

Mechanical activation of magnesium silicates for mineral carbonation, a review

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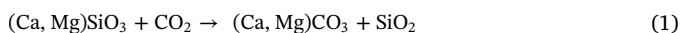
ABSTRACT

Mechanical activation is one of the most efficient pretreatment methods used to accelerate the reaction rate in the mineral carbonation technology. This paper reviews the current research related to this technique with a specific focus on three types of magnesium silicates - olivine and serpentine, and partially serpentinized olivine. First, the effects of mechanical activation on magnesium silicates are listed and compared with regard to a variety of milling conditions, including diverse energy inputs, grinding aids and mill types. Then, the existing literature determines the extents of mechanical activation, reviewed with respect to downstream carbonation processes, including direct gas/solid carbonation, direct aqueous carbonation, indirect aqueous carbonation, and indirect solid/solid mechanochemical carbonation. Finally, suggestions are made for further study on applying mechanical activation in an integrated mineral carbonation process, in order to lower the energy consumption of commercial systems.

1. Introduction

The average global temperature shows a warming of 0.8 °C in the last 100 years (Ray, 2015). The main cause of this rise in average global temperature is burning of fossil fuels to obtain cheap energy, which emit a large amount of greenhouse gases, such as CO₂. As reported by the NOAA (GMD, 2018), atmospheric concentration of CO₂ has increased at a rate of 2 ppm (parts per million) per year since 2000, and had reached 410 ppm by April 2018. The international consensus is that the global warming should be kept within 2 °C (UNFCCC, 2015). It is thus a matter of urgency to adopt efficient measures to reduce atmospheric CO₂ and/or ease the release of the gas in order to prevent hazardous consequences, including global warming, climate change, and ocean acidification (IPCC, 2013).

Mineral carbonation technology is one method that has been employed to permanently and safely decrease concentrations of CO₂ in the atmosphere by accelerating a nature weathering process in which CO₂ is chemically bound to calcium and/or magnesium silicates (reaction (1)) (Huijgen and Comans, 2005).



To overcome the slow reaction kinetics of the natural weathering process that occurs over thousands or even millions of years,

researchers have focused their efforts on carefully selecting process routes, process conditions, as well as pre-treatment options (Aminu et al., 2017; Boot-Handford et al., 2014; Huijgen and Comans, 2005, 2003; Olajire, 2013; Power et al., 2013; Romanov et al., 2015; Sanna et al., 2014; Sipilä et al., 2008; Yuen et al., 2016; Zevenhoven et al., 2011).

Mechanical activation, together with thermal activation and chemical activation are three classic pre-treatment options in mineral carbonation (Huijgen and Comans, 2005). All these pre-treatment methods enhance the carbonation rate of silicates through the destruction of their crystal structures (Aminu et al., 2017). Thermal activation could effectively dehydrate the hydrosilicates (i.e. serpentine), however, it does not work on igneous minerals (i.e. olivine). Also, the accurate control of the thermal activation process is necessary to prevent overheating, which induces recrystallization of minerals and greatly lowers the carbonation rate (Długogorski and Balucan, 2014). Chemical activation could effectively leach out the divalent cation from silicates, however it faces the challenge of recycling the chemicals that potentially pollute the environment (Azdarpour et al., 2015). Mechanical activation could effectively reduce the particle size of minerals and disorders their crystal structure. Mechanical activation of hydrosilicates could offer a high activation level that is similar to that of calcinations conducted with a delay of recrystallization, thus rendering

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it preferable to thermal activation (Dlugogorski and Balucan, 2014). Mechanical activation offers a fast leaching of divalent cation from silicates without adding chemicals, which saves the energy of recycling the chemicals. The only drawback of mechanical activation is the high level of operation energy consumed. However, when integrated as part of the complete milling process, energy consumption can be reduced, thus making mechanical activation a practical option (Haug et al., 2010). At present, the most effective pre-treatment method for olivine is mechanical activation in a high speed stirred mill, and the most effective pre-treatment method for serpentine is thermal activation at around 650 °C and chemical activation using $(\text{NH}_4)_2\text{SO}_4$ (Li et al., 2018). Literature related to chemical activation (pH-swing process) (Azdarpour et al., 2015) and thermal activation on serpentine (Dlugogorski and Balucan, 2014) has been reviewed in detail. However, mechanical activation of ultramafic rocks has received only simple mention in relation to mineral carbonation.

The objective of this review was to provide an up to date information on the mechanical activation of Mg-silicates for mineral carbonation. The basic theory of mechanochemistry is introduced in the first section. Then mechanical activation on Mg-Silicates with focus on olivine, serpentine and partially-serpentinized olivine are analyzed, followed by the evaluation of the carbonation technologies based on mechanically-activated Mg-Silicates. Finally, a section on the potential application in integrated mineral carbonation, limitations of the research up to now and suggestions for further development is presented.

2. Mechanical activation background

Mechanochemistry involves the application of mechanical energy to induce reactions (Avvakumov et al., 2001; Gutman, 1998). It has the same significance as thermochemistry, electrochemistry, and photochemistry in the area of physical chemistry (Baláz, 2003; Baláz, 2008; Takacs, 2013). Numerous publications on the topic of mechanochemistry have been published (Baláz et al., 2013; Braga et al., 2013; Juhász and Opoczky, 1990; Tkáčová, 1989; Zhu et al., 2013). Mechanochemistry has become one of the primary material-processing methods that is widely used in mineral processing, extractive metallurgy, pharmaceuticals, and preparative chemistry, including both organic and inorganic synthesis (Boldyrev, 2006; Boldyreva, 2013; James et al., 2012; Pourghahramani, 2007). As a sub-branch of mechanochemistry, mechanical activation (MA) also uses mechanical energy to create changes. However, instead of inducing a chemical reaction, the main purpose of mechanical activation is to enlarge the surface area and disorder the crystal structure, thus enhancing the reaction rate for subsequent processing by adding an input of energy into the system (Alex et al., 2016; Pourghahramani, 2007).

