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Nano-CaCO₃ synthesis by jet-reactor from calcium carbide slag

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Abstract

Nano-CaCO₃ was produced by using carbide slag in the self-designed jet-reactor. The effect of different operating parameter such as CO₂ flow rate and concentration, liquid flow rate and concentration of Ca(OH)₂ on the CaCO₃ particle size and morphology has been investigated in this paper. The obtained calcite particle were characterized using x-ray diffraction (XRD) and scanning electron microscopy (SEM). The results showed that the calcite about 50–200 nm was obtained under the reaction conditions of the saturation of calcium hydroxide as 1, the flow rate as 1.5 l min⁻¹, the CO₂ flow rate as 50 ml min⁻¹, the concentration as 100%, and the SDS amount 2%.

1. Introduction

Carbide slag is the waste residue produced in the process of acetylene production by calcium carbide method. Its main component is Ca(OH)₂, which is a waste with high calcium content [1–3]. At the same time, the production of calcium carbide slag continues to increase, and the slag are piled up or simply landfilled without treatment, which cause serious pollution to soil and water sources [4, 5]. Some scholars have prepared nano CaCO₃ using calcium carbide slag as raw material. For example, Lu zhongyuan et al used NH₄Cl to leach carbide slag and prepared calcium carbonate particles with good dispersion and particle size of 0.7 um under the condition of high speed stirring (>1000 rpm) [6]. Gao Chuanping et al used HCl to deal with carbide slag and obtained 80 nm CaCO₃ with 900 rpm [7]. And Nano CaCO₃ as a filler has reinforcing effect, good stability and dispersion, and non-toxic properties, which make it widely used and highly demanded in chemical industry, such as coatings, plastics, adhesives, papermaking, inks, toothpaste, cosmetics etc [8–10]. At present, the raw materials for the production of nano CaCO₃ are high-purity natural ores, such as limestone, shells, and corals and so on [11–16]. However, these valuable resources may be exhausted, due to the huge demand for the wide application of nano CaCO₃. Calcium carbide slag is a good alternative resource for calcium carbonate production because of its high yield and high calcium content. Thus how to use calcium carbide residue as raw material to prepare CaCO₃, and how to solve pollution problem and realize green production are urgent needs at present.

In industrial production of CaCO₃, it is necessary to control the optimal conditions, such as gas–liquid–solid three-phase uniform mixing, secondary nucleation, shape and particle size of CaCO₃. Therefore, it is best to use continuous method to produce nano calcium carbonate [17]. However, at present, there are very few literatures on the CaCO₃ continuous production. And only a few literatures have introduced the application of couetter-taylor reactor [18], continuous flow crystallizer [19], MSMPR removal crystallizer of mixed suspension products [20, 21], MSMPR reactor of microwave radiation source [22], and subsection flow tube reactor [23] in the production of CaCO₃. Wang Mo Jung investigated the effects of various operating conditions on the morphology and size of calcium carbonate particles produced by CO₂–Ca(OH)₂ reaction precipitation, and proposed the relative enhancement factor of operation variables in the process of calcium carbonate synthesis [18]. J Hostomsky studied the effects of residence time and pH on the particle size and agglomeration of CaCO₃ [19]. S R Shirsath compared the effects of varying calcium hydroxide slurry concentration and CO₂ flow on calcium carbonate particle size under ultrasonic and agitation conditions [17]. Hideo Watanabe studied the
effect of pH on the formation of calcium carbonate by continuously injecting CO\textsubscript{2} into CaCl\textsubscript{2} solution, and obtained hollow calcium carbonate particles \cite{24}. M M Nassar studied the effects of stirring speed and temperature on the particles and morphology of calcium carbonate by controlling the stirring and the addition of calcium nitrate, and prepared the nanoparticles and nanorods of calcium carbonate were prepared \cite{25}. Eda Ulkeryildiz used jet-flow and high-speed stirring to achieve carbon dioxide bubble refinement and separate calcium carbonate crystal zone from stable zone in the intermittent tank, and obtained nano calcium carbonate \cite{26}. Compared with these methods, the carbide slag was used to synthesis CaCO\textsubscript{3} as calcium sources in the self-designed jet-reactor. This can realize high value utilization of carbide slag without pollution. At the same time, the jet structure can make the gas-liquid mixing evenly and enhance the mass transfer between the gas and the liquid without high stirring. If the gas-liquid reaction can be completed by controlling the flow rate of reactants, the carbonization process can be realized continuously.

