

CHARACTERIZATION AND PROCESSING OF COMPOSITE PP/UHMWPE
FILAMENT FOR FUSED DEPOSITION MODELLING APPLICATION

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To the souls of my beloved mother and my great father

They are truly being missed



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May Allah bless us all!

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ABSTRACT

In this thesis Polypropylene/Ultra-high Molecular Weight Polyethylene (PP/UHMWPE) biomaterial composite was employed to manufacture implant to reconstruct large skull bone defect using Fused Deposition Modelling (FDM). This effort aimed to reduce the cost and processing time of manufacturing of a product like this and make it available to all. The effects of addition UHMWPE on the mechanical, thermal, rheology and toxicity of PP were investigated. All the blends composition were compounded using melt blending in an internal mixer and then extruded into single filaments characterized according to FDM filament specification. Subsequently, the prepared filament was underfed to FDM to manufacture tensile, flexural, and impact samples. This was done under the default setting of process parameters in order to investigate the mechanical behaviour of the composite. Structural morphology of the fracture surfaces of impact samples were investigated to explore microstructure changes related to UHMWPE content. Furthermore, thermal and rheological characterizations were conducted to explore the degradation temperature and process ability of the composites in order to accomplish successful processing in both filament extrusion and FDM processes. MTT assay was also conducted to investigate the composite toxicity. Full and Fractal Factorial Design were employed to investigate the effect of process parameters on the process response for filament extrusion and FDM processes respectively. The study results proved that the addition of 10% of UHMWPE resulted in 57% improvement in impact strength, 9.6% improvement in thermal stability, and 17.9 % in biocompatibility compared to pure PP. In addition, the impact strength improved once again at an estimation of 40.6% increment due to optimization of FDM setting parameters. As a case study, a skull implant was manufactured for a patient in USM-University Hospital with 88.13%-dimensional accuracy.

Keywords: Biomaterials melt blending, Polymer extrusion, Fused Deposition Modelling (FDM), Bone reconstruction,

ABSTRAK

Dalam tesis ini, komposit biomaterial *Polypropylene/Ultra-high Molecular Weight Polyethylene* (PP/UHMWPE) telah digunakan untuk pembuatan implan bagi tujuan pembinaan semula sebuah rangka tengkorak yang telah rosak dengan menggunakan kaedah *Fused Deposition Modeling* (FDM). Ini adalah untuk mengurangkan kos dan masa pembuatan sesebuah produk seperti ini serta membolehkan kaedah ini digunakan secara meluas. Kesan-kesan pertambahan UHMWPE (daripada 10% hingga 50%) ke atas aspek mekanikal, termal, reologi dan ketoksikan PP telah dikaji. Semua komposisi adunan telah disebatikan dengan menggunakan kaedah adunan leburan dalam sebuah pengadun dalaman yang disemperitkan kepada filamen-filamen individu yang setiap satunya mengikut ciri-ciri spesifikasi filamen FDM. Seterusnya, filamen-filamen ini telah melalui proses FDM untuk menghasilkan sampel-sampel yang mempunyai tegangan, lenturan dan impak. Ini dilakukan dengan mengikut tetapan asas proses parameter bagi mengkaji sifat mekanikal komposit berkenaan. Struktur morfologi pada permukaan yang telah pecah pada sampel impak telah dikaji untuk meneroka perubahan struktur mikro berkaitan isi kandungan UHMWPE. Berikutnya, proses pencirian termal dan reologi telah dijalankan untuk mengetahui penurunan suhu dan kemampuan komposit untuk diproses bagi menjayakan kedua-dua proses penyemperitan filamen dan FDM. Cerakin MTT juga telah dilaksanakan untuk mengkaji ketoksikan komposit. Rekabentuk *Full and Factorial* telah digunakan untuk mengkaji kesan proses parameter ke atas proses tindakbalas terhadap proses penyemperitan filamen dan FDM. Hasil kajian menunjukkan, berbanding dengan PP tulen, penambahan 10% UHMWPE telah menghasilkan 57% peningkatan kekuatan impak, peningkatan 9.6 % kestabilan termal dan peningkatan 17.9% pada aspek keserasian bio. Kekuatan impak juga telah meningkat sekali lagi pada jangkaan sebanyak 40.6% disebabkan tetapan FDM yang telah dioptimakan. Sebagai sebuah kajian kes, sebuah implan tengkorak telah dibina sebagai sebuah paten di Hospital Universiti di USM.

