微型Y型撞击流混合器强化快速沉淀反应可控制备碳酸锶微球

姚瀚植 游 攀 张俊杰 骆培成*
(东南大学化学化工学院,南京 211189)

摘要: 采用微型Y型撞击流混合器提高混合效率,利用共沉淀法制备碳酸锶($SrCO_3$)微球。采用乙二胺四乙酸二钠(EDTA)作为添加剂控制颗粒形貌。考察了EDTA浓度、反应物浓度对颗粒形貌和粒径分布(PSD)的影响。实验结果表明,以EDTA为添加剂,可以得到正交晶型球形$SrCO_3$颗粒。EDTA与氯化锶($SrCl_2$)的物质的量浓度之比R_E是影响颗粒形貌和粒径的关键因素。当R_E固定时,反应物浓度对形貌和粒径影响不大。在最佳条件下制得的微球粒径为2~3 μm,PSD非常窄。未添加EDTA时,只得到杆状颗粒,颗粒倾向于聚集成束。对EDTA调控$SrCO_3$形貌的机理进行了探讨。

关键词: 碳酸锶; 混合; 沉淀反应; Y型撞击流混合器; 形貌
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Controllable Preparation of Strontium Carbonate Microspheres by Fast Precipitation Reaction in a Miniature Y-Jet Mixer

YAO Han-Zhi YOU Pan ZHANG Jun-Jie LUO Pei-Cheng*
(*School of Chemistry & Chemical Engineering, Southeast University, Nanjing* 211189, *China*)

Abstract: In this work, a miniature Y-jet mixer was used to enhance the mixing efficiency to prepare strontium carbonate ($SrCO_3$) microspheres by using the co-precipitation method. Ethylenediamine tetraacetic acid disodium (EDTA) was used as the additive to control particle morphology. The effects of EDTA concentration and reactant concentrations on the morphology and particle size distribution (PSD) were investigated. Experimental results demonstrate that orthorhombic-type spherical $SrCO_3$ particles were obtained by using EDTA as an additive. The molar concentration ratio of EDTA to strontium chloride ($SrCl_2$), R_E, is the key factor affecting the particle morphology and size. When R_E was fixed, reactant concentrations had little effect on the morphology and size. The microspheres prepared under the optimum conditions were 2-3 μm with a very narrow PSD. Without EDTA, only rod-shaped particles were prepared and the particles tend to aggregate into bundles. The mechanism of how EDTA regulates the morphology is also discussed.

Keywords: strontium carbonate; mixing; precipitation reaction; Y-jet mixer; morphology

0 Introduction

As an important inorganic chemical raw material, strontium carbonate ($SrCO_3$) is widely used in the production of electronics, ceramics, and coating[1]. In recent years, the rapid development of the electronics industry has brought new opportunities and challenges to the development and utilization of $SrCO_3$ products. Among them, using various additives to synthesize functionalized $SrCO_3$ products with special forms,

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*通信联系人。E-mail:luopeicheng@seu.edu.cn

thereby giving them better application performance, has become an attractive research topic[1]. The morphologies of $SrCO_3$ prepared so far include spherical, needle, spindle, flake, dumbbell, olive, peanut, bunch, rod, *etc*., among which spherical $SrCO_3$ has attracted more and more attention due to its wide applications in electronic ceramics, catalytic support, and other fields.

In the past few years, many scholars have successfully prepared spherical $SrCO_3$ particles using a variety of additives as morphology control reagents. For example, Yu et al.[2] used poly-(stryrene-alt-maleic acid) (PSMA) as an additive to prepare spherical $SrCO_3$ with good dispersion. Zou et al.[3] prepared spherical $SrCO_3$ by gas-liquid precipitation method. Liu et al.[4] synthesized micron-sized $SrCO_3$ microspheres using phthalic acid and isophthalic acid as regulators. Du et al.[5] synthesized $SrCO_3$ microspheres with a particle size of 200-400 nm, using ionic liquid (1, 1, 3, 3-tetramylguanidylate) as a solvent and $SrCl_2·6H_2O$, NaOH, and CO_2 as raw materials. To avoid using expensive ionic liquid solvents, Cao et al.[6] prepared spherical $SrCO_3$ particles by hydrothermal method using cetyltrimethylammonium bromide (CTAB) as a soft template. These pioneering works have strongly promoted the development of spherical $SrCO_3$ preparation technologies. However, developing a high-efficiency and low-cost technology that can be used for large-scale industrial production remains a major goal.

