



# Ultra-fine grinding and mechanical activation of mine waste rock using a planetary mill for mineral carbonation



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## ABSTRACT

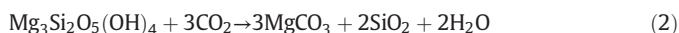
In order to mitigate the effects of climate change, a worldwide effort is being launched to use ultramafic mine waste as feedstock for CO<sub>2</sub> sequestration by mineral carbonation. To enhance the rate of mineral carbonation at any specific mine site, it is important to develop individualized methods for use at that specific mine site, and with respect to its particular waste mineralogy. This study examines mechanical activation as the pre-treatment method for ultramafic mine waste containing forsterite and serpentine. Particle size distribution, specific surface area, microstructure and direct aqueous mineral carbonation were measured to test the effects of mechanical activation. The Rietveld method was used to analyze the microstructure of untreated and mechanically-activated material. It was found that, as a result of the grinding energy input on olivine, particle size was decreased, surface area was increased, crystallite size was reduced and micro-strain was accumulated. During the dry mechanical activation of mine waste, serpentine content was partially dehydrated and converted to olivine. The concurrent grinding of serpentine with olivine promoted the formation of new surface area, but it hindered the disordering of the crystal structure of forsterite. Forsterite makes up the majority of the content and contributes to CO<sub>2</sub> sequestration under selected carbonation conditions. The CO<sub>2</sub> sequestration conversion of mechanically-activated olivine and mine waste are 22.5% and 31.5%, respectively, with 3600 kWh/t specific milling energy input after one hour direct aqueous carbonation. For the purposes of mineral carbonation, the mechanical activation of mine waste is preferable to simply grinding pure olivine.

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## 1. Introduction

Global climate systems have been observed to be warming since the 1950's, with increased temperatures being detected in the atmosphere and the oceans, snow and ice levels diminishing, and sea levels being raised. The dominant cause of climate change has been attributed to the cumulative emissions of global anthropogenic greenhouse gases, particularly carbon dioxide (CO<sub>2</sub>) (IPCC, 2014). CO<sub>2</sub> capture and sequestration (CCS) has been proposed as a solution for mitigating climate change through reducing CO<sub>2</sub> emissions. Of all the CCS methods, mineral carbonation (MC) provides an alternative strategy to geological sequestration, and is considered to be a preferred option with respect to the storage of CO<sub>2</sub> for small and medium-sized industrial emitters (<2.5 Mt CO<sub>2</sub>) (Sanna et al., 2014). MC mimics a natural weathering process, whereby magnesium or calcium silicates spontaneously react with CO<sub>2</sub> to form stable carbonates. The reaction of mineral carbonation on olivine and serpentine is described in Eqs. (1) and (2), respectively.

Due to the thermodynamically favorable processes, MC can guarantee the permanent and environmentally-friendly storage of CO<sub>2</sub>.



Since the process of MC was first proposed in 1990 (Seifritz, 1990), researchers have focused on finding ways to accelerate the process, including through the selection of process routes and conditions, and the pre-treatment of feedstock at low levels of energy consumption (Power et al., 2013). However, MC is currently not yet economically viable for large scale deployment. Magnesium silicates, olivine and serpentine are the most abundant feedstocks available for mineral carbonation (Lackner et al., 1999). Olivine is composed of a mixture of the magnesium-rich forsterite (Mg<sub>2</sub>SiO<sub>4</sub>) and the iron-rich fayalite (Fe<sub>2</sub>SiO<sub>4</sub>). Olivine has an orthorhombic structure with an independent SiO<sub>4</sub> tetrahedra linked by divalent ions in 6-fold coordination (Klein and Hurlbut, 1993). Serpentine (Mg<sub>3</sub>Si<sub>2</sub>O<sub>5</sub>(OH)<sub>4</sub>) consists of a group of common rock-forming hydrous magnesium phyllosilicate minerals. In all its polymorphs, lizardite, antigorite, and chrysotile are the most common serpentine minerals found in nature. The structure of serpentine consists

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of an overlapping sheet of SiO<sub>4</sub> tetrahedra and a sheet of Mg octahedral (brucite-like) in which the –OH base is bonded (Klein and Hurlbut, 1993). Both olivine and serpentine make up the dominant contents of mine waste rock and tailings in ultramafic deposits, such as nickel mines, diamond mines, PGE mines and former asbestos mines (Hitch et al., 2010). Turning ultramafic waste rocks or tailings into valuable CO<sub>2</sub> feedstock can benefit a deposit both environmentally and economically (Hitch and Dipple, 2012). With respect to the process of integrated mineral carbonation, the pre-treatment method of mechanical activation (MA) might become practical because the energy consumption of the operation is reduced when MA is integrated as part of the milling procedure in mineral processing (Haug, 2010).

MA uses mechanical force to accelerate a downstream process. Studies on the use of MA on ultramafic rocks are constantly expanding. They have focused on comparing individual mineral (i.e. olivine or serpentine) (Kim et al., 2008; Rigopoulos et al., 2015) responses to the grinding energy input (Atashin et al., 2016; Atashin et al., 2015), milling instruments (i.e. planetary mills, vibration mills, attrition mills, or tumbling mills) (Baláz et al., 2008; Haug, 2010), milling atmosphere (i.e. N<sub>2</sub>, CO<sub>2</sub> or air) (Turianicová et al., 2013), milling solutions (i.e. water, alcohol or acid) (Rigopoulos et al., 2015; Sandvik et al., 2011), and milling temperatures (Trapasso et al., 2012). These studies agree that MA induces changes in the surface properties and crystal structures of minerals, and that it thus enhances their rates of dissolution (Kleiv and Thornhill, 2006), –OH dehydration (Hrsak et al., 2008), carbonation (Summers et al., 2005), CO<sub>2</sub> adsorption (Fabian et al., 2010), and solid state reactions (Atashin et al., 2016). However, the concurrent grinding of olivine and serpentine was rarely studied. The authors have investigated fine grinding and mechanical activation of mine waste materials in a wet condition (Li and Hitch, 2016a, 2016b; Li and Hitch, 2015), the interactions between two minerals were confirmed through various characterization methods. Nevertheless, the mechanical activation of mine waste materials in dry condition has not yet been studied.

