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Bio-oil Production from Biomass: Steps toward Demonstration

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ABSTRACT: Metso, UPM, Fortum, and VTT have developed the world's first integrated bio-oil production concept to provide an alternative to fossil fuels. The consortium has constructed an up to 7 tons/day bio-oil production pilot unit, which uses a bubbling fluidized-bed (BFB) pyrolysis reactor integrated with a conventional fluidized-bed boiler. Proof-of-concept has been carried out; close to 90 tons of bio-oil has been produced from sawdust and forest residues at high availability. Around 40 tons of bio-oil has been combusted in Fortum's 1.5 MW district heating plant in Masala, Finland, with high efficiency. Flue gas emissions were close to those of heavy fuel oil, at 4% O₂, CO emissions ranged from 0 to 10 ppm and NO_x emissions ranged from 300 to 400 ppm. Organic compounds were under 5 mg m⁻³ N⁻¹, and particulate emissions were in the range of 150–200 mg m⁻³ N⁻¹. No odor emissions occurred. Development of the concept has been supported by experimental work on fast pyrolysis at VTT. This paper presents the recent results from the piloting project covering the whole chain from feedstock processing to bio-oil combustion, including the quality control system with online gas and liquid analyzers. The research supporting the pilot project, from various laboratory-scale units to systematic analytical development, is discussed, and the potential for market introduction of the new technology in forest product industries in western Europe and North America is described.

■ INTRODUCTION

Fast pyrolysis involves the rapid (1–2 s), thermal (about 500 °C) decomposition of biomass in an inert atmosphere to produce bio-oil, gases, and char. The yield of the main products of fast pyrolysis, i.e., liquid-phase organics, is expressed on the basis of dry and ash-free feed (Figure 1).

Fast pyrolysis of biomass was first proposed for alternative fuel production some 40 years ago in the U.S.A.¹ However, the work at the University of Waterloo² was especially instrumental in starting process development of fast pyrolysis. Several process development unit (PDU) and small pilot-plant scale technologies were developed during the 1990s, first in North America and later also in Europe. The first commercial plants to produce chemicals³ were built during the current decade. However, no real fuel markets have yet been established for this biofuel. Figure 2 presents some key developments in fast pyrolysis technology.

In Scandinavia, forest residues are the most feasible feedstock for pyrolysis.⁴ These residues contain extractives, which yield a second liquid phase. This is both an opportunity in terms of byproduct recovery and a challenge in terms of using both phases as fuel. Agro-biomasses are a more challenging feedstock for energy use because of the high alkali metals and nitrogen content of the resulting oil. They also yield more water during pyrolysis, causing phase instability.

The two key challenges here are the relatively high cost of production (per heat unit of product) and relatively low fuel quality. Production costs can be lowered by integration of fast pyrolysis with a fluidized-bed boiler. To address the second challenge, improvement of product quality for fuel use and development of critical stages throughout the use chain are being pursued.

The integration of a fluidized-bed boiler and fast pyrolysis is being currently developed by an industrial consortium^{5–8} to enable bio-oil to be used for the production of heat for both industry and communities. In addition, work to improve product quality is aimed at achieving higher value uses in the future. Research at VTT aims at supporting the industrial development of these efforts.

The aim of this paper is to provide new information about pyrolysis oil production in pilot scale and the means for quality control during oil production. An example on the controllability of the integrated process is provided. A comparison of oil quality from pilot and PDU scale is provided, which is important for scale-up. New experimental data on online water analyzers will be provided.

■ EXPERIMENTAL RESEARCH AT THE PDU SCALE (20 KG/H)

Pyrolysis Oil Production. VTT's PDU employs fluidized-bed reactors (Figure 3).⁴ Both circulated fluidized-bed (CFB) and bubbling fluidized-bed (BFB) reactors have been tested. The feedstock is fed to the reactor by a screw feeder. The pyrolysis temperature is about 480–520 °C, and the residence time for pyrolysis vapors is about 0.5–2 s. The majority of char particles are removed from the hot stream of product gases and vapors by one or two cyclones before entering recovery. The CFB reactor uses two cyclones, and the BFB reactor

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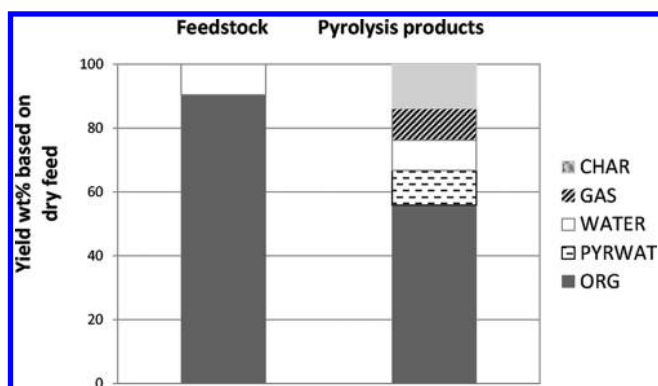


Figure 1. Origin of water in fast pyrolysis liquids.