Co-precipitation reaction of cationic salt solution and carbonate solution is a popular method to prepare micron-sized or nano-sized particles, in which the mixing efficiency of the reactants plays a vital role in the regulation of supersaturation level and its distribution in the crystallizer. Thus, many studies have focused on improving the mixing efficiency of the precipitation equipment to prepare particles with specific particle size, particle size distribution (PSD), and morphology. The crystallizers with mixing intensification studied are T-jet mixer[7-9], Y-jet mixer[10-12], impinging stream jet reactor[13-14], vortex reactor[15-16], multi-entry vortex reactor[17-18], rotating disc reactor[19-20], rotating liquid membrane reactor[21], multi-orifice impinging transverse (MOIT) jet mixer[22], among which the Y-jet mixer is a preferred device, due to not only its simple structure but also a rapid generation of high supersaturation, thereby producing nano-sized or micron-sized particles with a narrow PSD[11].

In this study, a miniature Y-jet mixer was used as the crystallizer to prepare micron-sized $SrCO_3$ particles by co-precipitation reaction of aqueous solutions of strontium chloride ($SrCl_2$) and sodium carbonate (Na_2CO_3). EDTA was used as the additive and its effect on the morphology of the prepared particles was discussed. We intend to study the effects of EDTA concentration (c_{EDTA}), and reactant concentrations on the morphology and PSD of the prepared $SrCO_3$ particles and explore how EDTA regulates the morphology of $SrCO_3$ particles in the miniature Y-jet mixer.

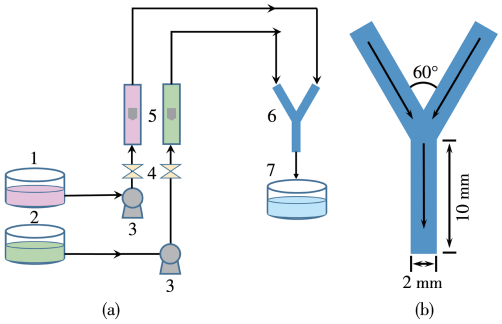
1 Experimental

1.1 Materials

Analytical grade $SrCl_2$ (99%, Shanghai D&B Biological Science and Technology Company), Na_2CO_3 (99.8%, Shanghai MERYER Company), EDTA (99%, Sinopharm Chemical Reagent Company), and distilled water were used to prepare $SrCO_3$ particles.

1.2 Apparatus and methods

The experiment was carried out at 25 ℃. In the experiment, an aqueous solution of $SrCl_2$ with (or without) EDTA was used as solution A and an aqueous solution of Na_2CO_3 as solution B. The schematic diagram of the experimental setup is shown in Fig. 1a. Two reactant fluids enter the Y-jet mixer at the same molar concentration and flow rate by gear pumps. Fig. 1b shows the configuration of the miniature Y-jet mixer adopted in this work. The inner diameters of the two inlet pipes and the mixing pipe were all 2 mm and the length of the mixing pipe was 10 mm. The angle of collision between two inlet streams was 60°. The flow rates of the two streams were all fixed at 100 mL·min^{-1}. The Reynolds number of the mixed stream in the mixing pipe, was 1 056, indicating that the flow belongs to the laminar regime. The precipitation process was carried out at room temperature. The concentrations of $SrCl_2$ (c_s) and Na_2CO_3 (c_c) remained the same, whereas the molar concentration ratio of EDTA to $SrCl_2$ ($R_E=c_{EDTA}/c_s$)



1: Feed tank for Solution A; 2: Feed tank for Solution B; 3: Gear pumps; 4: Valves; 5: Flowmeters; 6: Y-jet mixer; 7: Product collection tank

Fig.1　(a) Experimental setup of the co-precipitation process in (b) the miniature Y-jet mixer

was changed. $SrCO_3$ suspension was collected and rinsed several times with distilled water and pure ethanol, respectively. Wet particles were then dried in a vacuum drying oven at 80 ℃ for 24 h.