The size of nanoparticles depends on the nucleation rate and subsequent crystal growth rate \cite{27}. Both nucleation rate and crystal growth are related to the saturation of the reaction system. If supersaturation and nucleation rate are too high, the particles will aggregate to form irregular aggregates \cite{28}. The special structure of venturi jet throat can make the liquid cut the gas fiercely and emulsify it, forming the two-phase flow of gas-liquid emulsification, and the gas is further compressed and emulsified in the diffusion section, strengthening the mass transfer process between the gas and the solution \cite{29, 30}. In this way, the bubbles in the carbonization process can be refined to achieve the uniform mixing between gas and liquid, and provide good conditions for the synthesis of nanometer calcium carbonate.

In this paper, calcium carbonate particles were continuously synthesized by a self-designed jet reactor. The effects of different operating conditions, such as carbon dioxide flow and concentration, liquid flow and calcium hydroxide saturation, on the particle size and morphology of calcium carbonate were studied. A method is provided for the continuous preparation of calcium carbonate.

### 2. Materials and method

#### 2.1. Materials and equipment

The following are the materials and instruments used in the experiment: the gas of CO\textsubscript{2} (purity $> 99\%$, shenyang gas co.Ltd.), air, carbide slag (the main composition shown in table 1), sodium dodecyl sulfate (SDS, Chemical purity), sodium dodecylbenzene sulfonate (SDBS, chemical purity), cetyltrimethylammonium bromide (CTAB, Chemical purity), disodium ethylenediamine tetraacetic acid (EDTA, Chemical purity), glass rotameter, conduct meter, and jet-reactor.

The experimental device for the synthesis of precipitated calcium carbonate is shown in figure 1. The reaction device consists of a venturi jet reactor, a micro-diaphragm pump, a liquid storage tank, a carbon dioxide cylinder and an air cylinder. In the experiment, Ca(OH)\textsubscript{2} solution was pumped into the jet by the micro-diaphragm pump and mixed with carbon dioxide. And then the gas and liquid entered the reaction tank from the bottom with jet flow. Finally, the liquid returned to the storage tank through the upper overflow to form a
circulation. The conductivity in the storage tank was measured. The diameter of the reaction tank is 50 mm, the height is 150 mm, and the cone angle at the bottom is 60°. The jet is a 1/4-inch standard PVDF jet.

2.2. Nano-CaCO₃ synthesis
At first, sufficient amount of calcium carbide slag was leached with deionized water for 2 h and stirred at 200 rpm. Then filtered the suspension and obtained the filtrate (i.e. saturated Ca(OH)₂ solution). The 2 L solution was put into the storage tank and a certain amount of additives was added to it, with the stirring at a constant speed of 200 rpm. After stirring for 1 min, open the diaphragm pump to pump the solution into the jet, and open the gas valve to fill the jet with carbon dioxide. The progress of the reaction was detected by measuring the conductivity in the tank with a conductivity meter. The reaction ends, when the conductivity increases or does not decrease within 30 s. After the reaction ended, all the suspensions in the device were filtered and washed, and the obtained solids were dried in an oven at 80°C for 24 h to obtain calcium carbonate samples. In this experiments, carbonation was not continuous in strict sense, and all calcium carbonate was obtained after the experiment. But in the future, I will carry out experiments in a two-stage jet-reactor to realize continuous carbonation. In this paper, all the reactions were conducted at 25 ± 2°C. To investigate the effect of the designed bottom-flow non-stirred reactor on the synthesis of nano calcium carbonate, the effects of CO₂ flow rate, liquid flow rate, CO₂ concentration, Ca(OH)₂ saturation, additive amount and additive type on the morphology and particle size distribution of calcium carbonate were studied. Limited by the fluidizer, the range of liquid flow is 1.5 l min⁻¹ – 0.5 l min⁻¹, and the corresponding CO₂ flow is 25 ml min⁻¹ – 400 ml min⁻¹. The concentration of carbon dioxide is 0%–100%, the amount of additive is 2%–8% of the mass fraction of calcium hydroxide contained in the liquid, and the saturation of calcium hydroxide is 25%–1. During the experiment, the total volume of solution in the tank was 2 L.
2.3. Characterization
During the experiment, the conductivity was measured with a conductivity meter. The powder obtained from the experiment was analyzed with a powder x-ray diffractometer (XRD) at 15° to 90° with a scanning speed of 0.178° s⁻¹. And its morphology and size of calcium carbonate particles were studied by SEM (SU-8010, Hitachi, Japan).

