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# Flexible production of coke using alternative coals – effects on coke properties under blast furnace conditions

(FLEXCOKE)

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**Flexible production of coke using alternative coals – effects on coke properties under blast furnace conditions (FLEXCOKE)**

European Commission
Directorate-General for Research and Innovation
Directorate D - Industrial Technologies
Unit D.4 — Coal and Steel
Contact Hervé Martin
E-mail RTD-PUBLICATIONS@ec.europa.eu

European Commission
B-1049 Brussels

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European Commission

# Research Fund for Coal and Steel

# Flexible production of coke using alternative coals – effects on coke properties under blast furnace conditions

## (FLEXCOKE)


Maria Lundgren
Lena Sundqvist Ökvist

**SWEREA MEFOS AB (MEFOS) -** Box 812, SE-971 28 Luleå, Sweden

Janaina Brum, Alexandra Hirsch

**THYSSENKRUPP STEEL EUROPE AG (tk SE) -** Kaiser-Wilhelm-Strasse 100, DE-47166 Duisburg, Germany

Dan Hedkvist, Ted Svensson, Petra Sundkvist

**SSAB EMEA AB (SSAB) -** SE-97188 Luleå, Sweden

Katarina Pein

**SSAB EMEA AB (SSAB) -** SE-613 80 Oxelösund, Sweden

Per Lagerwall, Jenny Hagemalm

**SSAB EMEA AB (SSAB) -** SE-97188 Luleå, Sweden

Nicklas Eklund, Anna Dahlstedt

**LUOSSAVAARA-KIIRUNAVAARA AB (LKAB) -** Varvsgatan 45, SE-97128 Luleå, Sweden

Ruth Poultney

**Tata Steel UK Limited (Tata Steel UK) -** Teesside Technology Centre, Grangetown, Middlesbrough, TS6 6US, UK



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**Final report**

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# 3 FINAL SUMMARY

## 3.1 WP1: Laboratory cokemaking

### Task 1.1 Trial programme design

Alternative coals and C-containing materials for possible use in technical-scale carbonisation tests at DMT have been listed. Selected samples of pet coke (petrol coke), thermal coals and biomass were tested in an innovative devolatilsation thermal gravimetric analysing (TGA) program at Tata. Tests gave average proximate analyses with wide variations in their ash, volatile matter (VM) and fixed C contents. The five thermal coals showed more variable behaviour than the pet cokes, reflecting their variable VM and ash contents. With its very high VM content, the devolatilisation of the torrefied grot is different from the other materials. In contrast, the Swedish wood charcoal and the low volatile (LV) anthracite were least affected by heating, despite the charcoal having 15% VM and the anthracite 5% VM in the TGA analyses. The different VM and ash contents affect the fixed C content. Biomass without heat treatment has lower C and ash content and the highest VM. When torrefied, the VM decreases, ash and fixed C content increases with higher treatment temperature (grot, medium and high temperature torrefied wood). Charcoal has been subjected to the highest temperature, thus has the lowest VM etc. of the biomass samples. As grot contains bark and needles, the CaO, alkali and P content in the ash make it less suitable than the other biomasses.

### Task 1.2 Technical- and pilot-scale coke production

Selected alternative coals and biomasses were added to coal blends and carbonised in DMT 10 kg retort (technical-scale) and in the 500 kg semi-industrial oven (pilot-scale) and the produced coke were characterised in WP2. The alternative materials in technical-scale cokemaking were two thermal coals, pet coke, anthracite and three types of biomasses. In DMT first trial in pilot scale one thermal coal, anthracite, pet coke and charcoal were tested, some of them with lesser amounts than in technical-scale. A second trial of five tests in DMT semi-industrial oven was made due to delay in production of torrefied wood (TW). Carbonisation tests were made with 2% and 3% TW medium temperature and some repeated tests were made. New base blend coking coals were sent to DMT prior the tests. The DMT carbonisation tests showed lower maximum internal gas pressure for all test blends compared to the reference test in 10 kg retort tests and in 500 kg semi-industrial oven.

In the first semi-industrial trial the maximum wall load was lower than reference base blend for almost all alternative materials except for additions of 10% thermal coal, 3% thermal coal+3% anthracite and 3% anthracite. In the retort tests, calculated wall load was lower than for the reference except for 5% addition of charcoal and TW high temperature. When using 3% addition of charcoal in the semi-industrial oven, the wall load was lower than the reference blend.

Adding anthracite to the blend improves coke yield while adding thermal coal decreases the yield. Charcoal addition to the blend does not seem to affect the yield.

The shrinkage of the semi-industrial trial coke was sufficient in all tests, meaning that the coke will be easily pushed out of the coke oven. All test blends can be used when considering the safety of the coke plant except for test blend No.2, with 10% thermal coal addition to the base blend, that showed a higher wall load than other blends. As SSAB coke plant oven walls are sensible to high wall pressure the amount of 10% addition may be too high for industrial tests.

### Task 1.3 Selection of coke for further testing

Based on the results from technical-scale carbonisation tests (retort) selection of blends to be carbonised in pilot scale (semi-industrial) were made. They were; Base blend and 10% addition of HV thermal coal and 5% addition of anthracite. The result in the semi-industrial first trial with 10% addition of thermal coal was worse than indicated from the results in the retort tests, therfore it was decided to make a new coke with 5% addition of the thermal coal instead. Due to the decrease in either mechanical strength and/or hot strength in the retort tests, three blends were tested again in semi-industrial oven but with 3% addition instead of 5%; base blend mixed with either charcoal, thermal coal + anthracite and thermal coal + pet coke.

Seven of the semi-industrial carbonised alternative coke was chosen to be tested in an actual blast furnace (BF) and charged into basket samples in LKAB Experimental BF (EBF®) during campaingn 31. Standard coke type normally used in the EBF® was also tested in some baskets.

The later conducted second semi-industrial trial, with addition of 2% and 3% TW medium temperature to the blend as well as repeated tests of 5% anthracite and charcoal addition were investigated in WP2. The TW coke samples and repeated charcoal tests (3% addition) were also tested as basket samples in the FLEXCOKE EBF® campaign 32.

## 3.2 WP2: Laboratory characterisation of coke relative blast furnace conditions

### Task 2.1 Physical testing of coke

Mean grain size particle size (MPS) of coke and the particle shape using optical particle analysing system (PartAn 3D) at tk SE were determined of coke gained DMT pilot-scale 500 kg semi-industrial oven. The addition of alternative C-containing materials to the base blend mostly resulted in smaller coke pieces. For all considered coke pieces with a determined grain size smaller than 90 mm the length was greater than the thickness (L/T ratio>1). The same trend was seen in gravimetric sieving for coke produced in the 1$^{st}$ industrial scale cokemaking trial at SSAB. In contrast, the MPS was not notably affected during the 2$^{nd}$ industrial carbonisation trial producing coke for the full scale trial at SSAB BF No.3. Comparing the two alternative coke types produced in the 1$^{st}$ SSAB industrial trial with similar blends carbonised in DMT 500 kg semi-industrial oven the MPS was lower for the industrial produced alternative coke types.

Adding inert alternative C-containing materials decreases the cold mechanical strength and increases the abrasion. 3% addition of charcoal or pet coke is the best option for good cold, mechanical strength; also a mix of 3% thermal coal and 3% pet coke or 5% thermal coal can be an option. Adding anthracite has worse effect but recovered slightly by mixing with pet coke. Biomass has low bulk density which affects the amount added by weight. 3% addition of charcoal gives quite good result compared to 5% addition. Similar trend is observed in 2% TW compared to 3% addition. Cold, mechanical strength of coke produced at SSAB coke plant showed good strength regarding both Micum 40 ($M_{40}$) and Micum 10 ($M_{10}$) for the 1$^{st}$ carbonisation trial with only slightly lower values than the standard coke. In the 2$^{nd}$ industrial trial the $M_{40}$ was in general even higher than the standard coke but then the resistance to abrasion was lower as $M_{10}$ was higher than the standard coke.

Owing to the limited availability and elevated cost for a high-quality coking coal, the utilisation of alternative C-containing material in the coking coal blend to produce coke that meets the BF requirements were investigated. However, the addition of tested alternative materials (thermal coal, anthracite, pet coke, TW and charcoal) decreases the MPS of the produced coke compared to the reference blend. In addition, the cold mechanical strength are affected by adding alternative C-containing material to the coking coal but using 5% thermal coal in full-scale cokemaking trials at SSAB resulted higher cold strength than expected from retort and semi-industrial tests.

For industrial application a stepwise approach in adding alternative C-containing materials is recommended. It can be assumed that the impact in terms of the tested materials may be visible on industrial coke production (smaller coke pieces) but the absolute reduction on MPS and cold mechanical strength can be different. As seen in the FLEXCOKE project the effect on particle size was different between the two campaigns and the effect of smaller particle size was not seen during the 2$^{nd}$ full-scale trial at SSAB coke plant. It is also unknown which specific requirements on coke particle size and mechanical strength the different European BF operators have.

### Task 2.2 Analysis of coke microstructure

Studies have been made in order to understand changes in coke microstructure in relation to conventional coke quality and coke behaviour under metallurgical treatment during laboratory tests and in coke taken from the inside of the BF. For this purpose tk SE used their microscopically texture measurement method that has been developed to quantify the anisotropic and isotropic carbon components of coke.

Samples of coke produced from normal blends and blends using alternative coal/C-containing material were analysed before and after treatment in the CSR (Coke Strength after Reaction)/CRI (Coke Reactivity index) test, in the innovative test method and in actual BF, as the EBF®. The microstructure from coke samples produced at DMT 10 kg retort and 500 kg semi-industrial oven

were investigated as well as coke produced at SSAB coke plant 1st trial. In addition basket samples from EBF® campaign 31 and 32 were investigated.

Although the additives behave distinct under polarized light the addition of alternative C-containing materials to the base blend mainly increased the coke isotropy. Generally coke produced in the semi-industrial oven and in industrial coke ovens at SSAB were more anisotropic compared to the coke produced in the 10 kg retort. The examined samples after EBF® trials showed differences in the content of isotropic- and anisotropic total; however no trend could be concluded. In contrast to this, all samples after CRI/CSR tests became more anisotropic.

Evaluation of coke gasification (changes in pore area/distribution) has been made by LKAB using optical microscope studies for samples from DMT. Thermal history (graphitisation, Lc) evaluation of selected samples as basket samples from the EBF® trial (WP4) has been performed. The results of graphitisation are reported in Task 3.2. The study of gasification behaviour by measuring pores could not reveal any clear differences between untreated samples and samples reacted in the BF shaft (EBF® basket samples) or in the BF simulating tests. The variation between the cokes itself may influence the results more than changes in pore area as a result of gasification.

The forming microstructure in coke is a result of the properties of each single coal and also of the coking parameters. Therefore the coal quality and mode of operation of a coke plant has strong impact on the ratio between anisotropic and isotropic components. The BF size has increased since the past and the rate of pulverised coal injection (PCI) has been maximised over the years. With increasing PCI rate, the amount of charged coke per ton hot metal has been decreased and its residence time in the BF increased, as the coke carbon is partly replaced by the carbon from the pulverised coal (PC). Therefore quality demand on BF coke especially on CSR has increased. A higher CSR is desired, to ensure the drainage for liquids phases and the gas flow as well the support for the burden. Coke with higher amount of isotropic phases tends to have lower CSR values since the isotropic phases react more intensively with $CO_2$ in the CRI/CSR test, reducing the CSR of the coke.

Although some alternative C-containing material behaves anisotropically (pet coke, anthracite, HV thermal coal with FSI) and some behaves isotropically (charcoal, TW at medium and high temperature, HV thermal coal no FSI) under polarized light, the coke texture of the produced coke showed that the addition of these C-containing materials to the base blend mainly increases the isotropic phases and decrease the anisotropic total at the same time. This means that the alternative C-containing material deteriorate the coke quality by increasing the coke reactivity (CRI) and decreasing the CSR. Therefore not only the investigation of the microstructure from single coals and the alternative C-containing material is important but also from the coal blend as the single materials interact and optical behaviour is significantly influenced.

**Task 2.3 Ash amount and composition**

The ash amount and its composition of samples gained from DMT tests and SSAB coking plant during the two full scale trials were determined. The evaluation results of treatment in real BF, as in the basket sample treatment at EBF® are reported in WP3, Task 3.2.

Ash content decreased when charcoal, TW, pet coke or anthracite was added to the base blend. Thermal coal significantly increases the ash content. This effect can be compensated by bringing a low ash component such as pet coke or biomass into the blend. The effect on ash content was also partly seen in the 1st industrial trial produced alternative Test Coke 2 (TC2) with 3% thermal coal + 3% pet coke to the blend, as the ash content was similar as in standard coke while alternative Test Coke 1 (TC1), with 5% thermal coal to the blend had higher ash content. In the 2nd industrial trial the standard coke had similar ash content and chemical composition as the FLEXCOKE trial coke (FTC). In this coke, another type of thermal coal with lower ash was used and compared to the 1st industrial trial, the FLEXCOKE trial mix had higher amounts of medium volatile (MV) coal in the mix and more of the high volatile (HV) coal was replaced by thermal coal, which contributes to the total ash content.

The C content decreased and the ash content increased for all tested alternative coke samples after reacting in the CRI/CSR test. Comparing with baskets samples reacting in the EBF®, reported in WP3, the change in ash content was more notable after the CRI/CSR test due to the stronger effect on solution loss reaction in pure $CO_2$. The addition of alternative C-containing material always increased CRI and consequently decreased the CSR. The highest impact was observed for the blends with the additions of thermal coal and TW.

BF coke is an indispensable and cost-intensive raw material for the hot metal production. The characterisation regarding to its chemical composition is very important for the burden projection, since the content of carbon and ash as well its composition are parameters which influence directly the coke consumption. Also addition of fluxes is affected and in turn the productivity and quality from hot metal and slag. The gained results showed that mainly the selected thermal coal as raw material for cokemaking has a negative impact on the coke quality as ash content increases lowering the amount of C in coke. This negative impact can be compensated by parallel addition of a very low ash material as e.g. pet coke or biomass. All other alternative C-containing material did not show significant negative impact on coke ash amount/composition. However, not only the ash amount should be considered as also the effects on other parameter as for example cold mechanical strength, CRI, CSR and particle size distribution are important.

**Task 2.4 Metallurgical testing**

In the BF the most demanding tasks of coke is to ensure the gas flow and the drainage of liquid phases in the cohesive zone. In order to fulfil all tasks during its residence time, ensuring an optimal BF performance, coke with high hot strength after reaction (CSR) and low reactivity index (CRI) is required. In accordance with the literature the optimum value is <30 % for CRI and >58 % for CSR. A low CSR value means more generation of fines, which disturbs the gas flow and the drainage of the liquid phases in the BF. CRI and CSR tests according to ISO (International Standard Organisation) standard are worldwide known coke properties and permits the quality comparison regarding those parameters. However there are many differences between ISO test conditions and those being in an industrial BF. Therefore a realistic coke characterisation is important for an accurate prediction of coke behaviour in the BF.

CRI and CSR were determined for coke samples produced at DMT 500 kg semi-industrial oven and for TC1 and TC2 produced in SSAB coking plant during the 1$^{st}$ industrial trial.

The addition of alternative C-containing material always increased the CRI and consequently decreased the CSR values compared to the reference coke, which was produced using only coking coals (Base blend: LV 40%; MV: 32% and HV: 28%). The influence of biomass on the resulting reactivity is different. The addition of charcoal has less impact on the resulting CRI than the addition of TW. The highest impact could be observed when 10% thermal coal or 3% TW at medium temperature was added to the base blend. Though different blend compositions (base blend + 5% thermal coal and base blend + 3% thermal coal + 3 % pet coke), the industrial test samples, TC1 and TC2 produced by SSAB, have very similar CRI and CSR, even though they have at different recipes (base blend + 5% thermal coal, TC1, and base blend + 3% thermal coal + 3% pet coke, TC2).

The coke gained in the industrial trial has a higher quality regarding CRI/CSR compared to DMT semi-industrial coke. Both alternative cokes produced at SSAB 1$^{st}$ industrial trial coke, TC1 and TC2, had the required quality regarding CRI and CSR values to be charged in an industrial blast furnace. It can be thus concluded that a pilot-scale test can only give hints to the resulting quality on an industrial scale, but is not be able to give detailed information on expected CRI/CSR. According to DMT experience scaling-up from the semi-industrial oven to industrial coke ovens of 6 m height are that the coke quality, as CRI, CSR and the cold mechanical strength, are expected to improve up to ~ 5 points. This can be confirmed when alternative coke with 5% addition of thermal coal to the blend was carbonised in SSAB 2$^{nd}$ industrial trial. The FTC coke variations in CRI and CSR were in the range of normal variations when producing standard coke.

For a more realistic coke characterisation, simulating conditions were adapted closer to a real BF. For this purpose a new, innovative test facility, erected at tk SE, was used. The tests were carried out with tk SE coke and with TC1 (base blend + 5% thermal coal) from the SSAB 1$^{st}$ industrial trial. For dimensioning the new, innovative CRI/CSR test facility several pre-tests were carried out. After commissioning of the facility several tests using coke samples with two different grain sizes and different sample weights and gas compositions were performed. Results were compared to standard CRI/CSR tests. To simulate the mechanical strength, a load was applied on the coke column inside the reaction vessel.

Adding other gas components (CO, $N_2$ and $H_2$) to the test gas ($CO_2$) decreases the CRI and consequently increases the CSR compared to standard tests. According to the pre-tests series at tk SE (before erecting the innovative test facility) the CO content increases and $CO_2$ decreases in the arising gas inside the reactor during the solution loss reaction.

Some tests parameters influence the reaction rate, such as the particle size, temperature and gas concentration. In contrast the temperature and gas concentration are directly proportional to kinetics of the reaction. The ISO standard heating time of 45 min is sufficient for heating the coarser coke sample, i.e. larger particle size, to the target test temperature in the innovative test facility. The coke grains degrade proportionally more using the ½ Micum drum compared to the ISO drum. The addition of CO, $N_2$ and $H_2$ to $CO_2$ gas in the CRI test for a realistic coke characterisation led to a significant decrease of coke reactivity, i.e. the ISO standard CRI is significant higher than the CRI gained when coke is tested in the innovative test under gas atmosphere closer to those at the BF. The application of 2.8 bar load during the solution loss reaction increased the reaction rate, but only little change on grain size distribution was observed.

The parameter for CRI/CSR tests should be adjusted in order to characterise coke under conditions closer to a real BF. A first design of a "new CRI/CSR standard" at tk SE has been made with a sample weight of 1-2 kg, a particle size of 35-80 mm, a reaction temperature of 1100 °C (120 min) in a gas flow of 25-50 l/min $CO_2$, CO, $N_2$ and $H_2$ gas. A load on the sample bed of 2.8 bar is applied. A new calculation of the load exercised on the coke layer in the BF will be carried out taking in account the reduction development of $Fe_2O_3$ to metallic Fe.

Investigation of coke gasification in a BF simulating test at LKAB (TRT Mas test) shows that the weight loss of the coke is generally low at low $CO_2$ content in the gas at gasification temperatures. Mixing pellets with coke have a positive effect on the pellet reduction degree as all selected coke type samples give a higher reduction than single pellet tests. Alternative coke with 3% thermal coal and 3% pet coke addition to the blend showed the best influence on pellet reduction followed by coke types with charcoal addition. Coke with charcoal addition also showed higher C conversion and lowest activation energy during TGA tests while base blend coke has showed the opposite.

Comparison of cokes reacted in BF simulating test, standard CSR/CRI test and in the EBF® (basket samples), shows that the weight losses in the actual BF are much lower than in the standard test as the magnitude of the $CRI_{EBF}$ is much lower supporting the theory that BF atmosphere, with less $CO_2$, contribute less to solution loss in this region of the shaft. This results show similar trends as in the innovative test regime at tk SE pointing towards that the tests of coke reactivity should be performed closer to BF atmosphere to give more realistic values.

## 3.3 WP3: Technical- and pilot-scale test of coke relative blast furnace conditions

Original and alternative test coke that meets stated coke plant and BF requirements were produced at SSAB and selected coke types were subjected to actual BF conditions in basket samples charged into the EBF® (Task 3.2). During operational trial in the EBF® (Task 4.2) two raceway core samples and a number of hearth core-drilling samples have been taken out and characterised. Basket samples from the EBF® have been analysed and compared with laboratory test regimes with respect to graphitisation and gasification considering impact from in-furnace BF conditions. The results of the comparison are found in Task 2.4.

### Task 3.1 High-temperature behaviour

During the EBF® trial, two raceway (RW) cores have been drilled during short stoppages, one with standard coke normally used in the EBF® and the other with operation of alternative TC2. The RW drilling-cores have been characterised accordingly to previous projects. The EBF® trial ended with operation with TC1 and basket samples were charged before the stoppage and quenching of the EBF®. After cooling, the EBF® was excavated and samples were collected in the shaft and high temperature areas as raceway. In addition, core-drillings have been made into the EBF® hearth and characterised in similar way as the RW cores. In total, a large number of solid samples have been collected during the EBF® trial and during the excavation. The evaluation of the solid samples gives information of changes in size, distribution of coke/hot metal droplets/slag, chemistry/ash of high temperature coke, graphitisation degree (historical temperature) etc.

Based on coke graphitisation degrees, which gives historical temperature estimations, and chemical compositions of the RW drilling-cores, shows appearance of a bird's nest in both coke types of reference coke and TC2 although the reference drilling-core has higher amount of metal flowing in this area. Excavation samples of TC1 from raceways showed also indications of bird's nests but the graphitisation degrees indicated lower temperatures than in the RW drilling-cores.

Results from core-drilling made in the hearth of the EBF® were similar to earlier results from campaigns when operating with standard coke. Samples beneath the three tuyers contain more fines and have higher bulk densities, a large amount of aggregate were found in the middle of the hearth. Most TC1 samples have higher mount of ash in the hearth compared to charged coke.

Tumbling tests for determined the mechanical strength have been made in the CSR/CRI standard tumbler (I-tumbler) of selected coke samples reacted in EBF®, with special attention to the high temperature zone. Samples collected in WP4 were evaluated in Task 2.1 and the results are reported in WP3 Task 3.2.

The hot strength in raceway sub-samples ($CSR_{EBFraceway}$) shows high values compared to coke reacting in the standard CRI/CSR test atmosphere of pure $CO_2$ gas. Both the reference coke and TC2 withstand the conditions that occur in raceway high temperature area quite well even though the temperature is much higher than in the standard test, on the other hand the gas composition is not as aggressive as pure $CO_2$. The coke that probably been inside the raceway has somewhat lower $CSR_{EBFraceway}$.

The mechanical strength of TC1 excavation samples ($CSR_{EBF}$) from the shaft and from the high temperature area shows a decrease in $CSR_{EBF}$ during the descent in the BF. The $CSR_{EBF}$ are around 88% in a shaft sample positioned higher up in the shaft (at 3.0 m from the top of the EBF®) and at around 71% further down below tuyere level (7.9 m from the top of the EBF®) where the temperature in the BF is much higher than in the shaft. CSR for the charged TC1 was on average 70.9% and it can be concluded that all excavated TC1 coke samples have a higher hot strength after reaction in a real BF than after the standard CSI/CSR test.

The conclusions made of the coke behaviour in the BF high temperature area give valuable information, not only for the alternative coke tested but in general. The hot mechanical strength of high temperature coke is an important finding and proves earlier theories and studies that the gas atmosphere in a real BF does not affect the CSR nearly as much as the standard test performed in 100% $CO_2$ gas atmosphere. The large amount of samples characterised in the hearth indicates that the TC1 behaves similar to the standard coke as the results were similar to previous EBF® campaigns and it is believed that TC1 is an option for full scale trial without negative impact.

**Task 3.2 Basket samples in the LKAB EBF®**

Two EBF® basket charging schemes have been made for tests in actual BF shaft. The major part of the basket investigation was done in EBF® campaign 31 (C31). Selected alternative cokes were prepared in baskets, charged into the EBF® and excavated, all activities made by LKAB. After opening and weighing the baskets, LKAB sent the samples to tk SE for further evaluation. In campaign 32 (C32), the baskets were charged with alternative test coke from the 1st industrial trial (TC1 and TC2), standard coke, 2% and 3% TW addition and 3% addition of charcoal from the second trial in the 500 kg semi-industrial oven at DMT. Evaluations were made by MEFOS.

Basket sample evaluations of reference (standard) and alternative coke tested in the EBF® C31 and C32 showed that in general the ash content is higher in samples found further down in the EBF® shaft and that the increase probably is due to up-take of oxides. In both campaigns a significant increase of the alkali content ($K_2O$ and $Na_2O$) in the ash of the recovered EBF® samples are seen due to the alkali recirculation phenomena. TC1 has lower up-take of alkali than both standard coke and TC2 at 6.3 m position and further down in the EBF®.

Bio-coke has a moderate up-take of alkali in lower levels, i.e. down to around 6 m, but showed a sharp elevation of alkali contents in the coke ash at 6.3 m from the top. However, this may be linked to the in-furnace conditions more than to the coke type.

In C31, operating with a centre gas flow, the graphitisation degrees (Lc) were enhanced in the recovered basket samples compared to the charged coke types. In contrast to C31 the coke graphitisation degrees in C32, operating with a flat gas profile, showed that the temperature in the EBF® shaft down to around 6.3 m had not exceed the coking temperatures of the test cokes, i.e. around 1000 °C.

The evaluation of basket samples in the EBF® is a valuable indicator of different raw materials behaviour in actual BF atmosphere, even though sometimes difficult to point out if the changes are due to operational conditions, vertical and horizontal position, effects of sintered material, gas channels or the actual material itself. In C32, the standard coke was placed in the same basket row as TC1 and TC2 in an attempt to rule out some of the difficulties mentioned. Therefore it seems like the TC2 takes up more alkali due to alkali re-circulation than TC1, which has even lower up-take than the standard coke.

## 3.4 WP4: Operational tests in coke plant and in BF

### Task 4.1 Industrial-scale cokemaking

SSAB EMEA in Luleå has conducted two industrial trials in their coke plant. The 1st trial was to produce two types of alternative coke to be used in the EBF® trial. Before the decision of which type of alternative coke to be used a meeting was held to discuss the results. As 10% addition of thermal coal showed high wall load there was a concern of the impact on SSAB coke oven walls as they are sensitive to high wall pressures. Using charcoal was initially planned but was later not considered due to the concern of safety when crushing as charcoal is much softer than coal and can create small dust particles. Therefore it was decided to test 5% addition of thermal coal to the blend and also addition of 3% thermal coal and 3% pet coke to the blend.

400 tonnes of coke with 5% thermal coal addition (TC1) and 400 tonnes of coke with 3% thermal coal and 3% pet coke addition (TC2) were produced separately from their standard mix. The coke were analysed with standard analyses by SSAB. Samples of both TC1 and TC2 were sent to tk SE for evaluation in WP2.

After the EBF® trial in WP4 a meeting were held to present and discuss operational results. A decision of which coke to be used in industrial full scale was made which was TC1. Therefore, the 2nd trial was to produce alternative coke with 5% thermal coal addition to the mix at the same time the coke was sent to SSAB BF No. 3 in Luleå (BF industrial trial in Task 4.3). The same thermal coal used in TC1 could not be purchased but a similar type was used with some small modification of the blend. The alternative coke produced in the 2nd trial and used in the full scale BF trial at SSAB is called FLEXCOKE trial coke (FTC). The FTC was initially mixed with the standard coke with ramping up with series at the coke plant and mixes at the BF, 20%, 40%, 80% and finally around three weeks with 100% FTC at both the coking plant and at the BF.

SSAB full scale cokemaking trials were successful. Monitoring the coke ovens with alternative C-containing material (5% addition of thermal coal and 3% thermal coal and 3% pet coke addition to the blend in the 1st trial showed no harmful effects and no significant deviations in process parameters. The same was observed in the 2nd trial when the process operated smoothly even when all coke ovens contained 100% FLEXCOKE mix. A data set was collected and evaluated with a multivariable data analysis (SIMCA). The model pointed out some important process parameters that were evaluated further but variations shown were considered as normal variations. However a longer trial period is needed to explore possible long term effects with respect to the relative short trial and reference periods evaluated. Also a longer trial period is needed rule out that the coke breeze is not increasing even though the operators did not see any increase in dust.

As no harmful effects on the battery, coke oven gas or the BF could be seen using 5% thermal coal in the coal mix SSAB will proceed outside the project with a new longer test period of 10 weeks. This longer trial period makes it possible to rule out any possible long term effects not seen in the shorter full scale industrial FLEXCOKE trial, also enable better monitoring of coke breeze, energy consumption etc.

### Task 4.2 Blast furnace operational trials at the LKAB EBF®

The EBF® trials were conducted by LKAB with support from MEFOS in November 1st to 11th 2016 testing TC1 and TC2. The trial was made at the end of the 32nd EBF® campaign (C32) and data from a preceding operating period with standard coke was used as reference. Most process parameters were set to typical values and kept more or less constant throughout the operating period. LKAB has collected operational data and evaluated the trial results in cooperation with MEFOS. A large number of solid samples were taken during the trial, at stoppages and during the

shaft excavation performed by LKAB. The hearth was detached and core-drillings were made by MEFOS. Solid sample evaluation have been made by MEFOS and analysis made by SSAB. BF operational data for both EBF® trial and full scale trial at SSAB BF No. 3 have been evaluated in a heat- and mass model (MASMOD) by MEFOS with support from LKAB and SSAB.

Heat- and mass modelling in MASMOD of the EBF® trial shows that similar C-consumption likely can be achieved independent on if using standard coke, TC1 or TC2. Differences found were related to variations in the alkali output as well as changes that cannot be linked to the coke type.

Investigation of off-gas dust showed that the amounts of flue dust were less during the trial with the alternative coke although the difference was not that high compared to standard coke. On the other hand sludge amounts were higher with alternative coke, especially with TC2, giving the highest total off-gas dust. As the sludge in previous studies has been linked to gasification of elements in the high temperature zone does this fact indicate differences in energy consuming reactions in the lower part of the EBF®. Reference A coke period shows highest alkali and silica in in the coarser, dry flue dust (kg/tHM) while TC1 period shows the lowest. In sludge the highest amounts of alkali, C and other oxides was found in TC2 period. XRD measurements showed similar compounds regardless of test coke period.

Evaluation of coke collected from the EBF® with the upper and lower shaft probes during operation showed no significant differences between the reference periods and alternative coke periods (TC2 and TC1). Variations seen are typical for probe samples seen in earlier investigations. The graphitisation degrees did not increase compared to charged coke indicating lower or similar temperature in the shaft than coking temperature (~1000 °C).

Excavation of TC1 from the EBF® shaft at different heights shows typical patterns of alkali recirculation and alkali up-take. When compared with excavation samples at similar positions after operation with standard coke the same trends are observed. Dissimilarities are attributed to difference in charging pattern and/or operational conditions rather that differences in the coke types itself.

According to the evolution of the EBF® trial and MASMOD modelling the C-consumption are not affected using either TC1 or TC2. The exploitation of the results was that the project group decided to use TC1, with 5% addition of thermal coal to the blend, in SSAB full scale operational trials in coke plant and BF No. 3 simultaneously.

### *Task 4.3 Blast furnace trial on industrial-scale*

In the full scale BF FLEXCOKE trial at SSAB BF No. 3 no negative effects by operating with the alternative coke with 5% thermal coal addition to the blend, FTC, could be found. The conclusion is based on MASMOD model evaluation and by observations made by BF operators during the trial.

The period with 100% FTC showed lower or comparable C-consumption than during reference periods with standard coke despite the use of more limestone when operating with 100% FTC and higher heat level compared to the second reference period, R2. The off-gas dust did not increase during the FLEXCOKE trial compared to reference operation. Sludge amount seem to decrease when operating with 100% FTC. In order to fully investigate the effects on the off-gas dust, C-consumption etc., a longer trial period is of advantage.

The main objective of "FLEXCOKE" project was to increase the flexibility using alternative carbon sources in safe cokemaking and to produce coke of a quality that meets the requirements for stable BF operation. Another aim was to reduce costs for hot metal production with similar/lower carbon input and $CO_2$ emission. With the results obtained the goals have been met.

As mentioned before, the use of the FLEXCOKE trial coke was very successful and no harmful effects on the coke plant and in the BF could be seen using 5% thermal coal in the coal blend. Coke quality data such as CSR/CRI and Micum showed that the coke strength and reactivity are only slightly affected. To fully investigate the effect of FLEXCOKE trial coke on coke plant and in BF operation a longer time frame of production and operation are needed, therefore a new test period of 10 weeks is planned to be tested at SSAB after the project. During the test period the stability of the "dead man" will be monitored as well as other parameters as the energy consumption, possibly losses to off-gas dust etc.

# 5 SCIENTIFIC AND TECHNICAL DESCRIPTION

## 5.1 Objectives of the project

The main objective of "FLEXCOKE" was to increase the flexibility using alternative carbon sources in safe cokemaking and produce coke of a quality that meets the requirements for stable BF operation. This is of importance with a global market characterised by limited availability and high costs for high quality coking coals. another aim was to reduce the costs for HM (hot metal) production with similar or lower C (carbon) input and $CO_2$ emission. The research linked alternative raw material for cokemaking to actual coke properties and to behaviour in BF operation. Development and use of innovative test methods in combination with samples from BF testing improved the classification of coke quality relative BF conditions.

By carrying out the activities within the work program following were reached, which contributed to the overall objective;

- Properties of alternative C-containing materials or coals of economic and technical interest for cokemaking were compiled based on available data and characteristic testing was carried out;
- Test method for innovative coke test regimes were developed and used aiming for a method with development of coke properties correlated with the actual BF;
- The effects of selected coal blend/C-containing material on cokemaking and coke properties in standard coke tests, innovative tests simulating the BF and in the actual BF were stated;
- Selection and modification of coal blend for industrial test coke production and BF testing based on properties of laboratory and technical scale produced coke and coking behaviour of the latter for further evaluation as
    - High temperature characteristics of industrially produced coke based on behaviour in BF and solid sample evaluation from LKAB EBF® raceway and hearth;
    - Operational test data from the coke plant, the EBF® and an industrial BF of SSAB.
- Solid sample evaluation of microstructure for comparative examination (EBF® samples) regarding development of coke properties during laboratory treatment;
- Evaluated test result from operational tests in industrial coke plant, the EBF® and industrial BF assessing effects on coke making process and stable BF operation linking the effects on cokemaking with the BF operational results via reached quality of coke.

## 5.3 Activities

**WP1: Laboratory cokemaking**

***Task 1.1 Trial programme design***

Planned activities and work accomplished

List of coals and alternative materials, such as petroleum coke (pet coke), coke fines, charcoal, biomass, plastics etc. for cokemaking including the basic characteristics, were collected and discussed based on the existing data, especially the data available from the previous RFCS projects[(1),(2)]. The studies have examined additional products, such as waste biomass, sawdust, torrefied biomass, coal-tar emulsion and liquids derived from waste plastics, however these projects did not investigate coke behaviour in the BF (e.g. coke gasification behaviour, effects of blast parameters on bosh and raceway coke properties, textural changes, etc.) and the impact on BF operation, which were the focus in FLEXCOKE. Although some data already exist in terms of the alternative C-containing material, there are some missing data for some raw materials. Thus the missing data are provided by additional characterisation using standard and innovative methods.

Description of activities, results and discussion

A list of alternative coals and C-containing material, such as biomass, for possible use in the project were collected and are presented in Table 1 and Table 2.

**Table 1: Proximate and analysis of coking abilities. VM= volatile matter, FSI=Free swelling index, HGI=Hardgrove index**

| Thermal coal No. | Moist. (%) | Ash (%) | VM (%) | S (%) | Cl (%) | FSI | HGI |
|---|---|---|---|---|---|---|---|
| 1 | 8 | 20.9 | 25 | 0.78 | | | |
| 2 | 7 | 18.9 | 26 | 0.79 | | | 52 |
| 3 | 7 | 15.6 | 27 | 0.57 | | | 55 |
| 4 | 3.8 | 8.5 | 39 | 0.63 | 0.015 | 8 | 53 |
| 5 | 9 | 14.7 | 33 | 0.40 | 0.003 | | 55 |
| 6 | 7 | 14.5 | 31 | 0.88 | 0.120 | 6 | 48 |
| 7 | 6 | 12.2 | 36 | 0.92 | 0.070 | 2.5 | 41 |
| **Anthracite** | **Moist. (%)** | **Ash (%)** | **VM (%)** | **S (%)** | **Cl (%)** | **FSI** | **HGI** |
| 1 | 2.5 | 11.3 | 3.0 | 0.29 | | | |
| **Pet coke No.** | **Moist. (%)** | **Ash (%)** | **VM (%)** | **S (%)** | **Cl (%)** | **FSI** | **HGI** |
| 1 | | 0.60 | 10.0 | 7.0 | | 0 | |
| 2 | 9.0 | 0.93 | 13.6 | 4.3 | 0.05 | | |
| 3 | 12.0 | 1.51 | 11.8 | 2.24 | <0.01 | | |
| 4 | 9.2 | 1.84 | 11.7 | 0.70 | 0.022 | | |
| 5 | 10.4 | 1.19 | 12.4 | 1.76 | <0.01 | | |
| 6 | 9.1 | 0.79 | 14.5 | 4.06 | <0.01 | | |
| 7 | 8.1 | 1.17 | 14.3 | 4.26 | <0.01 | | |
| 8 | 12.9 | 0.41 | 10.8 | 2.67 | <0.01 | | |
| **Biomass** | **Moist. (%)** | **Ash (%)** | **VM (%)** | **S (%)** | **Cl (%)** | **FSI** | **HGI** |
| Swedish Charcoal (BBQ) | 1.4 | 0.9 | 12 | 0.01 | | | |
| Torrefied grot* | 21 | 1.1 | 74 | 0.04 | | | |
| Swedish wood pellets (3) Wood | 9.4 | 0.6 | 84 | | | | |
| Swedish wood pellets (3) Grot* | 9.5 | 0.8 | 83 | | | | |
| Swedish wood pellets (3) Bark | 8.6 | 3.1 | 76 | | | | |
| Brazilian Charcoal (4) *Eucalyptus* | | 3.1 | 24 | | | | |
| Torrefied wood, medium | 4.2 | 0.6 | 40 | <0.01 | <0.015 | | |
| Torrefied wood, high | <0.1 | 0.9 | 18 | <0.01 | <0.015 | | |

*Grot=top and branches

**Table 2: Chemical composition of the ash (wt.%). Samples without references are tested at SSAB in Oxelösund**

| Thermal coal No. | CaO | MgO | $Na_2O$ | $K_2O$ | $Fe_2O_3$ | $SiO_2$ | $Al_2O_3$ | P |
|---|---|---|---|---|---|---|---|---|
| 1 | 7.8 | 1.9 | 0.13 | 0.60 | 5.70 | 48.0 | 26.5 | 0.54 |
| 2 | 7.0 | 1.6 | 0.17 | 0.60 | 4.14 | 46.8 | 29.8 | 0.77 |
| 3 | 7.3 | 1.3 | 0.11 | 0.54 | 3.69 | 48.2 | 28.9 | 0.82 |
| 4 | 9.4 | 3.7 | 2.72 | 1.89 | 11.1 | 44.6 | 14.9 | 0.09 |
| 5 | 2.9 | *1.6* | 0.97 | 1.68 | 5.70 | 58.6 | 23.8 | 0.32 |
| 6 | 0.87 | 0.99 | 0.29 | 2.64 | 4.21 | 60 | 28.4 | 0.06 |
| 7 | 0.77 | 0.84 | 0.20 | 2.42 | 3.48 | 58.7 | 30.7 | 0.04 |
| **Anthracite** | **CaO** | **MgO** | **$Na_2O$** | **$K_2O$** | **$Fe_2O_3$** | **$SiO_2$** | **$Al_2O_3$** | **P** |
| 1 | 1.2 | 1.1 | 0.40 | 2.59 | 6.67 | 61.2 | 23.6 | 0.10 |
| **Pet coke No.** | **CaO** | **MgO** | **$Na_2O$** | **$K_2O$** | **$Fe_2O_3$** | **$SiO_2$** | **$Al_2O_3$** | **P** |
| 1 | 7 | - | 0.25 | 0.002 | 12 | 4 | 4 | |
| 2, HOD 364 | 4.8 | 0.79 | 0.86 | 0.66 | 5.15 | 28.4 | 11.81 | 0.16 |
| 3, HOD 048 | 3.22 | 0.65 | 0.53 | 0.83 | 14.6 | 47.0 | 23.91 | 0.31 |
| 4, HOD 365 | 8.52 | 1.26 | 1.57 | 0.95 | 21.5 | 41.6 | 20.44 | 0.39 |
| 5, HOD 701 | 3.42 | 1.96 | 1.36 | 0.98 | 13.4 | 40.0 | 17.4 | 0.27 |
| 6, HOD 746 | 9.27 | 1.14 | 1.67 | 1.12 | 15.7 | 34.1 | 16.33 | 0.32 |
| 7, HOD 156 | 3.96 | 0.74 | 1.42 | 0.67 | 12.7 | 30.4 | 14.06 | 0.29 |
| 8, HOD 267 | 7.29 | 1.72 | 2.53 | 0.95 | 11.3 | 30.9 | 9.7 | 0.36 |
| **Biomass** | **CaO** | **MgO** | **$Na_2O$** | **$K_2O$** | **$Fe_2O_3$** | **$SiO_2$** | **$Al_2O_3$** | **P** |
| Swedish wood Charcoal (BBQ) | 28.9 | 8.1 | 0.56 | 9.53 | 3.12 | 20.1 | 9.41 | 0.35 |
| Torrefied grot* | 37.8 | 6.0 | 1.24 | 13.5 | 1.29 | 10.1 | 3.67 | 2.79 |
| Swedish wood pellets(3) Wood | 27.0 | 5.1 | 1.19 | 10.0 | 3.55 | 24.7 | 4.91 | 1.02 |
| Swedish wood pellets(3) Grot* | 33.8 | 5.7 | 1.38 | 10.1 | 1.68 | 17.1 | 2.22 | 1.49 |
| Swedish wood(3) Bark | 38.0 | 3.4 | 1.06 | 7.10 | 0.97 | 13.1 | 2.16 | 1.08 |
| Brazilian Charcoal(4) *Eucalyptus* | 54.2 | 10.7 | 1.7 | 12.7 | 5.7 | 2.1 | 1.20 | 3.06 |
| Torrefied/pyrolysed wood, medium | 44.7 | 11.3 | 0.46 | 9.87 | 0.98 | 8.9 | 2.02 | 1.25 |
| Torrefied/pyrolysed wood, high | 48.3 | 12.4 | 1.07 | 7.67 | 0.82 | 7.3 | 2.05 | 1.26 |

*Grot=top and branches

Table 1 shows proximate analysis based on references or information from coal suppliers and coking abilities tested by SSAB in Oxelösund. In Table 2, the chemical composition of the ash is presented. Seven of thermal coals re listed together with one type of anthracite and eight types of pet coke. Biomass is an interesting additive for the coal blend, as it is a $CO_2$ neutral material. Wood materials without pre-heating contain too much VM to be feasible for cokemaking and therefore are charcoal and torrefied to pyrolysed wood better alternatives.

Some samples were selected to be analysed further at Tata Steel UK in an innovative devolatilisation TGA test program; thermal coals No. 1 to 5, pet cokes No. 2 to 8, torrefied grot and Swedish charcoal, in Table 1 and Table 2. Alkalis ($K_2O$ and $Na_2O_3$), CaO, MgO and $Fe_2O_3$ in coke ash are known to enhance the coke gasification reaction ($C+CO_2 \rightleftarrows 2\ CO$), and therefore the reactivity (CRI) of coke. Low amounts of these elements in the coking mix are preferable. Biomass has generally a quite high amount of these elements, but the contribution to the overall coke ash is estimated to be lower than for the thermal coals due to the low ash content. In addition, for Swedish hot metal production, low phosphorous amount is required.

To support the material selection process for alternative coal and other C-containing material, the missing data of selected samples were provided by carrying out characterisation tests using a thermogravimetric analyser (TGA), shown in Figure 1, at Tata. Using the TGA, innovative programmed devolatilisation analyses were carried out selected samples. In the TGA, the coal analysis method was selected to control the furnace, as the biomass samples had been heat-treated.

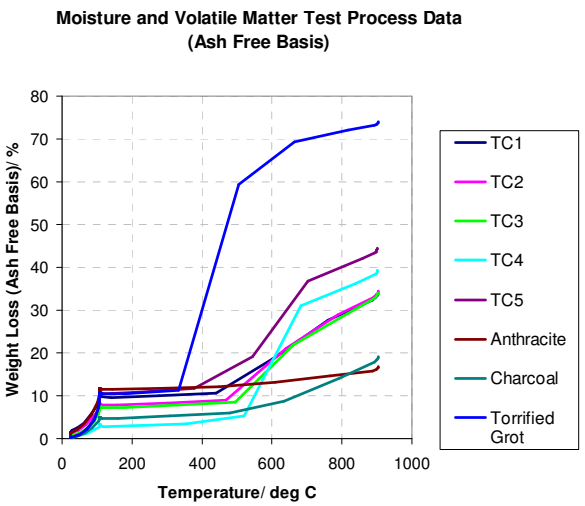
**Figure 1: Thermogravimetric analyser**

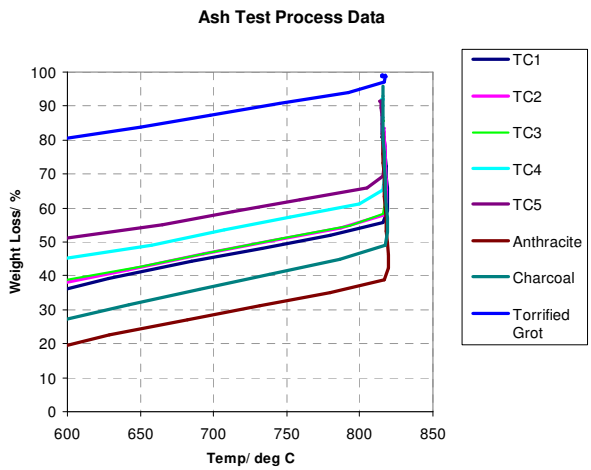
Duplicate, weighed samples of 1 g were run against standards for proximate analysis and a reference crucible was included in every run. The weight loss of each sample is monitored throughout the analysis and the percentage weight loss for each sample is collected automatically at the end of each analysis stage. The first stage is moisture analysis, which takes place in open crucibles. When it is complete, the furnace displays instructions to place lids on the crucibles for determination of volatile content. At the end of the volatile test at 900°C, the furnace displays instructions to remove the crucible lids. The furnace lid then closes automatically and heats up again from 600°C to determine ash content at 815°C (according to standard ISO 1171).

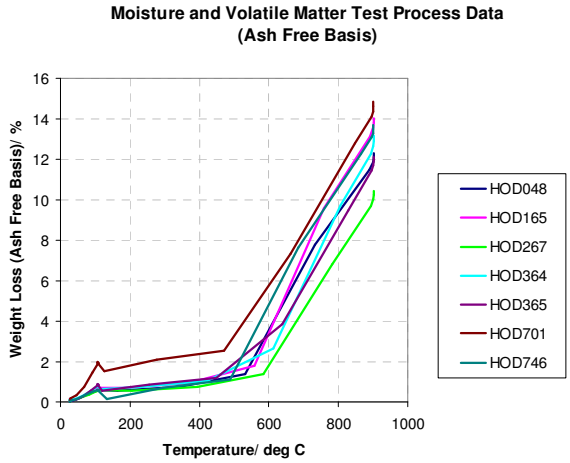
Trends were obtained for each material by plotting percentage weight loss against increase in temperature, as shown in Figure 2 and Figure 3. In order to make the results clearer, data for the petroleum cokes (labelled HOD) are shown separately from the other samples. The weight loss graphs up to 900°C have been calculated on an ash-free basis in Figure 2, so that the effects of temperature on the different materials can be observed. Figure 3 illustrates the weight losses during ashing.

Table 3 shows average proximate analyses for the selected samples with wide variations in their ash, VM and fixed carbon contents. The five thermal coals demonstrated more variable behaviour than the pet cokes, reflecting their variable VM and ash contents. With its very high VM content, the devolatilisation of the torrefied grot is very different from the other materials. In contrast, the Swedish wood charcoal and the low volatile anthracite were least affected by heating, despite the charcoal having 15% VM and the anthracite 5% VM in the analyses made by TGA. The very differing VM contents of the two torrefied biomasses are reflected in the weight loss graphs. They are similar up to 400°C, but above that temperature weight loss is much greater in the medium temperature sample, as expected. The seven pet cokes showed similar trends despite their VM

contents varying from 10.8-14.5%. The small weight gain in three samples after moisture was lost at 100°C was probably due to a minor error in the weighing system as the crucible lids were added.



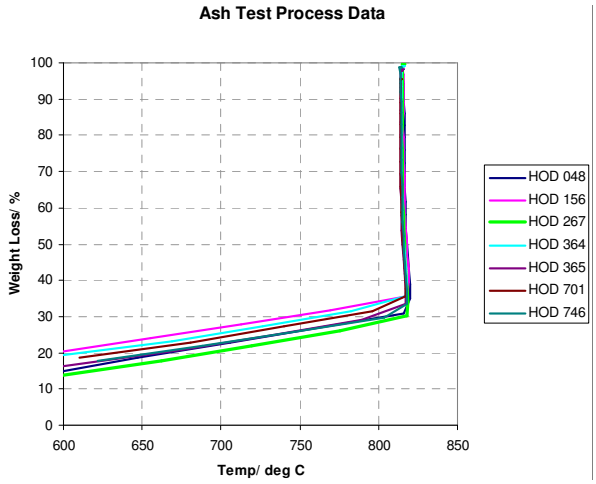




**Figure 2: Moisture and volatile matter weight loss as a function of temperature, ash-free basis**

**Figure 3: Weight loss on ashing vs: temperature for coals, biomass and petroleum cokes**

**Table 3: Proximate analyses of TGA samples**

| Sample | Moisture % | Ash % | VM % | Fixed C % | Ash % db | VM % db | Fixed C % db | VM % daf | Fixed C % daf |
|---|---|---|---|---|---|---|---|---|---|
| Thermal Coal 1 | 8.0 | 19.2 | 21.5 | 51.4 | 20.8 | 23.3 | 55.9 | 29.4 | 70.6 |
| Thermal Coal 2 | 6.7 | 16.6 | 24.3 | 52.3 | 17.8 | 26.1 | 56.1 | 31.7 | 68.3 |
| Thermal Coal 3 | 6.4 | 14.4 | 25.2 | 54.1 | 15.3 | 26.9 | 57.7 | 31.8 | 68.2 |
| Thermal Coal 4 | 2.9 | 7.8 | 36.9 | 52.4 | 8.1 | 38.0 | 54.0 | 41.3 | 58.7 |
| Thermal Coal 5 | 10.0 | 8.7 | 33.9 | 47.4 | 9.6 | 37.7 | 52.7 | 41.7 | 58.3 |
| Anthracite 1 | 10.5 | 9.3 | 5.1 | 75.1 | 10.4 | 5.7 | 83.9 | 6.3 | 93.7 |
| Swedish wood Charcoal BBQ | 4.7 | 3.4 | 15.2 | 76.7 | 3.5 | 16.0 | 80.5 | 16.6 | 83.4 |
| Torrefied Grot | 10.5 | 1.1 | 69.6 | 18.7 | 1.3 | 77.8 | 20.9 | 78.8 | 21.2 |
| Pet coke 2, HOD364 | 0.8 | 1.0 | 13.5 | 84.7 | 1.0 | 13.6 | 85.4 | 13.7 | 86.3 |
| Pet coke 3, HOD048 | 0.6 | 1.9 | 12.6 | 84.9 | 1.9 | 12.7 | 85.5 | 12.9 | 87.1 |
| Pet coke 4, HOD365 | 0.9 | 1.9 | 12.1 | 85.1 | 2.0 | 12.2 | 85.8 | 12.5 | 87.5 |
| Pet coke 5, HOD701 | 1.9 | 4.3 | 13.5 | 80.2 | 4.4 | 13.8 | 81.8 | 14.4 | 85.6 |
| Pet coke 6, HOD746 | 0.6 | 1.1 | 14.2 | 84.1 | 1.1 | 14.3 | 84.6 | 14.5 | 85.5 |
| Pet coke 7, HOD156 | 0.7 | 1.3 | 14.5 | 83.5 | 1.3 | 14.6 | 84.1 | 14.8 | 85.2 |
| Pet coke 8, HOD267 | 0.6 | 0.3 | 10.8 | 88.3 | 0.3 | 10.8 | 88.9 | 10.9 | 89.1 |

db = dry basis daf = dry-ash-free basis

Conclusions

The different VM and ash contents affect the fixed C content. Biomass without heat treatment has lower C and ash content and the highest VM. When torrefied, the VM decreases, ash and fixed C content increases with higher treatment temperature (grot, medium and high temperature TW). Charcoal has been subjected to the highest temperature, thus has the lowest VM etc. of the biomass samples. As Grot contains bark and needles, the CaO, alkali and P content in the ash make it less suitable than the other biomasses.

Exploitation and impact of the research results

These analyses have assisted determination of suitable candidates and their percentage amounts that could fit into the blend matrix for the technical-scale trial in Task 1.2.

### *Task 1.2 Technical- and pilot-scale coke production*

Planned activities and work accomplished

Selected alternative coal/C-containing material, based on the information in Task 1.1, and coal blends have been used in technical- and later pilot-scale production (i.e. DMT 10 kg retort and semi-industrial 500 kg oven) of coke for stating coking behaviour for industrial coke production in WP4 Task 4.1 and produce coke for further testing relative to BF conditions in laboratory tests (WP 2) as well as charged as basket samples (WP3 Task 3.2). Retort trials are used for selection of feasible blends relative to BF behaviour and cokemaking before scaling up to semi-industrial trials. In the first semi-industrial trial 10 test cokes were produced. It was planned to use torrefied wood, medium temperature, as an addition to the base blend but the manufacturing of the biomaterial was delayed and therefore, a second trial were made later. Due to the time elapsed from the previous test trial fresh coking coals were sent to DMT.

Description of activities, results and discussion

**Carbonisation tests in DMT 10 kg retort (technical-scale)**

A test plan set-up for the initial technical-scale carbonisation trials have been made, see Table 4. Coking coals of low, medium and high VM were mixed with either thermal coal No. 4 or 5 (see Table 1 and Table 2 or with pet coke 1, anthracite, charcoal and torrefied wood (medium and high). The carbonisation tests were carried out by DMT in their small-scale retort (10-11 kg) and coke was delivered to tk SE and to MEFOS for evaluation in WP2. Figure 4 shows the biomass used in the carbonisation tests.

(a) (b) (c)

**Figure 4: Biomass material used in retort (technical-scale) carbonisation trials. Charcoal (a), torrefied wood high temperature (b) and torrefied wood medium temperature (c)**

The alternative coals/C-containing material were crushed separate and added to the coking coals according to the blending test parameters (moisture 9%, wet bulk density 800 kg/m$^3$, particle size in blend 18-20% >3.15 mm and 30-34% <0.5 mm) and blended. The total blends were then crushed and the particle size distribution determined by sieve analyses. Moisture content was measured on the blend and adjusted to the desired value of 9 % during blending. The mixed coal according to Table 4 was filled into the steel tube and placed into the carbonisation furnace.

**Table 4: Test set-up for retort carbonisation tests at DMT. LV, MV, HV = low, medium and high volatile matter. TW = Torrefied wood**

| No. | "Coal" Blend % | LV coal | MV coal | HV coal | HV Thermal coal high FSI | HV thermal coal no FSI | Pet coke | Anthracite | Char coal | TW, high temp. | TW, medium temp. | Tot. |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **1** | Base Blend | 40 | 32 | 28 | | | | | | | | 100 |
| **2** | Base Blend + HV thermal coal high FSI | 40 | 25 | 25 | 10 | | | | | | | 100 |
| **3** | Base Blend + HV thermal coal no FSI | 40 | 25 | 25 | | 10 | | | | | | 100 |
| **4** | Base Blend + Anthracite | 40 | 30 | 25 | | | | 5 | | | | 100 |
| **5** | Base Blend + Charcoal | 40 | 30 | 25 | | | | | 5 | | | 100 |
| **6** | Base Blend + TW, high temp. | 40 | 30 | 25 | | | | | | 5 | | 100 |
| **7** | Base Blend + TW, medium temp. | 40 | 30 | 25 | | | | | | | 5 | 100 |
| **8** | Base Blend + HV thermal coal no FSI + Anthracite | 40 | 25 | 25 | | 5 | | 5 | | | | 100 |
| **9** | Base Blend + HV thermal coal No FSI + Pet coke | 40 | 25 | 25 | | 5 | 5 | | | | | 100 |
| **10** | Base Blend + HV thermal coal no FSI + Charcoal | 40 | 25 | 25 | | 5 | | | 5 | | | 100 |

The layout of the retort is shown in Figure 5 and the schematic layout of the test is shown in Figure 6. The height and diameter of coal bed are 505 mm and 180 mm, respectively and the exact weight of the charge depends on the desired density of coal blend. The final furnace temperature reached after approximately 4 hours is 1030 °C.

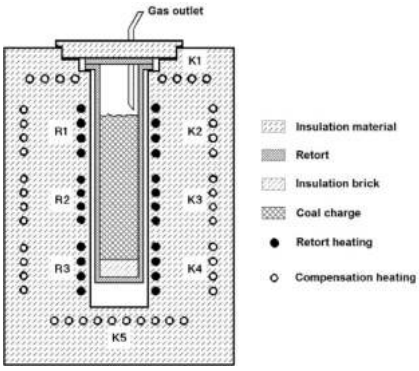



**Figure 5: Layout of the retort furnace [5]**

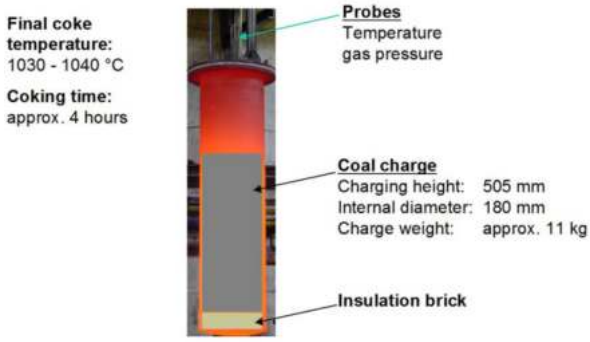



**Figure 6: Schematic layout of carbonisation tests [5]**

During the test the temperature and gas pressure is registered by a thermocouple and a pressure sensor that is located in the centre of the coal bed. The thermocouple and pressure sensor passes through the lid and perforated plate. The perforated plate is positioned above the bed; there is room for expansion of the coal blend and no force on the coke bed applied by the plate. When the coal is heated up the moisture and volatiles are released at the same time as some liquid phase is formed and blows up to a balloon and the internal gas pressure (IGP) is measured. The value of the measured IGP is influenced on the sustainability of the balloon of softening/liquid phase. If the balloon is disrupted the gas can leave and the internal pressure is lowered, if it sustains the gas is kept inside and a higher IGP is measured.

After conducted carbonisation tests the coke is allowed to cool in the steel tube before pushing it out [5]. The pieces are in maximum 90 mm in length and between 30 and 60 mm in width due to

the diameter of the retort. Before testing for mechanical strength and conducting CSR/CRI tests the coke is stabilised by dropping it 4 m plus additional 2 m. 5 kg of >40 mm coke that has been screened for particle size distribution is tumbled in the ISO drum for mechanical strength and then screened a second time. Factors have been determined for the relationship between ISO, MICUM, and JIS test results, respectively, for 50 kg coke samples produced in the semi-industrial coke oven and the smaller 5 kg coke sample from the retort tests from the same coking coal blend tumbled according to the ISO standard ISO 556 [(5)].

Coke from the retort has different particle size distribution than such from the semi-industrial oven. Due to the limited amount of coke available from the retort test the coke subjected to ISO drum test for mechanical strength is used for the CSR/CRI test. Before using already tumbled material as a routine DMT has made parallel tests in which tumbled and non-tumbled coke from the retort trials were compared. It was found that the material treated according to the $I_{40}$ tests could be used also in CSR/CRI tests without impact on the test result [(5)].

The results from analysing the produced alternative coke are presented in WP2. Physical testing, as cold mechanical strength, is presented in Task. 2.1, the results of coke microstructure are reported in Task.2.2, ash analysis is found in Task 2.3 and CRI/CSR results in Task 2.4 (Metallurgical testing).

*Coal blend and carbonisation test results*

The sieving results in fractions larger than 3.15 mm and less than 2.0 and 0.5 mm are shown in Table 5. The tests with anthracite as additive are diverging with lower amount in >3.15 mm and higher amount of fraction <2.0 mm due to different crushing instructions for anthracite particle size chosen to be comparable to the jaw crusher at SSAB coke plant.

**Table 5: Sieve analysis results for each coal blend (wt.%)**

| **Coal blend** | **Sieve analysis** | | |
|---|---|---|---|
| **No.** | **> 3.15 mm** | **< 2.0 mm** | **< 0.5 mm** |
| 1 | 20.5 | 65.2 | 29.0 |
| 2 | 17.4 | 71.0 | 36.6 |
| 3 | 18.0 | 65.7 | 28.3 |
| 4 | 12.6 | 76.7 | 34.5 |
| 5 | 20.7 | 64.2 | 32.3 |
| 6 | 21.4 | 67.3 | 31.6 |
| 7 | 20.5 | 65.8 | 33.5 |
| 8 | 12.8 | 74.2 | 31.9 |
| 9 | 18.8 | 66.7 | 31.7 |
| 10 | 19.9 | 65.9 | 33.2 |

From the measured maximum IGP shown in Table 6, the possibly internal gas pressure in the semi-industrial oven is estimated based on internal pressure measurements in the retort and semi-industrial coking oven for similar coal blends. Also the possible wall load is estimated. As can be seen from Table 6, using the HV thermal coal with no FSI reduces the wall load except when also pet coke is added to the mix. However, all test blends, with exception of high temperature torrefied wood and charcoal as additive, lowers the estimated wall load compared to the reference case.

The coke yield of each carbonisation tests are shown in Figure 7. Adding anthracite to the blend improves while adding thermal coal decreases the yield. Using charcoal or more highly torrefied/pyrolysed wood seems to not affect the coke yield in these tests. However, adding torrefied wood medium temperature decreases the coke yield due to its higher VM content.

**Table 6: Measured and calculated gas pressures and estimated wall load during carbonisation in the retort**

| Coal blend/ Test No. | Measured Max. Internal gas pressure, IGP (mbar) | Calculated * Gas Pressure (mbar) | Calculated * Wall load (mbar) |
|---|---|---|---|
| 1 | 104 | 30 | 14 |
| 2 | 67 | 22 | 10 |
| 3 | 16 | 16 | 7 |
| 4 | 71 | 22 | 10 |
| 5 | 44 | 34 | 16 |
| 6 | 32 | 33 | 15 |
| 7 | 38 | 17 | 8 |
| 8 | 18 | 27 | 12 |
| 9 | 52 | 13 | 6 |
| 10 | 12 | 12 | 6 |

* Calculation results are expected values that would be most likely obtained in the DMT 500 kg semi-industrial coke oven with movable walls

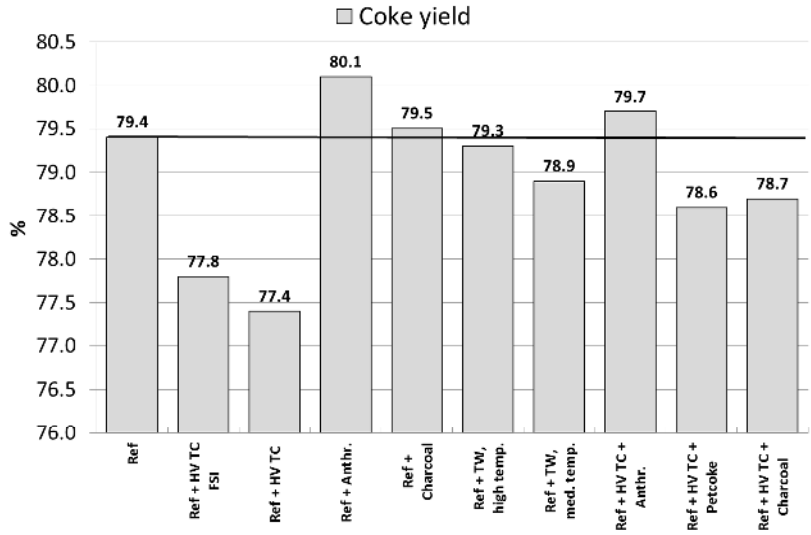



**Figure 7: Coke yield in retort carbonisation test**

**Carbonisation tests in DMT 500 kg semi-industrial oven (pilot-scale)**

SSAB performed trials in the DMT semi-industrial furnace with a movable wall (500–1000 kg charge of coal) with additions to the coal blend of alternative materials selected from the results of technical-scale characterisation and testing. The semi-industrial oven trials are important to predict the production conditions during carbonisation in the coking plant, but also for monitoring of the expected coke behaviour and coke quality tested in WP2.

Selected blends from technical-scale carbonisation tests to be carbonised at semi-industrial scale were: Base blend as reference, Base blend and 10% addition of HV thermal coal no FSI (No. 3 in technical-scale tests) and 5% addition of anthracite (No. 4 in retort tests). The test set-up is shown in Table 7. Due to the decrease in either mechanical strength and/or CSR/CRI in technical-scale, three blends were tested again in pilot-scale but with 3% addition instead of 5%; Base blend mixed with charcoal, thermal coal + anthracite and thermal coal + pet coke.

The results from Test No. 2, 10% addition of thermal coal, was surprisingly worse that indicated from the results in the retort tests so it was decided to make a new test coke with 5% addition of the thermal coal instead, test No. 2A. Also, as seen in Table 7, a number of new mixes with 3% addition were carbonised (thermal coal, anthracite and pet coke + charcoal). Total 10 carbonisation tests were performed during the first semi-industrial trial.

As mentioned earlier, it was planned to use torrefied wood, medium temperature, as an addition to the base blend but the manufacturing of the biomass was delayed and therefore, a second semi-industrial trial were made later. The test set-up is shown in Table 8. Due to the time elapsed from the previous test trial new fresh coking coals were sent to DMT and a new test with the base blend planned. The torrefied wood was tested with two levels of addition to the mix, 3% and 2%. As the

results with anthracite addition showed deviating results in wall load a new test with 5% anthracite was made along with a repeated test with 3% charcoal addition and the results are compared with results in the first semi-industrial trial. The torrefied wood medium temperature was in this case prepared from finer wood particles (saw dust), as seen in Figure 8, compared to the torrefied wood in the retort tests.

**Table 7: Set-up for the first semi-industrial (pilot-scale) trial at DMT. LV, MV, HV = low, medium and high volatile matter. TW = Torrefied wood**

| No. | "Coal" Blend % | LV coal | MV coal | HV coal | HV thermal coal no FSI | Pet coke | Anthracite | Char coal | TW wood medium | Tot |
|---|---|---|---|---|---|---|---|---|---|---|
| **1** | Base Blend | 40 | 32 | 28 | | | | | | 100 |
| **2** | Base Blend + HV thermal coal no FSI | 40 | 25 | 25 | 10 | | | | | 100 |
| **2A** | Base Blend + HV thermal coal no FSI, lower content | 40 | 30 | 25 | 5 | | | | | 100 |
| **3** | Base Blend + 5% Anthracite | 40 | 30 | 25 | | | 5 | | | 100 |
| **4** | Base Blend + Charcoal | 40 | 30 | 27 | | | | 3 | | 100 |
| **5** | Base Blend + Pet coke + Charcoal | 40 | 29 | 25 | | 3 | | 3 | | 100 |
| **7** | Base Blend + HV thermal coal no FSI + Pet coke | 40 | 29 | 25 | 3 | 3 | | | | 100 |
| **8** | Base Blend + HV thermal coal no FSI + Anthracite | 40 | 29 | 25 | 3 | | 3 | | | 100 |
| **9** | Base Blend + 3% HV thermal coal no FSI | 40 | 30 | 27 | 3 | | | | | 100 |
| **10** | Base Blend + 3 % Anthracite | 40 | 30 | 27 | | | 3 | | | 100 |

**Table 8: Set-up for the second semi-industrial (pilot-scale) trial at DMT. LV, MV, HV = low, medium and high volatile matter. TW = Torrefied wood**

| No. | "Coal" Blend % | LV coal | MV coal | HV coal | HV thermal coal no FSI | Pet coke | Anthracite | Char coal | TW medium Temp. | Tot |
|---|---|---|---|---|---|---|---|---|---|---|
| I | Base Blend | 40 | 32 | 28 | | | | | | 100 |
| II | Base Blend + TW, medium temp. | 40 | 30 | 27 | | | | | 3 | 100 |
| III | Base Blend + TW, medium temp. | 40 | 31 | 27 | | | | | 2 | 100 |
| IV | Base Blend + 5% Anthracite | 40 | 30 | 25 | | | 5 | | | 100 |
| V | Base Blend + 3% Charcoal | 40 | 30 | 27 | | | | 3 | | 100 |

**Figure 8: Torrefied wood in the second semi-industrial trial at DMT**

The set test parameters and conditions for both trials are listed in Table 9. During the tests the wall pressure force and internal gas pressure (IGP) is recorded. The horizontal shrinkage is measured automatically and shrinkage measurements are made manually at the end of the test.

**Table 9: Set test parameters for carbonisation tests at the DMT 500 kg semi-industrial oven with movable walls**

| Moist. | Bulk density (wet) | Bulk density (dry) | >3.15 mm in blend | <0.5 mm in blend | Coking time | Oven width | Heating wall temp. | Final coke temp. |
|---|---|---|---|---|---|---|---|---|
| 9 % | 800 kg/m³ | ~728 kg/m³ | 18-20% | 30-34% | 17.5 h | 0.45 m | 1220 °C | 1050 °C |

Anthracite particle size: Around 6% above 3.15 mm and 45 % less than 0.5 mm

*Coal blend and carbonisation test results*

Proximate analysis of alternative coal and C-containing materials for both trials are shown in Table 10. The results deviates slightly compared to Table 1 depending on laboratory and test methods. The proximate analyses of the blends in the first trial are shown in Table 11. The blends 2A (3% thermal coal addition) and 5 (3% charcoal and 3% pet coke) has slightly lower ash contents. Somewhat lower VM are noted for blend 3 (5% anthracite). The torrefied wood has almost double the amount of WM compared to charcoal and also lower ash content. The major difference for the alternative material between the first and the second trial are the ash content of anthracite, i.e. 2.8 % lower for the second trial.

**Table 10: Proximate analyse of alternative coal and C-containing materials**

| | **Moisture, air dried %** | **Ash, dry basis %** | **Volatile Matter, dry basis %** | **Volatile Matter, dry and ash free %** |
|---|---|---|---|---|
| **HV thermal coal no FSI** | 6.7 | 8.3 | 40 | 43.6 |
| **Pet coke** | 0.8 | 0.6 | 9.7 | 9.8 |
| **Anthracite, first trial** | 6.5 | 12.6 | 2.7 | 3.1 |
| **Anthracite, second trial** | 8.0 | 9.8 | 2.5 | 2.8 |
| **Charcoal, first trial** | 5.4 | 2.4 | 19.1 | 19.6 |
| **Charcoal, second trial** | 5.1 | 2.6 | 19.1 | 19.6 |
| **TW medium temp.** | 3.1 | 0.7 | 49.0 | 49.4 |

**Table 11: Proximate analyse of blends, first semi-industrial trial**

| **Test No.** | | **1** | **2** | **2A** | **3** | **4** | **5** | **7** | **8** | **9** | **10** |
|---|---|---|---|---|---|---|---|---|---|---|---|
| Moisture air dried | % | 1.8 | 1.4 | 1.7 | 1.4 | 1.7 | 1.4 | 1.6 | 2 | 1.7 | 1.8 |
| Ash dry basis | % | 9.3 | 9 | 8.5 | 9.1 | 9 | 8.4 | 8.9 | 8.9 | 9.4 | 9.4 |
| Volatile Matter dry basis | % | 23.7 | 24.3 | 24.1 | 22.8 | 23.7 | 23.3 | 23.5 | 23.9 | 24.2 | 23.4 |
| Volatile Matter dry and ash free | % | 26.1 | 26.7 | 26.3 | 25.1 | 26 | 25.4 | 25.8 | 26.2 | 26.7 | 25.8 |

In Table 12 the proxy analyses of the second trial are shown in comparison with same blend composition of the first trial. The VM of base blend is slightly higher for the second trial compared to the first trial that may influence the results.

**Table 12: Proximate analyse in the second semi-industrial trial with comparison of similar alternative blend in the first semi-industrial trial**

| **Test No.** | | **1** | **I** | **II** | **III** | **3** | **IV** | **4** | **V** |
|---|---|---|---|---|---|---|---|---|---|
| *Blend* | | *Base Blend (BB)* | | *BB+3% TW* | *BB+2% TW* | *BB+5% anthracite* | | *BB+3% charcoal* | |
| Moisture air dried | % | 1.8 | 1.1 | 1.5 | 1.2 | 1.4 | 1.9 | 1.7 | 1.5 |
| Ash dry basis | % | 9.3 | 9.2 | 8.7 | 8.8 | 9.1 | 9.2 | 9.0 | 9.1 |
| Volatile Matter dry basis | % | 23.7 | 24.2 | 24.4 | 24.3 | 22.8 | 22.6 | 23.7 | 23.2 |
| Volatile Matter dry and ash free | % | 26.1 | 26.6 | 26.7 | 26.6 | 25.1 | 24.9 | 26 | 25.5 |

The coal blend is sieved in fractions >10 mm, 10-7 mm, 7-5 mm, 5-3.15 mm, 3.15 -2 mm, 2-1 mm, 1-0.5 mm, 0.5-0.315 mm, 0.315-0.12 mm and 0.12-0.063 mm as seen in Figure 9 showing the particle size distribution of the first semi-industrial trial blends. Blend 2 with 10% addition of thermal coal deviates showing higher amount of larger particles. The reference blend 1 (base blend) has also a bit higher amount of larger particles in fractions 10-5 mm and lower amount in fraction 2-1 mm.

