

## Original Research Paper

Mechanically activated minerals as a sink for CO<sub>2</sub>

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## ABSTRACT

CO<sub>2</sub> removal from exhaust gasses from burning of fossil fuels is a political issue. If undertaken on a large scale, enormous amounts of carbon dioxide will have to be taken care of. This paper investigates how mechanical activation of olivine and other Ca or Mg containing silicates can increase the mineral's reactivity. Tests by grinding in a planetary mill have been done to investigate the effects of mechanical activation and other surface changes. The reactivity towards HCl and CO<sub>2</sub> has been determined as a function of grinding variables. Prolonged dry milling of olivine resulted in highly aggregated products, which were more reactive with respect to dissolution in acid than their respective BET surface areas would suggest. The initial breaking of bonds appears to give more reactivity on an energy usage basis than longer time activation. Most mechanical activation testwork so far has been carried out in batch mode. The result is diminishing effect of activation with increased grinding time. Continuous grinding and simultaneous reaction with some sort of classification may offer a chance of removing reaction products as soon as they are formed, giving a more energy effective process.

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## 1. Introduction

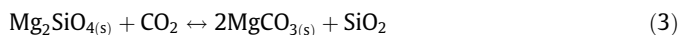
CO<sub>2</sub> removed from exhaust gases will represent a major disposal problem, but also a business opportunity as CO<sub>2</sub> may become the cheapest bulk chemical ever available. Mineral carbonation was proposed as CO<sub>2</sub> storage by Lackner et al. [1]. The knowledge of the reactions between minerals and CO<sub>2</sub>, mostly as carbonic acid, might be used to improve many mineral processes as CO<sub>2</sub> treatment of this kind can include dissolution of minerals and formation of new minerals.

A suited mineral for test purposes is the magnesium rich variety of olivine: forsterite. The simplified reaction between Mg-rich olivine and CO<sub>2</sub> in geological context can be described as the combination of two reactions. The first reaction is dissolution of CO<sub>2</sub> from air in water. pH will be around 5.6 and dissolved CO<sub>2</sub> will mainly exist as bicarbonate (Appelo and Postma [2]). The second reaction is described by the simplified Eq. (2).



olivine + acid + bicarbonate ↔ magnesite + silicic acid (dissolved silica)

The direct reaction is also a technical possibility:



Natural reactions require geological time spans. For industrial purposes alternatives have to be found. Testwork has been done with silicates containing magnesium. Both direct reaction with CO<sub>2</sub> as a gas and indirect reaction where CO<sub>2</sub> is dissolved in water which then has been reacted with the minerals has been tested (Gerdemann et al. [3], Giammar et al. [4], O'Connor et al., [5]). Heating will accelerate the reaction rate, but reaction (1) will be driven to the left if the temperature is increased. One solution to this problem is to run the reaction under high pressure in an autoclave as described by Haug et al. [6]. This is expensive. The alternative is to find other methods of speeding up the reaction. One possibility tested out here is pre-treatment of olivine particles to improve the dissolution process by mechanical activation.

The Slovak researchers Tkáčová and Baláž, have probably done more than anybody else to reap industrial benefits from mechanical activation and their monographs [7,8] give a good insight in the present state of the technology. Mechanical activation changes the character of the material beyond reduced particle size and increased surface area. During and after activation the crystal lattice is not in equilibrium and the excess energy caused by disordering contributes to lowering the activation energy of any further reaction of the material (Tkáčová [7]). The mills so far used to give mechanical activation have several times higher energy density than conventional tumbling mills.

Many experiments show that increasing milling time gives decreasing crystallinity, and this was found as increased amorphization from X-ray. (Baláž [8], Tromans and Meech [9], Pourghahramani and Forssberg [10]). Kleiv and Thornhill [11], and Haug et al.

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[6] describe specific details about the effect of mechanical activation of olivine. The purpose of this work is to evaluate the effect of mechanical activation of olivine especially the reactivity by dissolving olivine in acid (HCl) and then to look specifically on CO<sub>2</sub> in this context.

