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Study on the Formation of Calcium Carbonate by Carbon Sequestration of Phosphogypsum

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Currently, the utilization and treatment of solid waste resources and the recycling of greenhouse gas CO_2 need to be urgently addressed. Calcium carbonate powder was prepared by extracting Ca^{2+} from phosphogypsum, a by-product of wet-process phosphoric acid, with ammonium acetate solution and fixing CO_2 with leaching solution. The effects of ammonium acetate concentration, liquid-solid mass ratio, reaction temperature, and reaction time on the leaching rate of Ca^{2+}, and the effects of ammonia addition, CO_2 concentration, and carbonization time on the conversion rate of Ca^{2+} were systematically studied. SEM, XRD, and particle size analysis were used to analyze the morphological characteristics and formation mechanism of carbonized products under different ammonia additions and carbonization times. The results show that under the optimal conditions, the leaching rate of Ca^{2+} can reach 97.9%, the conversion rate of calcium carbonate can reach 91.78%, and the D50 of calcium carbonate powder is 14.7 μm.

Keywords: Phosphogypsum, Ammonium acetate, Calcium carbonate powder, Particle size analysis.

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INTRODUCTION

Phosphogypsum (PG) is a by-product of the wet process of phosphoric acid. Its global cumulative emissions are estimated to be about 6 billion tons[1], and the current global an*nual emissi*ons have reached 280 million tons. In the case of China, the annual PG emissions are about 70 million tons[2]. Due to the complexity of the impurity composition of phosphogypsum, its treatment and comprehensive utilization are considered a worldwide problem. The ninth meeting of the Central Financial and Economic Commission emphasized that China should strive to achieve carbon peaking by 2030 and carbon neutrality by 2060, which has a bearing on the sustainable development of the Chinese nation and the building of a community of human destiny[3]. Solid waste utilization is one of the most promising ways to reduce CO_2 emissions[4]. As one of the main means to achieve sustainable development, solid waste treatment will continue to improve the efficiency of resource utilization in the future, focusing on the development of more efficient and low-carbon energy treatment technologies[5]. Therefore, the use of industrial by-product phosphogypsum mineralization to fix CO_2 to prepare calcium carbonate powder is a ‘ win-win ‘ strategy, which has a positive effect on alleviating the problem of phosphogypsum storage and pollution, promoting the development of carbon capture and utilization technology, and protecting the atmospheric environment[6].

Before this, there have been many domestic and international reports on the preparation of calcium carbonate nanoparticles from calcium nitrate[7], limestone[8], calcium chloride[9], and other raw materials. Phosphogypsum has the advantage of low raw material cost compared with these raw materials, although it contains more types of impurities and has different impurity characteristics. Nevertheless, the purification of $CaSO_4 \cdot 2H_2O$ to a high degree of purity remains a challenging process, which in turn makes it difficult to prepare calcium carbonate of high purity from gypsum materials. The research on the preparation of calcium carbonate from phosphogypsum focuses on direct and indirect methods. Lu[10] prepared calcium carbonate with an average particle size of 86–104 nm under the conditions of 30–40 ºC and CO_2 flow rate of 251–138 mL/min by directly mixing phosphogypsum and ammonia with CO_2. Bao[11] investigated the impact of NH_3 and CO_2 pressure on the carbonation conversion of phosphogypsum. The findings revealed that the conversion rate could reach 97% within a relatively short timeframe. The disadvantage is that the researcher has certain requirements for the instrumentation used, and it is difficult to obtain calcium carbonate that is easy to prepare and at a low cost. The indirect method of extracting Ca^{2+} from gypsum involves the dissolution of acid[12, 13, 14], alkali[15, 16, 17], and salt[2, 18, 19], followed by the conversion of the resulting leachate to calcium carbonate. The acid dissolution method uses acid solution (sulfuric acid[12], nitric acid[13], hydrochloric acid[14]) to treat waste, and reacts with CO_2 or carbonate to form calcium carbonate. This method facilitates the dissolution of impurities in the gypsum, particularly those of a metallic nature, which can prove detrimental to the preparation of calcium carbonate. By adding weak alkali to remove metal ions, multiple filtration is complicated and uneconomical, and the preparation requires high corrosion-resistant equipment. The treatment of solid waste by the alkali dissolution method uses alkali solution (strong alkali NaOH[15], KOH[16, 17], weak alkali NH_4OH[15]) to form calcium carbonate with CO_2 or carbonate. Gypsum species have low solubility in weak bases and produce calcium carbonate with high purity but low reaction efficiency and low Ca^{2+} conversion. Qiao Jingyi[18] used ammonium chloride to leach phosphogypsum and remineralize CO_2 to prepare $CaCO_3$. The spherical vaterite $CaCO_3$ is prepared by controlling the conditions, but the ammonium chloride acid corrosion equipment affected the industrial utilization. Chen[20] dissolved phosphogypsum with NaCl and studied the effect of NaCl cycle times on carbonation efficiency and crystal phase. After optimization, the leaching rate of Ca^{2+} and carbonation rate are 49.42%

and 96.31%, respectively, and the particle size of calcium carbonate is 3~8 μm. Ding[20] used ammonium acetate to leach phosphogypsum. Under the optimal conditions, the leaching rate and carbonization rate reached 98.1% and 98.32%, respectively. However, the flow rate of CO_2 needs to be controlled, and the equipment requirements are high. Liu Lu[21] used sodium acetate as an additive to produce calcium carbonate under optimal conditions with 88.2% yield, 99.34% purity, and 2–9 μm particle size, but the reaction temperature is high.