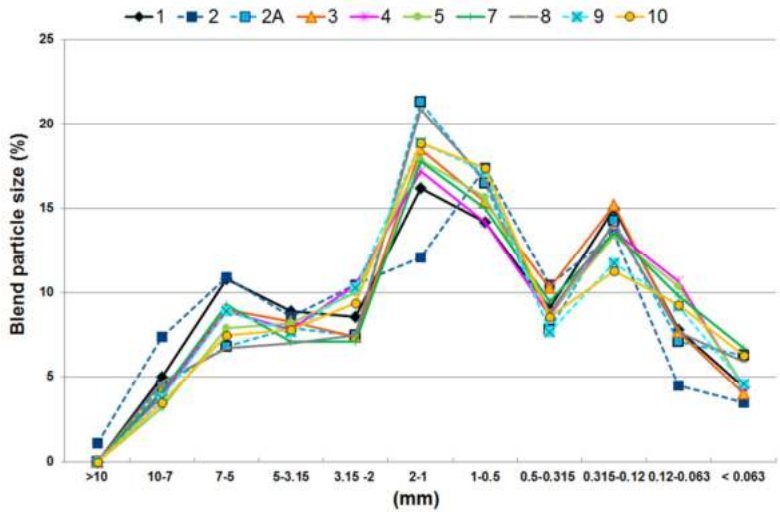



**Figure 9: Particle size distribution of the test blends in the first semi-industrial trial**

The coal blend particle size distribution of the same fractions are shown in Figure 10 blends with 3% (II) and 2% (III) addition of torrefied wood compared to the reference blend. In Figure 11, comparisons of repeated tests are plotted. Major differences are the particle size distribution for the Base blends (reference). The target size range of >3.15 mm is on a higher scale for Test trial 1 compared to additional test trial I. On the other hand, size <2.0 mm is higher for additional test I than for Test trial 1.

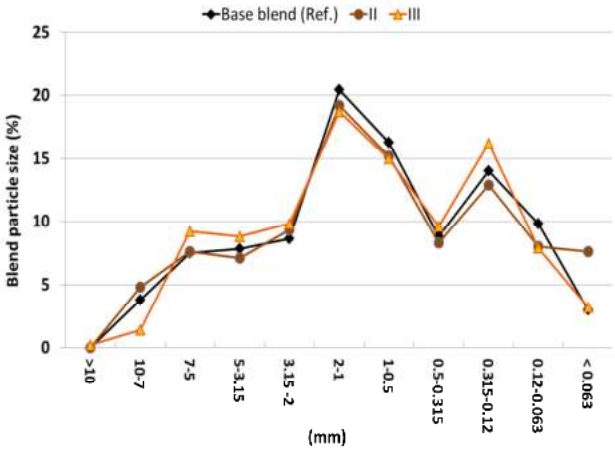



**Figure 10: Particle size distribution in blends with torrefied wood medium temperature, second trial**

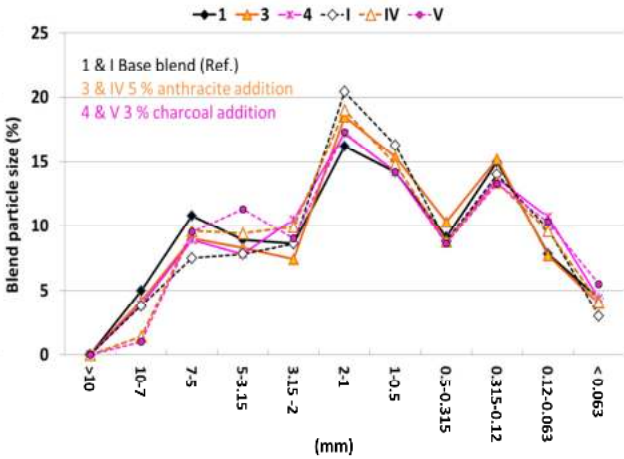



**Figure 11: Particle size distribution of the first trial compared to second trial blends**

*Pilot-scale (500 kg semi-industrial oven) carbonisation test results*

The carbonisation results for the semi-industrial first and second trials are listed in overview tables in Appendix 1 and 2, respectively.

As in the retort tests, the maximum internal gas pressure (IGP) are lower for all blends in the first trial compared to the reference Test No. 1 as seen in Figure 12. However, the maximum wall load exceeds the reference base blend in tests numbers 2, 8 and 10 with addition of 10% thermal coal, 3% thermal coal + 3% anthracite and 3% anthracite. As anthracite is expected to lower the wall load due to being inert during carbonisation it is not expected that 3% addition of anthracite shall give higher maximum wall load than the reference blend and it suggests an error in the wall load measurement for this sample. As expected, 5% anthracite addition to the mix (Test No. 3) showed lower maximum wall load than the reference blend.

A comparison of the maximum IGP and wall load for repeated tests are shown in Figure 13. The repeated test in the second trial shows notable lower values for both the maximum IGP and wall load. This influences the results for the repeated tests of 5% anthracite and 3% charcoal addition as they also have lower values than the first trial. The torrefied wood shows a bit higher IGP and

wall load but is well below the range of concern. The somewhat higher VM of additional test base blend may be the reason behind the lower maximum IGP and wall load.

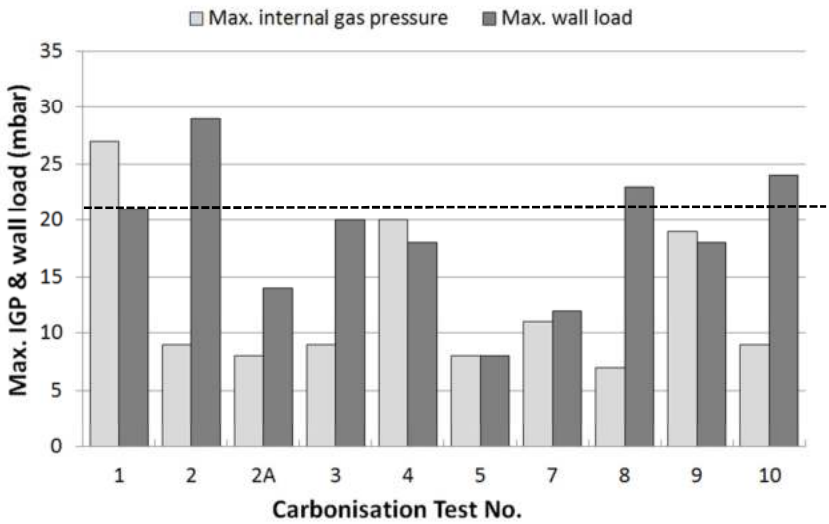



**Figure 12: Maximum internal gas pressure (IGP) and wall load, first trial**

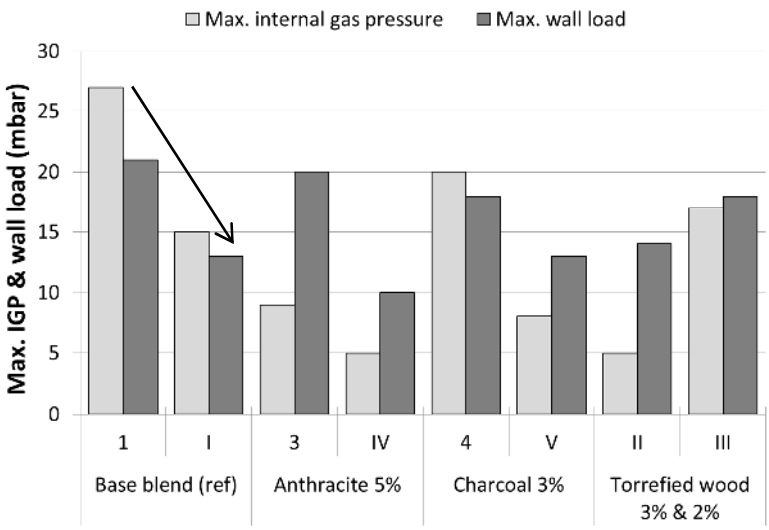



**Figure 13: Maximum internal gas pressure (IGP) and wall load, second trial compared with repeated tests from the first trial (No. 1, 3 and 4)**

The shrinkage of the coke was sufficient for all tests meaning that the coke will be easily pushed out of the coke oven. Looking at the final values in Appendix 1, four samples have lower shrinkage than the base blend, No. 4 (charcoal addition), No. 5 (charcoal and pet coke addition), No. 7 (3% thermal coal and 3% pet coke addition) and No. 9 (3% thermal coal addition). The lateral shrinkage during the tests are shown in Figure 14. Test No.s 4 and 9, and No. 7 to some extent, showed a time period with low or no shrinkage which differ from the other curves. Test 5 had faster and higher shrinkage than the base blend until the last hours of carbonisation, where the shrinkage is levelled out. The results from torrefied wood addition (second trial) and a comparison of the repeated tests (first and second trial) are presented in Figure 15. The new base blend (reference) used in the second trial showed 3.8 mm less shrinkage than in the first trial. However, the shrinkage of the coke was sufficient for all tests meaning that the coke will be easily pushed out of the coke oven.

The carbonisation rates are varying between 2.4 to 3.4 °C per minute during both trials. The coke yields for the first trial are shown in Figure 16. Anthracite and charcoal elevates coke yield while thermal coal and pet coke addition gives lower yield compared to the base blend. The coke yields in the second trial torrefied wood addition and a comparison for the repeated tests are shown in Figure 17. As in the first trial, anthracite and charcoal elevates coke yield. Torrefied wood, with its high content of volatile matter, decreases the coke yield.

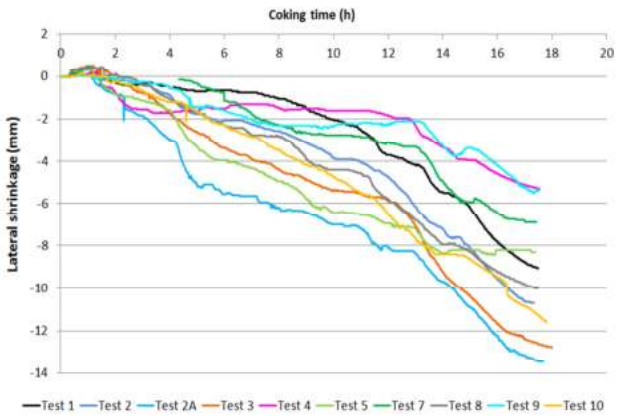



**Figure 14: Lateral shrinkage (mm) of the produced coke, first trial**

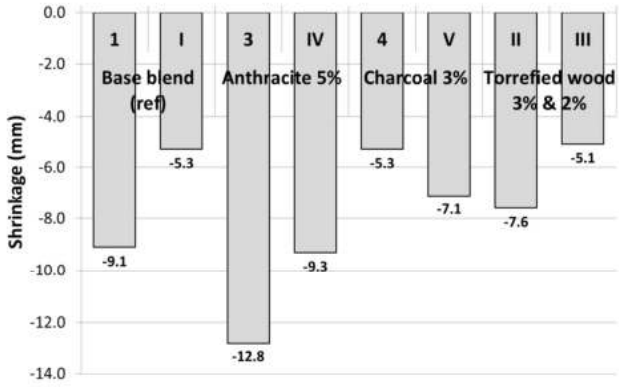



**Figure 15: Shrinkage (mm), comparison of second trial compared with repeated tests from the first trial (No. 1, 3 and 4)**

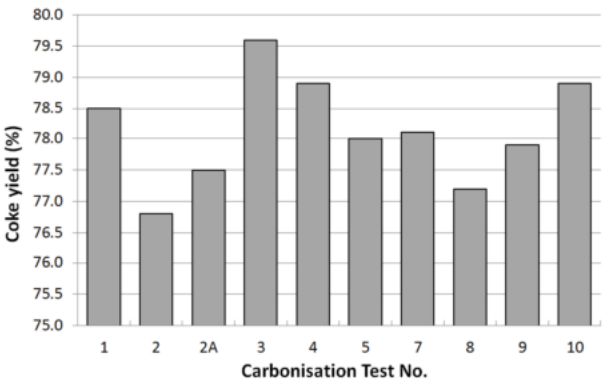



**Figure 16: Coke yield in semi-industrial carbonisation tests, first trial**

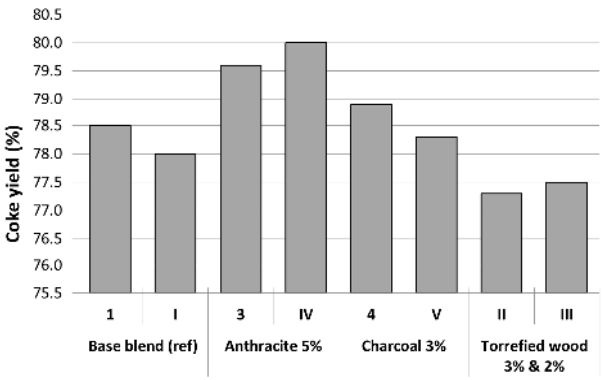



**Figure 17: Coke yield for torrefied wood in second trial and comparison with repeated tests from the first trial (No. 1, 3 and 4)**

Physical testing, as cold mechanical strength and particle size distribution, are presented in Task. 2.1, the results of coke microstructure are reported in Task.2.2, ash analysis is found in Task 2.3 and CRI/CSR results in standard and innovative tests in Task 2.4, Metallurgical testing. Also, selected coke samples from the pilot-scale coke oven tests have been tested in actual BF atmosphere as basket samples charged into the EBF before stoppage, quenching and excavation of the EBF shaft, presented in in Task 3.2.

Conclusions

- Addition of the HV thermal coal with no FSI reduces the wall load except when pet coke is added
- All test blends showed safe wall loads
- Adding anthracite to the blend improves the coke yield
- Adding thermal coal decreases the coke yield
- Using charcoal does not affect the coke yield but adding torrefied wood decreases the coke yield due to its higher VM content

Exploitation and impact of the research results

According to the semi-industrial testing, all test blends can be used when considering the safety of the coke plant except test blend No.2 with 10% thermal coal addition to the base blend that showed a higher wall load than other blends. As SSAB coke ovens are sensible to high wall pressure the amount of 10% addition may be too high for industrial tests.

### *Task 1.3 Selection of coke for further testing*

Planned activities and work accomplished

Based on the results from technical-scale carbonisation tests (retort) selection of blends to be carbonised in pilot scale (semi-industrial) were made. They were; Base blend and 10% addition of HV thermal coal no FSI (No. 3 in technical-scale tests) and 5% addition of anthracite (No. 4 in

technical-scale tests). As mentioned earlier the result in the semi-industrial first trial with 10% addition of thermal coal, was worse than indicated from the results in the retort tests, it was decided to make a new coke with 5% addition of the thermal coal instead. Due to the decrease in either mechanical strength and/or CSR/CRI in the retort trials, three blends were tested again in semi-industrial scale but with 3% addition instead of 5%; base blend mixed with charcoal, thermal coal + anthracite and thermal coal + pet coke.

Seven of the semi-industrial carbonised alternative coke was chosen to be tested in the LKAB Experimental BF (EBF®) shaft as basket samples in WP3, see Table 13. The standard coke type normally used in the EBF® was also tested in some baskets.

The later conducted second semi-industrial trial, with torrefied wood medium temperature, 2% and 3% addition, and repeated tests of 5% anthracite and charcoal addition were also investigated in WP2. The torrefied wood coke samples and repeated charcoal tests (3% addition) were also tested as basket samples in the FLEXCOKE EBF® campaign 32.

**Table 13: Selected alternative coke types to be tested in basket samples in the LKAB Experimental BF campaign 31**

| Test No. | Unit | 1 | 2A | 3 | 4 | 5 | 7 | 8 |
|---|---|---|---|---|---|---|---|---|
| LV coal | % | 40 | 40 | 40 | 40 | 40 | 40 | 40 |
| MV coal | % | 32 | 30 | 30 | 30 | 29 | 29 | 29 |
| HV coal | % | 28 | 25 | 25 | 27 | 25 | 25 | 25 |
| HV thermal coal no FSI | % | | 5 | | | | 3 | 3 |
| Pet coke | % | | | | | 3 | 3 | |
| Anthracite | % | | | 5 | | | | 3 |
| Charcoal | % | | | | 3 | 3 | | |

Description of activities, results and discussion

See results from the retort and semi-industrial carbonisation trials in WP2 and in WP3 Task 3.2.

Conclusions. Exploitation and impact of the research results

See WP2 and WP3 Task 3.2.

**WP2: Laboratory characterisation of coke relative blast furnace conditions**

Selected coke types have been characterised by standard methods (e.g. sizing, ash amount, composition, volatiles), metallurgically in standard tests (CSR/CRI and Micum/IRSID) and in innovative tests developed within the project by tk SE. The availability of samples from actual BF atmosphere in the LKAB EBF® makes it possible to outline changes of microstructure. Screening of produced coke is complemented by optical particle shape analysis. Thermal analyses for calculation of reaction rates were made. The microstructure and chemical changes of laboratory-treated coke have been compared with coke collected from different positions in the EBF® to study differences/similarities of coke properties.

***Task 2.1 Physical testing of coke***

Planned activities and work accomplished

Standardised, cold mechanical strength tests which measures coke size reduction and abrasion during handling and charging into the BF, as Micum, were carried out as well as coke particle size distribution for 10 kg retort and 500 kg semi-industrial test coke as well as in industrial produced coke used in BF trials. The particle shape of semi-industrial test coke from DMT were analysed using Optical Particle Analysing (PartAn 3D) system, installed to analyse the BF coke material in comparison to the gravimetric screen analysing methods at tk SE.[(6)]

Tumbling tests in the CSR/CRI test standard I-tumbler have been made on selected coke samples reacted in EBF®, including coke from high temperature zone. Samples collected in WP4 were evaluated in Task 2.1 and the results are reported in WP3, Task 3.2.

SSAB investigated the produced alternative coke properties carbonised in full scale trials at the SSAB coke plant in Luleå and results compared with the standard coke normally produced.

Description of activities, results and discussion

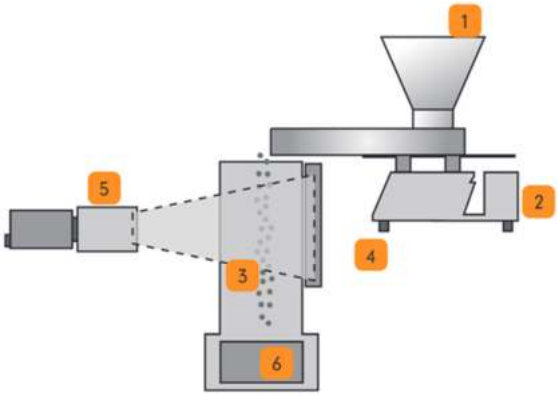
Alternative coke produced in the technical- and pilot-scale (retort and semi-industrial) carbonisation tests at DMT have been analysed and their particles sizes in terms of mean particle sizes and their cold, mechanical strengths are presented. The particle size distribution and cold mechanical strength for alternative coke produced in the SSAB industrial trials (Task 4.1) are also presented.

**Particle Analysis System – PartAn 3D**

The particle shapes of 15 coke types gained from DMT 500 kg semi-industrial oven were analysed. In addition to the coke particle shape, the mean grain size was also determined. Besides chemical and physical properties, the grain size distribution and shape factor are important parameters for optimal blast furnace operation, as they influence the gas flow and the drainage of liquids. To determine the grain size distribution tk SE uses an optical particle analysis system (PartAn) in addition to the gravimetric sieve analysis.

The layout of the optical particle analyser is displayed in Figure 18 and Figure 19.

**Figure 18: Measurement facility for PartAn (tk SE)**




1. Funnel
2. Vibrator feeder
3. Measurement area
4. Stroboscopic light
5. CCD - camera
6. Sample box

**Figure 19: Structure of PartAn (AnaTec AS)**

The sample to be analysed is fed into the equipment through a funnel. The material is then transported to the measuring area via a vibration feeder in which the particles are efficiently dispersed. With this mechanism, the overlapping of particles is prevented and the continuous flow of material is ensured. After leaving the vibration system, the particles fall between the stroboscope light and a CCD camera and are collected in a sample box.

There is a main difference between the 2D and 3D measuring system. With 2D, the first full frame image of a particle is used for the further evaluation, whereas with 3D several images are taken while the particle falls through the CCD camera and the stroboscope light. Due to the irregularity of coke particles, several shapes can be observed depending on the position. Therefore, a mean value of every image taken of a single particle is determined and used for further evaluation.

The resolution of the CCD camera can be up to 1600 x 1200 pixels, i.e. 1 920 000 pixels per image with a recording frequency of 25 Hz. This recording frequency ensures that each grain is measured while passing through the reflection area. Owing to the distance between the CCD camera and the

grain to be examined, it is possible to measure the grain size range from 0 mm up to 125 mm. This grain size range can be divided into different classes.

Figure 20 shows a typical picture of the optical particle analyser. Using the particle analysing system, the following parameters can be determined:

- Area (number of covered pixels)
- Circumference (number of covered pixels around the particle)
- Length (number of covered pixels for the longest expansion of the particle)
- Width (number of covered pixels for the longest expansion 90° to the length)

To control the vibration frequency and the conveying speed of the sample material, the following values are also determined:

- Quantity (number of particle per minute)
- Occupation (proportion of particles occupying image plane)

There are four different possibilities to determine the equivalent diameter, the conversion by area, the conversion by perimeter, the conversion by length and conversion by width, see Figure 21.

At tk SE the evaluation is based on the conversion by area.

**Figure 20: Typical picture of a coke piece taken from the optical particle analyser**

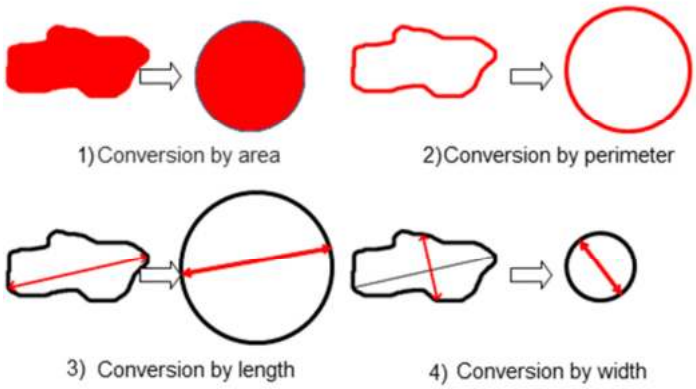



**Figure 21: Determination of equivalent diameter**

To determine the grain size diameter a square grid in the same size of the image resolution is set over the shade generated image. The pixels which are totally covered by the coke's shade are registered by the image analysing software (1). The total area of these pixels is converted first in a circle (2) and afterwards in a sphere with the same diameter (3), whereby the volume of the sphere can be calculated corresponding to the volume of the coke particle, see Figure 22.

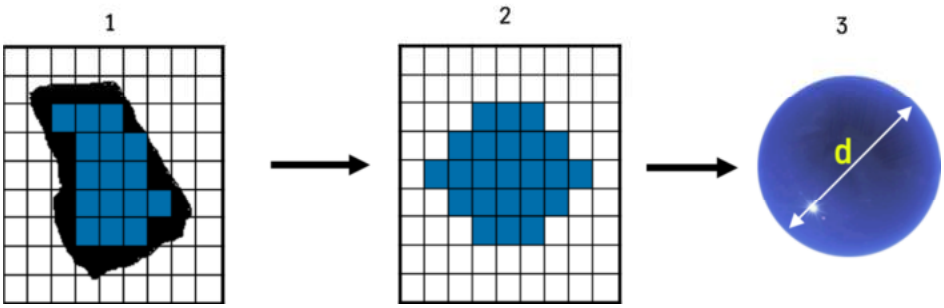



**Figure 22: Conversion steps**

*PartAn results of 500 kg semi-industrial tests at DMT*

To determine the grain size distribution and the particle shape of the coke samples gained from the DMT pilot trials a 30 kg coke sample was used for each test. This sample was divided into 3 parts of 10 kg each, resulting in three sub-samples. Every sub-sample was measured five times with the PartAn facility. For each sub-sample, the mean particle size distribution was calculated and used for the calculation of the overall mean value of the entire coke sample, see Figure 23.

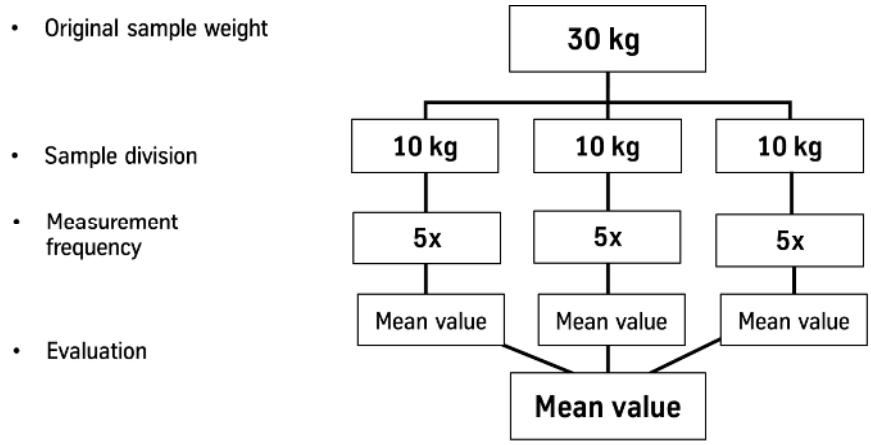



**Figure 23: Test preparation and evaluation**

Figure 24 and Figure 25 illustrate the mean particle size (MPS) gained with optical analysis for all coke samples from the 500 kg semi-industrial oven trials. The MPS varies between 59 mm (coal blend No. 5) and 70.8 mm (coal blend No. 1) for tests in the first trial and between 55.8 mm (coal blend No. II) and 64.4 mm (coal blend No. IV) for the second trial.

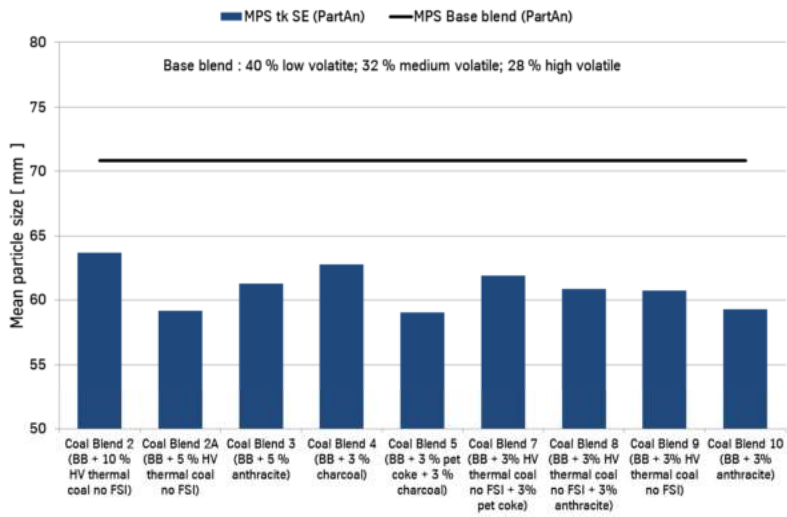



**Figure 24: Mean particle size – PartAn (first semi-industrial trial)**

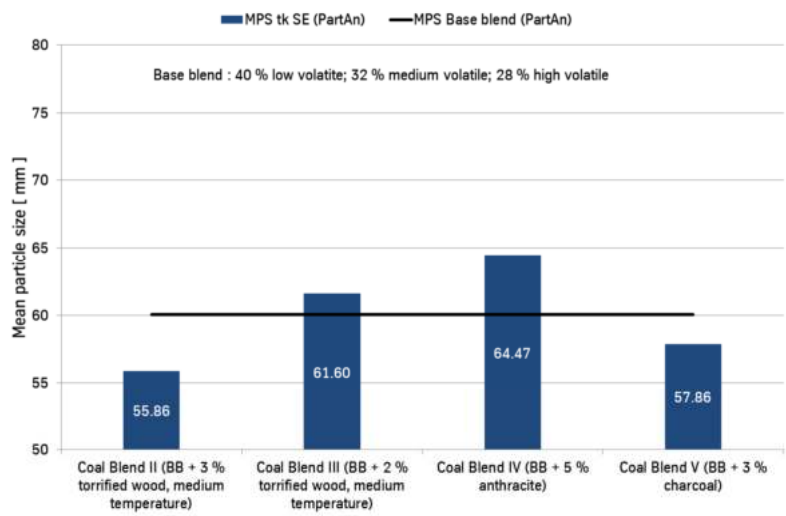



**Figure 25: Mean particle size - PartAn (second semi-industrial trial)**

The MPS gained for the reference blend in the first trial was with 70.8 mm 10.8 mm higher compared to the one from the second trial with 60 mm. Although the base blends were composed by the same coal types with the same ratio, the coals used in both trials derived from different batches, which can explain this divergence on the MPS results.

Regarding to the first trial series, the addition of alternative C-containing materials resulted always in significantly smaller coke pieces compared to the base blend. The strongest decrease was observed for coal blend 2A (addition of 5 % thermal coal), coal blend 5 (addition of 3% pet coke and 3% charcoal) and coal blend 10 (addition 3 % charcoal) with a mean MPS of only 59 mm.

The lowest reduction on coke piece size has been observed for coal blend 2 (addition of 10% thermal coal), coal blend 4 (addition of 3% charcoal) and coal blend 7 (addition of 3% thermal coal and 3% pet coke) where the resulting MPS was in the range of 62 to 64 mm (but still much lower compared to the base blend with 70.8 mm).

Considering the addition of anthracite and thermal coal to the base blend a trend can be seen. When increasing the anthracite content in the base blend from 3 % to 5 % a slight increase on MPS can be observed, whereas the addition of 10% instead 5% thermal coal to the base blend leads to the production of coke with a higher MPS, but of course still lower compared to the base blend.

Although all blends were used in the same 500 kg oven with similar test parameters for carbonisation tests it is difficult to compare the results from the first trial with those from the second trial. This shows for example the results of the base blend where a delta on MPS of 10.8 mm has been determined. Concerning the second trial series it can be summarised that the addition of 2% torrefied wood (coal blend III) and 5% anthracite (coal blend IV) apparently leads to the production of larger coke particles compared to the result of the repeated base blend test. Increasing the amount of torrefied wood from 2 to 3% decreases the MPS by 5 mm.

Coal blend IV (addition of 5% anthracite) and coal blend V (addition of 3% charcoal) were performed twice at the DMT 500 kg oven. The resulting MPS was rather close for the anthracite blend (61 mm vs. 64 mm) but divergent for the charcoal blend (63 mm vs. 58 mm).

As the shape factors, like convexity, sphericity and ratio length to thickness (L/T), were similar for all coke samples , Figure 26 - Figure 29 show exemplary 4 different coke samples, 2 from first pilot trial (coal blend 1 and coal blend 4) and 2 from the second one (coal blend I and coal blend III).

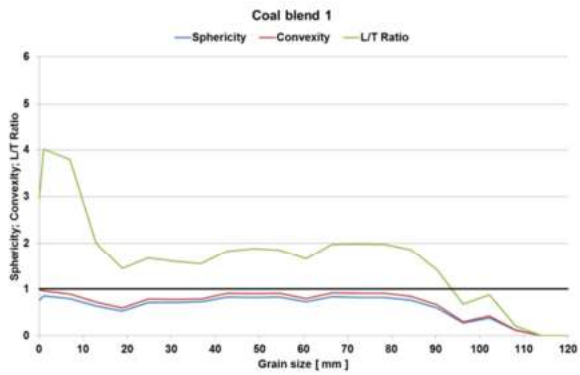



**Figure 26: Sphericity, convexity, L/T ratio - coal blend 1**

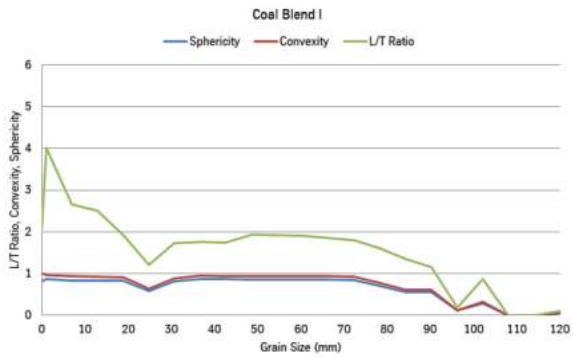



**Figure 27: Sphericity, convexity, L/T ratio - coal blend I**

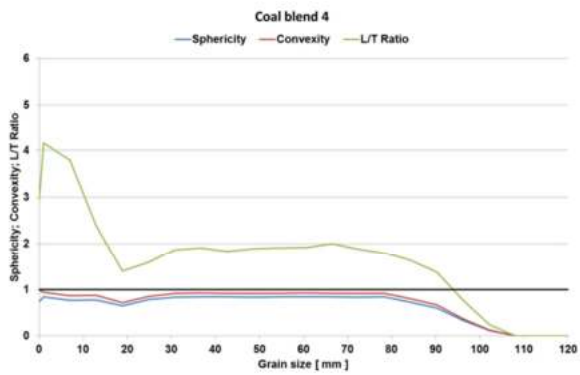



**Figure 28: Sphericity, convexity, L/T ratio - coal blend 4**

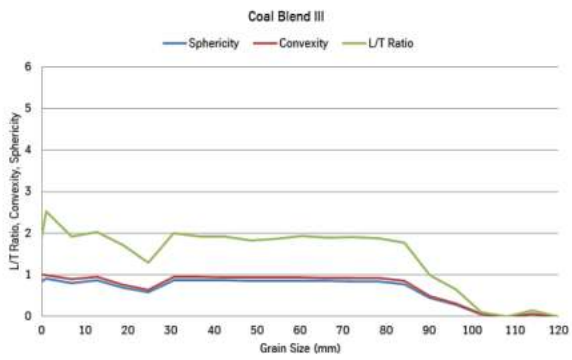



**Figure 29: Sphericity, convexity, L/T ratio - coal blend III**

With convexity the surface roughness of a particle is described whereas sphericity indicates how spherical the particle is. The scale for both shape factors reaches from 0 to 1. Convexity = 1 means that the particle has a smooth shape, whereas a convexity 0 means an irregular shape. Regarding sphericity, the higher the value the more spherical the particle is shaped.

From these results it can be concluded that for all coke pieces with a determined grain size smaller than 90 mm the length was greater than the thickness (L/T ratio >1). The trend in convexity and sphericity was also very similar. Coke particles with a grain size of less than 80 mm have in general a smooth surface and tend to be round.

**Particle size distribution of SSAB industrial scale carbonisation, WP4 Task 4.1**

*SSAB 1st cokemaking trial producing alternative coke for EBF® operational trials in Task 4.2*

As described in Task 4.1, it was decided in the project group that two coke types should be made for EBF® trials, i.e. coke with 5% thermal coal addition and coke with 3% thermal coal and 3% pet coke addition.

The sieve analyses of the produced coke were done on coke samples from the yard, which are not assured to be representative, and hence should be treated as an indication. Average sieving results of three tests of Mix 1 and Mix 2 coke types, i.e. Test Coke 1 (TC1) and Test Coke 2 (TC2) are reported in Figure 30 and an average of five tests for the standard coke produced. A higher abundance of medium sized coke and also for fines in the two test mixes of alternative coke versus normal production coke is noted. The coke MPS is plotted in Figure 31. As can be seen in the figure, the standard coke has a MPS of 60.1 mm and the MPS is reduced with 7.0 mm using 3% thermal coal and 3% pet coke addition (TC2) and with 8.4 mm with 5% thermal coal addition to the blend (TC1).

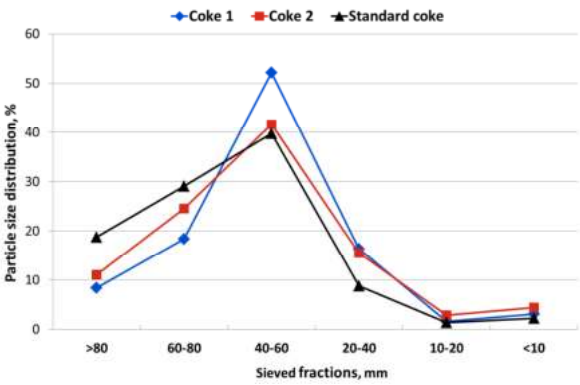



**Figure 30: Sieving curves of coke, showing standard coke Coke 1 (TC1) and Coke 2 (TC2)**

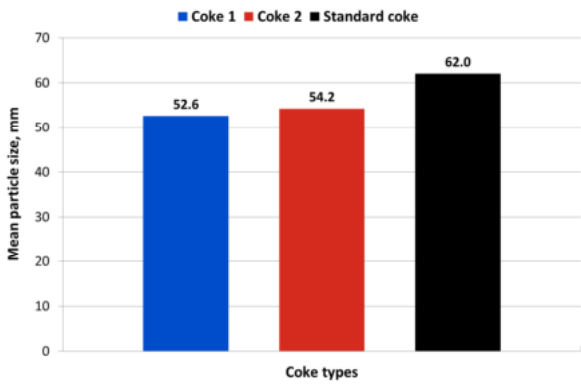



**Figure 31: Mean particle size (MPS), mm, of the alternative coke types and standard coke**

The SSAB industrial produced test coke are compared and illustrated with gravity sieving results from similar test blends carbonised at DMT 500 kg semi-industrial test oven, see Figure 32 for the sieving curves and Figure 33 for the MPS. Differences in the coal mixes are shown in Table 41, Task 4.1. Standard coke produced is compared with test No. 1 (base blend), Coke 1 is compared with test No. 2a (5% addition of thermal coal) and Coke 2 is compared with test No. 7 (3% thermal coal and 3% pet coke addition). The coke carbonised in the semi-industrial oven shows higher amounts of larger coke in fractions 60 to <80 mm and lower amounts of 20 to 60 mm than the SSAB industrial carbonised coke. The semi-industrial test coke No. 2A and No. 7 did not deviate that much compared to base blend No. 1. The higher amount of large coke influences the MPS, see Figure 33, as the DMT semi-industrial coke has higher MPS than the ones produced at SSAB 1st industrial trial.

The reduction in MPS when adding alternative carbon material to the blend is similar to the ones measured with PartAn, see Figure 24, in where coal blend 7 (3% thermal coal and 3% pet coke addition) has higher MPS than coal blend 2A (5% thermal coal addition) and all coal blends with alternative carbon material reduces MPS compared to base blend. The coke MPS resulting from the gravity sieving at DMT, see Figure 33, shows some diverging results as test No. 7 has higher MPS than test No. 2a but both of them have reduced MPS compared to without addition.

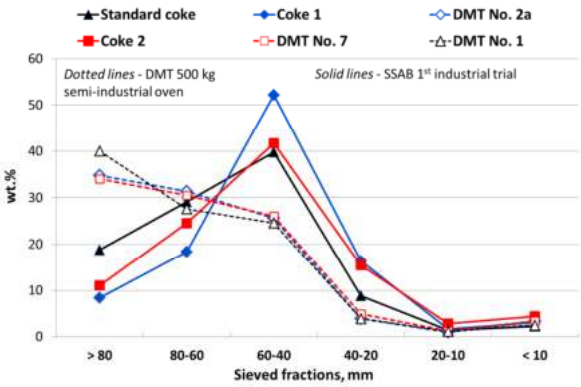



**Figure 32: Comparison of sieving curves of the industrial produced coke types and similar coal mixes in the semi-industrial tests**

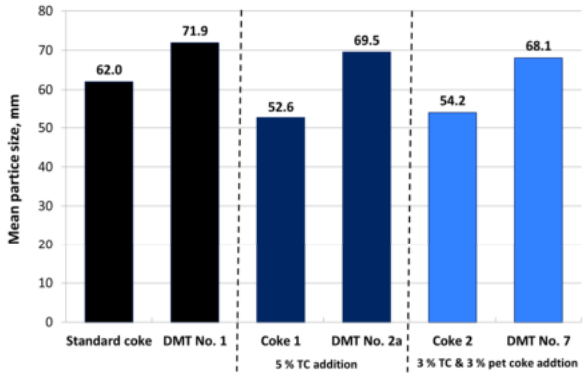



**Figure 33: Comparison of MPS of the industrial produced coke types and similar coal mixes in the DMT semi-industrial tests**

*SSAB 2nd cokemaking trial producing coke for full-scale industrial trials at SSAB BF No. 3 in Luleå, Task 4.3*

The second full-scale trial is described in Task 4.1. From the results of the FLEXCOKE EBF® trial it was decided to produce a trial coke similar to TC1, i.e. with 5% addition of thermal coal to the blend. Initially, 20% of the ovens in operation were filled with alternative coal blend. The number of ovens filled with FLEXCOKE trial blend was successive increased; 40%, 80% and 100% i.e. until all the ovens in the battery contained FLEXCOKE test coal blend and the produced FLEXCOKE trial coke (FTC) were transported and charged to the SSAB BF No. 3. The full scale trial at SSAB started the 9th of May and ended the 24th of June 2017. Weeks 15 to 18 are reference periods with carbonisation of the standard mix. The ramping up with mixture of the alternative test coke with 5% addition of thermal coal and standard coke was made during week 19 to 22, with 20% addition week 19 and 20, 40% during week 21 and 80% during week 22. 100% of FTC was produced during week 23-25. The particle size distribution during the FLEXCOKE trial is made on FTC with 5% addition of thermal coal to the mix.

The particle size distributions of the produced coke varied, not only for the FTC, but also in the standard, reference coke, see Figure 34, and can be consider as normal variation. Average data for the particle size distribution are plotted in Figure 35 and the FTC shows higher amounts in fractions 40-80 mm and lower amounts in 20-40 mm compared to reference coke.

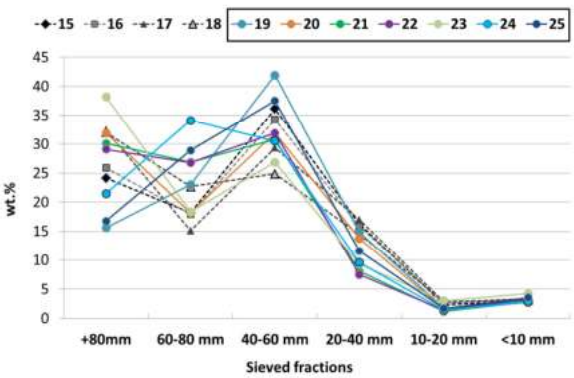



**Figure 34: Sieved analysis for reference coke (w. 15-18), black dotted lines, and FTC (w. 19-25)**

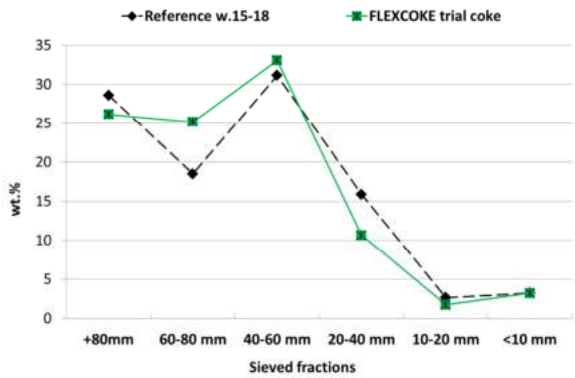



**Figure 35: Average particle size distribution reference coke and FTC**

The MPS of the produced coke are plotted in Figure 36. The MPS in reference coke varies between 59 to 62 mm and the average value is 59.6 mm. MPS in FTC varies between 56 to 64 mm and the average value is 61.3 mm. In contrary to the results in coke produced in the 500 kg semi-industrial trial and in the SSAB 1st industrial trial, has the addition of 5% thermal coal not resulted in smaller coke size.

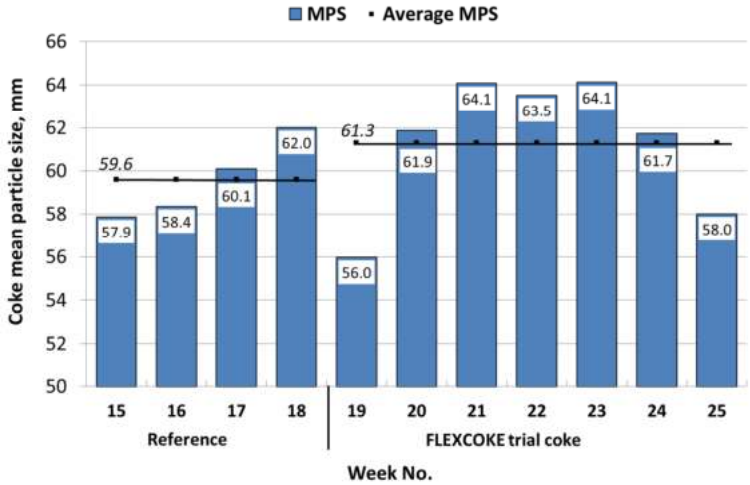



**Figure 36: MPS for reference coke and FTC. Averages are indicated**

## Cold mechanical strength

*Technical-scale (retort) carbonisation test coke*

The estimated values for mechanical strength are shown in Table 14**.** Both the Irsid $I_{40}$ and $I_{10}$ and the Micum values, Micum 40 ($M_{40}$) and Micum 10 ($M_{10}$) correlates. The Micum values are plotted in Figure 37 where all test coke samples have lower $M_{40}$ and higher $M_{10}$ than the reference coke blend (Test 1). The second best alternative regarding mechanical strength is Test 2 (HV thermal coal with high FSI), followed by Test 3 (HV thermal coal with no FSI) and 9 (HV thermal coal with high FSI and pet coke). However, the values of HV thermal coals with or without FSI showed very little difference. Adding anthracite and bioass to the blend have a negative effect on the mechanical strength in these tests, as Test 8 (Mix of thermal coal and anthracite) and Test 5 (charcoal) shows the worst mechanical strength. When mixed with HV thermal coal with no FSI in Test 10, the strength is slightly improved. As biomass as charcoal and torrefied wood have low bulk density the added amounts is much larger by volume which influences the inert part of the blend and therefore also the cold mechanical strength.

**Table 14: Estimated results of the mechanical strength in the DMT retort**

| | | JIS | $I_{40}$ | $I_{10}$ | $M_{40}$ | $M_{10}$ |
|---|---|---|---|---|---|---|
| **Test No.** | **Material** | **DI150/15** | **(Input >40mm)** | **(Input >40mm)** | **(Input >40mm)** | **(Input >40mm)** |
| 1 | Ref | 82.9 | 55.1 | 23.1 | 78.8, | 8.1 |
| 2 | Ref + HV thermal coal FSI | 82.2 | 53.5 | 23.8 | 77.8 | 8.3 |
| 3 | Ref + HV thermal coal | 81.9 | 52.8 | 24.1 | 77.4 | 8.4 |
| 4 | Ref + Anthracite | 81.1 | 51.2 | 24.7 | 76.3 | 8.7 |
| 5 | Ref + Charcoal | 79.9 | 48.5 | 25.9 | 74.6 | 9.1 |
| 6 | Ref + TW, high temp. | 81.5 | 52.0 | 24.4 | 76.9 | 8.6 |
| 7 | Ref + TW, med. temp. | 80.8 | 50.5 | 25.0 | 75.9 | 8.8 |
| 8 | Ref + HV thermal coal + Anthracite | 79.8 | 48.3 | 26.0 | 74.4 | 9.2 |
| 9 | Ref + HV thermal coal + Pet coke | 81.8 | 52.7 | 24.1 | 77.3 | 8.5 |
| 10 | Ref + HV thermal coal + Charcoal | 80.8 | 50.5 | 25.0 | 75.9 | 8.8 |

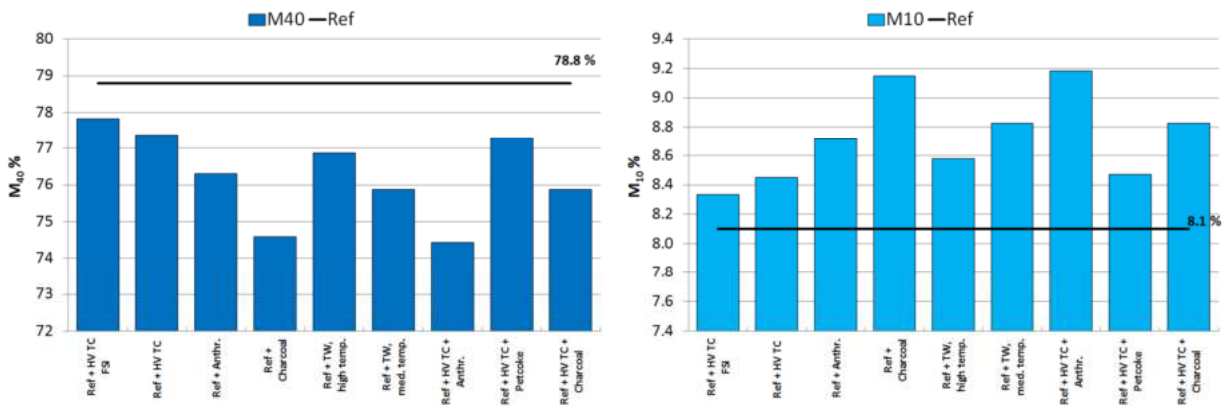



**Figure 37: Mechanical strength in coke produced in the retort, Micum 40 ($M_{40}$) to the left and Micum 10 ($M_{10}$) to the right side**

*Pilot-scale (semi-industrial) carbonisation coke, first and second trials*

The coke cold strength test result values of Micum and Irsid of the first semi-industrial trial are presented in the overview table in Appendix 1 and in Figure 38 for $M_{40}$ and $M_{10}$. As in the retort tests, all alternative carbon material degrade the mechanical strength as all test coke samples have lower $M_{40}$ and higher $M_{10}$ than the reference base blend.

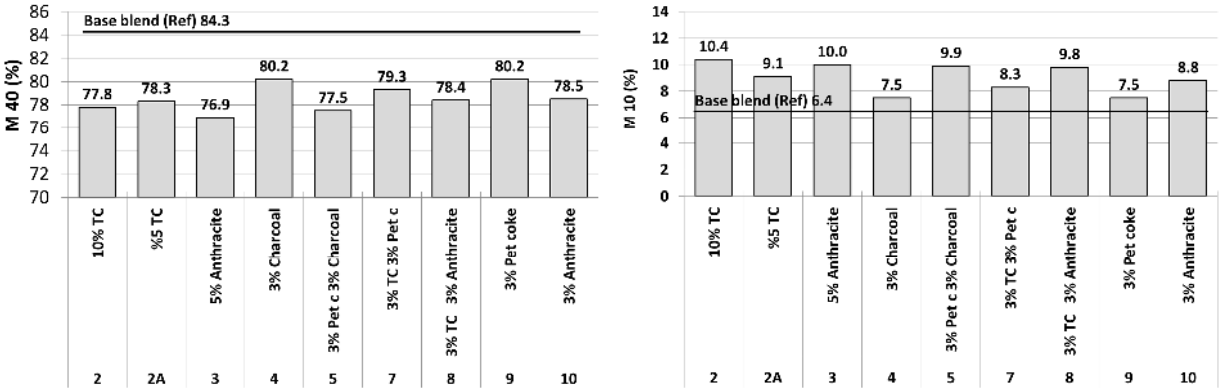



**Figure 38: Mechanical strength in coke produced in the 500 kg semi-industrial oven, first trial. Micum 40 ($M_{40}$) to the left and Micum 10 ($M_{10}$) to the right side**

By adding 10% thermal coal the effect on the cold strength was larger than with 5%, adding more inert into the blend. This can be noted for anthracite as well as the combined blends of pet coke and char coal (Test 5) and thermal coal and anthracite (Test 8). However, Test 7 (thermal coal and pet coke), also with 6% inert material in the mix seems to have a higher cold strength of $M_{40}$ and $M_{10}$. When adding 3% charcoal (Test 4), instead of 5% as in the retort tests, the mechanical strength is highest of alternative coke (in contrast to the results in the retort tests) in the semi-industrial carbonisation tests and addition of 3% thermal coal is the second best alternative.

The coke cold strength test result values of Micum and IRSID for the second trial are presented in the overview table in Appendix 2. The cold, mechanical strengths in terms of $M_{40}$ and $M_{10}$ are shown in Figure 39.

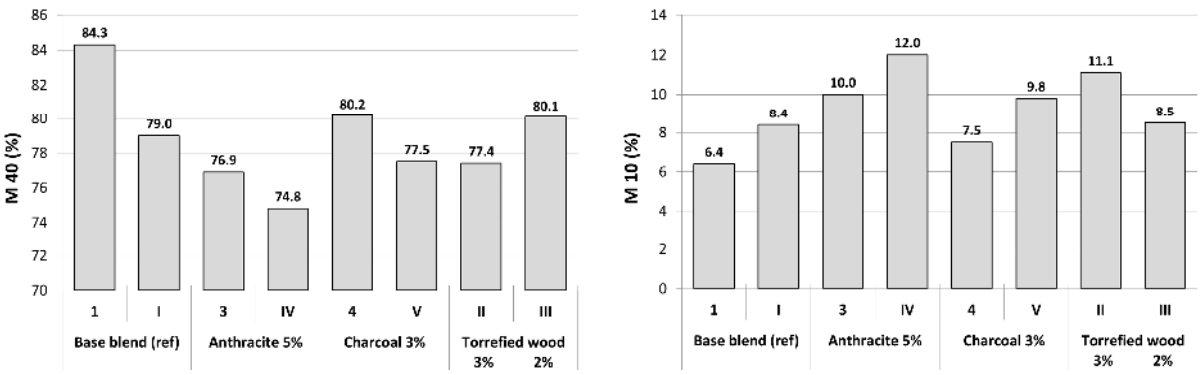



**Figure 39: Mechanical strength in coke produced in semi-industrial tests, Micum 40 ($M_{40}$) to the left and Micum 10 ($M_{10}$) to the right side. Second trial with torrefied wood medium temperature and repeated tests are compared with first trial (No. 1, 3 and 4)**

As in first trial, all alternative carbon materials lower the mechanical strength. However, the base blend (reference) for the new batch used in the second trial has considerable lower $M_{40}$ and higher $M_{10}$ than Test 1 in the first semi-industrial trial, probably due to the higher volatile matter of the base blend coking coals. This influences the results for the repeated tests of 5% anthracite and 3% charcoal addition. The addition of 2% torrefied wood showed quite good results, with somewhat higher $M_{40}$ but similar $M_{10}$, compare to the base blend in additional tests (I). 3% torrefied wood addition has more negative effect on the mechanical strength.

*SSAB 1st cokemaking trial producing alternative coke for EBF® operational trials in Task 4.2*

The cold mechanical strengths were tested by Micum tests. The Micum tests showed also good results, see Table 15 and Figure 40. Discussion regarding CRI/CSR is found in Task 2.4.

High $M_{40}$ results and also high and evenly analysed CSR are indicating a coke that is still very interesting to follow in EBF® trials. Both coke types are feasible to produce on full scale industrial trials.

**Table 15: The individual analysis of each series of test coke for CRI/CSR and Micum**

| Coal mix | CRI | CSR | $M_{10}$ | $M_{40}$ |
|---|---|---|---|---|
| Mix 1 Series 1 | 20.4 | 71.1 | 7.8 | 77.6 |
| Mix 1 Series 2 | 18.8 | 72.3 | 6.7 | 76.6 |
| Mix 1 Series 3 | 21.5 | 69.3 | 7.9 | 77.8 |
| **Average Mix 1 (Coke 1)** | **20.2** | **70.9** | **7.5** | **77.3** |
| Mix 2 Series 1 | 20.9 | 69.1 | 6.8 | 81.6 |
| Mix 2 Series 2 | 18.0 | 72.1 | 8.1 | 77.3 |
| Mix 2 Series 3 | 19.2 | 70.8 | 7.1 | 76.4 |
| **Average Mix 2 (Coke 2)** | **19.4** | **70.6** | **7.3** | **78.4** |

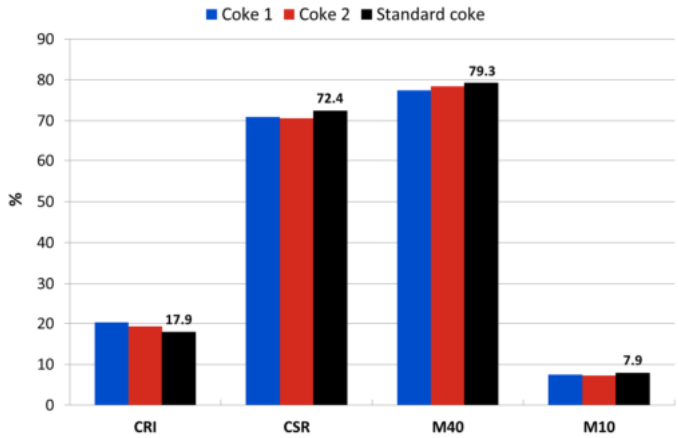



**Figure 40: Coke quality results of production of TC1 and TC2 versus standard coke**

*SSAB 2nd cokemaking trial producing coke for full-scale industrial trials at SSAB BF No. 3 in Luleå, Task 4.3*

The coke cold, mechanical strength represented by the Micum values in terms of Micum 40 and Micum 10 are shown in Figure 41 and Figure 42, respectively. Averages of are indicated as the black lines in the figures.

The $M_{40}$ in reference coke varied between around 72% to 79% and the average value is 76.1%, see Figure 41. $M_{40}$ in FLEXCOKE trial coke (FTC) showed higher values in general than the standard coke and varies from 73 % to 81 % with an average value of around 76.5 %. On the other hand, the FLEXCOKE trial coke seems to have slightly less resistance to abrading, as the $M_{10}$ values are generally higher than the standard coke, see Figure 42. However, there are some samples that also show very low $M_{10}$ values as week 24 and 25. The standard, reference coke, $M_{10}$ varies from around 8% to 9% with an average of 8.6% while the FTC has $M_{10}$ varying from around 7% to 12% with an average of 9.8%.

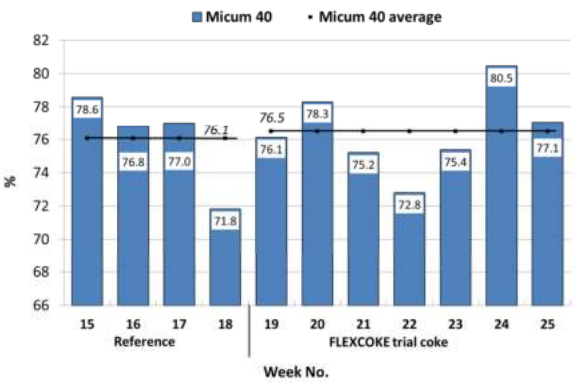



**Figure 41: Coke mechanical strength, $M_{40}$. Averages of $M_{40}$ tests indicated as the black lines**

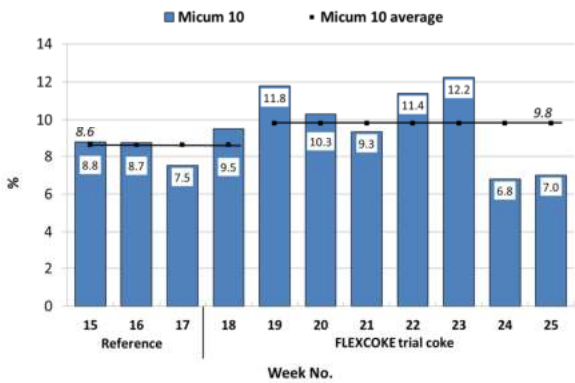



**Figure 42: Coke mechanical strength, $M_{10}$. Averages of $M_{10}$ tests indicated as the black lines**

Conclusions

The addition of alternative C-materials to the base blend mostly resulted in significantly smaller coke pieces measured by PartAn from the semi-industrial oven. However the amount of a certain material added to the base blend influences the size reduction of the coke pieces, as could be observed for the addition of anthracite and thermal coal. Increasing the percentage of alternative C-containing materials results in larger coke pieces, although still smaller than the reference base blend. It can also be concluded that for coke pieces smaller than 90 mm the length was larger than the thickness and coke particles with a grain size of less than 80 mm generally have a smooth surface and tend to be round. The same trend was seen in gravimetric sieving in the first industrial scale cokemaking at SSAB producing alternative coke for the EBF trial in WP4. The MPS was not notably affected during the second industrial carbonisation trial producing coke for the full scale SSAB BF trial. Comparing the two alternative coke types produced in the first SSAB industrial trial with similar blends carbonised in DMT 500 kg semi-industrial oven the MPS was lower for the industrial produced alternative coke types.

Adding inert alternative C-containing materials decreases the cold mechanical strength and increases the abrasion. 3% addition of charcoal or pet coke is the best option for good cold, mechanical strength. A mix of 3% thermal coal and 3% pet coke or 5% thermal coal can be an option.

Adding anthracite has worse effect but recovered slightly by mixing with pet coke. Biomass has low bulk density which effects the amount added by weight, 3% addition of charcoal gives quite good result compared to 5% addition. Similar trend is observed in 2% torrefied wood compared to 3% addition.

Alternative coke produced at SSAB coke plant in Task 4.1 was partly based on the conclusions above and the cold, mechanical strength of the actual coke produced showed good strength regarding both $M_{40}$ and $M_{10}$ for the first carbonisation trial with only slightly lower $M_{40}$ and higher $M_{10}$ than the standard coke. In the second industrial trial the $M_{40}$ was in general even higher than the standard coke but then the resistance to abrasion was lower as $M_{10}$ was higher than the standard coke. Comparing the two alternative coke types produced in the first SSAB industrial trial with similar blends carbonised in DMT 500 kg semi-industrial oven the $M_{40}$ was lower for the industrial produced alternative coke types but they had lower $M_{10}$ values.

Exploitation and impact of the research results

Owing to the limited availability and elevated cost for a high-quality coking coal, the utilisation of alternative C-material in the coking coal blend to produce coke that meets the BF requirements were investigated. However the addition of tested alternative materials (thermal coal, anthracite, petrol coke, torrefied wood and charcoal) decreases the mean particle size of the produced coke compared to the reference blend. In addition, the cold mechanical strength are affected by adding alternative C-material in the coking coal but using 5% thermal coal in full scale coking trials at SSAB resulted higher cold strength than expected from retort and semi-industrial tests.

For industrial application a stepwise approach in adding alternative C-materials is recommended. It can be assumed that the impact in terms of the tested materials is also visible on industrial coke production (smaller coke pieces) but the absolute reduction on MPS and mechanical strength can be different. As seen in the FLEXCOKE project the effect on particle size was different between the two campaigns and the effect of smaller particle size was not seen during the 2$^{nd}$ full scale trial. It is also unknown which specific requirements on coke particle size and mechanical strength the different European BF operators have.

### *Task 2.2 Analysis of coke microstructure*

The quality defined with the CSR/CRI test, which is the standardised test used for high-temperature properties of coke, correlates well with operational results and coke consumption. However, as the test is carried out in 100% $CO_2$ and at constant temperature, the structural change of coke and the gasification process is contradictory to the reactions under actual BF conditions. Microstructure studies by tk SE correlated the ratio of anisotropic phases in coke to the CSR.[(7),(8)] The relative proportion of the anisotropic phase in coke increases in the laboratory treatment of coke (CSR/CRI test or high temperature in inert atmosphere). However, coke samples from the BF showed the opposite result, i.e. the anisotropic part decreases, and an alternative test closer to the BF condition may reveal phenomena behind changes in coke microstructure in the BF.

Planned activities and work accomplished

Studies were made in order to understand changes in coke microstructure in relation to conventional coke quality and coke behaviour under metallurgical treatment in laboratory tests and in coke from the BF. It was planned that samples of coke produced from normal blends and blends using alternative coal/C-containing material would be analysed before and after treatment in the standardised CSR/CRI test, Tata tuyere rig and EBF® to evaluate the isotropy and anisotropy ratio. For this purpose tk SE uses their microscopically texture measurement method[8] that has been developed to quantify the anisotropic and isotropic carbon components of coke. However, measurements on coke samples derived from original planned tuyere rig trial were not carried out due to the withdrawal of Tata Steel from the project. Furthermore samples treated by using the innovative test method in Task 2.4 were investigated.

Following work has been accomplished by tk SE: The microstructure of coke produced at DMT in the technical and pilot trials (10 kg retort and 500 kg semi-industrial oven) as well at SSAB coke plant from the 1st industrial trial was investigated. Furthermore the change in the coke microstructure from coke basket samples (see WP3 Task 3.2) from EBF® C31 (campaign 31 autumn 2014) and C 32 (campaign 32 autumn 2016) as well from treated samples after standard and new innovative test method were determined. In total 84 samples were analysed.

In addition, gasification behaviour in selected samples from DMT and EBF® basket samples C31 (Task 3.2) have been examined and compared using a light optical microscope by LKAB with support from MEFOS. Changes in coke graphitisation degrees have been studied by MEFOS.

Description of activities, results and discussion

**Microstructure investigations using tk SE microscopically texture measurement method**

To investigate the microstructure of coke samples provided from DMT and SSAB as well of samples collected after EBF® trial and after CRI/CSR test, a microscopic method of tk SE was used.

The basis of the microstructure measuring technique is the quantitative detection of the optic bi-reflection of the carbon phases which results from the different reflection under polarized light. The basic physical principle for the detection of bi-reflection is the optical filtering of discrete directional oscillation of the white light, i.e. linear optical polarization seen in Figure 43. By turning two polarization filters of the same kind against each other, a complete extinction of the light can be observed (=crossed Nicols).

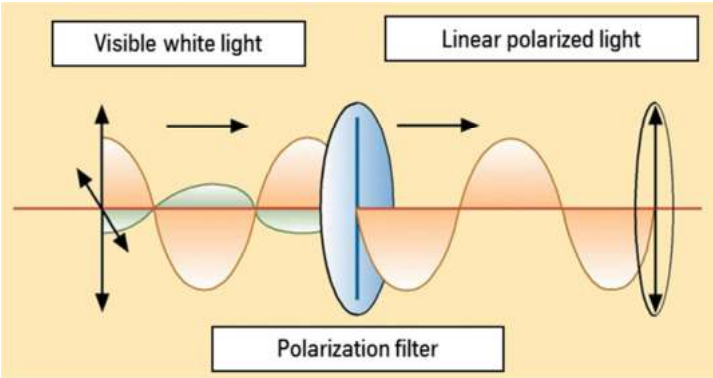



**Figure 43: Optical linear polarized light**

The concept of the coke microstructure measurement is based on the similarity of polarization effects between optically anisotropic materials and optical filters when exposed to white light. The anisotropic property of the coke microstructure component is used for the determination of the bi-reflection in such way that only one polarization filter is used. During the rotation of the polarization filter, the isotropic components do not change their reflectance, whereas the anisotropic phases change their reflection intensity from lighter to darker depending on the polarization filter position. The optical effects happen every 180° of the filter position.

Each pixel of taken images is associated with a grey value, which lies between 0 (black) and 255 (white) depending on the reflection intensity. The lightest and darkest reflections of each pixel are detected in order to generate the synthetic grey value image $Max_0$ and $Min_0$ respectively. The synthetic bi-reflectance (BiRef) image results from the difference between them as shown in Figure 44. These images are converted in a pseudo colour image as presented in Figure 45.

The coke microstructure has a wide range of reflection. Due to this, the measurement must be carried out twice at the same polarization position using two different camera gains; gain 0% and gain 100%. Thus, the low reflection structures (<8% reflection) and high reflection structures (0% to around 16%) can be properly distinguished.

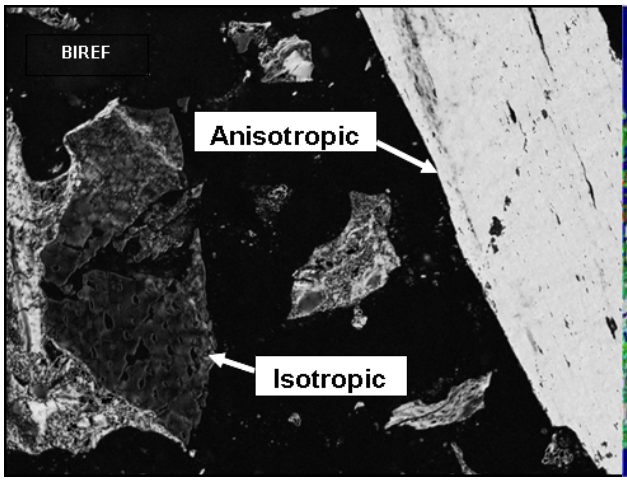



**Figure 44: Coke microstructure, Bi-reflection**

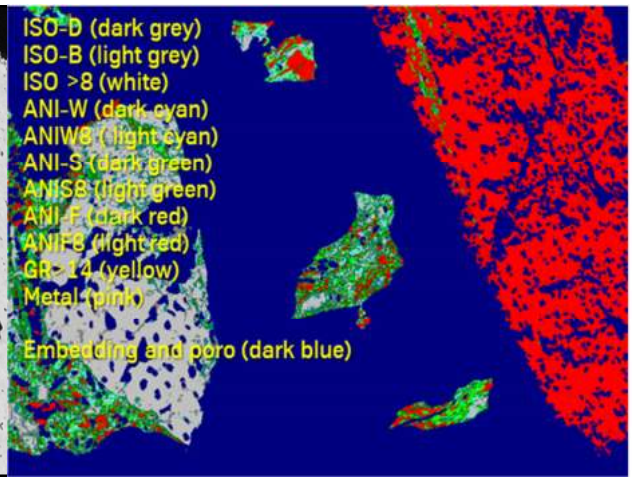



**Figure 45: Coke microstructure, isotropic and anisotropic components**

Due to different camera gains there are two corresponding classes for the same measured phase, gain 0% (isotropic dark-ISO-D, isotropic bright-ISO-B, anisotropic weak-ANI-W, anisotropic strong-ANI-S, anisotropic flow type-ANI-F) and the gain 100% (isotropic >8 % -ISO>8, anisotropic weak-ANI-W8, anisotropic strong-ANI-S8, anisotropic flow type-ANI-F8). The amount of pores and embedded material is not included in the results.
Depending on grey value, the isotropic and anisotropic phases are divided into several sub-phases. The isotropic phases are ISO-D, ISO-B and ISO > 8 and the anisotropic phases are ANI-W, ANI-W8, ANI-S, ANI-S8, ANI-F and ANI-F8. The isotropic and anisotropic total is determined by the sum of the respective phases.

Under consideration of the coke microstructure the following equation (1) has been evaluated by tk SE, taking into account the level of anisotropy (the higher the anisotropy the more it is considered in the equation).

$$\text{ANIQ} = \frac{\text{ISO-D}+2\times\text{ISO-B}+3\times\text{ISO}>8+4\times\text{ANI-W}+5\times\text{ANI-W8}+6\times\text{ANI-S}+7\times\text{ANI-S8}+8\times\text{ANI-F}+9\times\text{ANI-F8}+10\times\text{GR}>14}{\text{ISO-D}+\text{ISO-B}+\text{ISO}>8+\text{ANI-W}+\text{ANI-W8}+\text{ANI-S}+\text{ANI-S8}+\text{ANI-F}+\text{ANI-F8}+\text{GR}>14} \quad (1)$$

With respect to the composition of the coke carbon phases an increase in ANIQ is synonymous to an increase in the anisotropic parts of the coke microstructure.

*Coke microstructure results of technical-scale trial coke - 10 kg retort test at DMT*

The technical-scale trial was carried out at DMT in which 10 coke types out of different blends were produced in a 10 kg retort. Each mixture consisted of a base blend and had at least one addition of alternative C-containing material. The base blend was composed of 40% LV, 32% MV and 28% HV coal. The selected alternative C-containing material were HV thermal coal (with high or no FSI), anthracite, charcoal, pet coke and torrefied wood (TW) produced at high/medium temperature.

The results of the microscopic investigation turned out that the alternative C-containing material behave different under microscopic light as shown in Table 16.

The HV thermal coal with high FSI behaves anisotropic and was found in thin layers around the grains. In contrast, the HV thermal coal with no FSI has an isotropic property. Petrol coke (pet coke) is mostly anisotropic and showed a high reflection grade. Furthermore, anthracite also has an optical anisotropic property, whereby the most common phase was flow type. Charcoal behaves isotropic and could easily be detected see Figure 46. The TW (medium temperature) is also isotropic and was found quite individually, see Figure 47. Also in the wood material torrified at high

temperature an isotropic behaviour was observed. However, at some positions in the sample, a slight anisotropic behaviour was determined.

**Table 16: Behaviour of alternative C-containing material under polarized light**

| Anisotropic | Isotropic |
|---|---|
| Anthracite | Charcoal |
| Petrol coke | Torrified wood |
| HV Thermal coal high FSI | HV Thermal coal no FSI |

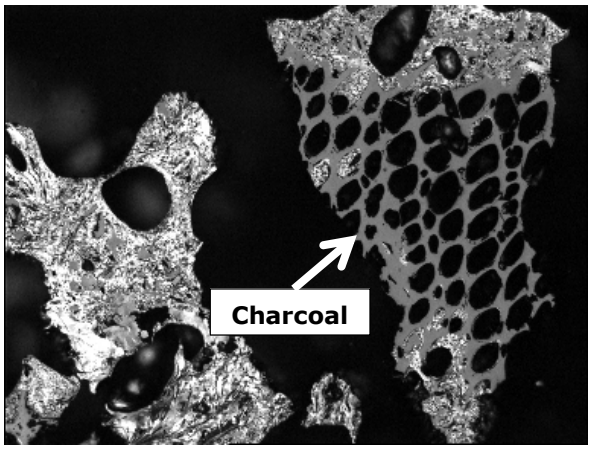



**Figure 46: Charcoal microstructure**

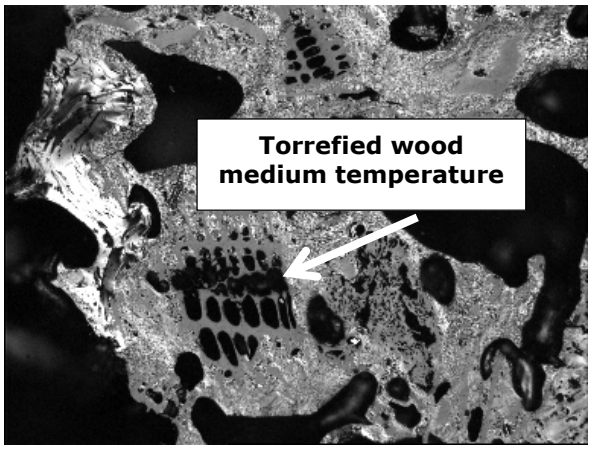



**Figure 47: Torrefied wood medium temperature microstructure**

The coke texture results were taken and correlated with a coke specific parameter - CRI, see Figure 48. The CRI and CSR tests for the 10 kg retort test were carried out at DMT. The samples are divided into two groups: orange = 5% addition of alternative C-containing material, blue = 10% addition of alternative C-containing material. The black dot represents the reference blend (base blend).