## 2. Materials

Forsterite is the simplest magnesium silicate available and therefore olivine foundry sand of the qualities AFS 50 and AFS 80 provided by North Cape Minerals from the dunite deposit at Åheim in Western Norway has been used in the current tests. X-ray diffraction shows that the sample contains 95% forsterite, the rest is chlorite and enstatite, chromite and traces of hornblende. AFS 50 had a specific surface area of 0.47 m<sup>2</sup>/g. The olivine used as test material for the dissolution experiment with HCl was refined from the AFS 80 quality sand by dry sieving to retrieve the fraction 74–147 µm and then washed with ethanol. The specific surface area was then 0.93 m<sup>2</sup>/g. The compositions of AFS 50 and the refined AFS 80 test sample are presented in Table 1.

## 3. Methods

Mechanical activation is a result of lattice disturbance of a mineral surface during grinding. A significant disturbance was in this case achieved by using a planetary mono mill, with a 250 cm<sup>3</sup> stainless steel mill chamber operating at 500 rpm which gave a grinding pressure about 15 G. Twenty Ø20 mm stainless steel balls weighing 645 g were used to grind 20 g olivine. The grinding was performed either dry, wet (using 100 ml distilled water) or moist (using 2.0 ml of water as a grinding aid). Three different series of tests were run.

### 3.1. Effect of grinding upon surface area and crystallinity

The specific surface area was measured using the BET method with N<sub>2</sub> adsorption. The X-ray diffraction analyses (XRD) was performed using a Philips PW1710 X-ray diffractometer.

The reduction of crystallinity due to milling can be calculated from X-ray diffraction analysis of the olivine samples (Ohlberg and Strickler [12], Baláz [8]). The average XRD-crystallinity, C<sub>XRD</sub>, of the olivine samples was calculated for the following 2θ peaks; 17.3; 22.9; 31.6; 35.7; 36.5; 41.6; 54.8. *I* is the intensity of the chosen peak above the constant background level found for the non-activated sample. The amount of material which not is crystalline is necessarily amorphous. Indices *i* and 0 refers, respectively to mechanically activated sample and original sample.

$$C_{XRD} = \frac{I_i}{I_0} * 100\%, \text{ or } (100 - C_{XRD})$$

will then be the degree of amorphization. (4)

**Table 1**  
Composition of AFS 50 and refined AFS 80 (REF).

Component	AFS 50 [wt. %]	REF [wt. %]
Fe <sub>2</sub> O <sub>3</sub>	7.13	7.54
TiO <sub>2</sub>	0.01	0.03
CaO	*	0.4
SiO <sub>2</sub>	41.64	42.29
MgO	50.62	48.39
MnO	0.1	0.1
LOI	0.34	0.41

\* Below detection limit.

### 3.2. Evaluation of olivine reactivity

In most tests 0.50 g ± 0.1% of sample was dissolved in 500 ml 0.01 M HCl in a Pyrex glass container. Haug [13] the olivine mass and acid volume was chosen so 30–35% of the olivine should be dissolved, the pH should change from 2 to at least 3. Finally the duration of the experiment should be between 1 and 24 h. The experiments were performed at 21.5 ± 1.3 °C and 1 atm. The pH in the suspension was logged automatically using a Metrohm lab 827 pH-meter. To avoid sedimentation a magnetic stirrer was used. The experiments were terminated when pH 3 was achieved. A 20 ml water sample was taken from the suspension at the end of each experiment. The sample was then filtered and transferred to a glass vial. Samples were not acidified.

The percentage of dissolved olivine was calculated for 30 min or the first hour of each experiment from the measured pH values based upon Eqs. (1) and (2) where 4 mol of H<sup>+</sup> is consumed for each mole of olivine dissolved. pH (initial) refers to initial pH in HCl solution as measured and *V* is the volume of 0.01 M HCl in liters used for the actual run. pH (time) is the measured pH at a given time. The activity factor γ<sub>H+</sub> was calculated PHREEQCi modeling USGS [14] <http://wwwbrr.cr.usgs.gov/projects/GWCcoupled/phreeqc/index.htm> (accessed 02.08.08) based upon solution analysis.

$$\text{Dissolved olivine (mol)} = \frac{(10^{-\text{pH (initial)}} - 10^{-\text{pH (time)}})}{4 \times \gamma_{H+}} \times V \quad (5)$$

The choice of milling parameters and other factors were chosen to obtain maximum difference in crystallinity as observed from XRD diffractograms and specific surface areas in order to get as wide variation in reactivity as possible.

