Nano-CaCO₃ synthesis by tangential jet from carbide slag

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Abstract
The nano calcium carbonate with a smaller particle size is prepared by carbide slag in the improved tangential jet-reactor, and is characterized by means of SEM, XRD, and the specific surface area. The results show that the particle agglomeration is mainly positively related to the calcium hydroxide saturation and liquid flow rate. And the particle size is correlated with liquid flow rate and carbon dioxide flow rate and concentration. And the sample obtained in the improved tangential jet-reactor has a smaller particle size than the bottom jet-reactor under the same conditions. Moreover, the specific area of calcium carbonate prepared under the optimal condition has increased from 8.3065 m² g⁻¹ to 11.3829 m² g⁻¹, and the particle size reduces from 266 nm to 199 nm by 27%.

1. Introduction
Calcium carbonate is an excellent inorganic filler, which is widely used in papermaking, plastics, food, coatings and other chemical fields [1–3]. Thus, the annual consumption of CaCO₃ is huge, and it consumed nearly 100 million tons in 2019 alone. Although shell and coral can be used as the source of calcium carbonate, the raw materials of nano calcium carbonate are high-purity natural ores (such as limestone and calcite) [4–9]. The large consumption of calcium carbonate will lead to the shortage of natural ores, and at the same time cause environmental damage, which is not conducive to the protection of local natural ecology. Therefore, it is feasible to prepare nano calcium carbonate from industrial waste residue with high calcium content, such as carbide slag. Its main components are calcium hydroxide, with a mass fraction of more than 90%. The main impurities are silica, alumina, iron oxide, volatiles, etc. Because of the impurity and calcium hydroxide similar in size and density, it is difficult to separate them by physical means. And MgO, SiO₂, Al₂O₃, and other impurities cannot be effectively removed by acid and alkali [10–12]. At present, carbide slag is simply landfilled or piled up. The high alkalinity of carbide slag will make the land and water alkalized, and the small particle size could cause air pollution [13, 14]. To synthesis nano calcium carbonate with high added value through carbide slag can not only solve the environmental pollution caused by simple accumulation or landfill of carbide slag to soil and water source, but also reduce the mining of natural ores and realize the green utilization of industrial wastes and the protection of natural resources.

There are many scholars having conducted researches on the preparation of nano calcium carbonate from carbide slag. It is common to use ammonium chloride or hydrochloric acid to treat carbide slag to obtain the solution of calcium chloride, and then use double decomposition method to product nano calcium carbonate under high-speed stirring. For example, Lu Zhongyuan used ammonium chloride to treat carbide slag and obtained 0.7 μm calcium carbonate particles at 1000 rpm [15], Gao Chuanping used hydrochloric acid to treat carbide slag and obtained 80nm calcium carbonate particles at 900 rpm. In these literatures, calcium carbonate was directly prepared by high-speed stirring, and no reactor was involved [16]. The structure of the reactor has an important influence on the mass transfer of chemical reaction. However, there are only a few literatures about the reaction equipment for the preparation of nano calcium carbonate. At present, the equipment for the preparing calcium carbonate mentioned in the literature are coetter-taylor reactor [17], continuous flow crystallizer [18], MSMPR removal crystallizer of mixed suspension products [19, 20], MSMPR reactor of
microwave radiation source [21], and subsection flow tube reactor [22], sonochemical reactor [23], jet reactor [24]. Wang Mojung studied the influence of stirring speed, flow rate, calcium hydroxide concentration and other factors on the particle size and morphology of CaCO₃ through the couette-taylor reactor, and raised the relative enhancement factor Er to reflect the particle size and morphology [17]. J Hostomsky found that the residence time of particles in the reactor, in addition to the concentration and pH value of reactants, also had an important effect on the calcium carbonate crystals during the process of preparing calcium carbonate by continuous flow crystallizer [18]. D Chakraborty found that the CaCO₃ nucleation was homogeneous in the process of preparing calcium carbonate by MSMPR, and the rate of phase transformation was positively related to the stirring speed [20]. R Vacassy prepared calcium carbonate powder with uniform particle size, due to the better plug flow obtained in the subsection flow tube reactor [22]. In the sonochemical reactor, S R Shirsath used ultrasound to break bubbles to increase the gas-liquid contact area and enhance the gas-liquid mass transfer, and prepared smaller calcium carbonate particles than those under mechanical stirring conditions. At the same time, it was found that as the flow rate of CO₂ increased, so did the particle size of CaCO₃ [23]. Eda Ülkeryıldız used a Venturi jet to break up the bubbles, and applied the mechanical stirring to disperse the calcium carbonate nucleus generated in the solution to separate the crystallization from the growth stability, and prepared the nano calcite particles with uniform size distribution [24]. It can be seen from the comparison of the above reactors that the purpose of reactor structure design is to promote the nucleation and prevent the growth of crystal nuclei. The process conditions of different reactors are suitable for the reactor structure. Therefore, on the basis of previous work [25], it is very important to optimize the jet reactor to re-study the influence of various factors on the calcium carbonate particles by changing the reactor structure.

