Key Engineering Materials Vols. 675-676 (2016) pp 667-670
© (2016) Trans Tech Publications, Switzerland
doi:10.4028/www.scientific.net/KEM.675-676.667

Submitted: 2015-06-08
Revised: 2015-08-17
Accepted: 2015-08-18

The Various Concentration Effected on Crystallite Size of Calcium Carbonate

Sunti Phewphong[1, a], Kanokwan Najai[1, 2, b] and Tosawat Seetawan[1, 2, c*]

[1]Extraction Research Laboratory, Thermoelectrics Research Center, Research and Development Institution, Sakon Nakhon Rajabhat University, 680 Nittayo Road, Mueang District, . Sakon Nakhon, 47000, Thailand

[2]Program of Physics, Faculty of Science and Technology, Sakon Nakhon Rajabhat University, 680 Nittayo Road, Mueang District, Sakon Nakhon, 47000, Thailand

[a]sunti-sc@hotmail.com, [b]Benzbenz_kanokwan@hotmail.com, [c]t_seetawan@snru.ac.th

Keywords: Extraction calcium carbonate; Crystallite size; Cherry sell; Hydrothermal method

Abstract. The calcium carbonate was extracted from *Pomacea Canaliculata Lamarck* (Cherry shell) by using the hydrothermal method. Cherry shell was washed and crushed by DI water and mortar. The powder size was analyzed by particle (aperture Size 63 μm), added x HCl (x = 2, 3, 4) and y Na_2CO_3 (y = 1, 1.5, 2) mixed with fine powder from Cherry shell. The mixed power was filtered annealed by autoclave at controlled temperature at 333 K for 20 h. The crystal structure was characterized by X–ray diffraction method. The phase transformation of $CaCO_3$ was analyzed by Fourier transform infrared spectroscopy (FTIR). The morphology of Cherry shell and $CaCO_3$ powders were observed by using scanning electron microscope (SEM). It was found that the extracted $CaCO_3$ 'shows single phase of $CaCO_3$ crystal structure. Smallest crystallite size was found about 0.54 μm.

Introduction

Calcium carbonate is a chemical compound with the formula $CaCO_3$. It is a common substance found in rocks in all parts of the world, and is the main component of shells of marine organisms, snails, coal balls, pearls, and eggshells. The vast majority of calcium carbonate used in industry is extracted by mining or quarrying. The two crystalline forms are calcite and aragonite [1, 2]. In the part synthesis of $CaCO_3$ was followed by two basic synthetic routes: (1) the solution route, through a double decomposition reaction, where in aqueous $CaCl_2$ and Na_2CO_3, or $CaCl_2$ and $(NH_4)_2CO_3$, or $Ca(NO_3)_2$ and Na_2CO_3 are combined in an equal molar ratio; and (2) the carbonation method, in which CO_2 gas is bubbled through an aqueous slurry of $Ca(OH)_2$ [3, 4].

In this paper, we are focused on the extracted calcium carbonate from shell by hydrothermal method. The extracted powder calcium carbonate was analyzed for crystal structure, and crystallite size. The phase transformation of $CaCO_3$ was analyzed by FTIR spectroscopy. The morphology of Cherry shell and $CaCO_3$ powders were observed by using SEM.

Experimental

The calcium carbonate was extracted from Cherry shell by using the hydrothermal method. Cherry shell was washed and crushed by DI water and mortar. The powder size was analyzed by a sieve (Aperture Size 63 μm). The starting powder is from 10 g of Cherry shell powder was digested in 100 ml hydrochloric acid 37% (QRëC New Zealand) and 100 ml sodium carbonate 99.99% (QRëC New Zealand). The mixed with fine powder from Cherry shell, Autoclave at controlled temperature at 333 K for 20 h. The crystalline $CaCO_3$ was rinsed with distilled water until nonionic residue by measuring the pH value of about 7, and then dried at 373 K for 20 h in an oven.

The overall reactions for the calcium carbonate extracted step can be summarized from Eq.

$$CaCO_{3(s)} + x\,HCl_{(aq)} \rightarrow CaCl_{2(aq)} + CO_{2(g)} + H_2O_{(l)}$$

$$CaCl_{2(aq)} + y\ Na_2CO_{3(aq)} \rightarrow CaCO_{3(s)} + NaCl_{(l)}$$

The crystalline phase of the synthesized $CaCO_3$ was characterized by X–ray diffraction with CuKα, λ = 0.15 nm (XRD–6100 Shimadzu, Japan).

The average crystallite size (D) of the calcium carbonate was calculated using Scherrer's equation [5].

$$D = \frac{0.9\lambda}{\beta \cos\theta}$$

D = shape factor, λ = X–ray wavelength, β = FWHM of diffraction peak, θ = Bragg angle.

The samples were measured by using FTIR spectroscopy analysis (FTIR–8900 Shimadzu, Japan) by the KBr pellet method. The morphology of the products was measured by virtue of SEM (SEM JSM–6301F JEOL, Germany).

