Powder Technology 284 (2015) 253–256

Contents lists available at ScienceDirect

Powder Technology

journal homepage: www.elsevier.com/locate/powtec

Short communication

The synthesis of calcium carbonate microparticles with multiple morphologies through self-assembly method

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ARTICLE INFO

Article history:
Received 30 November 2014
Received in revised form 17 June 2015
Accepted 28 June 2015
Available online 4 July 2015

Keywords:
Calcium carbonate
Mineralization
Microstructure
Microparticles

ABSTRACT

Taking advantage of the biocompatibility and biodegradability of calcium carbonate, a simple method was introduced to synthesize the calcium carbonate microparticles with different morphologies, including hollow (H-calcium), flower-like (P-calcium) and peanut-like (P-calcium). The possible formation mechanisms of these kinds of calcium carbonate microparticles were discussed in this study. Initially, the aminopropyltriethoxysilane (APTES) was possibly hydrolyzed, and octa(amino-propylsilsesquioxane) (R-NH_3^+) was obtained. The R-NH_3^+ might guide the $CaCO_3$ nanoparticles to form multiple calcium carbonate microparticles in aqueous phase because of their positive potential. Furthermore, the final treatments were very essential to obtain the calcium carbonate microparticles with satisfactory shape with this method. The XRD and FTIR results revealed the components and crystalline phase of these products. The morphologies were characterized by SEM and TEM. Furthermore, the potential value of these kinds of microparticles was discussed.

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1. Introduction

Calcium carbonate was a kind of material with good biocompatibility and suitable biodegradation rate [1–3]. There are different crystalline phases existed in calcium carbonate, such as calcite, aragonite, and vaterite [4]. The interactions between $CaCO_3$ species and proteins were very important [5–8]. Masahiro and his colleagues have studied the encapsulation of protein through the phase transition of calcium carbonate, from vaterite to calcite [5]. Moreover, some kinds of calcium carbonate biominerals that contained different proteins possessed some unique properties [9,10].

Taking advantage of the pH sensitivity and other excellent properties of calcium carbonate, a simple method was reported to obtain multiple morphologies of calcium carbonate microparticles in this paper.

2. Materials and methods

2.1. Materials

APTES and calcium chloride dihydrate ($CaCl_2 \cdot H_2O$) were purchased from the Aladdin Chemical Reagent Co., Ltd. Ammonia water was purchased from the Sinopharm Chemical Reagent Co., Ltd. Ethanol was bought from the KeLong Chemical Reagent Co., Ltd. Deionized water was produced by our own laboratory.

2.2. Synthesis of calcium carbonate microparticles with multiple morphologies

Solution I was a mixed solution by $NH_3 \cdot H_2O$ and ethanol with the volume ratio of 1:3; 0.120 g $CaCl_2 \cdot 2H_2O$, 1.1 ml aminopropyltriethoxysilane (APTES), and 9.0 ml ethanol were mixed to obtain solution II. Solution II (5 ml) was added into solution I (80 ml) under stirring to obtain solution III. Then solution III was stirred for 10 h at room temperature. Afterward, solution III was placed in the fuming cupboard to absorb CO_2 in the air. The white sediment was collected by centrifugation. Finally, the precipitates were treated with different measurements that were described as follows.

The precipitates were divided into three groups, group A, group B, and group C. Certain amount of deionized water was added into these precipitates. Precipitates in group A and group B were collected by centrifugation after being standing for 6 h at room temperature. After that, precipitate in group B was placed in deionized water for 7 days at room temperature. Precipitates in group C were stirred for 6 h with a certain amount of deionized water at room temperature. Then precipitates in group C were collected by centrifugation. All these samples by centrifugation were washed 3 times with deionized water. Finally, products of white powders in these groups were obtained after freeze-drying. The products of group A, group B, and group C were named H-calcium, F-calcium, and P-calcium, respectively.

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http://dx.doi.org/10.1016/j.powtec.2015.06.066
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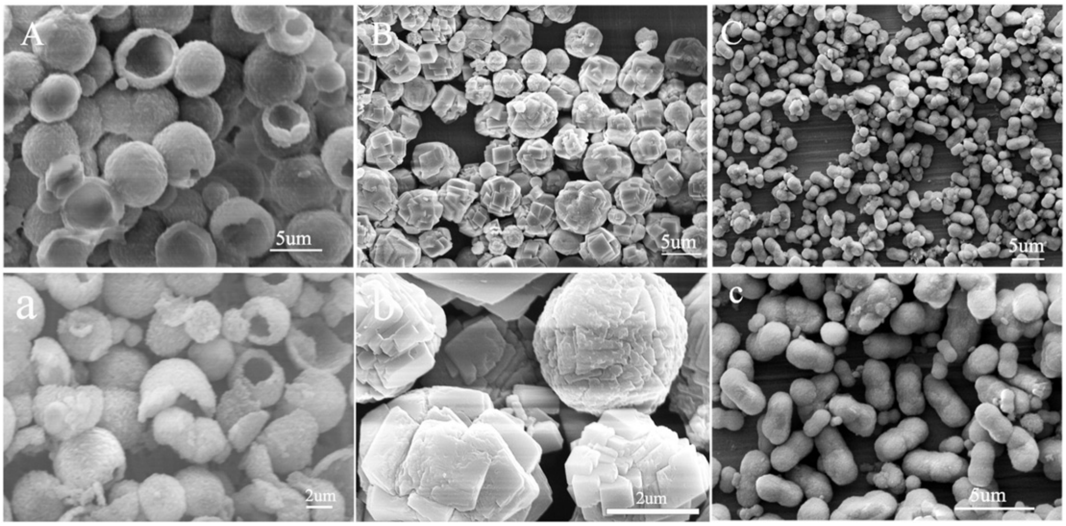

Fig. 1. The SEM images of these different products. Images A, B, and C show the microstructure of H-calcium, F-calcium, and P-calcium. Images a, b, and c are the magnified images of images A, B, and C.