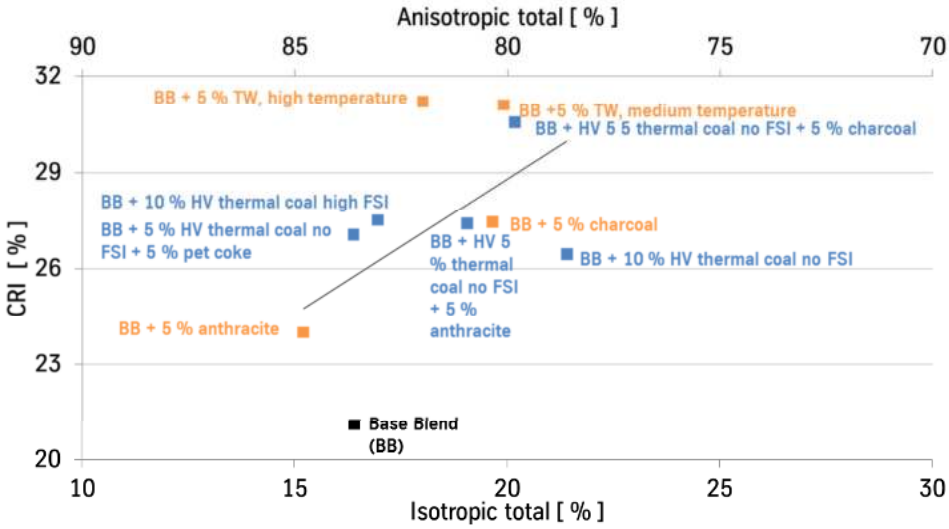



**Figure 48: Coke reactivity versus isotropic/anisotropic total**

Although some alternative C-containing material behaves isotropic (charcoal, torrefied wood, HV thermal coal no FSI) and other anisotropic (anthracite, petrol coal and HV thermal coal high FSI), the addition of alternative C-containing material to the base blend increased the isotropic phases in 8 out of 9 cases and increased the coke reactivity in all cases.

The results show two levels of reactivity, a group of blends with a resulting CRI between 26 and 28 % as well a group of blends with a CRI above 30% but below 32%. Most of the blends with thermal coal are found in the first group but adding charcoal to the thermal coal increases the reactivity up to the second level.

Regarding to the amount of added C-containing material (5% or 10%), no trend could be observed. The addition of 5% anthracite increased the anisotropy compared to the base blend. The opposite trend (increase isotropy) has been observed when 5% charcoal or torrefied wood was added.

The addition of thermal coal to the base blend showed different impact on coke texture depending whether a high FSI or no FSI quality was used. Adding thermal coal with no FSI significantly increased on isotropic total, whereas the addition of thermal coal with high FSI almost showed no change in isotropy compared to the base blend. The anisotropy increases by adding anthracite, charcoal or pet coke to the thermal coal no FSI. The highest increase can be observed when pet coke or anthracite was added to the thermal coal, since these alternative C-containing materials behaves anisotropically under polarized light.

**Coke microstructure results of pilot-scale trial - 500 kg semi-industrial test at DMT**

The pilot-scale trials at the 500 kg semi-industrial oven of DMT were performed in 2 campaigns, 1st series with 10 tests (first trial) and 2nd test series with 5 tests, (second trial).

The composition of the base blend was the same as for the 10 kg retort tests. But different batches of coal were used for the two campaigns which already resulted in different MPS of the base blend coke. In contrast to the 10 kg retort test the samples of the 500 kg semi-industrial oven tests partially show a higher isotropic total value and consequently lower anisotropic phases.

Also here the results of the coke texture measurements were taken and correlated with the corresponding CRI. Both DMT and tk SE performed CRI and CSR tests according to the standard on the produced material, however, for further evaluation only the tk SE results are considered. The coal blends were divided into four groups representing the amounts of addition of alternative C-containing material, red with 10%, dark blue with 6% (3+3%), green with 5% and light blue with 3% additives to the blend. The black dot represents the result of the base blend.

Figure 49 shows the results of the first campaign at the DMT 500 kg oven with a reference CRI of the base blend of 23%. It can be seen that the alternative C-containing materials lead to a rise in the isotropic phase content and in the coke reactivity index.

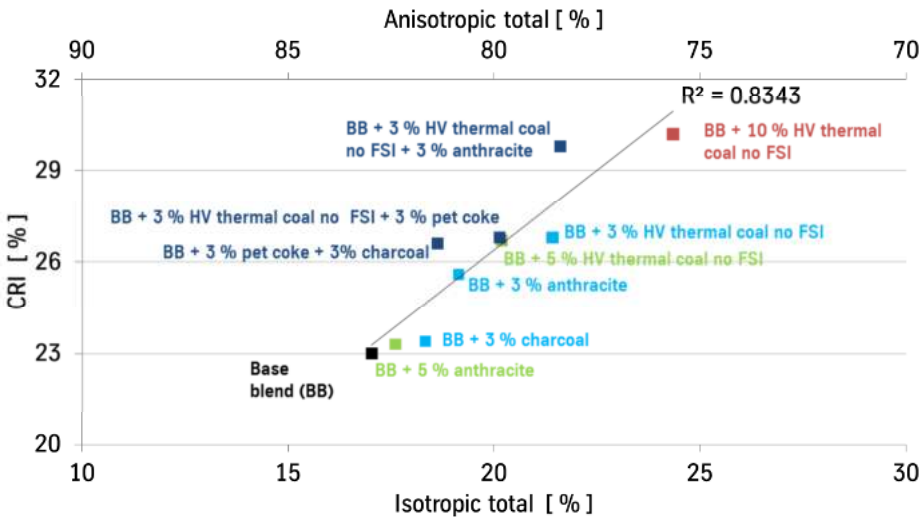



**Figure 49: Coke reactivity and isotropic/anisotropic total – first semi-industrial trial**

Figure 50 correlates the coke texture results with the corresponding CRI for the second trial. The samples are again divided in three groups: green = 5% addition of alternative C-containing material, light blue=3% addition of alternative C-containing material and purple = 2% addition of alternative C-containing material. Also from the second trial it can be concluded that the addition of charcoal, torrefied wood and anthracite to the base blend leads to an increase in both, coke

reactivity and isotropic total value. The strongest impact was observed for the sample with addition of 3 % torrefied wood. Decreasing the content of torrefied wood by 1% (from 3% to 2%) results only in a slight different result for the coke microstructure but the CRI significantly drops.

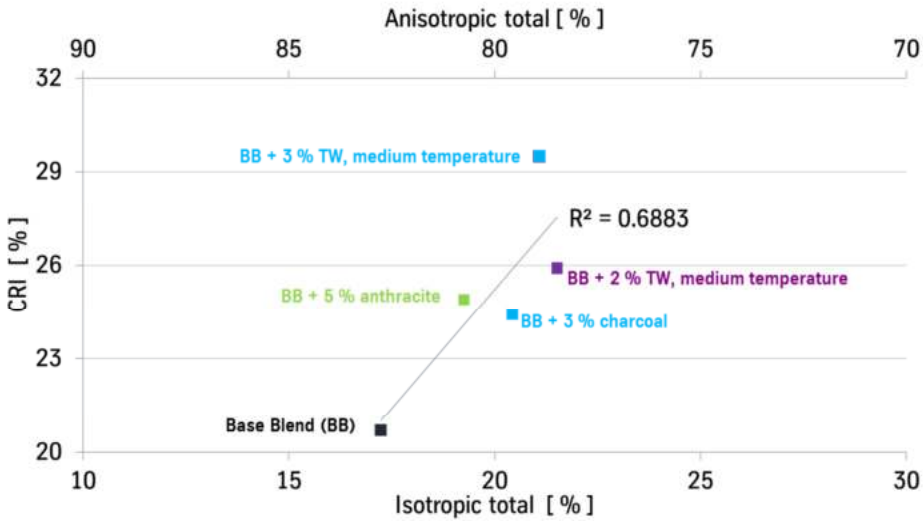



**Figure 50: Coke reactivity and isotropic/anisotropic total – second semi-industrial trial**

Although anthracite and charcoal behaves differently under polarized light, the addition of 3% charcoal or 5% anthracite almost show no impact on coke reactivity and only a slight increase in the isotropic phase content can be observed.

A moderate increase in coke reactivity (CRI between 25 and 27) and isotropic phase content can be observed for the blends with the addition of either 3% anthracite or HV thermal coal or the addition of pet coke + either thermal coke and charcoal respectively. Not consistent is the fact that the blend with the addition of 5% thermal coal leads to the same CRI as the addition of 3% and even lower isotropic phase content.

The highest CRI values (CRI around 30%) and isotropic phase content has been determined for the blend with the addition of 10% thermal coal and the blend with 6% addition of alternative C-containing material (3% thermal coal and 3 % anthracite). In contrast to the results of the 10 kg retort tests a higher correlation between isotropic/anisotropic content and resulting CRI can be found, see Figure 49 and Figure 50. An increasing isotropic content leads to higher CRI values.

When comparing coke microstructure gained from the 10 kg retort and 500 kg semi-industrial trials see Figure 51, it can be concluded that the CRI and isotropy for coke with same composition were different.

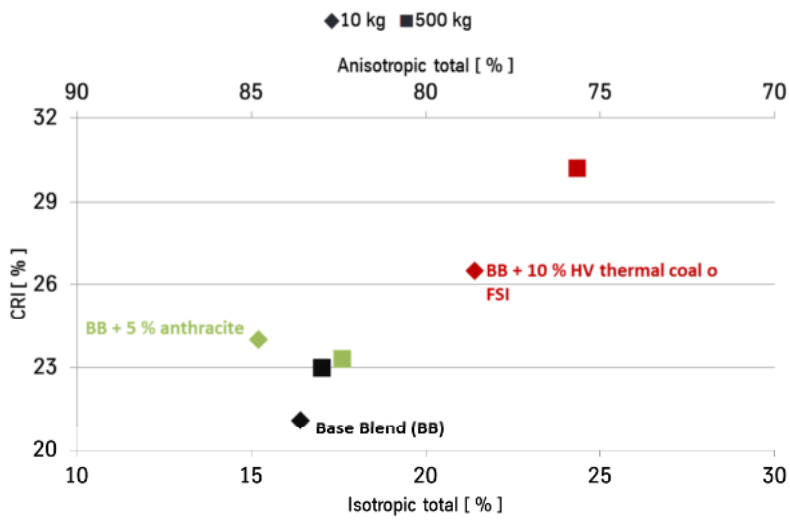



**Figure 51: Comparison of coke microstructure for tests in the 10 kg retort and first trial in the 500 kg semi-industrial oven**

The ratio of isotropic phases was higher and consequently the anisotropic phases lower, when coke was produced in the larger facility. Higher CRI values can also be observed except for the coal blend with anthracite addition. The strongest impact on isotropic total and CRI was when 10% HV thermal coal was added. As the same coal batches were used in the 10 kg retort and in the first semi-industrial tests, it can be assumed that coke produced in 500 kg semi-industrial oven tends to be more isotropic.

From Figure 52 no trend can be concluded, when correlating coke that was produced twice in the 500 kg semi-industrial oven. The measured isotropy and CRI was higher for blends with 5% charcoal or 3% anthracite in the second trial. The base blend show only slightly lower isotropy, but the CRI in the second trial base blend test was lower. Despite unchanged base blend composition, different batches of coal were used in the two campaigns. This could explain the difference on the isotropic and anisotropic total, since coke quality can differ depending on the batch, which can influence the formed microstructure in the produced coke.

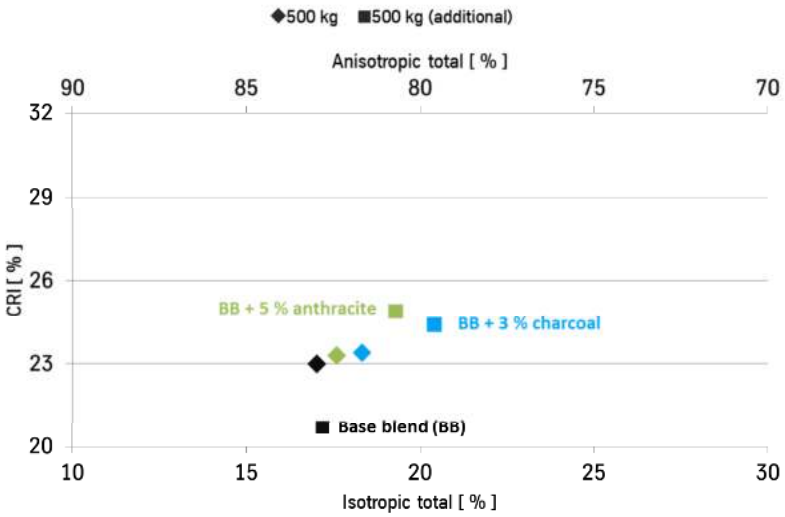



**Figure 52: Comparison of coke texture measurements – first semi-industrial trial and second trial repeated tests**

**Coke texture results SSAB 1st cokemaking trial producing alternative coke for EBF® operational trials in Task 4.2**

In the SSAB 1st industrial trial, see Task 4.1, two coal blends, which were already tested at DMT first trial 500 kg semi-industrial oven, were used at SSAB coke plant. However, the actual blends were slightly changed compared to DMT blends; see Table 41 in Task 4.1. Coke produced was TC1: base blend with 5% thermal coal no FSI addition and TC2: base blend and 3% thermal coal no FSI and 3 % pet coke addition.

From each coke type, three samples were analysed. The coke anisotropy increased when pet coke that contains more anisotropic phases was added together with the HV thermal coal. Comparing the coke texture results from measurements on DMT coke samples (No. 2A and No.7) with those from measurements on industrially produced coke at SSAB industrial trial test coke TC1 and TC2 (mean value) it can be concluded that in principle the DMT coke samples show higher amount of isotropic components than the SSAB coke samples, see Figure 53.

One reason for the increase of coke anisotropy can be explained by the change of the base blend composition for the industrial trial, where high volatile coal was replaced by higher qualitative coal (medium volatile and low volatile). This led to some modification of the in the single ratios for high, medium and low volatile coals.

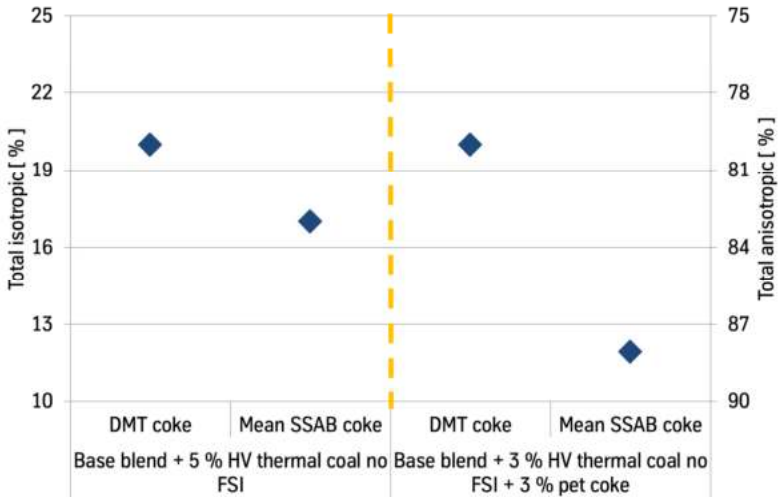



**Figure 53: Comparison coke texture measurements. DMT No. 2A and SSAB industrially produced coke TC1 (left). DMT No. 7 and SSAB industrially produced coke TC2 (right)**

**Coke microstructure results from EBF® basket samples**

Coke produced in the first DMT 500 kg semi-industrial trial was tested in basket samples in the EBF® campaign 31 (C31), see description in Task 3.2. From the 10 coke types derived from the DMT tests, 7 of them were charged in baskets and into the EBF® in 8 different layers. From the 48 charged baskets, 29 were recovered and 18 were selected for coke texture measurements, see basket charging scheme in Task 3.2, Figure 144. Additionally the standard SSAB coke used at the EBF® campaign was evaluated.

Summarised in Figure 54 the results of the coke texture measurements are shown distinguishing between the single coke types (Standard, Coke No. 1, 2A, 3 etc.) and its layer position found during excavation of the EBF® shaft. Furthermore for each coke type the corresponding Isotropic/Anisotropic content of the original material is shown.

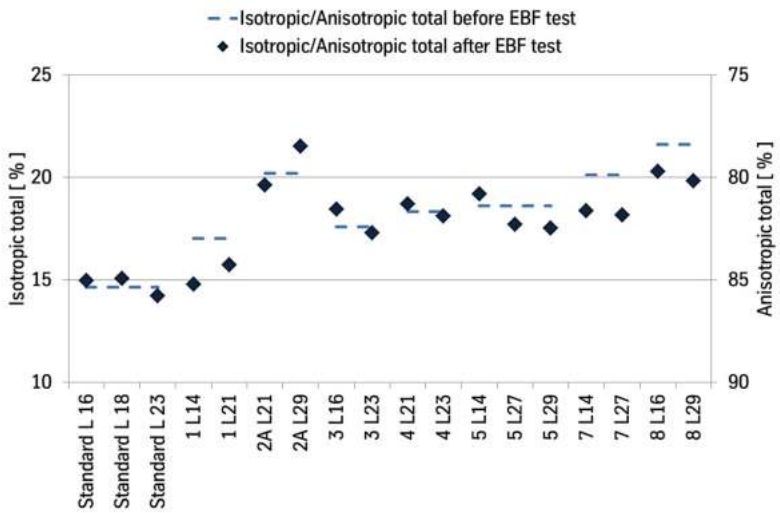



**Figure 54: Comparison coke texture measurement – original, standard coke and recovered basket coke samples – coke from first 500 kg semi-industrial trial**

It can be observed that some recovered coke samples became more isotropic and others more anisotropic. The highest increase could be observed for sample No. 7 (base blend + 3% thermal coal no FSI + 3% pet coke), whereas for standard coke, coal blend 3 (base blend + 5% anthracite) and coal blend 4 (base blend + 3% charcoal) only a slight change on the isotropy and anisotropy ratio could be noted.

Although a conclusion is difficult as only 2-3 samples from each coke type were analysed a first trend can be concluded. The lower position in the shaft, and higher layer No., of the recovered basket in the furnace is (= longer residence time in the furnace; higher temperatures, higher CO content in gas) the more anisotropic the samples became. This can be stated for coke type No. 3, 4, 5, 7 and 8. However, an opposite effect is observed for coke types 1 and 2A.

During the FLEXCOKE trial in EBF® campaign 32 (C32), 34 coke samples were charged in the EBF® in 10 different layers. Some baskets were divided with a mesh and contained two coke types, see description in Task 3.2. Beside the coke types TC1 and TC2 that were produced at the SSAB coking plant in the first industrial trial, some coke samples from the second 500 kg semi-industrial trial were charged. Chosen samples were the blends in where the biomass (torrefied wood and charcoal) were used as alternative C-containing material, see charging scheme in Figure 150.

From the charged baskets, 19 were recovered and 7 were selected for coke texture measurement. The coke used in EBF® campaign as reference (SC – standard coke) was also analysed.

The results of the coke texture measurement are shown in Figure 55. All coke types that were produced at SSAB coking plant (Standard, TC1 and TC2) show almost no difference in coke texture before and after EBF® tests. For the DMT semi-industrial second trial coke samples differences, especially for coal blend III but also blend II can be observed. Coal blend V was almost unaffected. Regarding the DMT coke, the isotropic and anisotropic total as well the measured CRI for coke produced with torrefied wood were higher compared to the coke with charcoal. Probably its isotropic phases reacted more intensively to weaker anisotropic phases in the EBF® compared to the blend with charcoal, resulting in moderate increase in anisotropy.

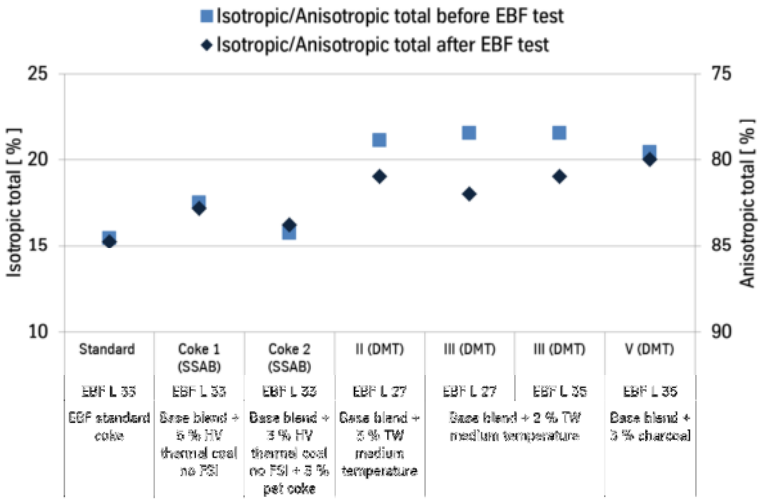



**Figure 55: Comparison coke texture measurement – original coke and recovered coke samples – SSAB (TC1 and TC2) and DMT coke with biomass addition (II, III and V)**

**Coke microstructure results of samples tested under standard CRI/CSR conditions – DMT and SSAB coke samples from the 1st industrial trial**

The coke texture was also determined for samples after performing CSR/CRI test for the DMT semi-industrial and SSAB full scale trial coke samples. For the DMT coke the results before and after CRI/CSR test are displayed in Figure 56. It can be observed that all samples became more anisotropic. Here the change in coke texture is more significant in comparison to results from the EBF® basket samples. The largest increase in the anisotropic phase content was observed for sample No. V (Base blend + 3% charcoal), despite the determined isotropic behaviour of charcoal.

Regarding the SSAB coke samples, it can also be seen that the isotropic components decreased and consequently the anisotropic part increased, see Figure 57. Despite the two coke types show different coke texture in the original state the isotropic and anisotropic proportions are very similar after being treated in the CRI/CSR test. During the CRI test, the coke carbon reacts with the carbon dioxide gas ($CO_2$) and a solution loss reaction ($C+CO_2=2CO$) takes place.

From former investigation at tk SE it was assumed that isotropic phases react more intensively with $CO_2$, resulting in a higher CRI. However this reaction does not explain the change in the coke isotropy, since plotting CRI value against the absolute delta isotropic total, no correlation between both can be observed, see Figure 58. As an example the marked samples (orange point) have similar CRI values, but large isotropic total deltas.

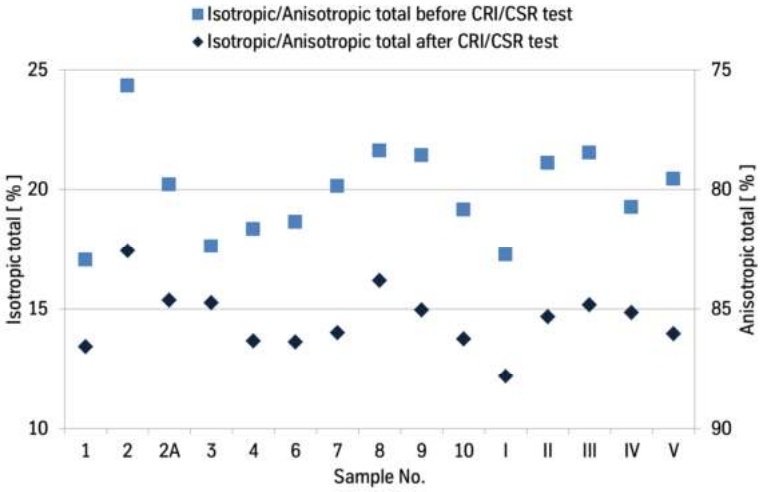



**Figure 56: Comparison coke texture measurement – before and after CRI/CSR test (samples with roman numerals are gained from the second trial at DMT)**

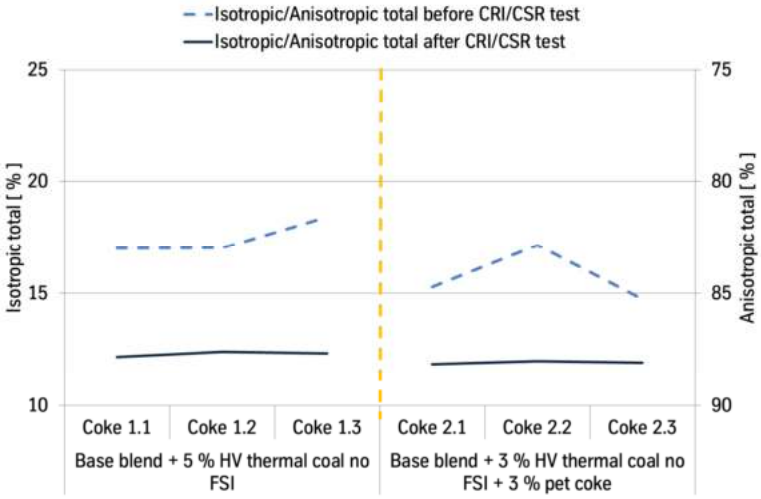



**Figure 57: Coke reactivity and isotropic/anisotropic total**

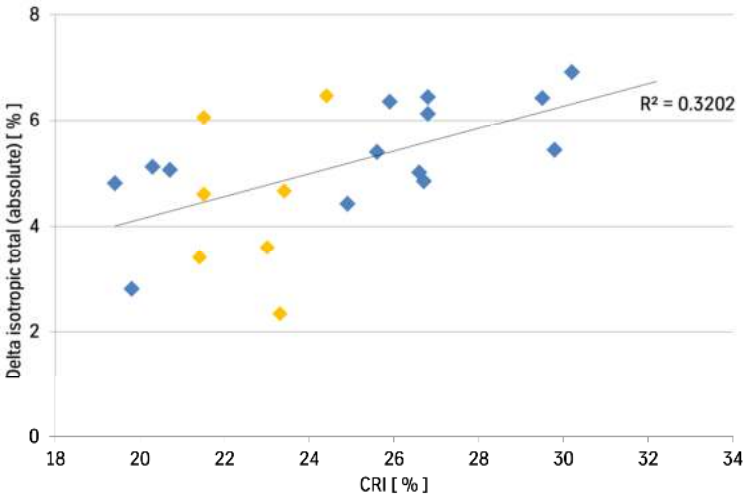



**Figure 58: CRI versus absolute delta isotropic total**

**Microstructure results for samples tested with innovative CRI/CSR test facility – tk SE coke**

The change in coke microstructure was also determined for coke samples tested under conditions different from those prescribed in the CRI/CSR ISO standard. For this purpose the new CRI/CSR facility at tk SE was used.

In Task 2.4 tests were performed using either 100% $CO_2$ or a gas mixture consisting of $CO_2$, CO, $H_2$ and $N_2$ in different ratios. This was done to simulate three different positions over the BF height (top, middle and bottom). For this test series, coke produced at tk SE coking plant was used. Selected particle size was 35 – 80 mm. Temperature of 1100 °C, heating time of 45 min and reaction time of 120 min were kept constant. In order to compare the change in coke microstructure, the original (=untreated) coke sample was also measured.

Figure 59 summarises the results for all coke samples. The black line represents the isotropic and anisotropic total for the untreated sample.

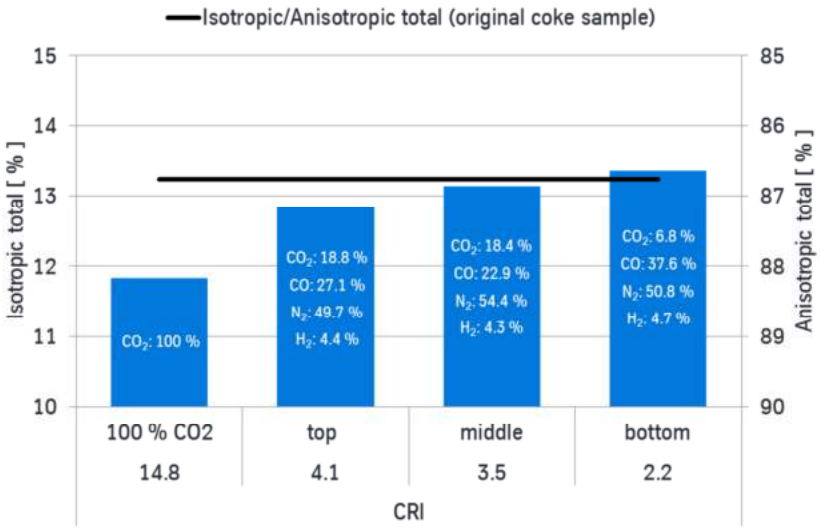



**Figure 59: Comparison coke texture measurement before and after CRI/CSR test (ISO and adapted gas composition). CRI given in %**

The coke sample tested in 100% $CO_2$ gas composition shows the highest change in isotropy/anisotropy. Switching to gas mixtures with $CO_2$, CO, $N_2$ and $H_2$ in different ratios has less impact on coke texture as less $CO_2$ in the gas means less solution loss reaction at the coke sample affecting the texture of the material. The content of isotropic phases increased for the test simulating the bottom position of a BF. This effect could also be observed for some of the EBF® basket samples even though different coke types were used there. As this diagram just show single results for each test condition further measurements will be carried out in the future in order to increase the database for further evaluation and quantification of the correlation between gas composition and the change in coke microstructure.

**Gasification behaviour in selected samples of BF simulating tests and basket samples from EBF® C31 using light optical microscope**

In total 18 samples have been investigated as summarised in Table 17 and Table 18. Four types of coke from the DMT first semi-industrial trial were selected to be analysed, i.e. Test 1 (base blend), Test 2A (base blend and 5% addition of high volatile thermal coal), Test 5 (base blend and 3% addition of pet coke and 3% addition of charcoal) and Test 7 (base blend and 3% addition of high volatile thermal coal and 3 % addition of pet coke).

The same coke types have been charged as basket samples into the EBF® in campaign 31, different layers/distance from top where these were found are presented in Table 17. In Table 18 the coke types, which have been tested in LKABs TRT Mas test in Malmberget, which is a BF simulating test in BF shaft atmosphere. As one of the tests with addition of 2A, K1913, has significantly higher coke weight loss both samples were investigated in the microscope. More information about these tests is found in Task 2.4.

The samples were prepared by LKAB and mounted in a 40 mm diameter sample mould and appropriate epoxy resin added. The epoxy mixture was allowed to cure, prior to subsequent polishing and instrument analysis. A Leica 4500 DP Light Optical Microscope (LOM) with a Leica DFC 290 digital camera was used as a platform for the image analysis conducted with Leica QWIN.V3 software and Leica LAS.V4 software. The QWIN.V3 software was used to obtain the total pore area as a fraction of total sample area. The LAS.V4 software enables the operator to conduct a pore size distribution and the automatic report feature delivers amongst a wide variety of particle data, a pore size histogram for each sample. A mosaic image of each sample was generated and created by stitching together several separate micrographs captured at 100X magnification. Phases that were either darker than pores or brighter than the matrix A phase, referred to as matrix B, was analysed in a Hitachi S3400N, a Scanning Electron Microscope (SEM), with a Bruker XFLASH 5010 SDD Energy Dispersive Spectroscopy (EDS) detector and a QANTAX 800 Esprit 1.9.4 software. The pore area of the samples, Method 1, and pore size distribution, Method 2, has been analysed by LKAB.

**Table 17: Selected samples of reference (untreated) coke types from DMT first trial and basket samples collected from the EBF® C31**

| Nr. | Coke type | Basket layer No. | Distance from top (m) |
|---|---|---|---|
| 1 | Base blend, untreated | 0 | 0 |
| 1 | Base blend, basket sample | 14 | 5.4 |
| 1 | Base blend, basket sample | 21 | 6.1 |
| 2A | Base Blend + 5% HV thermal coal no FSI, untreated | 0 | 0 |
| 2A | Base Blend + 5% HV thermal coal no FSI, basket sample | 21 | 6.1 |
| 2A | Base Blend + 5% HV thermal coal no FSI, basket sample | 29 | 6.5 |
| 5 | Base Blend + 3% Pet coke + 3% Charcoal, untreated | 0 | 0 |
| 5 | Base Blend + 3% Pet coke + 3% Charcoal, basket sample | 14 | 5.5 |
| 5 | Base Blend + 3% Pet coke + 3% Charcoal, basket sample | 27 | 6.0 |
| 5 | Base Blend + 3% Pet coke + 3% Charcoal, basket sample | 29 | 6.6 |
| 7 | Base Blend + 3% HV thermal coal no FSI + 3% Pet coke, untreated | 0 | 0 |
| 7 | Base Blend + 3% HV thermal coal no FSI + 3% Pet coke, basket sample | 14 | 5.5 |
| 7 | Base Blend + 3% HV thermal coal no FSI + 3% Pet coke, basket sample | 27 | 6.3 |

**Table 18: Selected samples of coke tested in a BF simulating atmosphere (TRT Mas)**

| DMT test No. and TRT Mas test No. | Coke type | Coke wt. loss (%) in TRT-Mas tests |
|---|---|---|
| 1, K1743 | Base blend mixed with MPBO | 0.97 |
| 2A, K1913 | Base Blend+5% HV thermal coalno FSI, mixed with MPBO | 4.12 |
| 2A, K2101 | Base Blend+5% HV thermal coal no FSI, mixed with MPBO | 0.94 |
| 5, K2194 | Base Blend+3% Pet coke+3% Charcoal, mixed with MPBO | 1.42 |
| 7, K1996 | Base Blend+3% HV thermal coal no FSI+3% Pet coke, mixed with MPBO | 1.04 |

*Total Pore Area, Method 1*

The mosaic images have been re-opened in the QWIN.V3 environment where they were subjected to a segmentation/detection of regions depending on the level of grayscale to obtain the total pore area as a fraction of total sample area. The principle of grayscale segmentation is based on the numbers of pixels compared to greyscale level and different layers represent different colours where zero (0) is total black and 255 is bright white. Blue represent greyscale up to 112, red from 214 and green in-between these two.

In Figure 60 the original grayscale image of coke sample No. 7, K1996 is presented as an example prior to segmentation to the left and after segmentation to the right. Here the pores and dark phases is blue, Matrix A is green and Matrix B is red. Figure 61 shows a magnified part of the small Matrix B area. The QWIN.V3 software is able to calculate the areas of the coloured parts of the image and the total area of the coke sample.

The results will then be; Pores and dark phases (Blue) 60.42%, Matrix A (Green) 38.08%, Matrix B (Red) 1.5% and the coke area $4.286*10^6$ $\mu m^2$. The other partial areas are given as a percentage of this total area for example the blue area (60.42 %) is actually $0.6042x4.286*10^6=2.59*10^6$ $\mu m^2$.

The segmented blue colour named "Pores and dark phases" includes not only the fractional pore area of the coke sample but also, as the name reveals, so called dark phases. These phases have a grey level just below the pores. They are rare but do have impact on the pore area measurement.

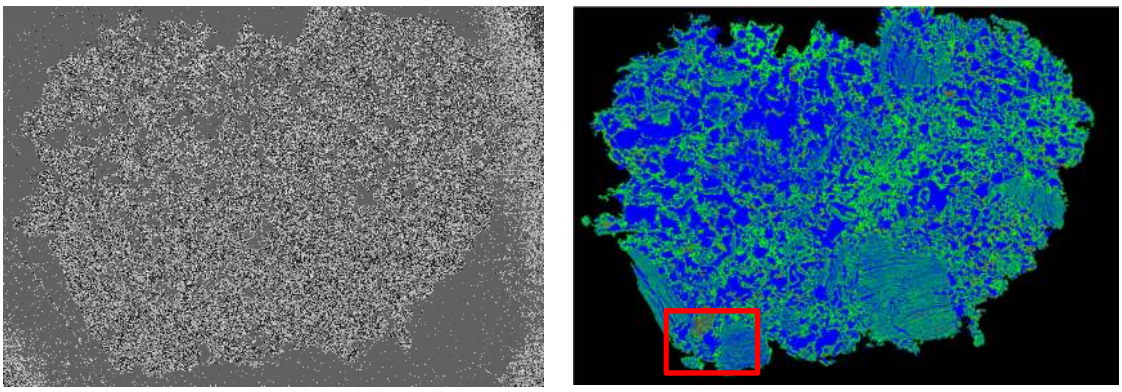


**Figure 60: Original grayscale mosaic image of sample No.7 K1996 prior to segmentation to the left and after segmentation to the right. Segmentation visualised via colours**

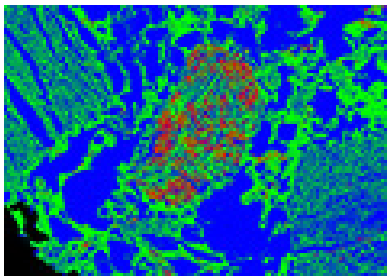


**Figure 61: Magnified rectangle, colours representing Pores and dark phases (Blue), Matrix A (Green) and Matrix B (Red)**

In Figure 62 sample No.7 K1996 is presented in a LOM image showing an example of where a dark phase. At the right side circular area is magnified 100 times. In order to investigate these dark phases the area was investigated in SEM equipped with EDS.

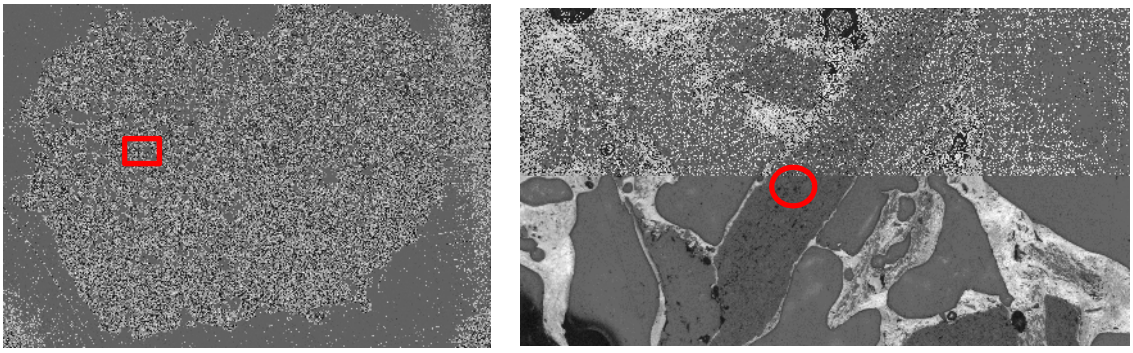


**Figure 62: Sample No.7 K1996 with red rectangle displaying a "dark phase" and magnified 100X from area marked with a rectangle at the right side (LOM)**

In Figure 63 as SEM image of dark phase and areas investigated are shown. Areas are P0 and P1 for the dark phase, P2 for Matrix A and a mixed phase (P3). In backscatter mode, the dark phase is brighter than the surrounding phases due to denser material. As carbon is a light element, the coke matrix containing mostly C is dark in SEM. In Table 19 the EDS-analysis is presented as concentrations calculated from X-ray spectra.

The dark phase (P0 and P1) comprises mainly of Al, Si – oxides with some amounts of Ca and K. The Matrix A phase (P2) has somewhat lower amounts of silica-alumina oxides but also comprises sulphur, S and the mixed phase (P3) also contains some S, K and Ti. All phases includes C and

analysed phases, mainly ash, are the normalised to 100%. As alumina-silicates are typical coke ash minerals points to that the dark phases are probably areas of coke ash which are not dispersed in the coke matrix or, in this case when reacted in the TRT Mas BF simulating tests, remains of coke ash when C in coke has reacted with $CO_2$ gas.

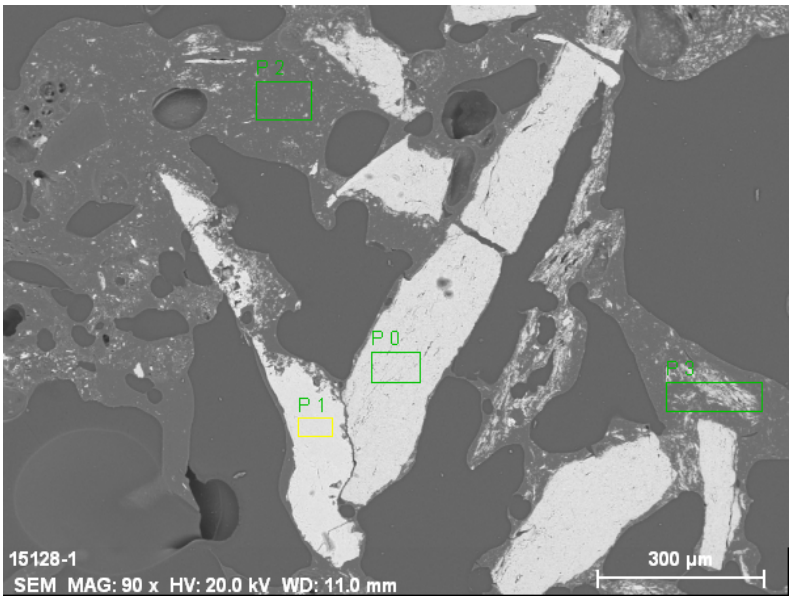



**Figure 63: Image of the dark phase (P0 and P1), Matrix A (P2) and a mixed area (P3)**

**Table 19: EDS-data from area measurements on dark phase, Matrix A and mixed phase**

| Spectra* | O | Al | Si | S | K | Ca | Ti |
|---|---|---|---|---|---|---|---|
| P0 | 26.5 | 35.9 | 36.7 | - | 0.92 | 0.05 | - |
| P1 | 27.7 | 36.6 | 36.7 | - | - | - | - |
| P2 | 20.9 | 24.3 | 28.7 | 26.1 | - | - | - |
| P3 | 19.7 | 34.7 | 39.9 | 3.3 | 1.88 | - | 0.56 |

*Elements in normalised mass percent (%)

The Matrix B phase is also investigated by SEM where the rectangular area showing the red coloured Matrix B in Figure 60 and Figure 61 are seen in the SEM image in Figure 64. The results from the EDS-analysis is presented in Table 20 and shows that the Matrix B phase (P0 and P1) is S rich and likely derived from ash. P2 and P3 are typical coke matrix areas, though with different textures, see Figure 64.

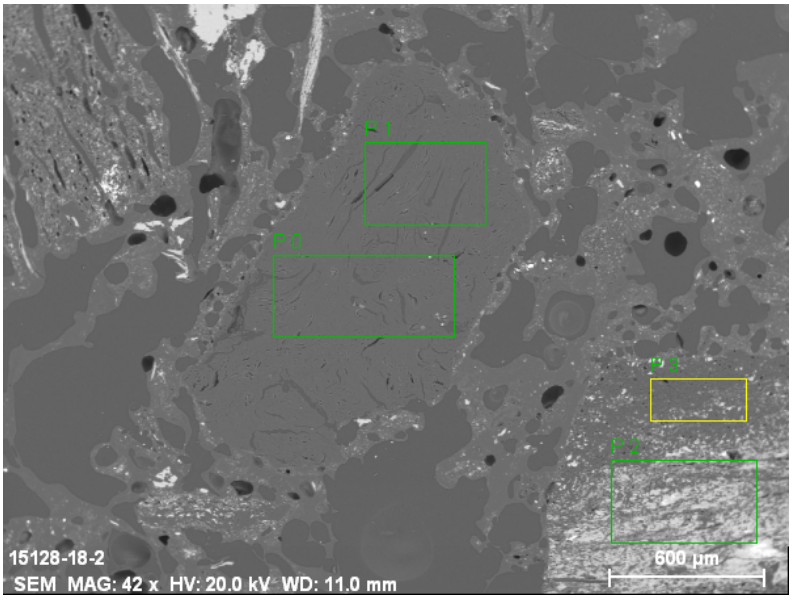



**Figure 64: Rectangle area in Figure 61 investigated in SEM**

**Table 20: EDS-data from area measurements on Matrix B (P0 and P1) and coke matrix (P3 and P4)**

| Spectra* | O | Al | Si | S | K | Ca |
|---|---|---|---|---|---|---|
| P0 | 33.3 | - | - | 60.3 | - | 6.45 |
| P1 | 41.7 | - | - | 58.2 | - | - |
| P2 | 22.7 | 37.1 | 37.8 | 0.88 | 1.58 | - |
| P3 | 22.4 | 32.8 | 37.8 | 4.13 | 2.87 | - |

*Elements in normalised mass percent (%)

*Results from Method 1*

The analysis separates three phases based on difference in grayscale. The software can only present the total pore area. Table 21 shows the results for un-treated samples compared to basket samples from different layers in the EBF® shaft excavated from campaign 31. In Table 22 TRT Mas test samples are presented.

**Table 21: Pore area, Matrix A and B and coke area for untreated coke and basket samples (bs) at different height in the EBF® shaft. BB=base blend**

| No. | Coke type | Basket layer No. | Pores/dark phases, % | Matrix A, % | Matrix B, % | Coke area, $mm^2$ |
|---|---|---|---|---|---|---|
| 1 | Base blend, untreated | 0 | 57.79 | 41.77 | 0.44 | 3.47 |
| 1 | Base blend, bs | 14 | 57.89 | 41.79 | 0.32 | 4.24 |
| 1 | Base blend, bs | 21 | 57.16 | 42.64 | 0.21 | 3.79 |
| 2A | BB + 5% HV thermal coal no FSI, untreated. | 0 | 56.35 | 43.17 | 0.48 | 4.77 |
| 2A | BB + 5% HV thermal coal no FSI, bs | 21 | 56.41 | 42.47 | **1.12** | 3.91 |
| 2A | BB + 5% HV thermal coal no FSI, bs | 29 | 60.93 | 38.93 | 0.15 | 4.78 |
| 5 | BB+3% Pet coke + 3% Charcoal, untreated | 0 | 62.35 | 37.28 | 0.38 | 3.72 |
| 5 | BB + 3% Pet coke + 3% Charcoal, bs | 14 | 61.15 | 38.69 | 0.16 | 4.19 |
| 5 | BB + 3% Pet coke + 3% Charcoal, bs | 27 | 55.59 | 44.09 | 0.32 | 3.26 |
| 5 | BB + 3% Pet coke + 3% Charcoal, bs | 29 | 60.05 | 39.79 | 0.15 | 3.26 |
| 7 | Bb + 3% HV thermal coal no FSI+3% Pet coke, untreated | 0 | 60.94 | 37.35 | **1.71** | 3.59 |
| 7 | BB + 3% HV thermal coal no FSI + 3% Pet coke, bs | 14 | 56.14 | 43.19 | 0.67 | 5.37 |
| 7 | BB + 3% HV thermal coal no FSI + 3% Pet coke, bs | 27 | 60.06 | 39.17 | 0.78 | 4.59 |

**Table 22: Pore area, Matrix A, B and coke area for untreated coke and after BF simulating tests mixed with MPBO iron ore pellets. BB=base blend**

| No. | Coke type | Coke wt. loss, % | Pores/dark phases, % | Matrix A, % | Matrix B, % | Coke area, $mm^2$ |
|---|---|---|---|---|---|---|
| 1 | Base blend, untreated | 0 | 57.79 | 41.77 | 0.44 | 3.47 |
| 1, K1743 | BB mixed with MPBO | 0.97 | 59.28 | 40.47 | 0.25 | 4.46 |
| 2A | BB + 5% HV thermal coal no FSI, untreated | 0 | 56.35 | 43.17 | 0.21 | 4.77 |
| 2A, K1913 | BB + 5% HV thermal coal no FSI, mixed with MPBO | **4.12** | 61.77 | 37.77 | 0.46 | 4.67 |
| 2A, K2101 | BB + 5% HV thermal coal no FSI, mixed with MPBO | 0.94 | 60.15 | 39.5 | 0.36 | 4.30 |
| 5 | BB +3% Pet coke + 3% Charcoal, untreated | 0 | 62.35 | 37.28 | 0.38 | 3.72 |
| 5, K2194 | BB+3% Pet coke + 3% Charcoal, mixed with MPBO | 1.42 | 59.85 | 39.8 | 0.35 | 4.48 |
| 7 | BB +3% HV thermal coal no FSI + 3% Pet coke, untreated | 0 | 60.94 | 37.35 | **1.71** | 3.59 |
| 7, K1996 | BB + 3% HV thermal coal no FSI+ 3% Pet coke, mixed with MPBO | 1.04 | 60.42 | 38.08 | **1.50** | 4.28 |

Looking at the column of the pore area (Pores/dark phases, %), there seem not to be any obvious difference between untreated samples and samples reacted in the BF shaft or in the BF simulating tests. The variation between the cokes itself may influence the results more than changes in pore area as a result of gasification.

A note is given to basket sample No. 2A Layer 21, untreated sample No. 7 and No.7 reacted in the BF simulating tests TRT Mas K1996, see Table 21 for basket samples from the EBF® and Table 22 TRT Mas test samples, due to an increased percentage of Matrix B phase. The later one is the samples used for describing the method. According to SEM investigation, Matrix B is related to S rich areas. The high weight loss of 4% obtained for one of the BF simulating tests with coke type 2A (K1913), see Figure 101, is most probably not valid as the changes in pore area and coke area is comparable small. Therefore should the weight loss in this sample be around 0.9 % instead of 4.1 %.

*Pore size distribution, Method 2*

The mosaic picture was imported into Leica LAS.V4 software that is able to identify individual pores and enables pore size distribution analysis. The LAS.V4 program prompts the user to adjust the grayscale to identify which part of the sample are pores; the program then uses the non-pore segments to separate the pores into individuals which are measured for area, size dimensions, roundness and equivalent diameter. For technical reasons a limit is set for the smallest measureable pore (6400 $\mu m^2$) and all pores larger than 1 500 000 $\mu m^2$ are considered oversized outliers and are excluded. Figure 65 shows how, similar to Method 1, that the greyscale (left side) is adjusted to only highlight the pores. This highlighting is shown to the right side where the program has identified and given individual identifiers to the pores.

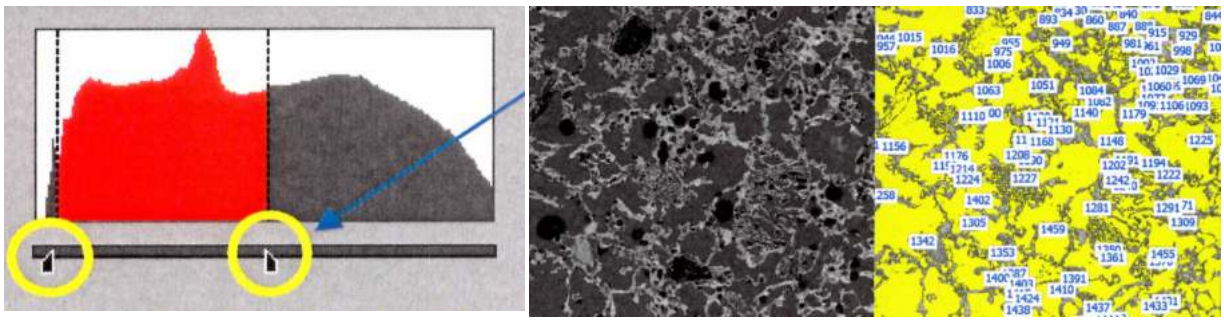


**Figure 65: Greyscale pixel histogram with selected pore segment (left) and an example of the picture in greyscale and using highlighted pores with individual markings (right)**

Table 23 show the pore percentage of the analysed coke area, this fraction is significantly smaller than from the one in Method 1, and subsequently, the coke area are much higher. The smaller pore % is due to the limitations and having to sort out all pores smaller than 6400 $\mu m^2$and the oversized ones. The normalised no. of pores shows that the pores counted are as few as 2-5 pores per $mm^2$.

Figure 66 show, as an example, the number of pores in the different size classes for the base blend untreated sample. As the number of pores decrease significantly when the pore sizes increases the larger pores could be excluded. Figure 67 show the different samples for the base blend (Layer 14, 21 and the TRT MAS sample) and how the different size classes of pores are distributed. It can be seen that the different samples follow each other quite closely; they do sort themselves such that the untreated sample have the largest number of small pores and the TRT MAS sample have the least. This sorting is only coincidental as no other coke blend show the same order.

The reacting gas would affect the smallest pores first as they give the largest surface area at these points. The problem with the analysis performed is that the limitations are that it is not possible to analyse the differences at the relevant scale. As Method 2 only catch 30-50% of the total amount of pores (as analysed by Method 1) it is clear that too much information gets lost to make a clear analysis of the data and therefore it is not suitable to use Method 2.

**Table 23: Calculated pore % and pore count information. BB=base blend, TRT MAS=blast furnace simulation tests –coke mixed with MPBO pellets**

| No. | Coke type | Basket layer No. | Pores, % | Wt. loss, % | Tot. No. of pores | Coke area, mm² | Norm. No. of pores, 1/mm² |
|---|---|---|---|---|---|---|---|
| **1** | Base blend, untreated | 0 | 14.65 % | | 1582 | 560 | 2.82 |
| **1** | Base blend, bs | 14 | 23.24 % | | 2634 | 550 | 4.79 |
| **1** | Base blend, bs | 21 | 25.87 % | | 2221 | 474 | 4.69 |
| **1** | Base blend, TRT MAS | 0 | 24.03 % | 0.97 | 2332 | 546 | 4.27 |
| **2A** | BB + 5% HV thermal coalno FSI, untreated | 0 | 25.01 % | | 2788 | 597 | 4.67 |
| **2A** | BB + 5% HV thermal coal no FSI, bs | 21 | 18.84 % | | 2472 | 628 | 3.94 |
| **2A** | BB + 5% HV thermal coal no FSI, bs | 29 | 15.24 % | | 2076 | 668 | 3.11 |
| **2A** | BB + 5% HV thermal coal no FSI, TRT MAS | 0 | 16.04 % | 4.12 | 2041 | 703 | 2.90 |
| **2A** | BB + 5% HV thermal coal no FSI, TRT MAS | 0 | 20.56 % | 0.94 | 2066 | 530 | 3.90 |
| **5** | BB +3% Pet coke +3% Charcoal, untreated | 0 | 13.47 % | | 1185 | 438 | 2.71 |
| **5** | BB + 3% Pet coke + 3% Charcoal, bs | 14 | 13.67 % | | 1720 | 597 | 2.88 |
| **5** | BB + 3% Pet coke + 3% Charcoal, bs | 27 | 22.31 % | | 1979 | 459 | 4.31 |
| **5** | BB + 3% Pet coke + 3% Charcoal, bs | 29 | 14.69 % | | 1511 | 546 | 2.77 |
| **5** | BB + 3% Pet coke + 3% Charcoal, TRT MAS | 0 | 17.84 % | 1.42 | 1965 | 588 | 3.34 |
| **7** | BB + 3% HV TC no FSI + 3% Pet coke, untreated | 0 | 20.11 % | | 1551 | 437 | 3.55 |
| **7** | BB + 3% HV thermal coal no FSI + 3% Pet coke, bs | 14 | 23.30 % | | 3171 | 636 | 4.98 |
| **7** | BB + 3% HV thermal coal no FSI + 3% Pet coke, bs | 27 | 13.85 % | | 1686 | 676 | 2.49 |
| **7** | BB + 3% HV thermal coal no FSI + 3% Pet coke, TRT MAS | 0 | 20.32 % | 1.04 | 1996 | 512 | 3.90 |

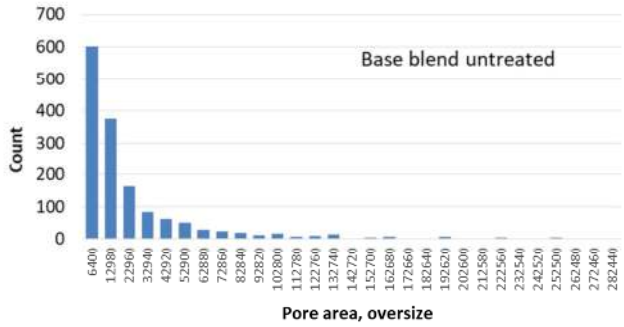



**Figure 66: Pore size histogram for untreated base blend coke**

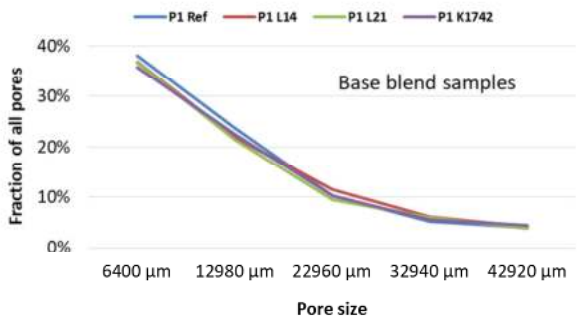



**Figure 67: Pore fractions for base blend samples**

### Evaluation description of coke graphitisation degree, Lc, and temperature estimation

Coke, coal and char are normally relatively amorphous sources of carbon. A higher degree of carbonisation during coking produces a more crystal coke structure, i.e. higher graphitisation degree. Graphitisation of the coke in the BF takes place in the high-temperature zone, i.e. when the coke is subjected to higher temperature than the coking temperature. The graphite structure consists of large sections of ordered carbon planes on top of each other. The highly ordered graphite structure can be described on the basis of the parameters Lc and La, which define the level of crystallinity. Lc represents the average height of the crystallite and La is the average length. Lc of graphite can be in the order of 100–300 Å and crystalline carbon in coal and coke can be considered to have a graphite type of structure, where the aromatic structures can align in a similar manner as graphite layers. The crystallites are less ordered over a shorter distance; about 2–30 Å.

Lc is determined by application of Scherrer's equation on the (002) carbon X-ray diffraction (XRD) peak:

$$L_c = \frac{0.89\lambda}{\beta \cos\theta}$$

where λ is the wavelength of the X-ray source, β is the full width at half maximum (FWHM) and θ is the corresponding Bragg angle. A sharper (002) peak corresponds to a higher Lc value, and represents a higher degree of ordering/graphitisation in the carbon structure. The development of crystalline order in coke structure could be used as a thermal signature to determine its history, i.e. the highest temperature experienced by the coke in the blast furnace.

The coke graphitisation evaluation method has been further developed during the course of the FLEXCOKE project. Earlier[9] the fitting of the coke peaks have been mostly by Gaussian distribution and now the algorithm carries out 4 different calculation methods of the Lc-value, the average of these are assumed to approximate the true Lc-value. Compared to previous method, the Lc values are lower with the new method but follow trends. The temperature study in the Tamman furnace have been converted into the new algorithms and the Lc versus temperature linear correlation was converted into an exponential curve fitting, see Figure 68. Previous study had a linear temperature fitting.

The results of graphitisation degrees are presented in each section when made, i.e. in Task 3.1 for the solid samples taken from the high temperature area of the EBF®, In Task 3.2 for the coke in basket samples excavated after EBF® campaigns 31 and 32 and in Task 4.2 for shaft excavation samples.

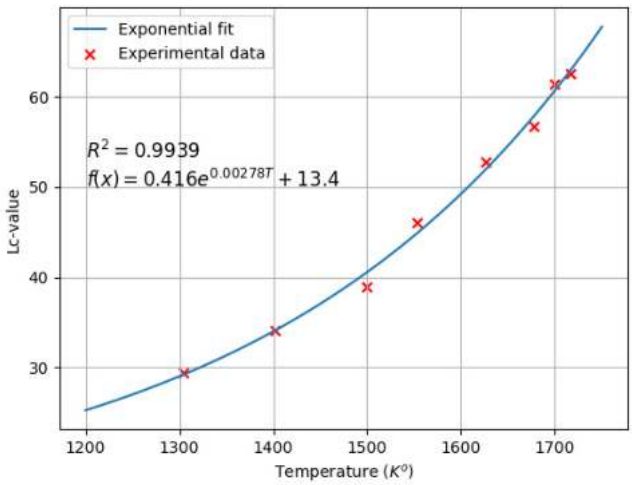



**Figure 68: Experimental data [9] of Lc versus temperature in the new method**

Conclusions

Although the alternative C-containing material behave distinct under polarized light (pet coke, anthracite, HV thermal coal with FSI – anisotropic; charcoal, torrefied wood at medium and high temperature, HV thermal coal no FSI – isotropic), the addition of alternative C-materials to the base blend always increases the coke isotropy. Only a slight impact on coke texture has been observed for the blends with the addition of 5% anthracite and 3% charcoal respectively.

No clear trend can be concluded for the basket samples charged and excavated in the EBF®. Most coke samples became more anisotropic, some more isotropic and some did not change. The change in the coke texture after standard CRI/CSR test was more significant than after reacting in the EBF® shaft. Here, for all samples a significant increase in anisotropy has been determined.

During the basket sample descent in the EBF® some anisotropic phases probably reacted to higher anisotropic phases and some isotropic to weak anisotropic phases, but there is only a slight change in the value of isotropic- and anisotropic total. However in the standard CRI/CSR test, the isotropic phases react mostly to weak anisotropic phases, increasing the coke anisotropy.

The study of gasification behaviour using LOM measuring pores could not reveal any clear differences between untreated samples and samples reacted in the BF shaft (EBF® basket samples) or in the BF simulating tests. The variation between the cokes itself may influence the results more than changes in pore area as a result of gasification.

Exploitation and impact of the research results

The forming microstructure in coke is a result of the properties of each single coal and also of the coking parameters. Therefore the coal quality and mode of operation of a coke plant has strong impact on directly the ratio between anisotropic and isotropic components.

The BF size has increased since the past and the rate of PCI has been maximised over the years. With increasing PCI rate, the amount of charged coke per ton hot metal has been decreased and its residence time in the BF increased, as the coke carbon is partly replaced by the carbon from the PC. Therefore quality demand on blast furnace coke especially on CSR has increased. A higher CSR is desired, to ensure the drainage for liquids phases and the gas flow as well the support for the burden. Coke with higher amount of isotropic phases tends to have lower CSR values since the isotropic phases react more intensively with $CO_2$ in the CRI/CSR test, reducing the hot strength (CSR) of the coke.

Although some alternative C-containing material behaves anisotropically (pet coke, anthracite, HV thermal coal with FSI) and some behaves isotropically (charcoal, torrefied wood at medium and high temperature, HV thermal coal no FSI) under polarized light, the coke texture of the produced coke showed that the addition of these C-containing materials to the base blend mainly increases the isotropic phases and decrease the anisotropic total at the same time. This means that the alternative C-containing material deteriorate the coke quality by increasing the CRI and decreasing the CSR.

Therefore not only the investigation of the microstructure from single coals and the alternative C-containing material is important but also from the coal blend as the single materials interact and optical behaviour is significantly influenced.

### *Task 2.3 Ash amount and composition*

Planned activities and work accomplished

Ash amount and composition of 10 coke samples gained from the technical (10 kg retort) and 15 samples gained from the pilot tests (500 kg semi-industrial oven) were determined. Changes in coke ash and composition of basket samples after EBF® trial C31 and after CRI/CSR test were analysed by tk SE, in total 58 samples were analysed. The results of the basket sample evaluation are reported in WP3, Task 3.2, and in Task 2.2 regarding coke micro structure. Selected samples collected at SSAB full scale trials (Task 4.1 and Task 4.3) and from EBF® trial (Task 4.2) were analysed by SSAB and MEFOS.

Description of activities, results and discussion

**Ash amount and composition of technical- and pilot trial - 10 kg retort and 500 kg semi-industrial oven tests**

In the carbonisation tests at DMT 10 coke types were produced in the 10 kg retort and 15 coke types in two campaigns in the 500 kg oven. 10 coke types during the first campaign and 5 during the second campaign. The base blend material was the same for the 10 kg retort tests and the first trial, at the 500 kg semi-industrial oven (40% LV coal; 32% MV coal; 28% HV coal). During the second trial new batches of coking coals were used.

Ash amount and ash analysis of the DMT samples are shown in Figure 69 and Figure 70 and in Table 24 and Table 25. For the 10 kg retort tests, Figure 69 and Table 24, it can be summarised that adding only thermal coal or thermal coal together with anthracite to the base blend always increased the ash content whereas the addition of the other alternative C-containing material (anthracite, charcoal, torrefied wood and thermal coal in combination with alternative C-containing material) always decreased the ash content. The lowest ash content with 10.8 % was observed for coal blend 9 where 5% thermal coal (no FSI) + 5% pet coke was added. This is understandable as pet coke has almost no ash. Regarding the ash composition it can be stated that no significant changes compared to the base blend coke ash are visible. Dominant mineral compound is in all coke samples $SiO_2$ and $Al_2O_3$.

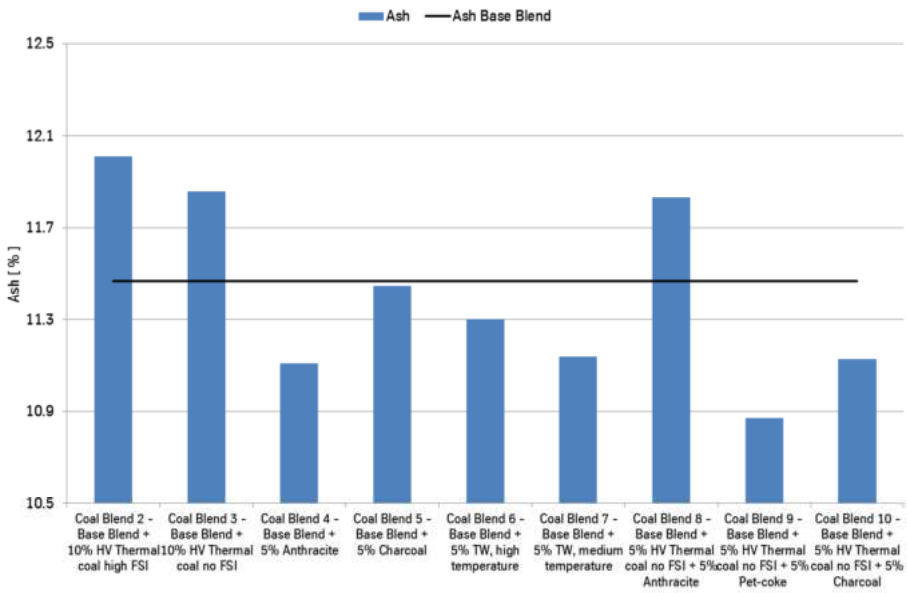



**Figure 69: Ash content - 10 kg retort tests**

**Table 24: Ash composition – samples 10 kg retort tests**

| | No. | Coal Blend | Ash % | Ash composition in % (dry basis) | | | | | | | | | | | Ash basicity |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Fe | $SiO_2$ | $Al_2O_3$ | MgO | CaO | $TiO_2$ | S | $Na_2O$ | $K_2O$ | Mn | P | B1 |
| Technical trial - 10 kg retort | 1 | Base blend | 11.5 | 3.44 | 61.50 | 26.10 | 0.51 | 1.22 | 1.52 | 0.04 | 0.40 | 1.25 | 0.03 | 0.17 | 0.02 |
| | 2 | Base blend + 10 % HV thermal coal high FSI | 12.0 | 3.72 | 59.70 | 25.30 | 0.84 | 2.08 | 1.48 | 0.14 | 0.70 | 1.31 | 0.02 | 0.17 | 0.03 |
| | 3 | Base blend + 10 %HV thermal coa lno FSI | 11.9 | 3.52 | 60.40 | 26.20 | 0.63 | 1.50 | 1.46 | 0.04 | 0.40 | 1.33 | 0.03 | 0.20 | 0.02 |
| | 4 | Base blend + 5 % anthracite | 11.1 | 3.38 | 60.50 | 26.30 | 0.54 | 1.34 | 1.49 | 0.03 | 0.50 | 1.29 | 0.03 | 0.20 | 0.02 |
| | 5 | Base blend + 5 % charcoal | 11.5 | 3.18 | 61.60 | 25.80 | 0.52 | 1.33 | 1.52 | 0.03 | 0.40 | 1.24 | 0.04 | 0.18 | 0.02 |
| | 6 | Base blend + 5 % torrified wood, high temperature | 11.3 | 3.14 | 61.00 | 26.30 | 0.56 | 1.41 | 1.51 | 0.03 | 0.50 | 1.34 | 0.05 | 0.18 | 0.02 |
| | 7 | Base blend + 5 % torrified wood, medium temperature | 11.1 | 3.40 | 60.60 | 26.40 | 0.56 | 1.38 | 1.52 | 0.02 | 0.40 | 1.31 | 0.05 | 0.19 | 0.02 |
| | 8 | Base blend + 5 % HV thermal coal + 5 % anthracite | 11.8 | 3.40 | 60.70 | 25.50 | 0.58 | 1.37 | 1.43 | 0.02 | 0.40 | 1.35 | 0.03 | 0.19 | 0.02 |
| | 9 | Base blend + 5 % HV thermal coal + 5 % pet coke | 10.9 | 3.55 | 60.60 | 25.90 | 0.57 | 1.46 | 1.48 | 0.02 | 0.40 | 1.29 | 0.03 | 0.20 | 0.02 |
| | 10 | Base blend + 5 % HV thermal coal + 5 % charcoal | 11.1 | 3.52 | 59.90 | 25.80 | 0.62 | 1.71 | 1.41 | 0.02 | 0.40 | 1.32 | 0.04 | 0.23 | 0.03 |

As in the reort tests, the ash content of the coke samples gained from the first trial at the 500 kg semi-industrial oven shows that the addition of thermal coal significantly increases the ash content, see Figure 70. Not only for the blends where only thermal coal was added but also in conjunction with other alternative C-containing material (except for the blend where thermal coal was added together with petrol coke). However, the resulting ash content for the blend where 5% anthracite was added is lower compared to the coke ash content where 3% anthracite was added and this appears not to be logical.

Table 25 shows the ash composition of the single coke types from the first semi-industrial trial. Here again no significant changes compared to the base blend are visible.

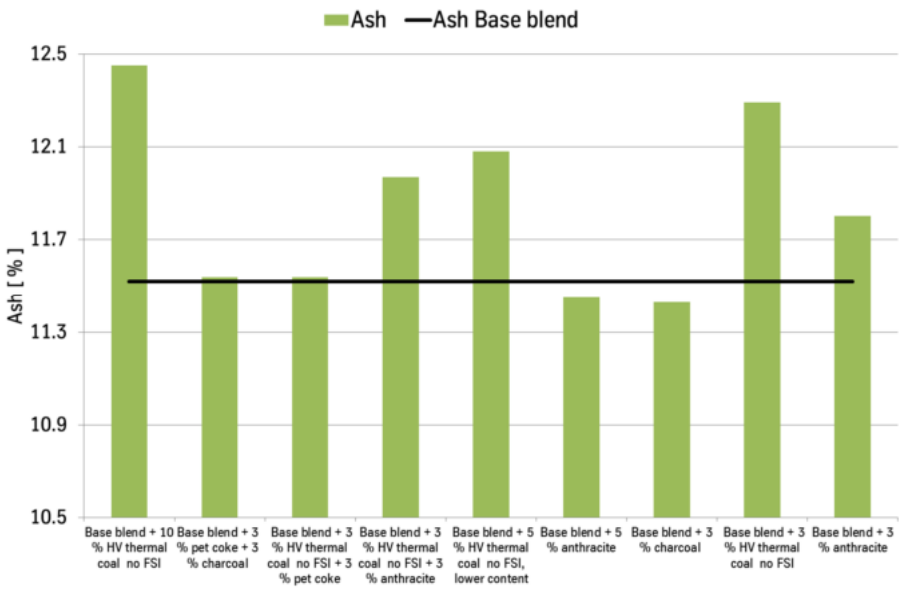



**Figure 70: Ash content – first semi-industrial trial**

**Table 25: Ash composition – samples from first semi-industrial trial**

| | No. | Coal Blend | Ash [ % ] | Ash composition in [ % ] | | | | | | | | | | | Ash basicity |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Fe | $SiO_2$ | $Al_2O_3$ | MgO | CaO | $TiO_2$ | S | $Na_2O$ | $K_2O$ | Mn | P | B1 |
| Pilot trial - 500 kg oven (1st campaign) | 1 | Base blend | 11.52 | 3.47 | 60.00 | 28.00 | 0.61 | 1.21 | 1.54 | 0.11 | 0.40 | 1.29 | 0.03 | 0.15 | 0.02 |
| | 2 | Base blend + 10 % HV thermal coal no FSI | 12.45 | 3.48 | 59.70 | 26.80 | 0.84 | 1.77 | 1.42 | 0.18 | 0.50 | 1.53 | 0.03 | 0.16 | 0.03 |
| | 2A | Base blend + 5 % HV thermal coal no FSI, lower content | 12.08 | 3.29 | 60.50 | 27.80 | 0.79 | 1.45 | 1.51 | 0.09 | 0.50 | 1.45 | 0.03 | 0.14 | 0.02 |
| | 3 | Base blend + 5 % anthracite | 11.45 | 3.18 | 60.20 | 27.90 | 0.62 | 1.20 | 1.51 | 0.07 | 0.40 | 1.32 | 0.03 | 0.14 | 0.02 |
| | 4 | Base blend + 3 % charcoal | 11.43 | 3.28 | 60.20 | 28.10 | 0.67 | 1.51 | 1.51 | 0.10 | 0.50 | 1.43 | 0.04 | 0.15 | 0.03 |
| | 5 | Base blend + 3 % pet coke + 3 % charcoal | 11.54 | 3.24 | 60.20 | 28.20 | 0.75 | 1.56 | 1.51 | 0.11 | 0.40 | 1.51 | 0.04 | 0.15 | 0.03 |
| | 7 | Base blend + 3 % HV thermal coal no FSI + 3 % pet coke | 11.54 | 3.43 | 59.70 | 27.50 | 0.77 | 1.40 | 1.51 | 0.08 | 0.50 | 1.42 | 0.03 | 0.14 | 0.02 |
| | 8 | Base blend + 3 % HV thermal coal no FSI + 3 % anthracite | 11.97 | 4.07 | 59.50 | 27.40 | 0.80 | 1.38 | 1.46 | 0.06 | 0.40 | 1.44 | 0.03 | 0.15 | 0.02 |
| | 9 | Base blend + 3 % HV thermal coal no FSI | 12.29 | 3.47 | 60.50 | 27.40 | 0.72 | 1.25 | 1.49 | 0.05 | 0.40 | 1.39 | 0.04 | 0.13 | 0.02 |
| | 10 | Base blend + 3 % anthracite | 11.80 | 3.25 | 60.50 | 27.90 | 0.67 | 1.27 | 1.51 | 0.07 | 0.40 | 1.33 | 0.03 | 0.15 | 0.02 |

The coke ash analyses of the coke produced in the second trial at the 500 kg oven are shown in Figure 71 and Table 26.