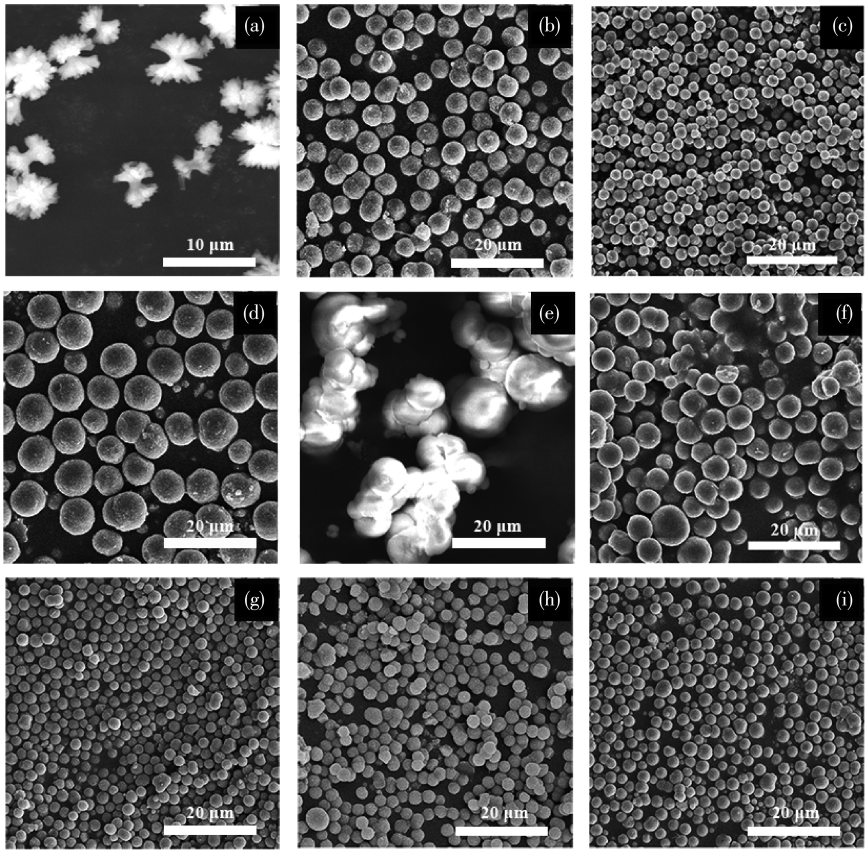
1.3　Characterization of $SrCO_3$ particles

The multifunctional energy-level X-ray powder diffractometer (XRD, Ultima Ⅳ) was used to identify the phase structure and purity of the $SrCO_3$ particles, with Cu $K\alpha$ (λ=0.154 06 nm) radiation at a scan rate of 0.02 (°)·s^{-1}. The accelerating voltage and applied current were 36 kV and 20 mA, respectively. The scanning electron microscope (SEM, Nova NaNoSEM450) was used to measure the morphology of the $SrCO_3$ particles, with an acceleration of voltage at 15 kV. PSD and volume-weighted average particle size (d_{43}) were measured by the Malvern Mastersizer 3000 particle size analyzer (MAN0475-06-EN-00).

