PREPARATION AND CHARACTERIZATION OF HYDROXYAPATITE (HA) FROM COW BONE AND ITS COMPOSITE WITH POLY(LACTIC ACID) FOR BONE REPLACEMENT

AKINDOYO JOHN OLABODE

Doctor of Philosophy

UNIVERSITI MALAYSIA PAHANG
SUPERVISOR’S DECLARATION

We hereby declare that we have checked this thesis and in our opinion, this thesis is adequate in terms of scope and quality for the award of the degree of Doctor of Philosophy in Chemical Engineering.

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(Supervisor’s Signature)

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Position     :
Date         :

________________________________________
(Co-supervisor’s Signature)

Full Name    :
Position     :
Date         :
STUDENT’S DECLARATION

I hereby declare that the work in this thesis is based on my original work except for quotations and citations which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at Universiti Malaysia Pahang or any other institutions.

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PREPARATION AND CHARACTRIZATION OF HYDROXYAPATITE (HA) FROM COW BONE AND ITS COMPOSITE WITH POLY(LACTIC ACID) FOR BONE REPLACEMENT

AKINDOYO JOHN OLABODE

Thesis submitted in fulfillment of the requirements for the award of the degree of Doctor of Philosophy

Faculty of Chemical & Natural Resources Engineering

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The Author.
ABSTRACT

The wide application of hydroxyapatite (HA) for medical applications such as bone tissue replacement sometimes constitutes environmental challenges as the conventional HA synthesis routes require the use of organic solvents. On the other hand, the current trend of research is to incorporate biomaterials such as HA into polymer matrices for some medical applications such as bone replacements. However, this often produces composites with inferior properties. This is due to poor HA dispersion within the composites as well as compatibility issues. In this study, natural HA was produced from cow bone through ultrasound and calcination processes at various temperatures. Composites then were produced from poly (lactic acid) (PLA) and hydroxyapatite (HA) through extrusion and injection molding. In order to foster good interaction between PLA and HA, and to impart antimicrobial properties onto the HA, surface of the HA was modified. On the other hand, impact properties of the PLA-HA composite was improved through the incorporation of impact modifier. Characterization of the produced HA was carried out through thermogravimetric (TGA) and field emission scanning electron microscope (FESEM) analysis. Spectrum obtained for the HA through Fourier Transform Infrared Spectroscopy was also compared with standard HA. Likewise, X-ray diffraction analysis of the HA in comparison with International Centre for Diffraction Data (ICDD) index for standard HA was conducted. On the other hand, Ca/P ratio of the produced HA was verified through Energy Dispersive X-ray analysis for elemental analysis. Likewise, different characterization techniques were used to characterize the composite produced. These include Fourier transforms infrared spectroscopy (FTIR), thermogravimetric analysis (TGA), Differential Scanning Calorimetry (DSC), Dynamic Mechanical Analysis (DMA), tensile, flexural and impact analysis. Also microbial properties of the produced HA and its composite with PLA were assessed. In addition, in vitro biocompatibility study was used to assess the cell attachment and cell proliferation properties of the composites. Results showed that modification of HA led to increased HA dispersion within the PLA matrix, which resulted into significantly higher mechanical, thermal and dynamic mechanical properties of the resulting composite. Similarly, impact properties of the PLA-HA composite was remarkably improved after incorporation of biostrong impact modifier. In addition, in vitro study revealed that the PLA-HA composite exhibits good biocompatibility properties. In general, the results from this study shows that combination of the salient properties of HA with the good mechanical properties of PLA holds great potential for production of bone replacement composite materials with good load bearing ability. The composite produced herein can help to overcome the secondary operation procedures often associated with the conventional bone replacement procedures.
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LIST OF ABBREVIATIONS

ACP  Amorphous Calcium Phosphate
ATCC  American Type Culture Collection
ALP  Alkaline Phosphate
ASCs  Adipose Derived Stem Cells
BS  Biostrong
BMD  Bone Mineral Density
CDHA  Calcium-deficient Hydroxyapatite
CHNS  Carbon, Hydrogen, Nitrogen, Sulfur
DB  Database
DCPA  Dicalcium Phosphate Anhydrous
DCPD  Dicalcium Phosphate Dihydrate
DLPLGA  D,L- Poly(lacti acid-co-glycolic acid)
DMA  Dynamic Mechanical Analysis
DMEM  Dulbecco’s modified Eagle’s medium
DSC  Differential Scanning Calorimetry
DTG  Differential Thermal Gravimetry
EDX  Energy Dispersive X-ray
FA, FAp  Fluorapatite
FBR  Foreign Body Response
FDA  Food and Drug Administration
FESEM  Field Emission Scanning Electron Microscope
FM  Flexural Modulus
FS  Flexural Strength
FTIR  Fourier Transforms Infrared Spectroscopy
FWHM  Full Width at Half Maximum
HA  Hydroxyapatite
HA-PLGA  Hydroxyapatite, Poly(lacti acid-co-glycolic acid)
HDPE  High Density Polyethylene
GP  Giga Pascal
ICDD  International Centre for Diffraction Data
IS  Impact Strength
LB  Luria Bertani
MPa  Mega Pascal
MSCs  Mesenchymal Stem Cells
MCPA  Monocalcium Phosphate Anhydrous
MCPM  Monocalcium Phosphate Monohydrate
PBS  Phosphate Buffered Saline
PBS  Poly(butylene succinate)
PCL  Poly(caprolactone)
PDLA  Poly(D- Lactide)
PEA  Poly(ester amides)
PEG  Poly(ethylene glycol)
PE  Polyethylene
PEO  Polyethylene Oxide
PET  Polyethylene Terephthalate
PGA  Poly(glycolic acid)
PHA  Poly(lactic acid), Modified Hydroxyapatite
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