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Technical note

A novel recirculating reactor for the leaching of magnesia by CO₂

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Abstract

A novel slurry recirculating reactor was designed to selectively leach caustic-calcined magnesia into soluble magnesium bicarbonate with CO_2 as the leaching reagent. Compared with the reactor without slurry recirculating, the leaching rate of magnesia could be enhanced by 80%-90% in this reactor. Parameters including mass concentration of the magnesia slurry, flowrate of slurry recirculating, injection rate of CO_2 and temperature were studied with respect to their effects on the leaching kinetics. A fast leaching of caustic-calcined magnesia could be achieved with a high CO_2 -consuming efficiency of 65–70% in this reactor. © 2008 Elsevier B.V. All rights reserved.

Keywords: Leaching; Recirculating reactor, Reactor design; Caustic-calcined magnesia; CO2

1. Introduction

Magnesite is a naturally occurring mineral with rich magnesium content. The total estimated reserve of natural magnesite is about 3.65 billion tons all over the world, mainly distributed in China, Russia, Brazil, Australia, Turkey, Greece, India and USA (Di, 2001). Magnesium products, such as magnesium oxide, magnesium hydroxide and magnesium hydroxy carbonate etc, have been increasingly developed and widely used as essential components in catalysts (Kim et al., 2002; Aramendía et al., 2003; Willems et al., 2002), ceramic materials (Yu et al., 2008; Rittidech et al., 2006), superconductors (Fletcher and Leach, 1995; Bhargava et al., 1998; Ma et al., 2002), flame retardants (Rothon and Hornsby, 1996) and pharmaceuticals (Botha and Strydom, 2001; Freitag and Kleinebudde, 2003). Except for the magnesium resource in sea water such as magnesium chloride, magnesium sulphate and so on, magnesite ores with huge global reserve provides abundant raw materials for the synthesis of the mentioned magnesium products.

The first step of exploiting magnesite is usually to decompose it into magnesia with desired adsorption capacity, refractoriness and electrical insulating property by calcination at different

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temperatures, varying from 850 °C to over 2800 °C (Birchal et al., 2000; Canterford, 1985). Specifically, to synthesize magnesium products using reactive magnesia as the starting material, magnesite ores with different crystal structure are often calcined at defined temperature range to yield caustic-calcined magnesia with high chemical reactivity (Canterford et al., 1985). Calcination itself still leaves metal oxide impurities (such as SiO₂, Al₂O₃, Fe₂O₃ and CaO, etc) in the magnesia, which will become a problem for in-depth development of magnesium products. Canterford et al. reported that iron was removed by the addition of aluminium sulphate (Canterford and Moorrees, 1984). To eliminate effectively these impurities, leaching of causticcalcined magnesia by carbon dioxide is a developed technique (Canterford and Moorrees, 1985), in which MgO can be carbonized into soluble $Mg(HCO_3)_2$ by CO_2 in the presence of H_2O . SiO₂, Al₂O₃, Fe₂O₃, CaO and other impurities can be practically left undissolved or precipitated at appropriate pH values and removed from the $Mg(HCO_3)_2$ solution by simple filtration. The obtained Mg(HCO₃)₂ solution can be converted to the higher purity MgO by heating and calcinations, MgO with high purity is the major raw materials for the production of any other magnesium product. However, the consumption of CO₂ gas is so much because of its low dissolving rate that the cost is very high for industrial scale-up. Therefore, it is necessary to improve experimental apparatus for higher production efficiency. Mesci



Fig. 1. Illustration of the leaching reactor.

and Sevim employed power ultrasound in their experiment to increase dissolved fraction of Mg^{2+} in the solution (Mesci and Sevim, 2006). The application of ultrasound in commercial production will increase greatly the cost.

In our previous studies, a remodelled stirring mill was employed as the leaching reactor to improve mass transfer and chemical reactions in the multiphase system via introducing the grinding force (Zhang and Zhu, 2004). The function of the grinding media accounted largely for the enhancement of the leaching rate, but this reactor might encounter some difficulty in industrial scale-up due to the following considerations: (a) Because the grinding media were usually high-density hard balls (such as steel or zirconia balls), there would be a huge demand of electricity energy to drive them in a large-capacity stirring mill reactor at the rotational speed of several hundred rpm, which was necessary for an obvious leaching enhancement; (b) Consequently, due to the capacity limitation of individual stirring mill reactors, the total batch operation of many such reactors lowered the production efficiency. Therefore, to build a more practicable large-scale production line, the design of the leaching reactor deserved second thoughts.