3. Results and discussion

3.1. Analysis of conductivity in carbonization process
As is known to all, the synthesis reaction of calcium carbonate is as follows [31]:

\[
\begin{align*}
\text{Ca(OH)}_2(s) & \rightarrow \text{Ca}^{2+} + 2\text{OH}^- \quad (1) \\
\text{CO}_2(g) & \rightarrow \text{CO}_2(aq) \quad (2) \\
\text{CO}_2(aq) + \text{OH}^- (aq) & \rightarrow \text{HCO}_3^- (aq) \quad (3) \\
\text{HCO}_3^- (aq) + \text{OH}^- (aq) & \rightarrow \text{H}_2\text{O} + \text{CO}_3^{2-} (aq) \quad (4) \\
\text{Ca}^{2+}(aq) + \text{CO}_3^{2-} (aq) & \rightarrow \text{CaCO}_3(s) \quad (5) \\
\text{CaCO}_3(s) + \text{CO}_2(aq) + \text{H}_2\text{O} & \rightarrow \text{Ca}^{2+}(aq) + 2\text{HCO}_3^- (aq) \quad (6)
\end{align*}
\]

Calcium hydroxide solution was used to conduct the experiment, so there would be no reaction (1) solid dissolution of calcium hydroxide, that is, there was no dissolution of calcium hydroxide in the solution during the carbonization process. In the early stage of carbonization, the conductivity of the solution was reduced due to the continuous binding of calcium ions to carbonate ions [32–34]. After the end point of the reaction, if carbon dioxide is continued to be injected into the solution, the reaction (6) (a phenomenon of calcium carbonate regurgitation) will occur, which will increase the conductivity of the solution. Therefore, the whole process can be detected by measuring the conductivity of the solution. At the same time, the concentration of calcium ions can be estimated according to the conductivity value. Burns et al believed that the concentration of calcium ions in calcium hydroxide solution and its conductivity could be expressed as follows [35, 36]:

\[
\text{Conductivity} = 0.4268 \times \text{concentration of calcium hydroxide} \quad (7)
\]

Where conductivity is mS/cm, and concentration of calcium hydroxide is in mM. And Eda Ulkeryildiz has proved that the conductivity is linearly correlated with the concentration of calcium hydroxide, and its solubility limit is about 20 mM [26]. The maximum solubility of calcium hydroxide was 17.5 mM in the experiment, less than 20 mM, so the concentration of calcium ions could be estimated by the conductivity.

Figure 2(A) shows the change of conductivity in the tank when the CO₂ concentration was 100%, the flow rate was 50 ml min⁻¹, the liquid flow rate was 1.5 l min⁻¹, the saturation of calcium hydroxide was 1, and the additive was SDS with the addition rate of 2%. It can be observed from the figure 2(A) that the conductivity of the solution was 6.38 ms cm⁻¹, and almost linearly decreased to 115 μS cm⁻¹ after CO₂ injection. When CO₂ was added to the solution, the conductivity increased, indicating that almost all of the Ca²⁺ was consumed when the conductivity decreased to 0.115 mS cm⁻¹ at 10 min of the reaction. In the later stage, calcium carbonate is
recirculated and the conductivity increases. This result is consistent with the change of conductivity in Eda Ulkeryildiz experiment.

