

# Synthesis of calcium carbonate using extract components of croaker gill as morphology and polymorph adjust control agent



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## ABSTRACT

Biomimetic synthesis of calcium carbonate with various polymorphs, sizes and morphologies by using organic substrates has become an interesting topic for the last years. Calcium carbonate has been synthesized by the reaction of  $\text{Na}_2\text{CO}_3$  and  $\text{CaCl}_2$  in the presence of extract components of croaker gill. The products were characterized by powder X-ray diffraction (PXRD) and Fourier transform infrared (FT-IR) spectrum, and particle morphologies were observed by scanning electron microscope (SEM). The results show that at lower concentration yellow croaker gill extract has no effect on calcium carbonate crystal polymorph. Calcite was obtained only. But the morphologies of calcite particle change with the increase of the concentration. The corners of the particle change from angular to curved. However, with the further increase of the concentration of yellow croaker gill extract, the calcium carbonate obtained is a mixture of calcite and vaterite. The vaterite component in the mixture rises with increasing concentration of extract solution, indicating that the proteins from the yellow croaker gill during growth play a crucial role in stabilizing and directing the crystal growth.

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## 1. Introduction

Biomimetic synthesis of calcium carbonate with various phases, sizes and morphologies by using organic substrates has become an interesting topic in recent years [1]. It is not only because many natural systems, such as seashells, can use the biomineralization (e.g. bone, tooth enamel or shell of the mollusc) principle of directed nucleation and regulated growth of calcium carbonate to form skeletons or shells [2,3], but also it has industrial applications due to its wide use as filler in paints, plastics, rubber, or paper, where controlling of polymorphism, morphology and crystal size distribution is important [4–10]. Calcium carbonate polymorphs are also important in geological, environmental and bio-sciences [9–11].

Calcium carbonate is one of the most abundant inorganic biominerals, existing as three anhydrous crystalline polymorphs of increasing thermodynamic stability: vaterite (hexagonal), aragonite (orthorhombic) and calcite (rhombohedral) [12–15]. It is well known that vaterite is the thermodynamically most unstable form of the three crystal phases of  $\text{CaCO}_3$ . Without any additives, the vaterite phase transforms quickly into stable calcite via a solvent-mediated process [16,17]. A number of biomimetic templates or additives such as lipid bilayer stacks, vesicles, functionalized micropatterned surfaces, and components extracted from  $\text{CaCO}_3$ -rich organisms have been used for the synthesis of  $\text{CaCO}_3$  [18–20]. The components extracted from organisms are mainly grouped in two groups depending on their solubility

in water solutions [21]. In this article, we show that the biomolecules, extracted from yellow croaker gill, especially the soluble biomolecules, can direct growth and control the polymorph, morphology of  $\text{CaCO}_3$ .

## 2. Experimental

### 2.1. Materials

AR-grade  $\text{CaCl}_2$  and  $\text{Na}_2\text{CO}_3$  were obtained from commercial sources without further purification. Ultrapure water was used throughout the experiment.

### 2.2. Preparation of $\text{CaCO}_3$

In order to investigate the impact of soluble biomolecules on the growth of  $\text{CaCO}_3$ , 18.5 g of yellow croaker gill was washed carefully. Then, the yellow croaker gill was smashed to juice by a pulverizer, and the juice was mixed with the proper amount of ultrapure water and filtered, obtaining 40.0 mL of yellow croaker gill extract aqueous solution. This solution was examined by UV–Vis and FT-IR spectroscopy for understanding the soluble biomolecules of yellow croaker gill extract.