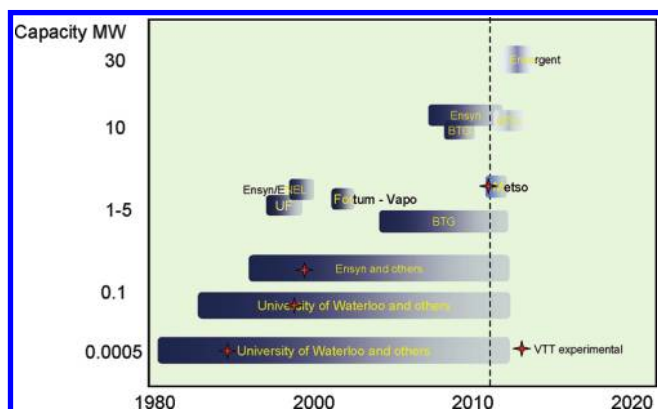


Figure 2. Development of fast pyrolysis: primary liquid production.

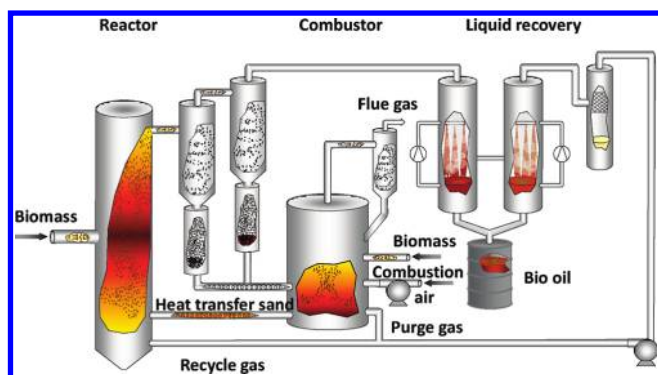


Figure 3. VTT PDU unit, 20 kg/h.

employs one cyclone. The product vapors are condensed in conventional liquid scrubbers, where the product liquid is used as a cooling agent. Typical product yields from pine wood are 62 wt % organic liquids, 12 wt % product water (chemically dissolved in organic liquids), 14 wt % char, and 12 wt % noncondensable gases. The unit has a total test run production time during 1996–2011 of 3100 h, with 42 tons of liquids produced. The availability (experimental time realized/planned) of the unit has been very good (Figure 4). Typical product yields from various feedstocks are shown in Figure 5. Organic yields have been highest with wood feedstocks and lowest with agro-biomass containing high amounts of ash.⁴

VTT has been developing an integrated concept, in which fast pyrolysis is integrated with a fluidized-bed boiler (Figure 6). Pyrolysis section is

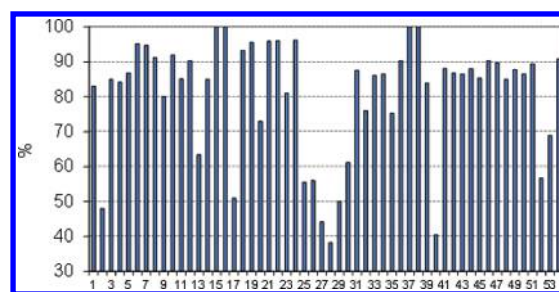


Figure 4. PDU availability in test runs with a duration of 3–4 days.

similar to stand-alone pyrolyzers. However, hot sand from the boiler provides heat for the pyrolysis reactor. Byproducts from pyrolysis, char, and off-gases are used as fuel in the boiler. Using the pyrolysis byproduct as fuel to replace boiler fuel improves the energy efficiency, because the byproducts are used in the production of power and heat.

Experimental work on fast pyrolysis has been targeted at supporting this concept. The integration is considered to offer the following technical and economic advantages: (i) high overall efficiency compared to stand-alone pyrolysis concepts, (ii) lower investment costs because of the eliminated need for a separate char combustor (effect on the main boiler cost is considered small), (iii) lower operating costs than stand-alone pyrolysis because of reduced manpower, (iv) operating flexibility because of full exploitation of the byproduct in main boiler, and (v) easier operation because of the eliminated need for byproduct char combustion in a small sub-optimal boiler (as typically required to meet plant energy requirements).

