Synthesis and Characterization of Eco-friendly Nanostructured Hydroxyapatite from Duck's Eggshells

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Abstract

Hydroxyapatite (HAp) is more suitable bioceramic materials for hard tissue replacement than metals (eg. steel). HAp can be prepared by using waste eggshells, natural snail shells and limestones as calcium originate that reacts with phosphate. In this work, HAp sample was synthesized by co-precipitation method using Duck's eggshells as Ca source. The obtained HAp sample was characterized by X-ray diffraction (XRD), energy dispersive X-ray fluorescence (EDXRF), Fourier transform Infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). All the diffraction peaks from XRD patterns confirm that the synthetic sample prepared at room temperature was hexagonal structure hydroxyapatite. The nanostructured hydroxyapatite sample was observed in the SEM image.

Keywords: duck's eggshells, hydroxyapatite, nanostructured, SEM, XRD

1. Introduction

The rise of accidents such as traffic accidents, accidents at work and other accidents led to high demand for materials which can repair damaged bones. Accidents can cause damage such as cracked or fractured bones. Implantation in the damaged bone is one of the good treatments in restoring the bone function. Bone implants in human body can use various synthetic materials of ceramic, metal, or polymer, such as powdered apatite [1].

Calcium phosphate group is the most important inorganic part of hard tissue constituting bones and detine materials in vertebrate animals. Calcium phosphates with a Ca/P ratio between 1.5 and 1.67 are called apatites (e.g., hydroxyapatite, fluorapatite and chlorapatite). Apatite [$Ca_{10}(PO_4)_6X_2$] materials have unique biocompatibility feature among calcium phosphate groups; X in the formula represents hydroxyl (OH^-) group for hydroxyapatite [$Ca_{10}(PO_4)_6(OH)_2$], fluoride (F^-) group for fluorapatite [$Ca_{10}(PO_4)_6F_2$], and chloride (Cl^-) group for chlorapatite [$Ca_{10}(PO_4)_6Cl_2$] [2].

Among the calcium phosphate groups, hydroxyapatite (HAp) is a thermodynamically most stable crystalline phase of CaP in body fluid, possesses the most similarity to the mineral part of bone. Currently, HAp is commonly the materials of choice for various biomedical applications, e.g. as a replacement for bony and periodontal defects, alveolar ridge, middle ear implants, tissue engineering systems, dental materials, bioactive coating on metallic osseous implants and regeneration [3].

Therefore, in this research, nanostructured HAp sample was synthesized by co-precipitation method using duck eggshells.

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2. Materials and Method

2.1 Synthesis

The major component of eggshells is more than 94% of calcium carbonate ($CaCO_3$). Therefore, in this study duck's eggshells were used as calcium source. Diammonium hydrogen phosphate ($(NH_4)_2HPO_4$) was used as phosphate source and distilled water (H_2O) was used as solvent.

First, Duck's eggs were bought from a shop at Myo Ma Market, Taunggyi and washed five times with water to remove the dust and other impurities. The cleaned eggs were boiled and immersed in cold water. After cooling, the eggshells were peeled and then sun dried for one day.

The dried eggshells were crushed and ground using a motor and a pestle to obtain ($CaCO_3$) powder. The obtained powder was calcined in furnace at 900ºC to attain the calcium oxide (CaO) powder. However, after a few days CaO phase transform into calcium hydroxide ($Ca(OH)_2$) phase due to the sensitivity of fine CaO powder with atmospheric air.

Calcium hydroxide and diammonia hydrogen phosphate were weighed. A 3 g of $Ca(OH)_2$ and 50 mL of distilled water were mixed in a beaker and the calcium solution was stirred at room temperature for 5 min with 700 rpm. The pH value of calcium solution was pH 12. A 3.16 g of $(NH_4)_2HPO_4$ and 50 mL of distilled water were mixed and then stirred at room temperature for 5 min with 700 rpm. The measured pH value of P solution was 8.

Then, P solution was added drop wise to Ca solution. The mixed solution was stirred at room temperature with 700 rpm for 30 min. All the steps were carried out at room temperature. The pH value of mixed solution was 9. The mixed solution was filtered and then dried for 24 h at room temperature. After that, the dried sample was calcined at 750ºC to obtain hydroxyapatite sample.

2.2 Characterization

The synthetic HAP sample was characterized by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR) and field emission scanning electron microscopy (SEM).