Results and Discussion

The X–ray diffraction patterns are shown 2 HCl + Na_2CO_3 (2:1), 3 HCl + 1.5 Na_2CO_3 (3:1.5) and 4 HCl + 2 Na_2CO_3 (4:2). The X–ray diffraction of calcium carbonate powder showed rhombohedral structure and agreed with ICUD PDF number 00–047–1743. The crystallite size of 2 HCl + Na_2CO_3 (2:1), 3 HCl + 1.5 Na_2CO_3 (3:1.5) and 4 HCl + 2 Na_2CO_3 (4:2) was calculated by the Scherrer's equation yield of 0.76 μm, 0.62 μm and 0.54 μm, respectively for crystallite size.

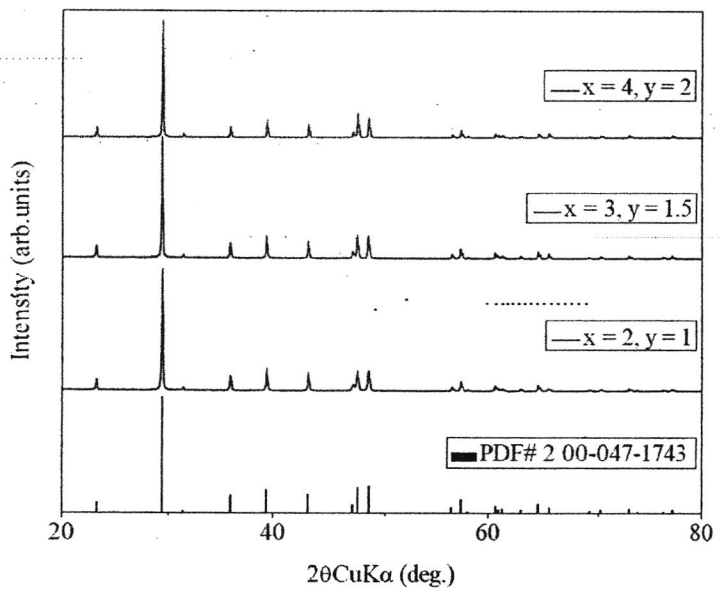

Fig. 1 X–ray diffraction patterns of Calcium carbonate powder for x HCl (x = 2, 3, 4) and y Na_2CO_3 (y = 1, 1.5, 2)

FTIR spectra of the calcium carbonate are presented in Fig. 2(a) and 2(b). FTIR spectroscopy is an important instrument used to identity different phases of organic and inorganic compounds. The spectral data obtained the absorption for a broad peak of CO_3^{2-} at wave number of ~1788.07 cm^{-1}, ~1082.10 cm^{-1}, ~875.71 cm^{-1}, ~862.21 cm^{-1}, ~713 cm^{-1}, and ~700 cm^{-1}, which have been reported to be the common characteristic features of the carbonate ions in calcium carbonate and are the fundamental modes of vibration for this molecule [1, 2, 6]. The characteristic peak of calcite is ~1788.07 cm^{-1}, ~875.71 cm^{-1}, and ~713 cm^{-1} and those of aragonite are ~1082.10 cm^{-1}, ~862.21 cm^{-1} and ~700 cm^{-1}.

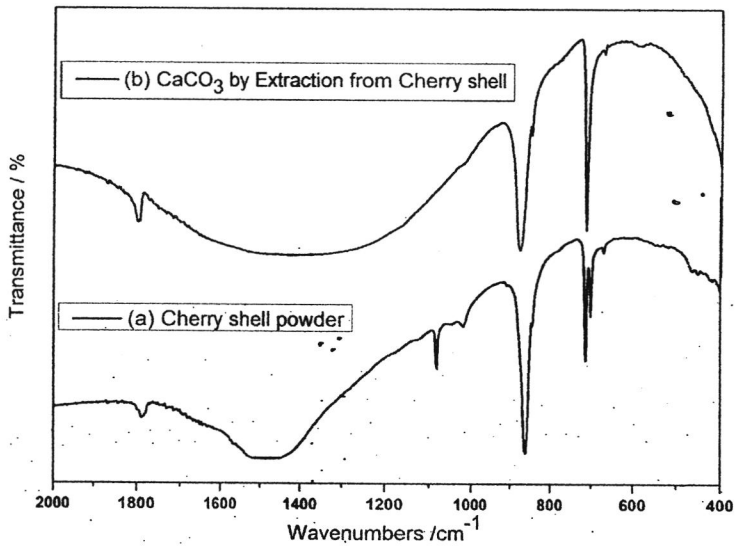

Fig. 2 FTIR spectra of (a) Cherry shell powder (b) Calcium carbonate has been extracted from Cherry shell

The morphology was analyzed by scanning electron microscopy. The SEM photographs of the calcium carbonate powder from Cherry shell as shown in Fig. 3(a), 3(b) and 3(c).
The concentration of particle size shows an effect on the samples. The calcium carbonate powders compared for x HCl (x = 2, 3, 4) and y Na_2CO_3 (y = 1, 1.5, 2) to the powders obtained particle size 1–2 μm. The morphology analyzed by SEM of particle size was decreased with increasing concentration of hydrochloric acid and sodium carbonate.