2　Results and discussion

2.1　Morphology and crystal form

The effect of the amount of EDTA added on the morphology of the prepared particles was first studied. It is seen that the prepared $SrCO_3$ particles are bundle-like structures with a particle size of about 5 μm when EDTA was not added, as shown in Fig. 2a. When the



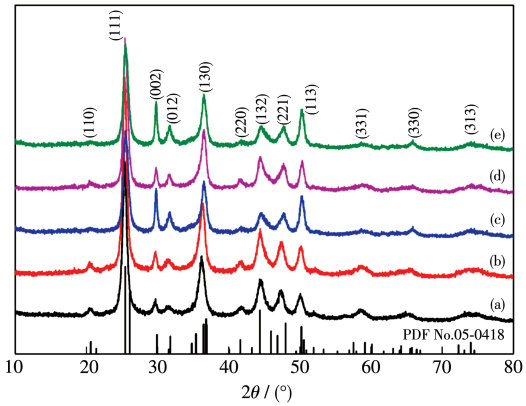
(a-e) c_s=0.1 mol·L^{-1}; (a) R_E=0; (b) R_E=0.1; (c) R_E=0.2; (d) R_E=0.3; (e) R_E=0.4; (f) c_s=0.2 mol·L^{-1}, R_E=0.1; (g) c_s=0.15 mol·L^{-1}, R_E=0.2; (h) c_s=0.2 mol·L^{-1}, R_E=0.2; (i) c_s=0.25 mol·L^{-1}, R_E=0.2

Fig.2　SEM images of $SrCO_3$ particles prepared under different conditions

reactant concentrations were kept constant (c_s=c_c=0.1 mol·L^{-1}) and a certain amount of EDTA was added, spherical $SrCO_3$ particles were obtained, as shown in Fig.2b-2d. Moreover, no obvious aggregation between the primary particles was observed, implying that the prepared particles are expected to have good dispersion in the liquid in practical applications. When R_E was increased, it can be seen intuitively from the images that the size of $SrCO_3$ particles decreased first and then increased. When c_{EDTA} exceeded 0.04 mol·L^{-1}, the precipitation reaction process slows down significantly, *i.e.* particulate matter in the suspension collected from the outlet of the Y-jet mixer was significantly reduced. In addition, serious aggregation between $SrCO_3$ particles was observed by SEM (Fig.2e).

The concentration of the reactants is another important factor affecting the precipitation process. At a lower concentration of the reactants, almost no solid particles were formed due to low supersaturation levels. Thus, c_s and c_c were first increased to 0.2 mol·L^{-1} with a lower R_E of 0.1. SEM image in Fig.2f shows that the particle size is similar to that in the case of c_s= 0.1 mol·L^{-1} and R_E=0.1 (Fig.2b). This indicates that the R_E of EDTA to $SrCl_2$ has a greater effect on the particle size than the reactant concentrations. To have a further check of this, R_E was fixed at 0.2, and c_s were changed from 0.1 to 0.25 mol·L^{-1}. From Fig.2g to 2i, the morphology and particle size of $SrCO_3$ particles obtained were very similar. This confirms that the molar concentration ratio of EDTA to $SrCl_2$ is the key factor affecting the particle morphology and size.

XRD measurements were then carried out to determine the crystal form of the prepared $SrCO_3$ particles. Fig.3 shows that the diffraction peaks of the prepared particles under different conditions are all consistent with those (110), (111), (221), (113), (132), and (002) crystal faces, which are characteristic peaks of orthorhombic $SrCO_3$ crystal (PDF No.05-0418). Moreover, there was no impurity peak, implying that the prepared $SrCO_3$ particles are very pure.



Conditions: c_s=0.1 mol·L^{-1}; (a) R_E=0; (b) R_E=0.1; (c) R_E=0.2; (d) R_E=0.3; (e) R_E=0.4

Fig.3 XRD patterns of $SrCO_3$ particles

2.2 Particle size distribution

From Fig.2, it is also seen that the amount of EDTA added has a significant effect on the size of the prepared particles. To have a quantitative analysis, the PSD profiles from experiments by using Malvern Mastersizer 3000 are compared in Fig.4. When EDTA was not added or at a low concentration (R_E=0 or 0.1), the PSD profiles exhibited a bimodal distribution.