Mill grinding, which typically involves a combination of the mechanical action of pressure and shear, is the most common operation used in both conventional comminution and mechanochemistry (Boldyrev, 2006). Conventional comminution and mechanical activation are divided by grinding limit, over which the particle deformation mechanism changes from brittle to plastic (Boldyrev et al., 1996). Mills used for mechanical activation also have several times higher energy density than do conventional tumbling mills. A level of mechanical activated status can be achieved in high intensity mills, such as the planetary ball mill, the vibratory ball mill, the stirred ball mill, the motor mill, and the jet mill (Baláz, 2008; Burmeister and Kwade, 2013). The electron energy at the contact spot of colliding particles can reach more than 10 eV, which is much higher than the thermal reaction at temperatures higher than 1000 °C (4 eV) and ultraviolet photochemical (6 eV) (Wei et al., 2009). This explains why some reactions, rarely achieved through traditional methods, can occur in a mechanically-activated state.

The phenomenon of energy relaxation during mechanical activation contributes to the activation status. With regard to relaxation time, Ljachov defined two types of disorder and instability in an activated

material: short-lived defects (relaxation time 10^{-7} – 10^{-3} s) and long-lived defects (relaxation time 10^{-3} – 10^6 s) (Avvakumov et al., 2001). Short-lived defects decay immediately as they are generated. When they reach their highest level of activation, a solid can be destroyed by fracturing over a short decay time. In contrast, some long-lived defects, which are very hot and unstable, are generated as a result of deep imperfections in an activated solid. Although activation energy decreases considerably during a long-lived period of time, it never returns back to its initial state, so a residual activity (residual disorder) remains (Avvakumov et al., 2001; Baláz et al., 2013). The influence of long-lived defects is the main reason for the modification of the results in the downstream process after mechanical activation. However, the reactions during mechanical activation take advantage of both types of defects, especially the short lived defects (Pourghahramani, 2007).

The solid state properties of materials can be modified through mechanical activation. In addition to reducing particle size and increasing surface area, mechanical activation changes the characteristics of the crystal lattice. The correlation of these changes to the rate of downstream reaction can be roughly described by Eq. (1) (Tkáčová and Baláz, 1988).

$$k_0 = a + b(S_{\text{BET}}/C_{\text{XRD}}) \quad (1)$$

where, k_0 is the initial rate constant, S_{BET} is the specific surface area and C_{XRD} is the degree of crystallinity after mechanical activation. a and b are coefficients.

From the view of energy transfer, excess energy is stored in non-equilibrium and disordered crystal lattice during mechanical activation, and the activation energy of any subsequent reaction of the material is lowered (Tkáčová et al., 1993). The most suitable description for characterizing the macro energy state of activated solid (ΔH_T) equals to the sum of the energy of microstates (Eq. (2)) (Pourghahramani, 2007).

$$\Delta H_T = \Delta H_d + \Delta H_s + \Delta H_A + \Delta H_p \quad (2)$$

Where, ΔH_d is the relative lattice distortion as a measure of dislocation density (Tromans and Meech, 2001). ΔH_s is the specific surface area as a measure of the grain boundary with the surrounding medium (Heegn, 1979). ΔH_A is the formation of amorphous material (Heegn, 1979). ΔH_p is the formation of new phases such as polymorphic transformation. Other defects, especially in short-term activated states, are essential too. However, they can only be quantified theoretically and cannot be determined experimentally. At present, the existing approximation does not consider these defects (Pourghahramani, 2007).

The techniques used to characterize the mechanically-activated materials include infrared spectroscopy (IR), X-ray photoelectron spectroscopy (XPS), scanning electron microscope (SEM), scanning tunneling microscope (STM), atomic force microscope (AFM), transmission electron microscopy (TEM), X-Ray Diffraction (XRD), Mossbauer spectroscopy, and electron paramagnetic resonance (EPR), thermal gravimetric analyses (TGA), analytical electron microscopy (AEM), and electron microprobe analyses (EMPA) (Baláz, 2008).

3. Mechanical activation on magnesium silicate

Due to their abundance in nature, relative high purity and low mass requirements for binding one unit of CO_2 , olivine ($\text{Mg, Fe}_2\text{SiO}_4$) and serpentine $\text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4$ are the most suitable Ca/Mg silicate mineral feedstock for CO_2 mineral carbonation (Goff et al., 2000). This review was focused mainly on the mechanical activation of olivine, serpentine, and their mixture, because these two magnesium silicates are the most abundant magnesium silicates for mineral carbonation. As a result of the differences in the crystal structures of olivine and serpentine, the effects of their mechanical activations differ from each other. Other Ca/Mg silicates such as wollastonite CaSiO_3 , enstatite MgSiO_3 , diopside $\text{CaMgSi}_2\text{O}_6$, and talc $\text{Mg}_3\text{Si}_4\text{O}_{10}(\text{OH})_2$ also provide suitable mineral feedstock. However, the mechanical activation on these minerals lies outside of the scope of this review.

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