The above studies show that the preparation of calcium carbonate by the salt solution method with phosphogypsum as raw material is simple, low-cost, recyclable, high carbon fixation rate, and a low leaching rate of Ca^{2+}, such as chloride salt. Ammonium acetate solution is a weak acid, weak base, and strong electrolyte, which is completely ionized in water and has a significant effect on the dissolution of gypsum in phosphogypsum[22]. The research process is carried out at ambient temperature and normal pressure, with simple operation, low equipment requirements, and low energy consumption. Ammonium acetate does not corrode the equipment, and the leaching solution can be recycled. In this paper, calcium ions in phosphogypsum and pure gypsum are leached with ammonium acetate solution, and calcium carbonate is prepared by adding ammonia and introducing CO_2. The effects of ammonium acetate concentration, liquid-solid mass ratio, reaction temperature, and reaction time on the Ca^{2+} leaching rate of phosphogypsum and pure gypsum are compared and analyzed. The effects of ammonia addition and carbonization time on Ca^{2+} conversion rate, calcium carbonate particle size distribution range, crystal type, and microstructure are also analyzed. This study is of great significance for the resource utilization of phosphogypsum solid waste as well as the utilization and sequestration of the greenhouse gas CO_2.

MATERIALS AND METHODS

Materials

The phosphogypsum used in this experiment is from the phosphogypsum yard of Hubei Yihua. Calcium sulfate dihydrate ($CaSO_4 \cdot 2H_2O$) and ammonium acetate (CH_3COONH_4) were purchased from Guoyao Group with purity ≥ 98%; Ammonium bicarbonate (NH_4HCO_3) was purchased from Comet Reagent, an analytically pure chemical reagent; Ammonia ($NH_3 \cdot H_2O$) was purchased from Cologne, purity 25%–28%; Sodium carbonate (Na_2CO_3) was purchased from Fuchen Chemical Reagent Company Limited, analytically pure chemical reagent; Disodium ethylenediaminetetraacetic acid ($C_{10}H_{14}N_2Na_2O_8 \cdot 2H_2O$), triethanolamine ($C_6H_{15}NO_3$) were purchased from Komeo Reagent, analytically pure chemical reagents; The CMP indicator was purchased from Pride; Potassium hydroxide (KOH) was purchased from Shanghai McLean Biochemical Company Limited, analytically pure chemical reagent.

Experimental Procedure

Preparation of leaching solution containing Ca^{2+}

Using single factor test, 5 g gypsum raw materials were weighed, mixed according to different liquid-solid ratios (3:1; 5:1; 10:1; 15:1; 20:1) and ammonium acetate concentrations (1 mol/L; 2 mol/L; 3 mol/L; 4 mol/L; 5 mol/L; 6 mol/L; 7 mol/L), and reacted at different temperatures (25 ºC; 45 ºC; 65 ºC; 85 ºC; 100 ºC) for different times (10 min; 20 min; 30 min; 60 min; 90 min). After cooling and filtration, the filtrate and impurities were obtained by washing the filter residue with deionized water. The leaching rate of Ca^{2+} in the filtrate was determined.

Preparation of $CaCO_3$ by carbonizing leaching solution

The single-factor test was used to add different amounts of ammonia (5 mL, 10 mL, 15 mL, 20 mL) to the Ca^{2+} leaching solution. The leaching solution was then placed in a carbonization box (HTX-12X) maintained at 20 ºC and exposed to different CO_2 concentrations (5%, 10%, 15%, 20%). Various carbonization durations (60, 90, 120, and 150 min) were also studied. The gas not only interacts with the liquid surface but also absorbed into the liquid to react. After the reaction, the solution was filtered using a 0.22 μm filter membrane to obtain a solid product of calcium carbonate, which was then dried in an oven at 100 ºC.

Experimental Setup

The chemical composition of phosphogypsum raw materials was analyzed by EDX-7000 X-ray fluorescence spectrometer (XRF) of Shimadzu Company in Japan; The particle size distribution of calcium carbonate powder was analyzed by Microtrac S3500 laser particle size analyzer; The phase of calcium carbonate powder was characterized by Japanese Rigaku SmartLab X-ray diffractometer (XRD); The crystal morphology of calcium carbonate powder was observed by JSM-IT800 scanning electron microscope (SEM).