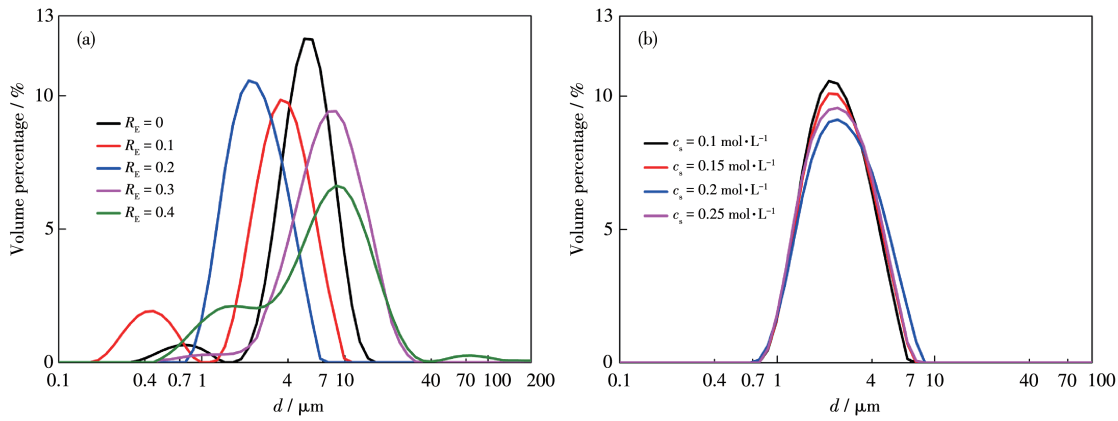
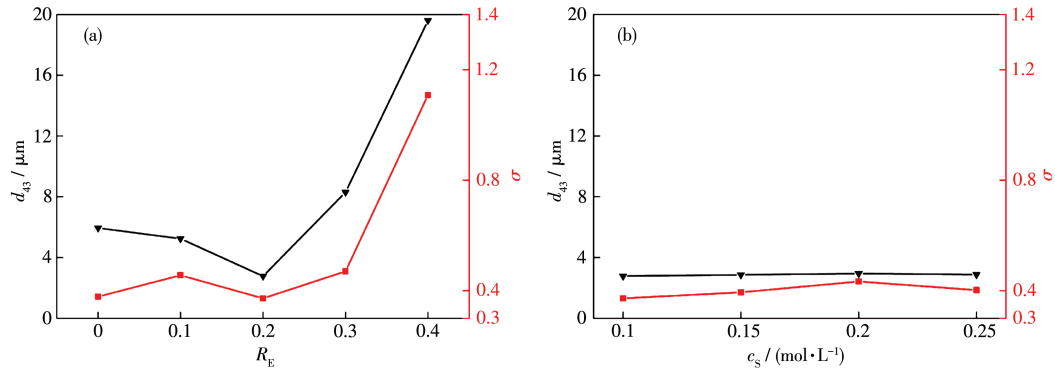


Fig.4 PSD of $SrCO_3$ particles prepared under different conditions of (a) c_s=0.1 mol·L^{-1} and (b) R_E=0.2

Compared with SEM images, it can be seen that the bundle-like particles were of different sizes when EDTA was not added, and there were some irregular small $SrCO_3$ particles when R_E=0.1. They are the reasons for bimodal distributions in the PSD profiles. Whereas the profiles were unimodal distributions when the amount of EDTA added increased. However, when R_E continued to increase to 0.4, the particle aggregation became more serious, and the spherical morphology deteriorated, resulting in a wider PSD with the multimodal distribution. In addition, when the molar concentration ratio was fixed at R_E=0.2 whereas the reactant concentrations were changed, particle size distribution curves almost coincided with each other, as shown in Fig.4b.

To have a quantitative analysis, d_{43}, and the uniformity, σ, were calculated and plotted against R_E and c_s in Fig.5. Here, σ is the degree to which the PSD deviates from the middle. The larger the value of σ, the wider the particle size distribution. When c_s was fixed at 0.1 mol·L^{-1}, increasing EDTA amount results first in a slight decrease followed by a rapid increase in d_{43}. This indicates there exists an optimized amount of EDTA added to regulate the particle size. While σ had a slight increase when the morphology changed from bundle to sphere after adding EDTA. A further increase in R_E led to a first slight decrease and then a sharp increase in σ. It is seen that the case of R_E=0.2 had the smallest size of d_{43}=2.77 μm and the most uniform size distribution of σ=0.372. When the molar concentration ratio was fixed at R_E=0.2 and c_s were changed, d_{43} and σ did not change significantly with the increase of c_s (Fig.5b). This indicates that the molar concentration ratio of EDTA to $SrCO_3$ has an important influence on the particle size and its distribution uniformity. There are relative maximum values of d_{43} and σ when c_s=0.2 mol·L^{-1}.

