

# The effect of mechanical activation in the production of olivine surface area



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## ABSTRACT

This study investigates the effect of dry mechanical activation of olivine as a pre-treatment step prior to wet milling with respect to the production of specific surface area. Milling experiments combining the two milling modes (i.e. dry and wet) were performed using a laboratory planetary mill. The specific surface area of the milled products was determined by N<sub>2</sub>-adsorption (BET), whereas their crystallinity was studied using X-ray diffraction (XRD). Combining dry mechanical activation with subsequent wet milling makes it possible to produce specific surface area at rates that exceed those that can be obtained by wet milling alone. Whereas a value of  $26.28 \pm 1.28 \text{ m}^2/\text{g}$  was obtained after 30 min of wet milling, 25 min of dry mechanical activation followed by 5 min of wet milling resulted in a specific surface area of  $64.44 \pm 2.13 \text{ m}^2/\text{g}$ . The concept could represent significant energy savings, and enable olivine products that combine high specific surface area with an activated structurally disordered surface.

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## 1. Introduction

Magnesium-rich olivine has received a lot of attention as a potential binding agent for carbon dioxide sequestration (e.g. Azdarpoura et al., 2015; Haug, 2010; Huijgen and Comans, 2005; Lackner et al., 1995; Seifritz, 1990), it has been proposed as source for hydrometallurgical magnesium production and has been used commercially as a heavy metal adsorbent (Kleiv and Sandvik, 2002; Kleiv and Thornhill, 2004; Rekkedal, 2013). All these applications rely on reactions (dissolution or adsorption) whose rates are directly proportional to the specific surface area of the olivine material.

Olivine is comparable to quartz in hardness, scoring 7 on Moh's scale. As with quartz the fracture is brittle, yielding small conchoidal fragments. The forsterite olivine from the Åheim deposit has been shown to be a very mechanically competent and hard to grind material with a Bond's work index of 19.6 kW h/tonne (Saramak and Kleiv, 2013). Hence, the production of surface area comes at a price. As a result, several investigators have studied the grindability of olivine, especially with focus on subsequent carbon dioxide sequestration (Atashin et al., 2015; Haug, 2010; Saramak and Kleiv, 2013; Summers et al., 2005).

In addition to a high specific surface area, the rate and extent of several heterogenic reactions could also benefit from a structurally

activated olivine surface. Mechanical activation, i.e. mechanically induced structural disordering of the mineral's crystal lattice through high intensity dry milling, could be used to render the olivine surface more reactive (Atashin et al., 2015; Fabian et al., 2010; Haug et al., 2010; Kleiv and Thornhill, 2006; Turianicová et al., 2013). Compared to wet milling, dry milling is usually more effective at achieving such activation. However, due to a higher degree of agglomeration, dry high intensity milling will usually yield relatively modest values for specific surface area.

The structural disordering that takes place during dry mechanical activation will undoubtedly affect the grindability of the resulting product, which could be exploited in a subsequent wet milling step. This paper presents the results from milling experiments in which the two milling modes are combined in order to study the effect on the overall production of new surface area.

## 2. Materials and methods

### 2.1. The original olivine feed

The material used in this study was supplied by the Norwegian mining company North Cape Minerals and originates from the dunite deposit at Åheim in western Norway. The material is a standard foundry sand (AFS50) produced by crushing, drying and sieving the dunite ore. Prior to sieving, fines are removed by air sweeping leaving a well sized material ranging from  $d_5 = 200 \mu\text{m}$  to  $d_{95} = 520 \mu\text{m}$ . This product consists of approximately 95%

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forsterite olivine ( $\text{Mg}_{1.86}\text{Fe}_{0.14}\text{SiO}_4$ ) in addition to minor quantities of chlorite, enstatite, calcic amphiboles, serpentine, talc, and chromite.

The olivine sand used in this study is identical to the material used by Baláž et al. (2008), Fabian et al. (2010), Haug et al. (2010, 2011), Kleiv et al. (2006), and Sandvik et al. (2011) in their studies of the effect of mechanical activation or mechanical activation combined with carbon dioxide sequestration.

## 2.2. Milling experiments

Three different experimental series were conducted in which the original olivine feed was subjected to mechanical activation using a *Fritsch Pulverisette 6* planetary mono mill. The two first series were designed simply to establish the respective relationships between the specific surface area and the milling time for the dry and wet milling modes. The third series consisted of batches that were subjected to dry milling prior to wet milling. This was achieved by adding distilled water to the mill chamber after the preset duration of dry milling had been reached.

All three experimental series were conducted by milling 20.0 g olivine feed material in a 250 cm<sup>3</sup> stainless steel mill chamber. Wet milling was achieved by adding 100 ml of distilled water to the mill chamber. The milling was conducted at 500 rpm using twenty Ø20 mm stainless steel balls with a total weight of 645 g. During the two first series the milling time varied from 2 to 30 min, whereas the total accumulated milling time of the third series was fixed at 30 min. A few selected batches were run in triplicate.

