UNIVERSITI TEKNIKAL MALAYSIA MELAKA

PREPARATION OF POROUS HYDROXYAPATITE VIA POLYMERIC SPONGE METHOD

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials)

by

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ABSTRAK

Kaedah span polimerik ialah satu kaedah yang digunakan untuk menyediakan sampel hidroksiapatit berliang dengan menggunakan serbuk hidroksiapatit komersial. Kesan-kesan kadar pensinteran, masa kacauan dan tumpuan hidroksiapatit pada keporosan, kekuatan mampatan, kehabluran badan-badan berliang, mikrostruktur, pengikatan dan molekul dan suhu akan dikaji. Ia dijangka bahawa peningkatan serbuk yang dimasukkan ke dalam komposisi sluri akan mengakibatkan ketumpatan yang lebih tinggi dan peningkatan kekuatan mampatan pada badan-badan berliang hidroksiapatit. Bagaimanapun, komposisi optimum pemuatan hidroksiapatit akan dikaji semasa kajian ini seolah-olah tumpuan serbuk hidroksiapatit di dalam sluri itu terlalu tinggi ia akan menghasilkan sluri dengan kelikatan tinggi yang mana membawa kepada kesukaran dalam mensenyawakan span berselulosa. Sebaliknya, jika tumpuan hidroksiapatit di dalam sluri itu adalah terlalu rendah, jumlah zarah-zarah hidroksiapatit akan berkurang mengakibatkan kekuatan mampatan lebih rendah dan ketumpatan yang rendah. Kadar pemanasan proses pensinteran hidroksiapatit berliang juga akan diliputi dalam kajian ini. Dijangka kadar pemanasan yang perlahan akan memberi ciri-ciri hidroksiapatit berliang yang lebih baik dalam soal ketumpatan, kekuatan mampatan dan kehabluran berbanding dengan pemanasan yang lebih cepat. Dalam kajian ini, peningkatan beban serbuk menyebabkan peningkatan dalam ketumpatan dari 0.37 g/cm^3 sehingga 0.66 g/cm^3 dan penghabluran. Kajian ini mendapati bahawa kadar serbuk pada 75% adalah komposisi optimum untuk hidroksiapatit. Untuk kadar pemanasan, keputusan yang diperolehi menunjukkan pemanasan yang lebih lambat memberikan ketumpatan lebih tinggi iaitu 0.48 g/cm^3 dan penghabluran yang lebih tinggi berbanding pemanasan yang lebih cepat. Sebuah masa pengacauan yang lebih lama juga menghasilkan keputusan yang sama seperti kadar pemanasan lambat dimana mengakibatkan ketumpatan dan penghabluran yang lebih tinggi.

ABSTRACT

Polymeric sponge method is a method that used to prepare porous hydroxyapatite sample by using commercial hydroxyapatite powder. The effects of sintering rate, stirring time and hydroxyapatite concentration on porosity, compressive strength, crystallinity of the porous bodies, microstructure, molecular and bonding and temperature will be studied. It is expected that increasing of powder loading in slurry composition will result in higher densities and increase of compressive strength of the porous hydroxyapatite bodies. However, the optimum composition of hydroxyapatite loading will be investigated during this study as if the concentration of hydroxyapatite powder in the slurry is too high it will produce slurry with high viscosity which leading to difficulty in impregnating the cellulosic sponge. On the other hand, if the hydroxyapatite concentration in the slurry is too low, the number of hydroxyapatite particles will be less resulting in lower compressive strength and low density. Heating rate of sintering process of porous hydroxyapatite also will be covered in this study. It is expected that slow heating rate will give better properties of porous hydroxyapatite in terms of density, compressive strength and crystallinity compared to faster heating. In this study, increase in the powder loading was resulted in increase of apparent density from 0.37 g/cm^3 to 0.66 g/cm^3 and crystallinity. The study found that powder loading of 75% is the optimum composition of hydroxyapatite. For the sintering rate, results obtained show the slower sintering give higher apparent density of 0.48 g/cm^3 and crystallinity than faster sintering rate. A longer stirring time also yielded the same results as slower sintering rate where it resulted in higher apparent density and crystallinity.