In this study, a new leaching reactor, incorporating a slurry recirculating unit and a slurry/ CO_2 pre-mixer, was built and expected to achieve a swift leaching of caustic-calcined magnesia. Our aim was to test the new design and investigate the effects of several operation parameters on the leaching process, thus providing theoretical basis for its scale-up applications.

2. Experimental details

2.1. Materials

The caustic-calcined magnesia was provided by Hengxin Magnesium Industrial Company in Shandong Province, China. The magnesia powders were produced from calcining magnesite ores at 700–800 °C, with the MgO content of 86.70%, SiO₂ 7.66%, Al₂O₃ 1.45%, CaO 1.26%, Fe₂O₃ 1.18%, and trace levels of other compounds. The particle size was far from uniform, practically ranging from several microns to a few millimeters. To avoid clogging the recirculating pipeline, the magnesia powders were screened through a 60 mesh sieve to exclude those coarser than 0.26 mm from the samples before their usage in leaching experiments. It should be noted that passing through a 60 mesh sieve was not a harsh qualification for the magnesia powders used in industrial processes where Raymond mills were employed as particle pulverizers. The CO₂ gas, with a purity of 99.99%, was purchased from Microelectronics R&D Center, Chinese Academy of Sciences.

2.2. Apparatus

The sectional view of the leaching reactor (with a working volume of 1.2 L) and the photo of the pre-mixer are shown in Fig. 1, where two marked designs merit more attention.

One is the slurry/CO2 pre-mixer consisted of cylindrical glass vessel with the diameter of 50 mm and a cone glass funnel with the diameter range of 5-10 mm in it, as shown in the dashed circle, a photo and an amplified illustration next to it. In leaching experiments, the recycled magnesia slurry runs into the pre-mixer and encounters the injected CO₂ flux at the inner funnel exit. CO₂ and the slurry experience the pre-mixing as they are sprayed down the long tube of the premixer. When the mixture gushes into the bulk slurry, a secondary mixing can be achieved by the intensive agitation of the stirrer. The other designs merit is the slurry recirculating unit driven by ES-B15 electromagnetic metering pump with the maximal flow of 65 mL/min and pressure of 0.7 MPa (Iwaki Walchem), as shown in the dashed frame. After the mixture is intensively agitated in the reactor, the undissolved magnesite that remained in the mixture is carbonized again in the pre-mixer when the mixture is transported from the rector to the premixer by electromagnetic metering pump. It should be mentioned that these two units must function simultaneously in that the lack of either one would nullify the other. The flow of CO₂ gas is controlled by LZB-6 glass rotor flowmeter with inside nominal diameter of 6 mm and working pressure of 1 MPa (China Yuzhao Yinhuan Flowmeter Co., Ltd.). The temperature in the leaching reactor is kept constant by the water bath with a ± 0.1 °C fluctuation bought from Chongqing Huida Instrument Company (the shape of water bath: cylindrical, size: Φ17.5×18.5, temperature range: 5-95 °C, power: 1.5 kW). The pH value in the bulk slurry can be measured in situ by the pH meter, and the pH measurement range of Orion 310P-02 pH Meter bought from Shanghai Nano Instrument Co., Ltd is -2.000-19.999.

