UNIVERSITI TEKNOLOGI MARA

SYNTHESIS AND MECHANICAL PROPERTIES OF NANO-HYDROXYAPATITE FROM EGG SHELLS AND EFFECTS OF NANO-HA-TITANIUM COMPOSITE USING MICROWAVE TECHNIQUE

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AUTHOR'S DECLARATION

I declare that the work in this thesis was carried out in accordance with the regulations of Universiti Teknologi MARA. It is original and is the results of my own work, unless otherwise indicated or acknowledge as referenced work. This thesis has not been submitted to any other academic institution or non-academic institution for any degree or qualification.

I, hereby, acknowledge that I have been supplied with the Academic Rules and Regulations for Post Graduate, Universiti Teknologi MARA, regulating the conduct of my study and research.

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ABSTRACT

Hydroxyapatite (Ca_{10} (PO_4) $_6$ (OH) $_2$,), is an interesting material of an inorganic compound whose chemical composition and crystallographic structures are similar to the composition of the bone. However, due to its poor mechanical properties, HA ceramics cannot be used for heavy load bearing applications. Natural source such as egg shells was composed by 94 wt. % of calcium carbonate ($CaCO_3$), which can be changed to calcium oxide (CaO) by calcinations process. The temperature and the duration to produced CaO was 900 ^{0}C for 2 hours. The synthesis of nano-HA was done by the mixtures of diammonium phosphate (DAP) and calcium hydroxide (CA (OH) $_2$) and subjected to heat using microwave for 30 minutes at 1100 W irradiation power. The X-Ray Diffraction (XRD) was used to characterize the phase and crystal sizes. As the results, nano-HA gave the highest peak that obtained clearly at 2θ~31.94 ° and located at (*211*) lattice. The nano-HA powder showed well defined broad crystalline peak. The crystalline size of nano-HA was in the range of 35 – 39 nm. Ball milled machine was used to mixed the nano-HA with different compositions of titanium (Ti) powder which were 10, 15, 20 and 25 wt.%. for 30 minutes and then was pressed by hand hydraulic pump with the force of 2300 psi to form the pellets. The pallets then were sintered at 1200 °C by the heating rate of 3 °C/min for 2 hours. Hardness, compression and wear test were tested. As a result, HA-25wt. %Ti composite gave the highest hardness test, 89.6 Hv, compressive test, 82.5 MPa and coefficient of friction for wear test,0.76 μ.The microstructure analyses showed, nano-HA particles have a high tendency to agglomerate and formed the shape likes needle morphology. The addition of 25wt %Ti, showed the distribution of each element in the composition have well distributed by using EDS analyses

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CHAPTER ONE
INTRODUCTION

1.1 BACKGROUND OF STUDY

Nowadays, egg shells that contain a high source of calcium had been studied as part of biological system especially bones. Highly complex organic–inorganic composite produced by nature can be found in bones, dentine, eggshells and shells of marine molluscs. In order to use these materials the characteristics are the most important factor to be controlled. From this biological material, the material that can be synthesised is hydroxyapatite (HA), [$Ca_{10}(PO_4)_6(OH)_2$](Dasgupta *et. al.*, 2004).

Drugs and chemical that is used in medicine today can cause side effect to human. Therefore, researcher is studying biomaterials to assist the replacement for organs and their functions. The new biomaterials have been developed with advance mechanical and biological properties. Biomaterials has been proven as a new alternative methods, especially in medical applications.

Calicum (Ca)-HA is the main inorganic component of hard tissues in the bones. It is one of 'apatite' that is similar in structure, but not necessarily identical in the composition. Similarity of synthetic HA in chemical and crystallographic structure with bone materials and its excellent biocompatibility can be used in clinical field applications. Calcium is one of the main components of synthetic HA. Therefore, Ca used to prepare HA can be obtained from living bodies and natural sources. The choice of natural sources can minimize the chances of finding impurities like silica in the materials, which will in turn help its implant in the living body. Moreover, the production cost can be reduced since no purifying process is required. Eggshells is one of the richest sources of Ca which composed of 94 wt. % calcium carbonate ($CaCO_3$).