### 3.3. Direct activation/carbonation

A series of batches were run where the dry activated products were exposed to gaseous carbon dioxide either during or after activation. These experiments were conducted using the mill chamber as a reactor utilising a special gas lid equipped with two valves, thus facilitating pressurisation of the chamber. Each batch was exposed to pure carbon dioxide gas by direct pressurisation of the filled mill chamber without previous flushing. An additional carbon dioxide pressure of 5.0 bar was applied. Consequently, approximately 1/6 of the free chamber volume consisted of air. The volume of the grinding balls constituted approximately 84 cm<sup>3</sup>. Assuming a density of 3.3 g/cm<sup>3</sup> for the original olivine feed, this yields a free chamber volume of 160 cm<sup>3</sup>. The gas was delivered at 20 ± 1 °C.

Three different combinations of mechanical activation and carbon dioxide exposure were studied; (1) exposure of the original olivine concentrate without any previous size reduction or mechanical activation, (2) exposure after mechanical activation, and (3) exposure during mechanical activation. The first combination involved placing the original concentrate and the grinding balls in the mill chamber before the gas lid was attached and the chamber was pressurised. Following 30 min of exposure to the gas, the chamber was depressurised and the final products were retrieved. The second combination was conducted by pressurising the chamber after the preceding 30 min of mechanical activation of the original olivine concentrate had been completed. This was done by first equalising the small overpressure in the chamber resulting from the increase in temperature during milling. Pressurisation of the mill chamber took place either immediately after activation (i.e., within 1 min) or after 60 min of relaxation. The third combination, exposure during activation, was performed by pressurising the mill chamber containing the olivine feed and the grinding balls prior to milling. A series of batches was produced in which the

duration of simultaneous milling and carbon dioxide exposure of the original olivine concentrate ranged from 5 to 60 min.

## 4. Results

### 4.1. Mechanical activation of olivine

In addition to the primary or initial comminution effects, the size distribution and the specific surface of the activated products will also depend on secondary processes like aggregation and agglomeration (Juhász [15]). Fig. 1 shows how the material that has been activated for 10 min contains a higher proportion of fines than the corresponding material activated for 30 min. A reduction of the amount of fines as the milling proceeds beyond its initial stage is commonly observed for dry milling. The specific surface exhibits a rapid initial increase before it peaks, reaching a value of  $5.08 \text{ m}^2/\text{g}$  after 10 min of mechanical activation. Milling beyond this point does not produce a further net increase in surface area. Instead, a small but significant decrease is observed. Similar results were obtained by Kleiv and Thornhill [11] when studying the mechanical activation of pure forsterite crystals using the same milling equipment. Here, a scanning electron microscopy analysis (SEM) produced direct evidence of agglomeration.

The results from the XRD analysis show a considerable reduction in peak height and signal to noise ratio with increasing activation time. (Fig. 2). The reduction is accompanied by a general broadening of the peaks. This suggests at least a partial structural modification and amorphization of the olivine lattice, even though the presence of small, but significant peaks even after 30 min of intense milling can be taken as an indication of the high milling resistance and mechanical strength of the submicron olivine particles. However, based on the diffractograms, it is reason to believe that prolonged milling have a significant effect on the crystal lattice. Dry grinding gave the lowest surface area, wet significantly higher and moist the highest surface area when conditions were the same. With regard to amorphization the situation was reversed. Water acts as a grinding aid in a way that reduces amorphization and increases surface area.

### 4.2. Material characteristics and pH change in dissolution experiments

Fig. 3 shows the measured pH versus experiment time. The reactivity of the reference sample was significantly lower than the mechanically activated materials.