Leaching and Conversion Rate of Ca^{2+}

Leaching rate of Ca^{2+}

In this paper, EDTA was used to titrate Ca^{2+} in the leaching solution. The experiment was repeated three times, and the average value was taken as the final reading. The molar number of Ca^{2+} in the leaching solution was obtained from the titration amount, and finally, the leaching rate of Ca^{2+} was calculated according to Formula (1).

$$p = \frac{n_2}{n_1} * 100\% \tag{1}$$

n_1 – moles of Ca^{2+} of calcium sulfate dihydrate in phosphogypsum, mol
n_2 – moles of Ca^{2+} in the leachate, mol
p – leaching rate of Ca^{2+}, %

Conversion rate of Ca^{2+}

The Ca^{2+} leaching solution after carbonization was filtered with 0.22 μm filter membrane, and the molar number of Ca^{2+} in the filtrate was determined. Finally, the conversion rate of Ca^{2+} was calculated according to Formula (2).

$$\theta = \frac{n_2 - n_3}{n_2} \times 100\% \quad (2)$$

n_3 – moles of Ca^{2+} in the carbonated filtrate, mol
θ – conversion rate of Ca^{2+}, %

RESULTS AND DISCUSSION

Leaching rate of Ca^{2+}

The Ca^{2+} leaching rate of gypsum at different CH_3COONH_4 concentrations at a reaction temperature of 25 °C, a liquid-solid mass ratio of 15:1, and a reaction time of 1h is shown in Fig. 1. With the increase of CH_3COONH_4 concentration, the Ca^{2+} leaching rate of pure gypsum and phosphogypsum increased significantly, and pure gypsum was better than phosphogypsum. CH_3COONH_4 is a weak acid, weak base, and strong electrolyte, which can be completely ionized in water, and the salt effect promotes the dissolution of phosphogypsum gypsum[22]. When the concentration of CH_3COONH_4 is 6–7 mol/L, the leaching rate of Ca^{2+} in pure gypsum does not increase. Because excessive CH_3COONH_4 makes the solution saturated to precipitate ammonium gypsum($(NH_4)_2Ca(SO_4)_2 \cdot H_2O$)[20], the content of Ca^{2+} does not increase.

The variation of the leaching rate of Ca^{2+} from gypsum with the liquid-solid ratio at a CH_3COONH_4 concentration of 7 mol/L, a temperature of 25 °C, and a time of 1 h is shown in Fig. 2. With the increase of liquid-solid mass ratio, the leaching rate of Ca^{2+} in pure gypsum and phosphogypsum increased significantly. When the liquid-solid mass ratio was 20:1, the leaching rate of Ca^{2+} could reach 89.15% and 83.75%, respectively. This is because, with the increase of ammonium acetate solution involved in the reaction, the increase of acetate ions will promote the leaching of Ca^{2+} in the system. The leaching rate of Ca^{2+} in pure gypsum is slightly higher than that in phosphogypsum, which is due to the combination of Ca^{2+} in solution with PO_4^{3-} ionized from soluble phosphate HPO_4^{2-} ionized from gypsum hydration process to form insoluble calcium phosphate[23].

Under the conditions of CH_3COONH_4 concentration of 7 mol/L, liquid-solid ratio of 20:1, and reaction time

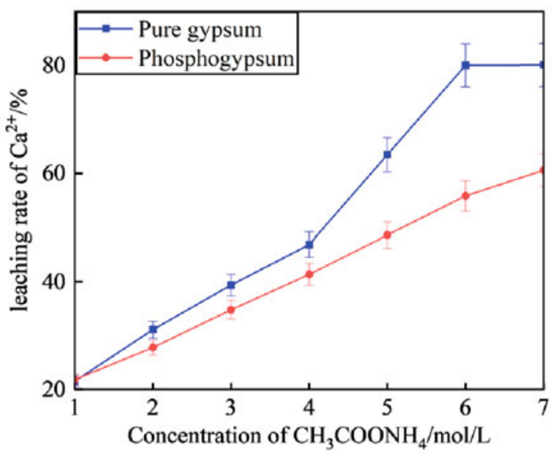

Figure 1. Ca^{2+} leaching rate of gypsum at different CH_3COONH_4 concentrations

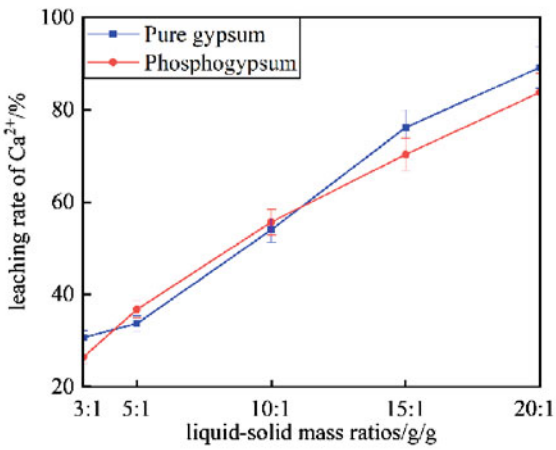

Figure 2. Ca^{2+} leaching efficiency of gypsum under different liquid-solid mass ratios

of 1h, the variation of Ca^{2+} leaching rate in gypsum with temperature is shown in Fig. 3. With the increase of reaction temperature, the leaching rate of Ca^{2+} in pure gypsum and phosphogypsum increased first and then decreased. The Ca^{2+} leaching rate in pure gypsum was 100% at 85 °C, and the Ca^{2+} leaching rate in phosphogypsum was 97.9% at 65 °C. With the increase in temperature, the dissociation degree of ammonium acetate increases, and the dissolution of gypsum increases. However, the impurities then generate ammonium gypsum to wrap phosphogypsum[24], which hinders the dissolution of calcium sulfate dihydrate, reduces the temperature requirement, and Ca^{2+} leaching is incomplete. Studies have shown that[25], Ca^{2+}, and SO_4^{2-} have a co-ion effect in the solution, hindering the dissolution of phosphogypsum. However, the dissolution of pure gypsum is not affected by this, and the solubility can reach 100%. This proves that the explanation of the ion effect is unreasonable, and the conclusion is consistent with the results of Shuang[26].

