

Preparation of Glass-Ceramic Biomaterial from Cow bone and Thai Mollusk shells

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Abstract:

Background: Hydroxyapatite material has excellent biocompatibility with the human body trust it has poor mechanical properties. Bioglass addition can be solving this problem. Glass-ceramic biomaterial from cow bone and Thai mollusk shells is a new material for repair the broken bone.

Objective: The purpose of this research is preparation of glass-ceramic biomaterial form cow bone and Thai mollusk shells, and to investigate their characteristics and properties.

Methods: Nano HA powder was prepared by thermal method for heating up to 950°C and ground with highspeed pot mill, beside the calcium phosphate glass was prepared by melted the mixture of CaO-P₂O₅-Na₂O at 1000°C, quenched and ground to fine powder by high speed pot mill. Then mix two type of powder in the various ratio. Samples of the composite were formed by a hydraulic press machine and sintered at temperature of 1300°C for 2 h in an electric furnace. Characterization by FTIR, XRD, SEM/EDX and UTM machine.

Results: The results from FTIR and XRD confirmed the functional groups and composition phase of HA powder that has the ion stretching vibration of hydroxyl group (OH⁻), carbonate (CO₃²⁻), and phosphate groups (PO₄³⁻) were also observed around 1045 and 560 cm⁻¹ respectively. XRD measurement showed that the ceramic contains hydroxyapatite crystals with β-tricalcium phosphate, calcium oxide and amorphous CaPO₄ phase. SEM measurement shows the porous, HA particles and amorphous phase. Mechanical property measurements show that the glass-ceramic contains flexural stress of 55.09 MPa.

Conclusion: In this experiment, hydroxyapatite powder and bioglass powder can be prepared for fabricating biomaterials. They could develop to apply as the production of scaffold bone, sponge bone, and bone filler.

Keywords: Hydroxyapatite, Biomaterial, Cockle shell, Bioceramic, Bioglass

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Introduction

Hydroxyapatite has been widely investigated due to their excellent biocompatibility with human's body which includes good bioactivity¹, but poor mechanical properties². Many calcium phosphate-based compounds can be synthesized by chemical techniques such as co-precipitation^{3,4}, sol-gel synthesis^{5,6}, hydrothermal method⁷ and thermal deposition⁸. Moreover, it can be synthesized from natural starting materials such as eggshell^{9,10}, coral¹¹ or bow bone^{12,13}. These natural materials pertain some advantage properties that they inherit from the raw materials such as pore structures. Mollusk shells (aragonite) is one of the more abundant crystalline polymorphs of calcium carbonate (CaCO_3). It is also a good biomedical material as it is denser than calcite and could be integrated with, resolved, and replaced the bone¹⁴.

Therefore, the aims of this work are; to fabricate dense HA ceramic from cockle shell which is the natural calcium source, and to investigate their characteristics and properties. Furthermore, the cockle shells can also be used over a wide range for medical applications

Objective

The aims of this work are to fabricate glass-ceramic from hydroxyapatite powder prepared from cow bone and co-bonding with glassy phase of calcium phosphate glass, these raw materials are the natural bone and calcium source, and to investigate their characteristics and properties.

Method

Nano HA powder was prepared by thermal method for heating up to 950°C and ground with highspeed pot mill, beside the calcium phosphate glass was prepared by melted the mixture of $\text{CaO-P}_2\text{O}_5\text{-Na}_2\text{O}$ at 1000°C , quenched and ground to fine powder by highspeed pot mill. To fabricate the dense HA, poly vinyl alcohol (PVA, Sigma-Aldrich, Singapore) and No-Tox HD food grade grease (Bel-Ray, USA) were used as binder and lubricant. HA and CaPO_4 glass powder were mixed with 0.05%wt PVA and moisture content of 3% in mortar. The mixtures were uniaxial pressed at 500 psi into a disc shape (0.5 cm high \times 1.5 cm diameter) by using stainless steel mold and dry pressing-hydraulic machine. The green body samples were sintered at three different temperatures of 1300°C for 2 h in electric furnace. Characterization by FTIR, XRD, SEM/EDX and UTM machine.

Results and Discussion

The FTIR spectra of the HA powder are displayed in Figure 1. The ion stretching vibration was around 3574 and 2002 cm^{-1} which confirms the presence of hydroxyl group (OH^-), 1451 cm^{-1} for carbonate (CO_3^{2-}) whereas phosphate groups (PO_4^{3-}) were also observed around 1045 and 560 cm^{-1} . Figure 2 show mineral composition in cow bone powder it confirmed that is the hydroxyapatite phase.

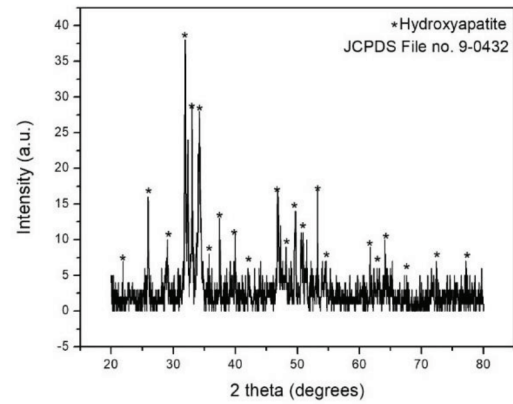
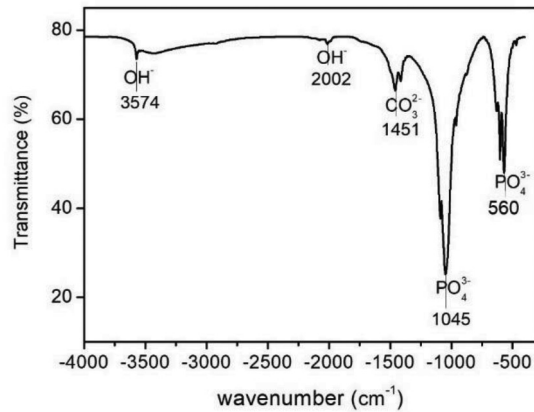


Figure 1 FTIR spectrum of synthesized HA powder. **Figure 2** XRD patterns of HA powder.