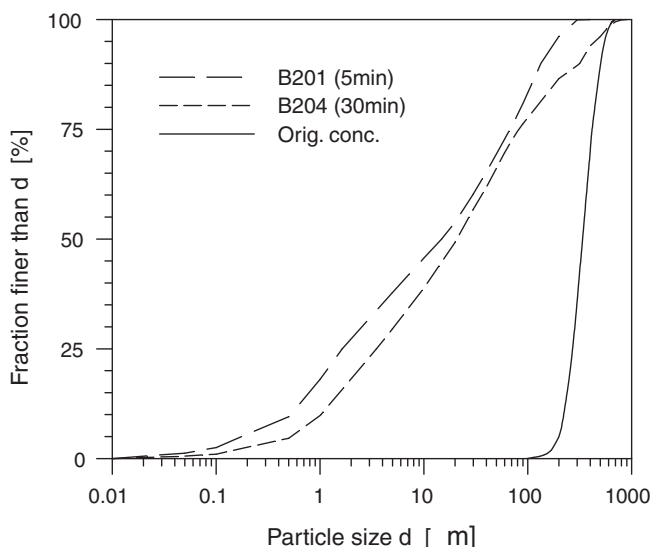


Fig. 1. Cumulative particle size distributions of selected activated olivine materials.

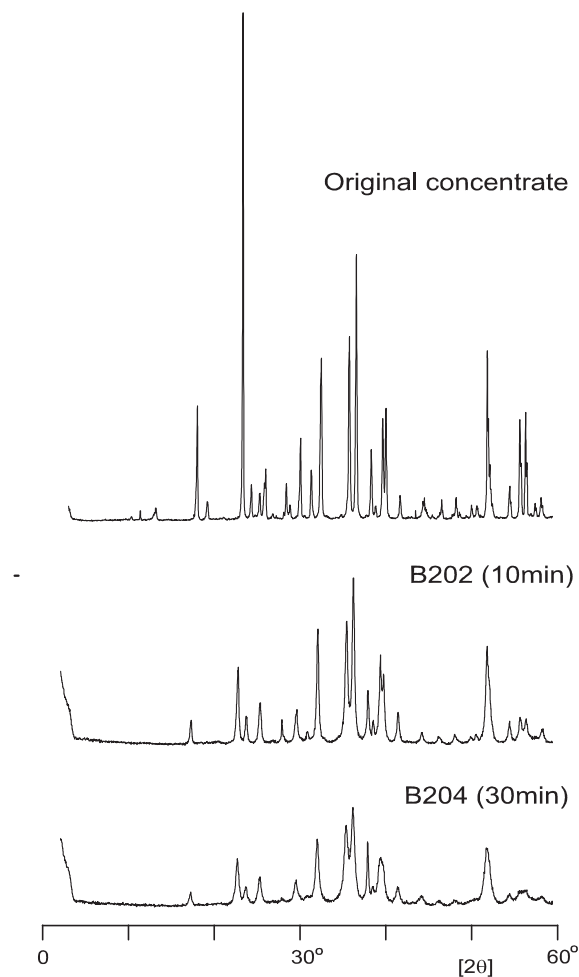


Fig. 2. XRD-diffractograms comparing the signature of the original concentrate to those of the activated products.

Generally the materials with the highest amount of fines should have been the most reactive and given the highest increase in pH. The opposite was found, and the wet milled materials (graph C in Fig. 3), having the highest amounts of fines were the least reactive materials. An increase in pH change rate corresponds to increased milling time and decreased water addition. The measured pH was used to calculate the total mass of olivine dissolved and there is a good agreement between the  $\text{Mg}^{2+}$ -content in the water samples and the content calculated from the pH change. The calculated value was less than 10% below the measured value which is within the uncertainty of the absolute calibration of the pH-meter,  $2.00 \pm 0.02$  units. The experiment was designed to have enough acid to dissolve approximately 35% and the result might have been different with a higher acid:olivine ratio. The pH at the end of the dissolution experiments varied from 2.7 to 6.0.