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LIST OF SYMBOLS AND ABBREVIATIONS

ABS	-	Acrylonitrile Butadiene Styrene
AFD	-	Adaptable Filament Deposition
AG	-	Air Gap
Ag-SiO ₂	-	Spherical Silica Containing Immobilized Nano-Silver
AM	-	Additive Manufacturing
ANOVA	-	Analysis of Variance
ASA	-	Acrylonitrile Styrene Acrylate
ASTM	-	American Society for Testing and Materials
ATOS	-	3-D Scanner Software
BD	-	Bone Density
°C	-	Degree Centigrade
CAD	-	Computer Aided Design
CaSt ₂	-	Calcium Stearate
CIM	-	Conventional Injection Moulded
Cu-SiO ₂	-	Spherical Silica Containing Immobilized Nano-Copper
DICOM	-	Digital Imaging and Communications in Medicine
DMSO	-	dimethylsulfoxide solution
DOE	-	Design of Experiment
DSC	-	Differential Scanning Calorimetry
3DP	-	Three-Dimensional Printing
E_{FF}	-	Young's Modulus for Pure PP
FDM	-	Fused Deposition Modelling
FETI	-	Finite Element Tearing and Interconnecting
FRFD	-	Fractional Factorial Design
FUFD	-	Full Factorial Design
g	-	Gram
GPa	-	Gigapascal
HBSS	-	Hank's Balanced Salt Solution

iPP	-	Isotactic polypropylene
J	-	Joule
LDPE	-	Low Density Polyethylene
LLDPE	-	Linear Low Density Polyethylene
LT	-	Layer Thickness
M	-	Molar
m	-	Meter
M_{ai}	-	Weight under Air Pressure
MFI	-	Melt Flow Index
Min	-	minute
mM	-	Milli-Molar
mm	-	Millimetre
Mpa	-	Mégapascal
MRI-	-	Magnetic Resonance Imaging
MTT	-	3-(4,5-dimethylthiazol-2-yl)-2,5-diphenyltetrazolium bromide
M_w	-	Molecular Weight
M_{water}	-	Weight under Water Pressure
N	-	Newton
NMWPE	-	Normal Molecular Weight Polyethylene
OSIM	-	Oscillatory Shear Injection Moulding
PC	-	Polycarbonate
PCL	-	Poly(ϵ -caprolactone)
PCL/MMT/HA-	-	Polycaprolactone/Montmorillonite/Hydroxyapatite Composite
PDLA	-	Poly-D Lactic Acid
PEEK	-	Poly(etheretherketone)
PEI	-	Polyetherimide
PGA	-	Polyglycolide
PIII	-	Plasma Immersion Ion Implantation
PLA	-	Polylactic Acid
PLLA	-	Poly-L Lactic Acid
PLT	-	Paper Lamination Technology
PMMA	-	Polymethylmethacrylate
PP	-	Polypropylene
PP-b-LLDPE	-	Polypropylene-Linear Low Density

PPSF	-	PolyPhenylSulfone
PU	-	Polyurethane
PVC	-	Polyvinyl Chloride
R ²	-	Coefficient of Multiple Correlations
RA	-	Raster Angle
RM	-	Malaysian Ringgit
RP	-	Additive Manufacturing
RPM	-	Revolution per Minute
RPS	-	Roller Pulley Speed
S ⁻¹	-	Per Second
SLA	-	System Stereolithography Apparatus
SL	-	Single Laser Beam
SLS	-	Selective Laser Sintering
SS	-	Screw Speed
SSSP	-	Solid-State Shear Pulverization
STL	-	Stereolithography
T _{10%}	-	Initial Decomposition Temperature
T _{99%}	-	Temperature at 99% Weight Loss
TEM	-	Transmission Electron Microscopy
T _c	-	Crystallization Temperature
TGA	-	Thermogravimetric Analysis
Ti	-	Titanium
T _m	-	Melting Temperatures
T _p	-	Peak of Decomposition Temperature
UHMWPE	-	Ultra-high Molecular Weight Polyethylene Polyethylene Compatibilizer
UV	-	Ultraviolet
VE	-	Vitamine E
VGCF	-	Vapour-Grown Carbon Fibres
VLDPE	-	Very Low-Density Polyethylene
WHO	-	World Health Organization
wt%	-	Percentage of weight
χ	-	Crystallinity
ρ	-	Density

ΔH_f - Fusion Heat

μL - Micro-litter



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