Fuel Oil Quality. The chemical composition of fast pyrolysis oil determines its physical properties⁹ and behavior during handling, storage, and combustion. A simple characterization method was developed^{10,11} to monitor key changes in pyrolysis oil quality. On the basis of the method, the main chemical compound groups of fast pyrolysis oil are carboxylic acids (4–6 wt %), carbonyl compounds (15–20 wt %), mainly cellulose-derived sugar-type compounds, “sugars” (25–35 wt %), water (20–30 wt %), and mainly lignin-derived water-insolubles (20–25 wt %).

For fast pyrolysis oil to be a viable fuel, it must have sufficient shelf-life stability. However, aging¹² reactions during storage of the oil, especially when heated, cause a considerable increase in viscosity. The main chemical changes during storage are a decreased carbonyl compound content and an increased water-insoluble fraction.^{12–14} The properties of the liquid need to remain constant during typical storage, i.e., at least 6 months at 15 °C. There are various methods of stability improvement, including dilution with alcohols,^{12,15} reduced pressure distillation,¹⁶ and hydrotreatment. Research in this area is ongoing.

EXPERIENCES FROM PILOT PROCESSING

Earlier Cooperation. VTT has participated in four projects involving pilot-scale development of fast pyrolysis: WFPP, Union Fenosa, Spain, Ensyn, ENEL, Italy, Foresteria, Fortum, Finland, and Integrated Pyrolysis, Metso, UPM, Fortum, VTT, Finland.

Metso's Integrated Pyrolysis Pilot Plant. Metso has built the world's first integrated pyrolysis pilot plant^{5–8} at the Metso R&D Centre in Tampere, Finland. An up to 2 MW_{fuel} fast pyrolysis unit has been integrated with Metso's 2–4 MW_{th} fluidized-bed boiler. In the concept, the pyrolysis pilot uses the hot sand from the fluidized-bed boiler as a heat source. Pyrolyzed vapors are condensed as bio-oil, and the remaining noncondensable gases and solids, including sand and fuel char, are returned to the

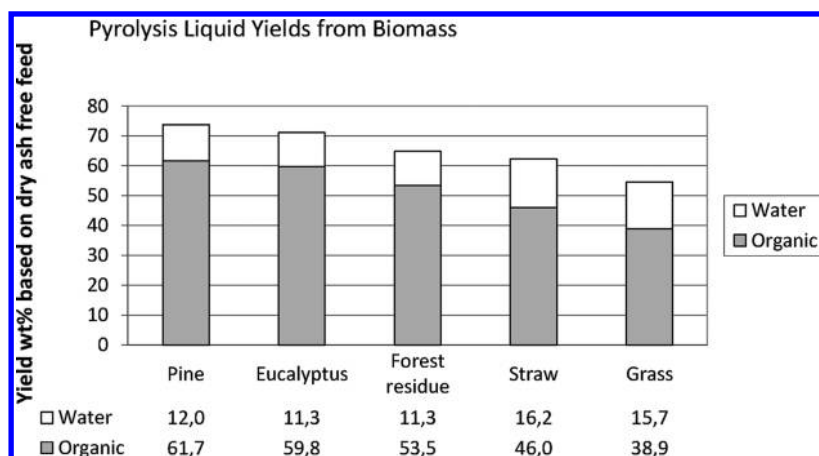


Figure 5. Organic yields from various biomasses in PDU.

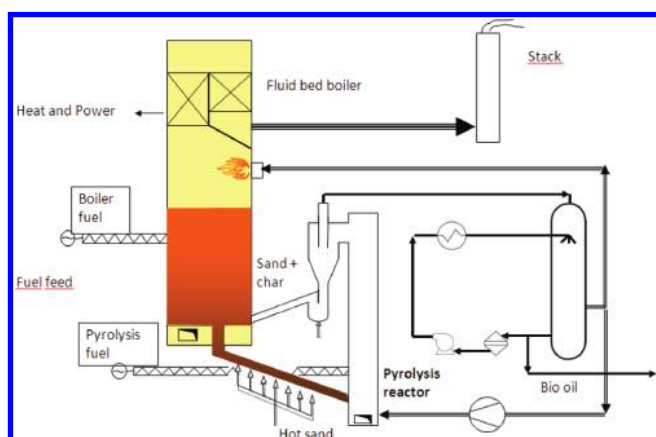


Figure 6. Example of the integrated pyrolysis.