We also compare the spherical $SrCO_3$ particles prepared in this work with those in the published works by different methods, as listed in Table 1. EDTA has also been used to control the morphology of $SrCO_3$ particles by a carbonization reaction of $Sr(OH)_2$ with CO_2[3]. Spherical $SrCO_3$ particles of 200-800 nm were obtained when R_e=0.2, which is very similar to this



(a) c_s=0.1 mol·L^{-1}; (b) R_E=0.2

Fig.5　Effect of the molar concentration ratio of EDTA to $SrCO_3$ on the particle size and the distribution uniformity

Table 1　Comparison of different methods for preparing spherical $SrCO_3$ particles

Method	Additive	Crystallizer	Particle size	t	Ref.
Carbonization	EDTA	Gas-liquid container	200-800 nm	10 min	[3]
Co-precipitation	PSMA	Flask with stirring	5-10 μm	5 min	[2]
Co-precipitation	—	Ultrasonic instrument	0.5-1 μm	0.5 h	[23]
Hydrothermal	Urea	Autoclave	300-500 nm	2 h	[24]
Solvothermal	CTAB	Autoclave	0.5-1 μm	8 h	[6]
Co-precipitation	EDTA	Y-jet mixer	2-3 μm	1 ms	This work

work. When PSMA was used as the additive by using the co-precipitation method in a stirring flask[2], changing the amount of PSMA results in different particle morphologies, such as bundles, dumbbells, irregular aggregates, and spheres. The obtained spherical $SrCO_3$ particles were about 5-10 μm. Ultrasonic has also been used to intensify the co-precipitation process[23]. In the absence of additives, when the solvent was water, the obtained $SrCO_3$ particles were fusiform structures with a particle size of about 3-5 μm. When the solvent was changed into ethylene glycol, it can be observed that the shape of the particles becames spherical and the particle size was about 0.5-1 μm. When the hydrothermal method was used[24], urea was added to the solvent as an additive. After 2 h of reaction at 120 ℃ and 24 h of etching by HCl, spherical $SrCO_3$ particles with a size of 300-500 nm were obtained. When the hydrothermal reaction time increased to 8 h, there were flower-like $SrCO_3$ particles with a particle size of about 10 μm, which are very similar to the particle prepared without EDTA additive in this work. In the microemulsion-mediated solvothermal method[6], the autoclave was used as a crystallizer with CTAB as an additive. Rod-shaped particles with a length of 1-2 μm can be prepared at a lower molar ratio of H_2O to CTAB, while the spherical $SrCO_3$ particles with a size of 0.5-1 μm were obtained at a higher molar ratio. This work uses the miniature Y-jet mixer as the crystallizer to intensify the co-precipitation process, with EDTA as additive to prepare spherical $SrCO_3$. The prepared particles had an average particle size of 2-3 μm and a narrow PSD. The method does not require complicated reaction devices, and the subsequent drying conditions are very convenient, making it easier for industrial applications.

2.3 Mechanisms of morphology regulation by EDTA

The above experiments have proved that the precipitation process can be well regulated by adding EDTA and micron-sized $SrCO_3$ microspheres were successfully prepared. This should be attributed to the carboxyl groups on the EDTA molecule, which makes EDTA a strong chelating agent to form stable complexes with many metal ions[25]. In the precipitation process of $SrCO_3$, when EDTA is not added, the short rod-shaped strontium carbonate grows along the axial direction and forms a bundle structure. When EDTA is added, a stable coordination compound of EDTA-Sr is formed, which is very easily adsorbed on the surface of $SrCO_3$ particles. EDTA-Sr formed a coating layer around $SrCO_3$ particles, leading to growth inhibition in the axial direction[26]. Thus, all crystal faces grow at the same rate, resulting in a decrease in the slenderness ratio and gradually growing into spherical particles. Fig.6 shows a schematic mechanism of how EDTA regulates the morphology of $SrCO_3$ from rod-like to spherical particles.