The specific surface area,  $S_A$ , is a better measure of the complex particle shape and surface topography than particle size. The measured specific surface areas are plotted against the fraction of dissolved olivine after 30 min reaction (Fig. 4). The wet milled samples and moist (10% water) milled samples have a good correlation between specific surface area and dissolved olivine. On the other hand, the specific surface area is very low for the dry milled samples, at the same time as they obtain a very high amount of dissolved olivine after 30 min. The calculated crystallinity,  $C_{\text{XRD}}$ , from XRD (Eq. (4)) or rather the degree of amorphization ( $100 - C_{\text{XRD}}$ ) was used as a measure of the crystal lattice disordering. The

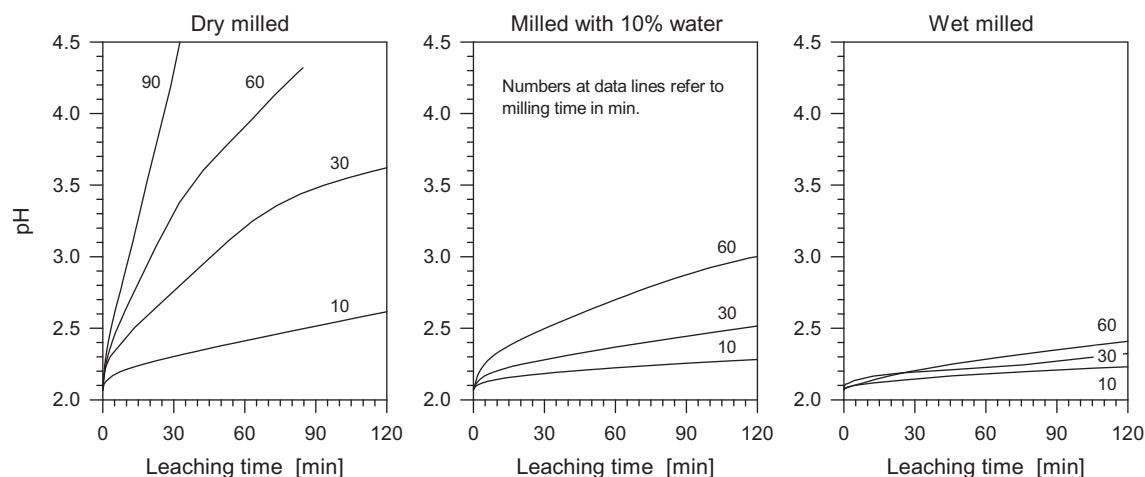


Fig. 3. pH versus leaching time in 0.01 M HCl.

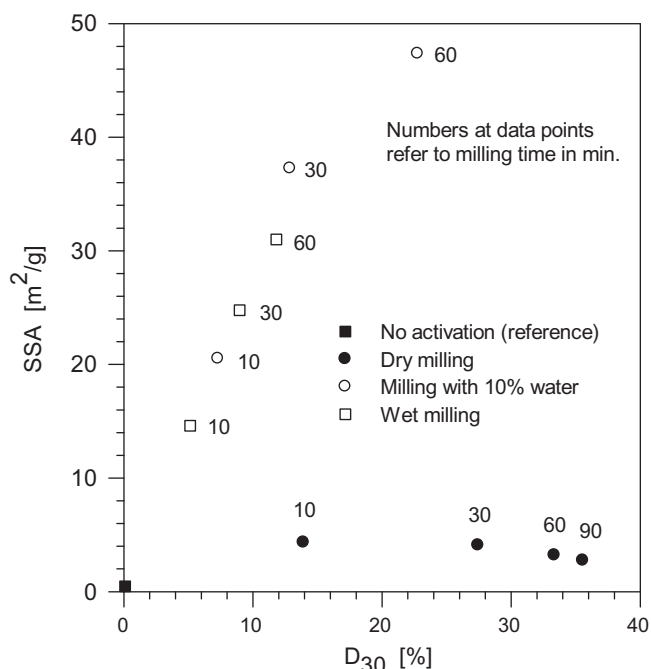


Fig. 4. Specific surface area (SSA) versus relative amount of olivine dissolved after 30 min ( $D_{30}$ ).

calculated  $C_{XRD}$  does not distinguish between deformation in crystal lattice and particle size effects, but describes the overall change in the peak intensity. For all three levels of water addition an increase of the milling time significantly decreased the crystallinity. The dry milled materials were most amorphous compared to wet and 10% wet milling. Fig. 5 shows the degree of amorphization for all samples versus the dissolved amount of olivine after 30 min of dissolution, as calculated by Eq. (1). Dry and moist milling follows the trend line, but wet milling might follow a trend where the effect of crystallinity is less important for the resulting amount of dissolved olivine. The difference can be related to surface topography, behavior of contamination or some other factor not included here. Fig. 4 shows that the specific surface area, seems to be most important for dissolution of olivine, when the crystallinity is high. For the mineral in amorphous state the specific surface area becomes insignificant as shown in Fig. 5.