In this paper, the self-designed jet reactor was changed from bottom flow jet to the tangential flow jet, and the effects of carbon dioxide flow and concentration, calcium ion solution flow and concentration, additive type and amount on the morphology of calcium carbonate were studied. Those experimental data would be provided to support the optimization of the jet reactor.

2. Materials and method

2.1. Materials and equipment
Carbide slag was obtained from a rubber factory in Henan Province, China, and the specific composition was shown in Table 1. Other supplies in the experiment, such as sodium dodecyl sulfate (SDS), sodium dodecyl benzene sulfonate (SDBS), ethylenediamine tetraacetic acid disodium (EDTA), and cetyltrimethylammonium bromide (CTAB), were all chemical purity, and were purchased from Sinopharm Chemical Reagent Co., Ltd. The CO₂ gas was purchased from Shenyang Gas Co., LTD, and the purity was more than 99%. The dilution gas of carbon dioxide was air.

The core equipment used in the experiment was the self-designed jet reactor (shown in figure 1), of which the jet direction, compared with the literature device [25], was not bottom jet, but horizontal along the tangent line of the circular, and the inlet of the jet was at the bottom of the reactor tank. Different from the equipment in Literature 25, after the fluid entered the reaction tank via the tangential jet, the liquid rose in a spiral pattern until it overflowed away from the top of the reactor tank to the liquid storage tank to form a circulation. While the carbon dioxide gas was confined in the axial part of the reactor tank until it left the reactor tank to the air due to inertia. The different flow patterns in the reactor tank could affect the preparation of calcium carbonate particles.

| Composition | CaO | SiO₂ | Al₂O₃ | TiO₂ | MgO | others |
|-------------|-----|------|-------|------|-----|--------|
| Content wt% | 64.39 | 3.65 | 1.26 | 0.26 | 0.34 | 30.1 |

2.2. Nano-CaCO₃ synthesis and characterization
The preparation process of nano-calcium carbonate was similar to the literature [25]. The filtrate obtained by leaching and filtering carbide slag with deionized water was placed in the storage tank with 21, and a certain amount of additive was added. The mixing speed was 200 rpm in the storage tank, and the conductivity was tested by a conductivity meter. After fully mixing, the diaphragm pump switch was turned on, the liquid flow was adjusted to a fixed valve, and then the gas flow was adjusted to the given valve. When the indication of conductivity meter did not decrease or increase within 30 s, the experiment ended. The gas supply should be stopped immediately, and the diaphragm pump switch should be closed. Then the suspension in the device was filtered, and the filter cake was washed multiple times. Finally, the washed cake was placed in an oven at 60 °C for
24 h to dry, which was the calcium carbonate powder used for XRD, SEM, and specific surface area tests. And all the experiments were carried out at 23 ± 2°C.

During the experiment, the conductivity was measured with a conductivity meter. The washed and dried powders 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), operating at an accelerating voltage of 3.0 kV and a working distance of 8.0 mm.

3. Results and discussion

3.1. Conductivity analysis in carbonization process

In the process of carbonization experiment, the calcium hydroxide solution is used to react with carbon dioxide, so the reactions are as follows [26].

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

With the gas of CO2 added, the reaction (1)–(3) are occurring, and the carbonation process begins. At the same time, the reaction (4) is also happening, i.e. calcium carbonate precipitates. And the ion concentration in the solution decreases, which leads to the change of the conductivity. Therefore, the process can be detected by the conductivity of solution [25, 27–29]. The relationship between the concentration of calcium ions and conductivity, meanwhile, can be expressed by the following formula:[25, 30, 31]

\[
\text{Cond.} = 0.4268 \times C_{\text{Ca(OH)}_2}
\]

Where cond. refers the conductivity in mS cm⁻¹, and the $C_{\text{Ca(OH)}_2}$ is the concentration of calcium hydroxide in mmol. And the conductivity of all experiments is within the range of formula.