2.3. Characterization

The X-ray diffraction (XRD) measurements of these products were realized by using D8 ADVANCE (German Bruker) diffractometer with Ni-filtered, Cu $K_{\alpha 1}$ target, $\lambda = 1.54183$ Å at 40 kV and 40 mA. Samples were scanned from $2\theta = 5°$ to $80°$ with a step size of 0.02° and a scanning speed of 0.1 s per step. The surface morphologies of these different microparticles were characterized by using NOVA NANOSEM 430 (Netherlands) instrument after coated with gold by using a sputter coater. The transmission electron microscopy (TEM) images were carried out by using the JEM-2100HR (JEOL) instrument. The Fourier transform infrared spectrometer (FTIR) results were recorded in the range of 400–4000 cm^{-1} by using Lambda950 (PerkinElmer Co., LTD. USA).

3. Results

Through this method, which was depicted in the experimental section, three kinds of $CaCO_3$ microparticles were synthesized. SEM (Fig. 1) images showed the different morphologies of these microparticles. FTIR and XRD results of these microparticles were listed in Fig. 2.

The differences of morphologies among three products were revealed from the SEM images (Fig. 1). The microstructure of H-calcium, F-calcium, and P-calcium were hollowed (Fig. 1A and a), flower-like (Fig. 1B and b), and peanut-like (Fig. 1C and c), respectively. It was shown from Fig. 1B and b that F-calcium was formed by many diamond-like particles. The component of diamond-like particles was confirmed by XRD and FTIR.

Fig. 2A showed FTIR results of three microparticles. Absorption peaks at 1467 cm^{-1}, 876 cm^{-1}, and 712 cm^{-1} were the absorption peaks of CO_3^{2-}. Absorption peaks at 2512 cm^{-1} and 1797 cm^{-1} could confirm the presence of calcium carbonate. Absorption peaks of H-calcium at 1087 cm^{-1} and 745 cm^{-1} showed the presence of vaterite polymorph, according to Gunawan Hadiko and his colleagues [2]. It was seen that absorption peak at 745 cm^{-1} was disappeared in the FTIR image (Fig. 2A) of P-calcium. These results demonstrated that the crystal phase of calcium carbonate microparticles was converted, from vaterite to calcite. The peaks at 1030 cm^{-1} and 1130 cm^{-1} were corresponding to the absorption peaks of Si-O bond in R-NH_3^+ [11]. Compared with the FTIR results of Dazhou Zhao and his colleagues [12], the peak at 712 cm^{-1} in our work showed that the H-calcium and F-calcium was not pure vaterite. XRD results (Fig. 2B) of H-calcium and F-calcium could further validate this conclusion. The diffraction peaks of the H-calcium at approximately 25°, 27°, and 33° indicated the existence of vaterite, while the peaks at approximately 30° indicated the existence of calcite [5,13]. These results confirmed that the components of H-calcium and F-calcium were not pure vaterite. Diffraction peaks at

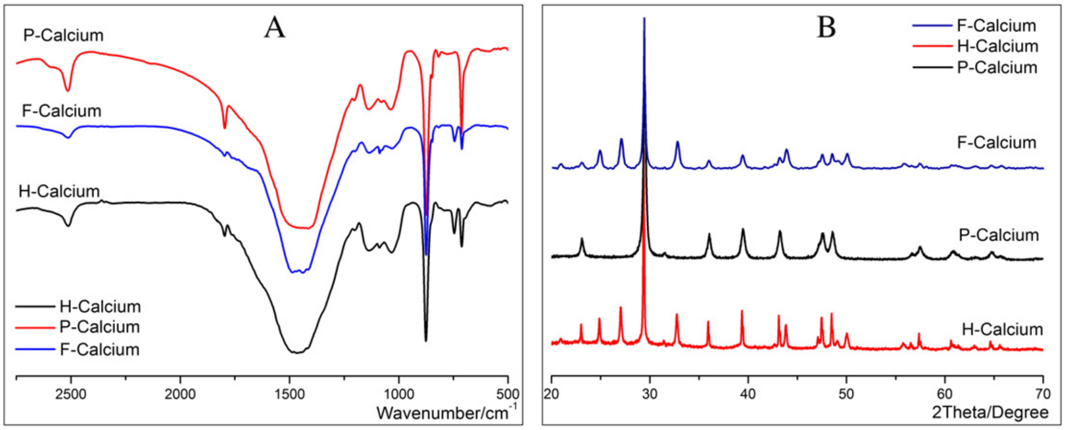

Fig. 2. FTIR (image A) and X-ray diffraction (image B) results of these microparticles.

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