

HYDROTHERMALLY EXTRACTED NANOHYDROXYAPATITE FROM
BOVINE BONE AS BIOCERAMIC AND BIOFILLER IN
BIONANOCOMPOSITE

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DEDICATION

To my supportive and loving parents (Mr. and Mrs. Muhammad Aslam Sandhu) whose prayer and affection are the source of strength and sign of success for my bright future. They always encourage me to get the highest goal of my life. Their everlasting love, guidance and encouraging passion will remain with me Insha'Allah till my last breath.



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ABSTRACT

Bones have an extraordinary capacity to restore and regenerate in case of minor injury. However, major injuries need orthopedic surgeries that required bone implant biomaterials. In this study, n-HAP powder was extracted from bovine bone by hydrothermal and calcined at different calcination temperatures (600-1100°C) without the use of solvents. The n-HAP powders produced were used to fabricate two types of biomaterials (HAP bioceramics and PLA/n-HAP bionanocomposite). The raw-HAP and calcined n-HAP powder samples were compacted into green bodies and were sintered at various temperatures (1000-1400°C) to produce HAP bioceramics. The best calcined n-HAP was mixed with PLA by melt mixing and injection moulding to fabricate PLA/n-HAP bionanocomposite. Characterizations of the n-HAP powder, n-HAP bioceramics and PLA/n-HAP bionanocomposite samples were done by Thermogravimetric analysis (TGA), X-ray diffraction (XRD), Fourier transforms infrared (FTIR), Field emission scanning electron microscopy (FESEM), Energy-dispersive x-ray spectroscopy (EDX), X-ray fluorescence (XRF) spectroscopy, universal testing machine (UTM) and melt flow index (MFI) analyses. TGA data revealed that n-HAP was thermally stable at 1300°C. The extracted n-HAP powder was highly crystalline and crystallite size was in the range of 10-83 nm as confirmed by XRD. Density and hardness of the n-HAP bioceramics increased as sintering temperature increased and showing maximum values at a temperature of 1400°C. The results of PLA/n-HAP bionanocomposite revealed that the higher n-HAP loaded (at 5wt%), the lower the tensile strength of bionanocomposite due to poor interfacial adhesion. The interfacial adhesion was improved by loading of 1.0 wt% maleic anhydride (MAH) as a compatibilizer. The biocompatibility of bionanocomposite was evaluated in simulated body fluids (SBF). The results showed that apatite layers were grown on the surfaces of both biomaterials. Therefore, both biomaterials formulated shall be promising medical biomaterials for orthopedic applications.

ABSTRAK

Tulang memiliki kemampuan pemulihan serta pembentukan semula yang luar biasa bagi kes kecederaan kecil. Namun, bagi kecederaan serius, pembedahan ortopedik melibatkan biobahan implan tulang adalah diperlukan. Dalam kajian ini, n-HAP diekstrak dari tulang lembu pada pelbagai suhu kalsin ($600-1100^{\circ}\text{C}$) menerusi kaedah hidroterma dan pengkalsinan, tanpa menggunakan sebarang pelarut. Serbuk n-HAP yang terhasil digunakan dalam fabrikasi dua jenis biobahan (bioseramik HAP dan nanobiokomposit PLA/n-HAP). Sampel-sampel HAP mentah dan n-HAP terkalsin telah dipadatkan sebagai jasad anum, lalu disinter pada pelbagai suhu ($1000-1400^{\circ}\text{C}$) untuk penghasilan bioseramik. n-HAP terkalsin yang terbaik dicampurkan dengan PLA secara percampuran lebur serta pengacuanan suntikan bagi proses fabrikasi nanobiokomposit PLA/n-HAP. Pencirian terhadap serbuk n-HAP, bioseramik n-HAP dan nanobiokomposit dilakukan menerusi analisis termogravimetri (TGA), pembelauan sinar-X (XRD), inframerah transformasi Fourier (FTIR), mikroskop elektron imbasan pancaran medan (FESEM), spektroskopi sinar-X serakan tenaga (EDX) dan pendafluor sinar-X (XRF), mesin ujian universal (UTM) dan indeks aliran lebur (MFI). Data TGA menunjukkan bahawa n-HAP mempunyai kestabilan terma pada 1300°C . n-HAP terekstrak mempunyai kehabluran yang tinggi, dan saiz kristalit berjulat 10-83 nm berdasarkan kepada ujian XRD. Ketumpatan serta kekerasan bioseramik n-HAP bertambah selaras dengan kenaikan suhu sinter, dan menunjukkan nilai maksimum pada suhu 1400°C . Keputusan ujian bagi nanobiokomposit PLA/n-HAP menunjukkan bahawa peningkatan jumlah n-HAP (pada 5wt%) menurunkan kekuatan tegangan nanobiokomposit berikutan pelekatan antaramuka yang lemah. Pelekatan antaramuka dipertingkatkan oleh penambahan 1 wt% maleik anhidrida (MAH). Aspek bioserasi bagi nanobiokomposit diuji di dalam simulasi cecair badan (SBF). Keputusan menunjukkan pertumbuhan apatit pada permukaan kedua-dua biobahan. Oleh itu, kedua-dua biobahan yang diformulakan adalah berpotensi sebagai biobahan perubatan untuk aplikasi ortopedik.