Figure 2 draws the change of solution conductivity and reaction rate after the detected data processing under the conditions that the concentration of CO2 is 100%, the flow is 25 ml min⁻¹, the liquid flow is 1.5 l min⁻¹, and the saturation of Ca(OH)₂ is 1, and 2% SDS is added as the additive. As is shown in figure 2, the solution conductivity gradually decreases from 7.0 mS cm⁻¹ to 0.116 mS cm⁻¹ within 28 min after the CO₂ was added. During this period, the carbonization reaction rate slowly decreases, from the initial 11 μmol s⁻¹ to the final 8.5 μmol s⁻¹. The reason is that as the reaction proceeds, the calcium ions in the solution decrease, resulting in the reaction rate slowly decreasing. When the reaction time exceeds 28 min, the conductivity begins to increase, which means that the reaction (5) begins to occur, that is, the calcium carbonate begins to dissolve back. And the reaction should be stopped immediately.

3.2. XRD analysis of product calcium carbonate

The phase structure of the calcium carbonate samples was tested by x-ray diffraction pattern. The XRD results of samples prepared under the conditions of 200 ml min⁻¹ CO₂ gas, 1.5 l min⁻¹ liquid flow rate, 1 saturation of Ca(OH)₂, and 2% SDS dosage are showed in figure 3. It can be observed that the characteristic peaks of the samples overlap with those of calcite phase in the standard card 00–005–0586 [32–34]. This indicates that the calcium carbonate prepared by tangential jet reactor is calcite phase. Through the XRD results of tangential jet
and bottom jet reactor [25], the phase of calcium carbonate is independent of liquid flow rate, CO2 concentration and flow rate, the types of additive, and the reactor structure.

3.3. SEM analysis
3.3.1. Effect of CO2 flow rate on calcium carbonate

Figure 4 draws the SEM images obtained under different carbon dioxide flow rates (25–400 ml min⁻¹), and the other conditions on 100% CO2 concentration, 1.5 l min⁻¹ liquid flow rate, 1 saturation of Ca(OH)2, 2% amount of SDS. It can be noticed from the figure 4 that the calcium carbonate size slowly grows from 100 nm to 2 μm with the CO2 flow rate increasing from 25 to 400 ml min⁻¹, and the calcium carbonate nucleus gradually develops completely, i.e. the particles change from an irregular shape to cubic. The Venturi jet has strong shearing and mixing effects on gas and liquid. The calcium hydroxide and carbon dioxide gas are mixed and emulsified in the jet throat, and at the same time the nano calcium carbonate nucleus is formed. The mixed emulsion and calcium carbonate nucleus enter the reaction tank from the diffusion section of the jet, and the velocity decreases significantly, and the sharing and mixing effects are weakened suddenly. However, compared to the bottom-flow in the literature, the gas-liquid-solid three-phase in this reactor spirally rises along the wall of the reaction tank due to inertia until it leaves the reactor and enters the storage tank. This state of flow can avoid the back-mixing phenomenon of gas-liquid-solid three-phase to a certain extent. Thus, the flow of gas, liquid and solid entering the reaction tank can be regarded as plug flow. When the CO2 flow rate is 25 ml min⁻¹, the calcium ions in unit volume are excessive, so the driving force of calcium carbonate nucleation is larger, and the nucleation mainly occurs. At the same time, due to the turbulent kinetic energy and the repulsive force formed
by calcium ions attached to the particles surface [17], the CaCO₃ particles have a good dispersion and less agglomeration. With the increase of carbon dioxide flow rate, the number of bubbles gets more, and gas-liquid contact area becomes bigger. This leads to the more carbon dioxide being dissolved per unit liquid volume, which is more conductive to the growth of calcium carbonate crystal. Calcium carbonate growth means that the calcium ions on the particle surface decrease, which causes the repulsive force between particles to become weaker, and the agglomeration phenomenon between the calcium carbonate particles intensifies. Therefore, as the increase of carbon dioxide flow rate, the agglomeration of calcium carbonate particles intensifies, the particle size increases, and the crystallization is more complete, showing cubic shape.

3.3.2. Effect of liquid flow rate on calcium carbonate

Figure 5 draws the SEM images obtained under different liquid flow rates (1.3–0.5 l min⁻¹), and the other conditions on 100 ml min⁻¹ and 100% concentration of CO₂ gas, 1 saturation of Ca(OH)₂ solution, 2% amount of SDS. It can be seen from the figure 5 that the proportion of large-size particles increases, as the liquid flow rate decreases, the largest particle size is about 400 nm, and the agglomeration of particles tends to intensify. From figures 5(a) to (e), with the decrease of liquid flow rate, the primary particles spacing becomes smaller, and even the particles grow together.