The variation of Ca^{2+} leaching rate with reaction time at 7 mol/L CH_3COONH_4, liquid-solid ratio of 20:1, and 45 °C is shown in Fig. 4. The leaching rate of Ca^{2+} in pure gypsum and phosphogypsum increased with the extension of reaction time, but it was not significant. At 60 min, the leaching rate of pure gypsum was 100%, and that of phosphogypsum was 97.9%.

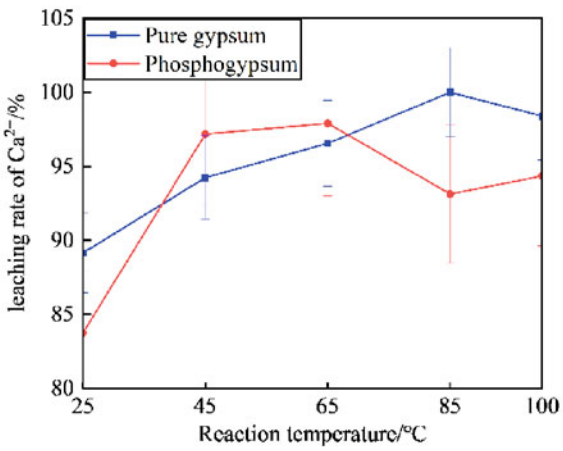

Figure 3. Leaching rate of Ca^{2+} in gypsum at different reaction temperatures

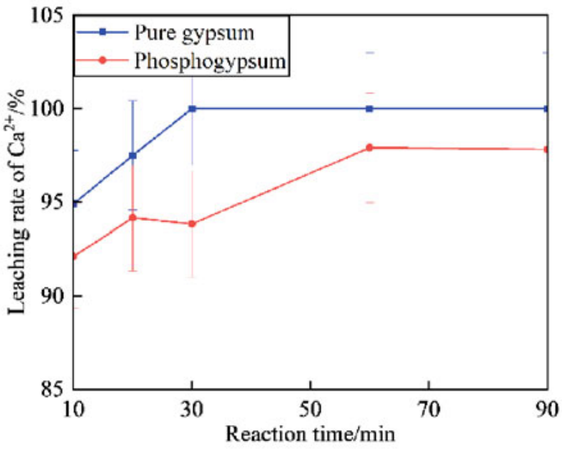

Figure 4. Leaching rate of Ca^{2+} in gypsum at different reaction times

Optimal phosphogypsum leaching conditions: ammonium acetate concentration of 7 mol/L, the liquid-solid ratio of 20:1, temperature of 65 ºC, reaction for 60 min, at this time the Ca^{2+} leaching rate of 97.9%.

The Ca^{2+} conversion rate of leaching solution under carbonation

Figure 5 shows the effect of ammonia addition on Ca^{2+} conversion at a CO_2 concentration of 20%, a temperature of 20 ºC, and a carbonization time of 150 min. With the increase in the amount of ammonia added, the Ca^{2+} conversion rate of pure gypsum and phosphogypsum leaching solution increased rapidly and then increased slowly. When the amount of ammonia added was 20 mL, the Ca^{2+} conversion rate reached 93.94% and 72.23%, respectively. At first, 5 ml ammonia was added, and Ca^{2+} was almost not converted because of the high viscosity of ammonium acetate solution, strong ion traction, limited diffusion, high ionic strength, slow diffusion, and slow reaction. The carbonation of Ca^{2+} solution is a long gas--liquid reaction. In a short period of time, the amount of ammonia added is small and the degree of ionization is weak, which can not reach the concentration required for critical supersaturation (1.5–3.0), resulting in almost no calcium carbonate precipitation. After that, increasing the amount of ammonia added increased the solubility and absorption rate of CO_2 in the solution, thereby increasing the Ca^{2+} conversion rate.

Figure 6 shows the effect of carbonization time on the conversion rate of Ca^{2+} under the conditions of ammonia addition of 20 mL, CO_2 concentration of 20%, and temperature of 20 ºC. With the increase of carbonization time, the Ca^{2+} conversion rate of pure gypsum and phosphogypsum leaching solution increased. After carbonization for 150 min, the conversions were 97.18% and 91.78%, respectively. Carbonization in ammonium acetate solution, due to the high viscosity of ammonium acetate, the mass transfer rate will slow down[27], and the entire carbonization process will be seriously slowed down. The whole reaction process did not completely end after 150 min of reaction, and it needed to be precipitated for one night before filtration.

