

**UNIVERSITI TEKNOLOGI MARA**

**EFFECT OF PALM STEARIN AS  
SINGLE BASED BINDER ON  
PROPERTIES OF CERAMIC  
INJECTION MOLDED OF  
HYDROXYAPATITE FOR SCAFFOLD  
APPLICATIONS**

**SITI NORAZLINIBINTIABD AZIZ**

Thesis submitted in fulfillment of  
the requirements for the degree of  
**Master of Science**

**Faculty of Mechanical Engineering**

November 2017

## ABSTRACT

Palm stearin (PSr) or known as vegetable fat has been introduced in injection moulding processes which has great potential as an alternative binder in a binder system. This study presents the processing of Hydroxyapatite ( $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ) mixed with 100 vol.% of single based binder palm stearin (PSr) using CIM. The main objective of the research was to determine the optimum formulation of powder-binder mixture via mixing torque and rheological analysis. The process generally started with the selected commercial HAp powder and the binder in correct proportions. From the CPVP test, the critical loading was 70.64%, thus the formulation was formulated below 2-5 vol.% critical loadings. Four different volume fractions of the powder were investigated: 62, 64, 66, and 68 vol.%. During the rheological test, the feedstock containing the mixture and the binder exhibited pseudoplastic properties, which was lower than 200 Pa.s with function of shear rate. The injection moulding process was successfully carried out in the injection temperature range of 65°C to 70°C which corresponded to 300 kPa to 400 kPa injection pressure being applied and followed the ASTM CI424-10. Compared to other previous research, the temperature used was slightly higher than 130°C due to the absence of a backbone binder. The moulded dumbbell shape was completely debound and sintered in one complete cycle using a single furnace with the presence of wicking media to remove the PSr binder. Next, the sample was subsequently sintered at three different temperatures of 900°C, 1000°C and 1100°C to allow diffusion of the particles. Besides that, the DSC result showed the melting point of the PSr binder was at 62°C, thus the mixing temperature was applied slightly higher at 70°C which allowed for homogenous mixing. The shrinkage result showed that higher formulation (68 vol.%) resulted in - lower shrinkage properties; higher sintering temperature contributed to higher shrinkage properties. This was due to the higher powder loading which meant smaller compact volume shrinkage and easier dimension tolerance control, which is very important for complex injection molded parts. Next, the pore size of the as-sintered sample was measured using a micrometric instrument. The porosity result obtained showed that higher volume fraction of the powder loading resulted in higher porosity; 62 vol.% resulted in about 38% porosity and 68 vol.% resulted in about 41% porosity respectively. The porous structure shown in SEM images was highly interconnected; thus it promoted better bone tissue ingrowth for implant application. The XRD result obtained showed the TCP formation was at 1100°C thus decreasing the compressive strength of HAp.

## ACKNOWLEDGMENT

In the name of Allah, the Most Gracious and the Most Merciful.

All praises and thanks to Allah that gave the direction to me to finish my studies and my whole life has always been a testament to His glory. I would like to say and express my deep and sincere gratitude to my advisor, Assoc. Prof. Dr. Mimi Azlina Abu Bakar, for her kind guidance, advice and support throughout my master journey at UiTM Shah Alam. Her wide knowledge and logical way of thinking became a great value for me. I also want to express gratitude to my supportive co-supervisor, Dr. Muhammad Hussain Ismail, who introduced me to ceramic injection moulding field. In addition, my thanks goes to those who gave me important guidance, supportive guide and motivation for me to finish my journey till my last word of the thesis.

I want to warmly thank Dr. Azudin Mamat, lecturer from UM and all lecturers, assistant engineers, FKM post graduates members and CAMAR from FKM for any kind of discussion, moral support, and encouragement that I cannot explain here. I would like to express heartiness gratitude to my family members especially my great husband, beloved mom and dad who always keep supporting me through my journey in this master studies. Not to forget the financial support from Exploratory Grant Scheme (ERGS,600-RMI/EGRS 5/3 (25/2013) Universiti Teknologi MARA Shah Alam.

# TABLE OF CONTENT

	<b>Page</b>
<b>CONFIRMATION BY PANEL OF EXAMINERS</b>	<b>ii</b>
<b>AUTHOR'S DECLARATION</b>	<b>iii</b>
<b>ABSTRACT</b>	<b>iv</b>
<b>ACKNOWLEDGMENTS</b>	<b>iii</b>
<b>TABLE OF CONTENTS</b>	<b>vi</b>
<b>LIST OF TABLES</b>	<b>x</b>
<b>LIST OF FIGURES</b>	<b>xii</b>
<b>LIST OF SYMBOLS</b>	<b>xvi</b>
<b>LIST OF ABBREVIATIONS</b>	<b>xviii</b>
<b>LIST OF CHEMICAL FORMULA</b>	<b>xx</b>
<b>CHAPTER ONE: INTRODUCTION</b>	
1.1 Background of Study	1
1.2 Problem Statement	4
1.3 Research Objectives	5
1.4 Scope of Study	6
1.5 Thesis Outline	7
<b>CHAPTER TWO: LITERATURE REVIEW</b>	
2.1 Demand of Ceramic Implant	8
2.1.2 Ceramic Material Used in Implant	10
2.1.2.1 Biomaterials	11
2.1.2.2 Bioceramics	13
2.1.3 Advantages of Ceramic Implant Over Metal Implant	17
2.1.4 Hydroxyapatite (HAp)	20
2.2 Processing of Ceramic Implant	23

2.3	Ceramic Injection Moulding Method	26
2.3.1	Powder Attributes	28
2.3.2	Binder Attributes	29
2.3.3	Binder System	30
2.3.3.1	Palm Stearin	33
2.3.4	Feedstock Preparation and Rheological Behavior	35
2.3.5	Injection Moulding	39
2.3.6	Debinding Process	41
2.4	Sintering Process	46
2.4.1	Solid State Sintering (SSS)	46
2.4.2	Liquid State Sintering	48
2.5	Conclusions	50

### **CHAPTER THREE: METHODOLOGY**

3.1	Introduction	52
3.2	Characterization of Ceramic Powder (Hydroxyapatite)	52
3.2.1	Particle Size Distribution and Density	52
3.2.2	X-ray Diffractometer (XRD)	55
3.2.3	Fourier Transform Infrared Spectroscopy (FTIR) Analysis	56
3.2.4	Energy Dispersive X-ray Spectroscopy (EDS)	57
3.2.5	Tracing Element Concentration Using Inductive Couple Plasma Optical Emission Spectroscopy (ICP-OES)	57
3.3	Characterization of Palm Stearin Binder System	57
3.3.1	Differential Scanning Calorimetry (DSC)	59
3.3.2	Thermogravimetric Analysis (TGA)	60
3.4	Hydroxyapatite Feedstock Preparation	60
3.5	Feedstock Characterization	61
3.5.2	Rheological Analysis	62
3.6	Injection Molding Process	63
3.6.1	Thermal Debinding & Sintering	64