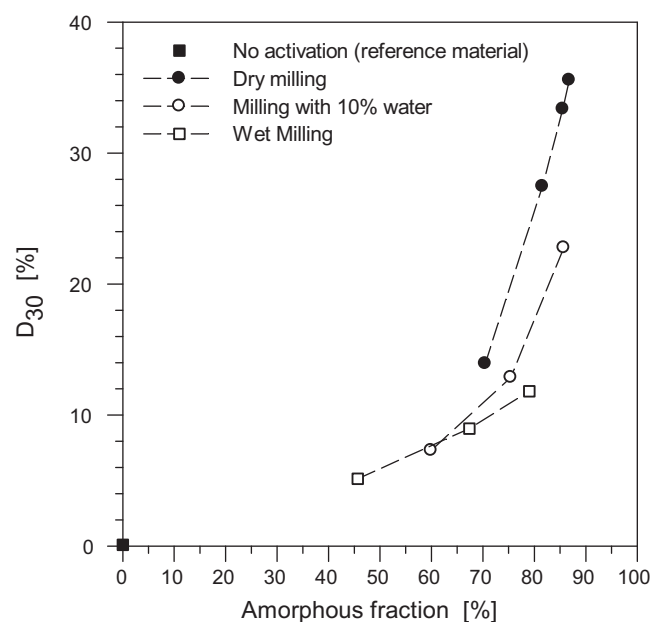


Fig. 5. Relative amount of olivine dissolved after 30 min ( $D_{30}$ ) versus relative amount of amorphous phase.

#### 4.3. Carbonization experiments

The amount of carbon dioxide sequestered during the carbonation experiments was calculated from the concentrations of total carbon in the final product as determined by the LECO analysis. The calculations were based upon the following assumptions: (1) the original feed material contained no carbon, (2) the number of moles of carbon dioxide sequestered during the carbonation experiments can be expressed by the following equation:

$$n_{\text{CO}_2, \text{seq}} = \frac{m_f \cdot x_{\text{C,p}}}{M_{\text{C}} - M_{\text{CO}_2} \cdot x_{\text{C,p}}} \quad (6)$$

where  $m_f$  is the mass of the original feed,  $x_{\text{C,p}}$  is the concentration (mass fraction) of carbon in the final product, and  $M_{\text{C}}$  and  $M_{\text{CO}_2}$  is the atomic and molecular weights of carbon and carbon dioxide, respectively (i.e.,  $M_{\text{C}} = 12.00$  g/mol and  $M_{\text{CO}_2} = 44.0$  g/mol).

The gas conversion ratio, that is, the ratio between the amount of carbon dioxide that has been sequestered and the amount initially present in the gas phase, can be defined as:

$$\gamma_{\text{CO}_2} = \frac{n_{\text{CO}_2, \text{seq}}}{n_{\text{CO}_2, \text{ini}}} \quad (7)$$

where  $n_{\text{CO}_2, \text{ini}}$  can be calculated from the ideal gas law. In this study, the solid conversion ratio is defined by making the simplifying assumptions that only magnesium is available for carbonation and that the stoichiometry of this reaction is determined by Eq. (3). The solid conversion ratio can then be defined in Eq. (7): where  $n_{\text{Mg}, \text{f}}$  represents the total number of moles of magnesium in the feed:

$$\gamma_{\text{Mg}} = \frac{n_{\text{CO}_2, \text{seq}}}{n_{\text{Mg}, \text{f}}} \quad (8)$$

The results from the total carbon analysis are presented in Table 2 along with derived values for both the gas and solid conversion ratios. As can be seen from these data, significant amounts of carbon dioxide were sequestered when the original olivine concentrate was exposed to the gas during milling, compared to when mechanical activation was used as a mere pre-treatment step. In the latter case, 30 min of mechanical activation immediately followed by 30 min of carbon dioxide exposure resulted in total carbon concentrations below the analytical detection limit for one out of three batch replicates. Unsurprisingly, exposure without previous activation and exposure following 60 min of relaxation also produced values below the detection limit. In contrast, a triplicate run comprising 30 min of simultaneous activation and exposure resulted in a concentration of  $0.69 \pm 0.026\%$  total carbon in the final product, corresponding to a gas conversion ratio of  $36 \pm 1.4\%$ .