The optimal conditions were as follows: addition of ammonium of 20 mL, CO_2 concentration of 20%, and carbonization time of 150 min. under these conditions,

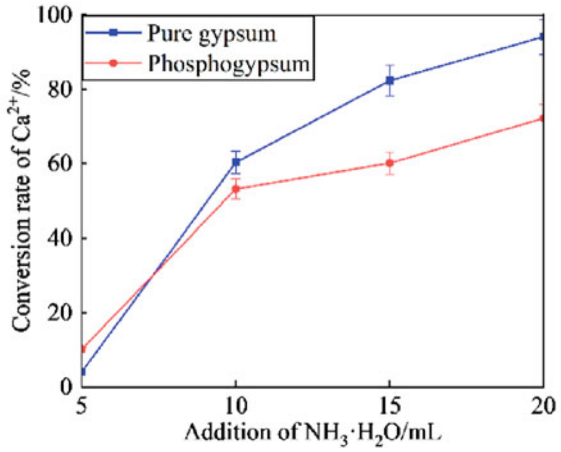

Figure 5. Ca^{2+} conversion rate of leaching solution under different ammonia addition rates

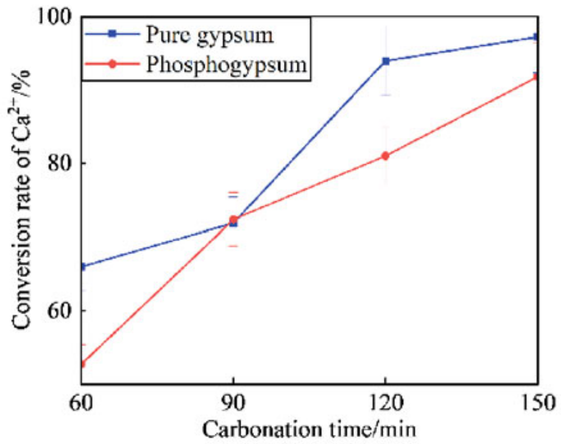

Figure 6. Ca^{2+} conversion rate of leaching solution at different carbonation times

the Ca^{2+} conversion rate was 91.78% after carbonization and standing for 24 hours.

Particle size distribution of calcium carbonate powders generated by phosphogypsum carbonation

Figure 7 shows the effect of different ammonia additions on the particle size distribution of calcium carbonate when the CO_2 concentration is 20%, the temperature is 20 ºC, and the carbonization time is 150 min. When 5mL ammonia was added, the particle size of calcium carbonate was concentrated in 10~100 μm and 500~1000 μm. When adding 10 mL, the particle size was mainly distributed in the range of 1~50 μm and 100~1000 μm, and there were two micro-convex peaks. With the increase in the amount of ammonia added, the particle size distribution of calcium carbonate is more concentrated. When the amount of ammonia added is 20 mL, the particle size of calcium carbonate micro powder is concentrated in 10~50 μm. When the addition of ammonia increased from 5 mL to 20 mL, the D_{50} of calcium carbonate were 44 μm, 18 μm, 11.75 μm and 15 μm, respectively.

The particle size of calcium carbonate was uneven and partly large when ammonia was added at 5~10 mL; the particle size distribution interval decreased at 15~20 mL. When the amount of ammonia added increases, the pH of the solution increases, which promotes the formation of carbonate ions in the solution. In addition, the supersaturation of the solution affects the particle size[28, 29]. As the

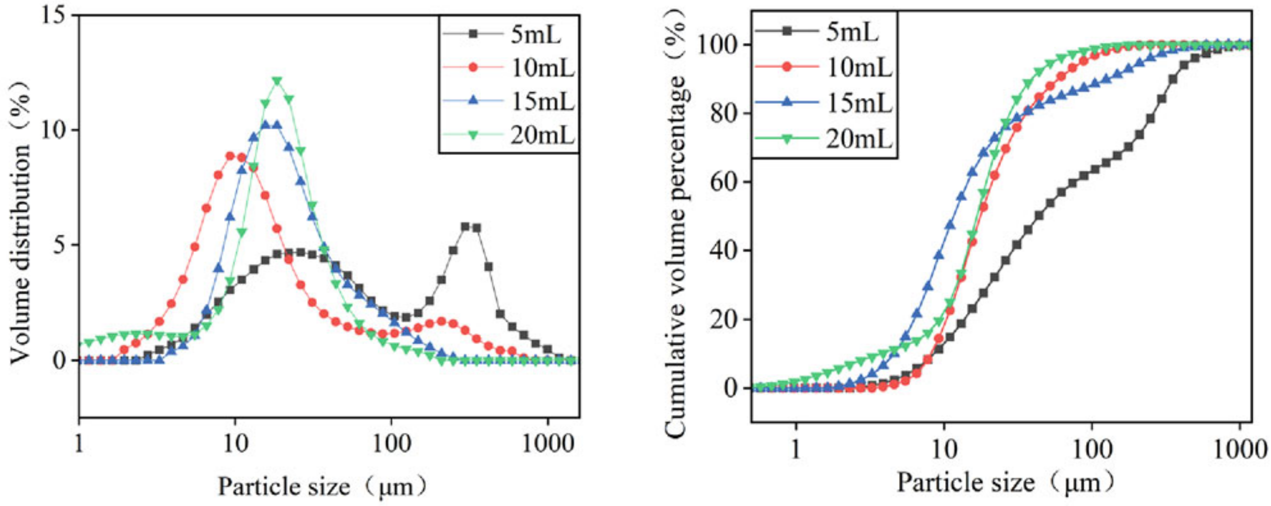

Figure 7. Particle size of calcium carbonate at different ammonia dosages

supersaturation increases, the crystal nucleation accelerates and the particle size becomes smaller[30]. However, only when the supersaturation is greater than a certain value, the effect on the crystallization rate will be accelerated. Therefore, with the increase of ammonia addition, the particle size distribution of calcium carbonate not only gradually decreases, but also the particle size distribution is concentrated in the range of 10 μm–100 μm.