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DEDICATION

First and for most I would like dedicate to my late father, Daud Bin Hassan, my siblings and especially my mother, Norisah@Merisah Binti Salleh. They have given me a lot of strength and spirit to wade all this with strength.

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LIST OF ABBREVIATIONS

HA	-	Hydroxyapatite
CHA	-	Carbo-hydroxyapatite
TCP	-	Tricalcium Phosphate
SEM	-	Scanning Electron Microscope
FTIR	-	Fourier Transform InfraRed
TGA	-	Themogravimetric analysis
XRD	-	X-ray Diffraction
Al_2O_3	-	Alumina Oxide
Ti	-	Titanium
ZrO_2	-	Zirconia
PMMA	-	Poly(methyl methacrylate)
UHMWPE	-	Ultrahigh-molecular weight polyethylene
PLA	-	Polylactic acid
PLGA	-	Poly(lactic-co-glycolic acid)
PCL	-	Polycaprolactone
OH	-	Hydroxide
CO_3	-	Carbonate
PO_4	-	Phosphate

CHAPTER 1
INTRODUCTION

Hydroxyapatite (HA) is one of the most biocompatible ceramics because of its significant chemical and physical resemblance to the mineral constituents of human bone and teeth (Swain, 2009). The excellent biocompatibility that it has, make it meets the requirement of any materials designed for bone repair and augmentation (Sopyan, *et al*, 2007). Its major advantage is that it is biologically active in a skeletal site, i.e. bone opposition, rather than fibrous encapsulation, is produced around the implant. It is this feature of favourable bioactivity which distinguishes hydroxyapatite from the various alloys and polymers used in skeletal implants and allows biological, 'cementless', fixation with enhanced long-term survival (Bonfield, 2006).

Hydroxyapatite is a bioactive coating that is usually prepared on the surface of biomedical metal implants, most commonly titanium and its alloys (Gilbert, 2008) to render good bioactivity between the host and the implant while the metallic implant provides mechanical strength for weight-bearing needs (Cheng, *et al*, 2006). Plasma spraying is the most extensively used for applying a hydroxyapatite coating to a metal surface. The hydroxyapatite powder is introduced into a flame that directs the particles for deposition onto the metal surface (Wise, 1996). Nevertheless, long term stability of the plasma-sprayed coatings is an exigent problem because of their high degree of porosities, poor bond strength, non-stoichiometric composition and amorphous structure. Then, the other techniques have been develop such as laser surfacing, sol-gel, magnetron sputtering, ion-beam deposition, pulse-laser deposition, electrochemical deposition and electrophoretic deposition (EPD) (Gilbert, 2008).

Hydroxyapatite can be produced into various forms like porous and dense bodies (Nicholson, 2002). Porous hydroxyapatite shows strong bonding to the bone compared to dense bodies. The pores provide a mechanical interlock leading to firm fixation of the material (Sopyan, *et al*, 2007). The strength of the hydroxyapatite implants will increase as the bone tissue grows well into the pores. When compared to the dense hydroxyapatite, hydroxyapatite in form of porous is more resorbable and more osteoconductive (Swain, 2009). The pores also provide a way for living bone to attach itself permanently to an implant (Ain, *et al*, 2008).

There are a lot of method to produce porous hydroxyapatite, including incorporation of volatile organic particles in hydroxyapatite powder, polymeric sponge method, gel casting foams, starch consolidation, microwave processing, slip casting and electrophoretic deposition technique (Sopyan and Kaur, 2009). In this project, polymeric sponge method is used to produce the porous hydroxyapatite. Polymeric sponge method is performed by impregnating porous cellulosic substrates (sponge) with hydroxyapatite slurry. Slurry is prepared by adjusting hydroxyapatite powder loading.