2.3. Leaching experiments

In a typical leaching experiment, 800 mL deionized water was added into the 1.2 L glass reactor. Then the stirrer (300 rpm) and the recycling pump were started, and the injection rate of CO_2 was regulated to the desired value (varying from 0.1 L/min to 0.3 L/min). Not until the temperature in the reactor reached the preset value were 10 g magnesia powders fed in. The main chemical reactions involved in the multiphase leaching system are as follows

$$\begin{split} & \text{CO}_2 + \text{H}_2\text{O}{\rightleftharpoons}\text{H}_2\text{CO}_3 \\ & \text{H}_2\text{CO}_3{\rightleftharpoons}\text{H}^+ + \text{HCO}_3^- \\ & \text{HCO}_3^-{\rightleftharpoons}\text{H}^+ + \text{CO}_3^{2-} \\ & \text{MgO} + 2\text{H}^+{\rightleftharpoons}\text{Mg}^{2+} + \text{H}_2\text{O} \end{split}$$

By adding them together, the total leaching process may be described by the following reaction equation

$$MgO + 2CO_2 + H_2O \rightleftharpoons Mg^{2+} + 2HCO_3^{-}$$
(1)

At certain time intervals, a 3 mL sample of the turbid slurry was drawn out from the reactor to be centrifugated for 2 min at the rotational speed of 3000 rpm. The upper clear liquids was fetched for EDTA titration method. The sample was firstly put in a conical flask, then 5 mL triethanolamine (1:3) and 10 mL ammonia–ammonium chloride buffer fluid (pH=10) were added in it. Eriochrome black T was used as indicator and the solution in conical flask became blue suddenly as stoichiometric point. The concentration of Mg²⁺ in the leachate could be determined by the consumed volume of EDTA, and the dissolved proportion of the caustic-calcined magnesia was calculated according to the ratio of the Mg content in total leachate and that in the caustic-calcined magnesia employed.

3. Results and discussion

3.1. Enhancement of leaching by recirculating and slurry/CO2 pre-mixing

To test the efficiency of the new reactor system, experiments were conducted to compare the leaching rates with and without the recirculating pump and the slurry/CO₂ pre-mixer.

The applied CO₂ injection rates were 0.1, 0.2 and 0.3 L/min respectively. The temperature in the leaching reactor was kept constant at 30.0 °C. With the initial slurry concentration of 12.5 g/L fixed, the curves of the leaching of caustic-calcined magnesia in the comparative reactors were depicted in Fig. 2. Fig. 2(a), (b) and (c) showed the conversion ratio of MgO into Mg(HCO₃)₂ within 1 h in the comparative reactors. Significant gaps between the leaching curves under each CO₂ injection condition were observed. Fig. 2(d) gave the calculated comparison of the leaching improvement at various CO₂ injection rates caused by the design of slurry recirculating and slurry/CO₂ pre-mixing.

It can be seen from Fig. 2(d) that, at lower CO_2 injection rates (0.1 and 0.2 L/min), the leaching of magnesia slurry could be improved by 80-90% in the starting period (the first 30 min). As concentration of magnesium in solution increased, the improvement dropped to around 60% after 1 h leaching. In the case of higher CO_2 injection rate (0.3 L/min), the leaching improvement was not as significant as those at lower CO_2 injection rates. The improvement in the first 15 min was slightly over 60%, and dropped to about 25% after 1 h leaching. In all cases, improvements in the leaching of magnesia had been observed, which confirmed the effectiveness of the design of slurry recirculating and slurry/ CO_2 pre-mixing in this leaching reactor.

3.2. Influence of CO_2 injection rate

It is natural that higher CO_2 injection rate will lead to higher concentration of CO_2 , which accelerates velocity of chemical reaction and leaching process. However, this also increases the production cost due to the increased consumption of CO_2 . It is supposed that there should be a balance point where both a moderate CO_2 consumption and a relatively high-speed leaching of magnesia can be achieved. To verify our supposition, leaching curves with respect to various CO_2 injection rates were compared at the same slurry recirculating flowrate of 2.5 L/ min, as shown in Fig. 3.

When the CO₂ injection rates were below 0.3 L/min (plotted in dashed lines), the leaching of magnesia did increased with the increment of CO₂ injection rate, though not very significantly. When the CO₂ injection rate was elevated to 0.4 L/min (plotted in solid line), the leaching of magnesia dropped, which was against our initial presumption. This could be explained by the different effects of slurry/CO₂ pre-mixing caused by the various CO₂ injection rates with the flowrate of slurry recirculating fixed.

At higher CO_2 injection rate, i.e. higher CO_2 pressure within the pre-mixer, the slurry stream was compressed into a line stream in the center of the CO_2 flux as shown in Fig. 4(a), which yielded a few large



Fig. 2. Leaching improvement caused by recirculating at CO₂ injection rates of (a) 0.1 L/min, (b) 0.2 L/min and (c) 0.3 L/min; and (d) is the calculated comparison of the leaching improvements.