Figure 8 shows the particle size distribution of calcium carbonate at different carbonization times under the conditions of ammonia of 20 mL, CO_2 concentration of 20%, and temperature of 20 °C. With the increase of carbonization time, the PPD of calcium carbonate decreased and the D_{50} decreased significantly, which were 44 μm, 33.2 μm, 25.3 μm, and 14.7 μm, respectively. When the carbonization time is 120 min and 150 min, the particle size of calcium carbonate is mainly 10–100 μm, and there are two micro-peaks. The carbonization time is prolonged, the particle size distribution is shifted to the left, and the small particles are increased.

The results show that the crystal size decreases with the increase in the concentration of the reactant solution[31]. The carbonization time is prolonged, the CO_3^{2-} concentration increases, and the solution supersaturation increases, which promotes the formation of calcium carbonate nuclei, reduces the Gibbs free energy, and improves the stability of microcrystals. The number of crystal nuclei is large, and the final particle size is small.

Microscopic morphology and composition analysis of product calcium carbonate powder

Figure 9 shows the SEM and XRD patterns of calcium carbonate carbonized for 150 min at 20% CO_2 concentration and 20 °C with different ammonia additions. Phosphogypsum was carbonized under the action of ammonium acetate and ammonia when ammonia was added at 5 mL or 10 mL at low temperature and atmospheric pressure. SEM images showed a large number of spherical particles agglomerated in the carbonation product[20]. The calcium carbonate morphology changed from uneven surface spherical to spliced spherical. When the ammonia water is added to 20 mL, as shown in Fig. 9(c), the particle size of calcium carbonate is 10–17 μm, the particles are uniform, the shape is complete, and the agglomeration is reduced. The XRD patterns were observed to have both calcite peaks and spherulite peaks at different ammonia additions.

Solution composition affects calcium carbonate crystallization. Ammonium acetate contains amino and carboxyl groups, Ca^{2+} is attracted by carboxyl groups, and CO_3^{2-} binds to Ca^{2+}. The electrolyte solution provides cohesion between the inorganic and organic layers to form double spherical calcium carbonate. Ammonia absorbs CO_2 to form CO_3^{2-}, and reacts with Ca^{2+} to form calcium carbonate crystal nucleus. After crystal growth, the alkalinity of the solution is weakened, and the nucleation rate is reduced. With the increase in the amount of ammonia,