## 5. Discussion

If ever mechanically activated minerals have to become attractive sinks or reactants for  $\text{CO}_2$  the energy used for activation has to be minimized. Fig. 3a shows that the output of leach able olivine is largest per min for the shortest grinding time (lowest energy use per amount of olivine reacted). Fig. 3b and c reveals that wet grinding or adding water as a grinding aid, decreases reactivity. Fig. 4 based upon the same data shows that for the dry milled materials, the energy has been spent making reactive material, again with a diminishing return compared to energy used. Wet grinding and water as grinding aid created surface area instead of reactive material. Fig. 5 may indicate that the relationship between amorphous phase determined by XRF and reactivity is not quite straightforward. Those findings have been compared to data given by Baláž

[16] (in Table 2). The data have been recalculated to show amorphization as a function of energy used and are given in Fig. 6. The figure shows the same trend as presented in this paper and demonstrates for a number of grinding devices a diminishing return of energy efficiency when it comes to mechanical activation. There are considerable differences between mills used. A wet attritor mill appears to be more efficient than the other pieces of equipment used, but nevertheless the picture is the same.

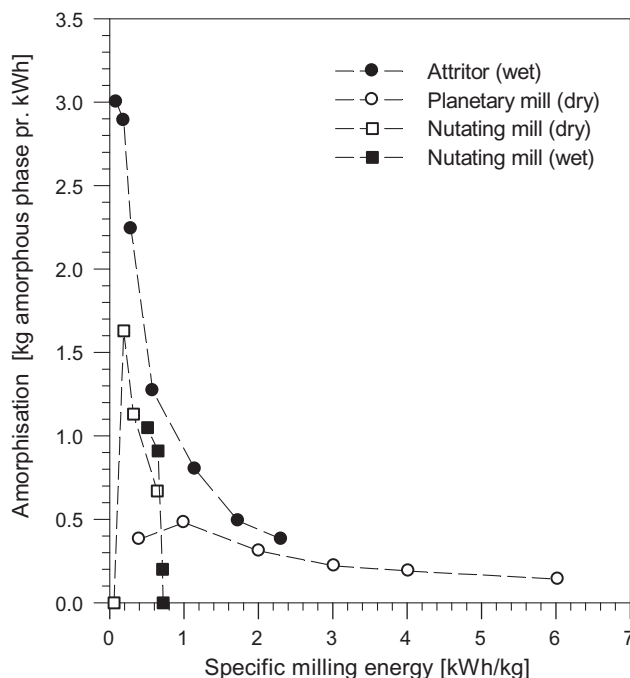
Two aspects of reactivity between  $\text{CO}_2$  were investigated. In the first, the experiments were conducted with same the energy input (30 min grinding time), the difference between the tests was the time of  $\text{CO}_2$  introduction. The significantly increased uptake when  $\text{CO}_2$  was added during grinding must be attributed to surface reactivity which appears to be greatest when breakage takes place. The relaxation effect comes quite rapidly. These transient reactivity levels appear to be significantly higher than the residual reactivity levels of the final products. Hence, the results clearly demonstrate how the concept of simultaneous mechanical activation and carbon dioxide exposure yield considerably higher sequestration rates than those obtained from the conventional sequential implementation of the two processes. This may explain why Fig. 5 does not show a proportional relationship between amorphous fraction and reacted material after 30 min dissolution of already ground samples.