Combined with the change of conductivity and formula (7), the consumption rate of calcium ions can be easily obtained, and the result is shown in figure 2(B). At the same time, according to the formula (7), the initial concentration of calcium ions was about 15 mmol l⁻¹, and when the crystallization reaction stopped, the concentration of calcium ions was about 0.27 mmol l⁻¹. It can be observed from the figure 2(B) that during the crystallization stage, the consumption rate of Ca²⁺ is approximately unchanged at about 25 umol/s, which is basically consistent with the literature [31, 37–39].

3.2. XRD analysis of calcium carbonate

The phase structure of the prepared product calcium carbonate particles was investigated by x-ray diffraction pattern. Figure 3 shows the XRD diagram of calcium carbonate under the conditions of 100% CO2 concentration, 50 ml min⁻¹ CO2 flow rate, 1.5 l min⁻¹ liquid flow rate, 1 saturation of calcium hydroxide, and 2% addition of different additives. It can be seen from the figure 3 that the diffraction characteristic peaks of the calcium carbonate particles obtained under the conditions are 2θ = 23.1°, 29.4°, 36°, 39.5°, 43.2°, 47.6°, 48.6°, 57.5°, 60.7°, 64.7°. These peaks overlap with the characteristic peaks of calcite phase in the standard card 00-005-0586, indicating that the calcium carbonate obtained by jet method is calcite phase [40–42].

3.3. SEM analysis

3.3.1. Effect of carbon dioxide flow rate on calcium carbonate

The effect of the flow rate of CO2 (25–400 ml min⁻¹) on the morphology and particle size of calcium carbonate was studied when the concentration of CO2 was 100%, the liquid flow rate was 1.5 l min⁻¹, the calcium hydroxide saturation was 1 and the additive amount was 2%. Figure 4 shows SEM images of calcium carbonate particles obtained at different CO2 flows. It can be observed from the SEM images that as the CO2 flow rate increased from 25 ml min⁻¹ to 400 ml min⁻¹, the particle size gradually increased from about 200 nm to 3 μm, while the calcium carbonate grains developed more completely, and changed from ellipsoid to cubic. However, when the CO2 flow rate increased from 25 ml min⁻¹ to 50 ml min⁻¹, the particle size did not increase significantly. When the flow rate exceeded 50 ml min⁻¹, the particle size increased significantly. The venturi jet has a strong shearing and mixing effect on the gas-liquid-solid three-phase [43]. The velocity and the turbulence intensity of liquid increases suddenly in short distance in the throat exit, and then they decreased rapidly due to the mixture of gas and liquid in the diffusion section of the jet. When the mixed fluid enters the upper of the reactor, the velocity and the turbulence intensity of the mixed fluid decreases to a small value [44]. So the throat and diffusion region of the jet reactor is conducive to nucleation, and the upper area of the reactor is conducive to crystal growth. And the jet structure also makes the bubbles smaller and more uniform. So the jet makes the CO2 gas become small bubbles distributed in calcium hydroxide solution. When the CO2 flow rate is small, the calcium ions in the solution are excessive and the saturation is high enough. At this time, the CaCO3 nucleation...
occurs mainly in the solution, so the calcium carbonate particles are smaller and more dispersed. With the increase of CO₂ flow rate, the number of bubbles increases, which makes the contact area between gas and liquid increase. Consequently, the amount of CO₂ dissolved in the liquid increased per unit time. With the nucleation of CaCO₃, the concentration of calcium ions in solution decreases, and the formation of CaCO₃ changes from nucleation to growth, which shows that calcium carbonate particles become larger. Therefore, with the increase of carbon dioxide flow rate, the rapid growth of crystal nucleus in the reaction tank makes the particle size larger, and its crystallization is more complete and presents a cubic shape.