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LIST OF SYMBOLS AND ABBREVIATIONS

<i>cm</i>	: Centimetre
<i>cm</i> ²	: Centimetre Square
<i>cm</i> ³	: Centimetre Cubic
°C/min	: Degree Celsius per Minute
<i>D</i>	: Diameter
<i>d</i>	: Interspacing between Diffraction Lattice Plane
ΔT	: Change in Temperature
ΔH_m	: Change in Melt Enthalpy
<i>F</i>	: Force (N)
<i>GPa</i>	: Giga Pascal
<i>h</i>	: Hours
<i>Kg</i>	: Kilogram
<i>kt</i>	: Metric kilo tons
<i>L</i>	: Length (cm)
λ	: Wavelength
μm	: Micrometre
μ	: Micron
<i>mm</i>	: Millimetre
<i>mg</i>	: Milligram
<i>MPa</i>	: Mega Pascal
<i>nm</i>	: Nanometre
<i>Pa</i>	: Pascal
%	: Percentage
<i>P</i>	: Pressure
<i>Q₃</i>	: Cumulative Distribution by Volume or Mass
<i>q₃</i>	: Density Distribution by Volume or Mass
ρ	: Density, g/cm ³

η	: Lattice Strain
θ	: Diffraction Angle
T_m	: Melting Temperature
T_c	: Crystallization Temperature
T_{cc}	: Cold Crystallization Temperature
T_g	: Transition Temperature
T	: Temperature (°C)
W	: Width (mm)
$wt\%$: Weight Percentage
X_c	: Crystallinity
ATR	: Attenuated Total Reflectance
AFM	: Atomic Force Microscopy
$ASTM$: American Society for Testing and Materials
Al_2O_3	: Aluminium Oxide
$\alpha-TCP$: Alpha Tri Calcium Phosphate
$\beta-TCP$: Beta Tri Calcium Phosphate
BET	: Brunauer–Emmett–Teller
$CAGR$: Compound Annual Growth Rate
Cl	: Chlorine
$CsCl$: Cesium Chloride
CaO	: Calcium Oxide
$CaCO_3$: Calcium Carbonate
$d-HAP$: Ca-Deficient HAP
DMA	: Dynamic Mechanical Calorimetry
DNA	: Deoxyribonucleic Acid
DCP	: Dicumyl Peroxide
DMA	: Dynamic Mechanical Analysis
DSC	: Differential Scanning Calorimetry
EDX	: Energy-dispersive X-Ray
FDA	: Food and Drug Administration
Fe_2O_3	: Ferro-oxide
$FTIR$: Fourier-Transform Infrared Spectroscopy
$FESEM$: Field Emission Scanning Electron Microscopy
$FWHM$: Full Width at Half Maximum

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