Fig. 3. Leaching of magnesia at various CO_2 injection rates with 2.5 L/min slurry recirculating.

 CO_2 bubbles and a poor contact between the slurry and the CO_2 gas. It is also visible from the glass pre-mixer. With lower CO_2 injection rates, the slurry stream was sprayed into the CO_2 flux at the inner funnel exit of the pre-mixer and a better slurry/ CO_2 contact was achieved, as shown in Fig. 4(b). The average size of the fragmentized bubbles was very small, which not only expanded the CO_2/H_2O interface area but also lengthened the residence time of these bubbles in the reactor before they overflowed from the bulk slurry. This was favorable for a well-developed contact between CO_2 and H_2O molecules and could improve the mass transfer on the gas/liquid interface. Therefore in this reactor, increasing the injection rate of CO_2 didn't always result in fast leaching of magnesia. It could be said that only a good match between the slurry recirculating flowrate and the CO_2 injection rate can yield a high-efficiency leaching process.

In fact, when the CO₂ injection rate was 0.1 L/min, the consumed amount of CO₂ was only a little excessive compared against the content of MgO in the slurry, which could be judged by a rough stoichiometric estimation according to Eq. (1). The CO₂-consuming efficiency was high up to about 65–70% (see Appendix). Plus the fact that there was no considerable improvement of the leaching rate by increasing the feeding of CO₂, it can be concluded that the design of recirculating and slurry/CO₂ pre-mixing made this leaching reactor a cost-effective one which performed well at lower CO₂ consumption.

3.3. Influence of the recirculating speed

By altering the recycling pumps with different powers, the recycling flowrate of the slurry can be adjusted. With the CO_2 injection rate of 0.3 L/min settled, the 16 W and 35 W pumps drove the recycled slurry



Fig. 5. Leaching of magnesia at various slurry recirculating speeds.

at the flowrate of 1.1 L/min and 2.5 L/min, respectively. The leaching curves were shown in Fig. 5, which indicated a clear tendency that increasing the recycling flowrate will enhance the leaching of magnesia. The difference in the leaching rates at various flowrates of recycled slurry was also caused by the different effects of slurry/CO₂ mixing as illustrated in Fig. 4, which was again in accordance with our conclusion that a good match between the flowrates of CO₂ and recycled slurry yielded a high-efficiency leaching of magnesia.

3.4. Effect of magnesia concentration in the slurry

Considering the batch operation in the leaching of magnesia slurry, it would be a waste of labor and time if the magnesia slurry was too diluted. If the slurry was over concentrated, other problems concerning the limited solubility of Mg(HCO₃)₂ in water might occur. To find the suitable magnesia concentration, various slurries with various magnesia concentrations ranging from 7.5 g/L to 25 g/L were prepared for leaching experiments, the curves of which were given in Fig. 6.

It can be seen that the leaching curves with magnesia concentrations of 20.0 g/L and 25.0 g/L at 30.0 °C both underwent a drop of conversion ratio after their maximum values were reached. The higher the magnesia concentration, the earlier this maximum value occurred. The calculation showed that at the maximum points on these two curves, the Mg^{2+} concentrations were nearly equal (0.267 and 0.264 mol/L). This indicated that the timing of Mg^{2+} concentration drop in the slurry was dominated by the solubility limitation of $Mg(HCO_3)_2$. Afterwards, the slurries turned into viscous gel-like mixtures with an obvious decrease of freely moving water in them. We considered this as due to the hydro-



Fig. 4. States of slurry/CO2 pre-mixing at various CO2 injection rates.