Adding biomass (torrefied wood or charcoal) or anthracite always led to lower ash contents compared to the base blend. Although anthracite has higher ash content (11.3 %) in comparison to charcoal (0.9 %) the coke ash content was very similar. The lowest impact on ash content shows the blend with the addition of 2% torrefied wood. The other tested blends show a stronger decrease in ash content.

Regarding the ash composition it can be summarised that in general no big differences were found. A visible change can only be observed for the blend where charcoal was added. Here the Fe content in the ash increased and the $SiO_2$ content decreased.

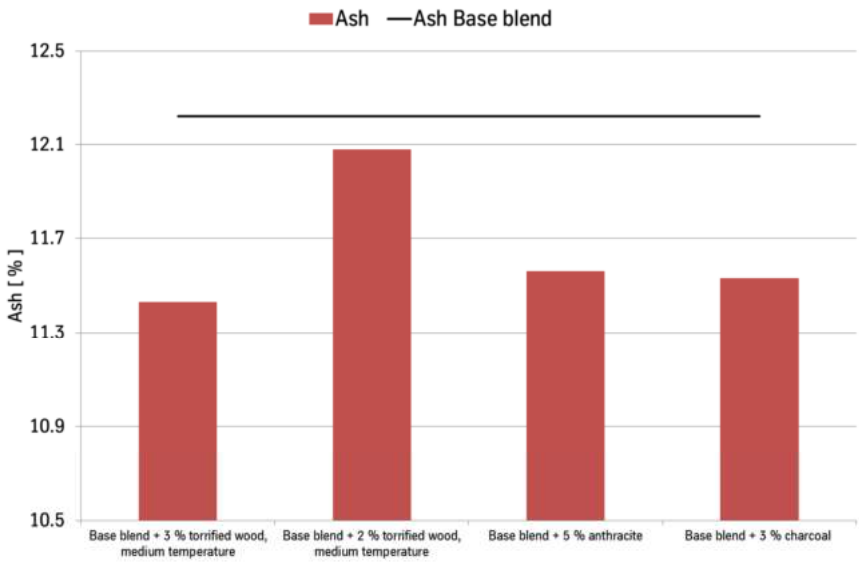



**Figure 71: Ash content – second semi-industrial trial**

**Table 26: Ash composition - samples from second semi-industrial trial**

| | No. | Coal Blend | Ash [ % ] | Ash composition in [ % ] | | | | | | | | | | | Ash basicity |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | Fe | $SiO_2$ | $Al_2O_3$ | MgO | CaO | $TiO_2$ | S | $Na_2O$ | $K_2O$ | Mn | P | B1 |
| Pilot trial - 500 kg oven (2nd campaign) | I | Base Blend | 12.22 | 3.02 | 59.70 | 29.80 | 0.45 | 1.05 | 1.51 | 0.06 | 0.30 | 1.34 | 0.03 | 0.18 | 0.02 |
| | II | Base blend + 3 % torrified wood, medium temperature | 11.43 | 3.11 | 59.10 | 29.90 | 0.47 | 1.17 | 1.60 | 0.04 | 0.50 | 1.31 | 0.04 | 0.18 | 0.02 |
| | III | Base blend + 2 % torrified wood, medium temperature | 12.08 | 2.98 | 59.20 | 29.70 | 0.46 | 1.10 | 1.49 | 0.05 | 0.40 | 1.36 | 0.03 | 0.18 | 0.02 |
| | IV | Base blend + 5 % anthracite | 11.56 | 3.16 | 59.10 | 29.20 | 0.52 | 1.06 | 1.56 | 0.04 | 0.40 | 1.38 | 0.03 | 0.17 | 0.02 |
| | V | Base blend + 3 % charcoal | 11.53 | 3.93 | 57.90 | 29.10 | 0.58 | 1.55 | 1.52 | 0.11 | 0.40 | 1.34 | 0.05 | 0.19 | 0.03 |

From former investigation it is known that some ash components such as Fe, CaO and MgO may influence the coke reactivity, due to their catalytic effect (reducing the temperature for the solution loss reaction). The evaluation shows a good correlation for the results gained from the first trial campaign at the 500 kg semi-industrial oven, see Figure 72.

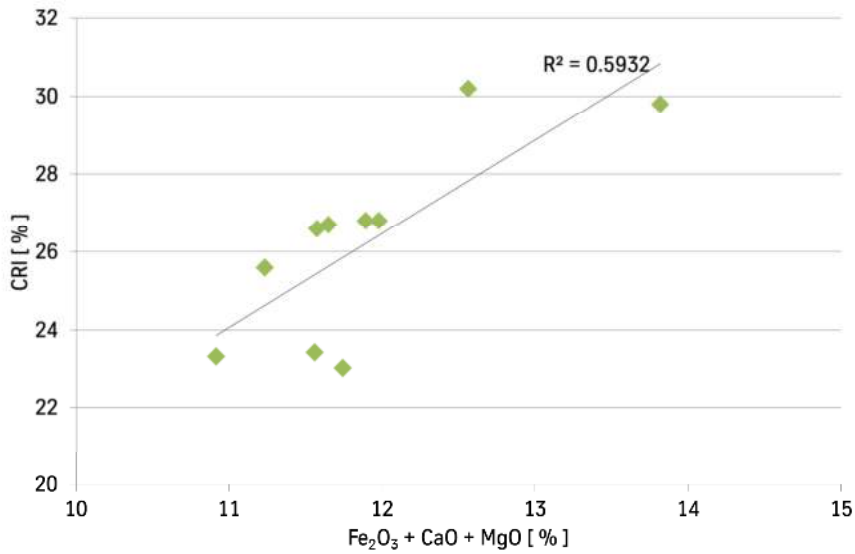



**Figure 72: $Fe_2O_3$, CaO, MgO in ash vs. coke reactivity – 500 kg first semi-industrial trial**

No correlation was determined for the tests at the 10 kg retort and for the second trial campaign of the 500 kg semi-industrial oven. It is assumed that the 10 kg retort does not provide reliable CRI values (only trends) allowing correlations and the number of tests during the second campaign was too low to have a sufficient database for statistical evaluation.

**Ash amount and composition of samples tested under standard CRI/CSR conditions – samples from DMT 500 kg semi-industrial oven**

The chemical analysis was done after performing CRI/CSR test and the variation ash content and composition are presented in Figure 73 and in Table 27. Due to the solution loss reaction the C content decreases and consequently ash amount increases in all cases. The change is more notable in comparison to the EBF® basket samples (Figure 146), as in the CRI/CSR test the coke is attacked by gas with 100% $CO_2$. The most significant impact on ash content with 4.3% was observed sample No. 2 (base blend + 10% thermal coal no FSI). Different from the EBF® basket samples, no specific changes in the ash composition can be observed.

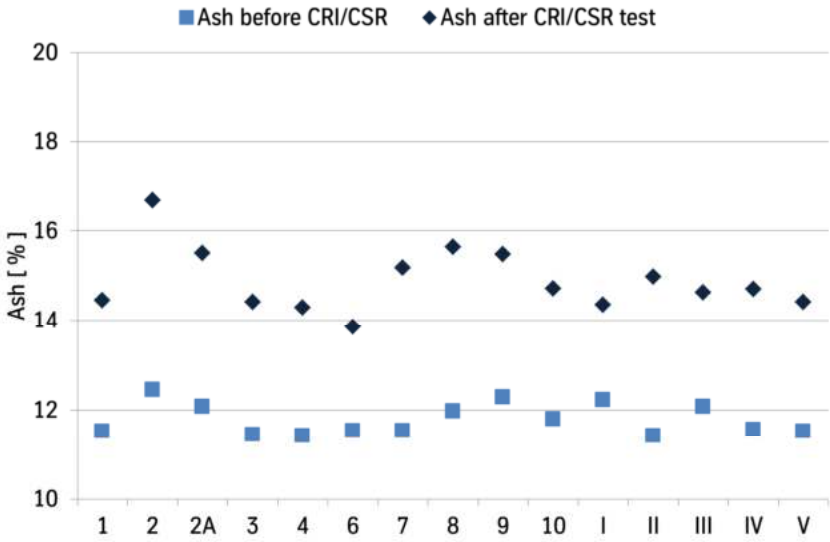



**Figure 73: Ash content before and after CRI/CSR test**

**Table 27: Ash composition before and after CRI/CSR test**

| | Fe before CRI/CSR test | Fe after CRI/CSR test | $SiO_2$ before CRI/CSR test | $SiO_2$ after CRI/CSR test | $Al_2O_3$ before CRI/CSR test | $Al_2O_3$ after CRI/CSR test | MgO before CRI/CSR test | MgO after CRI/CSR test | CaO before CRI/CSR test | CaO after CRI/CSR test |
|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 3.5 | 3.6 | 60.0 | 59.2 | 28.0 | 27.6 | 0.6 | 0.4 | 1.2 | 1.1 |
| 2 | 3.5 | 3.4 | 59.7 | 58.9 | 26.8 | 26.6 | 0.8 | 0.6 | 1.8 | 1.5 |
| 2A | 3.3 | 3.8 | 60.5 | 58.9 | 27.8 | 27.2 | 0.8 | 0.5 | 1.5 | 1.4 |
| 3 | 3.2 | 3.4 | 60.2 | 58.5 | 27.9 | 27.7 | 0.6 | 0.5 | 1.2 | 1.3 |
| 4 | 3.3 | 3.3 | 60.2 | 59.1 | 28.1 | 27.2 | 0.7 | 0.5 | 1.5 | 1.4 |
| 5 | 3.2 | 2.9 | 60.2 | 60.1 | 28.2 | 27.9 | 0.8 | 0.5 | 1.6 | 1.5 |
| 7 | 3.4 | 3.5 | 59.7 | 59.3 | 27.5 | 27.3 | 0.8 | 0.5 | 1.4 | 1.4 |
| 8 | 4.1 | 3.6 | 59.5 | 58.7 | 27.4 | 26.8 | 0.8 | 0.5 | 1.4 | 1.3 |
| 9 | 3.5 | 3.5 | 60.5 | 58.8 | 27.4 | 27.2 | 0.7 | 0.5 | 1.3 | 1.2 |
| 10 | 3.3 | 3.3 | 60.5 | 59.6 | 27.9 | 27.7 | 0.7 | 0.4 | 1.3 | 1.2 |
| I | 3.0 | 3.4 | 59.7 | 57.5 | 29.8 | 29.1 | 0.5 | 0.5 | 1.1 | 1.0 |
| II | 3.1 | 2.9 | 59.1 | 58.2 | 29.9 | 29.7 | 0.5 | 0.4 | 1.2 | 1.1 |
| III | 3.0 | 3.2 | 59.2 | 58.2 | 29.7 | 29.5 | 0.5 | 0.5 | 1.1 | 1.1 |
| IV | 3.2 | 3.0 | 59.1 | 58.5 | 29.2 | 29.4 | 0.5 | 0.5 | 1.1 | 1.0 |
| V | 3.9 | 3.0 | 57.9 | 58.4 | 29.1 | 29.5 | 0.6 | 0.5 | 1.6 | 1.2 |

**Ash amount and composition of SSAB industrial trials in WP4, Task 4.1**

The 1st SSAB industrial cokemaking trial produced alternative coke for EBF® operational trials in Task 4.2. Two types of *a*lternative coke to be tested in LKAB EBF® were produced in a couple of coke ovens at SSAB coke plant in Luleå. The ash contents and chemical compositions were made at SSAB laboratory.

The average values of the ash contents and chemical analysis made by X-Ray Fluorescence (XRF) are shown in Table 28 which also indicates standard deviations. TC1, with 5% thermal coal addition, has a bit higher ash content and subsequently higher amounts of oxides compared to standard coke.

TC2, with 3% thermal coal and 3% pet coke addition had similar ash content as standard coke but a bit higher S, $P_2O_5$ and CaO contents. The higher S content in TC2 is derived from the pet coke which has an S content of around 2.5%.

The VM is higher in the alternative test cokes compared to standard coke produced during the same week. The standard deviations are generally low, but $SiO_2$ and ash content shows higher variations.

**Table 28: Ash and VM content, and chemical composition (XRF analyses) of produced standard coke, TC1 and TC2 (wt.%). SSAB 1st cokemaking trial producing alternative coke for EBF® operational trial**

| | Fe | CaO | $SiO_2$ | MnO | $P_2O_5$ | S | $Al_2O_3$ | MgO | $Na_2O$ | $K_2O$ | $TiO_2$ | Ash | VM |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Standard coke** | **0.38** | **0.10** | **6.52** | **0.04** | **0.043** | **0.61** | **3.09** | **0.085** | **0.06** | **0.16** | **0.18** | **11.3** | **0.1** |
| Std. dev. | 0.00 | 0.01 | 0.16 | 0.00 | 0.00 | 0.03 | 0.08 | 0.01 | 0.00 | 0.00 | 0.00 | 0.2 | 0.1 |
| **TC1** | **0.44** | **0.14** | **6.84** | **0.04** | **0.049** | **0.59** | **3.20** | **0.094** | **0.07** | **0.16** | **0.18** | **11.8** | **0.7** |
| Std. dev. | 0.07 | 0.03 | 0.41 | 0.01 | 0.00 | 0.03 | 0.19 | 0.02 | 0.01 | 0.01 | 0.01 | 0.2 | 0.1 |
| **TC2** | **0.37** | **0.12** | **6.52** | **0.03** | **0.045** | **0.68** | **3.06** | **0.087** | **0.06** | **0.15** | **0.17** | **11.3** | **0.6** |
| Std. dev. | 0.02 | 0.04 | 0.12 | 0.00 | 0.00 | 0.01 | 0.04 | 0.02 | 0.00 | 0.01 | 0.00 | 0.4 | 0.2 |

For the SSAB 2nd full scale cokemaking trial producing coke for full scale trials at SSAB BF No. 3 in Luleå (Task 4.3) it was decided within the project group to go forward with TC1, i.e. 5% addition of thermal coal to the blend, during the full scale operational trial. It was however not possible to purchase the same type of thermal coal which was used in the EBF® trial for the large scale industrial trial but The same type of thermal coal which was used in the EBF trial could not be purchased for the large scale industrial trial but the thermal coals were quite similar. Therefore the coke is named FLEXCOKE trial coke (FTC). Differences were that the thermal coal used in the 2nd cokemaking trial had a bit higher ash, $SiO_2$, $Al_2O_3$ and alkali content than TC1 produced and tested in the EBF® trial which had a bit higher contents of VM, S, CaO, MgO and $P_2O_5$. Ash analyses of alternative coke produced in the full scale trial at the coking plant and used in full scale BF trial were made by SSAB.

Average values and standard deviations of the ash contents and chemical analysis are shown in Table 29. In addition, calculated values of the mixes of FTC and standard coke during ramping up in the industrial trials are presented in the table. Ramping up with alternative coke was made by mixing 20, 40, 80 and 100 % of FTC to the standard coke. As seen in the table, the standard, reference coke has quite similar ash, VM and chemical composition as the TFC.

**Table 29: Ash and VM content, and chemical composition (XRF analyses) of produced standard coke and FTC with 5% addition of thermal coal (wt.%) in SSAB 2nd cokemaking trial producing alternative coke for full scale BF trials at BF No. 3. Calculated analysis made on 20, 40 and 80 % mixing with standard coke during ramping up in the BF**

| | Fe | CaO | $SiO_2$ | $P_2O_5$ | S | $Al_2O_3$ | MgO | $Na_2O$ | $K_2O$ | $TiO_2$ | Ash | VM |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| **Reference coke** | **0.37** | **0.09** | **7.19** | **0.051** | **0.56** | **3.20** | **0.08** | **0.05** | **0.15** | **0.18** | **11.99** | **0.75** |
| Std. dev. | 0.04 | 0.04 | 0.34 | 0.007 | 0.02 | 0.16 | 0.03 | 0.01 | 0.01 | 0.00 | 0.25 | 0.12 |
| **Ind. trial coke 5% thermal coal addition** | **0.36** | **0.09** | **7.26** | **0.045** | **0.55** | **3.20** | **0.08** | **0.05** | **0.15** | **0.18** | **11.93** | **0.76** |
| Std. dev. | 0.04 | 0.03 | 0.42 | 0.005 | 0.01 | 0.17 | 0.01 | 0.01 | 0.01 | 0.01 | 0.20 | 0.11 |
| ***Calculated 20% mix to the BF*** | *0.37* | *0.09* | *7.21* | *0.050* | *0.56* | *3.20* | *0.08* | *0.05* | *0.15* | *0.18* | *11.98* | *0.75* |
| ***Calculated 40% mix to the BF*** | *0.37* | *0.09* | *7.22* | *0.049* | *0.56* | *3.20* | *0.08* | *0.05* | *0.15* | *0.18* | *11.97* | *0.75* |
| ***Calculated 80% mix to the BF*** | *0.36* | *0.09* | *7.24* | *0.046* | *0.55* | *3.20* | *0.08* | *0.05* | *0.15* | *0.18* | *11.94* | *0.76* |

Conclusions

When charcoal, torrefied wood, pet coke or anthracite was added to the base blend the coke ash content decreased dependent on the ash content of specific thermal coal selected. Thermal coal significantly increases the ash content. This effect can be compensated by bringing a low ash component such as pet coke or biomaterial into the blend by selecting another type of thermal coal.

This was also seen in the 1st industrial produced alternative TC2 as the ash content was at the same level as standard coke while the addition of 5% thermal coal in TC1 increased the ash content. However, during the 2nd industrial trial the standard coke has similar ash content and chemical composition as the FTC. In this coke another type of thermal coal with lower ash was used and compared to the 1st cokemaking trial FLEXCOKE trial mix had higher amounts of MV coal in the mix and more of the HV coal was replaced by thermal coal, which contribute to the total ash content.

The C content decreased and the ash content increased for all tested alternative coke samples after reacting in CRI/CSR test. Comparing with baskets samples reacting in EBF®, reported in WP3, the change in ash content was more notable after CRI/CSR test due to the stronger solution loss reaction in pure $CO_2$.

Exploitation and impact of the research results

Blast furnace coke is an indispensable and cost-intensive raw material for the hot metal production. The characterisation regarding to its chemical composition is very important for the burden projection, since the content of carbon and ash as well its composition are parameters which influence directly the coke consumption. Also fluxes addition is affected and in turn the productivity and quality from hot metal and slag.

The gained results showed that mainly the selected thermal coal as raw material for cokemaking has a negative impact on the coke quality as ash content increases lowering the amount of C in coke. This negative impact can be compensated by parallel addition of a very low ash material as e.g. pet coke or biomass. All other alternative C-containing material did not show significant negative impact on coke ash amount/composition. However, not only the ash amount should be considered as also the effects on other parameter as for example mechanical strength, CRI and CSR and particle size distribution are important.

### *Task 2.4 Metallurgical testing*

Planned activities and work accomplished

Both standard methods, like CSR/CRI and alternatively designed test regimes, have been used for a realistic characterisation relative to each section of the BF. Innovative tests for reactivity and hot strength was developed and used by tk SE. A new CRI/CSR measuring procedure has been developed to simulate the BF treatment conditions combining the mechanical load of the lower part in a BF.

Selected coke samples together with pellets were tested in an existing BF simulating test equipment at LKAB using test conditions designed for correlation with the EBF®. Evaluation was made by LKAB in cooperation with MEFOS and collected samples compared with the basket samples regarding gasification and graphitisation. Thermal gravimetric analyses were made by MEFOS for characterisation of reaction kinetics and calculation of reaction rates in selected samples from retort tests at DMT and the results compared with standardised reactivity tests.

Description of activities, results and discussion

An extended test method for determination of coke reactivity and hot strength is developed and used by tk SE. It is considered to develop a new CRI/CSR measuring procedure to simulate the BF treatment conditions combining the mechanical load of the lower part in a BF with the $CO_2$ gasification at 1100 °C eventually up to a defined rate of gasification.

Standard coke reactivity (CRI) and hot strength (CSR) were determined for coke samples produced at DMT 500 kg semi-industrial oven and for Test Coke 1 (TC1) and Test Coke 2 (TC2) produced in SSAB coking plant during the 1st industrial trial.

For a more realistic coke characterisation, simulating conditions were adapted closer to a real blast furnace. For this purpose the new test facility, erected at tk SE, was used. The tests were carried

out with tk SE coke and with TC1 (base blend + 5% HV thermal coal no FSI) from the SSAB 1st industrial trial.

**Standard CRI and CSR results: coke samples from DMT 500 kg semi-industrial oven**

The CRI and CSR results for the coke samples gained from the DMT 500 kg semi-industrial oven trials are shown in Figure 74 and Figure 75. Considering the percentages of alternative C-containing materials, the coal blends were divided in four groups marked in different colours; red with 10%, dark blue with 6%, green with 5%, light blue with 3 % and purple with 2% of additives. The black dot represents the base blend. As already reported in WP 2, Task 2.1, the alternative C-containing materials always increased CRI which subsequently reduces the coke strength CSR.

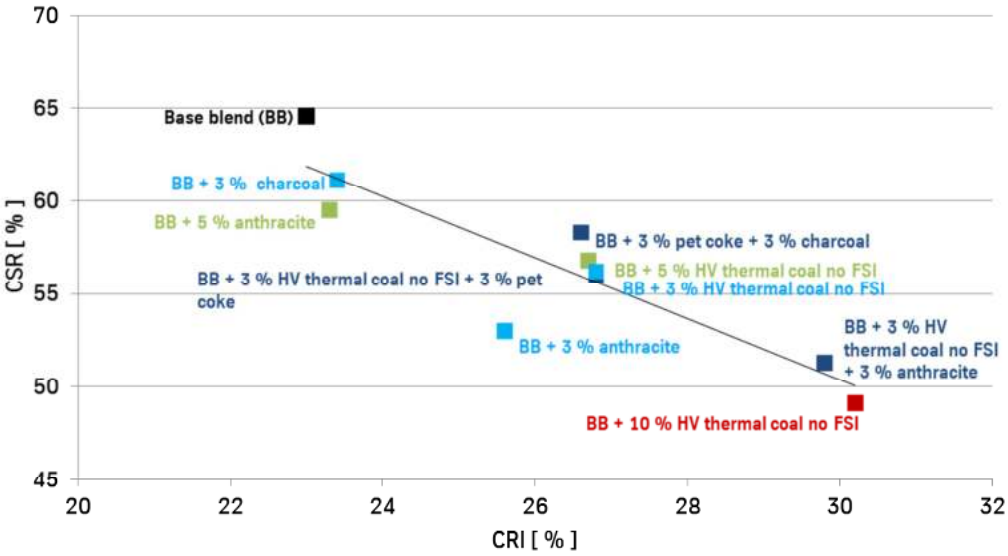



**Figure 74: Coke reactivity and coke strength after reaction – first semi-industrial trial**

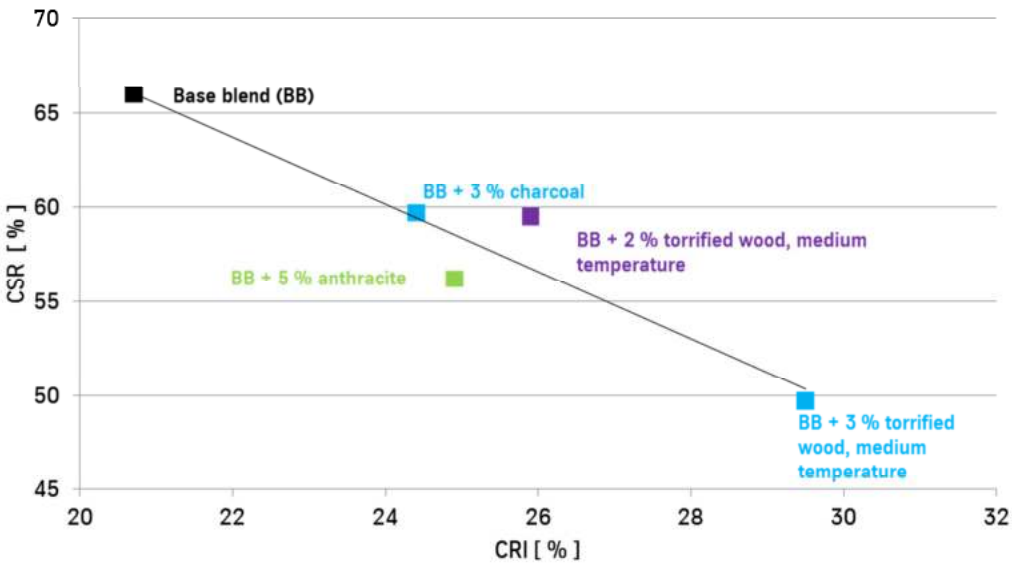



**Figure 75: Coke reactivity and coke strength after reaction – second semi-industrial trial**

In the first DMT trial the most significant impact on CRI and CSR can be observed for the blend with the addition of 10% HV thermal coal followed closely by the blend with the addition of 3% HV thermal coal together with 3% anthracite. With lower amount of thermal coal in the blend the negative impact on CRI and CSR is less. A group of quite similar CRIs and CSRs were determined for the blends with 3 or 5 % addition of HV thermal coal and for the blends where 3% pet coke was added together with either 3% charcoal or 3% thermal coal. The addition of charcoal led to slightly higher CSR values.

Considering the second DMT trial, a considerable effect could also be observed. Especially the addition of torrefied wood showed significant deterioration of coke reactivity and hot strength. The lowest CSR and highest CRI were determined for the blend with the addition of 3% torrefied wood. Although three recipes were repeated in the second trial, the results for CRI and CSR differ. An explanation for this can be the fact that a new batch of base blend coals has been used and resulting in a slightly different quality causing the differences with respect to CRI and CSR.

**Standard CRI and CSR results – coke samples from SSAB 1st industrial trial**

The CRI/CSR results tested at tk SE from the SSAB TC1 and TC2 samples are shown in Figure 76. For each blend three coke samples were tested. Although the two blend recipes are different, the CRI mean values for the produced coke are very similar, 20.8 % for TC1 and 20.5 % for TC2. Concerning CSR mean values the difference between both blends is slightly higher, 1.9 %.

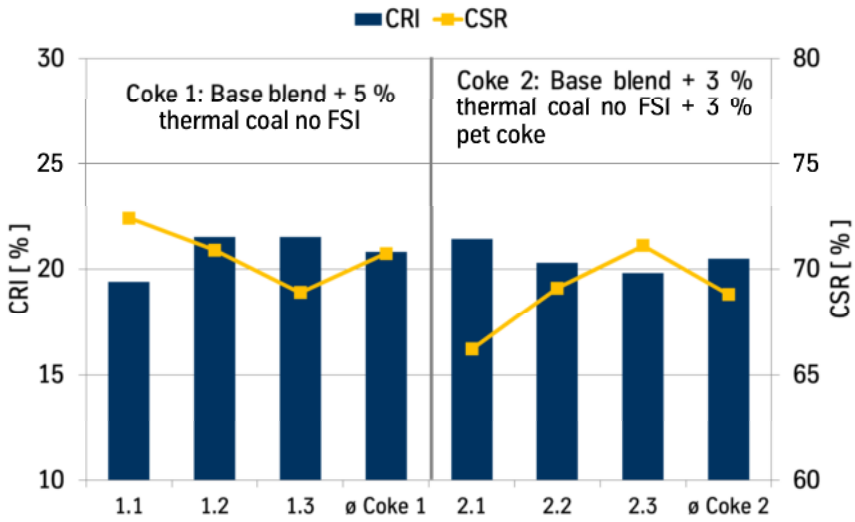



**Figure 76: Coke reactivity versus coke strength after reaction. ). Coke 1 =TC1, Coke 2 TC2**

Figure 77 shows a comparison of CRI/CSR results for coke samples with the same blend composition but produced either at DMT (No. 2A and No. 7) or SSAB (TC1 and TC22 – mean values).

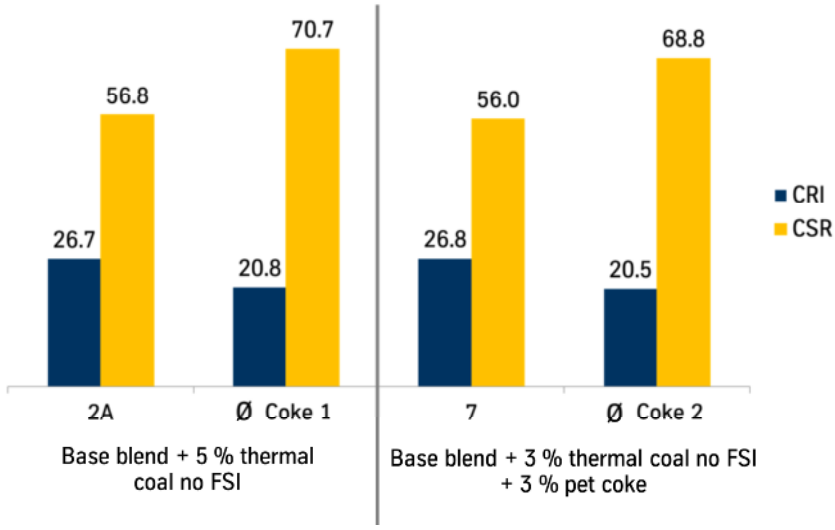



**Figure 77: Comparison between pilot test (DMT 500 kg semi-industrial oven) and industrial test (SSAB coking plant 1st trial - mean values). Coke 1 =TC1, Coke 2 TC2**

It can clearly be seen that the coke produced at the SSAB coking plant has a significantly higher quality compared be to the DMT pilot-scale (semi-industrial) coke. The SSAB industrial produced coke for both blends reaches CRI values around 20% to 21% and CSR values around 70%. For the

DMT semi-industrial coke samples a much higher CRI of around 27% and much lower CSR of around 56% to 57% was determined. Reasons for the difference are the different geometry of the coking devices and especially the resulting bulk density of the coal blend during charging into the chamber. In industrial coking plants the bulk density is in principle higher, especially with higher ovens, and the charged weight contributes to compaction on the coal bulk. Another contributing factor to for the difference can be the slight change in the base blend composition (low-, middle- and high- volatile coking coals) between DMT and the SSAB test, see Table 41 in Task 4.1, using higher amount of low volatile coal in the base blend, a coke of high quality regarding CRI/CSR values is produced.

**Innovative test equipment at tk SE for CRI/CSR determination**

The quality of the blast furnace coke charged is crucial for a smooth hot metal production as coke takes over several tasks in the furnace:

- providing heat for a partial gasification of coke
- providing CO as a reducing agent
- carburizes hot metal
- maintains drainage for liquid phases
- maintains gas permeability
- supports burden

Coke charged in the blast furnace is concurrently exposed to chemical, thermal and mechanical stress. ISO standard test considers all three forms of stress but not simultaneously. Chemical ($CO_2$) and thermal stress are applied for the determination of the CRI and mechanical stress (= tumbling) for determination of CSR. Although the ISO CRI/CSR results are known worldwide and used to compare coke quality, this test does not characterise coke in a realistic blast furnace condition. To investigate the coke reactivity and hot strength in conditions closer to those of an industrial blast furnace, innovative CRI/CSR equipment was designed and erected at tk SE.

The final design was agreed in summer 2014, see Figure 78. The first commissioning test at tk SE was carried out in spring 2015.

**Figure 78: Sketch (left) and overview (right) of the new CRI/CSR equipment erected at tk SE**

The electrical oven is designed for a maximum temperature of 1500 °C, but the retort, due its material heat resistance, is only allowed to be exposed to a maximum temperature of 1150 °C. Therefore the test temperatures are limited up to this. The larger retort volume compared to the ISO one allows testing sample weights up to 2500 g. The intention is also to use variable grain size distribution. The oval shape ensures a homogeneous temperature distribution inside the retort along the coke column.

The weight loss during the test can be controlled by a weighing system, which is installed under the oven. In order to obtain additional information from the test, the off gas can be also analysed using a mass spectrometer. To simulate the mechanical stress a pneumatic stamp is used, which is able to exercise a force up to 2.8 bar on the coke column during the test at a maximum temperature of 1150 °C

The test details are compiled in one recipe, where gas composition, flow rate, temperature, weight loss and test time can be established and varied if necessary. During the test a thermocouple monitors the middle temperature of coke layer, so that the temperature of the heating zones is automatic regulated in order to ensure the target test temperature.

Table 30 describes the main differences between the standardised CRI/CSR ISO test and blast furnace boundary conditions.

In the ISO test, coke is exposed to 100% $CO_2$ for two hours at a temperature of 1100 °C, whereas under real BF conditions its residence time is around 7.5 – 8 hours, in which the coke burden material sinks downwards in the BF facing increasing temperature and gas with different gas components such as $CO_2$, CO, $N_2$ and $H_2$. BF coke normally has a grain size of >35 mm, which differs considerably from the grain size distribution of 19 – 22.4 mm prescribed in the ISO standard.

**Table 30: ISO test- and blast furnace test conditions**

| | ISO standard test conditions | Blast furnace conditions |
|---|---|---|
| Reaction gas | 100 % $CO_2$ | $CO_2$, CO, $N_2$, $H_2$ |
| Temperature | 1100 °C | Different temperature zone |
| Reaction time | 2 hours | ca. 7.5 - 8 hours residence time |
| Grain size | 19 - 22.4 mm | > 35 mm |
| Sample weight | 200 g | ca. 300 - 350 kg/t hot metal |
| Stress | Thermal and chemical (CRI) separated from mechanical (CSR) | Simultaneous thermal, chemical and mechanical stress |

**Pre-Tests: Determination of coke reactivity index - CRI under different test conditions**

In order to investigate the impact of variable test conditions such as grain size, sample weight and gas flow rate/composition on the resulting CRI/CSR values, a series of experiments were carried out at the ISO furnace. For all tests the temperature of 1100 °C, the heating time of 45 min and the reaction time of 120 min were kept constant. Coke from the tk SE coking plant (KBS) was used in all tests as its quality is well known. The corresponding CRI and CSR values were determined in accordance with ISO standard.

*Influence of sample weight and gas composition on CRI and CSR value*

The influence of coke exceeding the ISO- sample weight and flow rate was evaluated. For this sample weights of 200 g and 400 g (19.0- 22.4 mm) and gas flow rates of 5.0 l/min (ISO) and 5.6 l/min $CO_2$ were tested. In some tests the coke column in the retort was divided into 2 or 4 fractions, using a sieve plate. This allowed a separate determination of reactivity and strength for those single fractions. The corresponding CRI/CSR results are marked with 1 (bottom fraction), 2, 3 and 4 (top fraction). Detailed test results are illustrated in Figure 79 and in Figure 80.

It can be clearly seen in Figure 79 that the reaction rate (i.e. CRI) with $CO_2$ is higher for the lower coke layer where the unreacted gas is introduced into the retort. This also results in lower CSR values. In the upper parts CRI is lower and CSR higher as the CO content (generated by solution loss reaction) in the arising gas increases, lowering the kinetics of solution loss reaction. This effect was gained for all experiments in which coke column was divided by a sieve plate.

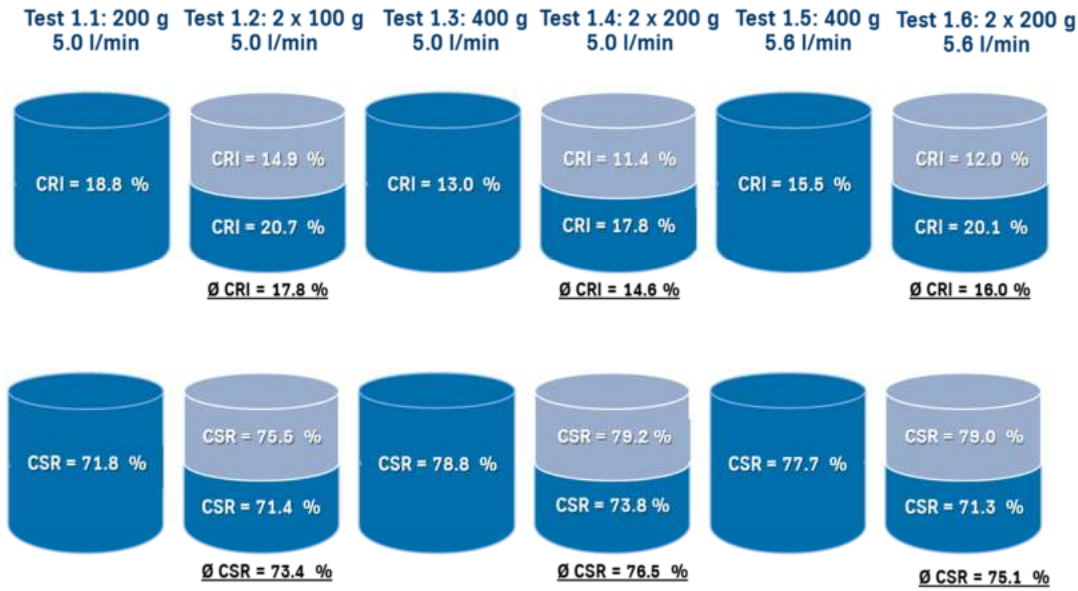



**Figure 79: Results of trial series 1 (test temperature: 1100 °C; reaction time: 120 min)**

Using twice the amount of sample weight (400 g) but keeping the ISO gas flow rate of 5.0 l/min led to lower CRI and higher CSR values, see Figure 80. However doubling the sample weight does not lead to a 50% decrease in CRI. A homogenous temperature distribution along the coke column in the reaction vessel was ensured for tests with 400 g.

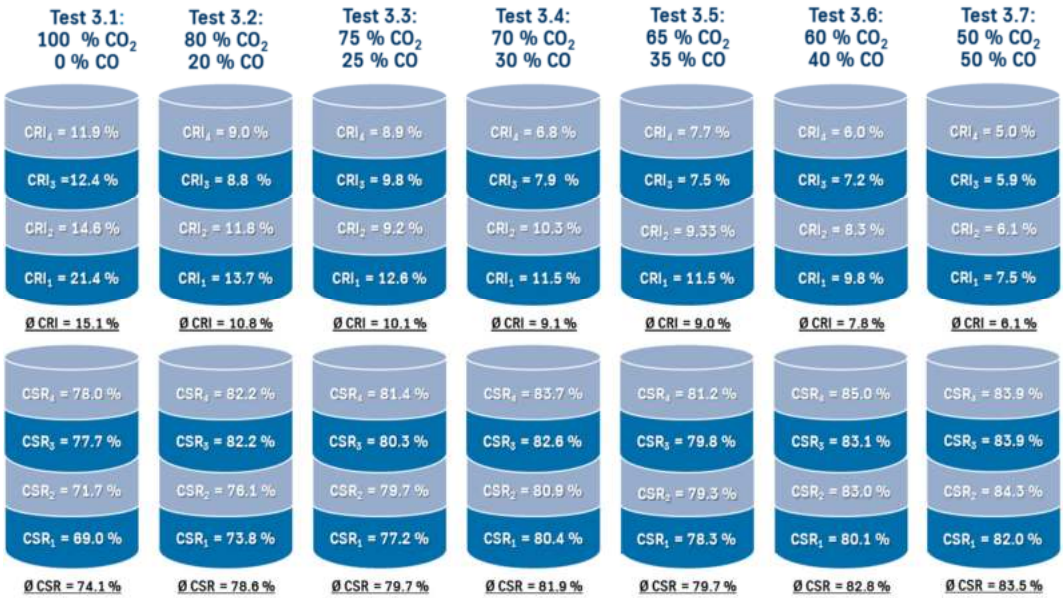



**Figure 80: Results of trial series 3 – 400 g – 4 x 100 g (test temperature: 1100 °C; reaction time: 120 min)**

Figure 81 shows the evaluation results of the tests 3.2 to 3.6 in order to create a gas profile along the retort height for test 3.1. The entering gas has a composition of 100% $CO_2$ but $CO_2$ content decreases when the gas ascends and solution loss occurs. The $CRI_2$ of test 3.1 is with 14.6 % slightly higher than the 13.6% of $CRI_1$ from test 3.2 where the corresponding gas composition is 80% $CO_2$ and 20% CO. Based on this, it can be assumed that the gas entering the second layer of test 3.1 has a gas composition with >80% $CO_2$ and <20% CO.

The achieved $CRI_1$ in test 3.3 is similar to the $CRI_3$ of test 3.1. Therefore it can be concluded that the gas composition in the third layer has a value of around 75% $CO_2$ and 25% CO.

The gained $CRI_1$ value test 3.4 and 3.5 are very similar to the $CRI_4$ of test 3.1. Based on this it can be concluded here that the gas entering the fourth layer has at least 65 % $CO_2$ content in the gas. This assumption can be confirmed by the analysis of the exhaust gas with the mass spectrometer

where 68% $CO_2$ and 32% CO were detected. This leads to conclusion that the gas in the fourth layer consists of approximately 70% $CO_2$ and 30% CO.

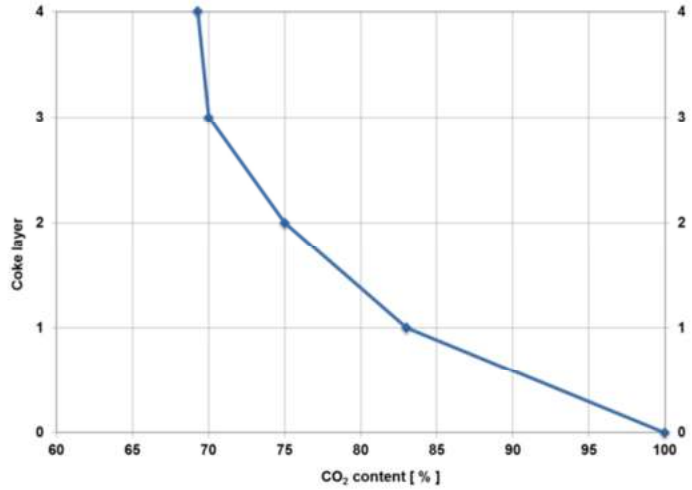



**Figure 81**: **Gas composition in the layers of test 3.1**

*Influence of gas flow rate and grain size on CRI value*

To determine the necessary gas amount to reach the same CRI in 120 minutes for a doubled sample weight of 400 g, gas flow rates from 6.0 -12.0 l/min and 15.0 l/min were tested (ISO = 5 l/min). The results are illustrated in Figure 82. The orange line represents the CRI value gained for 200 g.

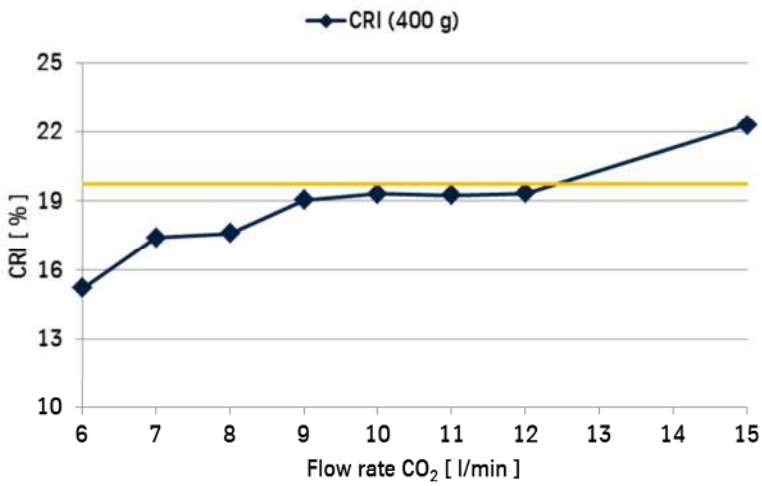



**Figure 82: CRI results for different flow rates**

It can be concluded that the gas flow rate influences the kinetics of solution loss reaction. For gas flow rates between 6 and 9 l/min an increase in CRI can be observed followed by a steady CRI of around 19 % for gas flow rates between 9 and 12 l/min. This confirms that doubling the gas flow rate and sample weight leads to similar CRI values as for the ISO conditions. Furthermore a CRI of 22% has been determined for the test with a flow rate of 15 l/min.

The influence of the grain size on the solution loss reaction was investigated by using coke samples with two different grain sizes (19.0 – 22.4 mm and 35 – 50 mm) similar to the standard test and the BF. The tests were performed under 100% $CO_2$ gas atmosphere. The gas flow of 5.0 l/min was doubled when the sample weight increased from 200 g to 400 g.

Figure 83 shows the results of the performed tests. It can be seen that an increase in grain size significantly reduces the corresponding CRI. In these tests, the CRI of the coarser fraction is around 4% lower compared to the ISO standard fraction. It is obvious that the surface area of the coke pieces is significantly different depending on the used fraction. For the ISO standard fraction around 37 coke pieces are necessary to create 200 g, whereas for the coarser fraction only 9

pieces are needed. From the literature it is known that the reaction rate increases by higher surface area. This explains thus the elevated CRI for smaller coke pieces.

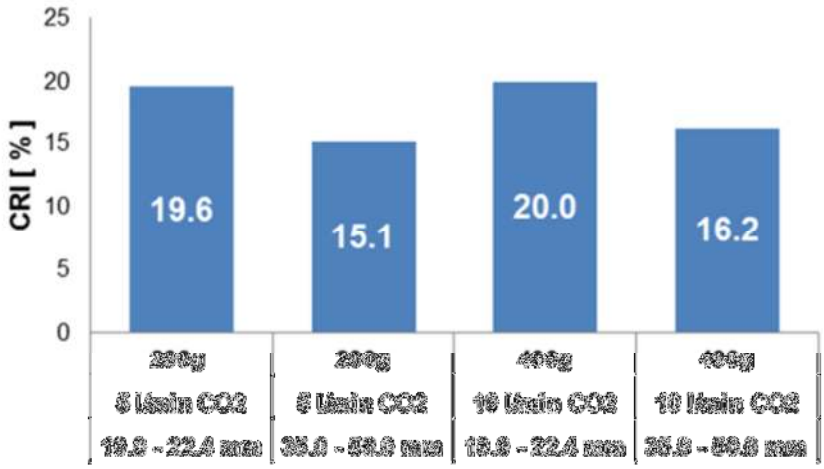



**Figure 83: Results of CRI and CSR for different grain sizes and sample weights**

**CRI/CSR – new coke characterisation**

*CRI/CSR comparison from ISO standard and new innovative test facility*

For the commissioning phase a test series was planned in order to gain experience about functionality of the new equipment, as the retort and oven geometry strongly differs compared to the standard ISO test retort and oven. To more clearly understand the results, the standard coke from tk SE coking plant (KBS) was used as its quality regarding to CRI and CSR is very well known.

The base area from the new retort is 3.13 times greater than the ISO retort. When 200 g of coke is filled into the ISO retort a bulk height of 80 mm is obtained, whereas the same amount only results in approximately 25 mm for the new retort.

In order to carry out the CRI test under the same conditions in both facilities (to check whether the new test rig can also be used for simulating ISO conditions), the test parameters were adjusted. It has been pre calculated that with a sample weight of 640 g (19.0 – 22.4 mm) and a gas rate of 15.7 l/min $CO_2$ the same gas amount per min/mm$^2$ for ISO test and new test rig is ensured. Consequently the reactivity index should also be the same.

The first commissioning results are shown in Figure 84. Comparing both facilities, the gained CRI values for the new test rig were slightly lower. In contrast to this the corresponding CSR was consequently higher. The difference for CRI was 2.6% and for CSR 4.3%.

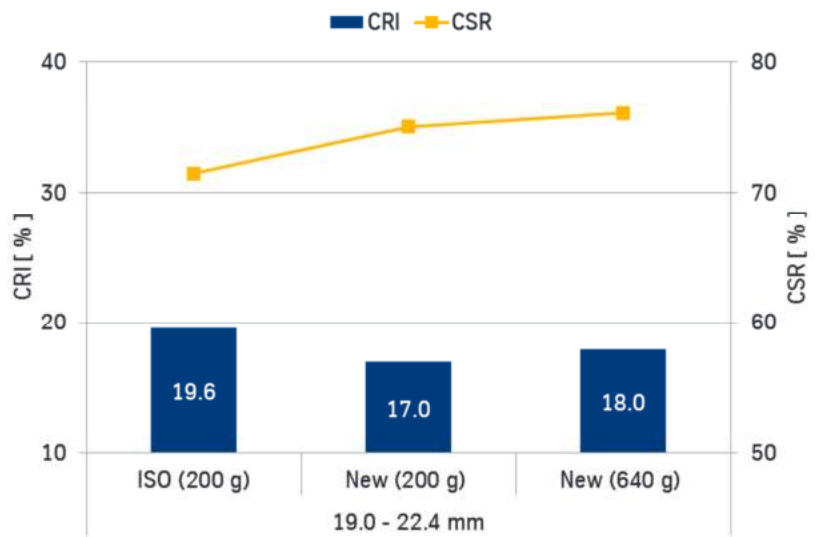



**Figure 84: CRI and CSR results**

From pre-tests it is known that the CRI decreases from the bottom to the top due to decreasing $CO_2$ concentration in the gas. According to this it could be assumed that when 200 g material is tested in the new facility a higher CRI than in the ISO test must be gained, since its bulk height is notable lower. However it can be observed that despite the strong difference in bulk height the CRI gained from the ISO and the new test are still similar.

When the bulk height is also kept at 80 mm (640 g) in the new rig and the sample weight and gas rate are adjusted, the CRI/CSR values are also nearly the same. Furthermore a good accordance regarding to CRI/CSR results can also be observed when comparing both tests performed in the new facility with different sample weight and bulk height.

The purpose of the new facility is to investigate coke under conditions closer to those of a blast furnace as they differ strongly from those prescribed in the ISO standard. In order to obtain a realistic coke characterisation with respect to coke reactivity index (CRI) and hot strength (CSR) tests with different grain sizes, sample weights and gas composition were carried out. For some tests a load of 2.8 bar was applied on coke column to simulate the mechanical stress. The entire tests were run at a temperature of 1100 °C and the flow rate for different sample weights was adjusted on the basis of ISO standard (5.0 l/min).

In order to ensure that larger coke bulk has reached target temperature of 1100 °C within the heating time of 45 min tests doubling the heating time to 90 min were carried out. The reaction time of 120 min was kept constant. Used fraction was 35 - 80 mm and the sample weight and gas flow rate were 2000 g and 50 l/min. The CRI results for both heating times are illustrated in Figure 85. The average CRI is very similar for both test conditions which indicates that a heating time of 45 minutes is sufficient in order to completely heat up even larger coke pieces.

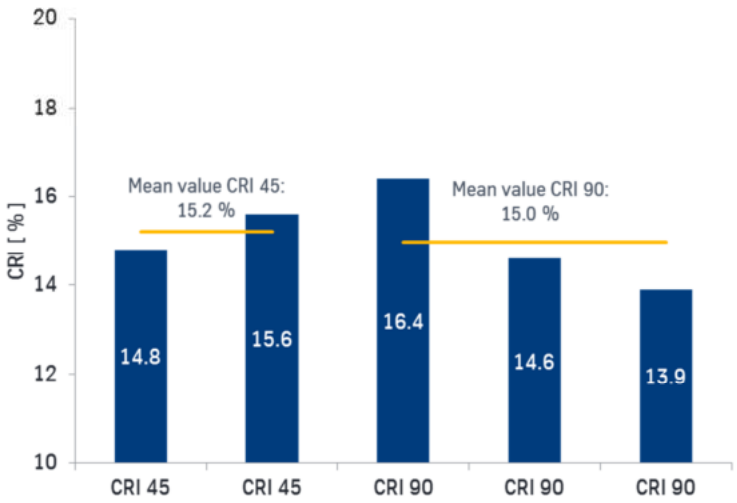



**Figure 85: Comparison of CRI for different heating times**

*Influence of grain size and type of tumbler on determined CRI and CSR value*

The weight loss was calculated according to ISO standard. Due to different sample weights two methods were used to determine the CSR, see Figure 86. The type of tumbler was selected depending on the sample size. The ISO tumbler was used for 200 g and 640 g and a larger tumbler (½ Micum) for 2000 g. For both methods 600 revolutions were set, with a velocity of 30 rpm for ISO and 24 rpm for ½ MICUM. The dimensions of both facilities are also summarised in Figure 86. Beside the conventional CSR evaluation, the change in the grain size was also investigated using the optical particle analysing facility – PartAn (Task 2.1).

Firstly the CRI and CSR values of coke with a grain size distribution of 35 - 80 mm were investigated and compared with the ISO standard grain size (19 – 22.4 mm). The tested sample weights were 200 g, 640 g and 2000 g. The tests were performed under 100% $CO_2$ gas atmosphere.

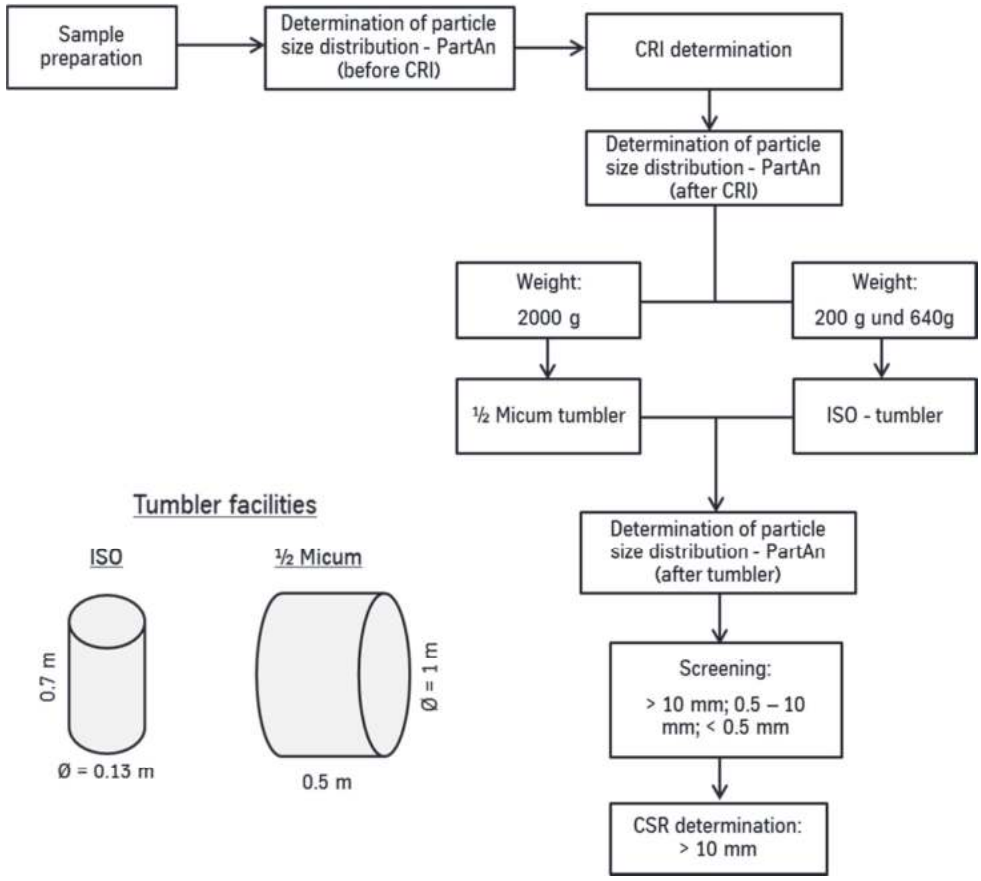



**Figure 86: CRI/CSR evaluation and the tumbler dimensions**

The CRI and CSR results are displayed in Figure 87. Beside the results obtained in the new facility, the CRI and CSR gained in the ISO test is also correlated. When comparing the CRI, it can be firstly concluded that coke reactivity drops with increasing grain size.

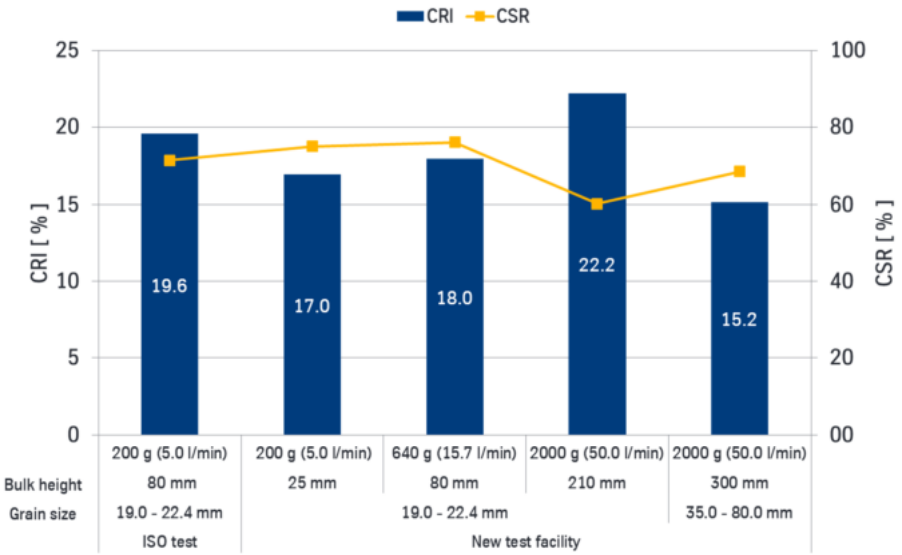



**Figure 87: CRI/CSR for different sample weights, flow rates and grain size distributions**

Comparing the results gained for the tests with grain size of 19 – 22.4 mm it can be concluded that increasing the bulk height in the retort (i.e. increasing sample weight and analogue increasing in gas flow rate) also increases the corresponding CRI.

Increased bulk height in a first step by 55 mm (25 mm to 80 mm) led to an increase in CRI of 1%. In a second step the bulk height was increased by 130 mm (80 mm to 210 mm) and the CRI increased by 4.2%. Here it can be assumed that an increase in bulk height also leads to a rise in gas resistance which leads to longer residence time of the gas in the coke column hereby affecting reaction rate.

CSR is inversely proportional to the coke CRI. The sample weights 200 g and 640 g presented similar CRI and also CSR, 75% and 76%, respectively. As the obtained CRI for 2000 g (19 – 22.4 mm) was the highest, it was also expected that CSR would be the lowest, which was confirmed. The CSR for 2000 g (35 – 80 mm) and ISO CSR were very similar (71% and 69 %). The samples were also measured using the PartAn facility before and after the CRI test as well after tumbling. Figure 88 - Figure 90 illustrate the frequency and MPS of three samples.

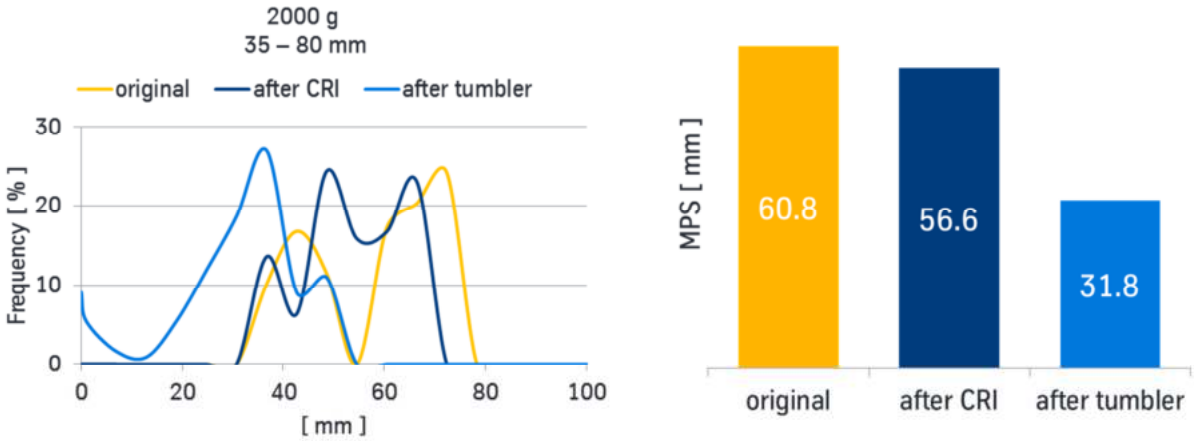



**Figure 88: PartAn results for samples treated in ½ Micum tumbler (39 coke pieces)**

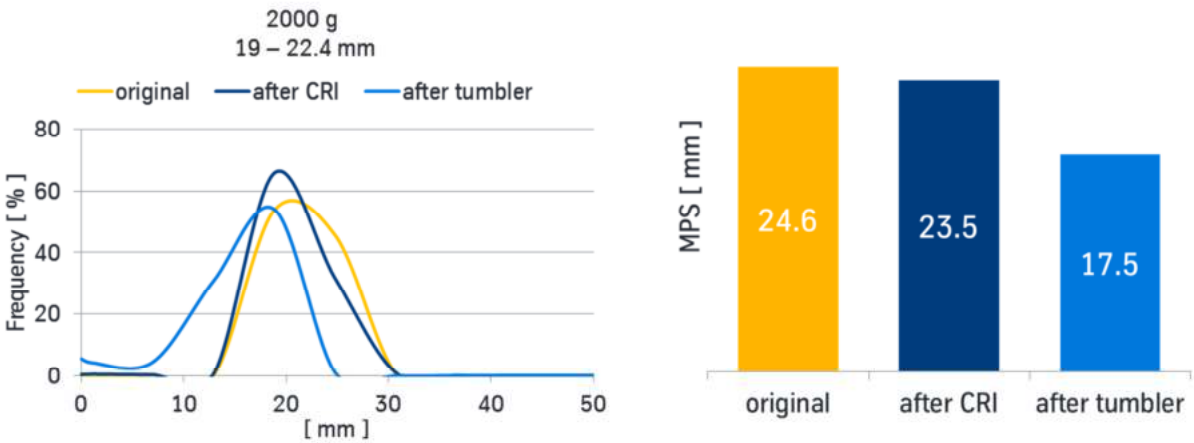



**Figure 89: PartAn results for samples treated in ½ Micum tumbler (375 coke pieces)**

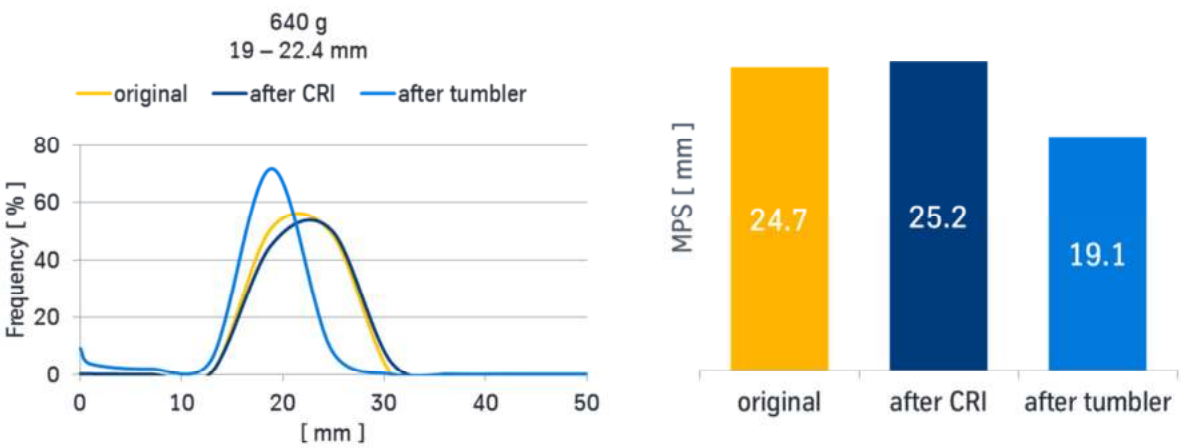



**Figure 90: PartAn results for samples treated in ISO tumbler (111 coke pieces)**

It is known from literature that during the CRI test the solution loss takes place preferentially on the particle core, i.e. there is almost no change in grain size after CRI test. This has been confirmed by the PartAn results as there is only a small change in the MPS from samples before and after CRI test. However after tumbling the MPS decreases by 44% and 25% for ½ Micum and

by 25% for ISO drum. Although the same decrease in the MPS for the samples with grain size 19 – 22.4 mm can be observed, significant particle degradation can be seen when the larger tumbler was used.

*Coke characterisation under gas composition similar to an industrial the blast furnace*

To test coke under gas atmosphere similar to a real blast furnace, $N_2$, CO and $H_2$ were added to $CO_2$ for the new test conditions. For this purpose the gas analysis of a gas sampling system at blast furnace No. 1 Schwelgern was evaluated and used. This gas sampling system measures the gas composition in 15 different positions distributed over a height of 21.5 m above tuyere level. To simulate the gas composition of the blast furnace 3 points were selected (named top, middle and bottom), see Figure 91. The composition at the top and middle are similar whereas at the bottom the CO concentration increases and consequently $CO_2$ decreases. The concentration of $H_2$ and $N_2$ is similar for all three positions.

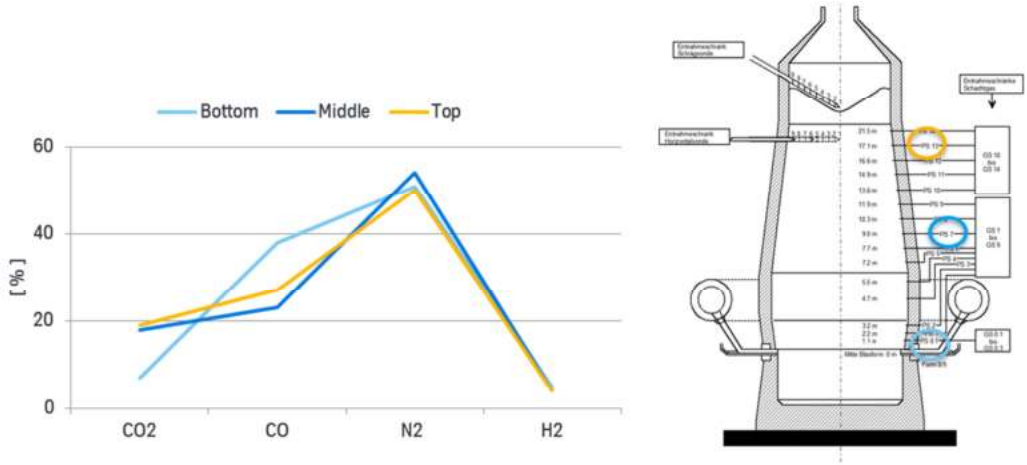



**Figure 91: Gas composition at top, middle and bottom**

The results of the performed tests under BF like conditions are displayed in Figure 92. Coke reactivity gained for the top and middle positions are very similar, due the similarity of the gas composition. The CRI obtained for the bottom was significantly lower, due to the increase of CO concentration in the gas mixture. In general the adapted gas composition generated much lower CRIs as known from the standard tests. Despite different CRIs values, the calculated CSR for all levels were very similar.

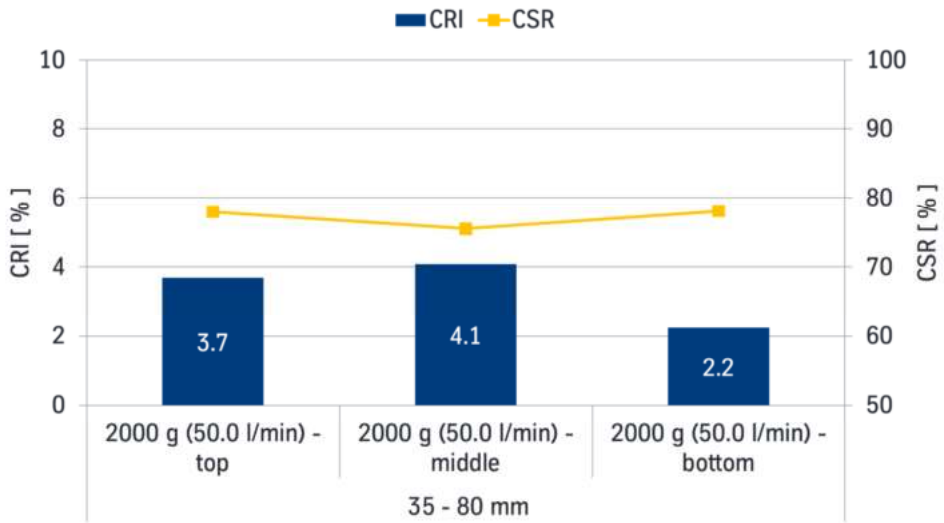



**Figure 92: CRI and CSR results for similar gas atmosphere to the blast furnace**

The addition of CO, $N_2$ and $H_2$ in the gas mixture lowered CRIs values considerably. In the ISO standard test, coke is tested under an extreme reactive gas atmosphere, 100% $CO_2$, but in the blast furnace this condition is found only for a short time in/near the raceway zone. However, due to the high temperature and the carbon availability in this area, the solution loss reaction takes

place and $CO_2$ is converted into CO. Therefore it could be useful to test coke under conditions closer to those at the blast furnace in order to establish a new characterisation for coke reactivity

*Coke characterisation under simultaneous thermal, chemical and mechanical stress*

As already reported, coke has to fulfil different tasks during its residence time in the blast furnace, such as a provider of heat and CO, maintainer of drainage for liquid phases and gas permeability and also supporter of the burden, in which coke is concurrent stressed thermally, chemically and mechanically.

To calculate the load which is exercised on the coke layer in the blast furnace, the charging profile of tk SE blast furnaces were used. It is known that the hematite ($Fe_2O_3$) is reduced to magnetite ($Fe_3O_4$) then to wustite (FeO) and finally to metallic iron (Fe) during the descent of the burden material. However in order to simplify the calculation, it was assumed that the burden layer contained only hematite from the top to the bottom of the blast furnace.

The maximum and minimum forces were calculated and the result for blast furnace No. 1 is presented in Figure 93. The maximum and minimum load is 0.13 bar/m and 0.05 bar/m. The lowest load derives from the middle of the blast furnace, where only coke is charged. Coke has bulk density of approximately 0.5 t/m$^3$ of. In contrast the maximum force was calculated at 2 to 4 meters along the furnace diameter, where the highest amount of burden is charged. The burden bulk density is ca. 1.7 t/m$^3$. At tuyere level the maximum and minimum load is 3.3 and 1.3 bar, respectively.

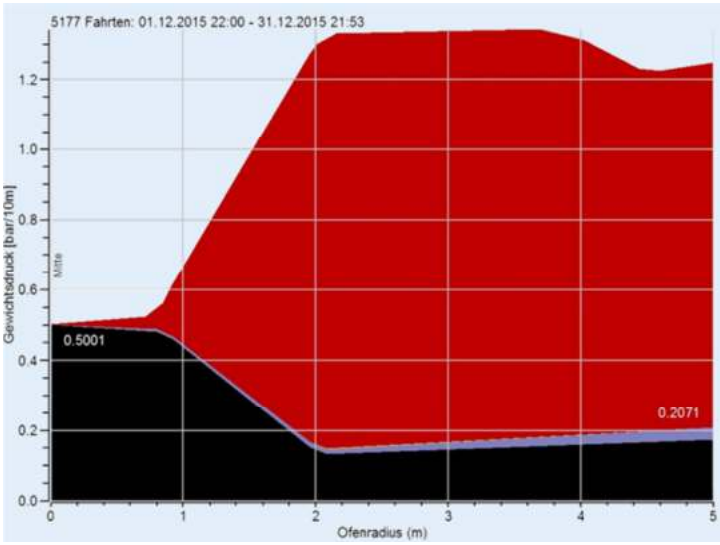



**Figure 93: Calculation of maximum and minimum load in the blast furnace No. 1 Schwelgern**

In order to obtain a first impression on the impact of the mechanical load applied during the solution loss reaction, two test series were designed. Besides tk SE coke, coke produced at SSAB coking plant in the 1$^{st}$ industrial trial (TC1: Base blend + 5 % HV thermal coal no FSI) was also tested. The gas atmosphere of 100 % $CO_2$ and the temperature of 1100 °C were kept constant. In order to ensure 1100 °C, the heating time of 45 min had to be adjusted to 75 min for tests with mechanical load. The sample weight and gas flow rate were 1000 g and 25 l/min respectively. However the reaction time and load applied were varied. In the reference trials no load was applied on the tested coke samples. The reaction time was set to 60, 120 and 180 min. In the second trial series a load of 2.8 bar was set, simulating the height of approximately 4.5 meters above tuyere level and a position between 2 and 4 m along the diameter, according to the calculation made for blast furnace No.1. For this trial the tested reaction times were 120 and 180 min.

The CRI and CSR results for the reference tests performed without mechanical load are presented in Figure 94. It can be seen that the CRI values increase proportionally with the reaction time. Although different coke types, the CRI and CSR values were very similar for the same reaction time.