Then, 0, 3.0, 5.0, 7.0 and 10.0 mL of the extract aqueous solutions of yellow croaker gill were measured and put into five beakers. The ultrapure water was added into each beaker up to 10.0 mL of total volume. After that, 0.1110 g (1.0 mmol)  $\text{CaCl}_2$  was added into each beaker under stirring for about 10 min. The 0.1060 g (1.0 mmol)  $\text{Na}_2\text{CO}_3$  was added into each beaker. The mixture solutions were stirred for another

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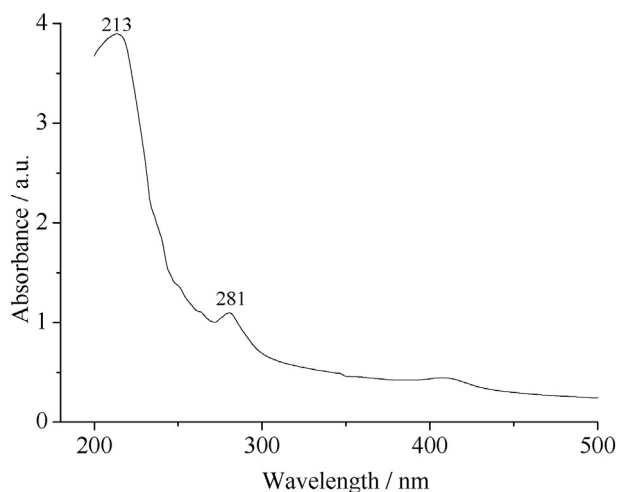


Fig. 1. UV-Vis spectrum of the extract components.

10 min. The precipitates produced were separated by centrifugation, washed three times with ultrapure water and anhydrous ethanol, and then vacuum dried at 40 °C for about 48 h. The size and morphology of the particles were examined by SEM. The crystal phases were determined by FT-IR spectroscopy and powder X-ray diffractometry.

### 2.3. Characterization

Powder X-ray diffraction patterns were performed using a Bruker D8 ADVANCE X-ray diffractometer with Cu-K $\alpha_1$  radiation at 40 kV and 40 mA. The samples were scanned over a  $2\theta$  range of 20–70° at a step size of 0.02°. Scanned electron microscopy (SEM) measurements for the crystals were performed on a JEOL JSM 5600LV scanning electron microscope. Before measurement, the samples were mounted on aluminum foil and sputter coated with gold. The FT-IR spectra were collected on a Nicolet 6700 spectrophotometer with KBr pellet and 32 scans were collected at a resolution of 4 cm $^{-1}$ . A UV-Vis double-beam spectrophotometer (Beijing Purkinje General Instrument Co., Ltd., China) was also used.

### 3. Results and discussion

Extract from gill of yellow croaker may consist of many biomolecules. Its chemical composition was studied by means of UV-Vis and FT-IR spectroscopy.

UV-Vis spectrum of the extract solution is shown in Fig. 1. The peak at 213 nm is attributed to the absorption of peptide bonds of protein in

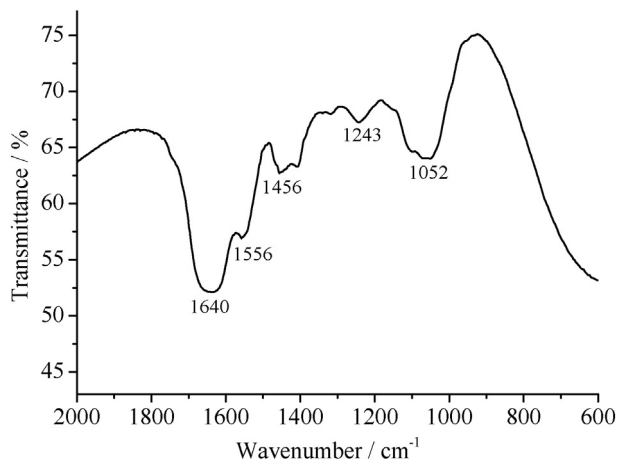


Fig. 2. FT-IR spectrum of the extract components.