Fig. 6. Leaching of magnesia with different mass concentrations of magnesia slurry at 30.0 °C.

decomposition of $Mg(HCO_3)_2$ into insoluble magnesium hydroxy carbonate, as expressed in the following reaction equation

$$(x+y)\mathrm{Mg}^{2+} + 2(x+y)\mathrm{HCO}_{3}^{-} + (z-y)\mathrm{H}_{2}\mathrm{O} \xrightarrow{\Delta} x\mathrm{MgCO}_{3}$$
$$\times y\mathrm{Mg}(\mathrm{OH})_{2}\cdot z\mathrm{H}_{2}\mathrm{O} + (2x+y)\mathrm{CO}_{2}\uparrow.$$
(2)

The generation of $xMgCO_3 \cdot yMg(OH)_2 \cdot zH_2O$ led to the decrease of soluble $Mg(HCO_3)_2$ in the leachate, which was not good for the leaching of magnesia and thus should be avoided. It should be mentioned that this phenomenon was closely related to the temperature at which the leaching of magnesia proceeded. There will be further comments concerning the effect of temperature in the following context.

3.5. Influence of temperature

Temperature was undoubtedly the most influential factor in the leaching of magnesia since it not only controlled the rate constants of the chemical reactions but also determined the mass transfer coefficients in the multiphase leaching system. We studied the effect of temperature on the leaching process using the magnesia slurry with a mass concentration of 12.5 g/L. The leaching curves at various temperatures were shown in Fig. 7.

As we can see, the leaching rate was improved with the temperature elevated from 17.0 to 30.0 °C during the whole leaching process. At 35.0 °C, the leaching curve underwent a similar change as in the case of leaching more concentrated magnesia (20.0 and 25.0 g/L) slurry at 30.0 °C as shown in Fig. 4, and the leaching ratio increased to the maximum value of 80% in the first 40 min and then dropped to 65% during the last 20 min. This was caused by the even smaller solubility of Mg(HCO₃)₂ at 35.0 °C than that at 30.0 °C, since the Mg(HCO₃)₂ was more inclined to change into insoluble magnesium hydroxy carbonate at higher temperatures. The calculated maximum Mg²⁺ concentration in the slurry at 35.0 °C was 0.217 mol/L, lower than that at 30.0 °C (0.266 mol/L).

To have a better understanding of the leaching process, pH values of the slurry were measured in situ at various leaching temperatures, as shown in Fig. 8. At the starting period of the leaching experiments, all the CO₂ solutions showed weak acidity with pH values of no less than 4.2, and a higher temperature corresponded to a higher pH value (i.e. weaker acidity) of the solution. This was because the solubility of CO₂ in H₂O decreased as the temperature was elevated. Once the magnesia powders were added into the reactor, the generated hydrogen ions were promptly consumed in the chemical reaction as expressed in



Fig. 7. Leaching of magnesia (12.5 g/L) at various temperatures.



Fig. 8. Evolution of pH value in the leaching of magnesia at various temperatures.

Eq. (1), which caused the sharp increment of pH values in the first several minutes. Then, for the rest part of the leaching experiments, the following phenomena were needed to be discussed:

- i. It was clear in Fig. 8 that the acidity of the magnesia slurry at 17.0 °C was stronger (which means the concentration of H⁺ in the slurry at 17.0 °C is higher) than those at 20.0, 25.0, 30.0 and 35.0 °C, but why was the leaching at 17.0 °C the slowest compared with others as shown in Fig. 7. This could be tentatively explained through some kinetics considerations. The rate of chemical reactions was usually determined by two factors, the concentration of reactants and the reaction rate constant. On the one hand, a higher concentration of hydrogen ions (due to the higher solubility of CO₂ in the slurry at lower temperature) favored the leaching reaction. On the other hand, the reaction rate constant was also smaller at lower temperature. When the effect of temperature on the reaction rate constant preponderated over its effect on the solubility of CO₂, the leaching curves at various temperatures exhibited a tendency as shown in Fig. 7, which answered the question.
- ii. For the leaching of magnesia at 35.0 °C, the pH value in the slurry started dropping at 28 min, which corresponded to the slowdown of the leaching reaction (but still faster than those at 17.0–30.0 °C). Under such circumstances, the hydrogen ions were not consumed in time and their accumulation led to the dropping of the pH value. Especially when the Mg(HCO₃)₂ started changing into the insoluble magnesium hydroxy carbonate, the released CO₂ from the decomposition of Mg(HCO₃)₂ also contributed to the dropping of pH value.
- iii. The leaching of magnesia at various temperatures took place in a slightly basic (nearly neutral) environment, where the main impurities of Fe₂O₃, SiO₂ and Al₂O₃ contained in the caustic-calcined magnesia didn't react with H₂CO₃ and was separated from the Mg(HCO₃)₂ solution by filtration. CaO was converted into CaCO₃, which can't be dissolved in the weak alkaline environment and therefore could also be removed from the solution by filtration. This was the mechanism how caustic-calcined magnesia could be selectively leached by CO₂.

