



Novel bead-milling mechanically pulverized bulk mineral particles to ultrafine scale: Energy storage and cleaner promotion of mineral extraction

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ABSTRACT

Novel bead-milling (BM), comprising of rough- and fine-milling processes, was developed and adopted to mechanically pulverize natural carbonate manganese (Mn_C) and zinc oxide (Zn_O). Real-time trackings showed that bulk mineral particles were finally pulverized to ultrafine scale (Z-avg hydrodynamic diameter ~244 nm for Mn_C, ~178 nm for Zn_O), implying excellent activation potential of BM. Meanwhile, tracking results strongly indicated that BM process was in the feature of initial coexisting mineral dissociation and subsequent mechanico-chemical reaction, even phase transformation. Besides initiating the decrease of mineral particle size (also the increase of surface area), fierce mechanico-chemical reaction caused the dislocation and edge/corner generation, crystalline structure destruction, which were among the effective approaches for defect formation. Note that all these processes were inherently interrelated and contributed to the energy storage of activated mineral, which was verified by the TG/FTIR characterization. The stored energy would make up for the energy required to break the lattice constraint by acidic extraction. Consistently, it was found that BM evidently promoted the Mn extraction efficiency from 46.5% to 74.8% and additional ligand complexation further promoted the extraction efficiency by another ~10%. As a result, less acid was consumed and accordingly less acidic wastes was discharged. This investigation might be enlightening for adopting proper strategies to promote the mineral extraction in a cleaner way.

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1. Introduction

Extraction was a central unit operation in the hydrometallurgical processings of different types of minerals (Crundwell, 2013). As an efficient extraction agent, acid was frequently employed to break the lattice constraint, thereby realizing the dissolution of various minerals, such as silicates (Mg₂SiO₄ (Pokrovsky and Schott, 2002), (Zn,Mn)₂SiO₄), carbonates (MnCO₃ (He et al., 2017), CaCO₃ (Liang et al., 1996)) and oxides (ZnFe₂O₄ (Muravyov et al., 2012), CuO (Majima et al., 1980)). However, though acid extraction could effectively transform the target ions from solid lattice to aqueous solution, it inevitable brought about severe combined pollution, of

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note was the heavy metal pollution (Schneider et al., 2016; Wei et al., 2016). Exposure to heavy metals (e.g., Cd²⁺, Pb²⁺, Hg²⁺, Ag⁺), even at trace levels, was believed to be a significant risk for ecosystem and human beings (Qiu et al., 2013; Chen et al., 2015). For combined pollution that induced by acid extraction was getting increasingly severer, valid technologies were developed for pollution prevention. In spite of the fact that these feasible technologies exerted satisfactory performances, they were featured to be energy/resource consuming and waste discharging (Neoh et al., 2016). The situation was getting worse with the grade of available mineral being lower due to the rapid depletion, which implied that increasingly more acids were required and correspondingly more wastes were generated (Duan et al., 2010).

The acid consumption curb emerged to be one of the most promising alternatives for pollution prevention, which would decrease the wastewater discharge at the source (He et al., 2017; Gasperi et al., 2010). The kinetics for an irreversible mineral

extraction was frequently expressed as follows (Alkattan et al., 1998):

$$\text{rate} = k[c]^n \exp(-E_A/RT) \quad (1)$$

where c represented the activity or concentration of a reactant, n the order of reaction, k the rate constant [$\text{mol}(\text{mol}/\text{m}^3)^{-n}/(\text{m}^2\text{s})$], E_A the activation energy [kJ/mol], R the gas constant [kJ/mol/K], and T the temperature [K]. Two crucial parameters were n and E_A . For most minerals, it was concluded that their extraction rates were half order in H^+ concentration (Pokrovsky and Schott, 2000). However, E_A varied distinctly, in which $E_A \leq 20$ kJ/mol suggested that the mineral extraction was diffusion controlled and the kinetics was chemical reaction controlled if $E_A \geq 40$ kJ/mol (Crundwell, 2014). In this case, rate of mineral extraction (Eq. (1)) could be accelerated by reducing the parameter of E_A with portable strategies, hence obtained higher extraction efficiency and less wastewater discharge. Mechanical activation (MA) came out to be a feasible strategy to decrease the E_A for mineral extraction (Kim et al., 2016). For example, MA was reported to decrease the E_A of indium-bearing ZnFe_2O_4 extraction from 68.8 to 44.7 kJ/mol (Zhang et al., 2010a, b). Similarly, effectiveness of MA in decreasing the E_A of rhodochrosite extraction was confirmed by us, and its significance in cutting down the wastewater discharge was forecasted (He et al., 2017). The excessive energy by MA that stored in the solid structure increased the internal entropy and energy, thus greatly enhanced the reactivities of solid particles in the acidic surroundings (Pourghahramani and Forsberg, 2006). It was reported that about 10% excessive free energy was associated with the surface area, which would lead to the surface area grew in an irreversible way (surface activation) (Sasikumar et al., 2004). In most cases, excess free energy was linked to the defect structure, where the acid attack was preferred to occur (Pourghahramani and Forsberg, 2006). Due to high feasibility and effectiveness, ball-milling was frequently adopted to mechanically crush the mineral particles (He et al., 2017; Zhang et al., 2010a, b; Pourghahramani and Forsberg, 2006). Its superiority in changing the critical structures and physicochemical properties of the solids by using friction, collision, impingement, shear and other mechanical actions was sufficiently revealed (He et al., 2017; Mulak et al., 2002). Nevertheless, ball-milling could only break the bulk particles (usually tens of microns) into the scale of several microns, which denoted that ball-milling might be limited in solid activation (Zhang et al., 2010a, b; Mulak et al., 2002). Recently, a novel bead-milling (BM) was reported to be able to comminute micro-sized aluminum trihydroxide into ~300 nm particles (Joni et al., 2010). Another study showed that BM could break conventional powdered activated carbon (typically 10–50 μm) into superfine scale (~169 nm) with entirely different morphologies (Partlan et al., 2016). Much higher activation potential of BM than that of ball-milling was reasonably speculated. If the detailed structure was considered, superfine particles had much larger fractions of atoms at edges and corners (Yang and Xie, 2006; Bian et al., 2011). These differences potentially made it easier for surface ions and small clusters to break away from the lattice structure, giving rise to enhanced solubility (Bian et al., 2011). Besides, continuous activation energy input would lead to instantaneous high temperature, which would favor the escape of ions from the mineral lattice within BM process.

