

**UNIVERSITI TEKNOLOGI MARA**

**POWDER INJECTION MOULDING OF  
SYNTHESIZED HYDROXYAPATITE  
POWDER FROM CLAMSHELL**

**NURUL HUDA BINTI M. ALI**

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

**Faculty of Chemical Engineering**

August 2015

## **CONFIRMATION BY PANEL OF EXAMINERS**

I certify that a Panel of Examiners has met to on 28<sup>th</sup> April 2015 to conduct the final examination of Nurul Huda Binti M. Ali on her Master of Science thesis entitled “ Powder Injection Moulding of Synthesized Hydroxyapatite Powder from Clamshell” in accordance with Universiti Teknologi MARA Act 1976 (Akta 173). The Panel of Examiners recommends that the student be awarded the relevant degree. The Panel of Examiners was as follows:

Jailani Salihon, Ir. PhD  
Professor  
Faculty of Chemical Engineering  
Universiti Teknologi MARA  
(Chairman)

Norliza Ibrahim, PhD  
Senior Lecturer  
Faculty of Chemical Engineering  
Universiti Teknologi MARA  
(Internal Examiner)

Abu Bakar Sulong, Ir. PhD  
Associate Professor  
Faculty of Mechanical and Materials Engineering  
Universiti Kebangsaan Malaysia  
(External Examiner)

**SITI HALIJAH SHARIFF, PhD**  
Associate Professor  
Dean  
Institute of Graduates Studies  
Universiti Teknologi MARA  
Date: 17<sup>th</sup> August, 2015

## ABSTRACT

Hydroxyapatite (HAp) is one of the most versatile bioceramic materials since it is widely used in biomedical and dentistry applications. This research focused on the effect of pH and calcination temperature on synthesized HAp powder from clamshells via chemical precipitation method. Besides that, the process of PIM using synthesized HAp powder mixed with PS based binder system had studied. The effect of sintering temperatures on the as-sintered HAp specimens had evaluated for its physical and mechanical properties. The process to synthesize HAp powder involves calcination of clamshells powder followed by the titration method using low concentration of phosphoric acid. Based on the analysis, synthesized HAp powder at the temperature of 850°C with the final pH solution of 6.5 has similar characterizations with commercial HAp powder. Then, the process is followed by a green processing route via PIM technique to produce the as-sintered HAp specimen. From the results attained, it shows that synthesized HAp feedstock prepared with the powder loading of 60 vol.% shows pseudo-plastic behavior. The feedstock was successfully injection moulded according to ASTM standard C1424-10 to produce green specimens at the temperature of 200°C with the pressure range from 4 to 7 bars. The green specimens were then successfully debound and sintered through single step wick-debinding and sintering process using alumina powder as an embedment agent. The sintered specimens were then evaluated on physical and mechanical properties. From the results obtained, sintering temperature above 1100°C is not preferable since HAp is start to decompose and forming TCP which not suitable for load bearing applications.

# TABLE OF CONTENTS

	<b>Page</b>
<b>CONFIRMATION BY PANEL OF EXAMINERS</b>	ii
<b>AUTHOR'S DECLARATION</b>	iii
<b>ABSTRACT</b>	iv
<b>ACKNOWLEDGEMENT</b>	v
<b>TABLE OF CONTENTS</b>	vi
<b>LIST OF TABLES</b>	xi
<b>LIST OF FIGURES</b>	xiii
<b>LIST OF SYMBOLS</b>	xvi
<b>LIST OF ABBREVIATION</b>	xviii
<b>LIST OF CHEMICAL FORMULA</b>	xx
<b>CHAPTER ONE: INTRODUCTION</b>	1
1.1 Research Background	1
1.2 Problem Statement	3
1.3 Research Objectives	4
1.4 Scope of the Research	4
1.5 Thesis Outline	6
<b>CHAPTER TWO: LITERATURE RIVIEW</b>	7
2.1 Introduction	7
2.2 Human Bone	7
2.3 Biomaterial	10
2.4 Bioceramic	12
2.5 Hydroxyapatite (HAp)	13
2.5.1 Synthetic Hydroxapatite (HAp) from Natural Material Sources	16

# CHAPTER ONE

## INTRODUCTION

### 1.1 RESEARCH BACKGROUND

Over the past of several years, owing to greater demands of implant material in biomedical sectors, it seems that research on hydroxyapatite [HAp,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ] powder has addressed remarkable attention by many materials researchers and medical practitioners. The main reason of HAp is widely used as an implant material (Prabakaran and Rajeswari, 2009) is due to its chemical similarity in natural bone material with the composition of 70% in human bone (Cui, Nelson, Peng, et al. 2012). In addition, HAp contains a compound of calcium (Ca) and phosphorus (P) with the molar ratio of Ca/P approximately equal to 1.67 which is almost equal to the Ca/P ratio for the natural bone (Mittal, Prakash, Nath and Sapra, 2010). Thus, it makes HAp becomes a suitable material for the implant purposes.

It is well known that HAp has been used extensively in various biomedical applications such as in bone repair, orthopedic, as coating for metallic prostheses and bone grafting applications (MacLaine, Meek and Brydone, 2010), (Afshar, Ghorbani, Ehsani, Saeria and Sorrell, 2003). This is due to it's an excellent biocompatibility, bioactivity, osteoconductivity and non-toxicity properties (Kumar, Thamizhavel and Girija, 2012). However, the critical issue in utilizing the HAp is its cost tremendously expensive. Therefore, researchers around the world have been attempting to find an alternative route in producing HAp by synthesis technique from any potential waste material using the concept of "waste to wealth".

Recently, HAp has been successfully synthesized from natural waste material sources such as oyster shells (Wu, Hsu, Wu and Ho, 2011), corals (Ripamonti, Crooks, Khoali and Roden, 2009) eggshells (Prabakaran and Rajeswari, 2009) (Kumar et al., 2012) and seashell (Vecchio, Zhang, Massie, Wang and Kim, 2007) owing to high content of  $\text{CaCO}_3$  which is the important calcium source for the preparation of HAp (Ho, Hsu, Hsu, Hung and Wu, 2013). However, the research on HAp from waste material of