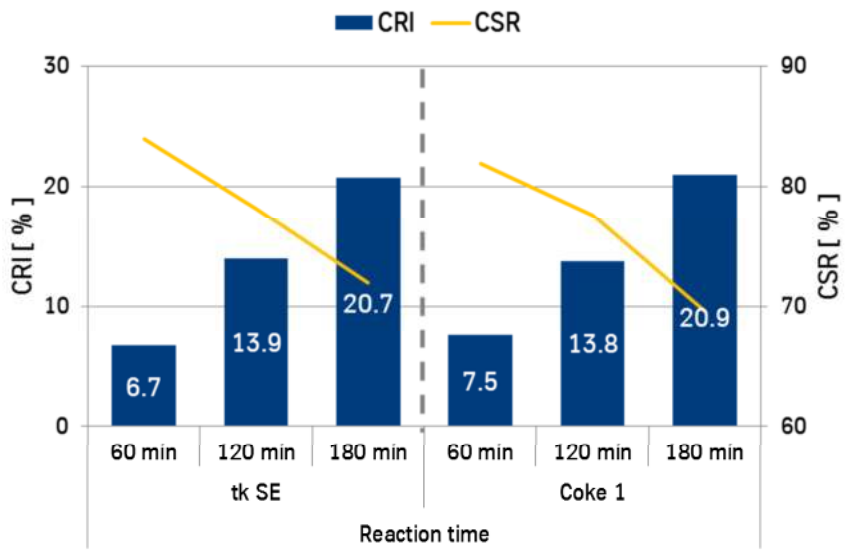



**Figure 94: CRI and CSR values (0 bar). Coke 1 =TC1**

Figure 95 and Figure 96 show the comparison of CRI and CSR values for the tests carried out with and without mechanical load. After performing the CRI tests, the change on grain size distribution was investigated using the PartAn facility (Task 2.1). Afterwards the coke samples were tumbled in the ISO drum in order to evaluate the coke strength after reaction. Here it can be seen that the application of 2.8 bar during the solution loss reaction slightly influences the reaction rate. The usage of a stamp enhances the gas resistance inside the retort which leads to higher residence time of the gas in the coke column increasing the reaction rate. The increase on CRI values was different depending on the set reaction time. The higher the reaction time is, the higher the CRI an absolute delta becomes.

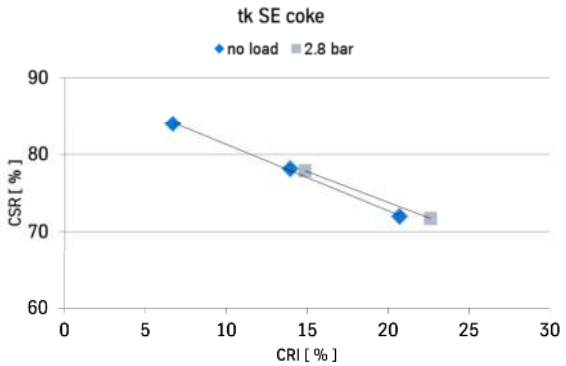



**Figure 95: Comparison of CRI and CSR values for tk SE coke**

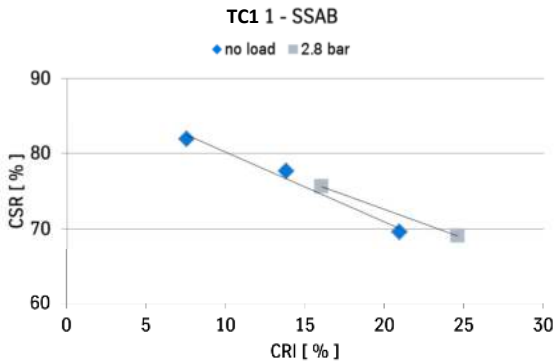



**Figure 96: Comparison CRI and CSR values for TC1 – SSAB 1st industrial trial**

For tk SE coke the CRI values increase by 1% and 2% for reaction time of 120 min and 180 min respectively, whereas the rise on CRI gained for TC1 (SSAB) was more significant, 2.2 % for 120 min and 3.6 % for 180 min reaction time. Although the CRI values were higher for the tests performed under 2.8 bar, the coke strength were very similar.

The grain size distribution of each coke sample was measured with PartAn facility before and after CRI test and finally after tumbling. The change in coke size distribution for the tests with 120 min reaction time is shown in Figure 97 and Figure 98.

After the gasification almost no change on the size of coke piece could be visually observed, but the PartAn results show that the coke pieces slightly shrink. It was expected that the application of 2.8 bar would deteriorate the coke pieces during the solution loss reaction (CRI test) much more, but only a slightly change could be observed when comparing the grain size distribution before and after CRI test. However the content of particles <20 mm is higher for the test when mechanical load was applied. Considering the amount >10 mm, the CSR determined by PartAn is higher than CSR evaluated according to ISO standard, see Figure 97 and Figure 98.

Due to several delays during the commissioning/trial phase the test series with mechanical load could not be fully performed. Therefore the investigations will be further continued.

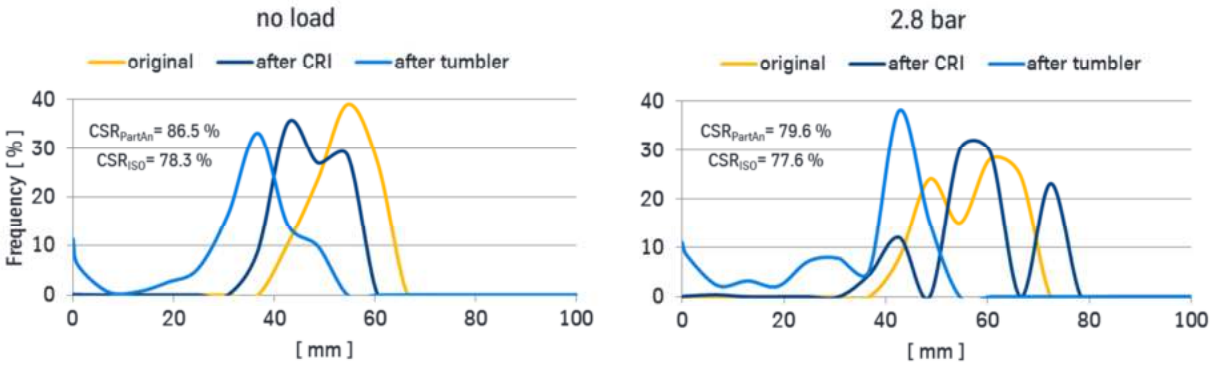



**Figure 97: Grain size distribution in tk SE coke**

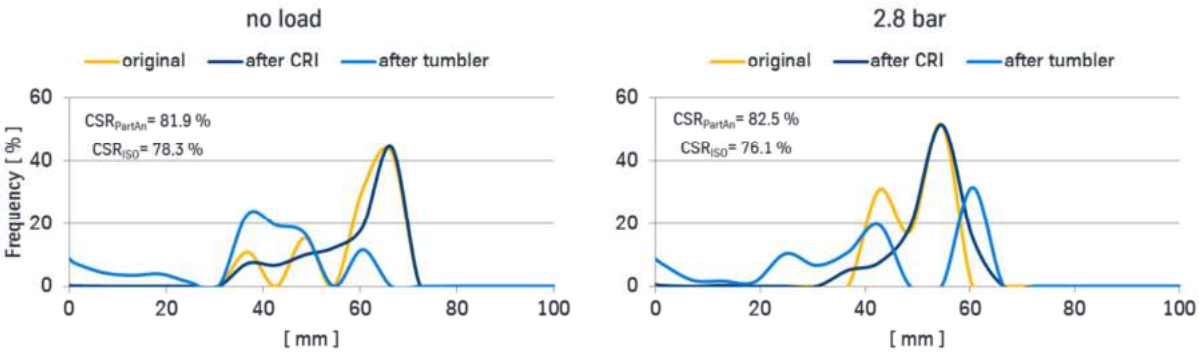



**Figure 98: Grain size distribution Test Coke 1 – SSAB 1st industrial trial**

### LKAB BF simulating test - TRT Mas

Selected alternative coke samples together with pellets have been tested in an existing BF simulating test equipment at LKAB using test conditions designed for correlation with the EBF® and evaluation was made in cooperation with MEFOS. The tests have been performed at LKAB test facility in Malmberget. Selected coke types from the first semi-industrial trial at DMT have been mixed with MPBO pellets and tested twice and average values of pellet reduction and coke loss were calculated. In addition, ordinary coke used (standard) in trials at EBF® was tested and the test set-up is shown in Table 31. The simulated BF test used is called "TRT Mas" test and the furnace and reduction tubes are shown in Figure 99.

**Table 31: Test set-up of BF simulating tests at LKAB**

| | | | |
|---|---|---|---|
| Mix MPBO + Coke No.1 | MPBO+No.1 | 1 | Base blend |
| Mix MPBO + Coke No.1 | | 2 | Base blend |
| Mix MPBO + Coke No.2A | MPBO+No.2A | 1 | Base Blend + HV thermal coal, 5% addition |
| Mix MPBO + Coke No.2A | | 2 | Base Blend + HV thermal coal, 5% addition |
| Mix MPBO + Coke No.3 | MPBO+No.3 | 1 | Base Blend + 5% Anthracite |
| Mix MPBO + Coke No.3 | | 2 | Base Blend + 5% Anthracite |
| Mix MPBO + Coke No.4 | MPBO+No.4 | 1 | Base Blend + Charcoal (3%) |
| Mix MPBO + Coke No.4 | | 2 | Base Blend + Charcoal (3%) |
| Mix MPBO + Coke No.5 | MPBO+No.5 | 1 | Base Blend + Pet coke + Charcoal |
| Mix MPBO + Coke No.5 | | 2 | Base Blend + Pet coke + Charcoal |
| Mix MPBO + Coke No.7 | MPBO+No.7 | 1 | Base Blend + HV thermal coal + Pet coke |
| Mix MPBO + Coke No.7 | | 2 | Base Blend + HV thermal coal + Pet coke |
| Mix MPBO + Coke No.8 | MPBO+No.8 | 1 | Base Blend + HV thermal coal + Anthracite |
| Mix MPBO + Coke No.8 | | 2 | Base Blend + HV thermal coal + Anthracite |
| Mix MPBO + Coke standard | MPBO + standard | 1 | Ordinary coke used at EBF® |
| Mix MPBO + Coke standard | | 2 | Ordinary coke used at EBF® |

**Figure 99: BF simulating test (TRT Mas) furnace (left), load devices and reduction tubes (right)**

The sample has a weight on top of the sample with increasing pressure, simulating a burden decent in the blast furnace. The standard test program was used and the temperature changes, time and changing gas mixtures are shown in Table 32. The temperature was stabilised for 15 minutes between heating and zone 1 and the gas flow was set to 50 l/min.

**Table 32: Programme settings during the blast furnace simulating tests**

| Level | Time (min) | Temperature (°C) | $CO/CO_2/H_2/N_2$ (%) | Load (kg/cm$^2$) |
|---|---|---|---|---|
| Heating | ~ 45 | 20-500 | 0/0/0/100 | 0-1 |
| Zone 1 | ~ 70 | 500-850 | 21-23/20-18/0/59 | |
| Zone 2 | ~ 95 | 850-950 | 23-39/18-2/0/59 | |
| Zone 3 | ~ 55 | 950-1100 | 39-41/2-0/0/59 | |
| Cooling | | 1100-100 | 0/0/0/100 | |

The temperature and gas profiles of the TRT Mas test are also shown in Figure 100. The figure also illustrates weight losses of the test run of pellet and standard coke mix. The dotted line represent total weight loss of mixed sample and the black line is the weight loss of pellets in the mixed sample calculated on an oxygen content of 28.6% in pellets.

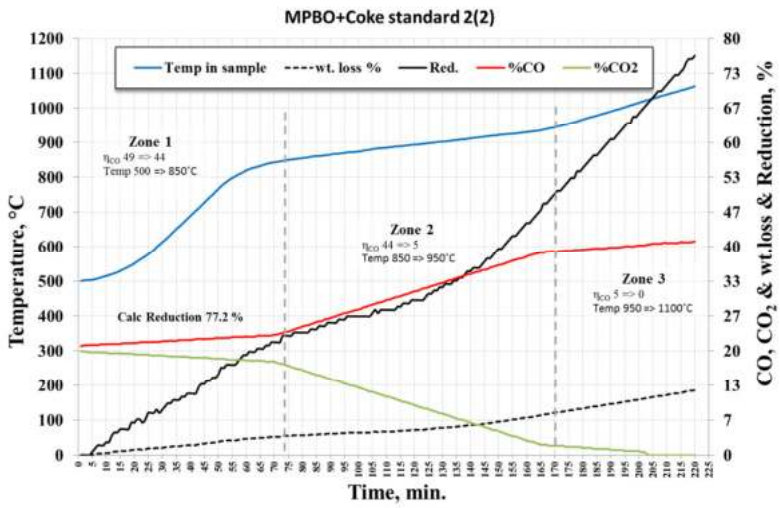



**Figure 100: Temperature, gas profiles, weight loss and MPBO pellet reduction for one sample mixed with standard coke**

Due to low $CO_2$ content (ramping down from 2% to 0) of the gas in TRT Mas tests at gasification temperatures (around 950-1100 °C) in zone 3 the weight loss of the coke is generally low. The weight loss of base blend coke (No. 1), standard coke used in the EBF® and a selection of alternative coke are shown in Figure 101 for all tests and average values in Figure 102.

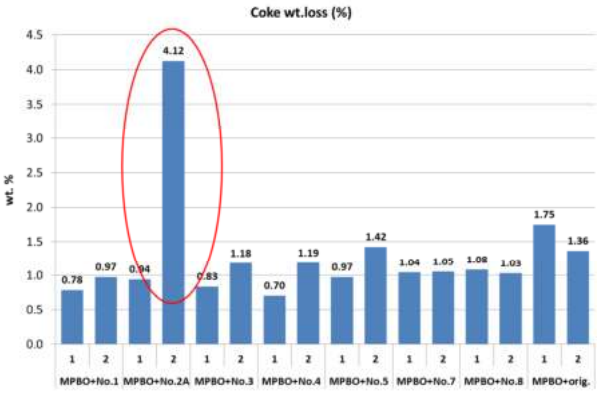



**Figure 101: Coke weight loss of all mixed tests**

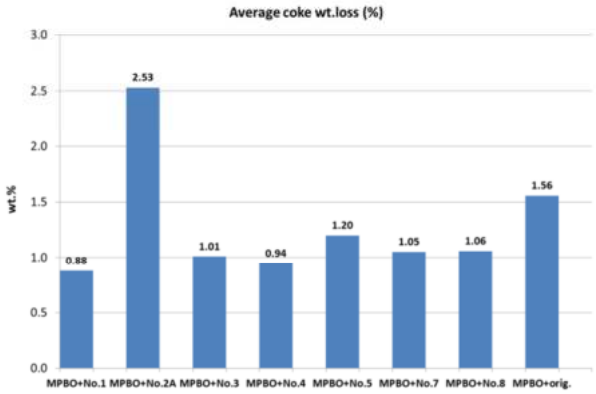



**Figure 102: Coke weight loss average values**

Notable is the large difference between test 1 and 2 for alternative coke type 2A, with 5 % addition of HV thermal coal. Looking at the pellet reduction for the same tests, the measured reduction for test 2 is much lower than test 1, see Figure 103. However, the difference is much smaller for the calculated reduction results in Figure 104.

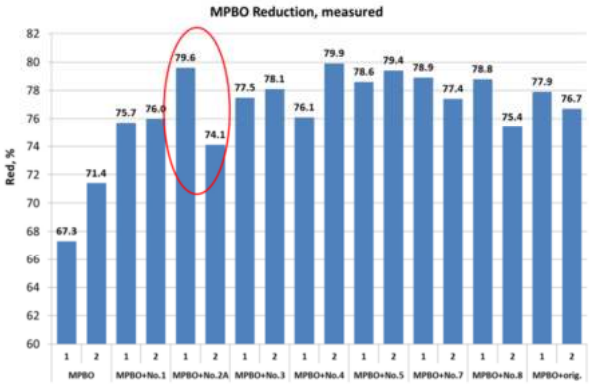



**Figure 103: Measured pellet reduction during the tests**

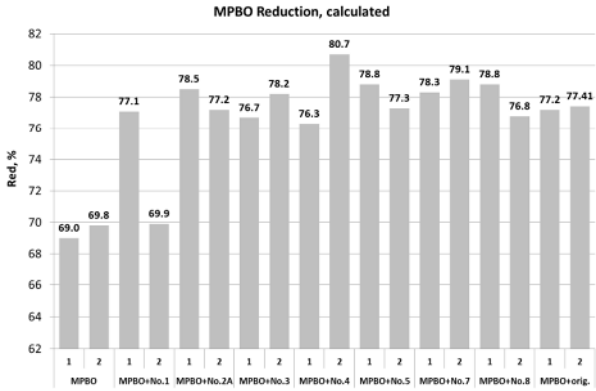



**Figure 104: Calculated pellet reduction (weight in-weight out)/$O_2$ in pellet sample)**

The test parameters could not explain the reason behind this high divergence. Therefore both samples were analysed in light optical microscope in Task 2.2 and it seems likely that the sample with 4% coke weight loss has an error and needs not to be considered. The result in Table 22 shows that the change in pore area and coke area is comparable small. Therefore should the weight loss in this sample be around 0.9 %.

Mixing pellets with coke have a positive effect on the pellet reduction degree as all coke type samples give a higher reduction than the single pellet tests, see Figure 103 to Figure 106.

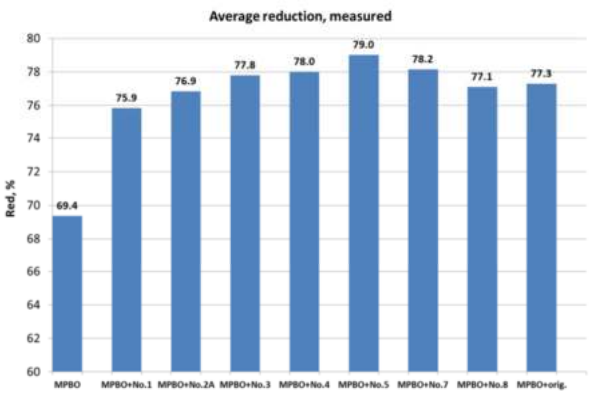



**Figure 105: Measured average pellet reduction degree of all mixed tests**

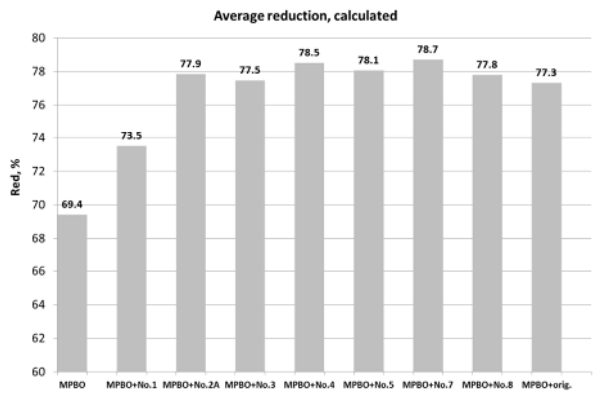



**Figure 106: Calculated average pellet reduction of all mixed tests**

The reference coke No. 1 and the standard coke used in LKAB EBF® are of similar type. Regardless of this the coke weight loss and pellet reduction is higher for the original coke than for coke type

No. 1. Looking at the test parameters, there were some inaccuracies regarding the temperature and $CO_2$ distribution, see Figure 107 and Figure 108 respectively. The temperatures were higher for test 2 of MPBO+Coke No.1 and for test 1 of MPBO + original coke. At the same time, the $CO_2$ gas did not reach 0% at the end of zone 3 for the same tests, which both can explain the higher gasification (weight loss) of coke for these tests. This gives an Eta CO of 5%, but was not seen in the other tests made.

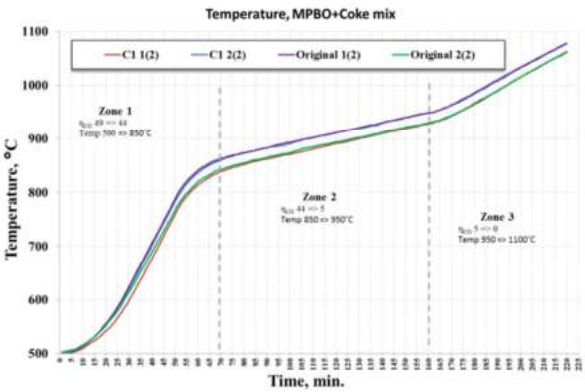



**Figure 107: Temperature profiles for coke type No. 1 (reference) and standard EBF® coke (original in figure)**

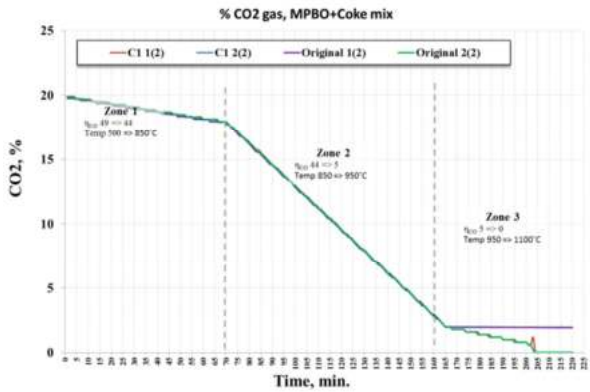



**Figure 108: $CO_2$ gas distribution for coke type No. 1 and standard EBF coke (original in figure)**

Calculated pellet reduction (wt. pellets in-wt. pellets out/wt. oxygen in pellet), not showed here, showed in some cases different results compared with measured pellet reduction during the tests. In the calculated pellet reduction alternative coke No.7 (3% HV Thermal coal and 3% pet coke), No.5 (3% charcoal and 3% pet coke) and No. 4 (3% charcoal) have the best influence on MPBO pellet reduction during these TRT Mas tests.

**Gasification reaction rates by using Thermal gravimetric analyser (TGA). Tests of selected technical scale carbonisation samples**

Selected samples from DMT retort tests (technical scale) in Task 1.2 have been analysed by MEFOS with thermal gravimetric analyser (TGA) using a Netzsch STA 409 graphite furnace.

The coke samples were crushed and screened to reach a particle size of 1-2 mm. Approximately 1 grams were placed in a crucible and an S-type thermocouple was used in the tests. The tests were conducted in 100% $CO_2$ gas atmosphere with the temperature programme showed in Figure 109. The flow rate of the $CO_2$ gas was set to 100 ml/minute.

The kinetic parameters as apparent reaction rate and activation energy for the gasification or solution loss reaction ($C+CO_2 \rightleftarrows 2CO$) in coke were calculated from TGA test data. The weight loss of carbon in segment 3, during slow heating of 3 degrees per minute, are assumed to entirely correspond to the apparent reaction rate (ρa) of C in coke.

The apparent reaction rate in grams of reacted C (dW) per time unit given in seconds (dt) relative to available amount of C in grams (W) according to eq. (2), is calculated based on the assumption that the weight loss entirely origins from loss of C due to coke gasification. Mean values of achieved apparent reaction rates and activation energies at temperature intervals of 950-990 °C, 990-1040 °C and 990-1120 °C were calculated, see Table 33.

$$\rho a = dW/dt * 1/W \qquad (2)$$

The activation energy for reaction of $CO_2$ with C in the coke can be calculated from the data assuming it to be a first order reaction following the Arrehenius relationship and following the equation (3)

$$\frac{dW}{dt} = k*W^n \text{ with } k=A\, e^{\frac{-E}{RT}} \text{ and } n=1 \qquad (3)$$

From a plot of ln[(dW/dt)*(1/W)] as a function of (1/T) in which T is given in Kelvin the activation energy (Ea) can be deduced for different temperature intervals by using the following relationship

$$Y=aX+b" \text{ where } a=-\frac{E}{R},\ X=\frac{1}{T} \text{ and } b=\ln(A) \qquad (4)$$

The mass loss of samples 1, 3, 4 and 8 are shown in Figure 110 and the C contents and conversions are presented in Table 34.

No 1 is the base blend coke of three coking coals and shows the smallest mass loss and C-conversion compared to the other samples with mixed alternative materials. The C-conversion is increased by 5 percentages when anthracite is part of the coking coal blend and the highest weight loss is shown when charcoal is added to the mix with around 12 percentages higher C-conversion compared to the base blend. When roughly 1% mass loss is reached, the temperatures are around 1000 °C for the base blend, 950 °C for No. 4 and 8, 930 °C for No. 3 and 730 °C for No. 5. The average reaction rates and activation energies in three different temperature intervals are presented in Table 33.

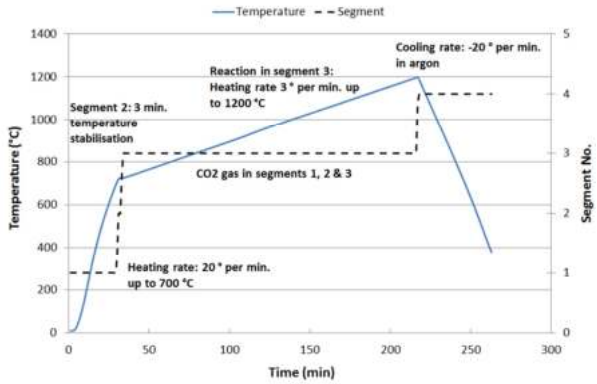



**Figure 109: TGA heating programme**

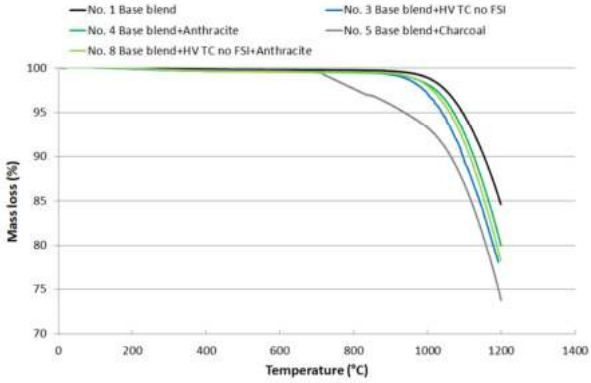



**Figure 110: Mass loss of selected samples from the DMT retort coke**

**Table 33: Average reaction rates and activation energies, different temperature intervals**

| Activation energies, Ea (kJ/molK) | | | |
|---|---|---|---|
| **Coke type/Temperature interval** | **950-990 °C** | **990-1040 °C** | **990-1120 °C** |
| No. 1 Base blend | 235 | 252 | 206 |
| No. 3 Base blend +HV TC no FSI | 246 | 163 | 129 |
| No. 4 Base blend +Anthracite | 209 | 170 | 132 |
| No. 5 Base blend +Charcoal | 75 | 165 | 161 |
| No. 8 Base blend +HV TC no FSI + Anthracite | 262 | 201 | 174 |
| **Average reaction rate of C (g/gs)** | | | |
| **Coke type/Temperature interval** | **950-990 °C** | **990-1040 °C** | **990-1120 °C** |
| No. 1 Base blend | 5.04E-06 | 1.20E-05 | 2.33E-05 |
| No. 3 Base blend +HV thermal coal no FSI | 1.36E-05 | 2.68E-05 | 4.06E-05 |
| No. 4 Base blend +Anthracite | 7.59E-06 | 1.54E-05 | 3.12E-05 |
| No. 5 Base blend +Charcoal | 1.46E-05 | 2.26E-05 | 3.77E-05 |
| No. 8 Base blend +HV thermal coal no FSI + Anthracite | 9.55E-06 | 2.07E-05 | 3.58E-05 |

**Table 34: C conversions and contents of selected samples**

| Coke type | Carbon conversion (%) | Carbon content (%) |
|---|---|---|
| No. 1 Base blend | 17.4 | 86.6 |
| No. 3 Base blend + HV thermal coal no FSI | 25.0 | 86.2 |
| No. 4 Base blend + Anthracite | 22.5 | 86.7 |
| No. 5 Base blend + Charcoal | 29.6 | 86.4 |
| No. 8 Base blend + HV thermal coal no FSI + Anthracite | 24.8 | 86.1 |

As expected, apparent reaction rate is increasing and the activation energy decreasing with increasing temperature. The coke with charcoal addition, No 5, has quite low activation energy in the lower temperature interval where the reaction is more towards chemical limited. This may imply that the raction in this case is more diffusion controlled than chemical. When temperature increases, the gasification reaction kinetics moves towards a mixed or diffusion limited regime.

Figure 111 shows correlations between the apparent reaction rates and CRI for two temperature intervals. At lower temperature interval charcoal is linear with the reference coke No 1 (lowest CRI), No. 4 (anthracite) and No. 3 (HV thermal coal) whereas No. 8 showed lower apparent reaction rate than No. 5 although they have almost the same high CRI values. At higher temperatures the apparent reaction rates of these two coke types becomes similar.

One reason for the different behaviour in the two temperature intervals for No. 5, with charcoal addition, might be that the isotropic charcoal structure, shown in Figure 46, is exposed to the $CO_2$ gas to a much higher degree than the more anisotropic coke structure. The charcoal structure might be consumed in the temperature interval of 950-990 °C °C leaving stronger coke structure with lowered apparent reaction rates in the higher temperature intervals. This can explain the low activation energy in the lower temperature interval and different slopes on the mass loss curve as seen in Figure 110. In addition, differences in porosity or microstructure effect might influence the CRI results more than the TGA results as a smaller size (1-2 mm) is used in the TGA.

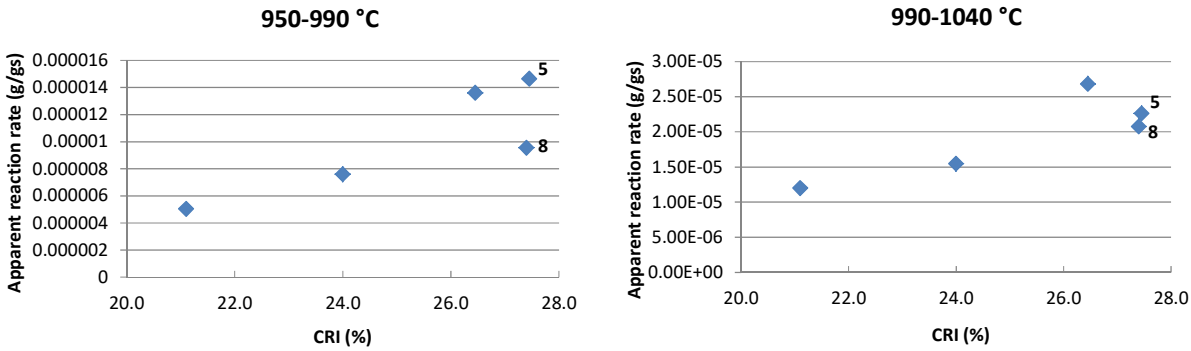
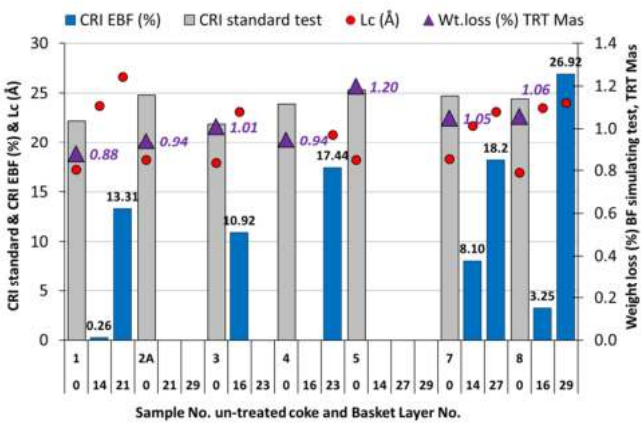



**Figure 111: Correlations between apparent reaction rates and CRI in selected samples from the DMT 10 kg retort tests**

**Comparison with CRI and CSR with coke gasification in BF simulating tests and in EBF® shaft**

No clear indications of gasification in the LOM study could be seen and therefore the study is not reported in this comparison. As the temperature in the BF simulating tests hardly exceed the coking temperature the graphitisation degree (Lc) of the coke is assumed to be as untreated, charged coke.

The coke weight losses in the BF simulating tests, LKAB TRT Mas tests, are assumed to represent reactivity in BF atmosphere. In Figure 112 the CRI values (table and grey bars) measured in the standard test at DMT are plotted together with coke samples reacted in BF simulating atmosphere (purple) and also in actual BF in basket samples (blue) excavated from the EBF shaft, campaign 31, see description in Task 3.2.




| Sample No. | DMT CRI (%) |
|---|---|
| 1. Base Blend | 22.2 |
| 2A. Base Blend+ 5% HV thermal coal no FSI | 24.8 |
| 3. Base Blend+5% Anthracite | 21.9 |
| 4. Base Blend+3% Charcoal | 23.9 |
| 5. Base Blend+3% Pet coke+3% Charcoal | 25.3 |
| 7. Base Blend+3% HV thermal coal no FSI+3% Pet coke | 24.7 |
| 8. Base Blend+3% HV thermal coal no FSI+3% Anthracite | 24.4 |

**Figure 112: Standard CRI with comparison of coke reacted in the EBF® and in BF simulating test**

Unfortunately there are some missing samples for $CRI_{EBF}$ as the baskets were torn and broken and the actual weight loss could not be calculated and all basket samples 2A and 5 are missing. As mentioned earlier the weight loss is low in the BF simulating tests due to the low amount of $CO_2$ reaction gas and therefore the reactivity is low compared to the standard test which has 100% $CO_2$ atmosphere.

Coke No. 5 (3% addition of charcoal and 3% pet coke) showed highest reactivity and the reactivity decreases in following order for BF simulating test; Cokes No. 8 (3% addition of thermal coal and 3% anthracite), No. 7 (3% addition of thermal coal and 3% pet coke), No. 3 (5% addition of anthracite), No. 2A and 4 (5% thermal coal and 3% charcoal respectively) and last with lowest

reactivity is No 1 (base blend). Standard CRI test showed different reactivity pattern, reactivity goes from high to low in following sample numbers; 5-2A-7-8-4-1-3. This means that addition of 5% anthracite (first semi-industrial trial) gives even lower CRI than base blend when testing in the standard CRI test.

Samples reacting in a real BF must be compared to the basket layer they were found as well as the position in the cross-section. Basket layer 14 is positioned 5.4 m down in the EBF® shaft, in relative from the top, and base blend (No.1) has almost no weight loss compared to No. 8 which had 7.8 percentages higher $CRI_{EBF}$. Basket layer 16 are positioned 0.3 m further down in the shaft and when comparing sample No. 3 (5% anthracite addition) with sample No.8 (3% addition of thermal coal and 3% anthracite) the sample with 5% anthracite addition had higher reactivity in the EBF®, 7.7 percentages higher. Note that this coke type had the lowest CRI under standard test conditions.

Basket layers even further down is located within 0.4 m from basket layer 21 (6.1 m) to basket layer 29 (6.5 m) and the $CRI_{EBF}$ increases with the depth of the shaft as well as the graphitisation degree (Lc) of each coke type. However, the weight losses in the actual BF are much lower than in the standard tests as the magnitude of the $CRI_{EBF}$ is much lower supporting the theory that BF atmosphere, with less $CO_2$, contribute less to solution loss in this region of the shaft. These results show similar trends as in the innovative test regime at tk SE pointing towards that the tests of coke reactivity should be performed closer to BF atmosphere to give more realistic values.

### CRI and CSR results - Industrial full scale trials at SSAB coke plant in WP4

The CRI/CSR results, made by DMT, in SSAB 1st cokemaking trial producing alternative coke for EBF® operational trials in Task 4.2 are shown in Figure 40 and in Table 15. In addition, tk SE has tested TC1 and TC2 in their standard CSR/CRI equipment, see results in Figure 77. The differences between the results were minor.

The CRI and CSR for SSAB 2nd industrial cokemaking trial producing alternative coke for full scale operational trials are shown in Figure 113. Averages of CRI and CSR are indicated as black lines in the figures. Some of the weekly samples showed a very small increase in CRI and decrease of CSR, but not at levels that are worrying.

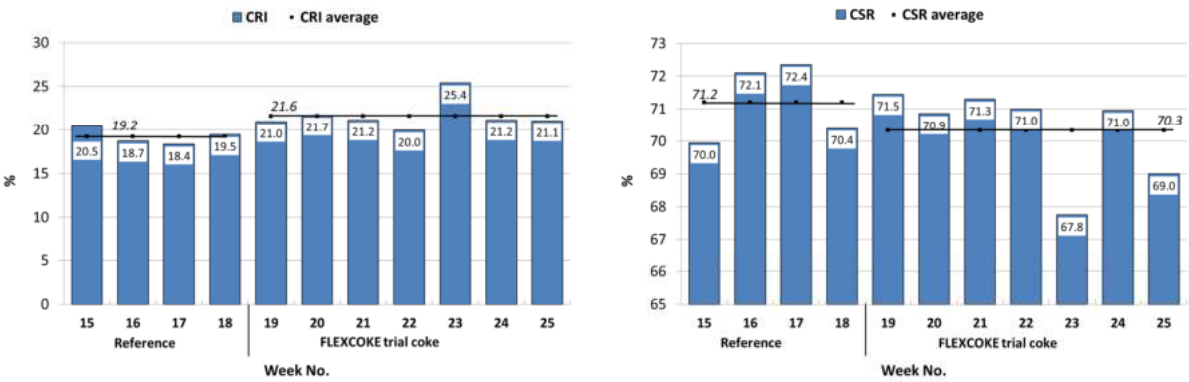



**Figure 113: Coke reactivity index, CRI (left) and Coke strength after reaction, CSR (right). Averages of CRI and CSR tests indicated as the black lines**

The valuations during a year were therefore plotted, see Figure 114. As can be noted there are variations using normal standard blend as well throughout a year and the FLEXCOKE trial coke fits well into these variations, se bold red dots in the figures.

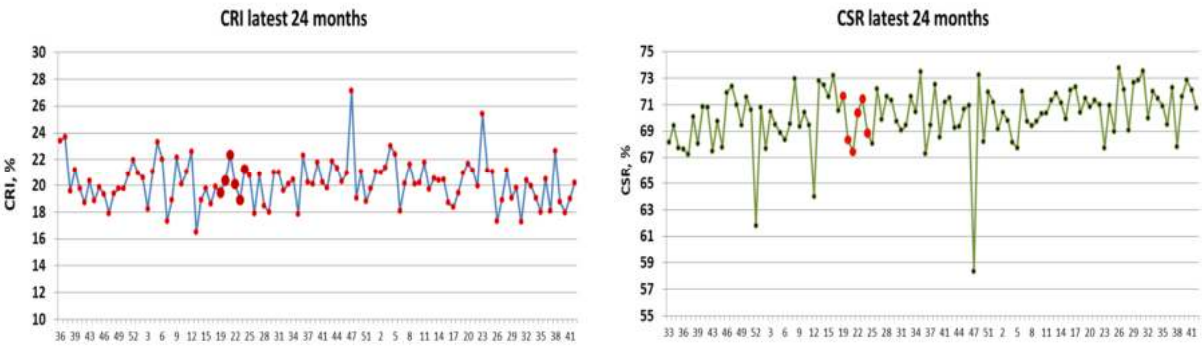



**Figure 114: CRI (upper) and CSR (lower) tested during 24 months, before and after the FLEXCOKE trial illustrated in red, larger dots in the figures**

Conclusions

The additions of the alternative C-containing materials to the base blend always increased the CRI and decrease the CSR. The highest impact could be observed when of 10 % HV thermal coal no FSI or 3% torrefied wood at medium temperature was added to the base blend.

Coke produced at SSAB 1st industrial trial presented similar CRI and CSR values, even at different recipes (Base blend + 5% HV thermal coal no FSI (TC1) and Base blend + 3 %HV thermal coal no FSI + 3% pet coke (TC2)). The CRI gained in the industrial trial at SSAB has a higher quality regarding to CRI and CSR compared to DMT 500 kg semi-industrial coke. It can be thus concluded that a pilot-scale test can only give hints to the resulting quality on an industrial scale, but is not be able to give detailed information on expected CRI/CSR. DMT experience when scaling-up from the semi-industrial oven to industrial coke ovens of 6 m height are that the coke quality as CRI, CSR and cold strength are expected to improve up to around 5 points. This can be confirmed when alternative coke with 5% addition of thermal coal to the blend was carbonised in SSAB 2nd industrial trial. The FTC variations in CRI and CSR were in the range of normal variations producing standard coke.

According to the pre-tests series at tk SE (before erecting the innovative test facility) the CO content increases and $CO_2$ decreases in the arising gas in the reactor during the solution loss reaction. Some tests parameters influence the reaction rate, such as the particle size, temperature and gas concentration. In contrast the temperature and gas concentration are directly proportional to kinetics of the reaction. The ISO standard heating time of 45 min is sufficient for heating the coarser coke sample to the target test temperature in the innovative test facility. The coke grains degrade proportionally more using the ½ Micum drum compared to the ISO drum.

The addition of CO, $N_2$ and $H_2$ to $CO_2$ in the CRI test for a realistic coke characterisation led to a significant decrease of coke reactivity, i.e. the ISO standard CRI is significant higher than the CRI gained when coke is tested in the innovative test under gas atmosphere closer to those at the blast furnace. The application of 2.8 bar during the solution loss reaction increased the reaction rate, but only little change on grain size distribution was observed.

The parameter for CRI/CSR tests should be adjusted in order to characterise coke under conditions closer to a real blast furnace. Table 35 lists a first design of a "new CRI/CSR standard" at tk SE. A new calculation of the load exercised on the coke layer in the blast furnace will be carried out taking in account the reduction development of $Fe_2O_3$ to metallic Fe.

**Table 35: CRI/CSR new standard**

| | CRI/CSR "new standard" |
|---|---|
| Reaction gas | $CO_2$, CO, $N_2$, $H_2$ |
| Temperature | 1100 °C |
| Reaction time | 120 min |
| Grain size | 35 - 80 mm |
| Sample weight | 1000 g up to 2000 g |
| Stress | Simultaneous thermal, chemical and mechanical stress |
| Tumbler type | 1/2 Micum |
| Flow rate | 25 - 50 l/min |
| Load | 2.8 bar |

Due to low $CO_2$ content of the gas in BF simulating test (TRT Mas tests) at gasification temperatures the weight loss of the coke is generally low. Mixing pellets with coke have a positive effect on the pellet reduction degree as all coke type samples give a higher reduction than the single pellet tests. Alternative coke with 3% thermal coal and 3% pet coke addition showed the best influence on pellet reduction followed by coke types with charcoal addition. Coke with charcoal addition also showed higher C conversion and lowest activation energy during thermal gravimetric analysing tests while base blend has showed the opposite. TGA results of selected samples show correlation with the apparent reaction rates and CRI, although coke porosity or microstructure plays a role in the latter.

Comparison with BF simulating test, Standard CSR/CRI test and samples reacting in the EBF® (basket samples) the weight losses in the actual BF are much lower than in the standard tests as the magnitude of the $CRI_{EBF}$ is much lower supporting the theory that BF atmosphere, with less $CO_2$, contribute less to solution loss in this region of the shaft. This results show similar trends as in the innovative test regime at tk SE supporting that the tests of coke reactivity should be performed closer to BF atmosphere to give more realistic values.

Exploitation and impact of the research results

In the blast furnace the most demanding tasks of coke is to ensure the gas flow and the drainage of liquid phases in the cohesive zone. In order to fulfil all tasks during its residence time ensuring an optimal blast furnace performance, the coke with high CSR and low CRI is required. In accordance with the literature the optimum value is <30 % for CRI and >58 % for CSR. A low CSR value means more generation of fines, which disturbs the gas flow and the drainage of the liquid phases in the blast furnace.

CRI and CSR test according to ISO standard is a worldwide known coke property and permits the quality comparison regarding those parameters. However there are many differences between ISO test conditions and those being in an industrial blast furnace. Therefore a realistic coke characterisation is important for an accurate prediction of its behaviour in the blast furnace.

The addition of alternative C-containing material always increased the CRI and consequently decreased the CSR decrease values compared to the reference coke, which was produced using only coking coal (Base blend: Low volatile 40%; medium volatile: 32% and high volatile: 28%). The influence of biomass on the resulting reactivity is different. The addition of charcoal has less impact on the resulting CRI than the addition of torrefied wood.

Both alternative cokes produced at SSAB 1st industrial trial coke, TC1 and TC2, had the required quality regarding CRI and CSR values to be charged in an industrial blast furnace.

The results in the 2nd industrial trial the results were comparable as the standard production coke and DMT experience between their semi-industrial oven and industrial coke ovens of 6 m height is an expected improvement up to around 5 points of CRI, CSR and cold strength. This must be considered when using their semi-industrial oven in the future, especially when adding alternative C-containing materials, which may differ from ordinary coking coals used. This difference between semi-industrial and industrial coke ovens are also important to consider if using biomass in the coal blend for possible future bio-coke production reducing the fossil impact on greenhouse gas emissions.

## WP3: Technical- and pilot-scale test of coke relative blast furnace conditions

### *Task 3.1 High-temperature behaviour*

Planned activities and work accomplished

Original and test coke that meets stated coke plant and BF requirements were produced at SSAB and selected coke types were subjected to actual BF conditions as in basket samples charged into the EBF® (Task 3.2). During operational trial in the EBF® (Task 4.2) two raceway core samples have been taken out and characterised. Basket samples from the EBF® have been analysed and compared with laboratory test regimes with respect to graphitisation and gasification considering impact from in-furnace conditions. The results of the comparison are found in Task 2.4.

Tumbling tests in the CSR/CRI test standard I-tumbler have been made on selected coke samples reacted in EBF®, with special attention to high temperature zone, an activity taken over by MEFOS due to the withdrawal by Tata. Samples collected in WP4 were evaluated in Task 2.1 and the results are reported in WP3 Task 3.2.

A large number of solid samples have been collected during the EBF® trial and during the excavation. The evaluation of the solid samples gives information of changes in size, distribution of coke/hot metal droplets/slag, chemistry/ash of high temperature coke, graphitisation degree (temperature) etc.

Description of activities, results and discussion

**Collection of solid samples from the raceway area of the LKAB EBF®**

*Raceway drilling-cores*

During the LKAB EBF® FLEXCOKE trial in campaign 32 two raceway core-drillings from tuyere 2 were conducted at the end of each evaluation period, i.e. reference period with standard SSAB coke normally used at the EBF® and trial with alternative TC2, i.e. addition of 3% thermal coal with no FSI and 3% of pet coke to the blend.

Operational conditions of raceway area of tuyere 2 four hours prior to drillings are presented in Table 36. The table also shows operational conditions four hours before the stoppage and quenching of the EBF® at the end of the C32 when operating with TC1. Figure 115 and Figure 116 shows the core-drilling steel tube and the radial position of the drilling-core, respectively.

**Table 36: Operational conditions of tuyere 2, four hours prior to stoppage and core-drilling and prior to stoppage before quenching and excavation, TC1 trial period**

| | Reference | | TC2 | | TC1 | |
|---|---|---|---|---|---|---|
| | Average | St. dev. | Average | St. dev. | Average | St. dev. |
| Temp. at tuyere 2, ºC | 1091 | 4.4 | 1094 | 4.3 | 1095 | 4.6 |
| Coal injection, g/Nm$^3$ | 127.0 | 3.4 | 127.2 | 1.3 | 127.7 | 3.5 |
| Blast temperature, ºC | 1198 | 9.4 | 1199 | 8.4 | 1198 | 8.9 |
| Blast flow, Nm$^3$/h | 1623 | 7.5 | 1626 | 9.6 | 1628 | 10.3 |
| Blast flow in tuyere 2, Nm$^3$/h | 506 | 9.0 | 521 | 7.2 | 516 | 6.2 |
| Blast speed, m/s | 134.7 | 0.78 | 135.7 | 0.98 | 134.7 | 0.81 |
| $O_2$ flow in lances, Nm$^3$/h | 53.7 | 0.66 | 53.7 | 0.54 | 54.0 | 2.58 |
| $O_2$ flow to blast, Nm$^3$/h | 50.3 | 1.19 | 50.4 | 1.23 | 51.1 | 2.83 |
| %$O_2$ | 24.3 | 0.06 | 24.3 | 0.06 | 24.3 | 0.13 |

**Figure 115: Drilling at tuyere 2**

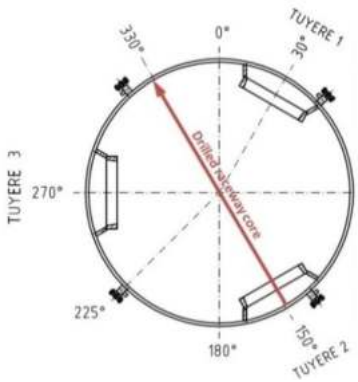



**Figure 116: Position of the raceway core**

The raceway drilling-cores have been opened and photographed before dividing the core into 0.1 m sub-samples as shown in Figure 117, left side shows the reference case when the EBF® was operating with standard coke and the right side operation with the alternative TC2.

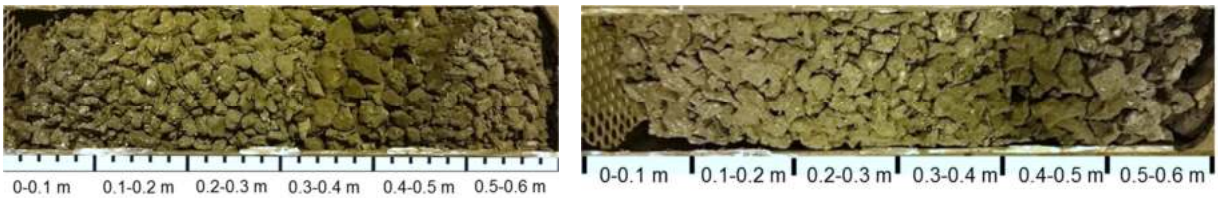



**Figure 117: Raceway drilling-cores when operating with reference (standard coke) (left) and when operating with alternative TC2 (right)**

Each sub-sample have been screened in fractions >16, 10-16, 5-10, 2.8-5, 1-2.8, 0.5-1 and <0.5 mm. Fractions >16 to 5-10 mm have been sorted in coke, slag and metal (magnetic). Agglomerates contain mainly coke pieces in where the slag or metal have acted as glue.

In addition, fractions 2.8-5 and 1-2.8 have been magnetically separated. The sorted coke was photographed as seen in Figure 118. The coke fraction 5-10 mm and the fines <0.5 mm from all sub-samples were chemically analysed and the graphitisation degree of coke was analysed.

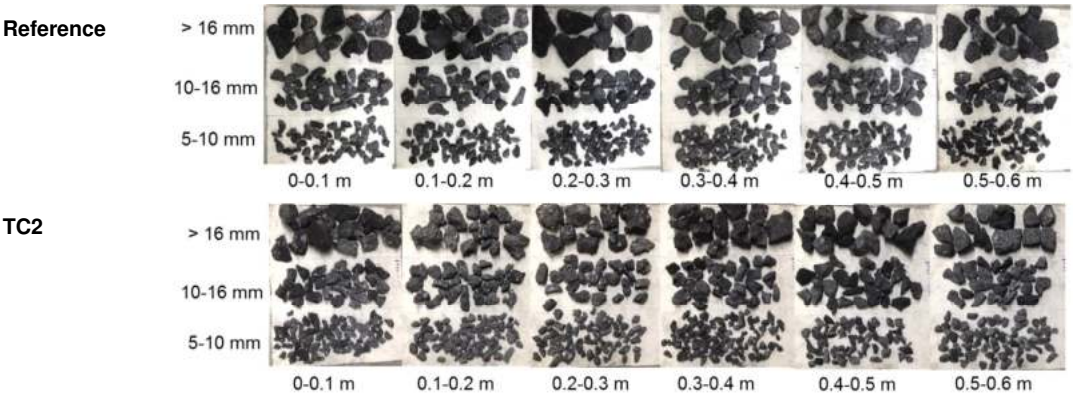



**Figure 118: Coke particles of the three coarsest fractions**

*Particle size and material distribution*

The weighed amounts of coke, slag, magnetics and aggregates in the raceway drilling-cores sub-samples separated in the fractions >16 mm, >10 mm and >5 mm are illustrated in Figure 119.

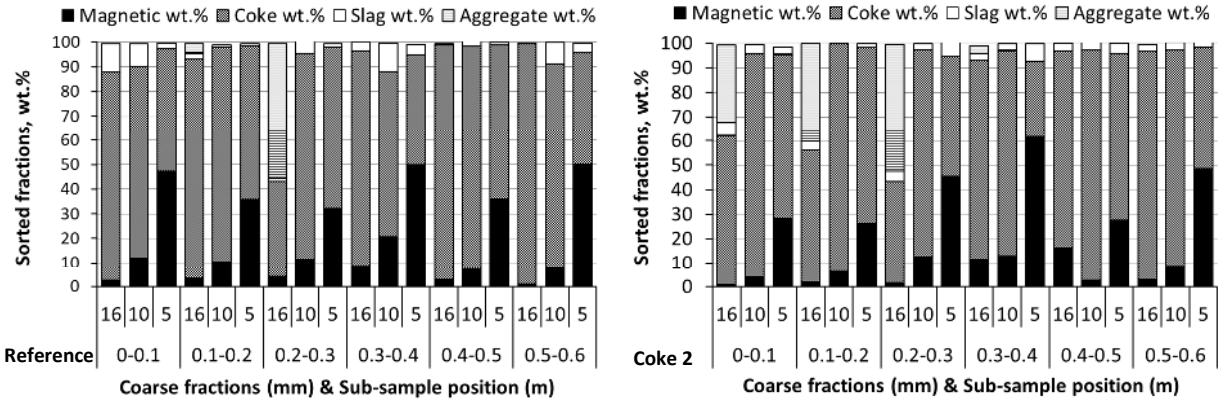



**Figure 119: Material distribution in fractions >16 mm, 10-16 mm and 5-10 mm for all sub-samples, reference coke (left) and TC2 (right)**

The particle size distribution of sub-samples in the drilling-cores for reference coke and TC2 with the three coarsest fractions showed that the formation of aggregates in the fraction >16 mm was found a higher extent during the trial with TC2 compared to with reference coke. The drilling-core with TC2 contained aggregates in the sub-samples from the wall up to the position 0.4 m towards the EBF® centre. Reference coke sub-samples contained fewer aggregates of material and therefore also higher amounts of coke in fraction >10 mm in the same position as for TC2. In general, the ratios of coke in the fraction >5 mm were higher in TC2, except in some sub-samples were the ratio of magnetic material was high and therefore the ratios of coke became lower.

The total weighed material >5 mm and the distribution of fines <0.5 mm in respective position in the cores can be seen in Figure 120. In addition the total amount of other material than coke >5 mm is plotted in Figure 120. This illustrates the occurrence of other sorted material as magnetic material, aggregates and slag and TC2 drilling-core has higher amounts of magnetic material, aggregates and slag than the reference coke drilling-core.

Both raceway drill-cores have lower amounts of coke at position 0.2 to 0.4 m and an increase in fines <0.5 mm at position 0.3-0.4 m indicates the end of raceway and a bird's nest. The amount of fines <0.5 mm was in almost all sub-samples lower in TC2 compared to the reference coke drilling-core.

The amounts of magnetic material indicate the possibility for molten metal to flow in the coke bed. As can be seen in Figure 121, the differences (gap) between the amounts of >5 mm and >1 mm was higher in drilling-core with reference coke than in the one with TC2, which indicates that the amounts of magnetic material in the particle size 1-5 mm is higher in the drilling-core with

reference coke, in particular in the sub-sample with position 0.3-0.4 m from the wall (at bird's nest position) and decreased after raceway towards the centre.

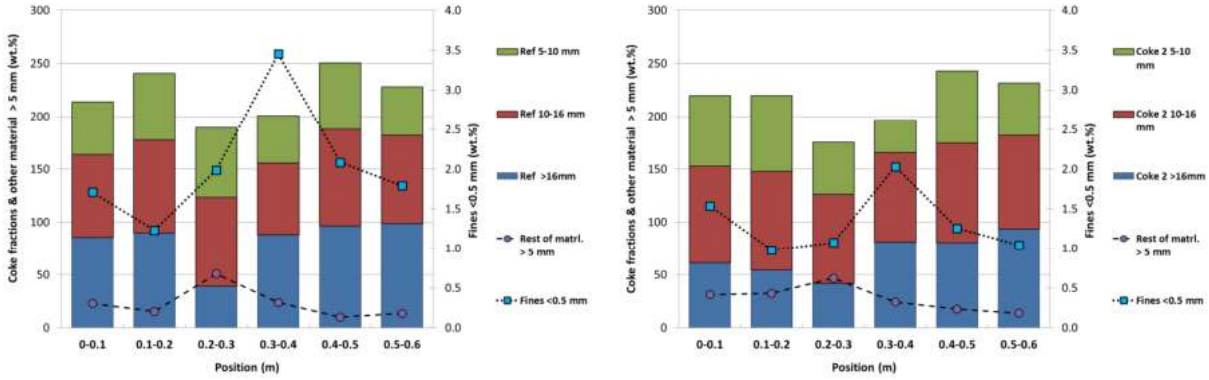



**Figure 120: Amounts (wt.%) in coke for the three coarsest fractions and amounts of fines <0.5 mm in each sub-sample. Reference coke (left) and TC2 (right)**

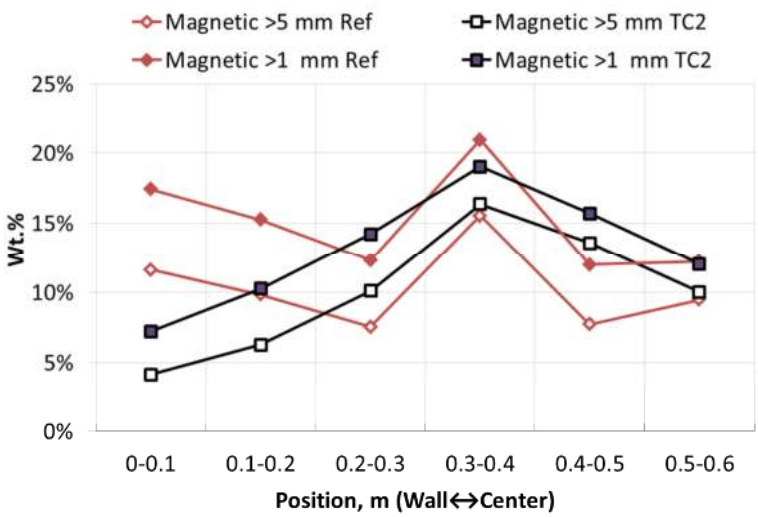



**Figure 121: Amounts of magnetic materials >5 mm and >1 mm in raceway core-drillings**

*Chemical composition and coke graphitisation degree*

The chemical composition of coke in fraction 5-10 mm has been analysed with XRF. In Figure 122, core-drilling samples from reference coke and TC2 are compared. The sum of oxides represents the ash content in coke. The sums of oxides in the charged coke are 11.6% in reference coke and 11.9 % in TC2, so it is clear that the ash components are significantly higher in the collected raceway coke.

The values for the ratio $SiO_2/Al_2O_3$ (round dots) in both coke types were lower than the ratio for charged coke values (~2.14 in standard coke and ~2.16 in TC2), which indicates a reduction and gasification of silica in the raceway high temperature area. The quotient in both standard coke and TC2 is lower in the position 0.1-0.3 m indicating a higher gasification of Si compounds in those positions. In general, the quotient was higher in the sub-samples close to the wall and at the position 0.4-0.5 m near to BF centre.

In addition, the alkali ($K_2O+Na_2O$) content (rhombic dots) was lower at the same positions (0.1-0.3 m) in both raceway drilling-cores; this indicates that the alkalis have been reduced and gasified in these positions, particularly for TC2. These samples contain mainly coke that has been rotating in the raceway.

The coke graphitisation degree with high Lc values of 76-108 Ångstrom indicates high temperatures as expected in this region. Figure 123 shows the raceway drilling-core coke graphitisation degrees in each sub-sample with corresponding estimated temperature made.

The estimated temperature profiles indicate a higher temperature initially behind the tuyere nose, at the wall, and then a temperature drop towards the end of the raceway, with lowest temperature at bird's nest position. Further in towards the centre an increase in graphitisation degree and estimated temperature are noted. The high temperature of coke at position 0-0.2 m may be

explained by a redirection of gas towards the wall by occurrence of bird's nest. The actual temperatures in the raceway are probably even higher than the estimated temperatures.

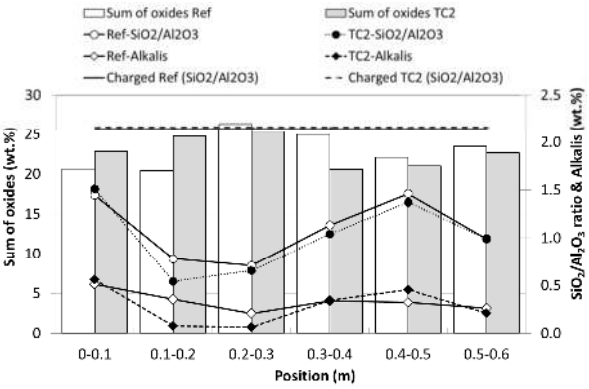



**Figure 122: Sum of oxides (ash content), alkalis and $SiO_2/Al_2O_3$ ratio in raceway drilling-cores**

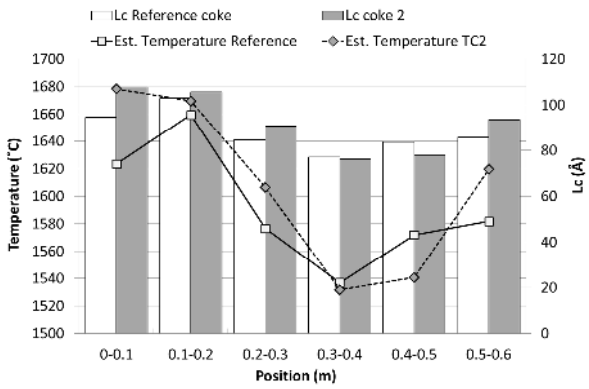



**Figure 123: Graphitisation degrees (Lc) of coke in raceway drilling-cores sub-samples with corresponding temp. estimations**

However, the higher $SiO_2/Al_2O_3$ ratios in the first sub-samples in Figure 122 do not correlate with the high Lc and temperature exposure. The alkali contents in some sub-samples do correlate to estimated temperature and the theory of reduction and gasification of alkali vapour from coke ash but not in all cases. Especially the high temperature in raceway, and to some extent also other high temperature zones contribute to the reduction of $SiO_2$ to $SiO_{(g)}$. Therefore a reduction of the $SiO_2/Al_2O_3$ ratio is often seen as an indicator as $Al_2O_3$ do not behave the same.

Gasification of C in coke by solution loss reaction in the high temperature area and in raceway weakens the outer layer of the coke. The abrasion caused by coke tumbled in raceway may have abraded some of these outer layers, leaving in the remaining, inner part of coke pieces that yet has not being reduced to the same extent than the outer layer. The C content of the fractions of fines <0.5 mm are higher in samples collected close to bird's nests (0.2-0.4 m) in both drilling-cores indicating abrasion of C in raceway coke. The theory may be supported to some extent by looking into the chemical composition of fines <0.5 mm, see Figure 124. At the same position the $SiO_2/Al_2O_3$ ratios goes up. The C content of the fines are higher in position close to bird's nests (0.2-0.4 m) in both drilling-cores indicating abrasion of C in raceway coke. However the elevated C may also origin from un-burnt char from PCI.

The Fe content in almost all sub-samples was slightly higher in TC2 than in reference coke, and also higher in the sub-samples close to the wall and the centre. In general, the Fe contents in the positions 20-40 cm, for both cokes, were lower than all the other sub-samples analysed. In addition, the C content in the fine fraction of TC2 was lower than in reference coke, which indicates a lower degradation or also a lower consumption of C from the coke.

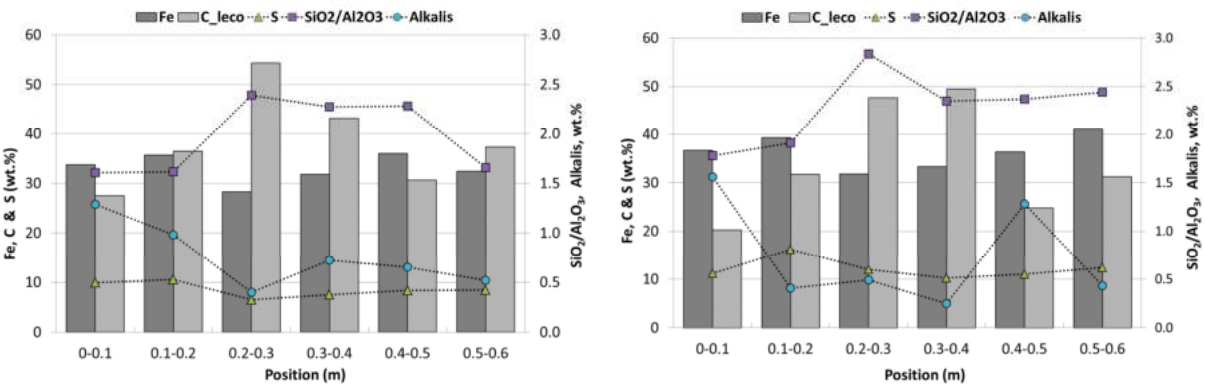



**Figure 124: Chemical analyse of fines <0.5 mm in each sub-sample of raceway drilling-cores. Reference coke (left) and TC2 (right)**

*XRD investigation of phases*

The coke sub-samples of fines <0.5 mm and 5-10 mm were analysed in X-Ray Diffraction (XRD) over an angle range from 10-90°, the illustration of the XRD diffractions patterns and identification of phases are shown in Figure 125.

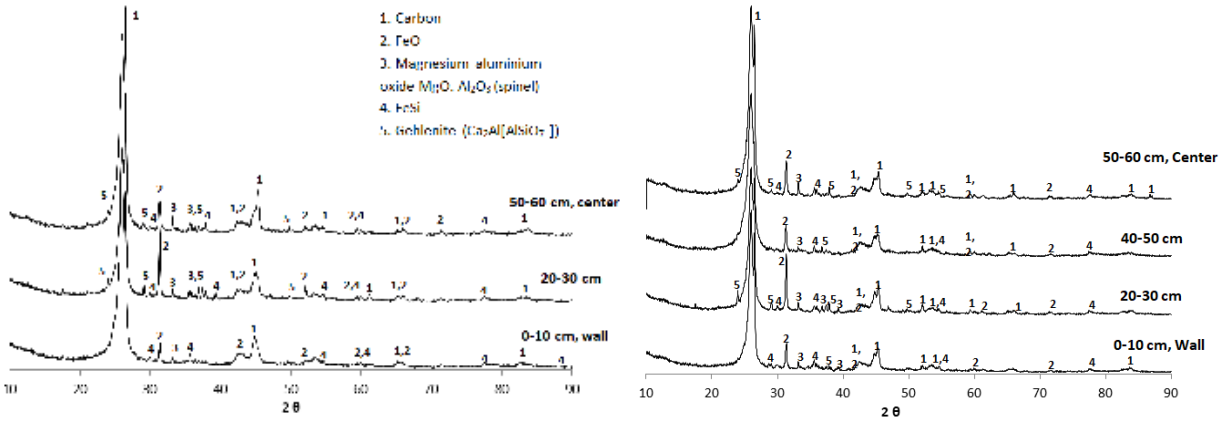



**Figure 125: XRD diffraction spectra in selected sub-samples 0-0.6 m for sub-samples of TC2 (left) and reference coke (right) from raceway drilling-cores**

According to the diffraction patterns in Figure 125, the dominating peaks observed corresponded to carbon in coke at ~20-26°. Other key components detected were for Wustite (FeO), Magnesium aluminium oxide ($MgO.Al_2O_3$, spinel), FeSi and Gehlenite. The peak corresponding to FeO varied in intensity, in particular at the position of 0.2-0.3 m. The peaks that correspond to Gehlenite, at ~20-22°, could only be observed in both drilling-cores at positions 0.2-0.3 m and 0.5-0.6 m.

The intensity of silica compounds in TC2 at ~30-40° was seen to decrease in the sub-sample close to the centre. Close to the centre at 50-60 cm and ~30-35°, the intensity of the spinel seemed to increase and other components as FeSi and Gehlenite partially decreased at high temperature. The last was more notable in the spectra for sub-samples from standard coke.

Figure 126 shows the diffraction patterns and phases in fines <0.5 mm for both drilling-cores and coke types. A peak corresponding to the carbon in coke was positioned at ~20-26° the coke carbon peak has very high intensity and in raceway fines samples corresponds to graphite rather than quartz as in in-going coke. Also, the peak at ~50-60° is present in all sub-samples and it can be caused by a carbon phase or a graphite phase. Almost all sub-samples contained two peaks of Gehlenite with difference intensities. In addition, small amounts of SiC and $Fe_3Si$ were also detected; the last mentioned was only detected in TC2 at the position 0.5-0.6 m.

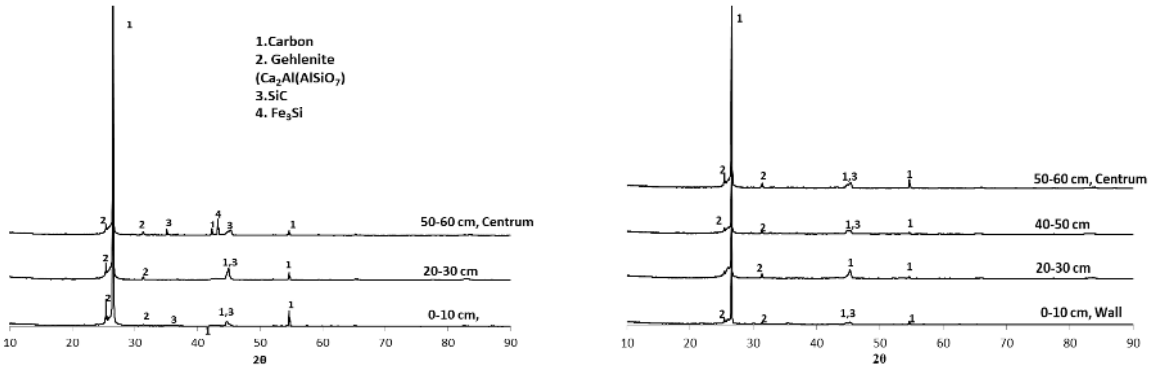



**Figure 126: XRD diffraction spectra for fines <0.5 mm in selected sub-samples 0-0.6 m for sub-samples of TC2 (left) and reference coke (right) from raceway drilling-cores**

Earlier laboratory graphitisation study[9] regarding with coke submerged into hot metal or slag showed additional peaks of $MgAl_2O_4$ spinel when submerged in slag and SiC and $Fe_3Si$ phases when submerged into hot metal. The XRD results indicates that the coke in raceway has been subjected to slag, or perhaps from own ash, according to the appearance of the spinel The outer part of coke, abraded into fines could have been subjected to the hot metal and ended up in the fines according to the phases shown. The fines may also come from char with similar behaviour.

*Mechanical strength of raceway drilling-core coke*

Coke acts as a mechanical support for the burden in the BF process. The coke strength tends to decrease when coke is exposed to high temperature, with carbon gasification leaving the coke outer layer more porous or open. It can also be abraded by the motion of particles in raceway. Additionally, ash components as silica and alkali oxides may be reduced and gasified.

The mechanical strength of the coke from the raceway area was analysed in order to compare the behaviour of standard coke (reference) and TC2. The tests were made as follows: 200 grams of coke from each sub-sample in fraction 19-22.5 mm were placed in the CRI/CSR standard test ISO tumbler and tumbled and sieved accordingly to the standard test. Coke strength after reaction in the BF raceway "$CSR_{EBF\ raceway}$" was determined by calculating the tumbled and sieved high temperature coke in the same way as in the standard CSR determination.

The mechanical strength ($CSR_{EBF\ raceway}$) of both reference and TC2 raceway drilling-core is illustrated in Figure 127. The lines represent the CSR determined by the standard test.

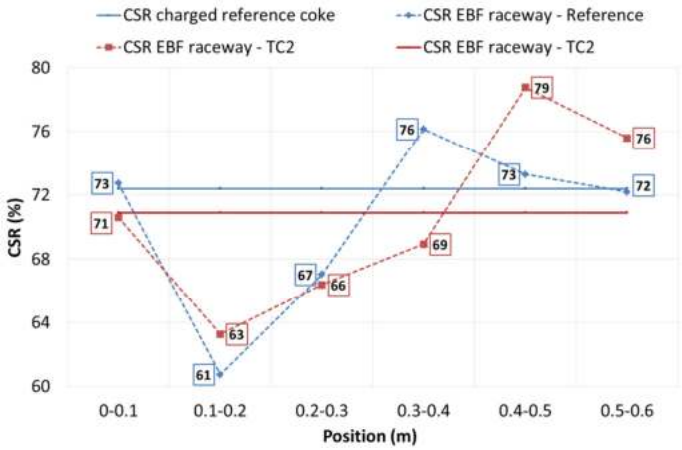



**Figure 127: Mechanical strength of raceway coke ($CSR_{EBF\ raceway}$) for reference coke and TC2 drilling-core sub-samples**

Figure 127 shows that the strength was lowest close to the wall in sub-samples at the position 0.1-0.2 m in both drilling-cores. The graphitisation degree in Figure 123 and temperature estimations were higher at the positions 0.1-0.2 m, which can explain the low strength in position 0.1-0.2 m but the trend with high temperature and mechanical strength was not consistent. TC2 has similar and in some positions even higher coke strength than the reference coke.

However, the $CSR_{EBF\ raceway}$ is still very high, almost as high as compared to CSR in the standard CRI/CSR test at position 0-0.1 m, indicating that colder bosh coke has fallen down in the raceway cavity when the EBF® was stopped. Further into the centre of the EBF®, raceway sub-samples at position ~0.4 to 0.6 m have even higher $CSR_{EBF\ raceway}$ compared to charged coke. The same trend can be observed in to the chemical composition of alkali and especially the $SiO_2/Al_2O_3$ ratio in Figure 122. To sum up the results pointing towards evidence for a bird's nest formed in the end of the raceway when operating on both coke types. The high values of $CSR_{EBF\ raceway}$ compared to coke reacting in the standard CRI/CSR test atmosphere shows that both the reference coke and TC2 withstand the conditions that occurs in the high temperature area quite well even though the temperature is much higher than in the standard test. The cokes that probably have been inside the raceway have somewhat lower $CSR_{EBF\ raceway}$.