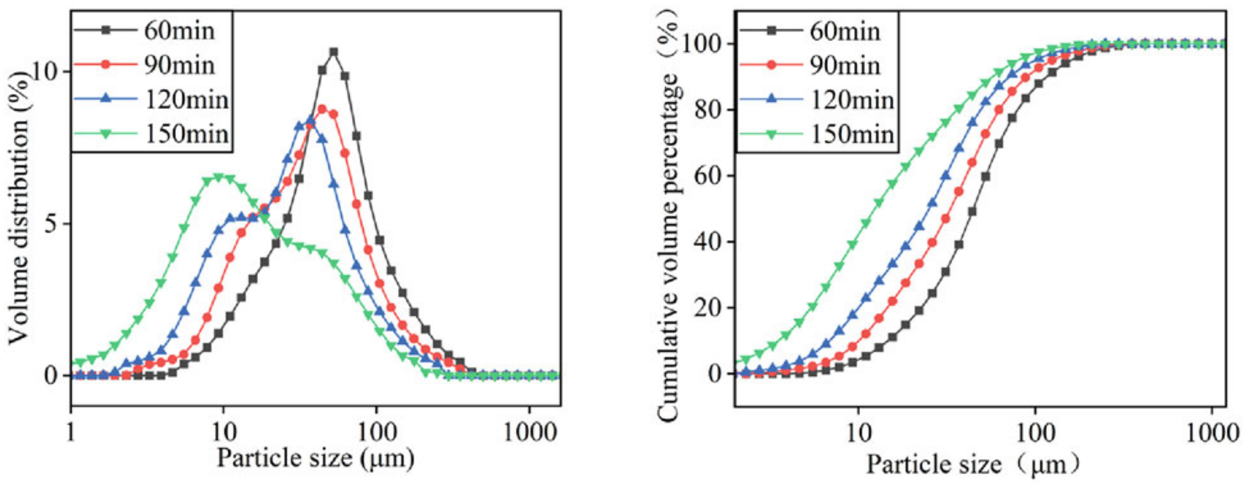

Figure 8. Particle size of calcium carbonate at different carbonization times

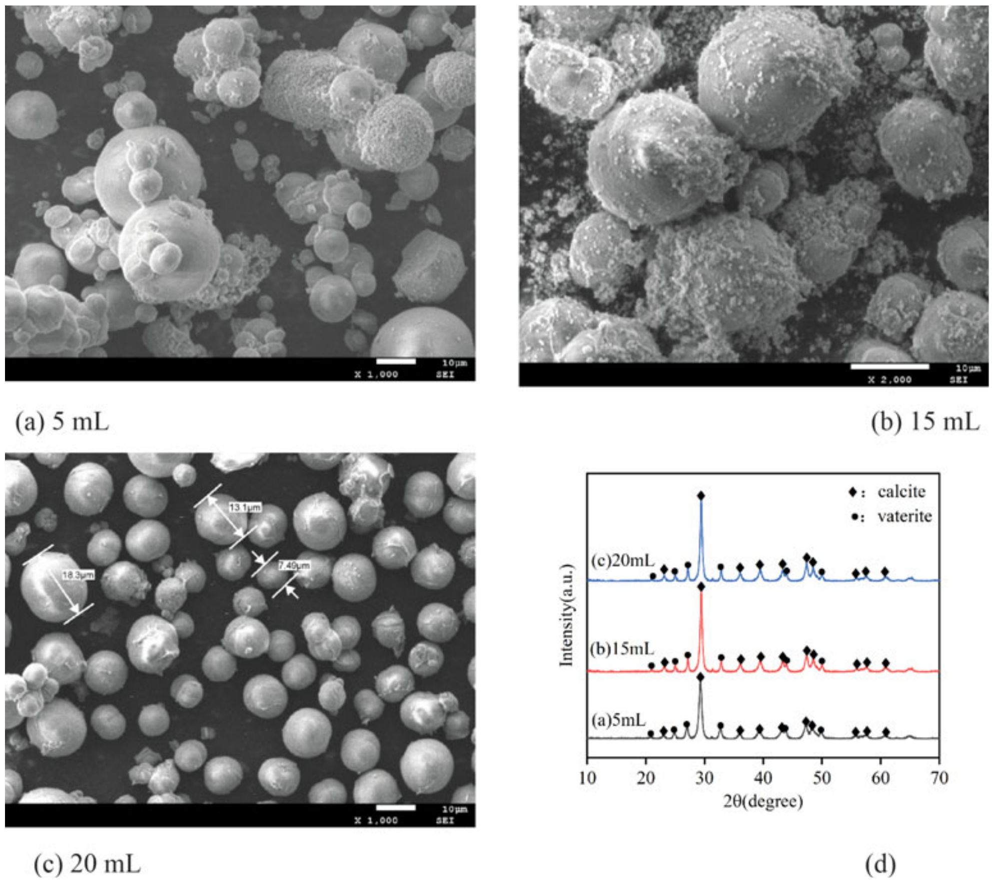

Figure 9. SEM images (a-c) and XRD patterns (d) of carbonization products under different ammonia water additions

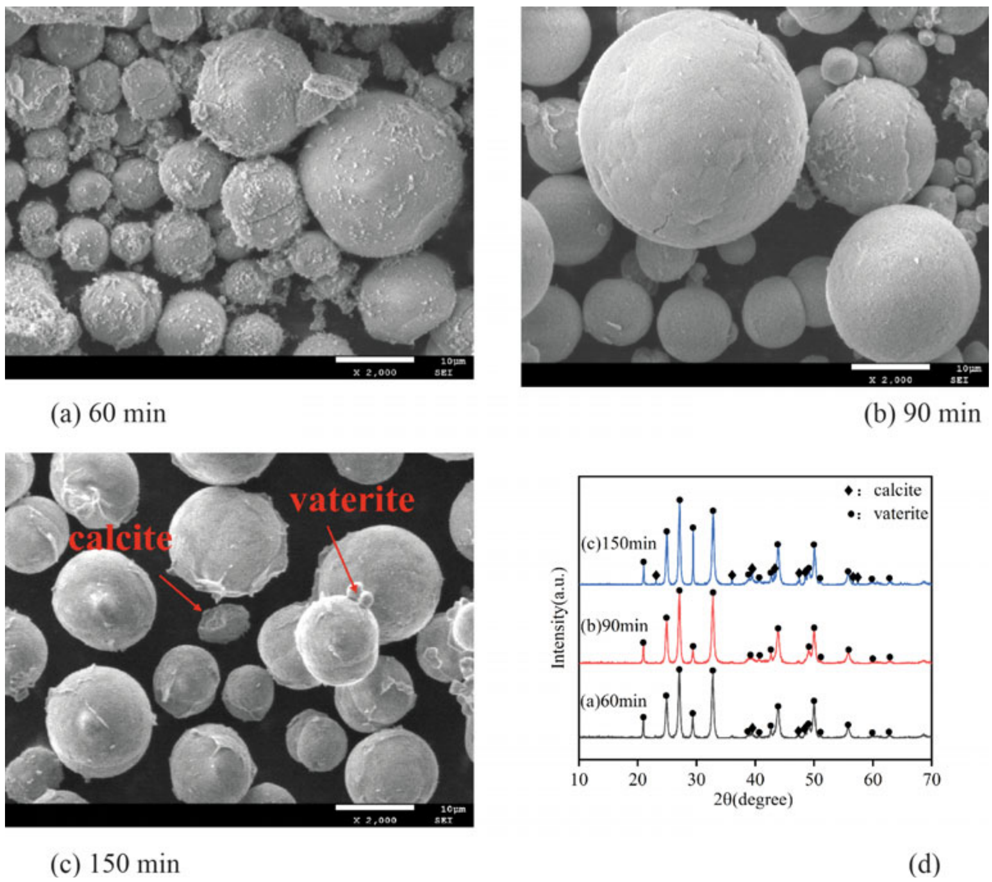

Figure 10. SEM images (a-c) and XRD patterns (d) of carbonization products at different carbonation times

the particle size of calcium carbonate decreases, which indicates that the absorption of CO_2 is accelerated, the precipitation rate is increased, and the small spherical calcium carbonate particles are formed, but the particle size is uneven. Increasing the pH value of the reaction solution promotes the dispersion of calcium carbonate particles[32] and reduces agglomeration.