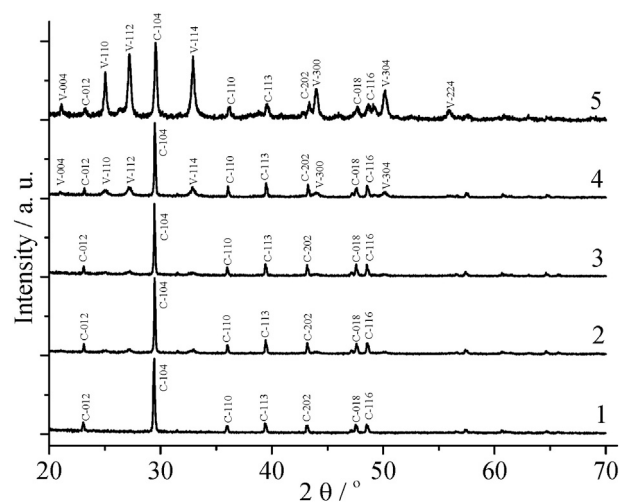


Fig. 3. XRD patterns of the as-synthesized samples, the volume of extract solution (mL): 1: 0.0 mL; 2: 3.0 mL; 3: 5.0 mL; 4: 7.0 mL; 5: 10.0 mL.

the extract, which is from the  $n-\pi^*$  transition of the CO group. The peak at about 281 nm belonged to the  $\pi-\pi^*$  transition of tyrosine, tryptophan, or phenylalanine residues of proteins. Fig. 2 is the FT-IR spectrum of the extract. The absorption bands at 1640 and 1551 cm $^{-1}$  are assigned to amides I (CO) and II (N–H) of protein, respectively. The peaks located at 1056 cm $^{-1}$  may correspond to the chlorophyll macrocyclic skeleton vibration. The FT-IR further confirms that the extract contains protein(s) [22–24].

Fig. 3 shows the XRD patterns of the samples isolated from all five control systems, which are compared with the XRD patterns of the respective standards of calcium carbonate polymorphs. The Bragg reflections marked with “C” correspond to calcite and “V” to vaterite polymorphs, respectively. It is evident that in systems 1, 2 and 3 only diffraction peaks of calcite were observed, while in systems 4 and 5 diffraction patterns of both calcite and vaterite were found (standard JCPDS files are as follows: calcite, 47-1743; vaterite, 33-0268). The contents of vaterite in 4 and 5 can be calculated based on equation  $I_C^{104}/I_V^{110} = 7.691(X_C/X_V)$  [25], where  $I_C^{104}$  and  $I_V^{110}$  stand for the diffraction peak intensities at (104) for calcite and (110) for vaterite. In 4, the content of vaterite is 36.9%, while in 5, the content of vaterite rises up to 82.4% with increasing amount of extract solution.

The as-synthesized samples of the CaCO $_3$  products were further characterized by FT-IR spectroscopy analysis. The results are

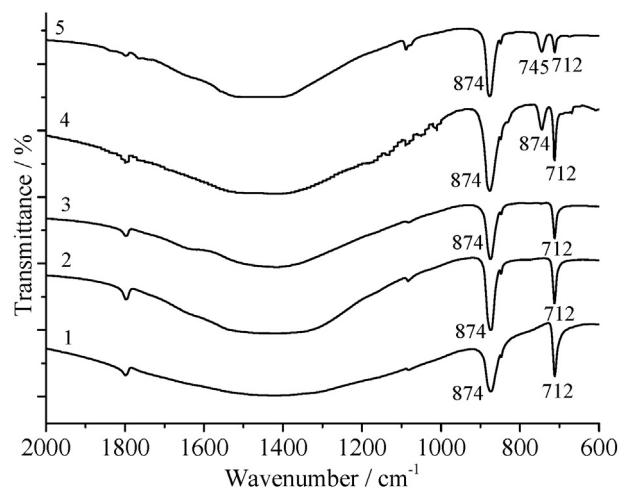


Fig. 4. The FT-IR spectra of the as-synthesized samples, the volume of extract solution (mL): 1: 0.0 mL; 2: 3.0 mL; 3: 5.0 mL; 4: 7.0 mL; 5: 10.0 mL.

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