Fig. 7 shows that the total carbon concentration in the final product increases with the exposure time, reaching some 1.08% after 60 min of mechanical activation. This corresponds to 0.82 g sequestered carbon dioxide, thus yielding gas and solid conversion ratios of 57% and 7.5%, respectively. The ratio between the two conversion rates can be manipulated by changing the balance between the amount of gas and solid in the feed. Future research is needed to find a balance that will maximise carbon dioxide sequestration per time unit, energy unit and volume unit. Fig. 7 further indicates that the reaction with  $\text{CO}_2$  is more or less proportional with the grinding time. The amorphous material reacts and disappears as soon as it is formed. The curve might be flattening as the surface is covered by reaction products. Only a small amount of the

**Table 2**  
Results from the carbonation experiments showing the total carbon concentration ( $x_{\text{C,p}}$ ) and the corresponding gas ( $\gamma_{\text{CO}_2}$ ) and solid ( $\gamma_{\text{Mg}}$ ) conversion ratios.

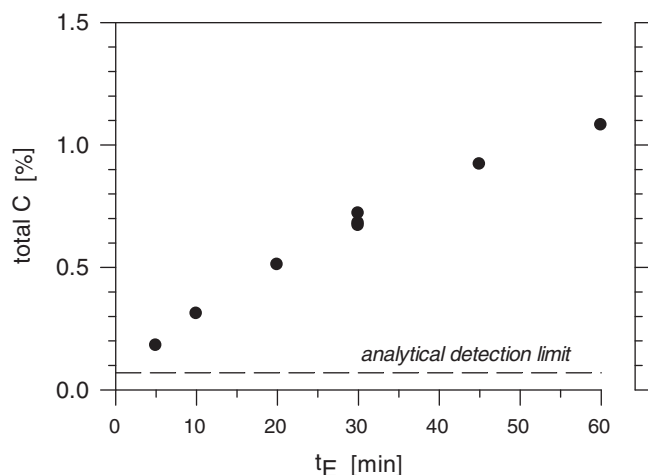
Mode of $\text{CO}_2$ exposure	$t_m$ [min]	$t_E$ [min]	$x_{\text{C,p}}$ [%]	$\gamma_{\text{CO}_2}$ [%]	$\gamma_{\text{Mg}}$ [%]
Without MA	0	30	<0.07	<3.6	<0.47
Immediately after MA	30	30	0.10	5.1	0.67
Immediately after MA	30	30	<0.07	<3.6	<0.47
Immediately after MA	30	30	0.08	4.1	0.54
After MA (60 min relax.)	30	30	<0.07	<3.6	<0.47
During MA	5	5	0.18	9.2	1.2
During MA	10	10	0.31	16	2.1
During MA	20	20	0.51	26	3.5
During MA	30	30	0.68	35	4.7
During MA	30	30	0.67	35	4.6
During MA	30	30	0.72	37	4.9
During MA	45	45	0.92	48	6.4
During MA	60	60	1.08	57	7.5

$t_m$  and  $t_E$  denotes the milling time and the  $\text{CO}_2$  exposure time, respectively.



**Fig. 6.** Amorphisation as a function of specific milling energy (from Baláž et al. [16]).





**Fig. 7.** Total carbon concentrations as a function of exposure time ( $t_E$ ) for materials exposed to carbon dioxide during mechanical activation.

olivine did react during the time studied, but a major part of the carbon dioxide, this may also be a reason the reaction rate appeared to decrease.

A natural step further in order to minimize energy use, would be to introduce an (air)/CO<sub>2</sub> swept tumbling ball mill where the activated/reacted products are effectively removed as soon as they are formed, while the coarse crystalline material is given another beating. Thus the original breaking of chemical bonds, which should give the most efficient activation, can be fully utilized.

## 6. Conclusion

Mechanical activation of olivine increases the specific bulk reaction rate [% dissolved after 30 min] when olivine is dissolved in a 0.01 M HCl solution. The particle size distribution can not be used to explain the reaction rates found, but the measured specific surface area and the crystallinity,  $C_{XRD}$ , (from XRD) are very important.

Differences in the degree of amorphization and the specific surface area are directly reflected in the measured pH change.

Direct contact between carbon dioxide and olivine in a dry mill gives more efficient reaction possibilities than separate grinding and then mixing. It is therefore a possibility that conventional grinding in a tumbling mill in a CO<sub>2</sub> atmosphere may compete with the usage of high energy efficiency mills when CO<sub>2</sub> sequestration is concerned.

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