Table 1. Metso's Pyrolysis Feedstocks

		forest residue	pine
moisture, as received (DIN 51718)	wt %	10.9	10.8
sulfur, dry basis (CEN/TS 15289)	wt %	0.03	ND
ash, dry basis (CEN 335)	wt % (db)	1.5	0.6
volatiles, dry basis (DIN 51720)	wt % (db)	80.2	83.1
carbon (C), dry basis (ASTM D5373)	wt % (db)	51.1	50.7
hydrogen (H), dry basis (ASTM D5373)	wt % (db)	6.0	5.9
nitrogen (N), dry basis (ASTM D5373)	wt % (db)	0.4	0.1
higher heating value, dry basis (DIN 51900)	MJ/kg	20.6	20.3
lower heating value, dry basis DIN 51900)	MJ/kg	19.3	19.0
lower heating value, as received (DIN 51900)	MJ/kg	16.9	16.7

fluidized-bed boiler. In the boiler, char and off-gases are combusted to produce heat.

Feedstock Processing. Fuel preparation is an essential part of the process. The main feedstocks for the pilot plant were forest residues and sawdust. The feedstock was dried at 40–50 °C to about 10% moisture and ground to below 5 mm particle size. Two feedstock compositions are presented in Table 1.

Pilot Plant Operation and Bio-oil Production. The first bio-oil batches were produced during the very first test run. More than

90 tons of bio-oil have since been produced from pine and forest residues. The longest continuous test run lasted 9 days and was terminated because of the lack of feedstock. The main focus has been on the development of the fast pyrolysis reactors and minimizing solids content in the product oil. Different measurement technologies for bio-oil moisture analysis were tested during the test runs. Bio-oil samples were analyzed in an on-site laboratory by means of Karl Fischer (KF) titration as a reference. In parallel, an automatic online KF sample analyzer was used as well as a Metso microwave consistency transmitter (MCA) mounted in-line. The online KF titrator is an Applikon Analytical/ADI 2040 process analyser. It is a continuous titrator, which takes 0.5 mL of sample 1–2 times an hour. According to the standard American Society for Testing and Materials (ASTM) E203, the solvent is chloroform/methanol (1:3). The titration reagent is KF Reagents 5, which is 2-methoxy ethanol with an anion of the alkyl sulfurous acid, iodine, and pyridine. The equipment works well when the solids content of the sample is reasonable, approximately below 0.5 wt %. Filters (i.e., 225 µm) for solids were useful but needed maintenance. The continuous MCA is based on microwave technology and is propriety technology by Metso (Kajaani, Finland). The results from both methods correlated with laboratory water measurements (Figure 7).

In comparison to a stand-alone pyrolysis unit with a non-optimal small boiler for combustion of the pyrolysis byproduct (char and pyrolysis gases), the integrated concept is easy and smooth to operate and has a high efficiency. For pyrolysis, having a steady and smooth flow of input energy (i.e., boiler sand) is a considerable advantage from the operator point of view.

Figure 8 depicts an example of pilot operation. The quench liquid has high initial water content, but during the first day as the feed moisture is decreased, a constant level of product water of about 25 wt % is achieved. As a result of stable pilot plant operation, product water is constant as long as feed moisture is constant. After day 3, when feed moisture is gradually increased from 8 to 12 wt %, product liquid moisture increases. Three different wood fuels were tested during the shown period.

Controllability of the integrated concept has been excellent. One of the critical features of pyrolysis is the ability to maintain a constant reactor temperature during operation. An example of the controllability of the integrated concept is shown in Figure 9. Because the pyrolysis reactor is integrated directly with the

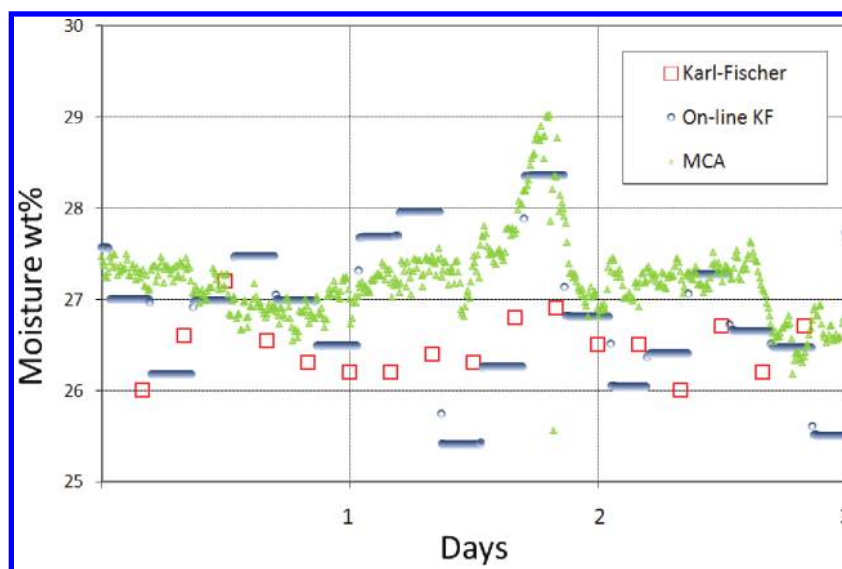


Figure 7. MCA for in-line water content, results from pilot plant. KF, Karl Fischer titration in laboratory; On-line KF, online Karl Fischer analyzer; MCA, microwave consistency transmitter.