At the end of each individual batch the final product was carefully retrieved by either using a brush and a spatula (dry mode) or a rinse bottle (wet mode). Wet samples were left to settle for 24 h, after which the majority of the water was carefully decanted. Finally, the samples were dried in a heating cabinet at 35 °C for 72 h.

## 2.3. Specific surface area and size distribution of milled products

The specific surface area of the milled products was determined using the N<sub>2</sub>-adsorption technique (i.e. the BET equation) and a *Flow Sorb II 2300* volumetric gas adsorption analyser equipped with a degassing unit, whereas their size distributions were obtained from Mie diffraction analysis using a *Malvern Mastersizer 3000*.

## 2.4. Crystallinity of milled products

The crystallinity of the milled products was studied using X-ray powder diffraction (XRD) employing a *Philips PW1710* diffractometer with a *PW 1830* generator. The XRD analysis was performed with monochromatised CuK<sub>α</sub> radiation, scanning from 2–60° with a step size of 0.04° (2θ). The reference sample for the XRD-analysis, representing the nominal crystalline basis, was produced by milling the original feed material for 30 s in a *Siebtechnik* disk mill.

The relative fraction of crystalline phase ( $X_{\text{XRD}}$ ) in the milled products was calculated according to the method proposed by Ohlberg and Strickler (1962):

$$X_{\text{XRD}} = \frac{U_0}{U_x} \cdot \frac{I_x}{I_0} \quad (1)$$

Here,  $U$  represents the background level,  $I$  represents the integral intensity of a selected diffraction peak, and the subscripts 0 and  $x$  denotes the reference sample and the activated sample, respectively. The forsterite diffractogram is characterised by a number of closely spaced diffraction lines resulting in overlapping peaks that make evaluation difficult. The 020 ( $hkl$ ) peak was chosen as it is one of the few peaks that lends itself to a closer analysis. This

peak has also been evaluated by Baláž et al. (2008) and Haug et al. (2010).

The relative degree of structural disorder was also expressed by Patzak's  $F$ -value (Patzak, 1966 in Baláž, 2000):

$$F = \frac{ILB_x}{ILB_0} \quad (2)$$

where  $ILB$  is the integral width of the 020 ( $hkl$ ) peak.

## 2.5. SEM microphotographs

Scanning electron microscope (SEM) microphotographs of selected products were obtained using the secondary electron detector (i.e. SE images) of a Hitachi SU-6600 SEM.

## 3. Results and discussion

### 3.1. Specific surface area and size distribution as a function of milling time

The main results of the investigation are presented in Fig. 1, which shows the specific surface area  $S_A$  as a function of the milling time  $t_M$ . For the third experimental series,  $t_M$  represents the total accumulated milling time (i.e. the combined duration of dry and wet milling). The different products are labelled with reference to the production variables. To exemplify, the notation  $w15$  refer to a material that has been produced by 15 min of wet milling, whereas  $d25w5$  represents the combination of 25 min of dry milling followed by 5 min of wet milling.

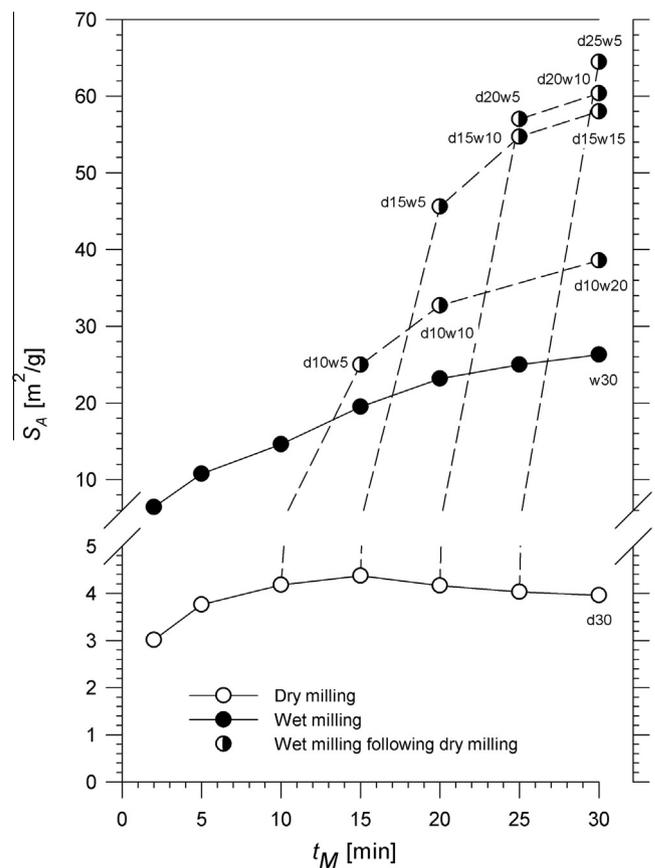


Fig. 1. Specific surface area as a function of total milling time. Note the break in the ordinate axis.

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