Figure 10 shows the SEM images and XRD patterns of calcium carbonate with different carbonation times under the conditions of ammonia addition of 20 mL,

CO_2 concentration of 20% and temperature of 20 ºC. When the carbonization time is 60 min, the morphology of calcium carbonate is a large number of spherical vaterite and a small amount of massive calcite, both of which aggregate with each other. At the same time, the XRD pattern can be observed as most vaterite peaks and a small amount of calcite peaks. When the carbonization time increased to 90 min, the aggregation of carbonation products decreased, and the shape was complete. The only crystal form of calcium carbonate was vaterite. After the carbonization time increased to 150 min, rounded diamond calcite appeared, and some calcite peaks appeared in the XRD pattern. This is since as the reaction time increases, the less stable spherical aragonite dissolves and recrystallizes[33], producing some calcite.

Process of phosphogypsum carbonation to make calcium carbonate powder

Figure 11 shows the process of phosphogypsum carbonization to produce calcium carbonate micro powder. Ammonium acetate solution to leach Ca^{2+} in phosphogypsum, generating $Ca(CH_3COO)_2$. pumping filtration and removal of impurities to get a Ca^{2+} leaching solution, add ammonia through the CO_2 carbonization of calcium carbonate micro powder. At the beginning of carbonization, calcite, and vaterite agglomerate; after 90 min, the calcium carbonate is all vaterite, and the agglomeration is reduced; with the extension of carbonization time, vaterite dissolves and recrystallizes to form rounded diamond calcite.

CONCLUSION

When phosphogypsum was dissolved in ammonium acetate solution, the leaching rate of Ca^{2+} increased with the increase of concentration and liquid-solid ratio. When the reaction temperature increased, the leaching rate of Ca^{2+} increased first and then decreased. The leaching rate of Ca^{2+} reached 97.9 % at 65 ºC.

The conversion rate of Ca^{2+} increased with the increase of ammonia addition, CO_2 concentration, and carbonation time. When the ammonia addition was more than 10 mL, the growth rate slowed down. With the increase of ammonia, the particle size distribution of calcium carbonate decreased, and the minimum D_{50} was 11.75 µm at 15 mL.

The microstructure and composition analysis of calcium carbonate showed that the additional amount of NH_3H_2O and carbonization time had a significant effect on the particle size of calcium carbonate. When the additional amount of ammonia was 20 mL and the carbonization time was 90 min, the only crystal form of the carbonization product was vaterite.

The optimum process of preparing calcium carbonate powder from phosphogypsum is as follows : the concentration of ammonium acetate is 7 mol/L, the ratio of liquid to solid is 20:1, the reaction temperature is 65 ºC, the reaction time is 60 min, the amount of ammonia is 20 mL, the carbonization temperature is 20 ºC, the carbonization time is 150 min. Under these conditions, the leaching rate of Ca^{2+} and the conversion rate of calcium carbonate in phosphogypsum can reach 97.9% and 91.78%, respectively, and the D_{50} of calcium carbonate powder is 14.7 µm.

Data availability

Data can be available upon reasonable request to the corresponding author.

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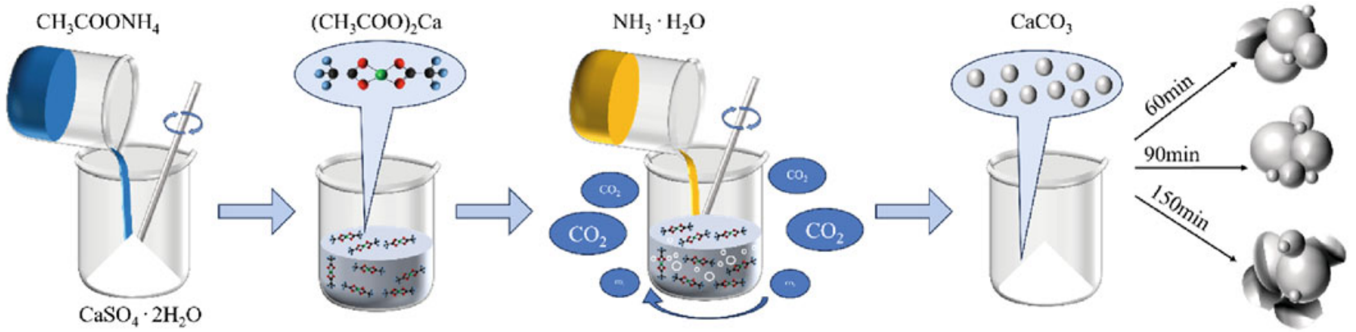

Figure 11. Process of phosphogypsum carbonization to calcium carbonate powder

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