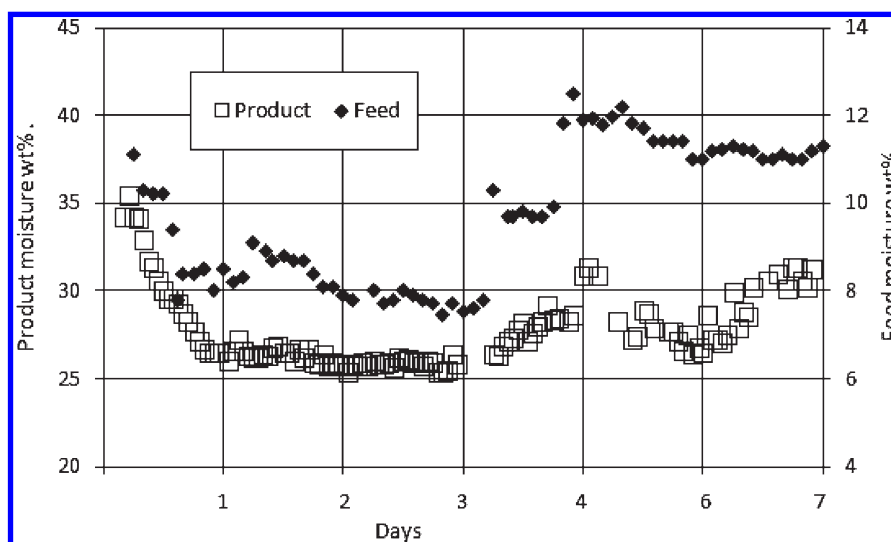


Figure 8. Example of bio-oil–water content during a 6 day period.

boiler, the effect of potential boiler temperature variations on the pyrolysis temperature is a key area of interest. Boiler temperature disturbances can be seen in Figure 9. As the figure shows, rapid changes in the boiler sand temperature are smoothly compensated by pyrolysis temperature control.

The pilot plant availability has improved continuously during the development phase (Figure 10).

Quality Control. Quality monitoring during bio-oil production ensures the production of a uniform quality product and helps minimize potential production problems. Feedstock moisture is the main parameter monitored. Excessive (>10 wt %) feedstock moisture may cause the formation of a multiphase liquid product. A first indication of expected organic liquid yields from any biomass may be obtained by analyzing the volatiles/fixed carbon of the feedstock to be pyrolyzed (Figure 11).

The main product gases, carbon monoxide and carbon dioxide, can be monitored by an online gas chromatograph.

The main liquid quality parameters to be monitored are water and solids content (Figure 12). An increase in water may indicate a change in feedstock moisture or processing conditions or the presence of catalytic reactions. An increase in solids content of the liquid may indicate a failure in cyclone operation or a blockage. For laboratory measurements, liquid samples are recommended to be taken from the condensers at fixed intervals.

Chemical characterization is carried out on an as-needed basis. For quality monitoring, water and water-insolubles contents typically provide sufficient product composition information for control purposes. An increase in water-insolubles is related to factors such as polymerization. It increases the viscosity of the oil and has an effect on its combustion properties. Additional information is easily obtained by analyzing the water-soluble fraction for acids by capillary electrophoresis (CE), with the total acid number (TAN),¹⁷ for “sugars” (total amount of carbohydrates) by BRIX,¹¹ or for carbonyls by titration.¹⁴ The oil

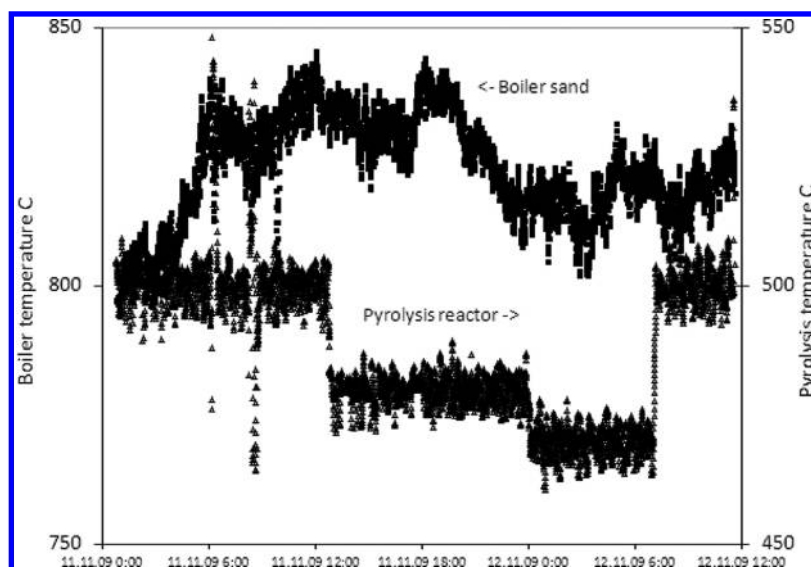


Figure 9. Smooth reactor temperature control.