4. Conclusions

In this study, a novel leaching reactor with the design of slurry recirculating and slurry/CO₂ pre-mixing is used to leach caustic-calcined magnesia into soluble magnesium bicarbonate. With this design, the leaching efficiency can be improved by 80-90%.

The leaching of caustic-calcined magnesia takes place in a weak alkaline (nearly neutral) environment, where the pH value in the slurry fluctuates in the range between 7.00 and 7.35. The impurities of Fe₂O₃, SiO₂, Al₂O₃ and CaO remain undissolved or precipitated during the reaction with CO₂ in such environment. A high-purity Mg(HCO₃)₂ solution can be obtained after removing the insoluble impurities by filtration.

There are two sets of parameters, (a) temperature/mass concentration of magnesia slurry and (b) flowrate of slurryrecycling/injection rate of CO_2 , which must be coordinated to make the leaching process highly efficient. The mass concentration of magnesia slurry should be reasonably high at certain temperature to avoid the hydro-decomposition of Mg(HCO₃)₂ into insoluble magnesium hydroxy carbonate. A good dispersion of CO_2 into the slurry can be achieved to facilitate the leaching process when the CO_2 is fragmentized into small bubbles by the recycled slurry stream in the pre-mixer, which can be realized by an appropriate match between the flowrate of slurry-recycling and the injection rate of CO_2 .

The design of slurry recirculating and slurry/CO₂ pre-mixing makes this reactor cost-effective in that it performs well at lower CO₂ consumption with a consuming efficiency of 65-70%.

Appendix A. Estimation of CO₂-consuming efficiency (0.1 L/min)

The CO₂-consuming efficiency (*E*) is defined as the ratio of the reacted CO₂ to the total amount of CO₂ fed into the reactor.

Given the CO₂ injection rate (v, L/min) 0.1 L/min, CO₂ pressure (P, MPa) 0.28 MPa in the pipeline, room temperature 25 °C, the molar flowrate of CO₂ (Q, mol/min) can be calculated as follows:

$$Q = \frac{Pv}{RT} = \frac{0.28 \times 10^6 \times 0.1 \times 10^{-3}}{8.314 \times (273.2 + 25.0)} = 0.0113 \text{ (mol/min)}.$$

According to Fig. 3, when the CO₂ injection rate is 0.1 L/min, the conversion ratios of magnesia (η) in the first 15 and 30 min are 27.9% and 53.1%, respectively. Given the mass concentration of magnesia slurry (*C*, g/L) 12.5 g/L, the volume of slurry (*V*, L) 0.8 L, the MgO content (*x*) in the caustic-calcined magnesia 86.7%, the molar weight of MgO (*m*, g/mol) 40.304 g/mol, the amount of generated Mg(HCO₃)₂ (*M*, mol) for 15 and 30 min can be calculated as follows:

15 min:

$$M_1 = \frac{C \times V \times x}{m} \times \eta_1 = \frac{12.5 \times 0.8 \times 0.867}{40.304} \times 0.279$$
$$= 0.060 \text{ (mol)}$$

30 min:

$$M_2 = \frac{C \times V \times x}{m} \times \eta_2 = \frac{12.5 \times 0.8 \times 0.867}{40.304} \times 0.531$$

= 0.114 (mol).

Therefore the $\rm CO_2$ -consuming efficiency after leaching for 15 and 30 min are

15 min:

$$E_1 = \frac{M_1 \times 2}{O \times 15} \times 100\% = \frac{0.060 \times 2}{0.0113 \times 15} \times 100\% = 70.8\%$$

30 min:

$$E_2 = \frac{M_2 \times 2}{Q \times 30} \times 100\% = \frac{0.114 \times 2}{0.0113 \times 30} \times 100\% = 67.3\%$$

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