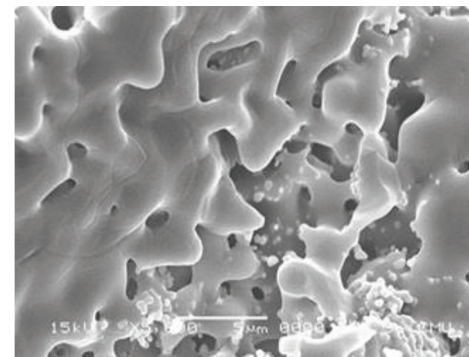
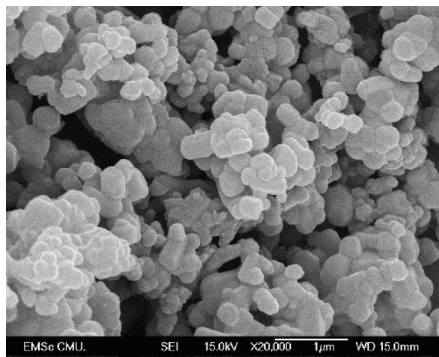


Figure 3 SEM micrograph of HA powder. **Figure 4** SEM micrograph of CaPO₄ glass-HA.

Figure 3 shows the morphology of HA powder that was agglomerated after the powder sample. For sintered CaPO₄ glass-HA ceramic shows in Figure 4 grain size range between 0.5–1 mm on the fracture surface of sample with sintered at 1300°C. An apparent porosity occupies approximately 50% of total area. The pore size ranges between 1–10 mm, while smaller pores range between 1–2 micron and bigger

pore has size that range between 3–8 mm. This sintered temperature showed the formation of closed pores. Strength of materials was shown in Figure 5, the flexure stress and strain of 10% Glass: 90% HA sample, sintered at 1300°C are 55.09 MPa and 10100.19 MPa. It was lower than flexure stress and strain of dense human bone (156 MPa).

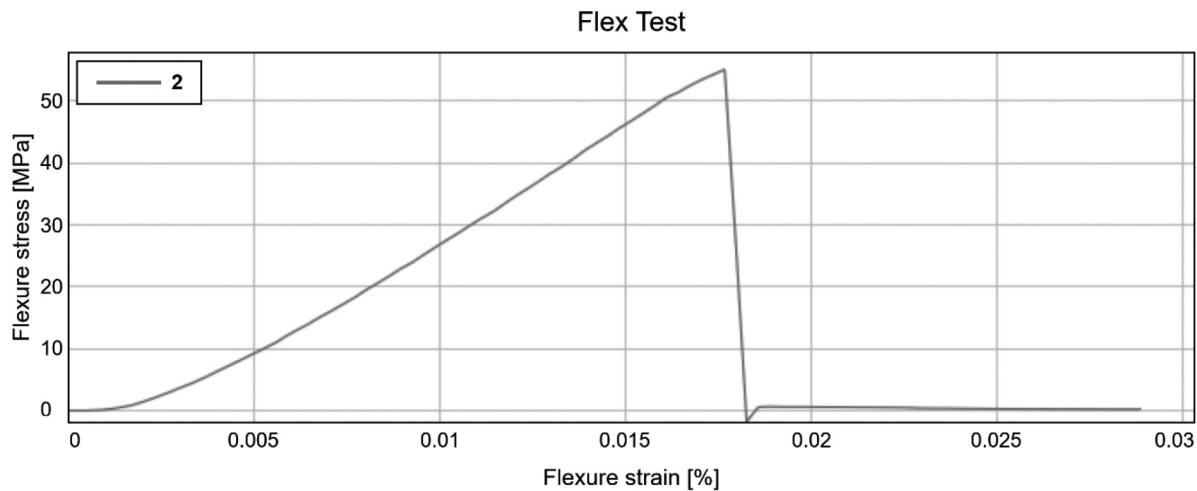


Figure 5 Flexural modulus of 10% Glass: 90% HA sample, sintered at 1300°C.

Conclusion

In this experiment, nano HA powder was prepared by thermal method and the calcium phosphate glass was prepared at 1000°C. Then, the samples of glass-ceramic composite were formed by a hydraulic press machine and sintered at temperature of 1300°C for 2 h in an electric furnace. The results from characterization technique; FTIR, XRD, SEM/EDX and UTM machine can be investigated that hydroxyapatite or HA powder and bio-glass powder were prepared from natural resource like cow bone and mollusk shells. SEM measurement shows the porous, HA particles and amorphous phase that given low mechanical property that the glass-ceramic contains flexural stress of 55.09 MPa. So, they could develop to apply as the production of scaffold bone, sponge bone, and bone filler.

Acknowledgement

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