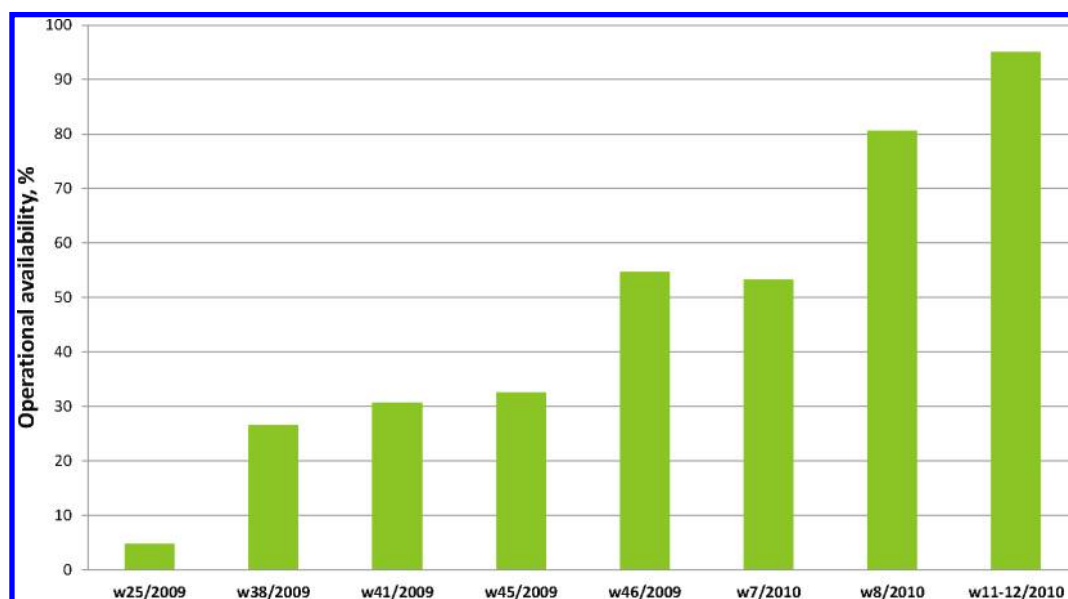


Figure 10. Integrated pyrolysis pilot plant availability (weeks/year).

quality of the pilot plant has been similar to that of the PDU by VTT (Figure 13), which is very important in scaling-up.

■ PYROLYSIS OIL USE FOR HEATING

Bio-oil Combustion for District Heating. Bio-oil was combusted in Fortum's 1 MW district heating plant in Masala, Finland. The existing burner was replaced with a new bio-oil burner consisting of a modified heavy fuel oil burner. The total amount of bio-oil combusted has been about 40 tons. Two main topics for the tests were the overall functionality of the bio-oil receiving, storing, and pumping system and the function of the burner. Both topics were successfully addressed. The receiving system and oil tank were located outside of the boiler building. The system worked well, despite the outside temperatures, which varied from -20 to $+10$ °C during the test periods. As a result,

good reliability and a satisfactory turn-down ratio of 1:3 were achieved. Flue gas emissions were close to those of heavy fuel oil, at 4% O_2 , CO emissions ranged from 0 to 10 ppm and NO_x emissions ranged from 300 to 400 ppm. Organic compounds were under $5 \text{ mg m}^{-3} \text{ N}^{-1}$, and particulate emissions were in the range of $150\text{--}200 \text{ mg m}^{-3} \text{ N}^{-1}$. No odor emissions occurred.