1.1 Problem Statement

There are several biocompatible metallic materials that are frequently used as implanting materials to replace damaged bone or to provide support for healing bones or bone defects such as stainless steel, titanium, aluminum, vanadium, cobalt, chromium and nickel (Davis, 2003). However, the main disadvantage of metallic biomaterials are their lack of biological recognition on the material surface and possibility release of toxic metallic ions and/or particles through corrosion or wear possible that lead to inflammatory cascades and allergic reactions, which reduce the biocompatibility and cause tissue loss (Alvarez and Nakajima, 2009). For example the excessive cobalt may lead to polycythemi, hypothyroidism, cardiomyopathy and carcinogenesis. Nickel can lead to eczematous dermatitis, hypersensitivity and carcinogenesis. Aluminum also has it drawbacks where it has been associated with

anemia, osteomalacia and neurological dysfunction. Titanium that regarded as inert has been associated with pulmonary disease (Ratner, 2004).

The limitations of those materials was overcome by the introduction of synthetic hydroxyapatite, a calcium phosphate compound which approximates to the bone mineral phase that comprises about 45% by volume and 65% by weight of human cortical bone (Anonymous, 2010). Then the hydroxyapatite in form of porous are then further developed because their interconnected pores can provide a favorable environment for bone ingrowth and osseointegration (Jo, *et al*, 2009). Pores are important, they are conduits for blood flow (blood is generated in bone marrow) and they allow bones to be strong without being too heavy (Ain, *et al*, 2008).

1.2 Objective

The objectives of this project are:

(i). To characterize the physical and chemical properties of hydroxyapatite powder and cellulose sponge used for preparing porous hydroxyapatite.
(ii). To produce porous hydroxyapatite via polymeric sponge method and characterize its physical and chemical properties.
(iii). To optimize the slurry composition in order to achieve the best properties (porosity, compressive strength and crystallinity) of porous hydroxyapatite.
(iv). To evaluate the effect of heating rate on porosity, compressive strenght and crystallinity of porous bodies.

1.3 Scope of Study

Hydroxyapatite powder and cellulose sponge is the main raw material in this project. The porous hydroxyapatite was prepared through polymeric sponge method. In polymeric sponge method, it involves preparation of slurry where hydroxyapatite powder was mixed with distilled water. The cellulose sponge was soaked into the

prepared slurry. It follows by drying the samples at room temperature for 72 hours. The dried samples were then subjected to heat treatment at 600°C in order to burn out the organic matrix. Sintering at 1250°C was then carried out to the samples with variation heating rate at 5°C/min and 20°C/min. The evaluation on effect of heating rate and hydroxyapatite powder loading on the porosity, compressive strength and crystallinity of porous bodies will be implemented.

CHAPTER 2
LITERATURE REVIEW

2.1 Biomaterial

A biomaterial can be defined as any material used to make devices to replace a part or a function of the body in a safe, reliable, economic and physiologically acceptable manner. The purpose of using biomaterials is to improve human health by restoring the function of natural living tissues and organs in the body (Park and Lakes, 2007). Table 2.1 shows the biomaterials classifications and examples.

Table 2.1: Biomaterial classifications (Anonymous, 2001).

Metals	Ceramics	Polymers
316L stainless steel Co-Cr Alloys Titanium Ti6Al4V	Alumina Zirconia Carbon Hydroxyapatite	Ultra high molecular weight polyethylene Polyurethane

Biomaterials are used for some application such as orthopedics applications. All the three types of the material that show in Table 2.1 are used in biomaterial applications. Metallic materials are used for load bearing members like pins, plates and femoral stem. In dental applications, metallic biomaterials are used for anchoring tooth implants and as parts of orthodontic devices. Ceramics like hydroxyapatite is used for bone bonding applications to assist implant integration, coating on metallic pins and to fill large bone voids that caused by disease or trauma. Other ceramic materials like alumina and zirconia are used in wear applications such as joint replacements. Polymers also used in orthondontic devices such as plates and

dentures. Silicones are polymer material that used in cosmetic surgery such as breast augmentation (Anonymous, 2001). Table 2.2 shows the summary of the three types of the materials (metals, ceramics and polymers) used in orthopedic application.

Table 2.2: Orthopedic biomaterials and their primary use (Basu, *et al,* 2009).