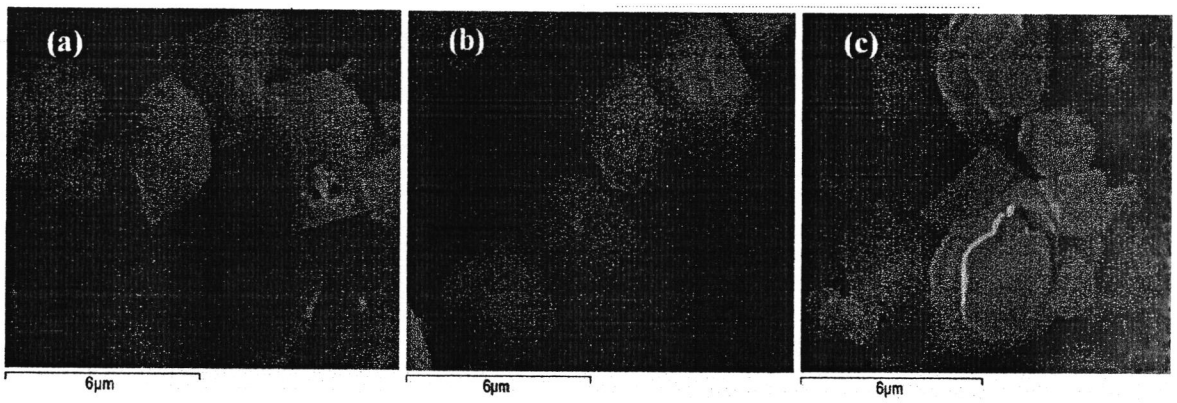

Fig. 3 SEM photograph of calcium carbonate from Cherry shell of HCl : Na_2CO_3 (a) 2:1, (b) 3:1.5 (c) 4:2

Summary

The analysis of crystal structure using XRD illustrates that the calcium carbonate powder extracted from Cherry shell. The concentration HCl and Na_2CO_3 were obviously effects of calcium carbonate crystallite size. The characteristic FTIR spectra peak of calcite is ~1788.07 cm^{-1}, ~875.71 cm^{-1}, and ~713 cm^{-1} and those of aragonite are ~1082.10 cm^{-1}, ~862.21 $cm^{-1}$and ~700 cm^{-1}. SEM images confirmed that the extracted calcium carbonate powder from cherry shell. Calcium carbonate powders compared for x HCl (x = 2, 3, 4) and y Na_2CO_3 (y = 1, 1.5, 2) to the powders obtained particle sizes 1–2 μm. The results XRD and SEM of particle size were decreased with increasing concentration of hydrochloric acid and sodium carbonate.

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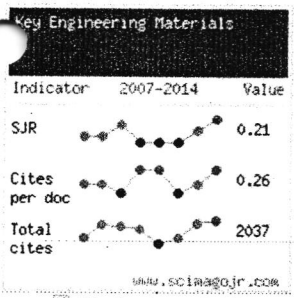

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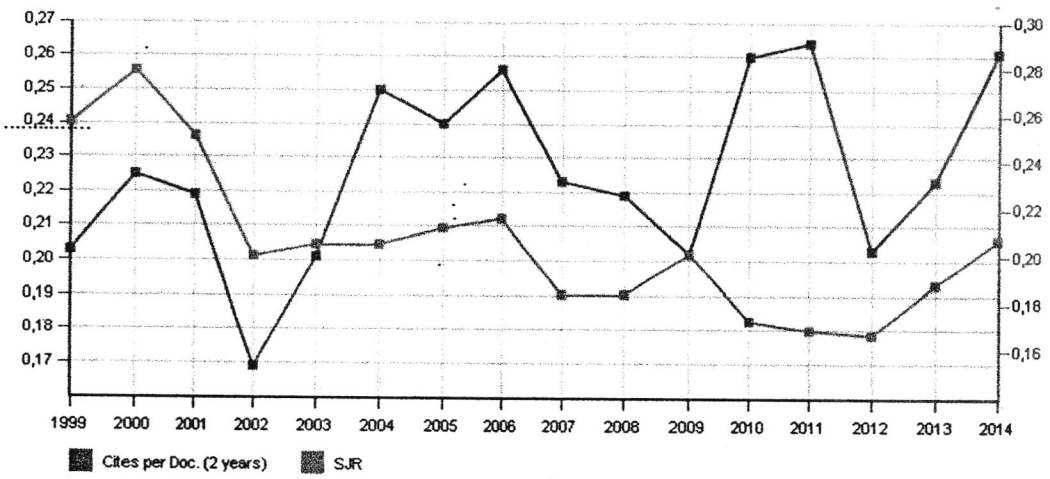

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