Therefore, as the potential alternative to promote the mineral extraction, BM was extensively explored in the MA of manganese carbonate and zinc oxide in this study. The pH, leached ion and particle size distribution within the BM were real-time monitored and discussed, to provide sufficient cognitive into the process. The aggregation of ultra-fine particles, which was a spontaneous response to high enthalpy and free energy (Echigo et al., 2012;

Zhang et al., 2008), was mentioned and discussed. Meanwhile, the impact of BM on the physico-chemical properties of minerals was investigated in details using XRD, SEM-EDX mapping, FTIR and BET characterizations. Particularly, TG/DSC and TG/FTIR were creatively adopted to disclose the energy storage feature of mechanically-activated mineral (Garcia-Ten et al., 2011; Pan et al., 2016). Finally, extraction kinetics and efficiency of minerals before and after BM involving proton extraction and ligand complexation were compared, which would be helpful to quantify the maximal promotion capability of MA towards the mineral extraction. Ligand complexation was quite effective for mineral dissolution (Chen and Grassian, 2013; Saad et al., 2017), but its effect on thorough-mechanically activated mineral has been not yet reported.

2. Materials and methods

2.1. Reagents

All reagents (Sinopharm Chemical Reagent) were analytical grade and used without further purification. Ultrapure water (18 $\text{M}\Omega\text{cm}^{-1}$, Millipore) was used for all the experiments. The 0.45 μm PTFE syringe filters were obtained from ANPEL Scientific Instrument. Two absolutely different minerals, namely manganese carbonate (Mn_C) and zinc oxide (Zn_O), were selected for BM and subsequent experiments. The details of both ores would be presented later. Briefly, raw Mn_C was composed of kunohorite ($\text{CaMn}(\text{CO}_3)_2$) with quartz (SiO_2), gypsum ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), dolomite ($\text{CaMg}(\text{CO}_3)_2$) and magnesian ($\text{CaMg}(\text{CO}_3)_2$) as the gangue minerals. For raw Zn_O , it was mainly composed of zinc oxide (ZnO), zinc ferrite (ZnFe_2O_4) and Zn-Al composite oxide ($\text{Zn}_6\text{Al}_2\text{O}_9$).

2.2. Bead-milling and extraction experiment

Bulk Mn_C and Zn_O particles were pulverized to ultrafine scale using a novel BM (Netzsch, Germany) and schematic illustration of the experimental set-up was shown in Fig. 1. Considering the relatively larger particle size distributions of raw Mn_C and Zn_O as well as the goal to sufficiently pulverize the particles, BM process that composed of rough-grinding and fine-grinding was designed. For 1.0 mm ZrO_2 ceramic beads mediated rough-grinding, mineral and ultrapure water were added into the grinding chamber simultaneously with 30% solid/liquid ratio at a total volume of 1.0 L. As soon as rough-grinding was completed, the solid/liquid mixture underwent fine-grinding with 0.1 mm ZrO_2 ceramic beads. All the milling procedures were identical using the same flow rate, milling loading and milling rotational speed, in order to obtain steady energy transfer to the mineral particles. Particularly, by connecting the output line back to the input line, recirculation of the solid/liquid mixtures was achieved, which implied the full pulverization of mineral particles. In each pass scenario that mineral gone through input line to output line, the true milling took approximately 30 s. However, considering that all milling parameters kept identical and constant throughout the investigation, total milling time, instead of true milling time, was reasonably adopted. 3.0 h, 2.0 h were selected for rough-grinding and fine-grinding, respectively. To track the impact of gradually increased applied energy on the physico-chemical properties of minerals, solid/liquid mixtures within both the rough- and fine-grinding processes were quickly sampled at selected time intervals in the output, followed by analysis and characterization as soon as possible.

Mineral extraction was performed in a glass vessel containing 250 mL aqueous solution with the solid loading fixed to be 8.0 g L^{-1} . In order to determine the promotion efficiency of BM on mineral extraction, diluent H_2SO_4 was employed. To reveal the additional promotion of ligand complexation on mineral extraction, 0.1 mol/L

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