As is mentioned above, the flow of jet-reactor used in the experiment can be considered as plug flow. Thus, the back-mixing phenomenon is relatively low, and the particles distribution is more uniform. With the liquid flow rate decreasing, the calcium ions per unit volume become smaller, while the CO₂ flow rate does not change. It means that the ratio of carbon dioxide to calcium ion in solution gets reduced. Therefore, the driving force for CaCO₃ nucleation becomes too weak to form new nucleus. And the crystal nucleus grows and becomes bigger due to the excess carbon dioxide. However, when the liquid is too low, the calcium ions are too seriously insufficient, so that the crystal nucleus cannot grow up, resulting in a smaller primary size. In addition, with the liquid flow rate decreasing, the liquid velocity becomes small and the turbulent kinetic energy decreases. At the same time, the calcium ions per unit volume decrease, the calcium ions on the surface of the particles decrease, and the repulsion between particles reduces sharply, due to the decrease of liquid flow rate. The agglomeration between particles is intensified, because the turbulent kinetic energy decreases and the repulsive force between particles becomes smaller. As the liquid velocity decreases, the residence time of particles in the reaction tank is longer, which is conductive to the agglomerated particles continuing to grow. And the gaps between the agglomerated particles are gradually replaced by calcium carbonate, so that a few small particles eventually form a large particle. Therefore, with the decrease of liquid flow rate, the primary particle size first increases and then decreases, and the phenomenon of particle agglomeration intensifies.

3.3.3. Effect of CO₂ concentration on calcium carbonate

Figure 6 draws the SEM images obtained under different CO₂ concentration (10%–100%), and the other conditions on 200 ml min⁻¹ of CO₂ gas, 1.5 l min⁻¹ and 1 saturation of Ca(OH)₂ solution, 2% amount of SDS. It
can be seen from the figure 6 that the particle size of calcium carbonate is the largest, about 1.5 μm, when the CO₂ concentration is 30%. As the carbon dioxide concentration increases from 10% to 50%, the particle size increases first and then decreases. And the particle size increases when the carbon dioxide concentration continues to increase. The reason is that the dissolved amount of carbon dioxide is small, resulting in the driving force for nucleation of calcium carbonate insufficient when the carbon dioxide concentration is low. So the nucleation is less, and the particles formed cannot grow up. With the CO₂ concentration increasing, the dissolved carbon dioxide increases, and it is still not enough to form more crystal nucleus. The surplus dissolved carbon dioxide promotes the growth of calcium carbonate particles, resulting in larger particle size. However, when the carbon dioxide concentration further increases, the driving force for nucleation increases, and the more nucleus of calcium carbonate is formed, which results in a decrease in the particle size of calcium carbonate. If the carbon dioxide concentration is further increased, the excess dissolved carbon dioxide cannot make the lower concentration of calcium ions nucleate, which in turn promotes the growth of calcium carbonate nucleus. Therefore, the calcium carbonate particles in figures 6 (b), (c), (e) are cubic, and the particles in figures 6 (a), (d) are not fully grown. But all of the sample are calcite (as shown in figure 3).
3.3.4. Effect of the additives amount on calcium carbonate

Figure 7 draws the SEM images obtained under different SDS amount (8%–0), and the other conditions on 100 ml min\(^{-1}\) and 100% concentration of CO\(_2\) gas, 1.5 l min\(^{-1}\) and 1 saturation of Ca(OH)\(_2\) solution. It can be seen from figure 7 that the particles size tends to increase, the proportion of large-size increases, and all particle sizes are between 50 nm and 400 nm with the decrease of SDS dosage. When the amount of SDS is 0, the particle size becomes largely apparent, between 0.5 \(\mu\)m and 1 \(\mu\)m. The reason why the proportion of large particle increases and the agglomeration between particles intensify with the amount of SDS dosage decreasing is that the additive promotes the nucleation and inhibits the crystal growth \cite{35,36}. Thus, the additive is very effective in reducing the size and the degree of agglomeration of the particles.

3.3.5. Effect of calcium hydroxide saturation on calcium carbonate

Figure 8 draws the SEM images obtained under different Ca(OH)\(_2\) saturation (0.25, 0.5, 0.75, 1), and the other conditions on 50 ml min\(^{-1}\) and 100% concentration of CO\(_2\) gas, 1.5 l min\(^{-1}\) of Ca(OH)\(_2\) solution, 2% amount of SDS. It can be seen from figure 8 that with the increase of the saturation, the particle size is significantly reduced from 2 \(\mu\)m to about 150 nm. Moreover, the agglomeration weakens as the calcium
Figure 9. SEM images of CaCO$_3$ obtained at different additives, (a) SDS, (b) EDTA, (c) SDBS, (d) CTAB.