3. Results and Discussion

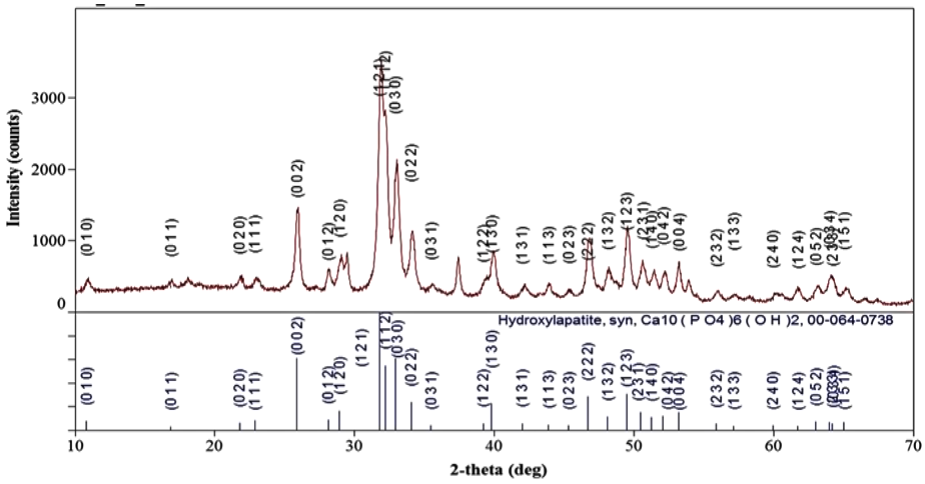

Figure 1 XRD pattern of synthesized HAp powder

Figure 1 shows the XRD pattern of HAp sample. All the diffraction peaks of synthesized HAp are in agreement with the standard hexagonal structure hydroxyapatite ICDD card No.00-064-0738. Diffraction peaks with higher intensity at angle 2θ values of 25.89°, 31.87°, 33.08° and 34.13° corresponding to (002), (121), (030) and (022) crystal planes confirm that the synthesized HAp is pure hydroxyapatite phase. The crystallite size calculated from the main diffraction peak of HAp sample at 2θ value of 31.87° by Debye-Scherrer's equation ($D = k \lambda / \beta \cos \theta$) is 19.85 nm.

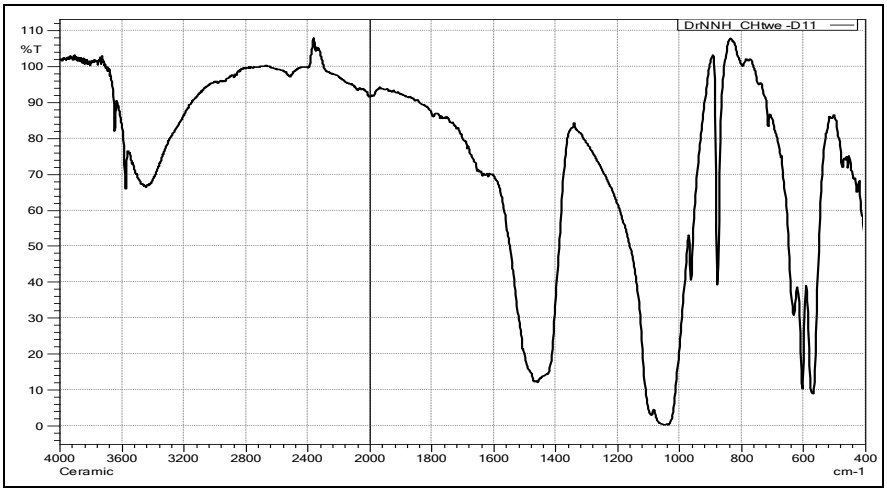

Figure 2 FTIR spectrum of synthesized HAp powder

Figure 2 shows the FTIR spectrum of HAp sample. The presence of characteristic HAp bands including phosphate group and hydroxyl group can be observed. The bands between 3600 cm^{-1} and 3440 cm^{-1} are attributed to O-H stretching vibrations and around 1630 cm^{-1} are due to the H-O-H bending vibrations of hydroxyl group. The characteristic bands of PO_4^{3-} are observed around 1050 cm^{-1}, 962 cm^{-1}, 620 cm^{-1} and 575 cm^{-1}. Furthermore, the bands around 1400 cm^{-1} and 874 cm^{-1} were attributed to the CO_3^{2-} group and this is due to the presence of carbon dioxide during synthesis.

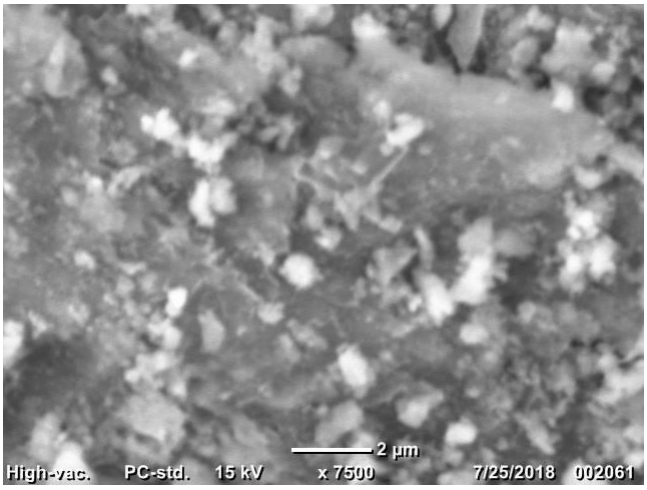

Figure 3 SEM image of synthesized HAp powder

Figure 3 shows the SEM image of HAp sample. In the SEM image of HAp sample, irregular-shaped nanoparticles and few nanorods are observed.

4. Conclusion

In this study, eco-friendly nanostructured (HAp) sample was successfully synthesized by co-precipitation method. XRD results show that pure HAp sample prepared at room temperature is in agreed with the standard HAp. FTIR spectrum confirms the XRD result. SEM image exhibits that nanostructured HAp sample can be synthesized by simple co-precipitation method at room temperature.

5. Acknowledgements

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6. References

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