In addition, the obvious separation of nucleation and growth stages is the basic requirement for the formation of homogeneous particles[27]. It has been reported that adding EDTA to the system can delay the precipitation of $SrCO_3$ and prolong the nucleation induction period of crystals[28], which is beneficial to the separation of two stages. From Fig.2, 4, and 5, one can see that the size of $SrCO_3$ particles was very uniform when enough EDTA was added to the system. Moreover, the miniature Y-jet mixer can mix two solutions evenly within several milliseconds, leading to uniform distribution of the supersaturation level[29], which is also

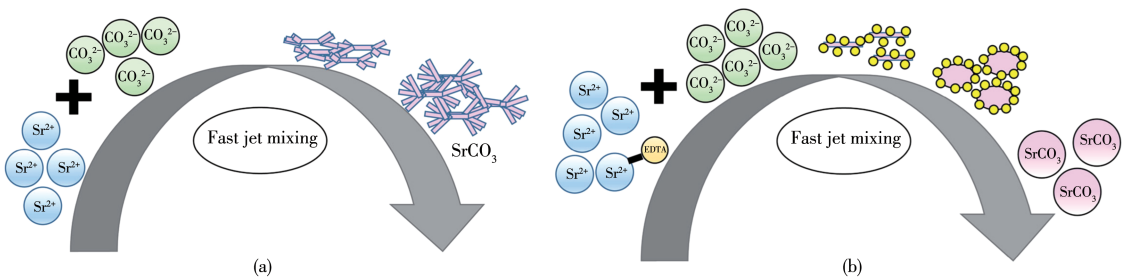


Fig.6 Mechanism diagrams of $SrCO_3$ formation in the miniature Y-jet mixer (a) without or (b) with adding EDTA

favorable to prepare $SrCO_3$ particles with a narrow PSD. With a further increase in the amount of EDTA added, more strontium ions are consumed to form the coordination compound of EDTA-Sr, leading to a further decrease in the supersaturation level and nucleation rate. This is conducive to the growth into large particles, which has been proven by the above experimental results. Nevertheless, a more mechanistic approach such as molecular simulation should be used in the future studies to further prove the morphology regulation mechanism by EDTA.

3 Conclusions

In this work, the co-precipitation method was used to prepare $SrCO_3$ particles. A miniature Y-jet mixer was used to intensify the mixing efficiency of the reactant solutions and EDTA was used as the additive to control the particle morphology. The particles were characterized by SEM, XRD, and PSD measurements.

In the absence of EDTA, $SrCO_3$ particles with bundle-like morphology could be obtained. When EDTA was added, uniform spherical $SrCO_3$ particles were prepared continuously for several milliseconds. The prepared particles are all orthorhombic phase crystals with very narrow PSD. The molar concentration ratio of EDTA to $SrCl_2$, R_E, is the key factor affecting the particle morphology and size. When R_E is fixed, the reactant concentration of $SrCl_2$, c_s, has a slight effect on the particle size and its uniformity. In the case of R_E= 0.2 and c_s=0.1 mol·L^{-1}, the prepared particles have the smallest size of d_{43}=2.77 μm and the most uniform size distribution of σ=0.372. The mechanism has been proposed that EDTA-Sr is adsorbed on the surface of $SrCO_3$ particles, which inhibits the axial growth rate of crystals, making all crystal faces have the same growth rate and gradually grow into spherical particles. At the same time, the addition of EDTA facilitates the separation of nucleation and growth stages, and the use of a miniature Y-jet mixer redistributes supersaturation, all of which make the $SrCO_3$ particle size more uniform. Compared with the methods in the published works, the preparation process in this work can be accomplished in a continuous mode within several milliseconds, thus, providing a high-efficiency and low-cost route to prepare micron-sized $SrCO_3$ spheres.

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