3.3.2. Effect of liquid flow rate on calcium carbonate
The effect of liquid flow rate (1.3–0.5 l min⁻¹) on the morphology and particle size was studied when the concentration of CO₂ was 100%, the flow rate was 100 ml min⁻¹, the calcium hydroxide saturation was 1 and the amount of additive was 2%. Figure 5 shows SEM images of calcium carbonate particles obtained at different liquid flows. From the SEM images, it can be observed that, as the liquid flow rate decreases, the particle size gradually increases but the increases are small, from 200 nm to 500 nm, which is only 2.5 times. The agglomeration is more and more obvious, and the diameter of agglomerated particles exceeds 4 μm. With the
decrease of liquid rate, the amount of calcium ions flowing into the reactor decreases per unit time, so does the nucleation number. Moreover, the CO$_2$ flow rate remains unchanged, that is, the dissolved CO$_2$ per unit time remains unchanged. The formation of CaCO$_3$ is mainly growth. So the particle size of CaCO$_3$ becomes larger. At the same time, with the decrease of liquid flow rate, the turbulent kinetic energy of liquid decreases exponentially. When the turbulent kinetic energy can not overcome the attraction between calcium carbonate particles, the generated calcium carbonate particles agglomerate. So the agglomeration of particles becomes more and more obvious with the decrease of liquid flow rate.

3.3.3. Effect of CO$_2$ concentration on calcium carbonate

The effects of different CO$_2$ concentrations (10%, 20%, 30%, 50%, 100%) on the morphology and particle size of calcium carbonate were studied when CO$_2$ flow rate was 200 ml min$^{-1}$, liquid flow rate was 1.5 l min$^{-1}$, the calcium hydroxide saturation was 1 and additive amount was 2%. Figure 6 shows SEM images of calcium carbonate particles obtained at different CO$_2$ concentrations. It can be seen from figure 6 that with the increase of CO$_2$ concentration, the particle size first decreases and then increases, and the agglomeration phenomenon first becomes serious and then reduces. When the CO$_2$ concentration is 20%, the particle size is the smallest, about 250 nm. At this time, the agglomeration phenomenon is the most obvious, and the aggregated large particle size exceeds 3 um. When the concentration of CO$_2$ is small, the unit dissolved amount of CO$_2$ is small. And this causes the driving force of calcium carbonate nucleation is not enough and nucleation is reduced. The formation of CaCO$_3$ is mainly growth. So the particle size is larger. With the increase of CO$_2$ concentrate, the amount of carbon dioxide in unit time increases. The driving force of nucleation increases, and more nucleation is formed. So the particle size decreases correspondingly. When the CO$_2$ concentrate is too large, the CO$_2$ is excessive which leads to a decrease of calcium ions concentrate with the nucleation of CaCO$_3$. And the formation of CaCO$_3$ changes from nucleation to growth, which shows that calcium carbonate particles become larger.

3.3.4. Effect of the additives amount on calcium carbonate

The effect of additive amount (8%, 4%, 2%, 1%, 0.5%, 0) on the morphology and particle size of calcium carbonate was studied when CO$_2$ flow rate was 100 ml min$^{-1}$, CO$_2$ concentration was 100%, the calcium hydroxide saturation was 1 and liquid flow rate was 1.5 l min$^{-1}$. Figure 7 shows SEM images of calcium carbonate particles obtained at different additive amount. As can be observed from figure 7, the particle size does not change significantly with the decrease of additive amount, and the particle size is approximately in the range of 100 nm to 300 nm. When no additives is added, the particle size is significantly increased, exceeding 500 nm, and the agglomeration is serious. The particle size after agglomeration is over 4 um. The additives can promote the nucleation and inhibit the growth of crystals [45, 46]. Therefore, adding additives can effectively reduce the size and agglomeration of particles.
3.3.5. Effect of calcium hydroxide saturation on calcium carbonate