*Excavation of TC1 at high temperature tuyere level*

At the end of campaign 32 the excavation of the EBF® was made on TC1, i.e. coke with addition of 5% thermal coal with no FSI to the blend. The excavation of the high temperature area was made according to Figure 128. In the evaluation most focus has been made to tuyere 2 samples, the same position of the raceway drilling-cores, at Level 2. Samples from above tuyere level, Level 1, and below, Level 3 were also studied.

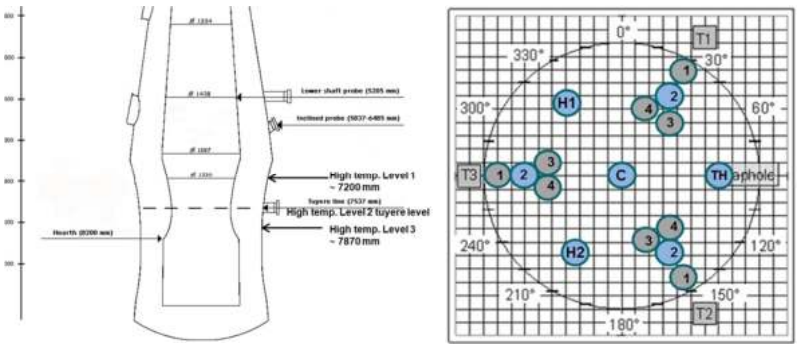



**Figure 128: Positions of high temperature level sampling during excavation, TC1**

*Level 2, tuyere level and raceway excavation samples of TC1*

The material distributions at tuyere level in all three tuyeres for the coarser fractions are plotted to the left in Figure 129. To the right the distribution of the coarser fractions and the amount of fines >0.5 mm are shown.

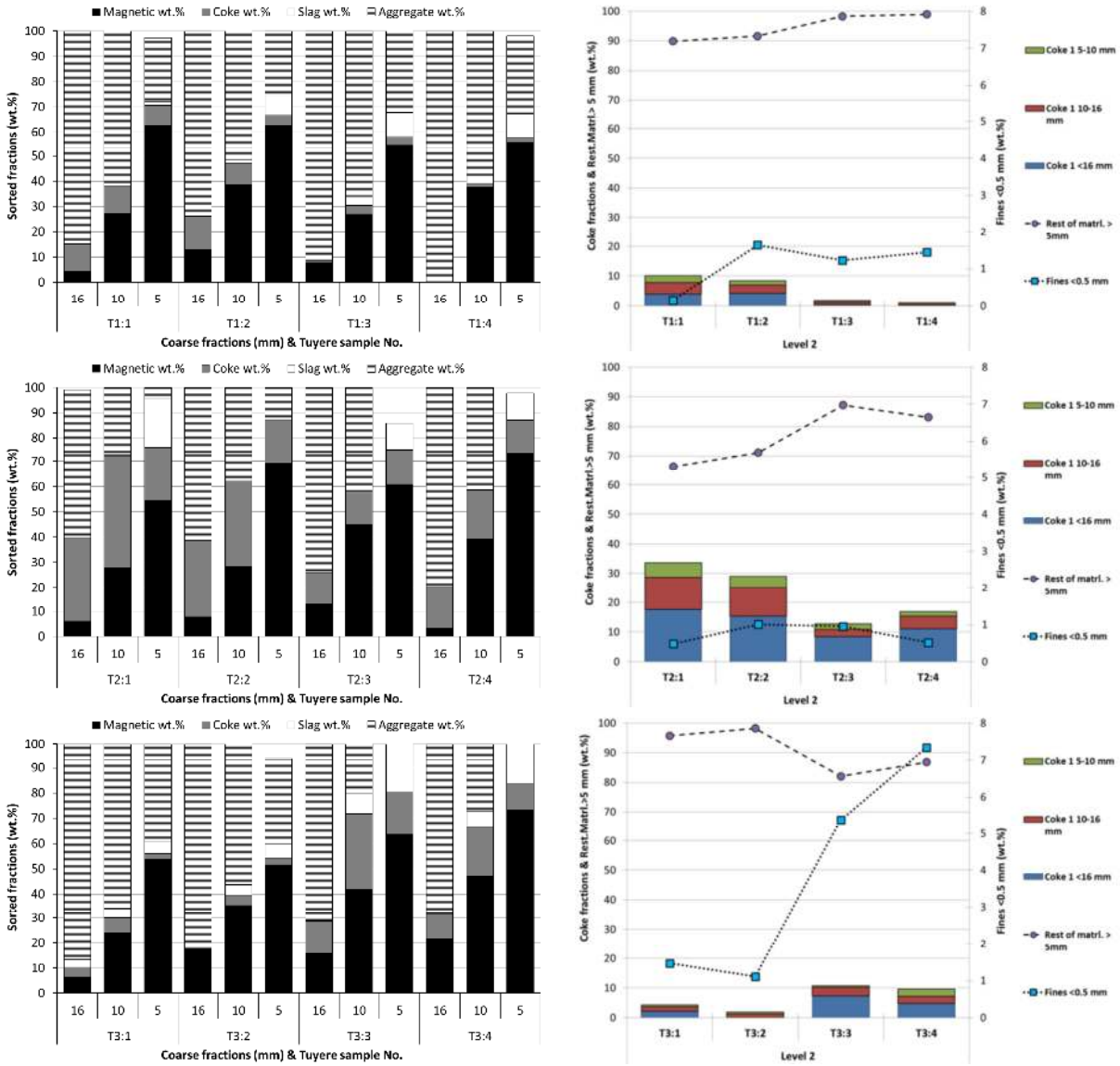



**Figure 129: Material distribution at tuyere level in fractions >16 mm, 10-16 mm and 5-10 mm (left side) and amounts in TC1 for the three coarsest fractions and fines <0.5 mm (right side). Tuyere 1 (upper), tuyere 2 (middle) and tuyere 3 (bottom)**

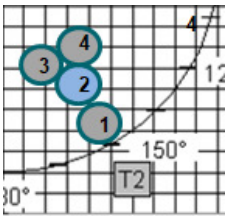
The highest wt.% of magnetic material in the tuyere area of Level 2 was found in the particle sizes 5-10 mm. Samples taken close to the wall (T1-3:1) and the samples placed 14-15 cm from the wall (T1-3:2) contained a higher wt.% of coke than the samples toward the centre, in particular in the fractions larger than 16 mm and 10-16 mm. The formation of aggregates indicates a lower temperature in the area. It is also an indication of the formation of Bird's nest at the end of the raceway. Tuyere 1 (T1) and 2 (T2) showed a high formation of aggregates in the samples toward the centre, T1-2:3 and T1-2:4.

The total amounts of material larger than 5 mm in comparison with the coke amounts in the three coarsest fractions indicates that the samples taken in the area consisted mainly of other materials (slag, aggregates and magnetic material). The gaps between the wt.% of other materials >5mm and wt.% of coke was high, aside from samples in T2 that contained a higher wt.% of coke. Tuyere 3 (T3) contained a higher content of fine material <0.5 mm. This can be an indication of higher degradation of the coke. The increase of small fractions can also be a contribution of char particles from coal injection.

Based on the graphitisation degrees and the temperature estimations, see Table 37, it seems like T2 raceway TC1 had been subjected to the lower side of the temperatures estimated for the coke samples collected by core-drillings in raceway, see Figure 123. The position of excavation samples

are not as accurate as for the samples collected by core-drillings and some coke from above or the side of raceway may be part of the analysed sample.

**Table 37: Graphitisation degrees and temperature estimations of T2 raceway excavation sample 1-4 of TC1**




| Position T2 | Lc (Å) | Temperature estimation (°C) |
|---|---|---|
| 1 | 78.3 | 1543 |
| 2 | 87.7 | 1592 |
| 3 | 87.6 | 1591 |
| 4 | 85.7 | 1582 |

Figure 130 shows the chemical composition of the TC1 fraction 5-10 mm for excavated raceway samples at Level 2. T2 and T3 contained normally higher content of CaO and the sum of oxides were lower in almost all of the samples toward the centre of the furnace. The $SiO_2/Al_2O_3$ ratio was lower than the one for charged coke which also indicates that silica was reduced and $SiO_{(g)}$ evaporated due to the high temperature and strongly reducing conditions further down in the BF. Also the alkali was low in almost all samples indicating that accumulated alkali has been reduced and evaporated in the high temperature area. The ash content in coke, which is presented in form of the sum oxides, decreased from the wall towards the centre of the furnace.

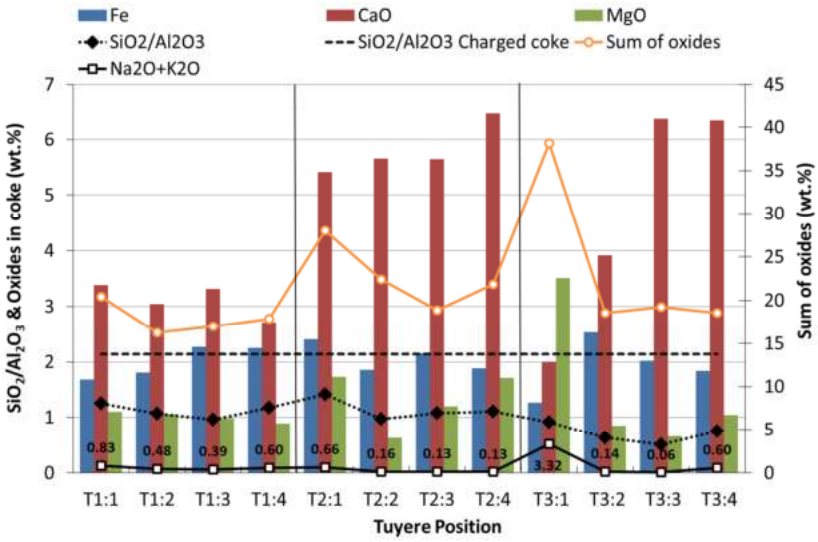



**Figure 130: Chemical analyse of raceway samples at tuyere level, Level 2**

Figure 131 indicates that the material at the centre of the level contained higher amount of slag and aggregates and less wt.% of coke compared to the tap hole (TH) and in-between the tuyeres (H1 and H2). Magnetic material was most commonly in the fraction 5-10 mm. This indicates a higher flow of melt in-between tuyeres in the EBF®.

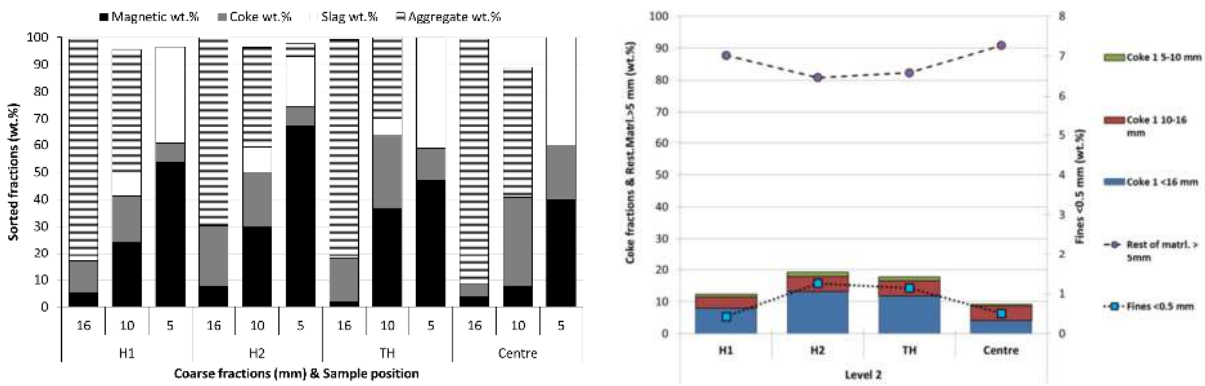



**Figure 131: Material distribution in samples H1, H2, TH and Centre at tuyere level, fractions >16 mm, 10-16 mm and 5-10 mm (left side) and amounts in coke for the three coarsest fractions and fines <0.5 mm (right side)**

The chemical analyses performed on coke samples positioned in-between the raceway area, see Figure 132, at the tuyere level showed an increased amount of ashes and CaO. The alkali content in coke in-between raceways is not as low as in the raceway samples.

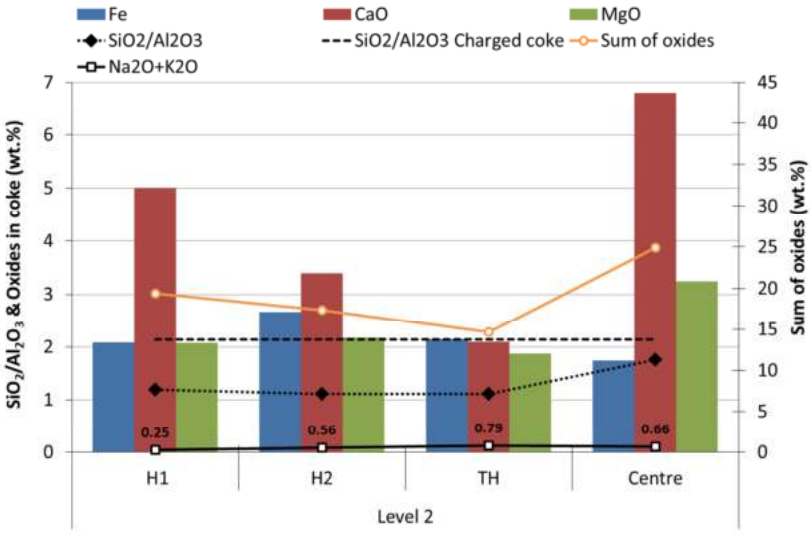



**Figure 132: Chemical analyse of in samples H1, H2, TH and Centre at tuyere level, Level 2**

*Level 3, around 0.3 m below tuyere level*

Excavation samples below the tuyere level were screened and sorted the same was as in Level 2. The material distribution in the three coarsest fractions can be seen in Figure 133 as well as the coke distribution and the amount of fines 0.5 mm.

At Level 3 it can be seen that the wt.% of magnetic material and coke were higher in major extent than in samples at Level 2. The formations of aggregates were much lower. It can also be seen that the coke proportions were higher in this level of the high temperature area. Material at this level might as well be material from the combustion zone and the beginning of the melt bath which explain the increased amounts of magnetic.

The amounts of fines <0.5 mm was much higher at Level 3 than at tuyere level. The three samples closest to the centre and in particular in the area below T2 contained higher amount of fines. Char particles coming from the raceway area and increased degradation of the coke can be the explanation.

The material distributions of samples excavated below the tuyere level in the centre and in-between the raceways and above the tap hole are shown in Figure 134.

The distribution of coke in the centre of the furnace was lower than above the tap hole position and in-between the tuyeres (H1, H2) positions. A higher amount of magnetic material was found in the particle sizes 5-10 mm. The material distributions in samples excavated in the centre and in-between the tuyere areas, are similar to the distribution of material excavated at Level 2, see Figure 131.

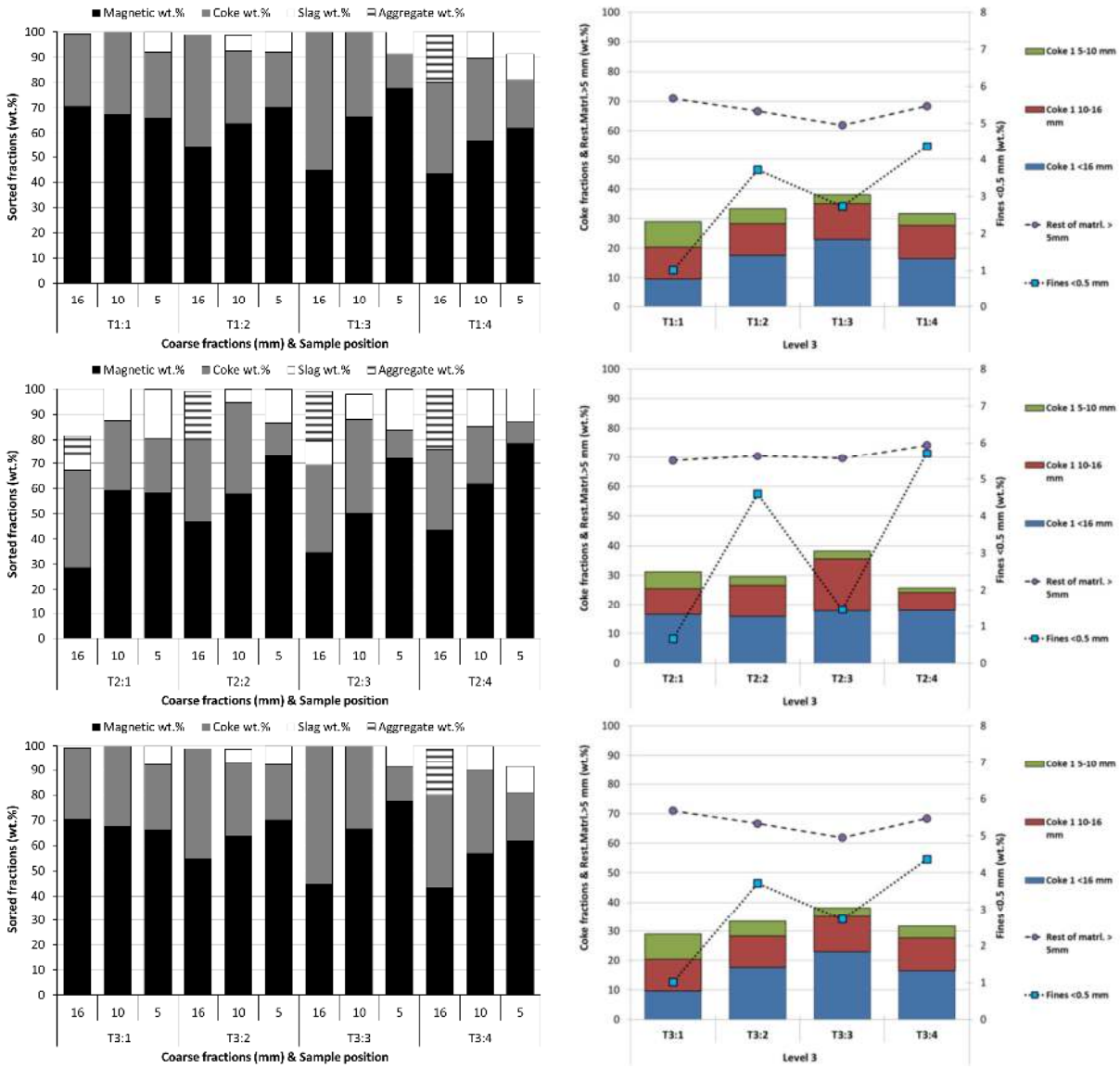



**Figure 133: Material distribution below tuyere level in fractions >16 mm, 10-16 mm and 5-10 mm (left side) and amounts in coke for the three coarsest fractions and fines <0.5 mm (right side). Below tuyere 1 (upper), tuyere 2 (middle) and tuyere 3 (bottom)**

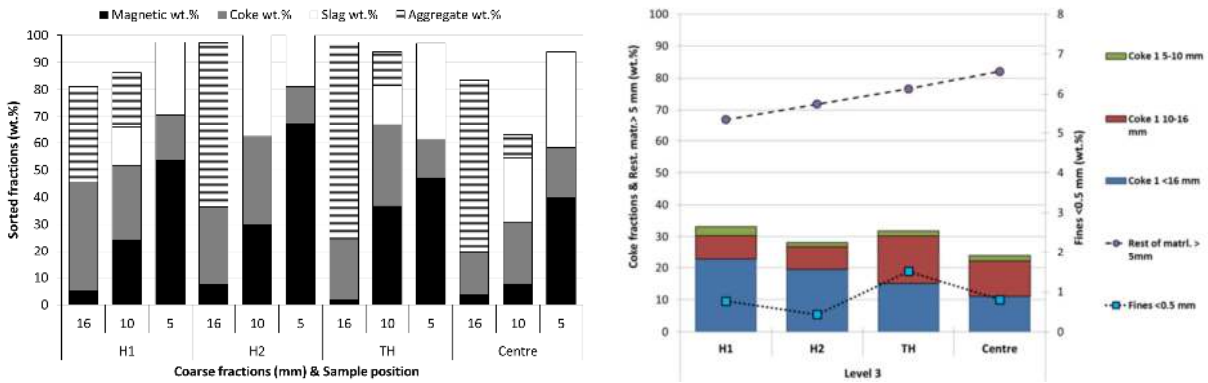



**Figure 134: Material distribution in samples H1, H2, TH and Centre below tuyere level, fractions >16 mm, 10-16 mm and 5-10 mm (left side) and amounts in coke for the three coarsest fractions and fines <0.5 mm (right side)**

*Mechanical strength of high temperature TC1 excavation samples*

Coke mechanical strength of samples collected during excavation of the shaft (described in more detail in Task 4.2) and the high temperature area mechanical strength of the EBF® ($CSR_{EBF}$) were determined, see Table 38. Samples with similar chemical content and from the same level was needed to be mixed in order to get representative amount of sample for $CSR_{EBF}$ analyses. The

$CSR_{EBF}$ were obtained as described earlier in the section of the race way drilling-cores determination of $CSR_{EBF\ raceway}$.

Selected shaft samples in Task 4.2 was also investigated and reported in Table 38. The coke strength normally decrease on its way down to the bottom of the BF, in particular in the high temperature area, this was also the behaviour of TC1. It can be seen in Table 38 that the coke strength, $CSR_{EBF}$, was around 88% in shaft sample Coke layer 4 and around 71% further down at Level 3 (below tuyere level) where the temperature in the BF is much higher than in the shaft. It was noted that the coke surface structure seemed to be more deteriorated in the centre of the Level 2 which indicates a higher temperature in that area. CSR for the charged TC1 was on average 70.9%, see Table 15. This means that all tumbled TC1 in Table 38 has a higher strength after reaction in a real BF than after the standardised CSI/CSR test.

**Table 38: Mechanical strength ($CSR_{EBF}$) of TC1 collected in the shaft and the high temperature area during excavation of EBF® C32**

| Position | Sample No. | $CSR_{EBF}$ [%] | Level from top of the EBF® [m] |
|---|---|---|---|
| Shaft | Coke layer 4 (1) | 88.1 | 3.9 |
| | Coke layer 7 (1) | 84.8 | 5.9 |
| | Coke layer 8 (2-3) | 84.8 | 6.5 |
| Level 1 | T1 (3-4) | 82.4 | 7.1 |
| | T2 (3-4) | 82.8 | 7.1 |
| | T3 (3-4) | 80.7 | 7.1 |
| | Centre | 82.4 | 7.1 |
| Level 2 | T2 (2-4) | 77.3 | 7.5 |
| | Centre | 68.3 | 7.5 |
| Level 3 | T1 (3-4) | 71.1 | 7.9 |
| | T2 (3-4) | 71.0 | 7.9 |
| | T3 (3-4) | 71.1 | 7.9 |

**Collection of solid samples from the hearth of the LKAB EBF®, C32**

After excavation of the shaft and high temperature level above the hearth the shaft was detached and the materials in the hearth of the EBF® were exposed. The solid samples, which later on referred to as core-drilling samples, in the hearth of the EBF® was procured by drilling steel tubes (diameter 130 mm and height 500 mm) into the bed of the hearth, as shown in Figure 135.

**Figure 135: Attaining the solid samples by core-drilling into the bed of the EBF®'s hearth**

The core-drilling samples were procured from two layers and a number of different positions (with respect to the positions of tuyeres and tap hole) in the hearth of EBF®. The core-drilling has in general made the distribution of materials in the tube the same as it was in the hearth. In total 28 core-drilling samples were obtained: 7 from the first layer and 21 from the second layer. The positions with respect to the three tuyeres and tap hole of these core-drilling samples as well as their reference numbers are illustrated in Figure 136.

From Figure 136 it is also seen that some tubes, especially the tubes from the second layer core-drilling, are protruding above the bed level. This is due to the presence of hard materials, essentially the metals and metal-containing aggregates, which have hindered the core-drilling further down into the bed. Therefore, some of the drilling tubes are not fully filled with solid samples. In these cases some filling materials (clay pellets) as shown Figure 137 were placed in the tube to make the materials stabilised. Figure 137 shows the appearance of the materials in the tubes and the relative positions of the drilling cores into the hearth of the EBF®.

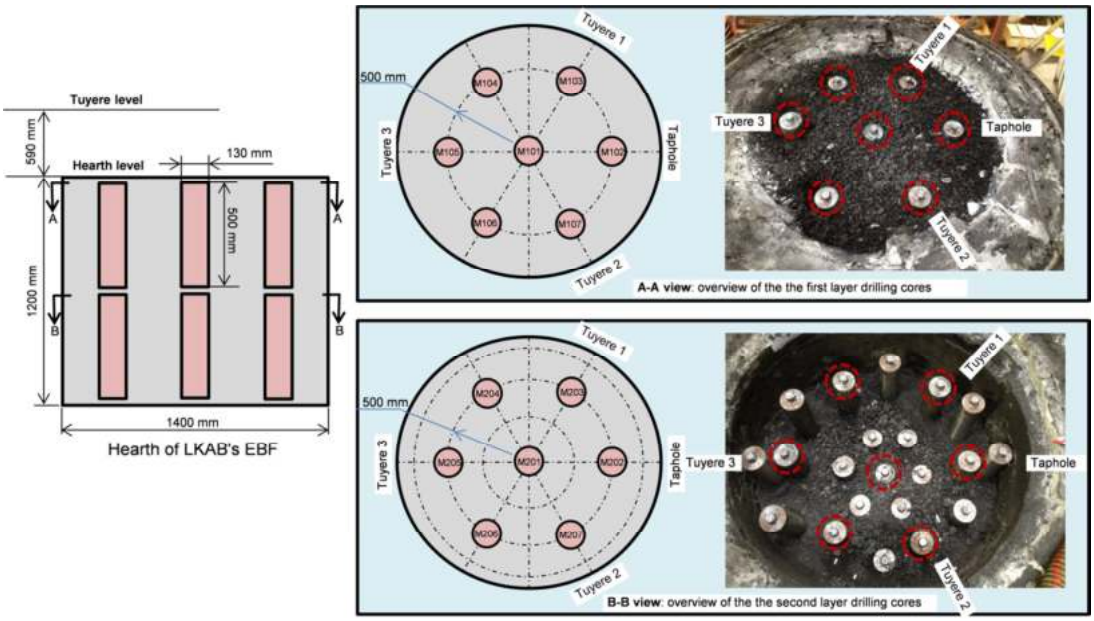



**Figure 136: Illustration of the sampling positions of the core-drilling samples as well as their reference numbers. FLEXCOKE core-drilling tubes are circled with red-dash lines in the figure**

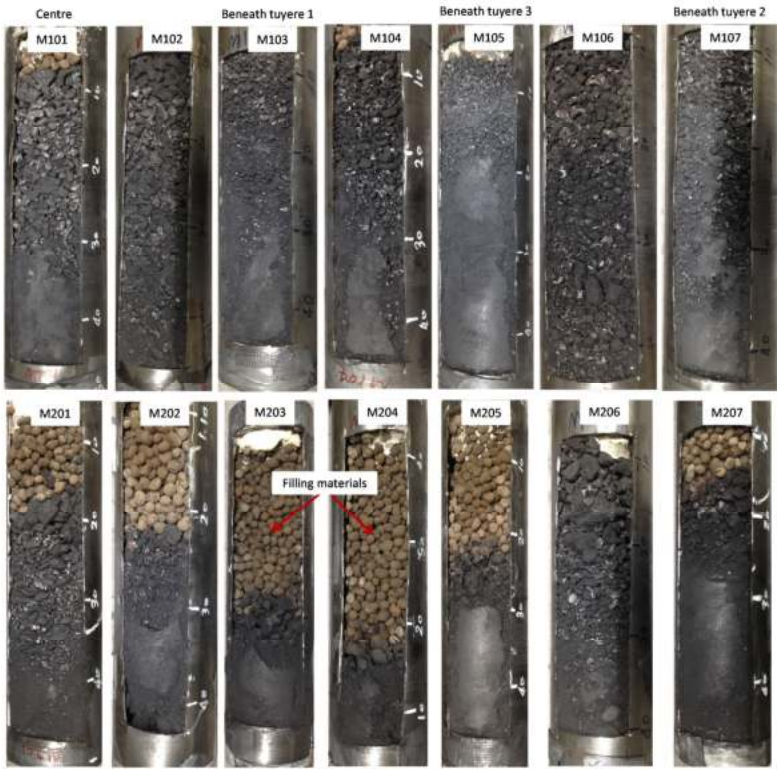



**Figure 137: Appearance of the materials in the core-drilling tubes**

The relative positions (with respect to the depth into the hearth) of the procured core-drilling samples are shown in Figure 138.

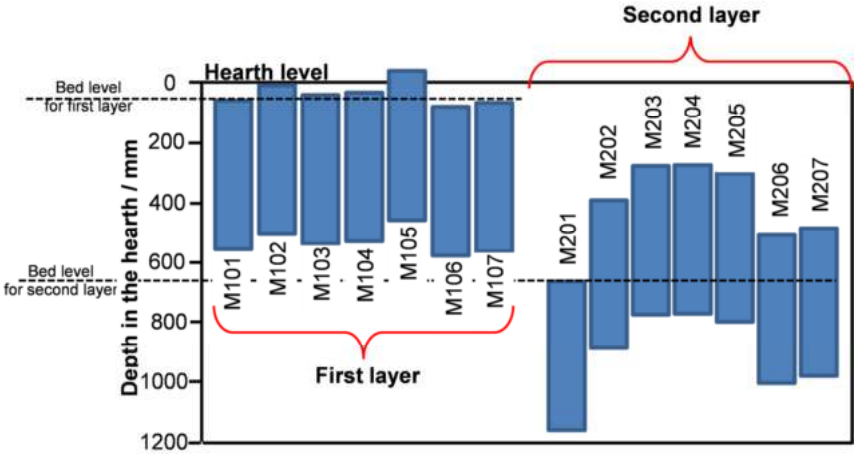



**Figure 138: Illustration of the sampling positions with respect to the depth in the hearth**

*Physical characterisation*

The core-drilling samples procured from two layers in the hearth of the EBF® were firstly physical characterised with the same procedure as in raceway drilling cores. Each of the core-drilling samples was opened by cutting a window from the side of the drilling tube and the materials in the tube photographed and divided into 10 cm sub-samples (up to 5 sub-samples in each hearth drilling-core), see Figure 139.

Each sub-sample was screened into same fractions as reported earlier and was manually separated in the same way as the raceway drilling-core. The average bulk density of the materials in the core-drilling samples as well as the bulk density of the materials in each sub-sample is calculated according to the following equation.

$$\text{Bulk density} = \frac{\text{weight of the sample}}{\text{the volume that the sample occupied in the core} - \text{drillig tube}}$$

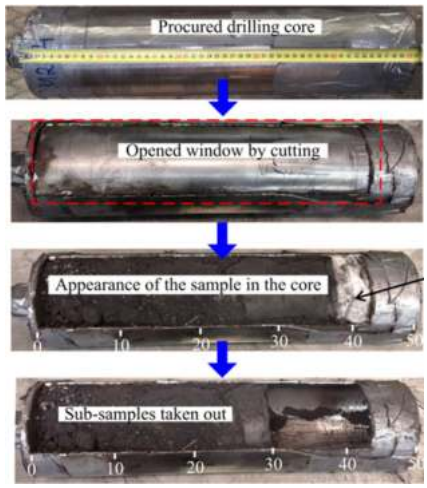



**Figure 139: Procedure of the sub-samples from the core-drilling tube**

*Chemical characterisation*

The coke samples (5-10 mm fraction) from selected positions, as shown in Figure 140, of the core-drilling tubes in the EBF® hearth were chemically analysed (C and S by LECO analysis and the other elements by XRF analysis). Further, the same coke samples were also analysed by XRD for graphitisation determination (Lc) as described in Task 2.2.

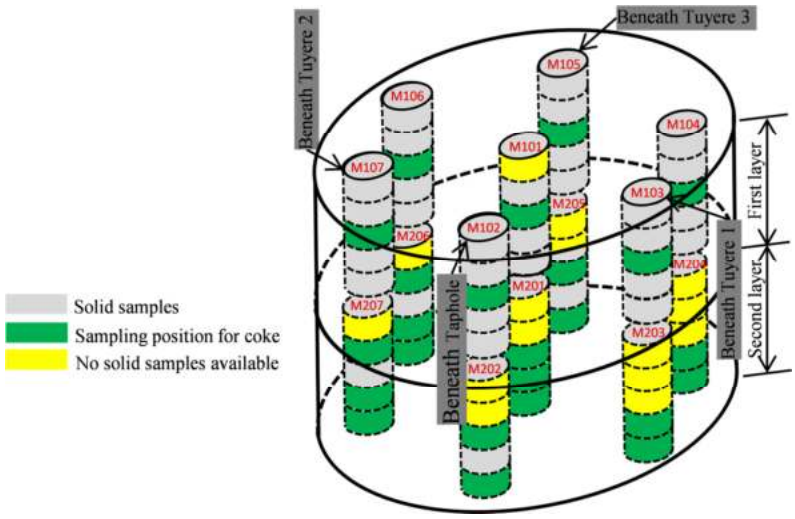



**Figure 140: Sampling positions of the coke from the core-drilling tubes in the EBF® hearth**

Figure 141 shows the particle size distribution of the materials as well as the average bulk densities of the materials in the drilling cores. Firstly, it can be seen that the materials from the first core-drilling layer is much coarser than those from the second core-drilling layer. Secondly, it can also be seen that the first core-drilling layer materials obtained from beneath the three tuyeres are denser than the materials obtained from other positions. These phenomena can be reflected by the percentages of fines found in these samples as well as by the measured bulk densities of the materials from different core-drilling tubes. These findings are in general consistent with findings from previous EBF® campaigns. Further, it has been found that in the second layer drilling cores, significant amounts of aggregates, which actually are mixture of slag, metal and coke, were also found.

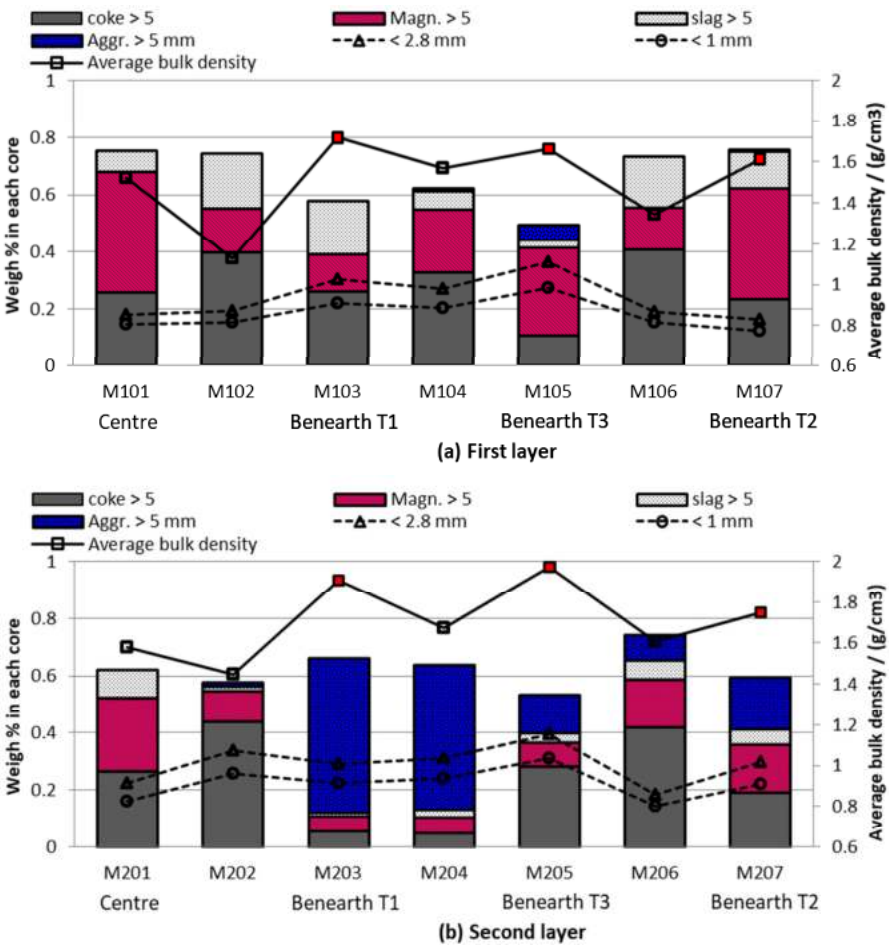



**Figure 141: Particle size distribution of (a) first layer drilling-cores and (b) second layer drilling-cores into the hearth of the EBF® as well as the bulk densities of the materials in each drilling-core**

Figure 142 shows the distribution of coarse fractions (>5 mm fractions) of coke, the magnetic, slag and aggregate in each sub-sample of first layer drilling cores beneath the three tuyeres (M103, M105 and M107, as shown in Figure 140. It is seen that aggregates are dominantly in the >16 mm fraction; slag, metal and coke can be found in all the coarse fractions.

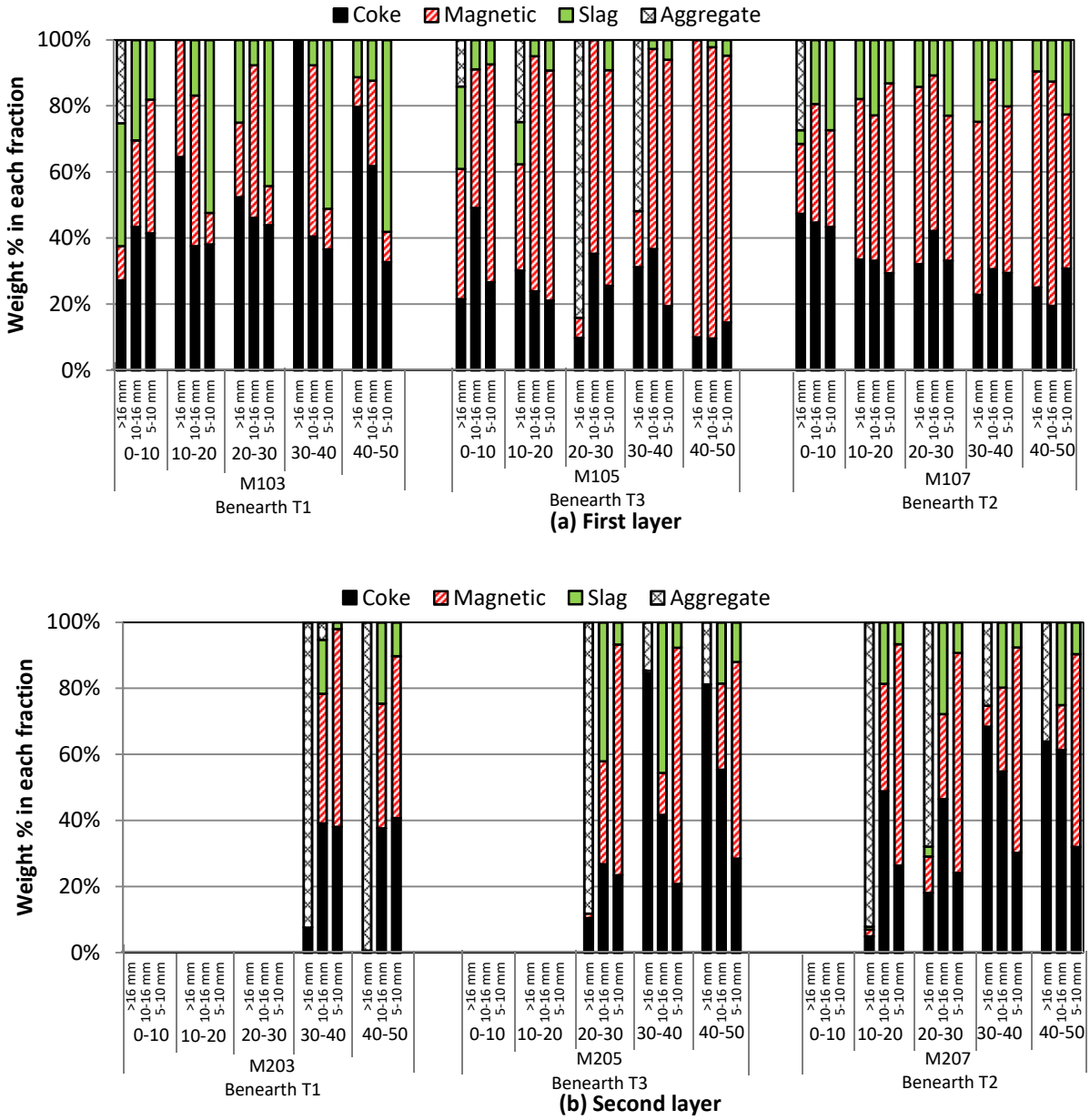



**Figure 142: Distribution of the coarse fractions (>5 mm) of coke, the magnetic, slag and aggregate in each sub-sample beneath the three tuyeres. (a) First layer; (b) Second layer**

*Chemical characterisation results and the calculated historical temperature*

Figure 143 shows the chemical composition (in the terms of total ash content and alkali content) as well as the estimated historical temperature of the selected coke samples from the EBF® hearth. In the coke the ash mainly consists of CaO, MgO, $SiO_2$ and $Al_2O_3$. It is seen that the total ash contents in most of the coke samples are higher than in the original coke (around 10%). This is due to both gasification of C in coke and penetration of slag and metal into the coke at the sites where molten slag and iron have contact with coke. In the raceway, alkali will be reduced and collected in the ascending gas. Alkali are oxidised and condensed at low temperature positions e.g. behind the birds nest, in the shaft or in the lower part of the hearth (as shown in Figure 143 (b)). From Figure 143 (a) it is seen that the ash contents of the coke beneath the tuyeres are higher than that of the coke from other positions while from Figure 143 (b) the alkali content of the coke beneath the tuyeres are in general lower than that of the coke from other positions. This is due to that the coke from beneath the tuyeres could have experienced a higher thermal history. And this could be confirmed by the estimated historical temperature of the coke shown in Figure 143 (c).

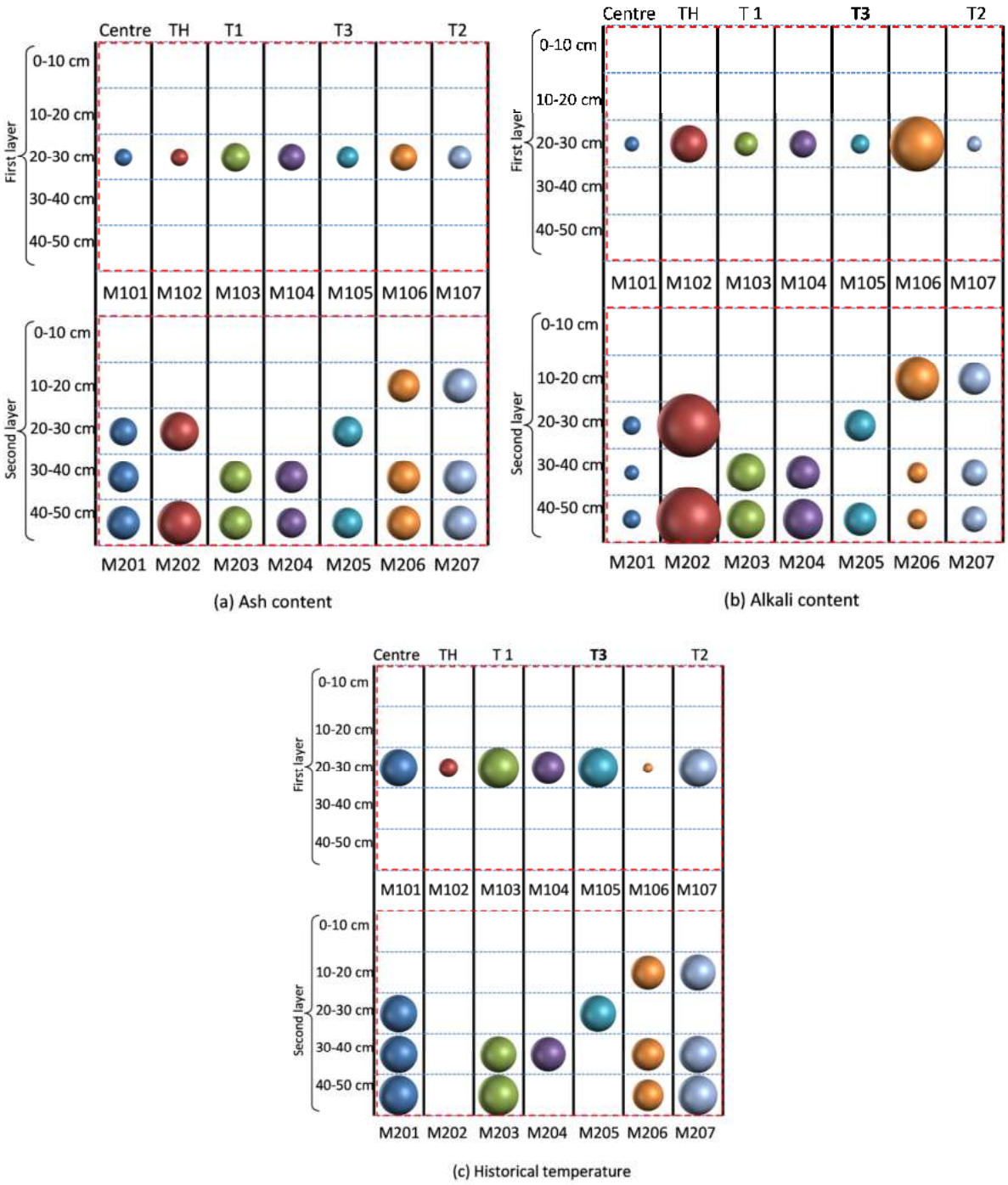



**Figure 143: (a) Ash content (wt.%), (b) alkali content (wt.) and (c) estimated historical temperature (˚C) of the coke samples from the EBF® hearth**

Conclusions

Following conclusions on standard coke and FLEXCOKE trial cokes TC1 and TC2 reacting in EBF® raceway and in excavation samples from the high temperature area could be made:

In the raceway drilling-cores, based on coke graphitisation degrees, temperature estimations and chemical compositions both coke types of reference coke and TC2 shows appearance of a bird's nest although reference drilling-core has higher amount of metal flowing in this area. XRD measurements indicate occurrence of similar phases seen in an earlier study [9] meaning that the coke has been subjected to slag and hot metal. The fines <0.5 mm shows even stronger correlation of contact with hot metal with presence of SiC. Excavation samples of TC1 in raceway showed also indications of bird's nests but the graphitisation degrees indicated lower temperatures than in the raceway drilling-cores.

The mechanical strength determined by tumbling the coke in similar way as the standard CRI/CSR tests in raceway sub-samples ($CSR_{EBF\ raceway}$) shows high values compared to coke reacting in the standard CRI/CSR test atmosphere of pure $CO_2$ gas. Both the reference coke and TC2 withstand

the conditions that occur in raceway high temperature area quite well even though the temperature is much higher than in the standard tests, on the other hand the gas composition is not as aggressive as pure $CO_2$. The coke that probably been inside the raceway has somewhat lower $CSR_{EBF\ raceway}$.

The mechanical strength of high temperature TC1 excavation samples ($CSR_{EBF}$) in shaft and in high temperature area TC1 shows a decrease in $CSR_{EBF}$ during the descent in the BF. The $CSR_{EBF}$ is around 88% in shaft sample positioned higher up in the shaft (at 3.0 m from the top of the EBF®) and around 71% further down below tuyere level (7.9 m from the top of the EBF®) where the temperature in the BF is much higher than in the shaft. CSR for the charged TC1 was on average 70.9% and it can be concluded that all excavated TC1 has a higher strength after reaction in a real BF than after the standardised CSI/CSR test.

Core-drilling samples were procured from the hearth of the EBF® and the samples were physically and chemically characterised. According to the characterisation results, following conclusions can be obtained:

- The samples procured from beneath the three tuyeres contain more fines and have higher bulk densities;
- Significant amounts of aggregates were found in the second layer drilling-cores;
- Most of the coke samples from the hearth have higher total ash contents than the charged TC1 coke;
- According to the Lc values the historical temperatures of the coke samples from the hearth were calculated and the results indicate that the coke from beneath the tuyeres could have experienced a higher thermal history. This could also be confirmed by the condensation of alkali at low temperature positions in the lower part of the EBF® hearth.

Exploitation and impact of the research results

The conclusions made of the coke behaviour in the high temperature area give valuable information, not only for the alternative coke tested but in general. The mechanical strength of high temperature coke is an important finding and proves earlier theories and studies [10] that the gas atmosphere in a real BF does not affect the CSR nearly as much as the standard test performed in 100% $CO_2$ gas atmosphere.

The large amount of samples characterised in the hearth indicates that the TC1 behaves similar to the standard coke as the results, as more fines beneath the tuyeres and has higher bulk density, were quite similar to previous EBF® campaigns. Therefore it is believed that TC1 is an option for full scale trial without negative impact on the hearth.

### *Task 3.2 Basket samples in the LKAB EBF®*

Planned activities and work accomplished

Coke samples were selected from the coke produced in pilot-scale (DMT semi-industrial 500 kg carbonisation oven) in WP1. A defined fraction of selected coke were charged as basket samples according to a charging scheme at the end of an EBF® trial campaign, stopped by nitrogen quenching. After cooling the basket samples were collected by LKAB and sent to tk SE for characterisation in WP2, using selected methods for classifying the coke behaviour relative to the BF. As there were two carbonisation campaigns in the DMT semi-industrial oven, selected samples from the first trial were charged in baskets at the end of EBF® campaign 31 and selected samples from the second trial, reference (standard coke) and test trial coke types TC1 and TC2 were charged as basket samples in EBF® campaign 32.

Description of activities, results and discussion

**Basket sample evaluation LKAB EBF®, C31**

Alternative coke produced in the first trial at DMTs 500 kg semi-industrial oven was tested in BF atmosphere as basket samples in the EBF® campaign 31. Some baskets also contained the standard coke that is normally used in the coke layers in the EBF®. A basket charging program was designed using the selected samples from the first semi-industrial trial, see Table 13 in Task 1.3, and presented in Figure 144. The baskets were charged into the EBF® at the end of the campaign

(24th of October 2014) and after the basked charging scheme was completed, the EBF® was stopped and cooled down by a flow of nitrogen. After the cooling down period, the excavation of the EBF® started and each layer was documented by LKAB. From the 10 coke types derived from the DMT tests, 7 of them were charged in baskets in the EBF® in 8 different layers. From the 48 charged baskets, 29 were recovered. In Figure 145 the EBF® is shown in where all baskets were found beneath the lower shaft probe positioned at 5.2 m from the top. To the right side the investigated baskets vertical position from the top and the time spent in the EBF® after charging are shown. Two baskets have a hold-up in the burden, indicated in red circles in the figure. The standard coke (Layer 18) has the same position of 5.8 m as sample No. 3 (Layer 16) but has been in the EBF® 16 min longer and more notably sample No. 5, which was charged in Layer 27, was found 0.3 m higher up than sample No. 7 charged at the same time in Layer 27.

**Figure 144: Charging scheme EBF® C31 (autumn 2014)**

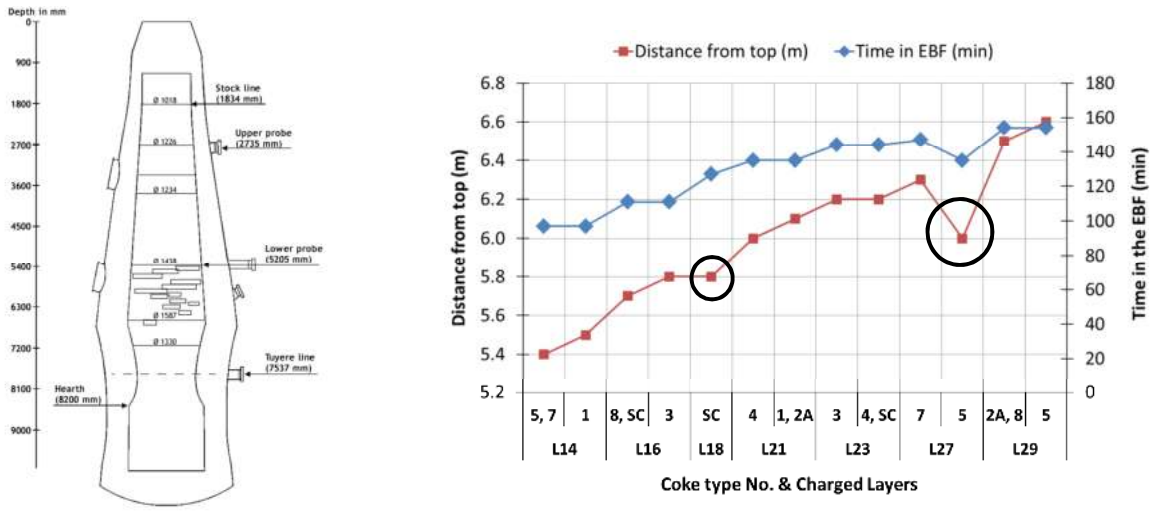



**Figure 145: Vertical position of basket samples recovered (left) and the distance from the top and time in the EBF of each sample No. and basket layer (right)**

*Ash amount and composition of EBF® basket samples - C 31*

Figure 146 shows the ash content of the standard coke compared to the coke samples that were recovered after EBF® trial. Due to gasification or up-take of oxides in the furnace the C content decreases and ash content increases.

In Table 39 the ash composition of the standard coke material and the recovered samples are listed. The ash analysis for some recovered samples (Standard L23, 2A L29 and No.5 L29) was not carried out, since not enough material for analysis was available. Comparing the results it can be concluded that $SiO_2$ and $Al_2O_3$ content was lower and MgO content higher in the ash. No trend can be observed for Fe and CaO content, as its change depends on the coke type.

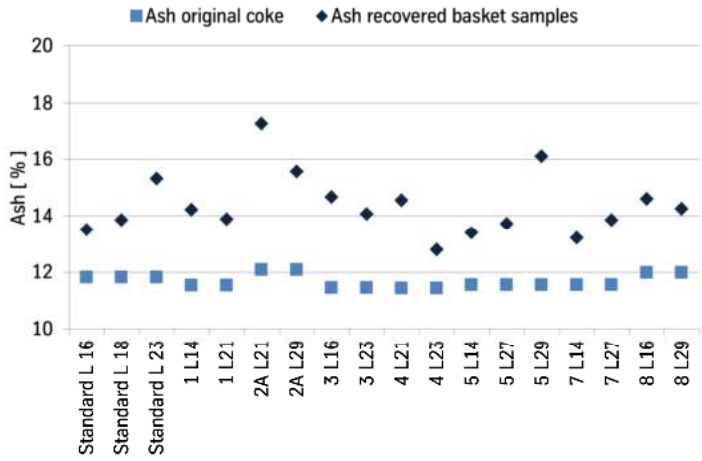



**Figure 146: Ash content in original coke and recovered basket samples, C31**

**Table 39: Ash composition (standard coke and recovered basket samples), C31**

| | Fe before EBF test | Fe after EBF test | SiO2 before EBF test | SiO2 after EBF test | Al2O3 before EBF test | Al2O3 after EBF test | MgO before EBF test | MgO after EBF test | CaO before EBF test | CaO after EBF test |
|---|---|---|---|---|---|---|---|---|---|---|
| **Standard L16** | 3.7 | 4.7 | 60.1 | 57.4 | 27.8 | 26.7 | 0.7 | 0.9 | 1.1 | 1.0 |
| **Standard L18** | 3.7 | 3.1 | 60.1 | 56.3 | 27.8 | 25.4 | 0.7 | 1.0 | 1.1 | 1.1 |
| **Standard L23** | 3.7 | - | 60.1 | - | 27.8 | - | 0.7 | - | 1.1 | - |
| **1 L14** | 3.5 | 3.3 | 60.0 | 52.9 | 28.0 | 23.6 | 0.6 | 1.0 | 1.2 | 1.3 |
| **1 L21** | 3.5 | 2.8 | 60.0 | 55.5 | 28.0 | 25.2 | 0.6 | 0.8 | 1.2 | 1.1 |
| **2A L21** | **3.3** | **9.2** | 60.5 | 48.0 | 27.8 | 21.1 | 0.8 | 0.9 | 1.5 | 1.4 |
| **2A L29** | 3.3 | - | 60.5 | - | 27.8 | - | 0.8 | - | 1.5 | - |
| **3 L16** | **3.2** | **7.1** | 60.2 | 52.1 | 27.9 | 23.9 | 0.6 | 1.1 | **1.2** | **2.4** |
| **3 L23** | 3.2 | 2.2 | 60.2 | 55.0 | 27.9 | 25.3 | 0.6 | 0.6 | 1.2 | 0.8 |
| **4 L21** | 3.3 | 3.4 | 60.2 | 51.6 | 28.1 | 23.1 | 0.7 | 0.9 | 1.5 | 1.5 |
| **4 L23** | 3.3 | 4.4 | 60.2 | 57.2 | 28.1 | 26.4 | 0.7 | 1.1 | 1.5 | 1.8 |
| **5 L14** | 3.2 | 2.6 | 60.2 | 58.0 | 28.2 | 26.5 | 0.8 | 0.9 | 1.6 | 1.4 |
| **5 L27** | 3.3 | 3.5 | 60.2 | 54.3 | 28.2 | 25.0 | 0.8 | 0.7 | 1.6 | 1.4 |
| **5 L29** | 3.3 | - | 60.2 | - | 28.2 | - | 0.8 | - | 1.6 | - |
| **7 L14** | 3.4 | 3.4 | 59.7 | 58.0 | 27.5 | 25.9 | 0.8 | 1.1 | 1.4 | 1.5 |
| **7 L27** | 3.4 | 3.4 | 59.7 | 57.7 | 27.5 | 26.3 | 0.8 | 1.2 | 1.4 | 1.6 |
| **8 L16** | 4.1 | 3.2 | 59.5 | 52.3 | 27.4 | 23.4 | 0.8 | 1.3 | 1.4 | 1.6 |
| **8 L29** | 4.1 | 4.7 | 59.5 | 54.5 | 27.4 | 24.8 | 0.8 | 1.0 | 1.4 | 1.4 |

Figure 147 and Figure 148 show the significant increase of the alkalis content ($K_2O$ and $Na_2O$) in the ash of the recovered EBF® samples. This increase can be explained by the alkali recirculation phenomena.

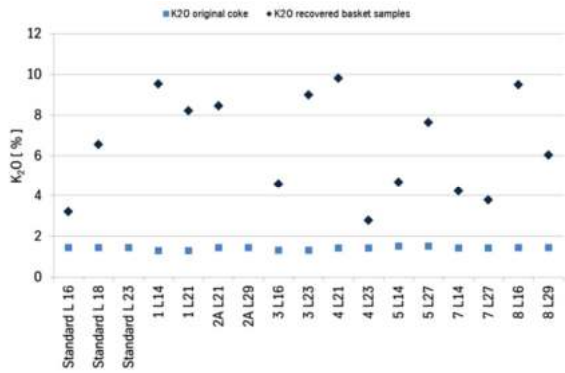



**Figure 147: $K_2O$ content (original coke and recovered basket samples)**

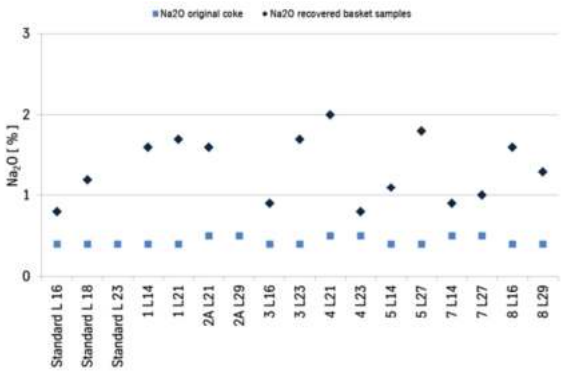



**Figure 148: $Na_2O$ content (original coke material and recovered basket samples**

It could be expected a decrease in C content and therefore increase in ash content for the basket samples that were recovered further down in the furnace due to longer residence time, higher exposed temperature and therefore stronger gasification. However this cannot be confirmed for all

blends. From experience in previous EBF® campaigns and from the excavation results reported in Task 4.2, indicates that coke in basket samples in general is is not that affected by gasification in the EBF shaft and that the lowering of C content is most likely due to up-take of oxides, mainly alkali when descending in the shaft.

*Graphitisation results*

Coke in basket samples have been evaluated and compared with graphitisation values (Lc) of original, charged coke. The standard coke normally used at the EBF® and the base blend and six alternative cokes from the first semi-industrial trial were tested, analysed and evaluated as described in Task 2.2. Figure 149 shows the Lc of charged coke in comparison of recovered basket samples.

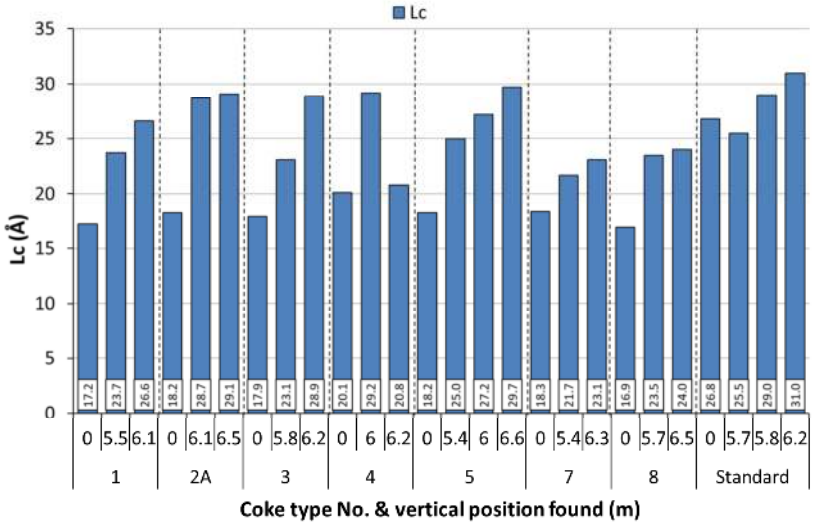



**Figure 149: Graphitisation degree, Lc (Å). Lc of charged coke and vertical position found, EBF® C31**

The graphitisation degree has increased for all EBF® samples compared to each charged coke type. Initial values are different for each coke type and the standard coke has the highest Lc value of all coke types (~27 Å). The Lc values of base blend (No. 1) from the first semi-industrial trial is ~17 Å as well as in No. 8 (3% addition of anthracite and 3% thermal coal). Samples with 5% addition of thermal coal (2A) and anthracite (No.3) as well as samples No. 5 and No. 7, addition of 3% pet coke and 3%charcoal and 3% addition of thermal coal and 3%pet coke, respectively, showed just slightly higher Lc of ~18 Å. The samples with 3% charcoal had higher graphitisation of ~20 Å (No. 4). However, the small differences may be variations in the base blend mix and that relative amount of added alternative C-containing material contributes less to the graphitisation.

Comparing coke types from the first semi-industrial trial, the base blend (No.1) has similar increase in Lc in samples from lower levels, highest Lc is found in No.5; No.3 and No. 2A. Here, not only the vertical position must be considered, also the horizontal position were the baskets are found contributes as local temperature conditions can influence the graphitisation.

**Basket sample evaluation LKAB EBF®, C32**

At the end of EBF® campaign 32, operating with TC1, baskets were charged according to the charging scheme in Figure 150.

Due to low sample amount, some baskets were divided into two parts with a mesh in-between. Basket samples of TC1 and TC2 from SSAB 1st industrial trial were charged as baskets together with standard coke (SC)/reference coke and some blends in where the biomass (torrefied wood 2% and 3% addition and 3% addition of charcoal to the blend) were used as alternative C-containing material in the second semi-industrial trial at DMT.

34 coke samples were charged in the EBF® in 10 different layers. From the charged baskets, 26 were recovered and 7 were sent to tk SE for coke texture measurement in Task 2.2. The chemical analyses were made by SSAB and MEFOS investigated the coke graphitisation degrees.

*TW2%=coke with 2% torrefied wood addition, TW3%=coke with 3% torrefied wood addition and Charcoal=coke with 3% charcoal addition. SC= standard coke (reference)

**Figure 150: Charging scheme EBF® C 32 (autumn 2016)**

Figure 151 shows the distance from the top of the EBF® (see Figure 145) where the basket samples were found during excavation and an example of a basket row containing coke samples with biomass addition (L23) is shown in Figure 152.

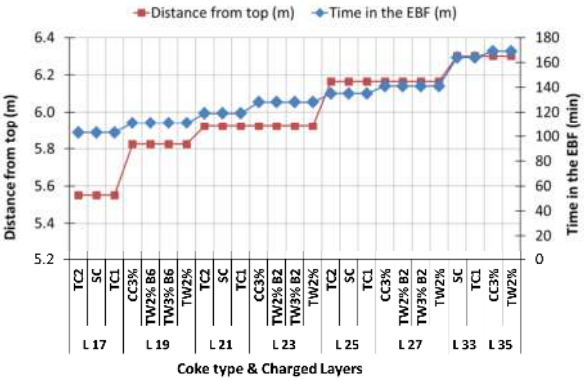



**Figure 151: Distance from the top and time in the EBF of coke type and basket layer. CC=charcoal, TW=torrefied wood, SC=standard coke**

**Figure 152: Example of an excavated basket row, here L23**

*Ash amount and composition of EBF® basket samples - C 32*

Figure 153 shows the ash content of the standard coke, trial coke TC1 and TC2 and also bio-coke from the second semi-industrial trial. Generally the ash content increases in samples found further down in the EBF®. The increase of the ash contents tends not be as high in samples with 2% and 3% TW additions when descending in the EBF®. For the bio-coke with 3% charcoal addition the two samples found at 6.2 and 6.3 m from the top has high ash values. The high value of ash in Level 27 was mainly due to a high amount of MnO (~40%), not shown here. Important to note is that the differences may also be due to different local BF conditions and horizontal position and not the behaviour of specific coke type.

In Table 40 the ash composition of the standard coke and the recovered samples in C32 are listed. In basket investigation of C31 it was seen that the MgO content decreases in the EBF® and no trend could be noted for Fe and CaO. In C32 both CaO and MgO is higher after test in the EBF® and that the Fe was in principle un-changed, even though some samples are increased in the upper part of levels investigated. In baskets at lower levels a clear trend can be observed as from Layer 27 (6.2 m) and downwards the Fe, CaO and MgO are lower as well as the $SiO_2/Al_2O_3$ ratio. This may indicate reduction of CaO, MgO and $SiO_2$ in coke ash due to higher temperatures at these levels.

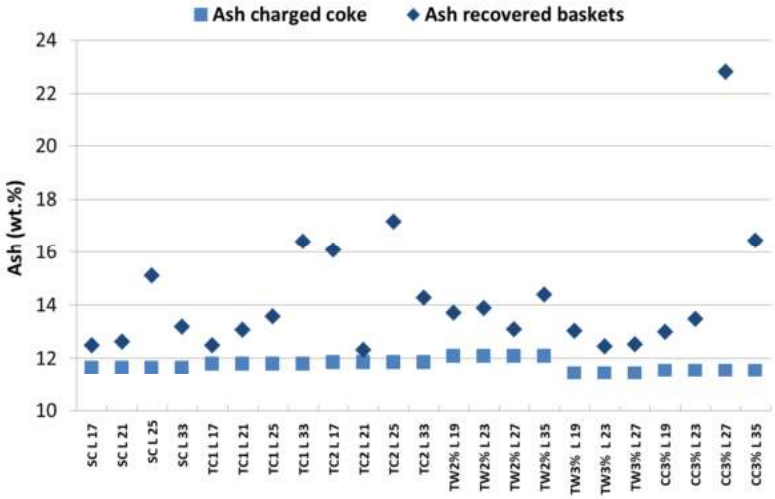



**Figure 153: Ash content in original coke and recovered basket samples, C32**

**Table 40: Ash composition (standard coke and recovered basket samples), C32**

| | Fe before EBF test | Fe after EBF test | $SiO_2$ before EBF test | $SiO_2$ after EBF test | $Al_2O_3$ before EBF test | $Al_2O_3$ after EBF test | $SiO_2/Al_2O_3$ before EBF test | $SiO_2/Al_2O_3$ after EBF test | MgO before EBF test | MgO after EBF test | CaO before EBF test | CaO after EBF test |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| SC L 17 | 3.50 | 3.50 | 58.8 | 57.3 | 28.3 | 28.4 | 2.08 | 2.02 | 0.64 | 0.96 | 0.69 | 0.73 |
| SC L 21 | 3.50 | 3.50 | 58.8 | 57.1 | 28.3 | 25.6 | 2.08 | 2.23 | 0.64 | 1.37 | 0.69 | 1.41 |
| SC L 25 | 3.50 | 3.30 | 58.8 | 50.8 | 28.3 | 23.5 | 2.08 | 2.16 | 0.64 | 3.41 | 0.69 | 1.66 |
| SC L 33 | 3.50 | 3.10 | 58.8 | 47.5 | 28.3 | 22.3 | 2.08 | 2.13 | 0.64 | 0.86 | 0.69 | 0.33 |
| TC1 L 17 | 3.50 | 4.10 | 59.0 | 57.9 | 26.4 | 25.6 | 2.23 | 2.26 | 0.67 | 0.91 | 1.08 | 2.09 |
| TC1 L 21 | 3.76 | 3.60 | 59.0 | 56.6 | 26.4 | 25.7 | 2.23 | 2.20 | 0.91 | 1.38 | 1.08 | 2.04 |
| TC1 L 25 | 4.12 | 4.20 | 59.0 | 53.2 | 26.4 | 23.7 | 2.23 | 2.25 | 1.38 | 1.03 | 1.08 | 1.22 |
| TC1 L 33 | 3.57 | 2.50 | 59.0 | 47.8 | 26.4 | 22.5 | 2.23 | 2.13 | 1.03 | 2.22 | 1.08 | 1.61 |
| TC2 L 17 | 3.64 | 3.09 | 59.5 | 47.9 | 26.9 | 21.0 | 2.21 | 2.28 | 0.65 | 2.34 | 0.98 | 1.36 |
| TC2 L 21 | 3.64 | 4.37 | 59.5 | 55.1 | 26.9 | 24.9 | 2.21 | 2.22 | 0.65 | 1.64 | 0.98 | 2.81 |
| TC2 L 25 | 3.64 | 7.48 | 59.5 | 40.1 | 26.9 | 18.3 | 2.21 | 2.19 | 0.65 | 1.63 | 0.98 | 1.86 |
| TC2 L 33 | 3.64 | 1.72 | 59.5 | 46.2 | 26.9 | 21.4 | 2.21 | 2.16 | 0.65 | 0.99 | 0.98 | 0.56 |
| TW2% L 19 | **2.98** | 1.95 | **59.2** | 50.8 | **29.7** | 23.9 | 1.99 | 2.12 | **0.48** | 0.93 | **1.10** | 0.30 |
| TW2% L 23 | **2.98** | 3.95 | **59.2** | 54.3 | **29.7** | 25.4 | 1.99 | 2.14 | **0.48** | 2.10 | **1.10** | 2.82 |
| TW2% L 27 | **2.98** | 1.97 | **59.2** | 53.4 | **29.7** | 25.3 | 1.99 | 2.11 | **0.48** | 0.89 | **1.10** | 0.44 |
| TW2% L 35 | **2.98** | 1.54 | **59.2** | 46.7 | **29.7** | 22.6 | 1.99 | 2.06 | **0.48** | 0.91 | **1.10** | 0.23 |
| TW3% L 19 | **3.11** | 4.22 | **59.1** | 54.2 | **29.9** | 26.6 | 1.98 | 2.04 | **0.47** | 1.13 | **1.17** | 0.52 |
| TW3% L 23 | **3.11** | 2.57 | **59.1** | 58.6 | **29.9** | 26.3 | 1.98 | 2.23 | **0.47** | 1.09 | **1.17** | 1.02 |
| TW3% L 27 | **3.11** | 1.79 | **59.1** | 50.2 | **29.9** | 23.3 | 1.98 | 2.15 | **0.47** | 0.78 | **1.17** | 0.32 |
| CC3% L 19 | **3.93** | 3.59 | **57.9** | 57.2 | **29.1** | 26.7 | 1.99 | 2.14 | **0.58** | 1.46 | **1.55** | 1.06 |
| CC3% L 23 | **3.93** | 2.81 | **57.9** | 56.0 | **29.1** | 25.9 | 1.99 | 2.16 | **0.58** | 1.70 | **1.55** | 2.32 |
| CC3% L 27 | **3.93** | 0.95 | **57.9** | 33.7 | **29.1** | 15.6 | 1.99 | 2.16 | **0.58** | 0.57 | **1.55** | 0.31 |
| CC3% L 35 | **3.93** | 0.97 | **57.9** | 41.3 | **29.1** | 20.0 | 1.99 | 2.07 | **0.58** | 0.96 | **1.55** | 0.16 |

**Bold characters**-analyse made by tk SE. SC=standard coke, TC1=Test Coke 1, TC2=Test Coke 2, TW=torrefied wood, CC=charcoal

Figure 154 and Figure 155 show the significant increase of the alkali content ($K_2O$ and $Na_2O$) in the ash of the recovered EBF® samples. As in C31 this increase are explained by the alkali recirculation phenomena. In is noted that the amounts of alkali in coke ash are much higher in the recovered baskets in C32 than in C31. This can partly be explained by different laboratories making the analysis, SSAB (C32) and tk SE (C31). Also the alkali recirculation and $K_2O$-yiedls may be different at the end of each campaign.