■ STANDARDS AND NORMS

Bio-oil differs from conventional liquid fuels and must therefore overcome both technical and marketing hurdles prior to its acceptance in the market. To standardize bio-oil quality in the liquid fuels market, specifications are needed. The first bio-oil burner fuel standard in ASTM D7544 was approved in 2010.¹⁸ CEN standardization has been pushed forward in Europe, although there is no formal fuel specification. Fast pyrolysis pilot

plants for fuel applications producing tons of bio-oil are in operation, and commercial plants are under design. Bio-oil transportation by water and land is increasing in volume, leading to a need for specifications and support documentation. A comprehensive Material Safety Data Sheet is required, backed by independent testing and certification. This work is currently under way under IEA Pyrolysis Task 34.¹⁹

DEMONSTRATION AND FUTURE ASPECTS

Initial development of fast pyrolysis of biomass can be traced back to the development program by Occidental Petroleum¹ in

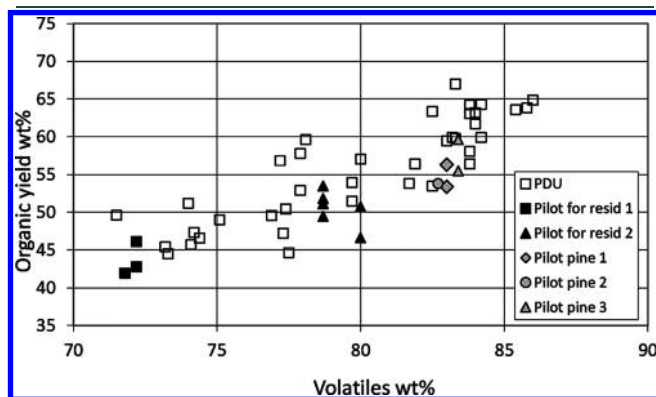


Figure 11. Organic yield as a function of volatiles.

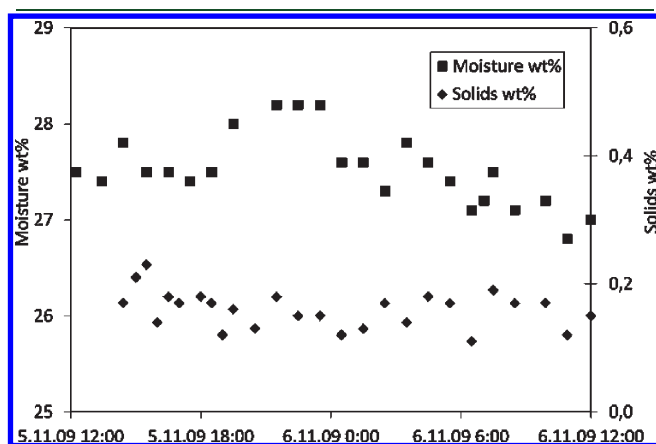


Figure 12. Product quality follow-up.

the early 1970s in the U.S. However, the most important development work in this field is indebted to the pioneering work of Prof. Scott and co-workers at the University of Waterloo, Canada, on whose work (e.g., ref 2 and numerous subsequent publications) our modern understanding of fast pyrolysis is largely based. Another important development initiated by the University of Western Ontario eventually led to the establishment of Ensyn Technologies.³ Considerable early fundamental work was also carried out at the National Renewable Energy Laboratory (NREL) in the U.S.²⁰ In Europe, development work initiated at the University of Twente, Enschede, The Netherlands, has led to process development at the Biomass Technology Group (BTG).^{21,22}

A few companies are currently pushing to commercialize bio-oil for energy applications: Ensyn Technologies/Envergent,^{23,24} Karlsruhe Institute of Technology (KIT)²⁵ with its partners, BTG,^{21,22} and Metso^{5–8} with its partners, which have probably the most advanced initiatives aimed at larger scale operations.

VTT and its partners have identified considerable potential for the deployment of fast pyrolysis in industries with established infrastructures, such as the pulp and paper and mechanical wood processing industries in Europe²⁶ and North America.²⁷ Integration of fast pyrolysis and forest industry boilers is technically viable, and the opportunity is considered industrially relevant. The first uses for the bio-oil would probably be in replacing fuel oils in industrial ovens and boilers. However, a longer term goal would be to produce co-feedstock for mineral oil refineries for the production of transportation fuels and petrochemical feedstocks. This approach has been developed, for example, within the European Union (EU)'s recently completed BIOCOUP project.²⁸

Phased construction of biofuel capacity may be a technically low-risk approach to build new second-generation transportation biofuel capacity. Technically less demanding uses for bio-oil may

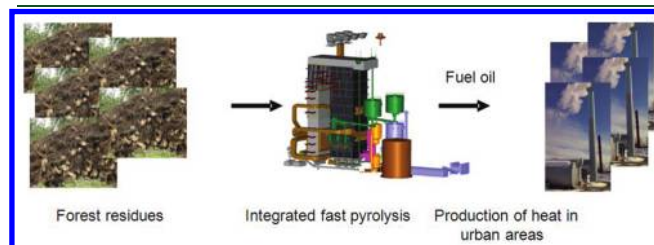


Figure 14. Phased implementation of the biofuel industry based on integrated pyrolysis: the first demonstration plant.