The effect of the saturation of calcium hydroxide (0.25, 0.5, 0.75, 1) on the morphology and particle size of calcium carbonate was studied when the CO₂ flow rate was 50 ml min⁻¹, the concentration was 100%, the liquid flow rate was 1.5 l min⁻¹, and the additive amount was 2%. Figure 8 shows SEM images of calcium carbonate particles obtained under different saturation of calcium hydroxide. As can be seen from figure 8, with the saturation increase, the particle size decreases remarkably from 1.5 um to about 100 nm. That is, the higher the saturation of calcium hydroxide, the smaller the particle size of calcium carbonate. According to the nucleation theory, the smaller the concentration of solution, the greater the resistance to nucleation, the smaller the nucleation probability, and the larger the particle size of nucleation. Therefore, as the saturation of calcium hydroxide increases, the number of calcium carbonate crystal nuclei increases and the particle size of crystal nuclei decreases.

3.3.6. Effect of additive type on calcium carbonate

The effects of different additives (SDS, EDTA, SDBS, CTAB) on the morphology and particle size of calcium carbonate were studied when the flow rate of carbon dioxide was 100 ml min⁻¹, the concentration was 100%,
Figure 9 shows SEM images of calcium carbonate particles obtained with different additives. As can be seen from figure 9, EDTA as an additive made little contribution to the decrease of the particle size of calcium carbonate. SDS has the best effect, both in terms of agglomeration effect and particle size. Under the same conditions, the particle size of SDS was the smallest, ranging from 50 nm to 400 nm.

According to the above single factor experiment results, the higher the saturation of calcium hydroxide, the lower CO₂ flow rate, and the higher concentration, the larger liquid flow rate, the smaller particle size of calcium carbonate can be obtained. Thus, experiments under the following conditions were carried out. The experimental conditions were as follows: the CO₂ flow rate was 50 ml min⁻¹, the concentration was 100%, the liquid flow rate was 1.5 l min⁻¹, the calcium hydroxide saturation was 1, the additive was SDS, and the amount was 2%. The SEM result is shown in figure 10. It can be seen from figure 10 that the particle size is between 50 nm and 200 nm, of which less than 100 nm occupies the majority. Moreover, the particles have good dispersion, and there is no obvious agglomeration phenomenon, and the particles do not adhere to each other. Its XRD is shown in figure 3. Through card comparison, it can be seen that the obtained nano calcium carbonate is calcite type. And the specific surface area of the CaCO₃ synthesized under the conditions is 8.3065 m²/g. According to the results, it can be calculated that the particle size is about 266 nm.

4. Conclusion

In this work, the new reactor designed by venturi jet was used to study the influence of operating conditions such as CO₂ flow rate and concentration, liquid flow rate, calcium hydroxide saturation, additive type and amount on the particle size and morphology of nano calcium carbonate, and the continuous precipitation of nano calcium carbonate particles at room temperature is successfully achieved. When CO₂ flow rate is 50 ml min⁻¹, the concentration was 100%, the liquid flow rate was 1.5 l min⁻¹, the calcium hydroxide saturation was 1, the additive was SDS, and the addition amount was 2%, the calcite with particle size between 50 nm and 200 nm was obtained.

As the liquid flow rate increases, the shearing effect increases dramatically. And the smaller the agglomeration degree of calcium carbonate is obtained in the conditions of larger shearing. Therefore, the agglomeration degree is determined by the liquid flow rate. The flow rate and concentration of CO₂ have a great influence on the particle size of CaCO₃. The larger the flow rate of carbon dioxide, the larger the particle size of calcium carbonate is obtained. In addition, the additives can effectively reduce the particle size of calcium carbonate. The particle size of calcium carbonate is smaller as the saturation of calcium hydroxide is larger.

In view of the wide particle size distribution, the next step will be to obtain a calcium carbonate with narrow particle size distribution and better dispersion by changing the structure of the reactor and other conditions.

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