The aim of the present paper is to investigate the physical, structural and chemical properties of mine waste rock (that contains both olivine and serpentine), and which has been subjected to mechanical activation under dry conditions. MA was induced using a planetary mill since this is a type of high-intensity mill that is commonly used for MA. A direct aqueous mineral carbonation process was used to test the effects of the MA due to it being the most competitive route for industrial application. Different types of characterization techniques were used to recognize mechanically-induced changes in mine waste rock.

## 2. Materials and experiments

### 2.1. Materials

The olivine foundry sand (TSO) was obtained from the Twin Sisters Deposit in northwest Washington State, USA. TSO samples were used as a control. The mine waste rock or whole ore (TAW) was provided by Hard Creek Nickel Corp. from their Turnagain deposit in northern British Columbia, Canada. All received materials were ground by a laboratory bond ball mill, and screened using 140 mesh Tyler sieve (106 μm). The undersized materials were used as the starting sample for the planetary milling and the chemical and mineralogical compositions tests.

Table 1 shows the content of major elements of samples, expressed as wt% oxide, which were analyzed using X-ray fluorescence (XRF) at Acme Analytical Laboratories Ltd. Canada. The mineral compositions of

the samples received were tested using X-ray powder diffraction (XRPD) analysis in the Earth and Ocean Science Department at the University of British Columbia. XRPD detected 97.9% forsterite, 1.5% chromite, <1% lizardite, and 0.4% quartz in TSO. TAW contains 64.7% forsterite, 29.6% lizardite, 4.2% magnetite, 1.2% brucite, and <1% quartz.

### 2.2. Grinding test

The laboratory grinding tests were conducted using a planetary ball mill (Micro Mill Pulverisette 7, Fritsch GmbH, Germany) from Advanced Materials and Process Engineering Laboratory, UBC. The two bowls of the Pulverisette 7 have a planet-like movement, where the bowls rotate around a central axis and also simultaneously around their own axis in opposite directions with the same rotational speed (Abdellahi and Bahmanpour, 2015). The rotational speed of the mill was kept constant at 500 rpm. In each test, 10 g of feed material was ground with seven 15 mm zirconium oxide balls in a 45 mL zirconium oxide mill bowl (95% ZrO<sub>2</sub>, density 5.7 g/cm<sup>3</sup>) under a dry atmospheric condition. The ball to solid weight ratio was fixed to 7:1. The samples were ground for 0 min, 30 min, 60 min, and 120 min, and were then selected, carbonated and characterized. Every 15-min of grinding was followed by a 15-min rest interval to prevent the overheating of the material and of the milling ball. The milling temperature was maintained below 30 °C, as monitored by a thermocouple.

The net specific energy consumption  $W_N$  (kWh/t), which is the energy transferred to the powder, is calculated according to Eqs. (3) and (4).

$$P_N = P_0 - P_{NL} \quad (3)$$

$$W_N = \frac{P_N}{m} \times t_{MA} \quad (4)$$

where  $P_N$  is the specific power (kW),  $P_0$  is the operational power (kW), and  $P_{NL}$  is the power draw (kW) when running the mill empty with no material or media.  $P_0$  and  $P_{NL}$  are monitored by a Watts-up?™ Pro electrical watt meter (Think Tank Energy Products Inc., Vermont, USA) at a 0.2 s interval.  $m$  is the mass of sample milled (t).  $t_{MA}$  is the MA time (hour). As no changes have occurred in the milling parameters, the net specific energy consumption for a specific duration remains the same. The materials that were mechanically-activated for 30, 60, and 120 min, correspond to the net specific energy consumption of 900, 1800 and 3600 kWh/t, respectively. Grinding time and specific milling energy input are considered to have the same influence on the effects of mechanical activation.

### 2.3. Carbonation test

The carbonation condition is chosen according to the optimum conditions for the direct aqueous mineral carbonation of olivine (O'Connor et al., 2005) except for the working pressure which was reduced from 15 MPa to 6 MPa due to limitations of the experimental set-up, in which gas was supplied directly from a gas cylinder without the aid of a booster pump. 7.5 g of samples were mixed with 50 mL solutions containing 1 mol/L NaCl and 0.64 mol/L NaHCO<sub>3</sub>, and transferred to a 100 mL Hastelloy-4566C Parr bench-top stirred autoclave (Parr Instruments, Moline, IL, USA). The maximum working temperature and pressure of the autoclave are 500 °C and 20.7 MPa, respectively. Through increasing the concentration of HCO<sub>3</sub><sup>-</sup> in the aqueous solution and maintaining a constant pH of between 7 and 8, the solution can be used to help achieve the optimum extent of carbonation. The sample slurry was carbonated at 185 °C and 6 MPa CO<sub>2</sub>, with a stirring speed of 1500 rpm for 1 h.

**Table 1**  
XRF results for the TSO and TAW (wt%).

Component	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	MnO	TiO <sub>2</sub>	Cr <sub>2</sub> O <sub>3</sub>	LOI	TOT/C
TSO	40.7	0.15	8.39	0.09	50.94	0.12	0.02	0.677	-0.3	<0.02
TAW	38.7	0.15	10.39	0.3	45.51	0.16	0.03	0.671	4.09	0.05

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