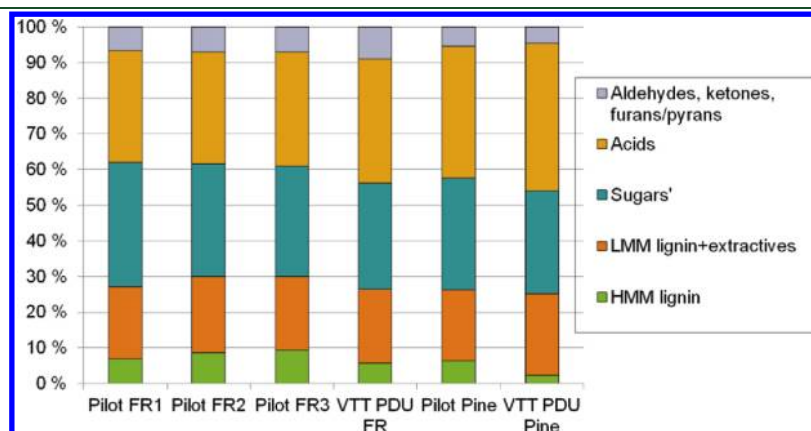


Figure 13. Bio-oil composition, VTT/PDU versus Metso pilot.

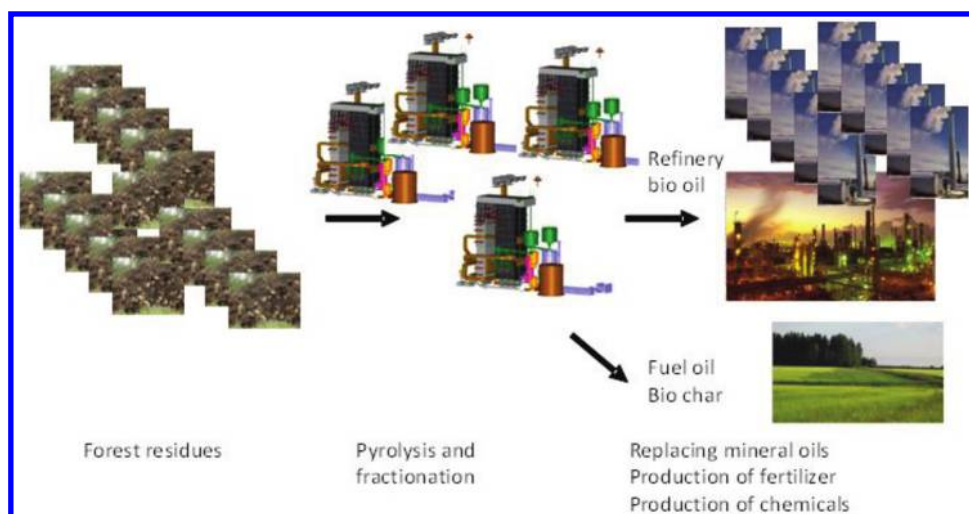


Figure 15. Phased implementation of biofuel, fertilizer, and chemical co-production: building more biofuel capacity.

be developed initially. The risk for an investor is manageable, because the initial investment would be an order of magnitude 20 M€. This may be compared to other second-generation bio-fuel technologies, where investments required for demonstration require considerable funds.

Industrial demonstration of pyrolysis is expected to take place in a couple of years. The phased investment strategy, which is especially feasible for biomass fast pyrolysis technologies, has been outlined in Figures 14 and 15.

SUMMARY

Systematic research on the production, handling, and use of pyrolysis oil has been carried out. Together with industry, VTT has developed and tested an integrated pyrolysis concept, which offers a viable route to replace fossil fuels. Metso has built and operated the world's first pilot plant at up to 2 MW_{th} integrated with a fluidized-bed boiler (4 MW_{th}), where proof-of-concept has been verified. In comparison to a stand-alone pyrolysis unit with a non-optimal small boiler for combustion of pyrolysis byproduct (char and pyrolysis gases), the integrated concept is easy and smooth to operate and has a high efficiency. For pyrolysis, having a steady and smooth flow of input energy (i.e., boiler sand) is a considerable advantage from the operator point of view. A first indication of expected organic liquid yields from any biomass may be obtained by analyzing the volatiles/fixed carbon of the feedstock to be pyrolyzed. The main liquid quality parameters to be monitored are water and solids content. Two automatic online water analyzers were tested. Both online KF titrator and MCA analyzer provided similar results to laboratory measurements. However, the KF titrator was more sensitive toward variation in solids content.

Use of bio-oil has been carried out by the industry in a district heat boiler at 1 MW_{th}. Analyses and test methods for physicochemical characterization and quality control of fast pyrolysis oils have been developed, and standardization work has been initiated.

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