Figure 10. SEM images and the XRD result of the CaCO$_3$ obtained at the optimal conditions.
hydroxide saturation increases. Based on the nucleation theory, the nucleation resistance becomes greater with the decrease of the calcium hydroxide saturation, which leads to the less number and bigger of the crystal nucleus. At the same time, the calcium ions on the particles surface increase with the saturation increasing, which makes the repulsion between particles become bigger. It causes the agglomeration weaker. So, the nucleus of calcium carbonate is formed more, and the nucleus size becomes smaller with the calcium hydroxide saturation increasing.

3.3.6. Effect of additive type on calcium carbonate

Figure 9 draws the SEM images obtained under different additives (SDS, EDTA, SDBS, CTAB), and the other conditions on 100 ml min\(^{-1}\) and 100% concentration of CO\(_2\) gas, 1.5 l min\(^{-1}\) and 1 saturation of Ca(OH)\(_2\) solution, 2% amount of additive. It can be seen from figure 9 that when the EDTA is used as an additive, the particle size is the largest, exceeding 1.3 \(\mu\)m, and the agglomeration phenomenon is the most serious. The other additives have a better effect on inhibiting the particle size of calcium carbonate, and the SDS and the CTBA have the best effect, followed by SDBS. In addition, it can be observed from figures 9(a), (d) that the large particles obtained after agglomeration with the SDS additive used are smaller than those with the CTAB used. Therefore, SDS has a good effect on particle agglomeration and particle size. Under this condition, the particle size of SDS is the smallest, between 50 nm and 500 nm.

Based on the experimental results, the experimental results of the optimal conditions, which are 25 ml min\(^{-1}\) and 100% concentration of CO\(_2\) gas, 1.5 l min\(^{-1}\) and 1 saturation of calcium hydroxide solution, 2% dosage of SDS, are shown in figure 10. The SEM images show that the particle size is between 50 nm and 150 nm, and the majority is less than 100 nm. Moreover, the particles have good dispersibility, no obvious agglomeration, and no adhesion between the particles. It can be seen from figure 10(c) that the calcium carbonate obtained is calcite by comparing the standard card. The specific surface area of the calcium carbonate sample is 11.3829 m\(^2\) g\(^{-1}\), and the average particle size, calculated through the formula of the cubic specific surface area, is about 193 nm, which is 27% smaller than that of the 266 nm calcium carbonate obtained by the bottom flow jet [25]. When the jet direction is changed from bottom to tangential, the back mixing is avoided. Meanwhile, the fluid in the reactor tank moves in a spiral way, due to inertia, the solid particles tend to move in large circular sizes, and the gas is confined to the axial part. This to some extent achieves the separation of calcium carbonate particles from carbon dioxide gas, resulting in smaller calcium carbonate particles.

4. Conclusion

In this paper, the effects of various single factors on the morphology, particle size and crystal form of calcium carbonate are studied by changing the reactor structure. It is found that the particle size of calcium carbonate obtained by the tangential jet is smaller than that by the bottom jet, and the average particle size of 199 nm nano calcium obtained by the tangential jet is 27% smaller than the 266 nm calcium carbonate obtained by the bottom flow jet under the optimal conditions. This shows that the reactor structure has a great impact on the particle size of calcium carbonate. In addition, the calcium carbonate obtained is calcite under these conditions with different jet-reactors.

The results in the paper show that the agglomeration degree of calcium carbonate is related to the liquid flow rate and the calcium hydroxide saturation. The larger the liquid flow rate is, the higher the saturation is, and the smaller the agglomeration degree of calcium carbonate is. The particle size is related to the liquid flow rate, the CO\(_2\) flow rate and concentration, and the calcium hydroxide saturation. The higher the liquid flow rate and the saturation are, the smaller the particle size is. There is an optimal condition for the CO\(_2\) flow rate and concentration, and the particle size would become bigger if the CO\(_2\) flow rate and concentration are greater than or less than this condition. Additives can effectively reduce the particle size and make the agglomeration weaker. And the nano calcium carbonate obtained has a particle size range from 50 nm to 150 nm, a specific surface area of 11.3829 m\(^2\) g\(^{-1}\) under the optimal conditions, which are 25 ml min\(^{-1}\) and 100% concentration of CO\(_2\), 1.5 l min\(^{-1}\) and 1 saturation of calcium hydroxide solution, 2% SDS dosage.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

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