Comparing the standard coke with TC1 and TC2 in baskets from L33 (6.3 m from EBF® top), TC1 has lower up-take of alkali than both standard coke and TC2 where TC2 has the highest up-take. The TC1 and SC were placed in the same basket and the local BF conditions should be similar for

these two. The TC2 basket was found a bit further apart and the higher alkali content may be due to the fact that this basket was found closer the wall in the EBF where the oxide and alkali content generally is higher. Bio-coke has a moderate up-take of alkali in lower levels, i.e. down to around six m, especially with 3% addition (TW and charcoal) but showed a sharp elevation at lower levels as for 3% charcoal addition in L35, at 6.3 m from the top.

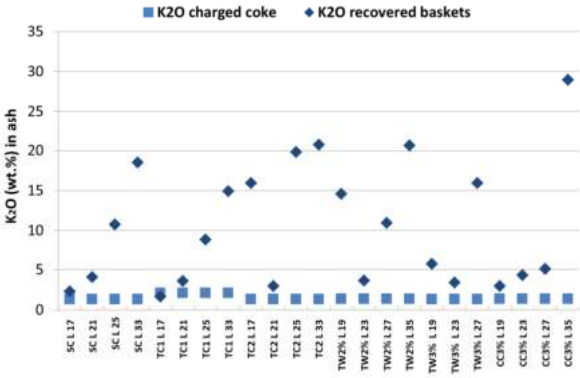



**Figure 154: $K_2O$ content (original coke and recovered basket samples)**

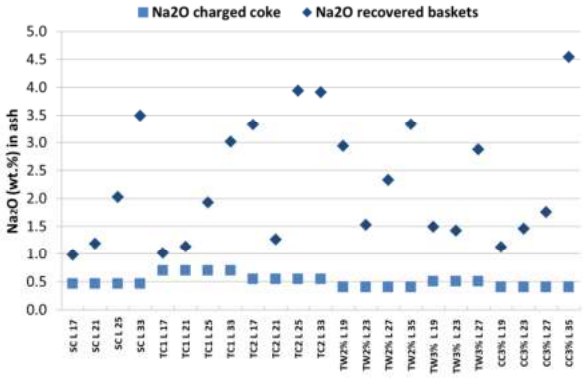



**Figure 155: $Na_2O$ content (original coke material and recovered basket samples**

*Graphitisation results*

Coke in basket samples have been evaluated and compared with graphitisation values (Lc) of original, charged coke of each type. The standard coke and the FLEXCOKE trial coke TC1 and TC2 and alternative cokes contain biomaterial from the second semi-industrial trial were tested, analysed and evaluated as described in Task 2.2.

Figure 156 shows the Lc of charged standard coke in comparison of recovered TC1 and TC2 basket samples. The graphitisation degree seems to be lower than the charged coke in many samples. Similar trend was seen in bio-coke samples, see Figure 157. However, the differences are quite small and the results point at that the temperature in the EBF® shaft down to a level of 6.3 m from the top had not exceed the coking temperatures of the test cokes, i.e. around 1000 ˚C, in C32.

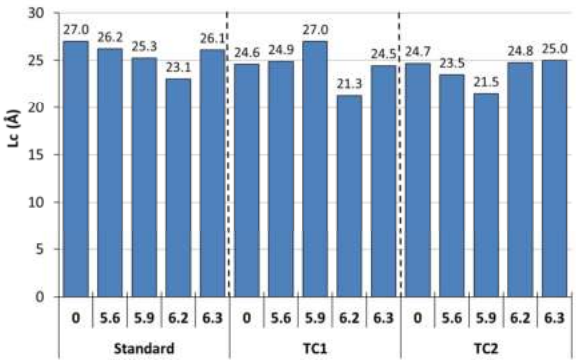



**Figure 156: Lc in standard coke, TC1 and TC2 basket samples**

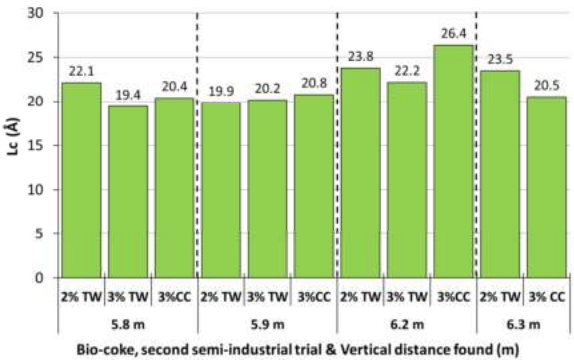



**Figure 157: Lc for biomass addition to the coke blend, second semi-industrial trial basket samples**

Conclusions

Basket sample evaluations of reference and alternative coke tested in the EBF® campaigns 31 and 32 showed that in general the ash content in general is higher in samples found further down in the EBF® and that the increase probably is due to up-take of oxides. In both C31 and C32 a significant increase of the alkali content ($K_2O$ and $Na_2O$) in the ash of the recovered EBF® samples are seen due to the alkali recirculation phenomena.

TC1 has lower up-take of alkali than both standard coke and TC2 at 6.3 m in the EBF®. Bio-coke has a moderate up-take of alkali in lower levels, i.e. down to around 6 m, but showed a sharp elevation of alkali contents in the coke ash at 6.3 m from the top. However, this may be linked to the in-furnace conditions more than to the coke type.

In C31, operating with a centre gas flow in EBF®, the graphitisation degrees (Lc) were enhanced in the recovered basket samples compared to the charged coke types. The standard coke has the highest Lc and the increase was also highest looking at lower levels in the EBF®. Comparing coke

types from the first semi-industrial trial, the base blend (No.1) has in general similar increase in Lc in samples from lower levels as coke type's No. 7 and No. 8, highest Lc was found in No.5; No.3 and No. 2A.

In contrast to C31 the coke graphitisation degrees in C32, operating with a flat gas profile, points at that the temperature in the EBF® shaft down to a level of 6.3 m had not exceed the coking temperatures of the test cokes, i.e. around 1000 °C.

Exploitation and impact of the research results

The evaluation of basket samples in the EBF® is a valuable indicator of different raw materials behaviour in actual BF atmosphere, even though sometimes difficult to point out of the changes are due to operational conditions, vertical and horizontal position, effects of sintered material, gas channels or the actual material itself. In C32, the standard coke was placed in the same basket row as FLEXCOKE trial coke TC1 and TC2 in an attempt to rule out some of the difficulties mentioned. Therefore it seems like the TC2 takes up more alkali in the BF alkali re-circulation than TC1, which has even lower up-take than the standard coke.

## WP4: Operational tests in coke plant and in BF

### *Task 4.1 Industrial-scale cokemaking*

Planned activities and work accomplished

Production of coke for operational trials in the EBF® and for full scale BF testing involves both a careful consideration of coking properties to prevent damage at the coke plant and a systematic logistic arrangement of a specific coal blend. SSAB in Luleå has a unique flexible coal preparation and handling layout at their coke plant. The material handling includes 12 separate bins for different coals that can be precisely added to the mix. This makes it possible, although requiring close control in order to keep separate qualities apart, to prepare a specific blend with selected alternative coal or C-containing material in order to produce test coke at a few coke ovens followed by separate quenching and handling. Therefore the needed amount of alternative coke for testing in the LKAB Experimental BF can be made. The produced alternative coke combination with BF trials offers a possibility to link the test properties of coal/C-containing materials with the BF behaviour of produced coke.

The test cokes for the EBF® were produced in five coke ovens, with a mix of original and test coal blend in adjacent ovens, and handled separately during quenching and needed specific logistics and control during production. To meet the requirements for coke in EBF® around 500 tonnes of coke were produced. Coke on fraction of 15-30 mm was prepared by crushing and screening according to a standard procedure used by LKAB and losses during preparation was considered.

Production of coke for industrial scale trials in the BF had to be carried out with alternative coal blend that do not risk the restrictions at coke plant. The results from DMT semi-industrial testing were considered and the quality of coke produced had to meet the specifications for BF operators. The operational trials were evaluated and compared with results when using the standard coal blend. Evaluation included process data from coking plant, coke quality and the effects on off-gas and its treatment in the by-product and gas cleaning facilities.

Description of activities, results and discussion

**SSAB 1st cokemaking trial producing alternative coke for EBF® operational trials in Task 4.2**

It was decided in the project group that two coke types should be made for EBF® trials. One option discussed was using charcoal addition but shortage of production amounts needed, but mostly for safety reason during crushing with risk for dust explosion, the only realistic coking coal substitutes were thermal coal and pet coke. As 10% addition of thermal coal in the first semi-industrial trial showed high wall load during carbonisation and worse cold mechanical strength and CRI/CSR the better option was to choose the lower amount of 5% thermal coal. The main reason was however the wall load as SSAB coke ovens are sensitive to high wall pressures. The project group decided on the following test production:

1. 400 tonnes of coke with 5% thermal coal addition
2. 400 tonnes of coke with 3% thermal coal and 3% pet coke addition

The mixes had to be slightly adjusted compared to those at DMT, for production safety and coking purposes. Table 41 shows the mix properties in comparison with DMT mixes coke No. 2a and No. 7. These changes should not have any significant effect on the resulting coke but was important to assure the coking process together with the normal operation of the battery.

**Table 41: The small changes of the mixes to assure production in the coke plant**

| Mix name: | LV part | MV part | HV part | Non-coking part |
|---|---|---|---|---|
| **Mix 2a DMT** | 40% | 30% | 25% | 5% thermal coal |
| **Mix 1 EBF® trial coke** | 43% | 32% | 20% | 5% thermal coal |
| **Mix 7 DMT** | 40% | 29% | 25% | 3% thermal coal + 3% pet coke |
| **Mix 2 EBF® trial coke** | 39% | 33% | 22% | 3% thermal coal + 3% pet coke |

Five ovens (out of 54) were charged with mix 1 three times (a total of 15 ovens) and then the same five ovens were charged with mix 2 three times (15 ovens). Bunkers were cleaned before crushing and filling with pet coke and thermal coal. All conveyer belts were emptied from "ordinary mix" before loading the test mixes, and the test mixes were kept in an own coal tower bunker. A separate charging car was used for the test mix.

All these precautions made it possible to keep separate the test mix and ordinary mix in such a way that there is no doubt that the correct mix ended up in the test ovens.

The test ovens were carefully monitored and no deviations at all occurred during the campaign. Everything ran smoothly and the produced coke was quenched and emptied at a separate wharf where a tractor collected each coke oven, see Figure 158, before transported to storage under roof.

**Figure 158: Emptying the alternative coke from a separate wharf after quenching**

Before the crushing of the thermal coal the parameters of the crusher were set according to the reported hardness, measured as Hardgrove Grindability Index (HGI). A sample of the crushed coal was dried and sieved to compare its fineness to the rest of the mix. It was seen that the thermal coal was a bit coarser than the normal mix curve, see Figure 159. The mixes as a whole though, were quite close to the normal curve but particle size distribution of the thermal coal differs significantly from the one of the blend.

The produced two alternative coke types, carbonised from Mix 1 and Mix 2, were named Test Coke 1 (TC1) and Test Coke 2 (TC2).

TC1 and TC2 physical characterisation data as particle size distribution and cold mechanical strengths are reported in Task 2.1. Microstructure (Iso- and anisotropy analyses, graphitisation etc.) analyses are presented in Task 2.2 and chemical analyses in Task 2.3. Results from metallurgical testing as for example CSR/CRI, both in standard and innovative tests are reported in Task 2.4.

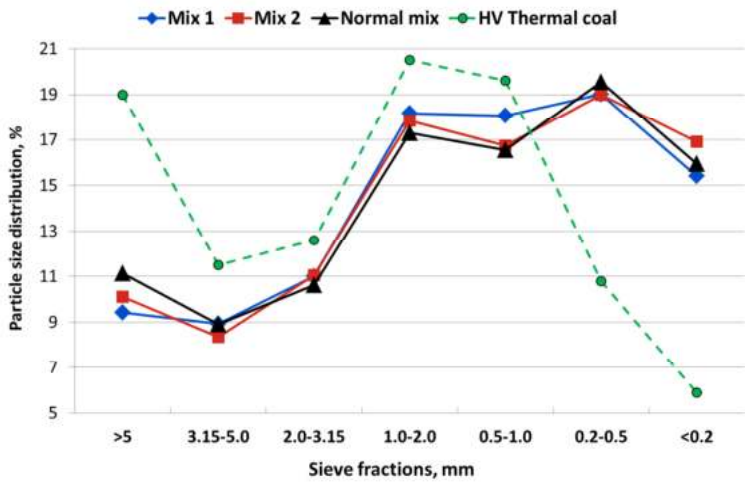



**Figure 159: Sieving curves of coal, showing normal mix, mix 1, mix 2 and thermal coal**

**SSAB 2nd cokemaking trial producing coke for full-scale industrial trials at SSAB BF No. 3 in Luleå (Task 4.3)**

Since no harmful effects seen in the EBF® trials it was decided within the project group to go forward with TC1 (Coke 1), i.e. 5% addition of thermal coal to the blend, during the full scale operational trial. The same type of thermal coal which was used in the EBF trial could not be purchased for the large scale industrial trial but one with properties expected to behave quite similar could be found. Differences were that the thermal coal used in the 2nd cokemaking trial had a bit higher ash, $SiO_2$, $Al_2O_3$ and alkali content than TC1 produced and tested in the EBF trial which had a bit higher contents of VM, S, CaO, MgO and $P_2O_5$. Therefore the produced coke from the full scale industrial trial is named FLEXCOKE Trial Coke (FTC).

It was also decided to produce the alternative coke at the SSAB coke plant in Luleå at the same time the coke is charged into SSAB BF No. 3 in Luleå (Task 4.3). The number of ovens filled with alternative C-containing material during the FLEXCOKE trial (i.e. 5% thermal coal to the coal blend) were successive increased so that a mixture of FTC and standard coke produced and sent the BF were; 20%, 40%, 80% and 100% i.e. until all the ovens in the battery contained the FLEXCOKE trial mix. The distribution of coals in the mixes during the trial is shown in Table 42.

Compared to the 1st cokemaking trial, see Table 41, the FLEXCOKE trial mix had higher amounts of MV coal in the mix and more of the HV coal was replaced by thermal coal.

**Table 42: Distribution of coals in the mixes during the FLEXCOKE trial**

| Name: | LV part | MV part | HV part | Non-coking part |
|---|---|---|---|---|
| **FLEXCOKE trial mixe** | 42% | 36% | 17% | 5% thermal coal |
| **Standard (Reference) mix** | 42% | 36% | 22% | |

The full scale trial at SSAB started the 9th of May and ended the 24th of June 2017 and was successful and no harmful effects on the battery could be seen. The operators monitoring the ovens and the dust at pushing and could not note any increase.

A data set was collected and evaluated with a multivariable data analysis done in SIMCA. An orthogonal partial least square with discriminant data analyses (OPLS-DA) was created in order to compare the drift data collected, during 100% test coke period and a reference period. If differences are found between two data sets, the model will indicate which parameters or variables that causes the differences. The data set was divided into two sets, see (A), where DA 1 is with 100% alternative FLEXCOKE trial blend in the battery and DA 2 is the reference period.

The plot (A) in Figure 160 is a coefficients overview plot; it illustrates the variables that affect the two data sets. The blocks for the variable Flue temp, that is the temperature measured in the walls of the ovens, is a systematic difference in the two data sets (the bars are in the opposite direction of the other parameters in each data set). Plot (B) is a VIP plot, shows which variables that contributed most to the variations from the average in the data set. According to the plot, the variable with major importance was the charging weight.

From a process point of view, the charging weight varied most during test period and the reference period. The charging weight can as for example affect the pushing pressure. The pushing pressure can in turn also be influenced by the swelling of the coal during cooking, which can in turn be influenced by other parameters such as coal moisture, particle size distribution and coking time.

The Flue temp was significant difference in-between the two periods, the differences found between the periods could in the model be caused by decreased Flue temperature measured during 9th of June to 14th June, see Figure 163. Any of the other parameters measured seemed to affect or deviate in the model made in SIMCA. The important process parameters are explained in Table 43.

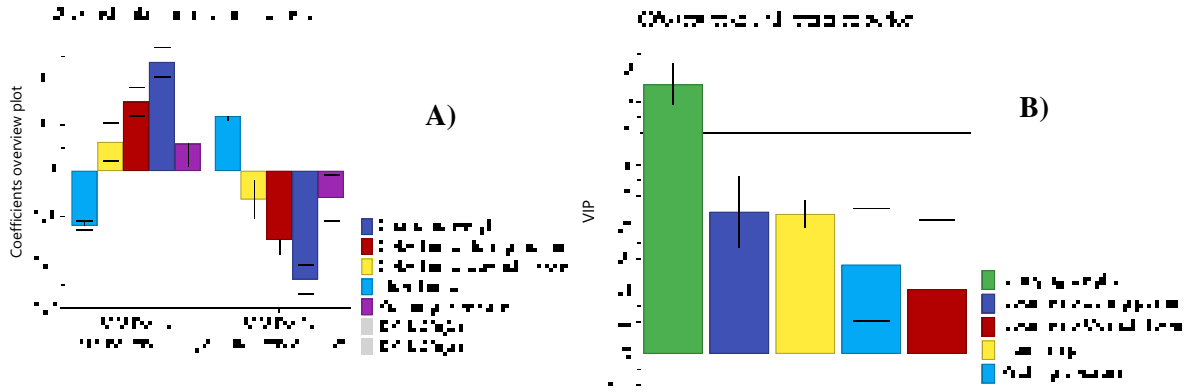



**Figure 160: Some results from SIMCA analysis made**

**Table 43: Important parameters found in SIMCA modelling**

| Process parameter | |
|---|---|
| Coke temp during pushing (°C) | Measure temperature (average) in the coke when pushed to the shield in the coke guiding car |
| Charging weight (kg) | Weight of coal blend when coke oven is filled |
| Flue Temp. (°C) | Temperature measurements at the walls of coke oven, manual measurements done once per shift |
| Pushing Pressure (Ampere) | Pressure when the coke is pushed out of the oven by the pushing machine |
| Coke temp Quench Tower (°C) | The temperature of the coke before quenching |

The process parameters pointed out in SIMCA modelling were investigated and the charging weight is plotted in Figure 161. The charging weight seems to be decreasing changing to the standard mix (Ref) but the variations are considered to be within the normal range and no significant differences could be seen. The same considerations were made for the pushing force in Figure 162 and for flue temperature and coke pushing temperature in Figure 163 and in Figure 164, respectively.

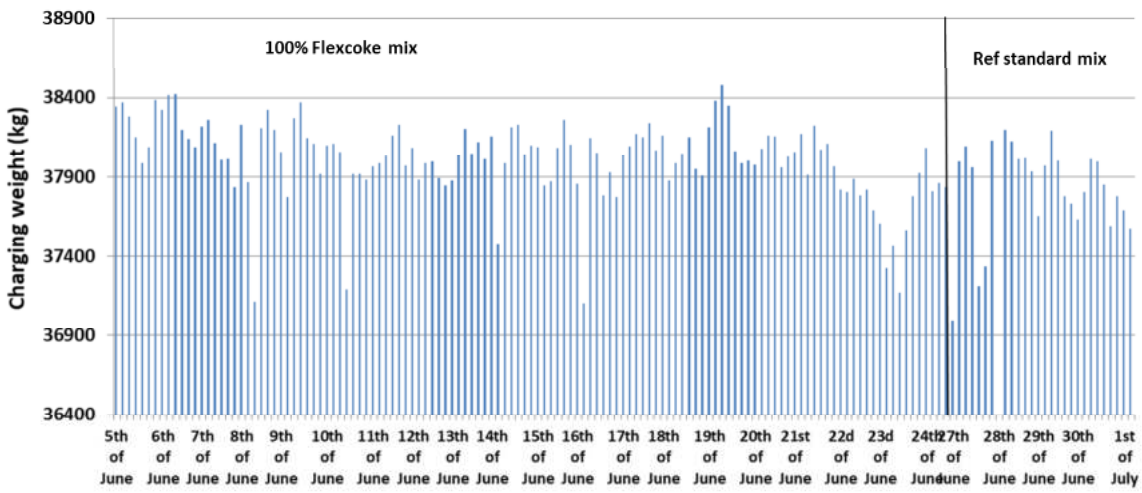



**Figure 161: Charging weight average values of each series of 11 ovens during the FLEXCOKE trial (100% battery) and reference period with standard mix at the end**

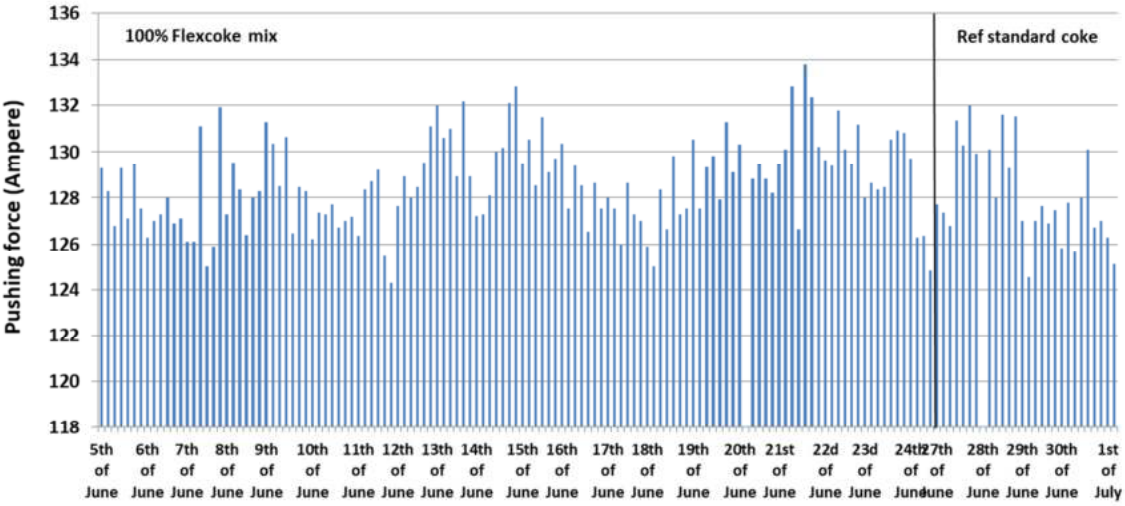



**Figure 162: Pushing force average values of each series of 11 ovens during the FLEXCOKE trial (100% battery) and reference period with standard mix at the end**

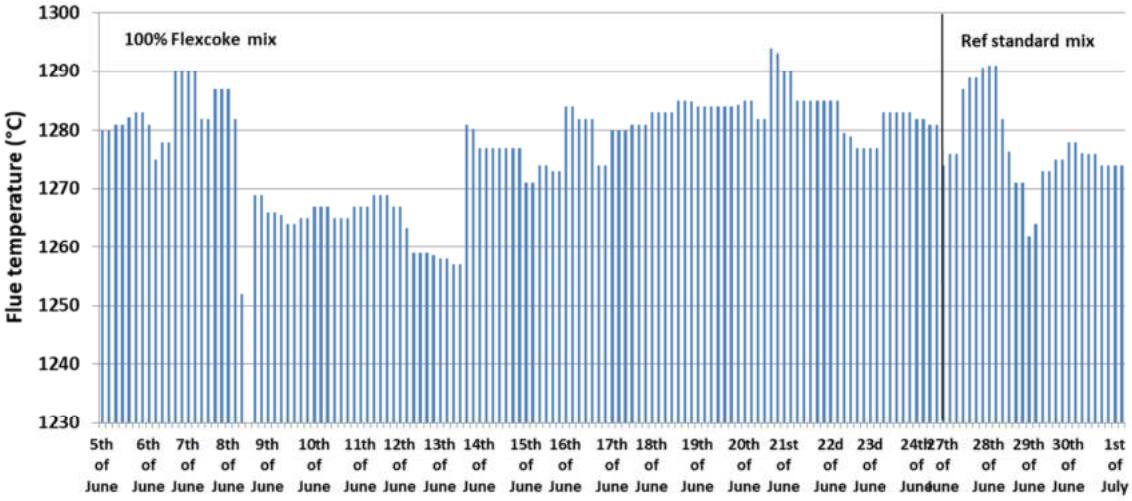



**Figure 163: Flue temperature average values of each series of 11 ovens during the FLEXCOKE trial (100% battery) and reference period with standard mix at the end**

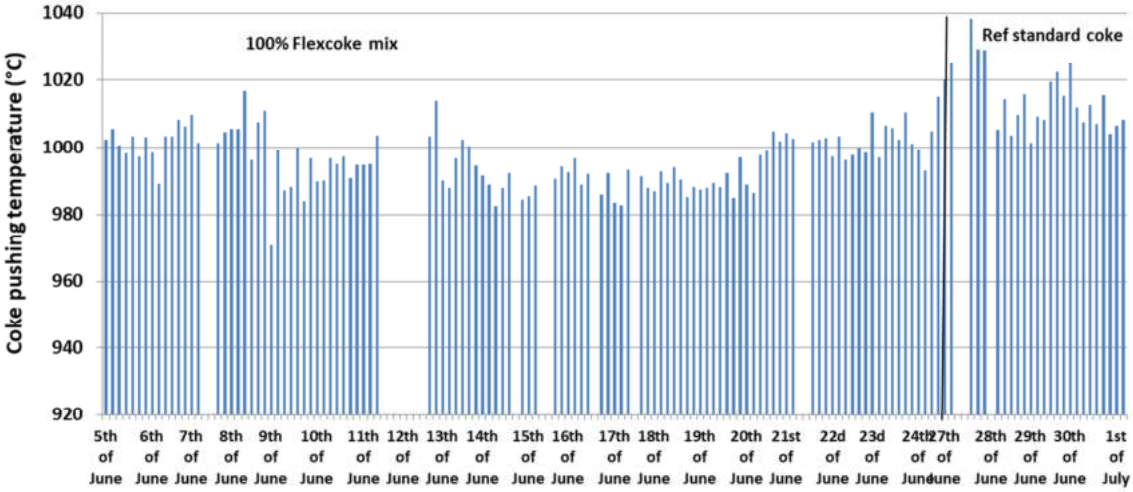



**Figure 164: Coke pushing temperature average values of each series of 11 ovens during the FLEXCOKE trial (100% battery) and reference period with standard mix at the end**

The effects on off-gas and its treatment in the by-product and gas cleaning plant were evaluated by Energy Thechnology Center, RISE, ordered by SSAB. The evaluation was done in order to investigate the gas composition when the FLEXCOKE test mix was added to the coal blend. Gas samples were analysed with gas chromatograph composed of three detectors, a thermal

conductivity detector for analysis of $H_2$, $O_2$, $N_2$, CO, $CH_4$ and $CO_2$ ethylene, ethane and acetylene. Also, a flame ionization detector (FID) was used for detection of other hydrocarbons and a pulsed flame photometric detector (PFPD) was used for detection of sulphur components.

Normally, components such as benzene, toluene and Xylene (BTX) are removed from the gas in the gas cleaning plant. During the test trial the benzene plant was not in operation which can change the gas composition. Figure 165 shows some of the components in the analysed in the gas, during normal operation in the gas cleaning plant, during test trial and a reference period with the benzene plant out of operation. According to the results showed in the external report, no negative impact on the gas composition could be seen during the industrial-scale FLEXCOKE cokemaking trial.

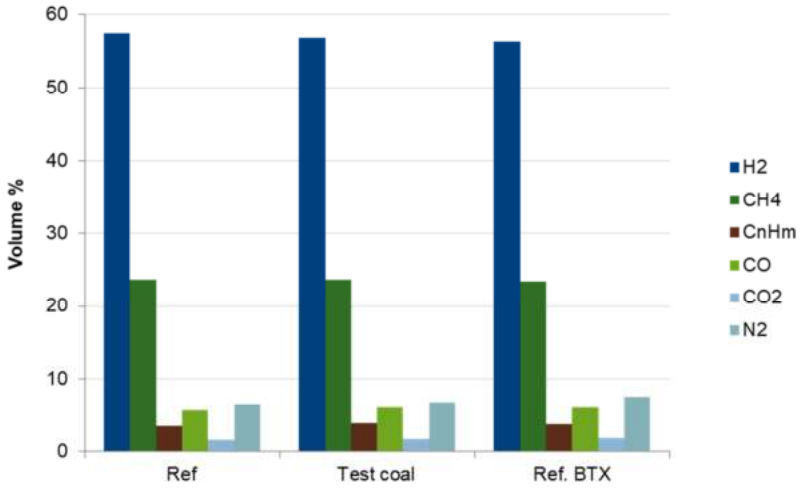



**Figure 165: Results of gas measurements made during the FLEXCOKE trial and reference**

The produced FLEXCOKE trial cokes physical characterisation data as particle size distribution and cold mechanical strengths are reported in Task 2.1, the results from ash analyses in Task 2.3 and the CRI and CSR results in Task 2.4.

Conclusions

Monitoring the ovens with alternative coke mix with 5% addition of thermal coal showed no harmful effects and no significant deviations in process parameters and both trial campaigns were operated smoothly even when all coke ovens contained 100% FLEXCOKE mix. However a longer trial period is needed to explore possible long term effects with respect to relative short trial period and reference period evaluated. Also a longer trial period is needed rule out that the coke breeze is not increasing even though the operators did not see any increase in dust.

Exploitation and impact of the research results

As no harmful effects on the battery, coke oven gas or the BF could be seen using 5% thermal coal in the coal mix SSAB will proceed outside the project with a new longer test period of 10 weeks. This longer trial period makes it possible to rule out any possible long term effects not seen in the shorter full scale industrial FLEXCOKE trial, also enable better monitoring of coke breeze, energy consumption etc.

### *Task 4.2 Blast furnace operational trials at the LKAB EBF®*

Planned activities and work accomplished

Ordinary SSAB standard coke and, at the SSAB coke plant, two alternative coke types for test in the LKAB Experimental BF (LKAB EBF®) were charged according to a trial program designed by the project group. Tests in the EBF® allow a larger deviation from original coke type regarding BF behaviour for coke, and 100% charging trials can be carried out without risking the steel production. Therefore, the EBF® trial was undertaken to evaluate performance of these two coke batches. Based on the results SSAB decided what test coke to produce in larger quantity, and in

what ratio to test it in the industrial scale BF trial. The EBF® operates 24 hours per day and the plant process management were the responsibility of LKAB that carried out the work with support from MEFOS, SSAB and tk SE. During the EBF® trials, LKAB measured the vertical temperature profiles by introducing a thermocouple from the top and the horizontal temperature and gas profile by using the shaft probes installed at two levels in the shaft. Solid samples were collected using the two shaft probes which are operated by LKAB. Two raceway cores were collected by MEFOS at short stoppages corresponding to standard coke and one type alternative coke charging. Selected sub-samples from the collected material were analysed in WP2 and evaluated and reported in this task and in WP3.

After the trials, the EBF® were quenched with nitrogen and excavated by LKAB, with support from MEFOS, according to routines and experiences of previous campaigns. During excavation, coke samples of one type of alternative were collected along the height and diameter of the EBF®. Careful sampling and investigation were also carried out for the raceways and the surrounding coke at the high temperature area. Additionally, cores were drilled out from the EBF® hearth in order to explore the coke properties. MEFOS and LKAB have, with support from tk SE and SSAB cooperatively evaluated the operational tests at the EBF® by collection of material and operational data during reference period and during the trials with alternative coke.

Description of activities, results and discussion

The two alternative coke types, produced as described in Task 4.1, TC1 and TC2 have been tested in LKAB EBF®. The EBF® is operated in campaigns, typically lasting for six to eight weeks. Since the construction of the EBF® in 1996-1997, usually two and in some cases one campaign have been carried out per year. The operation and control of the EBF® are comparable with the industrial BF but the response time is shorter. Figure 166 gives an overview of the EBF® plant and a photo image of a tapping. The process is operated continuously during campaigns and in all relevant aspects comparable to how production scale blast furnaces are operated. Raw materials (pellets, sinter, coke and slag formers) are screened and brought to day bins. From there they are weighed batch wise, brought by a skip to the furnace top and distributed across the shaft using bell-less top. Blast air is compressed and preheated to around 1200 °C in heat exchangers. Oxygen can be added, to the blast and to the pulverised coal injection (PCI) lances via the oxy-coal system. Pulverised coal (PC) is supplied daily from the nearby SSAB Luleå plant. Top pressure is usually controlled to 1 bar above atmospheric pressure and off-gas particles are removed from the gas by a dust catcher separating the dry, coarser flue dust. The finer part is removed from the gas stream by using a venture scrubber in combination with a wet electrostatic filter.

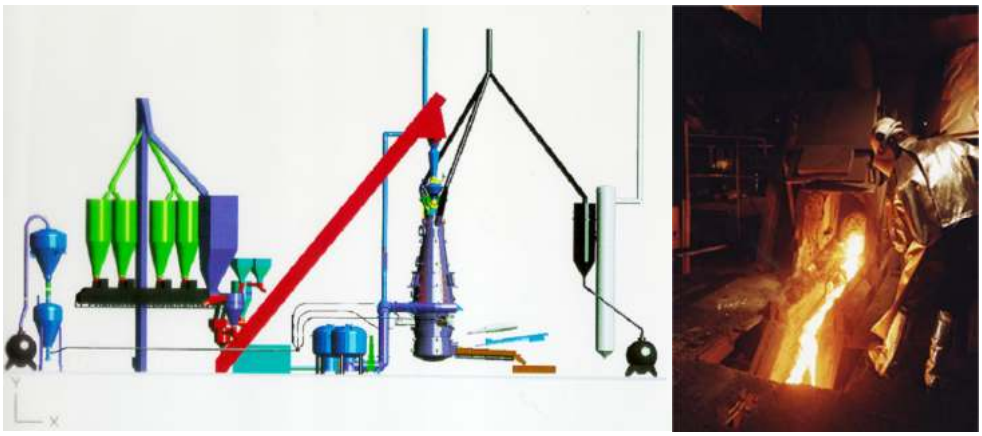


**Figure 166: Overview of the EBF® plant and a tapping**

FLEXCOKE test cokes were tested in the 32nd EBF® campaign, run September to November 2016. A two-week period at the end of the operating period starting with a reference and followed by operation with test coke, November 1st to 11th was allotted for the evaluation of the two coke batches. Data from a the preceding operating period, with standard coke (from the SSAB coke plant in Luleå) was used as reference data but due to some process irregularities an additional reference period was evaluated as well.

Apart from the two batches of test coke the EBF® was operated using what is considered as "standard materials". Chemical compositions of all charged materials are compiled in Table 44. The burden is similar to what is charged to the SSAB Blast Furnace in Luleå. The ore was 100% LKAB Malmberget Olivine Pellets (MPBO). The MPBO used in the trial originate from three different batches, all within normal product specification but with individual material codes. Reference coke, pulverised coal, and LD-slag from SSAB were used, together with limestone and some quartzite.

The quartzite is needed in the EBF® to compensate for high silica content of the hot metal (HM) compared to larger blast furnaces. Typical consumption figures are compiled in Table 45.

**Table 44: Chemical composition, in wt.%, of charged materials used during the trial**

| | MPBO -322 | MPBO -324 | MPBO -329 | Lime stone | LD slag | Quartzite | Coke Ref | TC2 | TC1 | PC |
|---|---|---|---|---|---|---|---|---|---|---|
| **Fe** | 66.64 | 66.94 | 66.96 | 0.46 | 19.86 | 0.15 | 0.42 | 0.37 | 0.44 | 0.73 |
| **CaO** | 0.45 | 0.43 | 0.39 | 54.46 | 40.73 | 0.20 | 0.10 | 0.12 | 0.14 | 0.59 |
| **$SiO_2$** | 2.00 | 1.73 | 1.74 | 1.68 | 9.10 | 97.9 | 7.07 | 6.52 | 6.84 | 4.08 |
| **P** | 0.005 | 0.007 | 0.007 | 0.007 | 0.253 | 0.01 | 0.016 | 0.020 | 0.021 | 0.022 |
| **$Al_2O_3$** | 0.44 | 0.31 | 0.33 | 0.45 | 1.51 | 0.75 | 3.28 | 3.06 | 3.20 | 2.01 |
| **MgO** | 1.13 | 1.22 | 1.21 | 1.00 | 10.37 | 0.22 | 0.08 | 0.09 | 0.09 | 0.23 |
| **$Na_2O$** | 0.044 | 0.034 | 0.034 | 0.03 | 0.01 | 0.07 | 0.07 | 0.06 | 0.07 | 0.03 |
| **$K_2O$** | 0.014 | 0.008 | 0.009 | 0.15 | 0.01 | 0.25 | 0.12 | 0.15 | 0.16 | 0.16 |
| **$TiO_2$** | 0.37 | 0.31 | 0.29 | 0.08 | 1.73 | 0.03 | 0.18 | 0.17 | 0.18 | 0.06 |
| **$V_2O_5$** | 0.27 | 0.23 | 0.24 | 0.05 | 4.38 | 0.00 | | | | |
| **S** | | | | 0.07 | 0.06 | 0.03 | 0.55 | 0.68 | 0.59 | 0.27 |
| **MnO** | 0.04 | 0.06 | 0.05 | 0.04 | 4.05 | 0.01 | | | | |
| **Ox deg** | 1.49 | 1.49 | 1.49 | 1.0 | 1.29 | 1.0 | 1.5 | 1.5 | 1.5 | 1.5 |
| **Volatiles** | | | | | | | 0.8 | 0.6 | 0.7 | 20.7 |
| **Ash** | | | | | | | 11.3 | 11.3 | 11.8 | 8.23 |
| **C** | | | | | | | 86.7 | 87.4 | 86.9 | 80.9 |
| **Moisture** | 5 | 4 | 1.2 | 0.1 | 0.1 | 0.1 | 10 | 2.7 | 5.4 | 0.5 |

**Table 45: Specific consumption of charged and injected materials (kg/tHM)**

| MPBO | LD-slag (from SSAB) | Limestone | Quartzite | Pulverised coal | Coke |
|---|---|---|---|---|---|
| 1380 kg | 40-45 kg/tHM | 40 kg/tHM | 18-22 kg/tHM | 130 kg/tHM | ≈ 420 kg/tHM |

It was decided to use standard process settings for the EBF® during the FLEXCOKE trial period. These settings were initially inspired by SSAB operation but have changed slightly over time. Key values are stated in Table 46. A PCI rate of 130 kg/tHM is regarded as a rather moderate amount for the EBF® (at the set blast volume and oxygen enrichment) and it allows for increased PCR to be used as a means of quickly adjusting the heat level of the process. The only planned change during the operating period is the changes in coke type. The trial plan is shown in Table 47.

**Table 46: Process Settings during FLEXCOKE Trial**

| **Set Points for Selected Process Parameters** | |
|---|---|
| Expected production rate | 1.50-1.55 tHM/h |
| PCI | 200 kg/h (130 kg/tHM) |
| Expected total reductant rate | 550 kg/tHM (Coke Rate 420 kg/tHM) |
| Slag rate | 150 kg/tHM ($b_2$ 0.95) |
| Expected HM Si | 1,5% |
| Top pressure | 1.0 bar |
| Blast volume (before additives) | 1550 $Nm^3/h$ |
| Oxygen enrichment to lance and/or blast | 105 $Nm^3/h$ (25.5% $O_2$ in blast)<br>55 to lance + 50 to blast |
| Moisture content in blast | 13 $g/Nm^3$ |
| Blast temperature | 1200°C – set point, from heaters |
| Calculated RAFT | 2250°C |

**Table 47: FLEXCOKE Trial Plan. TC2 = Test Coke 2, TC1 = Test Coke 1**

| Start | Hours | Trial focus | Coke | Pellet |
|---|---|---|---|---|
| 2016-10-30<br>4 pm | *40* | Reference | Reference<br>(standard coke) | MPBO324 |
| 2016-11-01<br>8 am | *16* | Preparation for stop,<br>Drilling and restart | Reference - TC2 | MPBO324 |
| 2016-11-02<br>midnight | *54* | Operation on TC2 and sampling | TC2 | MPBO324 |
| 2016-11-04<br>6 am | *16* | Preparation for stop,<br>Drilling and restart | TC2 - TC1 | MPBO324 |
| 2016-11-04<br>10 pm | *86* | Operation on TC1 and sampling | TC1 | MPBO324 |
| 2016-11-08<br>noon | *68* | Operation on TC1 and sampling<br>(preparation for quenching) | TC1 | MPBO329 |
| 2016-11-11<br>8 am | *4* | Basket charging | TC1 | MPBO329 |
| 2016-11-11 noon | | Quenching | TC1 | MPBO329 |

The main purpose of the EBF® test was to reduce the risk of encountering process difficulties caused by insufficient performance of the altered coke, in the subsequent full scale test at SSAB. Coke evaluations have very seldom been attempted in the EBF® and a key concern is the time needed to fully (or sufficiently) exchange coke in the furnace, especially in more inactive regions of the belly and hearth. As large changes can be made in the EBF®, where a stable hot metal production is not an issue, it was decided to do the complete changeover from one coke to another in just one step. It should be noted that from quality testing of the industrially produced test cokes the difference compared to standard coke was quite small, and less than expected from laboratory and semi-industrial scale produced samples, earlier within the FLEXCOKE project.

An additional objective of the trial was to retrieve samples of all three coke grades from the raceway region. Therefore, each test period was preceded by a planned stop when raceway sampling was done. Prior to charging TC2, a sample of the reference coke was taken during a stop on November 1$^{st}$. And prior to charging TC1 on November 4$^{th}$, the operation was stopped, and a raceway sample was retrieved of TC2. The sampling of TC1 was done during the excavation, after quenching and cooling. Therefore, it was necessary to consider that the FLEXCOKE trial needed to be in the end of the operating period as the quenching of the furnace should be made with TC1. Prior to quenching, numerous test materials are charged as basket samples and there are several stakeholders to consider in setting the operating parameters for the last two or three days of the campaign. By selecting standard process setting (the same were used throughout most of the campaign), data from any operating could be used as reference if needed.

Main evaluation criteria for the comparison are process performance, in terms of process efficiency and stability. For the comparison, operating periods have been chosen, representing each coke grade. The length of the test periods vary significantly, due to how the trial was planned. TC2 was charged for three days, while TC1 was charged for a full week. Operation during the planned reference period (Reference A, Ref A) just prior to the FLEXCOKE trial was a bit problematic and some changes in charging were done that were not maintained in the FLEXCOKE trial. Therefore a second reference period (Reference B, Ref B) has also been included, from about a week earlier in the campaign. These periods are described below. In the evaluation presented here we have chosen to compare what we regard to represent the maximum relevant evaluable operating period, based on reasoning described below. The selected periods ranges from 52 to 67 hours and in addition to these, some calculations have also been performed including the entire time period from the start of the first reference period on October 20$^{th}$ till the end of the campaign, on November 11$^{th}$ (Total)

**Operating description of FLEXCOKE and Reference periods**

During the reference periods and in the FLEXCOKE trial three different batches of MPBO were used, produced under slightly different conditions but with no significant difference in metallurgical properties within normal specification. The only difference between these batches, that has an influence on some of the results considered here, is in moisture content. It does affect top gas

temperature slightly, how much water spraying is needed to protect equipment at the furnace top, and possibly also the amount of dust collected from the top gas.

Figure 167 shows some key operating parameters for the trial periods and later in the text described briefly in terms of main observations and actions for each test period. Figure 167 illustrates evaluated test periods, were a 4-day period is plotted and the period chosen for analysis is indicated by a "bluish block". The process parameters displayed are; blast flow (in black) where any stops or reductions in production rate are easily seen, "gas utilisation" ($\eta CO=CO_2/(CO+CO_2)$) in red, and calculated "direct reduction rate" (DRR, in green) as indications on process efficiency and stability, hot metal sulphur content (S, as grey bullets) is included as increased S content in hot metal (HM) is a reliable indication of low heat level in the BF. Top gas temperature (a 10 min moving average) is also plotted (blue), as is water spraying to the top gas (which is done when the temperature exceeds 280 °C, also blue but as "spikes"). Total reductant rate (RR) according to the active recipe is included, in light green, to indicate if active changes in charging have been done. This data does not consider charging of extra coke dumps.

The Ref B period (operation on 100% MPBO pellet and standard coke) started in the morning of October 19 and ended on the 24$^{th}$. As the trial period was preceded by a test period on mixed sinter and pellet burden some time was needed for transition, so process data from the 19$^{th}$ is not included in the evaluation. The process was rather undisturbed during the Ref B period, especially during the 20$^{th}$-22$^{th}$ of October and very few coke rate adjustments were done. The adjustments made were in the latter part of Ref B when the coke rate was increased due to cooler conditions. A period with cool taps (high S) can be seen in the afternoon of the 21$^{st}$, when RR is increased. Late on the 22$^{nd}$ there is a problem with some of the data collection and a few hours later a short stop is needed. We have chosen not to include process data from this period in the comparison.

Ref A period was run October 28 to early morning November 1. As already stated there were some process disturbances and episodes with cool taps during this period, and some alterations were made to the charging pattern. Therefore, the Ref B period was added for the evaluation. Nevertheless, a 52-h period from Ref A has also been used in the evaluation. Transition from summer time to normal time took place at 2 to 3 am on October 30, and we have chosen to start the evaluation period after this, and end it in the morning of November 1, when charging of extra coke for the core drilling stop was initiated.

The period at which TC2 was charged started and ended with a stop for sampling in the raceway region, on November 1$^{st}$ and 4$^{th}$ respectively. These stops required additional coke some hours before the stop, and afterwards it took at least 8-10 hours to fully recover to normal operating conditions. Two episodes with slow burden descent and cooler taps occurred during TC2 and the blast was reduced a brief period, but without additions of extra reductants. We have used the time period from midnight between November 1$^{st}$ and 2$^{nd}$ until the morning of November 4$^{th}$ for the comparison presented here.

The evaluated period with TC1 is November 4$^{th}$ to 7$^{th}$ and is representing an operating period with rather few disturbances. From the afternoon on November 7$^{th}$ until noon on the 10$^{th}$ considerable problems were experienced and operational data from this period is therefore not included in this evaluation. The problems were due to equipment failures and coke quality is not doubted.

The moisture content in both test cokes were lower compared to the reference standard coke, with only ~3% for TC2 and ~5% for TC1, compared to ~10% for the reference coke. As a consequence of the low moisture content, the top gas temperature was higher and varied more for the test cokes, and more frequent water spraying was also needed.

The selected time periods for comparison and evaluation are summarised in Table 48. "Total" is the long, modelled period that includes all periods, starting from Ref B and continuing to the end of the campaign. Unlike the other evaluation periods, this one also includes stoppages and some larger disturbances. This results in a significantly lower average production rate (about 100 kg less per hour compared to the other periods) lower gas utilisation (ηCO%) and also higher coke rate, see Table 48. The large standard deviation of for instance HM temperature, top gas temperature and the gas efficiency is also a result of the disturbances included.

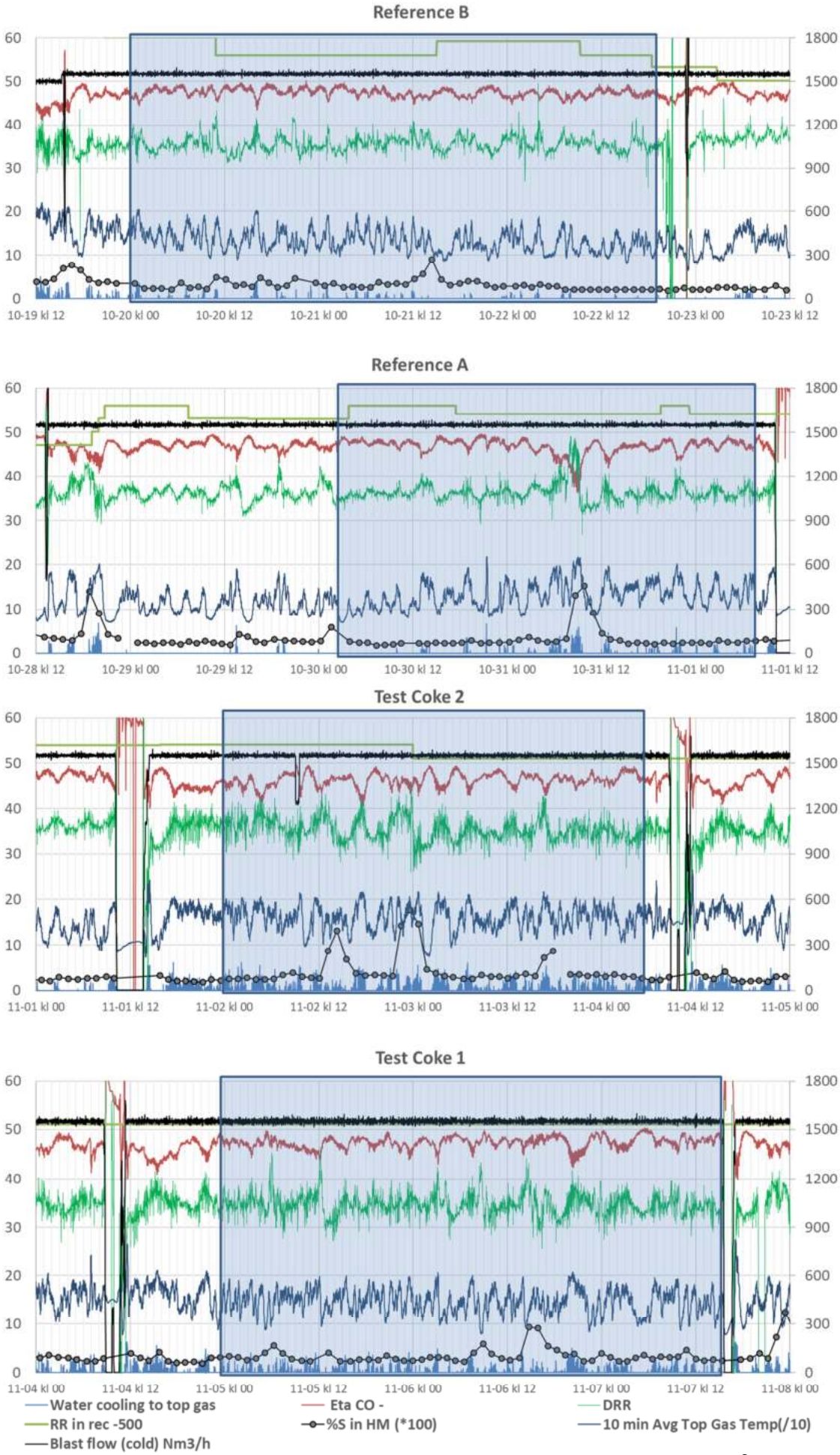



**Figure 167: Process overviews of reference and test coke periods in EBF® trial. Process data from time periods marked by blocks have been evaluated and used to compare performance**

**Table 48: EBF® periods for evaluation in the heat and mass model (MASMOD)**

| | Ref B | Ref A | TC 2 | TC 1 | Total |
|---|---|---|---|---|---|
| Starting | 10/20, 12 am | 10/30, 3 am | 11/01, 12 am | 11/04, 12 am | 10/20, 12 am |
| Ending | 10/22, 7 pm | 11/01, 8 am | 11/04, 6 am | 11/07, 3 pm | 11/11, 2 pm |
| Length (h) | 67.1 | 52.3 | 54.3 | 63.6 | 542.3 |
| | | | | | |
| Production rate (tHM/h) | 1.52 | 1.51 | 1.50 | 1.53 | 1.42 |
| Coke rate (kg/tHM) | 421 | 423 | 421 | 418 | 439 |
| PCI rate (kg/tHM) | 128 | 132 | 131 | 129 | 135 |
| Charged and injected C (kg/tHM) | 469 | 474 | 474 | 469 | 486 |
| Spec blast ($Nm^3$/tHM) | 1026 | 1035 | 1045 | 1029 | 1078 |
| Blast & lance $O_2$ - tot (%) | 25.9 | 25.9 | 25.9 | 25.9 | 25.9 |
| Gas utilisation ηCO (%) | 47.4 | 46.8 | 46.3 | 47.4 | 46.9 |
| Std. ηCO | 1.0 | 1.7 | 1.5 | 1.2 | 3.4 |
| Top gas temp (°C) | 132 | 132 | 160 | 146 | 142 |
| Std. TT | 35 | 39 | 46 | 42 | 47 |
| Charged water (kg/tHM) | 104 | 106 | 70 | 79 | 93 |
| Sprayed water (kg/tHM) | 1.7 | 3.5 | 11.4 | 6.3 | 8.2 |
| | | | | | |
| HM Temp (°C) | 1450 | 1448 | 1444 | 1451 | 1445 |
| Std. HM Temp | 25 | 42 | 29 | 19 | 34 |
| HM C (wt.%) | 4.48 | 4.35 | 4.31 | 4.33 | 4.38 |
| Std. HM C | 0.17 | 0.19 | 0.19 | 0.11 | 0.18 |
| HM Si (wt.%) | 1.60 | 1.58 | 1.51 | 1.68 | 1.55 |
| Std HM Si | 0.24 | 0.29 | 0.30 | 0.30 | 0.30 |
| HM S (wt.%) | 0.03 | 0.03 | 0.05 | 0.03 | 0.04 |
| | | | | | |
| Slag rate (kg/tHM) | 144 | 147 | 151 | 145 | 126 |
| B2 - $CaO/SiO_2$ | 0.96 | 0.92 | 0.88 | 0.92 | 0.92 |
| Std. B2 | 0.07 | 0.08 | 0.08 | 0.08 | 0.10 |
| B3 – $(CaO+MgO)/SiO_2$ | 1.42 | 1.37 | 1.31 | 1.35 | 1.37 |
| BR - Bells ratio – $(CaO+0.69*MgO)/(0.93*SiO_2+0.18*Al_2O_3)$ | 1.24 | 1.21 | 1.16 | 1.20 | 1.22 |
| $K_2O$ yield (%) | 88 | 96 | 99 | 83 | 79 |

Referring to process parameters in Table 48, the highest gas utilisation is found in Ref B and TC1 periods, indicating that the process was running most efficiently, and undisturbed at these times. Total charged carbon is also lowest in these periods, as is the standard deviation in most parameters. Note that the total carbon charged, given in the table is based both on the coke and PC rates and C contents on the respective materials, stated in Table 44.

During the Ref A and TC2 periods, the total C input was higher. For Ref A, this can partly be explained by some episodes with temperature drops in the early part of the period, resulting in extra coke and PC additions. The charging recipe was adjusted in halftime to have less central gas flow due to high top gas temperatures. TC2 is the period with the highest $K_2O$ yield and lowest heat level. This is indicated by low hot metal temperature and Si content. As a consequence of this, slag basicity is low and slag rate high. The gas utilisation was found to be lowest in TC2 followed by Ref A, which can be linked to higher amount of C available. Both periods includes episodes with slow burden descent, which is also known to have a detrimental effect on the gas efficiency.

### Description and results of heat and mass balance calculations in MASMOD

The different operational cases at the SSAB industrial BF No. 3 (see Task 4.3) and at the LKAB Experimental blast furnace have been studied in a static heat and mass balance model, MASMOD.[(11),(12)] A principle sketch of the model is seen in Figure 168. The BF process is divided into an upper zone and a lower zone that are connected by the equilibrium between the gaseous

components $CO/CO_2$ and $H_2/H_2O$ together with solid Fe/FeO in the thermal reserve zone. The input to the BF model is by injectants and blast in the lower zone as well by top charged raw materials via the burden model. In the burden model, the required production rate, slag basicity and reducing agents in the BF for the specific case is achieved by calculating the amounts of raw materials. The balances for material, slag, metal, gas components and energy are closed by iterative calculations. For each calculation case, the thermal reserve zone temperature has been adjusted and the shaft efficiency deduced as a first step. Thereafter, the shaft efficiency, heat losses and their distribution between the lower and upper zone were fixed to calculate the coke rate needed to close the balances. If the coke rate, heat losses and the gas balance could be closed with rather small adjustments, the calibration can be regarded as successful. For the opposite case when there are large deviations between the coke rate needed to close the balances and the actual amount of charged coke to the process, the difference is assumed to be related to changes in coke reserve. For instance; if the measured coke rate is larger than iterated by the model, the coke reserve is assumed to increase and vice versa.

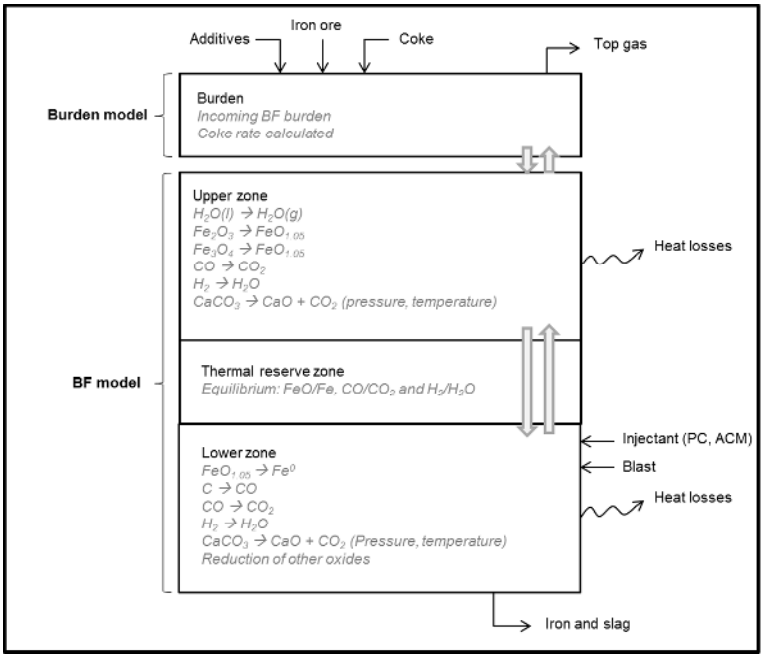



**Figure 168: Heat and mass balance model of BF process [12]**

In Table 49, the shaft efficiency, thermal reserve zone temperature (TRZ temp) and heat losses for evaluation cases in MASMOD are listed. The shaft efficiency has been iterated to close the balances at a fixed TRZ of 1030 °C. The total heat losses have been calculated based on the cooling losses from the water-cooled part of the shell, a fixed value for the remaining area of the shell and also as a function of the top gas temperature. The highest total cooling losses were found in the TC2 period even though the losses to the water cooled shell was moderate. This is likely due to the high top gas temperatures during this period, see Table 48.

**Table 49: MASMOD Parameters for each EBF®-case**

| Parameter | Ref B | Ref A | TC2 | TC1 | Total |
|---|---|---|---|---|---|
| TRZ temp (°C) | 1030 | 1030 | 1030 | 1030 | 1030 |
| Shaft efficiency (%) | 96 | 95 | 94 | 96 | 95 |
| Heat losses from water cooled shell (MJ/h) | 639 | 605 | 630 | 634 | 616 |
| Total heat losses (MJ/h) | 905 | 868 | 1032 | 968 | 930 |

The calculated C-consumptions for the evaluation periods can be seen in Figure 169. The Si content in HM has also been plotted in Figure 169 to give an indication on the heat level. The highest calculated C-consumption was 479 kg/tHM corresponding to the period when operating with TC2. Referring to the heat level indicated by the Si-content in HM, TC2 have the highest C-consumption but lowest heat level. Highest Si content in HM is found for the TC1 period but the theoretical amount of C required by the process was only 473 kg/tHM, which is the same rate as in Ref A. The "Total" period starts at the same time as Ref B and is overlapping all other evaluation periods as well as stoppages during- and in-between these periods. This period proceeds to the final end of the campaign. The calculated C-consumption for Total period at 475 kg/tHM is based on average HM- and slag analyses as well as process data from this time slot and the data should therefore represent the overall process state, which includes both stable and less stable periods.

In all periods except for the "Total" period, the calculated C-consumption in Figure 169 is higher than the total C-input from coke and PCI given in Table 48. The difference being 4-5 kg/tHM for periods operating with alternative coke and highest for the "Total"-period at 11 kg/tHM.

The heat loss from the water cooled shell was more or less similar in periods Ref B and TC2 (see Table 49) but the top gas temperature differed, being significantly higher in TC2. As previously mentioned, the total heat loss value has been calculated by considering the heat losses from the water cooled shell, the non-cooled shell, which is a fixed value, and the top gas temperature. The lower moisture content in alternative cokes, especially in TC2, can partly explain the higher top gas temperature, thereby giving the higher heat losses in total for TC2 compared to Ref B. Hence, applying the same heat losses as in Ref B could indirectly be a way to better compare the periods despite different coke moistures. Therefore, C-consumption has been calculated for evaluation periods with normalised heat losses. As the Ref B period is representing a rather smooth process with few disturbances, this period was selected as the base case and the same heat losses as in Ref B have been assumed for the other cases (Ref A, TC2, TC1 and Total) to investigate how it impacts on the reductant rate while keeping the other parameters fixed in the model.

The C-consumptions in Figure 170 are compared between the original cases (Figure 169) and the normalised cases with respect to heat losses. When applying the same heat losses as in Ref B, the C-consumption decreases in all cases by 1-3 kg/tHM, except for Ref A which increased the C-consumption with 1 kg. The difference in the total C-consumption between TC1 and Ref B is less than 1 kg/tHM when normalising the heat losses and the difference between TC2 and Ref A is about 2 kg/tHM.

To further study the impact of coke moisture on top gas temperature and consequently the total heat losses and C-consumption, a new case for the TC2 period was calculated. A coke moisture content comparable to the reference coke at 10% resulted in a decreased top gas temperature from 160 °C to 126 °C. The total heat losses for case TC2 was thereby recalculated to 859 MJ/h resulting in a C-consumption of 475 kg/tHM, being 4 kg/tHM less than the original case but only 1 kg less than when only considering the same heat losses as in Ref B. By this it can be assumed that the top gas temperature and consequently the total heat losses in both TC2 and TC1 should have been more comparable to the reference periods if the coke moisture was equal. Hence, the elevated C-consumption calculated for the TC2-period can partially be explained by this theory.

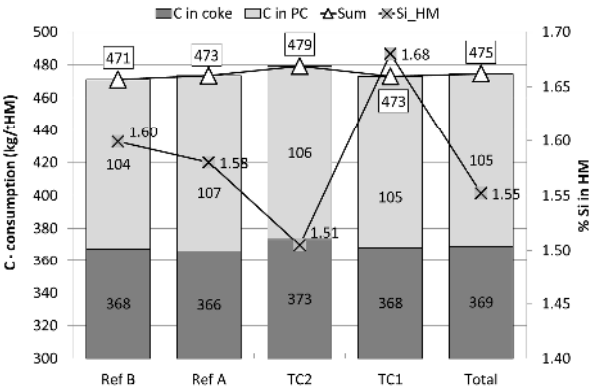



**Figure 169: C-consumption from coke and PC for evaluation periods**

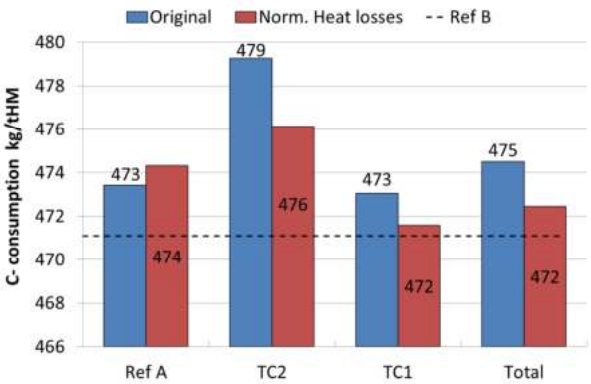



**Figure 170: C-consumption of normalised cases with respect to heat losses and HM composition**

In both test coke periods (TC1 and TC2), the calculated C-consumption in MASMOD was 4-5 kg/tHM higher than the charged and injected amounts given in Table 48. This is reasonable, as both periods where started after stops for core-drilling when substantial amounts of extra coke were charged. Depending on the actual coke rate needed by the process, this extra coke addition might have been consumed first within TC2 and/or TC1 period which partly could explain the gap between calculated C-consumption and the total C input. This since additions made before or after has not been considered in the total C-input (Table 48) for the evaluation periods. The fact that no compensations in coke or PCR was necessary in order to recover from the episodes with high alkali output and colder taps in TC2 period supports the theory on a sufficient coke reserve. This is also supported by the large gap between calculated C-consumption (Figure 169) and the C-input presented in Table 48 for the "Total"-period which implies that coke was charged in excess to what was needed by the process in overall. The Ref A period also contained some episodes with colder conditions but in contrast to the TC2 period, the coke reserve was assumed to be smaller. Thus, extra coke- and PC additions were necessary at these occasions and the calculated C-consumption is therefore closer to the charged and injected amount in this period.

**Probe measurements in the shaft and from the top**

Gas profile in terms of temperature and gas composition (CO, $CO_2$ and $H_2$) in the EBF® shaft are monitored daily, using shaft probes installed at two levels. The shaft probes are water cooled and measurements are carried out at nine predetermined positions along the furnace diameter. At each measuring position the probe is kept stable for one to two minutes, and a pipe containing a thermocouple and gas inlet is pushed some centimetres into the burden. Gas is cooled and analysed for CO, $CO_2$ and $H_2$. For evaluation the stabilised readings of temperature and gas components are recorded. Results are plotted in Figure 171 (temperature) and Figure 172 gas utilisation, ηCO, for the upper shaft probe and in Figure 173 and Figure 174 for the lower shaft probe. The positions of the probes are illustrated to the left in Figure 175.

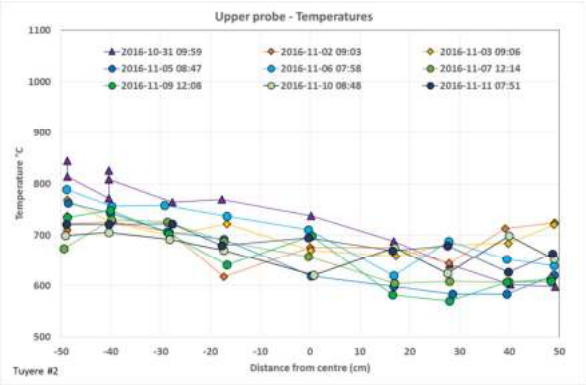



**Figure 171: Temperature profiles from FLEXCOKE periods, measured by the upper shaft probe**

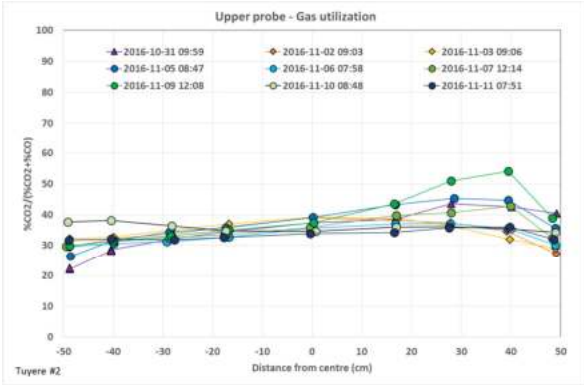



**Figure 172: Gas utilisation profiles from FLEXCOKE periods, measured by the upper shaft probe**

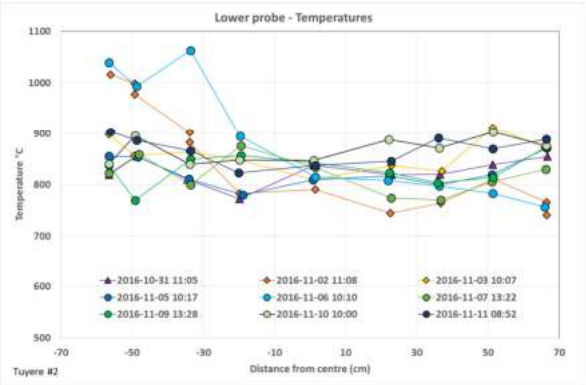



**Figure 173: Temperature profiles from FLEXCOKE periods, measured by the lower shaft probe**

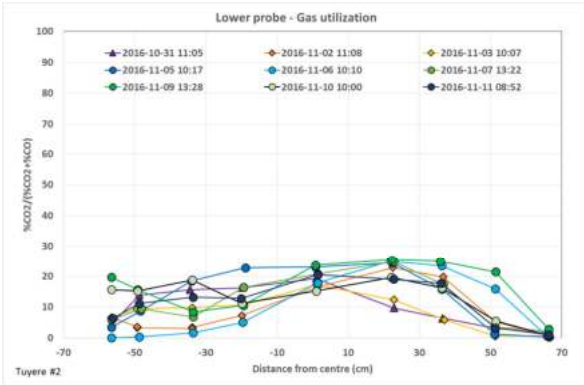



**Figure 174: Gas utilisation profiles from FLEXCOKE periods, measured by the lower shaft probe**

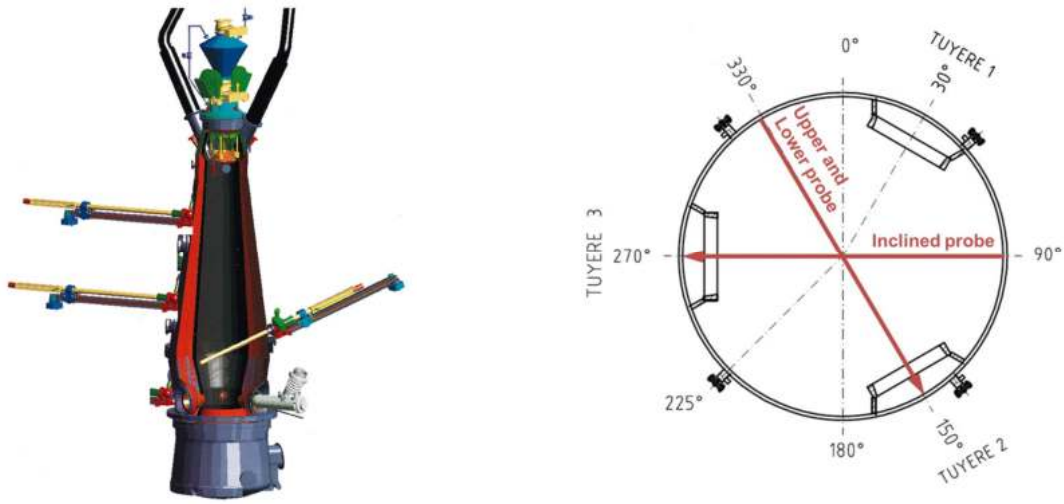



**Figure 175: Position of the EBF® shaft probes (left) and radial position (right). Inclined probe not used in the FLEXCOKE trial**

Compared to recent EBF® campaigns when the central coke chimney has been rather pronounced, the charging pattern was modified in this campaign to get more equal ore to coke ratio across the shaft diameter. Probe profiles indicate that this was achieved.

The shaft probe measures along a diameter were tuyere 2 (out of three) is situated at (below) one of the endpoints, as indicated to the left in the diagrams, and showed to the right side in Figure 175. The gas profiles show differences between the regions right above a tuyere and across from one. In this respect the EBF® with only three tuyeres 120° apart, differs from a production scale blast furnace. In this trial there is no indication of a central "gas chimney" but gas temperature is often a bit higher right above the tuyere while gas utilisation is slightly higher across from the tuyere. Most of the displayed measurements were done during the operation with TC1 (circle shaped bullets) and substantial variation can be seen within this period. Data from the TC2 period (rhombic bullets) do not differ from data from the TC1 period.

On few occasions LKAB also measured a vertical temperature profile by introducing a thermocouple from the top of the furnace and let it descend through the shaft along with the solid burden. Some examples from the FLEXCOKE trial period are shown in Figure 176.

When this kind of measurements is done the radial position of the thermocouple is not known and may vary, so several measurements are recommended. Specific for these measurements were also some "handling issues" that made the recording of how deep into the shaft the thermocouple had travelled a bit uncertain. What we can see is a rather quick temperature increase, to around 700°C within 10-20 minutes and thereafter a slower increase of the temperature.

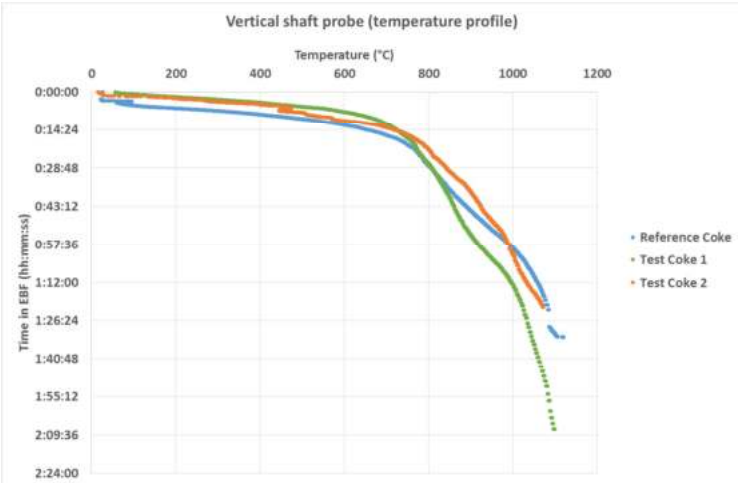



**Figure 176: Temperature measurements with thermocouple following the solid burden from stock line, until the thermocouple breaks (at around 1100°C)**

*Shaft probe solid samples taken out during operation*

Solid samples have also been collected using the two shaft probes shown in Figure 175. The probes have an inner diameter of around 6 cm and reach across the full diameter of the Experimental blast furnace. The diameter varies along the furnace height, from 1.1 m at the upper probe position and 1.4 m at the lower probe position respectively. The probes are forced through the burden by vibrations in horizontal and rotating directions, and pulled back after having reached their end positions, close to the far wall. Probes are water cooled (between an outer and inner steel tube) and the material inside is further cooled by purging nitrogen gas. When the temperature is brought down, the probe is emptied, in portions representing about 20 cm length. How well they are filled varies, but typically two to four sub-samples are collected, as illustrated in Figure 177.

During the FLEXCOKE period each probe were operated ten times (roughly once a day) resulting in a total of 67 sub-samples. Examples of how the material from the shaft probes may look are shown in Figure 178. The material from each samples have been screened and sorted. Square sieves are used and all material collected on the 12.5 mm, 10 mm and 6.3 mm sieve have been sorted into five categories; pellets, coke, slag formers, mixed aggregates and on a few occasions larger pieces of what looks like accretions have been found. However these seem to be rather brittle and it is more common that they break into smaller pieces. Material collected on 3.3 mm, 0.5 mm and <0.5 mm sieves have not been sorted, only weighed. However, results presented are within the selected, stable time periods shown in Figure 167 and Table 48.