Material	Primary uses
Metals Ti alloy (Ti-6%, Al-4%), Co-Cr-Mo alloy, Stainless steel	Bone plates, screws, total-joint arthroplasty (TJA) components, cabling
Polymers Poly(methyl methacrylate) (PMMA), Ultrahigh-molecular weight polyethylene (UHMWPE), PLA, PLGA, HA/PLGA, PCL	Bone cement, low friction inserts for bearing surface in TJA, bone tissue engineering scaffolds, bone screws
Ceramics Alumina (Al_2O_3), Zironia (ZrO_2)	Bearing surface TJA components, hip joints, coating on bioimplants, bone filler, alveolar ridge augmentation
Composites HA/collagen, HA/gelatin, HA/PLGA, PLGA	Bone graft substitute and tissue engineering scaffolds.

2.2 Types of Implant Tissue Response

No material implanted in living tissues is inert; all materials elicit a response from the host tissue. (Hench and June, 1993). In general, materials can be placed into three classes that represent the tissue response they are elicit, which are inert, bioresorbable and bioactive (Ain, *et al*, 2008) as shown in Table 2.3.

Table 2.3: Classes of biomaterials according to tissue response (Ain, *et al*, 2008).

Classes of Biomaterial	Tissue Response	Examples
Bioinert	Mechanical interlock, separation by a fibrous tissue of various thickness	Tantalum, Titanium, Alumina, Zirconia (PSZ), UHMW Polyethylene, Stainless Steel
Bioactive	Direct biochemical bond	High Density Hydroxyapatite, Glass Ceramics A-W, Certain Bioglasses
Bioresorbable	Gradual dissolution, replacement of implants by the tissue	Porous Hydroxyapatite, Tricalcium Phosphate Polyurethane, Polylactic-polyglycollic Acid Copolymer

2.2.1 Bioactive

Bioactive materials are a group of biocompatible materials that can attach directly with body tissues and form chemical and biological bond during early stages of the post implantation period (Basu, *et al*, 2009). The concept of bioactive material is intermediate between a bioinert material and biodegradable or resorbable material. Upon implantation in the host, surface reactive ceramics form a strong bond with an adjacent tissue. The bioactive materials (ceramic, glasses and glass-ceramics) bone to living bone through a carbo-hydroxyapatite layer (CHA) biologically active, which provides the interface union with the host. This phase is chemical and structurally equivalent to the mineral phase of the bone and the responsible of the interface union (Caruta, 2006).

2.2.2 Bioinert

Bioinert materials are biocompatible materials but cannot induce any interfacial biological bond between implants and bone. When a bioinert material is implanted, a capsule-like layer forms on the surface of the implant to keep it isolated from the living part of the body. For example, bioinert ceramics such as alumina or zirconia, develop fibrous capsules at their interface when implanted. However, the thickness of an interfacial fibrous layer depends upon motion and the extent of required fit at the interace. Therefore, bioinert materials are not useful for long-term application (Basu, *et al*, 2009).

2.2.3 Biodegradable / Bioresorbable

Bioresorbable materials are the type of biocompatible materials that are gradually resorbed before they finally disappear and are totally replaced by new tissues *in vivo*. This kind of material that is bioresorbable, degrades with time inside the body's environment. The degradation rate should be such that the regeneration rate of new tissue will be same as the material resorption rate. Tricalcium Phosphate (TCP) and bone cement are the two examples of bioresorbable materials (Basu, *et al*, 2009).

2.3 Metal

Metal are used as biomedical materials because of their excellent mechanical properties and fair biocompatibility but there are some of them that exhibit good biocompatibility, i.e. they do not cause serious toxic reactions in the human body such as stainless steel, cobalt alloys, titanium alloys and noble metals (Shi, 2006). Some metals are used as passive substitutes for hard tissue replacement such as total hip and knee joints as shows in Figure 2.1. The figure shows fracture healing aids of bone plates and screws, spinal fixation devices and dental implants. Those devices have excellent mechanical properties and corrosion resistance. Some metallic alloys