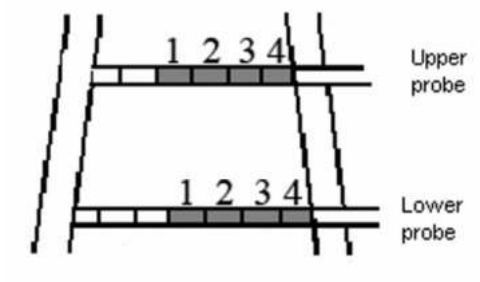



**Figure 177: Est. position and extension of sub-samples 1–4 taken out with EBF® shaft probes [13]**

**Figure 178: Examples from visual inspection of unsorted sub-samples from shaft probes**

Figure 179 show results from the sorting and sieving of some sub-samples gained from two shaft probing made during TC1, upper probe to the left side and lower probe to the right side of the figure. Generally, the variations between samples are considerable and may have several reasons, for example to what extent the probe are made in a coke or pellet layer and to what extent material has started to sinter together. Aggregates are in principle only found in samples from the lower probe, and these also contain more fines. Also, the sub-sample taken out close to the BF wall, i.e. the sub-sample with the highest number, generally contains more fines as seen in Figure 179 and in earlier investigations [13] In addition, more fines >0.5 mm are present in the lower shaft probe samples as seen in Figure 180 and earlier investigations. Figure 180 illustrates the ore to coke ratio and amount of fines >0.5 mm in the upper (to the left side) and lower (to the right side) shaft probes taken during Ref B, TC2 and TC1 test periods. A low ore to coke ratio means that the larger amount of coke is found in the sub-samples, indicating probing in coke layer.

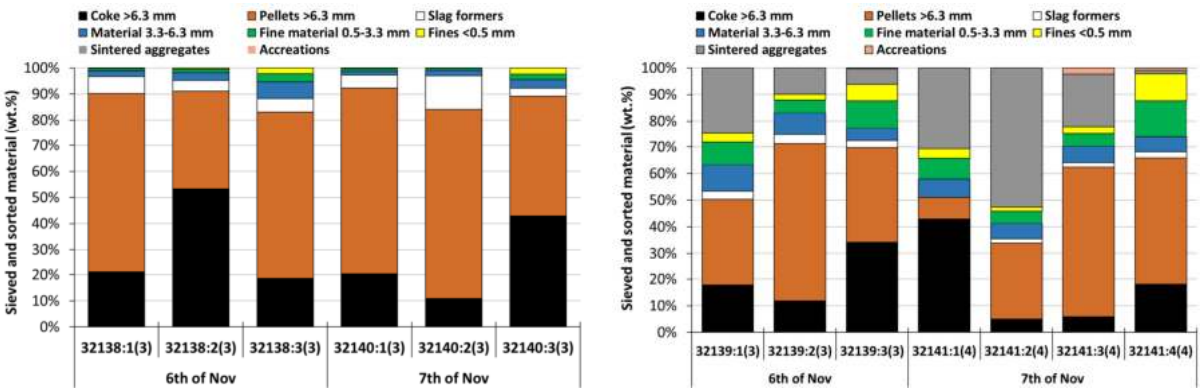



**Figure 179: Examples of material distributions after sorting and sieving of sub samples from two material probings during operation with TC1. Upper shaft probe (left) and lower shaft probe (right)**

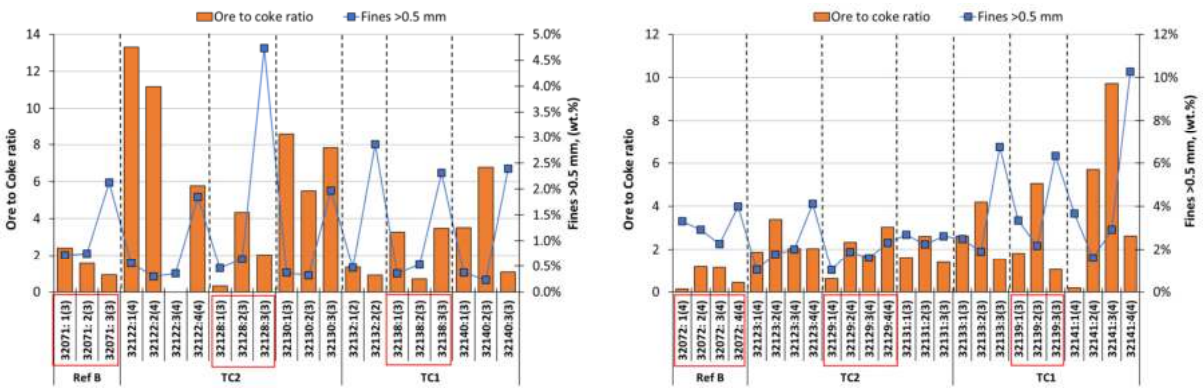



**Figure 180: Ore to coke ratios and amounts of fines >0.5 mm in shaft probe sub-samples from upper probe (left) and from lower probe (right) for Ref B, TC2 and TC1**

Chemical XRF analyses were made by SSAB and the ash contents, representing by sum of oxides, which are plotted in Figure 181. Generally the ash content is higher in the lower shaft probe sub-

samples comparing to the upper probe. Sub-sample 1, which is positioned towards the centre of the EBF®, shows higher amounts of ash in coke than the sub-samples closer to the wall.

No significant differences in the ash contents generated in the shaft can be noted for the standard coke (Ref B and Ref A) and test cokes TC2 and TC1, even though TC1 has slightly higher ash content. As the majority of each investigated coke samples showed similar results, the chemical analyse and graphitisation degree results in one probing (upper and lower) of each test periods are presented.

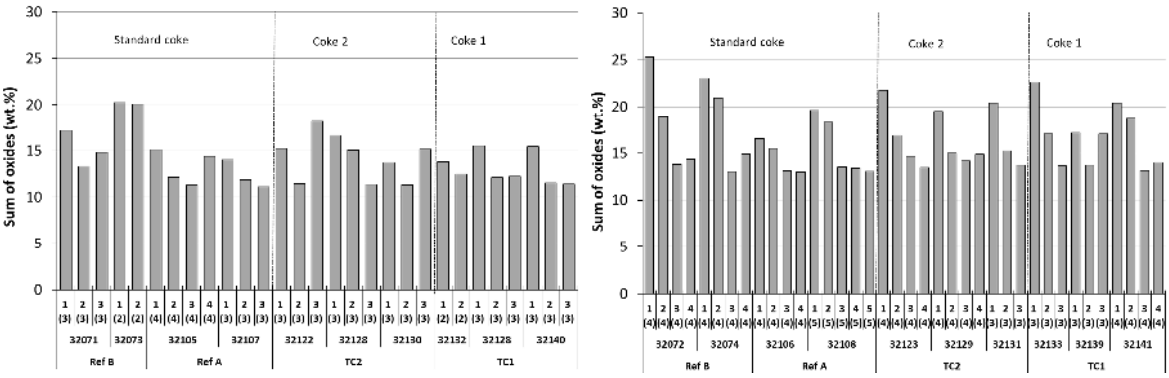



**Figure 181: Ash content (sum of oxides) in sub-samples from upper probe (left) and from lower probe (right) for Ref B, Ref A, TC2 and TC1**

The high ash content in sub-sample 1 and, in some cases 2, is mainly related to elevated contents of CaO and MgO in coke in the upper and S, alkalis in the lower shaft probes, see Figure 182.

Fe is increased more evenly in the sub-samples with an increase in lower shaft probe and towards the EBF® wall (high sub-sample no.). $SiO_2/Al_2O_3$ ratios are increased compared to each charged coke type but similar in both upper and lower shaft probes with an exception of high values in upper probe in Ref B. The alkali content is low in the upper shaft probe sub-samples in all coke types. The alkali recirculation contributes to alkali up-take in coke during its decent in the BF. The sub-samples in the lower shaft probes shows higher amounts of alkali, especially in Ref B standard coke type but also with TC2. The TC1 coke has lower up-take of alkali than the others.

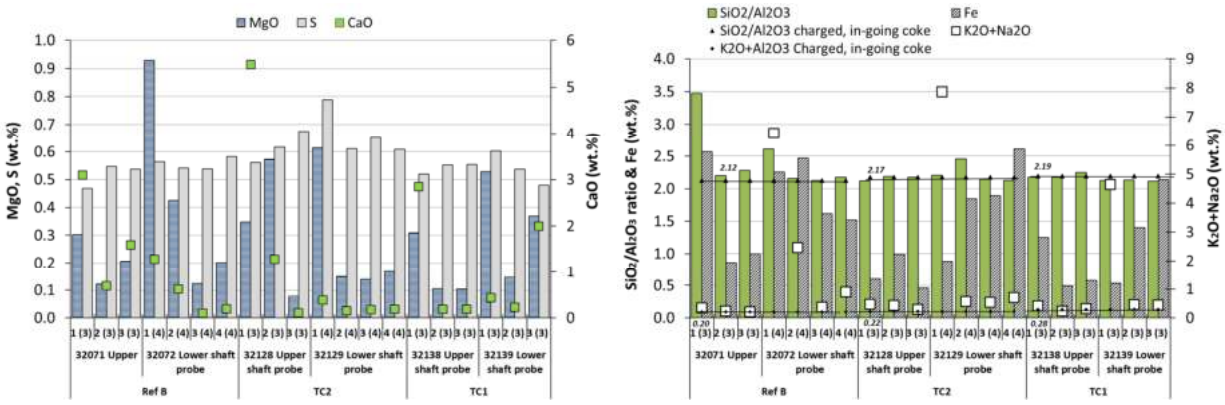



**Figure 182: Chemical composition of coke in shaft probe sub-samples. MgO, S, CaO (left) and Fe, alkalis and $SiO_2/Al_2O_3$ ratio (right)**

The graphitisation degrees, i.e. Lc, of coke taken out in with shaft probes are plotted in Figure 183. The figure shows that the graphitisation degree of the majority of shaft coke samples has not exceed the one of the charged coke.

There is a general trend indicating that almost all types of coke shows the highest graphitisation degrees at sub-samples 1 and 2 located closer to the centre of the furnace at the upper shaft probe while the graphitisation degrees are lower closer to the wall particularly at the lower shaft probe. Lc can be used as a temperature indicator. The carbonisation temperature of the coke is around 1000 to 1040 ˚C which means that the temperature in the shaft measured by the shaft probes (around 600 to 800 ˚C) do not increase the coke graphitisation at the position of the shaft probes. However, the actual temperatures may be higher due to the effect of water cooled probes and locally higher temperatures may occur when the gas speed can be locally higher.

The fact that the Lc is lower in many cases than for the charged coke may be due to up-take of oxides, which is higher at the wall position that affects the coke or more that there are natural variations in coke graphitisation.

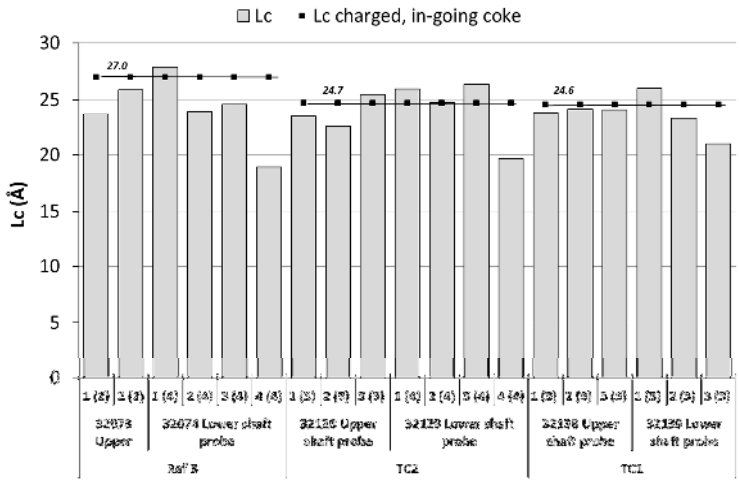



**Figure 183: The graphitisation degree (Lc) of coke in shaft probe sub-samples. Lc of charged coke indicated**

### Investigation of off-gas dust samples taken during operation of LKAB EBF®, C32

40 dry, coarse flue dust samples and 40 sludge samples were collected during Ref A, TC2 and TC1 periods. Sludge samples were taken out at the same time as the flue dust samples. The sludge were filtered and dried and from the wt.% solids measured the amounts in kg/h were calculated. Chemical analyses and XRD were made and evaluated in all samples.

Flue dust and dried sludge sampled during stable periods, i.e. same evaluation periods as in Table 48 are presented here. The average amounts of off-gas dust in kg/tHM are presented in Figure 184. The flue dust expelled from the top was less during the trial with the alternative coke although the difference was not that high. On the other hand were sludge amounts higher with alternative coke, especially with TC2, giving the highest total off-gas dust of around 7.9 kg/tHM.

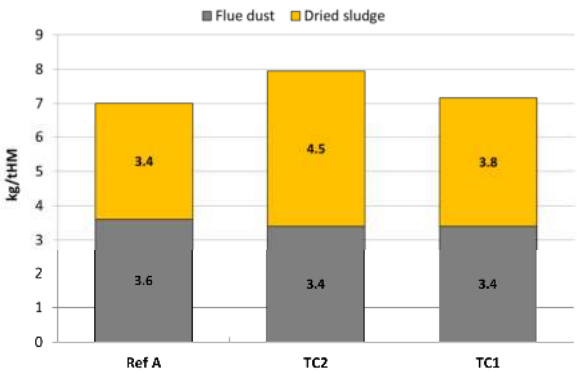



**Figure 184: Amounts of average off-gas dust (kg/tHM) during Ref A, TC2 and TC1 test periods**

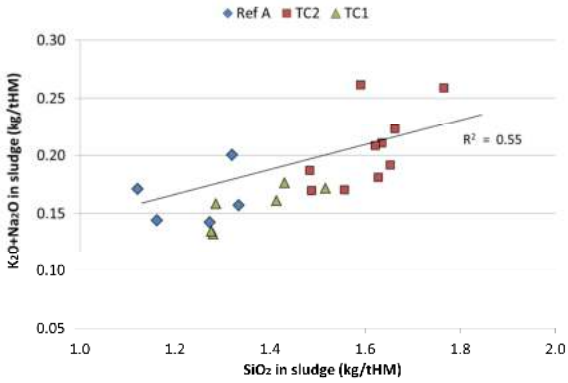



**Figure 185: Correlation between alkali and the $SiO_2$ amounts in sludge**

The chemical analyse, re-calculated into kg/tHM, for the coarser dry, flue dust and in the dried sludge are presented on an average basis in Table 50. The flue dust samples do not vary that much between the evaluated periods but Ref A coke period shows highest alkali and silica while TC1 period shows the lowest alkali and silica. The C contents in flue dust expelled from the top are similar. TC2 has the highest amount of sludge and has also the highest amounts C, S and all oxides as alkali, CaO, $SiO_2$, etc. This indicates that the reduction and evaporation of silica and alkali is high during this period as in a previous study [14] it was a correlation between raceway conditions (as PCR and flame temperature etc.) and these elements in the EBF® sludge. The correlation between $SiO_2$ and alkali in the sludge is shown in Figure 185. The $R^2$ value is lower than previous study probably due to the more flat gas profile in this EBF® campaign compared to earlier centre gas profiles.

**Table 50: Average chemical analyse in flue dust (top) and in sludge (bottom), in wt.% and in kg/tHM**

| Flue dust (wt.%) | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | **Fe** | **CaO** | **$SiO_2$** | **MnO** | **$P_2O_5$** | **$Al_2O_3$** | **MgO** | **$K_2O+Na_2O$** | **$TiO_2$** | **C LECO** | **S LECO** |
| **Ref. A** | 15.4 | 5.1 | 15.8 | 0.12 | 0.12 | 2.84 | 1.54 | 0.66 | 0.21 | 48.0 | 0.47 |
| **TC2** | 16.1 | 5.3 | 13.7 | 0.18 | 0.13 | 2.60 | 1.37 | 0.61 | 0.21 | 49.1 | 0.46 |
| **TC1** | 16.0 | 6.2 | 10.9 | 0.17 | 0.13 | 2.80 | 1.68 | 0.52 | 0.22 | 51.3 | 0.46 |
| **Flue dust (kg/tHM)** | | | | | | | | | | | |
| **Ref. A** | 0.55 | 0.18 | 0.55 | 0.004 | 0.004 | 0.10 | 0.054 | 0.024 | 0.008 | 1.73 | 0.016 |
| **TC2** | 0.55 | 0.18 | 0.47 | 0.004 | 0.004 | 0.09 | 0.046 | 0.021 | 0.007 | 1.67 | 0.015 |
| **TC1** | 0.54 | 0.21 | 0.37 | 0.006 | 0.004 | 0.10 | 0.057 | 0.018 | 0.007 | 1.74 | 0.015 |
| **Sludge (wt.%)** | | | | | | | | | | | |
| | **Fe** | **CaO** | **$SiO_2$** | **MnO** | **$P_2O_5$** | **$Al_2O_3$** | **MgO** | **$K_2O+Na_2O$** | **$TiO_2$** | **C LECO** | **S LECO** |
| **Ref. A** | 5.3 | 9.9 | 36.4 | 0.64 | 0.42 | 14.0 | 10.66 | 4.78 | 0.10 | 6.39 | 0.19 |
| **TC2** | 6.2 | 9.2 | 35.4 | 0.70 | 0.41 | 12.0 | 9.40 | 4.54 | 0.10 | 8.26 | 0.28 |
| **TC1** | 5.5 | 9.6 | 36.4 | 0.64 | 0.45 | 13.0 | 10.34 | 4.14 | 0.09 | 6.43 | 0.26 |
| **Sludge (kg/tHM)** | | | | | | | | | | | |
| **Ref. A** | 1.82 | 3.39 | 12.42 | 0.22 | 0.14 | 4.76 | 3.64 | 1.63 | 0.04 | 2.18 | 0.06 |
| **TC2** | 2.84 | 4.16 | 16.08 | 0.32 | 0.19 | 5.34 | 4.27 | 2.06 | 0.05 | 3.75 | 0.13 |
| **TC1** | 2.06 | 3.63 | 13.67 | 0.24 | 0.17 | 4.81 | 3.89 | 1.56 | 0.04 | 2.42 | 0.10 |

XRD measurements and evaluation of flue dust and sludge samples have been made. In Figure 186 examples of phases found in the flue dust during Ref A, TC2 and TC1 trial periods are shown and in in Figure 187 examples of phases found in sludge during the same periods. XRD analysis of flue dust exhibited that all of dust samples consists of four main phases; hematite, graphite, quartz and calcite and low concentration of feldspar "anorthite". Many samples showed presence of aluminium silicate "kyanite" while few samples showed presence of sodium silicate. XRD diffractogram of the sludge samples are very amorphous and made the evaluation of its peaks difficult. The intensity of silica and iron oxide in sludge during TC2 and TC1 trials are higher than that in Ref A trial. In addition, the intensity of calcium carbonate is also higher.

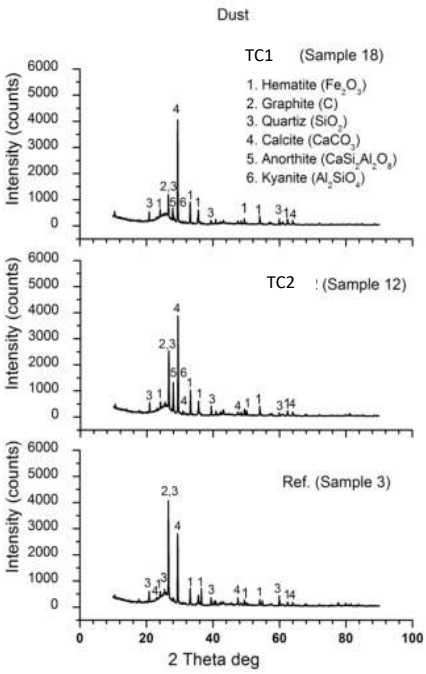



**Figure 186: Examples of XRD diffractograms of flue dust samples**

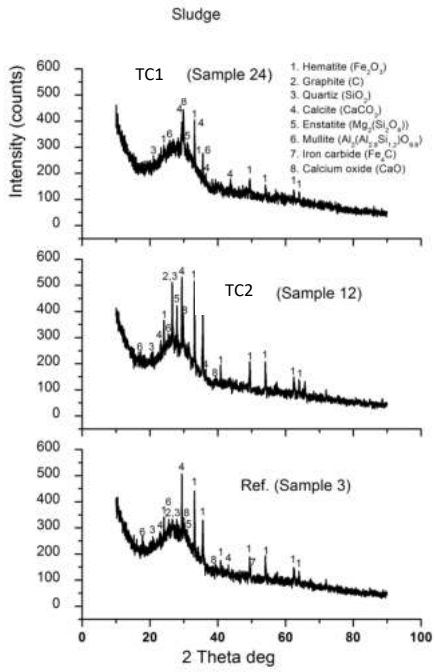
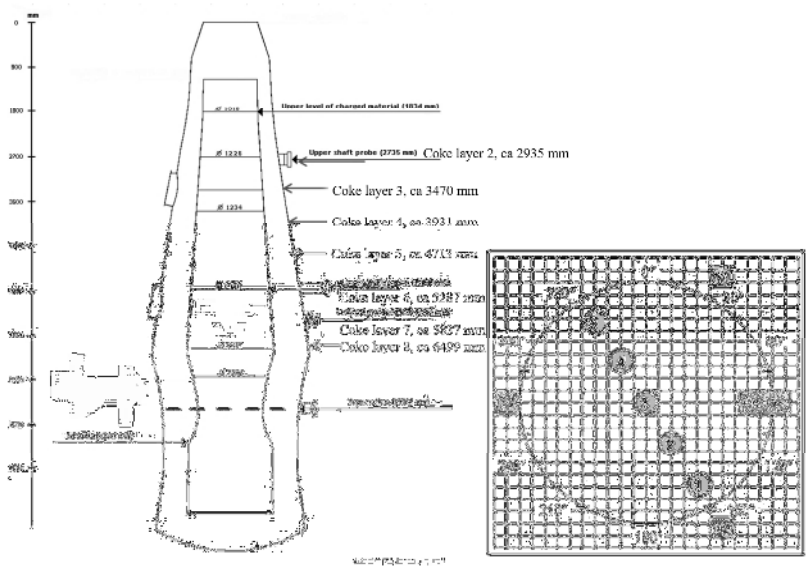



**Figure 187: Examples of XRD diffractograms of sludge samples**

**Excavation TC1 samples taken in the LKAB EBF® shaft, C32**

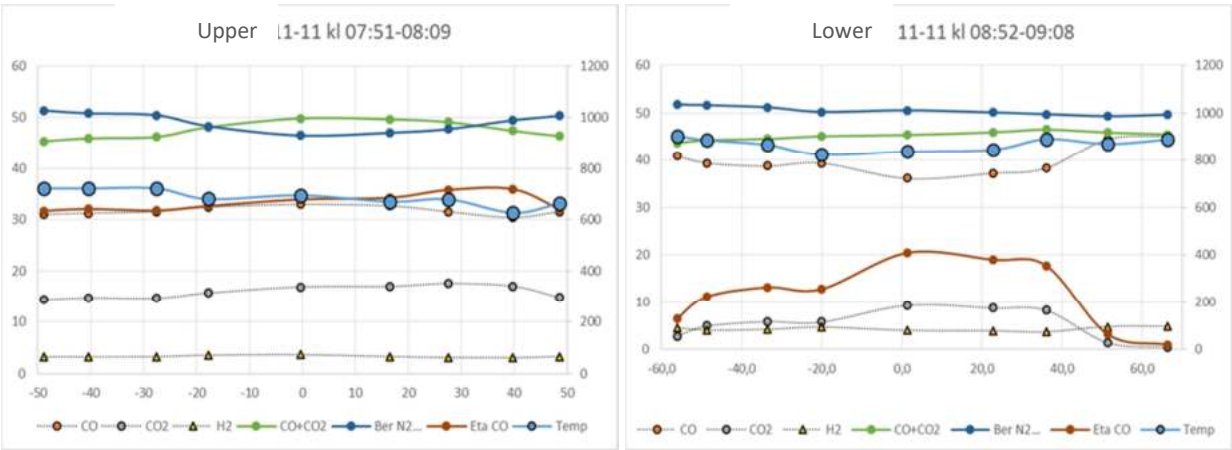
Excavation of TC1 samples were made by LKAB at sample layers 2 to 8, see (a) in Figure 188. At each layer five samples were collected according the horizontal pattern in (b). It is the same horizontal position as the shaft probe gas, temperature and solid sample position, see Figure 175. 35 samples in total were excavated and analysed by XRF for chemical analyse. Based on the result, XRD measurements were carried out for evaluation of the graphitisation degrees (Lc) on 20 samples in Coke layers further down in the shaft, i.e. Coke layers 4, 6, 7 and 8.




(a) Vertical position of coke layers (b) Horizontal position and sample No.

**Figure 188: Vertical position of excavation sample layers in the EBF® (a) and horizontal position (b) [15]. Same excavation procedure made in both C31 and C32**

Gas- and temperature probing made before the stoppage of the EBF® at the 11th of November 2016 are illustrated in Figure 189. The gas utilisation, Eta CO, and temperature is natural higher in the upper probe position in the shaft (2.7 m from the top) and the other way around in the lower probe position (5.2 m from the top). In the lower probe Eta CO increases slightly in the centre region.




FLEXCOKE trial C32 upper probe (left) FLEXCOKE trial C32 lower probe (right)

**Figure 189: Gas- and temperature shaft probing before basket charging, stoppage and quenching of the EBF®**

*Ash and chemical evaluation, comparison of C31 and C32*

For comparison, TC1 is compared to excavation samples from the previous EBF® campaign (C31) where standard coke was charged. The charging pattern between C31 and C32 differ and the charging of coke was with thicker coke layers in the centre creating a centre gas profile. During C32, coke was more evenly distributed over the diameter.

Figure 190 and Figure 191 shows the ash content in sum of oxides in excavation coke from C31 and C32, respectively. The charged standard coke used in C31 has 1% lower ash content compared

to TC1. In C32 the TC1 coke has similar ash content in all sample positions down to 5.9 m in the shaft, just below the lower shaft probe, and further down the ash content increases indicating some gasification of C or up-take of oxides. However, as the coke sample at 5 position shows higher ash content than the others the latter option is more likely. The horizontal position of sample 5 is at the wall in-between tuyere 1 and tuyere 3 and it is likely that the conditions is a bit colder than in sample 1 positioned above tuyere 2 in the shaft, see Figure 188. The ash in C31 with standard coke lacks the clear up-take in the lowest vertical position and has a higher ash already at 3.5 m from the top, this difference can be partly linked to different in-furnace conditions between campaigns.

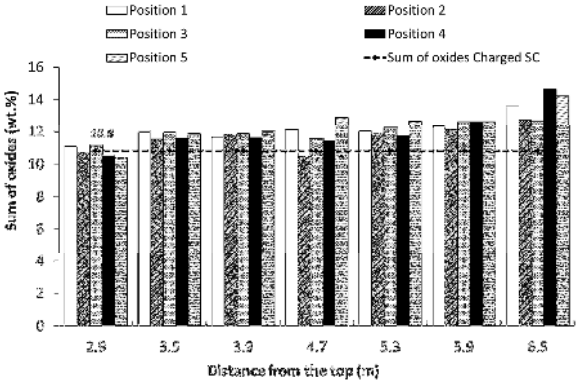



**Figure 190: Ash content, in sum of oxides, at different depth in the EBF® shaft C31 operating with standard coke (SC)**

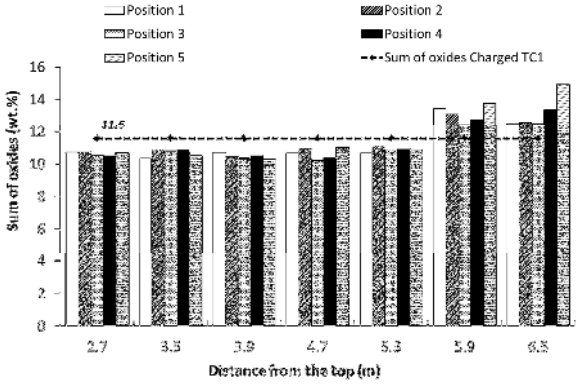



**Figure 191: Ash content, in sum of oxides, at different depth in the EBF® shaft C32 operating with TC1**

The alkali content ($K_2O$+$Na_2O$) shows typical behaviour of the coke up-take due to recirculating alkali during the descent in the BF shaft, see Figure 192 for the standard coke in C31 and Figure 193 for TC1 in C32. The standard coke has around 0.1% lower alkali content in the charged coke than TC1. The alkali contents are similar as the charged coke down to position 3.9 from the top and then starts to increase, initially on position 1 and 5 (wall samples). At 5.9 m from the top, at bosh level and the position of the inclined probe the alkali contents starts to be significant higher and increases even more at 6.5 m, especially at position 4 and 5 towards the wall in-between tuyeres.

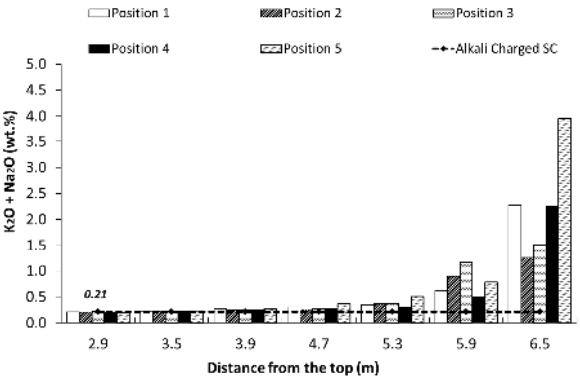



**Figure 192: Alkali content at different depth in the EBF® shaft C31 (Standard coke, SC)**

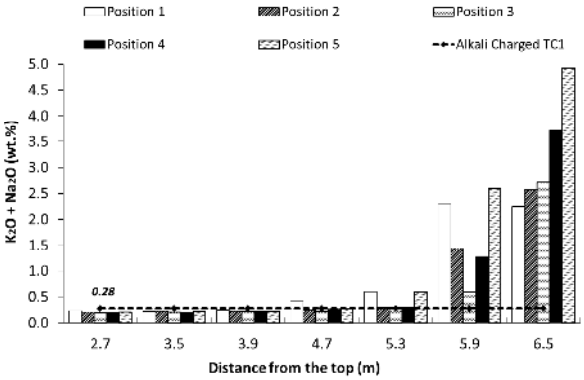



**Figure 193: Alkali content at different depth in the EBF® shaft C32 (TC1)**

*Graphitisation and temperature estimation of TC1, C32*

In Task 2.2 the evaluation of coke graphitisation degree, Lc, and temperature estimation method is described. The results of the evaluated graphitisation degrees of TC1 in the horizontal directions and at different heights in the shaft are plotted in Figure 194. The temperature in the EBF® shaft has not affect the TC1 at 3.9 m as the Lc are in the range of the charged coke. At 5.9 m from the top, the same level that alkali content started to increase significantly, Lc is increased especially at position 1, which is located above tuyere 2 where more and hotter gas flowing upwards.

The estimated temperatures are plotted in Figure 195. The estimated charged TC1 temperature of 910 ˚C seem quite low as the coke temperature during pushing varied between 980 to 1060 ˚C during the SSAB 1st trial period with and average coking temperature of around 1000 to 1030 ˚C However, the estimated temperature trend can be followed in the figure but actual temperatures are likely higher.

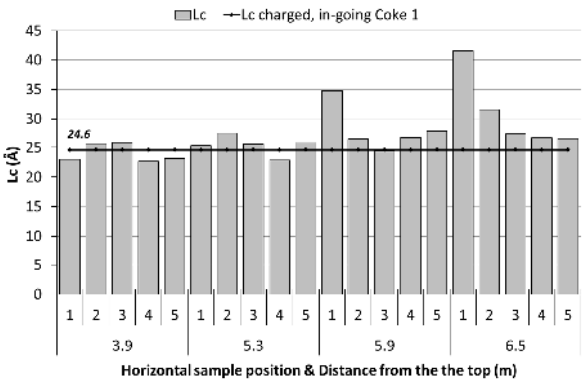



**Figure 194: The graphitisation degree (Lc) of TC1 at different depth in the EBF® shaft. Lc of charged coke indicated**

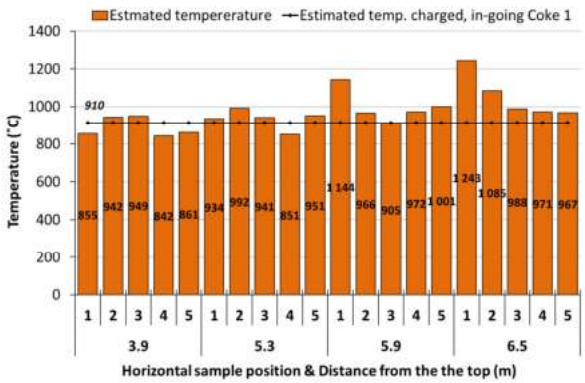



**Figure 195: Estimated temperatures of TC1 at different depth in the EBF® shaft, line shows estimated temp. of charged coke**

Conclusions

Investigation of coke collected with upper and lower shaft probes during operation of the EBF® showed no significant differences between the reference periods and alternative coke periods TC2 and TC1. Variations seen are typical for the probe samples seen in earlier investigations. The graphitisation degrees did not increase compared to charged coke indicating lower or similar temperature in shaft than coking temperature (~1000 ˚C).

Heat- and mass modelling shows that similar C-consumption likely can be achieved independent on if using standard coke, TC1 or TC2. Differences found were related to variations in the alkali output as well as changes cannot be linked to the coke type.

Investigation of off-gas dust during C32 shows that the amount of flue dust is less during the trial with the alternative coke although the difference was not that high compared to standard coke. On the other hand sludge amounts were higher with alternative coke, especially with TC2, giving the highest total off-gas dust. As the sludge in previous studies has been linked to gasification of elements in the high temperature zone does this fact indicate differences in energy consuming reactions in the lower part of the EBF®. Ref A coke period shows highest alkali and silica in kg/tHM while TC1 period shows the lowest alkali and silica in the coarser, dry flue dust. In sludge the highest amounts of alkali, C and other oxides was found in TC2 period. XRD measurements showed similar compounds regardless of test coke period.

Excavation of TC1 from the LKAB EBF® shaft at different heights show typical patterns of alkali recirculation and alkali up-take and comparison with excavation samples at similar positions after operation with standard coke has the same trends and dissimilarities are attributed to difference in charging pattern and/or operational conditions rather that differences in the coke types itself.

Exploitation and impact of the research results

According to evolution of the EBF® trial and MASMOD modelling the C-consumption are not affected using either TC1 or TC2. The exploitation of the results was that the project group chose to use TC1, with 5% addition of thermal coal to the blend, in SSAB full scale operational trials in coke plant and BF No. 3 simultaneously.

### *Task 4.3 Blast furnace trial on industrial-scale*

Planned activities and work accomplished

Industrial scale trials involve testing of a coke, produced from alternative coal blend, which needs to fulfil the requirements for coke to be charged at the industrial BF of SSAB. Specifications deduced in the project were the base in the design of the test program for charging of alternative coke.

A trial program with a ramp up of selected coke type was designed prior to the test. For each trial step, the SSAB analysed the operational state carefully in real-time before the next step is started to secure stable operational conditions. The operational results were collected and evaluated in comparison with the original coke used at the BF with a heat- and mass model (MASMOD) in order to evaluate C-consumption.

Description of activities, results and discussion

As described in Task 4.1, it was decided within the project group to go forward with TC1, i.e. 5% addition of thermal coal to the coking coal blend, during the full scale operational trial. The same type of thermal coal which was used in the EBF trial could not be purchased for the large scale industrial trial but were similar. Differences were that the thermal coal used in the 2nd cokemaking industrial trial had a bit higher ash, $SiO_2$, $Al_2O_3$ and alkali content than TC1 (Test Coke 1) produced and tested in the EBF trial which had a bit higher contents of VM, S, CaO, MgO and $P_2O_5$. Therefore the produced coke is named FLEXCOKE trial coke (FTC).

It was also decided to produce the alternative coke at the SSAB coke plant in Luleå and to charge it at the same time into SSAB BF No. 3 in Luleå (Task 4.3). The number of ovens filled with alternative C-containing material during the FLEXCOKE trial (i.e. 5% thermal coal to the coal blend) were successive increased so that a mixture of FTC and standard coke produced and sent the BF were; 20%, 40%, 80% and 100% i.e. until all the ovens in the battery contained the FLEXCOKE trial mix. The distribution of coals in the mixes during the trial is shown in Table 42. Compared to the 1st cokemaking industrial trial, see Table 41, the FLEXCOKE trial mix had higher amounts of MV coal in the mix and more of the HV coal was replaced by thermal coal.

The full scale trial at SSAB started the 9th of May and ended the 24th of June 2017. The ramping up of the FTC was closely monitoring for any harmful effects on the BF operation.

**Heat and mass balance modelling results for the FLEXCOKE trial at industrial trials at SSAB BF No. 3**

The periods when the BF was operated with the alternative coke with 5% addition of thermal coal to the blend, i.e. FTC, have been compared to reference periods with standard coke by means of heat-and mass balance calculations in MASMOD, see description of the model in Task 4.2. FTC was charged to the BF in ramp up steps by 20, 40, 80 and 100% of the total coke charged. The time periods for reference and ramp up periods are listed in Table 51.

The charged amount of C-containing materials per tonne HM in each period is presented in Figure 196 with the total fuel rate included.

**Table 51: Charged amounts of coke, PCI, BF dust inj., dust briquettes and the total fuel rate in each period. FTC=FLEXCOKE trial coke**

| Period | ID | Start date | End date | Duration (days) |
|---|---|---|---|---|
| Reference | R1, R2 | 2017-04-10 | 2017-05-08 | 28 |
| 20% FTC | T-20 | 2017-05-09 | 2017-05-18 | 9 |
| 40% FTC | T-40 | 2017-05-19 | 2017-05-22 | 3 |
| 80% FTC | T-80 | 2017-05-23 | 2017-05-30 | 7 |
| 100% FTC | T-100 | 2017-05-31 | 2017-06-24 | 24 |
| Reference, standard coke | R3 | 2017-06-30 | 2017-07-02 | 2 |

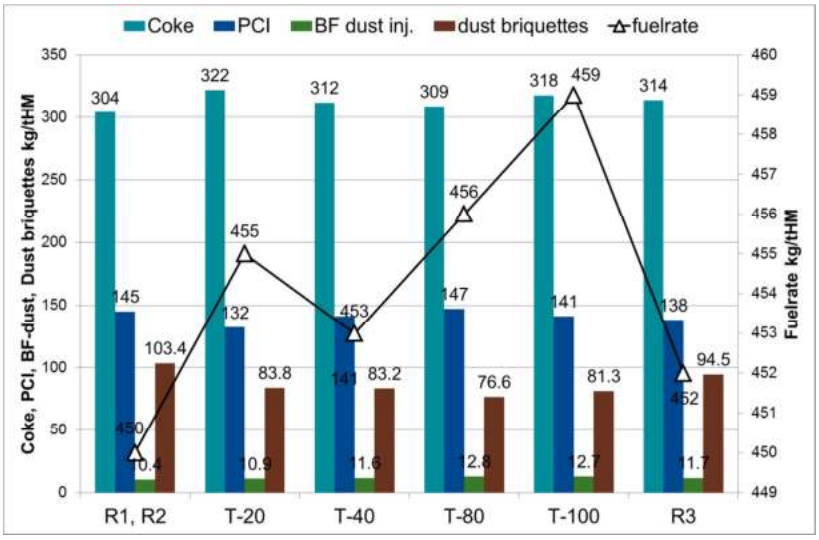



**Figure 196: Charged amounts of coke, PCI, BF dust inj., dust briquettes and total fuel rate**

According to the figure, the highest fuel rate was 459 kg/tHM and is corresponding to the period when the BF was operated with 100% FTC. This period had the second highest coke rate (318 kg/tHM), the highest being in the ramp-up at 20% alternative coke with 322 kg/tHM coke charged. The lowest fuel- and coke rate are both found in the reference periods. Hence, the trend in Figure 196 implies that the fuel rate increases with increased addition of the alternative coke. To have this verified, the different periods should be compared equally i.e. without occasions with stoppages and other disturbances that can have effect on the fuel consumption not related to coke quality.

To get an overview on the characteristics of each trial period and also identify episodes with disturbances, statistical modelling of the process data was conducted as a first step by using the multivariate analytical software SIMCA 14.1. In Figure 197, the blast flow has been plotted over time and it is shown that a significant part of the trial periods, mainly T-20 and T-100, are corresponding to rather unstable BF operation with low production. According to the data evaluation, statistical modelling and observations made by BF operators, these stoppages and disturbances were caused by equipment failures and not by the charged coke. Hence, none of these episodes were included in the condensed periods for MASMOD evaluation, which can be distinguished by the red lines. Mean values of some operational parameters in the condensed MASMOD evaluation periods are summarised in Table 52.

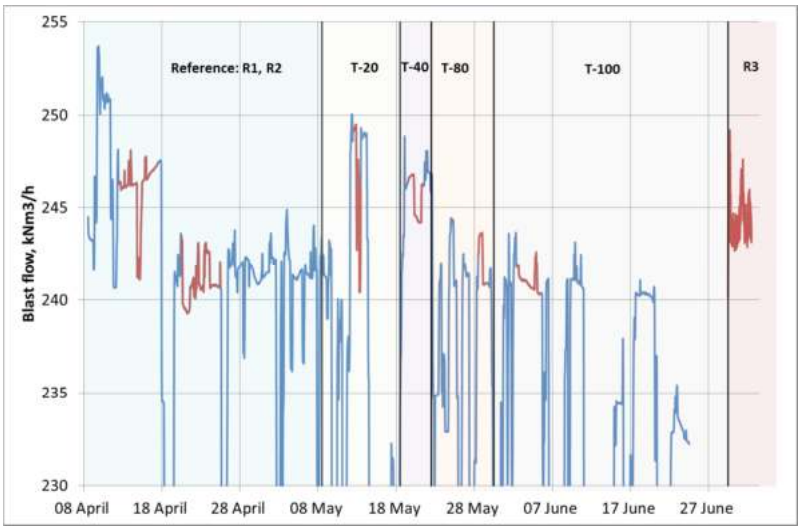



**Figure 197: Blast flow with highlighted test sequences for MASMOD evaluation in red**

**Table 52: Start- and end date of selected evaluation periods and production parameters**

| | R1 | R2 | T-20 | T-40 | T-80 | T-100 | R3 |
|---|---|---|---|---|---|---|---|
| Starting | 04/13 | 04/21 | 05/13 | 05/20 | 05/29 | 06/03 | 06/30 |
| Ending | 04/17 | 04/25 | 05/13 | 05/21 | 05/29 | 06/05 | 07/02 |
| Length (days) | 5 | 5 | 1 | 2 | 1 | 3 | 3 |
| | | | | | | | |
| Production rate (tHM/h) | 279 | 270 | 281 | 278 | 256 | 282 | 276 |
| Coke rate (kg/tHM) | 300 | 300 | 337 | 312 | 306 | 303 | 314 |
| PCI rate (kg/tHM) | 146 | 151 | 108 | 146 | 139 | 152 | 138 |
| Spec. blast ($Nm^3$/tHM) | 883 | 898 | 905 | 889 | 878 | 885 | 914 |
| Blast $O_2$ - tot (%) | 4.50 | 4.50 | 3.86 | 4.50 | 4.50 | 4.50 | 3.45 |
| ηCO (%) | 55.6 | 55.2 | 55.8 | 55.1 | 55.6 | 55.8 | 55.3 |
| Top gas temp. (°C) | 135 | 147 | 94 | 93 | 122 | 139 | 127 |
| Charged water (kg/tHM) | 28 | 25 | 27 | 36 | 24 | 24 | 25 |
| Sprayed water (kg/tHM) | 1 | 5 | 0 | 0 | 1 | 5 | 3 |
| | | | | | | | |
| HM Temp. (°C) | 1480 | 1482 | 1497 | 1475 | 1492 | 1483 | 1479 |
| HM C (%) | 4.68 | 4.65 | 4.69 | 4.65 | 4.75 | 4.70 | 4.63 |
| HM Si (%) | 0.33 | 0.38 | 0.40 | 0.35 | 0.44 | 0.42 | 0.46 |
| HM S (%) | 0.043 | 0.038 | 0.046 | 0.048 | 0.038 | 0.045 | 0.062 |
| Slag rate (kg/tHM) | 167 | 166 | 165 | 167 | 159 | 157 | 162 |
| B2 - $CaO/SiO_2$ | 1.04 | 1.03 | 0.97 | 0.98 | 1.00 | 1.00 | 0.94 |
| B3 – $(CaO+MgO)/SiO_2$ | 1.46 | 1.46 | 1.38 | 0.98 | 1.43 | 1.40 | 1.31 |
| BR (Bells ratio) | 1.32 | 1.32 | 1.25 | 1.25 | 1.29 | 1.27 | 1.18 |
| $K_2O$ yield (%) | 108 | 113 | 140 | 114 | 98 | 97 | 108 |
| Heat losses (MJ/tHM) | 345 | 378 | 365 | 301 | 315 | 324 | 321 |

The duration of each period differs in between the cases. For instance, the duration of the reference periods, R1 and R2, were lasting for 5 days respectively whereas the evaluation of T-20 and T-80 is corresponding to 1 day each. As also revealed by Table 52, the periods are representing different production rates and operational conditions. For instance, the production rate was highest in the T-100 period at 282 tHM/h compared to the lowest found in R2 at 270 tHM/h.

For T-100 and R3, the charging of BOF-slag was less and the limestone addition higher compared to the other cases due to restrictions of the vanadium content in HM at steel plant, see Table 53. The charging of dust briquettes was highest during R1 (115 kg/tHM) compared to the lowest amount in T-100 (69 kg/tHM) and T-80 (69.5 kg/tHM). Steel scrap was only charged in R3, this period together with T-100 corresponds to the highest additions of Manganese (Mn).

**Table 53: Charged amounts of top charged materials in each case**

| | Pellets | BOF slag | Limestone | steel scrap | Dust briquette | Mn source | Unit |
|---|---|---|---|---|---|---|---|
| *Fe-type* | *Hematite* | | | *Met. Fe* | | *Met. Fe & Mn* | |
| R1 | 1329.8 | 42.3 | 19.6 | 0 | 114.6 | 3.3 | kg/tHM |
| R2 | 1341.1 | 42.2 | 24.9 | 0 | 95.4 | 2.3 | kg/tHM |
| T-20 | 1344.4 | 49.7 | 24.1 | 0 | 80.1 | 2.9 | kg/tHM |
| T-40 | 1348.5 | 47.2 | 27.2 | 0 | 80.5 | 2.9 | kg/tHM |
| T-80 | 1351.5 | 44.1 | 29.4 | 0 | 69.5 | 3.1 | kg/tHM |
| T-100 | 1354.2 | 36.5 | 33.4 | 0 | 69.3 | 4.9 | kg/tHM |
| R3 | 1322.2 | 12.0 | 31.8 | 39.3 | 89.6 | 4.9 | kg/tHM |

The MASMOD calculated C-consumption based on charged coke, dust injection (BF flue dust injection), dust briquettes and injected PCI and also the sum for each case are shown in Figure 198 together with the Si content in HM. The lowest C-consumptions are found for the reference cases R3 and R1 with rates of 397 and 398 kg/tHM respectively. The highest rates are found in R2, T-80 and T-100, all having C-consumptions of 402 kg/tHM.

Referring to the heat level, T-100 and T-80 are both corresponding to higher Si contents in HM compared to R2. R3 was shown to have the lowest C-consumption among all cases but still having the highest Si content in HM. This could be explained by the charging of scrap during this period which will decrease the C demand in the process by approximately 8-9 kg/tHM. Hence, the C-consumption in R3 without any scrap additions is expected to be 405 kg/tHM, which then is the highest rate among all cases.

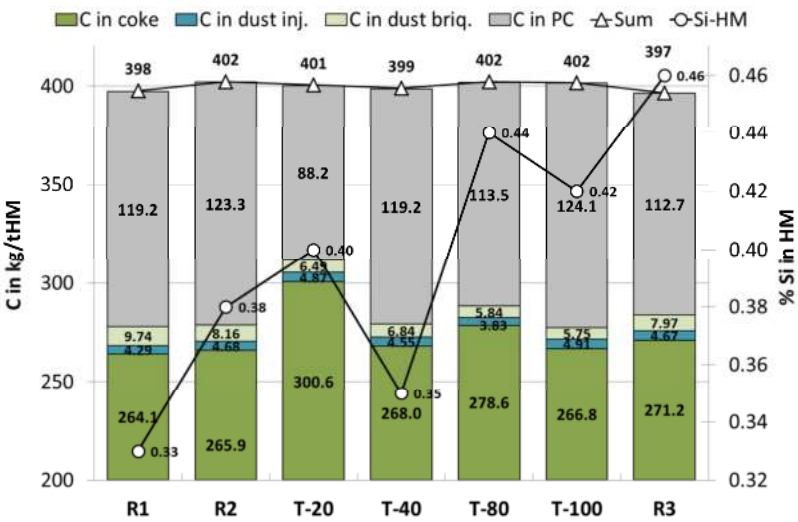



**Figure 198: C-consumption for each case in MASMOD**

A comparison between calculated C-consumption and charged- and injected C for the different cases is shown in Figure 199. The smallest gap between the modelled values from the input amounts can be found for R1 and T-10 with deviations of less than 0.5 kg/tHM. When the C-consumption based on the actual coke rate is higher than needed to close the balances, it is assumed that that the coke reserve is increasing. This seems to be the case at T-40, T-100, R3 and R1 whereas the opposite is shown for the other cases.

However, a difference in calculated C-consumption and input for the shorter periods could also be attributed to a time delay between the process demand of reductants and the charging of burden materials. For shorter periods, coke additions before starting a period might be consumed within the period and hence, the C input by charging and injection becomes lower as this value only considers dumps within that period. This could be the case for T-80 and T-20. In T-40, the reverse case is indicated with higher charged and injected amount of C than theoretically needed by the process.

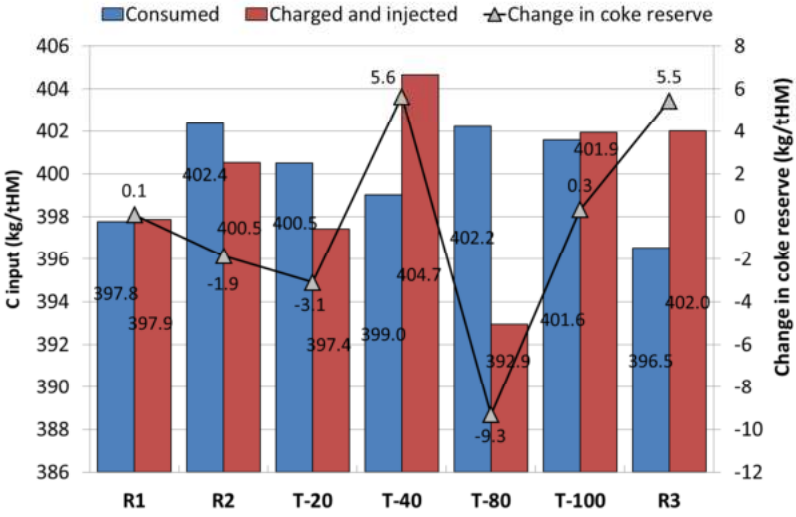



**Figure 199: Comparison of calculated C rate and charged amounts for each calculation cases**

To investigate how the C-consumption differs between the cases if the same heat level is assumed, each period was normalised with respect to the HM analysis and HM temperature of R1. R1 was selected as base case because this period was corresponding to the lowest Si-content in HM and also lowest C-consumption, see Table 52. For R3, the reduced C-consumption by the addition of scrap has been considered in the total consumption to make it more comparable to the other cases.

In Figure 200, a comparison between the original C-consumption i.e. the consumption reported in Figure 198 and the consumption when applying the HM-analysis of R1 is shown for each period. The normalised cases show slightly lower C-consumption than the original cases except for T-40. Largest difference is found in T-20 and T-80 with a reduced C-consumption of 1.5-2 kg/tHM when assuming the same HM-analysis and temperature as in R1. These periods were corresponding to higher Si contents and HM temperatures. In T-40, the Si-content was higher but HM-temperature lower, which might explain why the C-consumption in this period still is higher than in R1. The difference in C-consumption between R2 and T-100 becomes slightly larger compared to the original cases with less consumption in T-100.

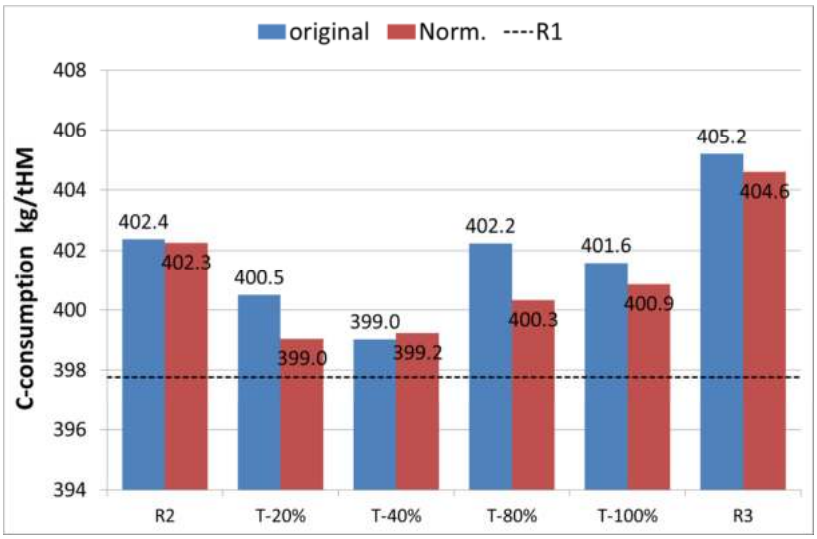



**Figure 200: C-consumption of cases with normalised HM analyses. C equivalents for scrap in R3 included**

To avoid elevated vanadium contents in HM at the steel plant, the BOF-slag was partly substituted by limestone during T-100 and R3. In T-100, the limestone charged to the BF was almost 10 kg/tHM higher than in R2. The effect on C-consumption by limestone additions have been studied for periods T-100 and R2, see Figure 201.

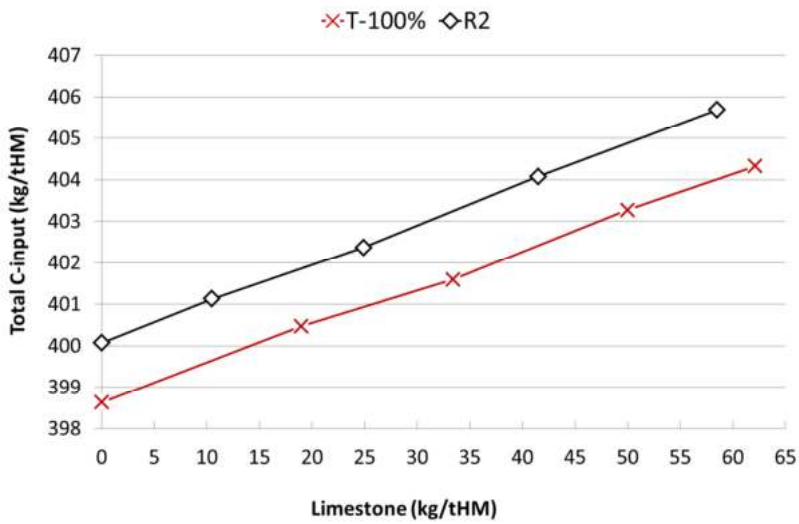



**Figure 201: Effect of limestone addition on the total C-consumption for R2 and T-100**

According to this study, there is a more or less linear relationship between limestone additions and C-consumptions with roughly 1 kg/tHM increased C-consumption per 10 kg limestone added. This means that the total C-consumption in T-100 is expected to be 1 kg/tHM lower if 10 kg/tHM of limestone were substituted with BOF-slag. This added to a normalized heat level between the two cases could mean 2 kg/tHM less C-consumption in T-100 compared to R2.

### Off-gas dust during the FLEXCOKE trial

The amounts (in kg/tHM) of the off-gas dust expelled from the BF top during the full scale FLEXCOKE trial are plotted in Figure 202 and the chemical composition of the off-gas dust is shown in Table 54. The chemical analyses of the off-gas dust have been made by XI (Iso-formation of melt) analyses.

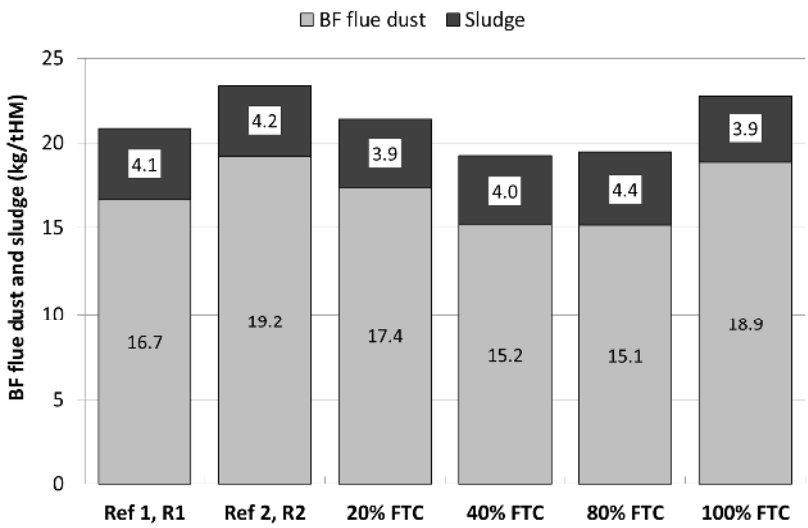



**Figure 202: Off-gas dust during the reference periods, during ramping up and with full operation of FLEXCOKE trial coke (FTC)**

The variation between the reference periods and the 100% operation with FTC is small where 100% FTC shows slightly higher amounts of BF flue dust than R1 and lower amounts than R2. The sludge amount is the lowest during 100% FTC compared to all test periods. In the BF flue dust

(kg/tHM) alkali varies and is slightly higher in both Ref and TFC 80% samples. Fe, CaO and $SiO_2$ decreased when ramping up FTC. The C content in flue dust decreases also with high amount of FTC charged. Fe and C (kg/tHM) increased slightly in sludge ramping up with TFC while CaO and alkali decreases. The $SiO_2$ content in sludge is unchanged.

**Table 54: Chemical analyse in flue dust (top) and in sludge (bottom), in wt.% and in kg/tHM, in samples taken un Ref 1, Ref 2 and TFC 20%, 40% and 80%**

| Flue dust, wt.% | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | **Fe** | **CaO** | **MgO** | **MnO** | **$SiO_2$** | **$Al_2O_3$** | **$K_2O+Na_2O$** | **P2O5** | **S** | **$TiO_2$** | **C LECO** |
| **R1** | 24.1 | 12.4 | 1.55 | 0.51 | 6.34 | 2.54 | 0.11 | 0.07 | 0.56 | 0.36 | 36.5 |
| **R2** | 16.7 | 11.4 | 1.55 | 0.44 | 6.10 | 2.52 | 0.26 | 0.07 | 0.51 | 0.35 | 40.8 |
| **FTC 20%** | 22.6 | 10.7 | 1.44 | 0.41 | 5.96 | 2.40 | 0.10 | 0.08 | 0.46 | 0.32 | 40.8 |
| **FTC 40%** | 24.2 | 11.8 | 1.62 | 0.43 | 5.69 | 2.07 | 0.06 | 0.09 | 0.42 | 0.33 | 37.3 |
| **FTC 80%** | 21.1 | 9.81 | 1.43 | 0.57 | 6.18 | 2.40 | 0.29 | 0.07 | 0.46 | 0.31 | 45.1 |
| **Flue dust, kg/tHM** | | | | | | | | | | | |
| **R1** | 4.03 | 2.08 | 0.26 | 0.09 | 1.06 | 0.42 | 0.018 | 0.012 | 0.09 | 0.06 | 6.10 |
| **R2** | 3.20 | 2.19 | 0.30 | 0.08 | 1.17 | 0.48 | 0.050 | 0.013 | 0.10 | 0.07 | 7.84 |
| **FTC 20%** | 3.94 | 1.86 | 0.25 | 0.07 | 1.04 | 0.42 | 0.017 | 0.014 | 0.08 | 0.06 | 7.12 |
| **FTC 40%** | 3.68 | 1.79 | 0.25 | 0.07 | 0.87 | 0.31 | 0.009 | 0.014 | 0.06 | 0.05 | 5.66 |
| **FTC 80%** | 3.19 | 1.48 | 0.22 | 0.09 | 0.94 | 0.36 | 0.044 | 0.011 | 0.07 | 0.05 | 6.82 |
| **Sludge, wt.%** | | | | | | | | | | | |
| | **Fe** | **CaO** | **MgO** | **MnO** | **$SiO_2$** | **$Al_2O_3$** | **$K_2O+Na_2O$** | **P2O5** | **S** | **$TiO_2$** | **C LECO** |
| **R1** | 33.1 | 9.73 | 1.53 | 0.27 | 5.12 | 2.20 | 0.17 | 0.17 | 0.52 | 0.29 | 24.6 |
| **R2** | 32.2 | 10.1 | 1.56 | 0.24 | 5.11 | 2.25 | 0.16 | 0.19 | 0.42 | 0.28 | 25.4 |
| **FTC 20%** | 34.5 | 8.68 | 1.67 | 0.22 | 5.36 | 2.35 | 0.26 | 0.22 | 0.48 | 0.27 | 23.4 |
| **FTC 40%** | 31.2 | 7.88 | 1.64 | 0.19 | 5.14 | 2.34 | 0.10 | 0.20 | 0.35 | 0.25 | 29.3 |
| **FTC 80%** | 32.1 | 7.90 | 1.51 | 0.22 | 5.09 | 2.29 | 0.12 | 0.16 | 0.33 | 0.26 | 28.9 |
| **Sludge, kg/tHM** | | | | | | | | | | | |
| **R1** | 1.35 | 0.40 | 0.06 | 0.01 | 0.21 | 0.09 | 0.007 | 0.007 | 0.02 | 0.01 | 1.01 |
| **R2** | 1.35 | 0.42 | 0.07 | 0.01 | 0.21 | 0.09 | 0.006 | 0.008 | 0.02 | 0.01 | 1.06 |
| **FTC 20%** | 1.35 | 0.34 | 0.07 | 0.01 | 0.21 | 0.09 | 0.010 | 0.009 | 0.02 | 0.01 | 0.91 |
| **FTC 40%** | 1.26 | 0.32 | 0.07 | 0.01 | 0.21 | 0.09 | 0.004 | 0.008 | 0.01 | 0.01 | 1.19 |
| **FTC 80%** | 1.40 | 0.34 | 0.07 | 0.01 | 0.22 | 0.10 | 0.005 | 0.007 | 0.01 | 0.01 | 1.26 |

Conclusions

No negative effects by operating with the alternative coke could be found based on the MASMOD evaluation and by observations made by BF operators during the trial. The period with 100% alternative coke (T-100) showed lower or comparable C-consumption than during reference periods (R2-R3) with standard coke despite the use of more limestone in T-100 and higher heat level compared to R2.

The off-gas dust did not increase during the FLEXCOKE trial compared to reference operation. Sludge amount seem to decrease when operating with 100% FTC. In order to fully investigate the effects on the off-gas dust, C-consumption etc., a longer trial period is of advantage.

Exploitation and impact of the research results

As described before the use of the FLEXCOKE trial coke, FTC, was successful and no harmful effects on the coke plant and in the BF could be seen using 5% thermal coal in the coal blend. Coke quality data such as CSR/CRI and Micum showed that the coke strength and reactivity are only slightly affected. To fully investigate the effect of FTC on coke plant and in BF operation a longer time frame of production and operation are needed, therefore a new test period of 10 weeks is planned to be tested at SSAB after the project. During the test period the stability of the "dead man" will be monitored as well as other parameters as the energy consumption, possibly losses to off-gas dust etc.

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# 8 LIST OF ACRONYMS AND ABBREVIATIONS

2D Two dimensions

3D Three dimensions

BF Blast Furnace

BTX Xylene

C Carbon

C31 Campaign 31

C32 Campaign 32

CCD Charge-Coupled Device

CO Carbon monoxide gas

$CO_2$ Carbon dioxide gas

CRI Coke Reactivity Index

CSR Coke Strength after Reaction

daf dry ash free

DRR Direct Reductant Rate

EBF Experimental Blast Furnace

EDS Energy Dispersive Spectroscopy

Eta CO, ηCO Gas utilisation, gas efficiency

FSI Free Swelling Index

FTC FLEXCOKE trial coke

g grams

$H_2$ Hydrogen gas

HGI Hardgrove Grindability Index

HM Hot metal

HV High volatile

Hz Hertz

IGP Internal Gas Pressure

ISO International Standard Organisation

l litre

Lc Graphitisation degrees, i.e. height of graphite like crystal

LKAB Luossavaara Kiirunavaara AB

LOM Light Optical Microscope

LV Low volatile

m meter

$M_{40}$ Micum 40

$M_{10}$ Micum 10

MASMOD Heat- and mass model for the BF

MEFOS Swerea MEFOS AB

min minute

mm millimeter

| | |
|---|---|
| MPS | Mean Particle Size |
| MV | Medium volatile |
| $N_2$ | Nitrogen gas |
| $O_2$ | Oxygen gas |
| P | Phosphorus |
| PC | Pulverised Coal |
| PCI | Pulverised Coal Injection |
| Pet coke | Petroleum coke |
| RAFT | Calculated Adiabatic Flame Temperature |
| Ref | Reference |
| RFCS | Research Fund for Coal and Steel |
| RW | Raceway |
| SC | Standard Coke |
| SEM | Scanning Electron Microscope |
| SIMCA | Multivariate data analysis |
| SSAB | SSAB EMEA AB |
| Tata | Tata Steel UK |
| TC1 | Test Coke 1 |
| TC2 | Test Coke 2 |
| Temp | Temperature |
| TGA | Thermo Gravimetric Analyser |
| TRZ | Thermal Reserve Zone |
| tk SE | thyssenkrupp Steel Europe AG |
| TW | Torrefied wood |
| UK | United Kingdom |
| VM | Volatile matter |
| XI | Iso-formation of melt (chemical analyse) |
| XRD | X-ray Diffraction |
| XRF | X-ray Fluorescence (chemical analyse) |

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## 11 APPENDICIES

### Appendix 1: Overview table of DMT carbonisation tests in DMT 500 kg oven with movable walls, first trial

| Test No. | Unit | 1 | 2 | 2a | 3 | 4 | 5 | 7 | 8 | 9 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|---|
| LV coal | % | 40 | 40 | 40 | 40 | 40 | 40 | 40 | 40 | 40 | 40 |
| MV coal | % | 32 | 25 | 30 | 30 | 30 | 29 | 29 | 29 | 30 | 30 |
| HV coal | % | 28 | 25 | 25 | 25 | 27 | 25 | 25 | 25 | 27 | 27 |
| Thermal coal | % | | 10 | 5 | | | | 3 | 3 | 3 | |
| Pet coke | % | | | | | | 3 | 3 | | | |
| Anthracite | % | | | | 5 | | | | 3 | | 3 |
| Charcoal | % | | | | | 3 | 3 | | | | |
| Moisture | % | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 |
| Bulk density (wet) | kg/m3 | 798 | 799 | 792 | 796 | 783 | 783 | 799 | 799 | 804 | 804 |
| Bulk density (dry) | kg/m3 | 727 | 727 | 721 | 724 | 713 | 713 | 727 | 727 | 732 | 732 |
| Chamber width | mm | 450 | 450 | 450 | 450 | 450 | 450 | 450 | 450 | 450 | 450 |
| Heating flue temperature | °C | 1200 | 1200 | 1200 | 1200 | 1200 | 1200 | 1200 | 1200 | 1200 | 1200 |
| Coking time | h | 17.5 | 17.4 | 17.7 | 18.0 | 17.6 | 17.4 | 17.4 | 17.5 | 17.5 | 17.8 |
| Carbonisation rate | °C/min | 2.8 | 3.1 | 3.1 | 2.5 | 2.8 | 2.7 | 3.2 | 2.6 | 3.0 | 2.4 |
| Coke end temperature | °C | 1057 | 1057 | 1053 | 1056 | 1058 | 1060 | 1058 | 1056 | 1059 | 1057 |
| Coke yield | % | 78.5 | 76.8 | 77.5 | 79.6 | 78.9 | 78 | 78.1 | 77.2 | 77.9 | 78.9 |
| Max. internal gas pressure | mbar | 27 | 9 | 8 | 9 | 20 | 8 | 11 | 7 | 19 | 9 |
| Max. wall load | mbar | 21 | 29 | 14 | 20 | 18 | 8 | 12 | 23 | 18 | 24 |
| Shrinkage | mm | -9.1 | -10.7 | -13.5 | -12.8 | -5.3 | -8.5 | -6.9 | -10.0 | -5.5 | -11.6 |
| Coke mean size | mm | 71.9 | 67.7 | 69.5 | 77.4 | 69.0 | 67.6 | 68.1 | 72.3 | 70.0 | 73.0 |
| M40 | % | 84.3 | 77.8 | 78.3 | 76.9 | 80.2 | 77.5 | 79.3 | 78.4 | 80.2 | 78.5 |
| M10 | % | 6.4 | 10.4 | 9.1 | 10.0 | 7.5 | 9.9 | 8.3 | 9.8 | 7.5 | 8.8 |
| I40 | % | 61.1 | 53.9 | 55.8 | 51.2 | 58.2 | 52.8 | 56.5 | 55.0 | 56.7 | 53.4 |
| I10 | % | 21.3 | 27.9 | 23.7 | 28.4 | 22.1 | 25.7 | 24.0 | 26.2 | 23.2 | 25.0 |
| **CRI** | **%** | **22.2** | **28.7** | **24.8** | **21.9** | **23.9** | **25.3** | **24.7** | **24.4** | **22.8** | **21.7** |
| **CSR** | **%** | **67.8** | **53.7** | **60.7** | **63.9** | **64.1** | **60.8** | **59.7** | **62.5** | **63.9** | **63.8** |

**Appendix 2: Overview table of DMT carbonisation tests in DMT 500 kg oven with movable walls, second trial**

| Test No. | Unit | I | II | III | IV | V |
|---|---|---|---|---|---|---|
| LV coal | % | 40 | 40 | 40 | 40 | 40 |
| MV coal | % | 32 | 30 | 31 | 30 | 30 |
| HV coal | % | 28 | 27 | 27 | 25 | 27 |
| Torrefied wood, medium temperature | % | | 3 | 2 | | |
| Anthracite | % | | | | 5 | |
| Charcoal | % | | | | | 3 |
| Moisture | % | 9.0 | 9.0 | 9.0 | 9.0 | 9.0 |
| Bulk density (wet) | kg/m3 | 796 | 804 | 805 | 800 | 778 |
| Bulk density (dry) | kg/m3 | 724 | 732 | 734 | 728 | 708 |
| Chamber width | mm | 450 | 450 | 450 | 450 | 450 |
| Heating flue temperature | °C | 1200 | 1200 | 1200 | 1200 | 1200 |
| Coking time | h | 17.5 | 17.5 | 17.5 | 18.0 | 17.5 |
| Carbonisation rate | °C/min | 2.8 | 2.9 | 3.3 | 2.4 | 3.1 |
| Coke end temperature | °C | 1060 | 1056 | 1056 | 1058 | 1061 |
| Coke yield | % | 78.0 | 77.3 | 77.5 | 80.0 | 78.3 |
| Max. internal gas pressure | mbar | 15 | 5 | 17 | 5 | 8 |
| Max. wall load | mbar | 13 | 14 | 18 | 10 | 13 |
| Shrinkage | mm | -5.3 | -7.6 | -5.1 | -9.3 | -7.1 |
| Coke mean size | mm | 68.3 | 66.8 | 68.4 | 77.7 | 67.2 |
| M40 | % | 79.0 | 77.4 | 80.1 | 74.8 | 77.5 |
| M10 | % | 8.4 | 11.1 | 8.5 | 12.0 | 9.8 |
| I40 | % | 58.0 | 51.3 | 57.2 | 48.0 | 52.4 |
| I10 | % | 23.4 | 28.0 | 24.1 | 29.6 | 26.1 |
| **CRI** | **%** | **20.8** | **25.3** | **23.7** | **23.2** | **23.1** |
| **CSR** | **%** | **68.8** | **61.0** | **62.8** | **61.0** | **62.5** |

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Main objective of the project "Flexible production of coke using alternative coals – effects on coke properties under blast furnace conditions"- FLEXCOKE - was to increase the flexibility using alternative carbon sources in safe cokemaking and to produce coke of a quality that meets the requirements for stable blast furnace (BF) operation aiming to reduce costs for hot metal production with similar/lower carbon input and CO2 emission. This is of importance with a global market characterised by limited availability and high costs for high quality coking coals. The uniqueness of the research was to link alternative C-containing material in cokemaking to coke properties and behaviour in BF operation with use of new innovative test methods in combination with samples from BF testing.

Properties of alternative C-containing materials/coals of economic/technical interest for cokemaking were collected. A test method for innovative test regimes has been developed and used aiming for a method with correlation of coke properties with the actual BF. Selected coal blend/C-containing materials effects on cokemaking, coke properties in standard and innovative coke tests and in the BF have been identified. Microstructure evaluation during standard and innovative coke tests were compared with samples from the LKAB Experimental BF (EBF▪). Industrial alternative coke production and BF testing gave valuable information of the effects using alternative materials. High temperature characteristics and behaviour were studied with solid sample evaluation from the EBF▪ high temperature area. Operational test data from the EBF▪ and SSAB coke plant and industrial BF using alternative coke have been evaluated.

*Studies and reports*

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