

SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING

UNIVERSITI SAINS MALAYSIA

**OPTIMISATION OF APATITE BIOCERAMIC FORMATION FROM
WASTE DENTAL MOULD**

By

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of the requirements for the degree of Bachelor of Engineering with Honours
(Materials Engineering)

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitles “**Optimisation of Apatite Bioceramic Formation from Waste Dental Mould**”. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or university.

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LIST OF ABBREVIATIONS

DTS	Diametral Tensile Strength
FESEM	Field Emission Scanning Electron Microscope
FTIR	Fourier Transform Infrared Spectroscopy
HA	Hydroxyapatite
ICSD	International Centre of Standard Data
SBF	Simulated Body Fluid
XRD	X-ray Diffraction
XRF	X-ray Fluorescence

LIST OF SYMBOLS

$^{\circ}$	Degree
θ	Theta
% D_s	Percentage of shrinkage in diameter
% t_s	Percentage of shrinkage in thickness
σ_{ult}	Ultimate tensile strength
%T	Percentage of Transmittance
n_1	Number of mole
M1	Molarity of the solution
V1	Volume
λ	Wavelength
d	Interplanar spacing
θ	Scan angle for XRD
D	Dried weight
W	Saturated weight
S	Suspended weight

PENGOPTIMUMAN PEMBENTUKAN APATIT BIOSERAMIK DARI SISA ACUAN GIGI

ABSTRAK

Menurut Pusat Perubatan dan Pergigian Termaju (AMDI), 50 kilogram sisa acuan gigi telah dibuang pada setiap bulan. Acuan gigi tersebut mempunyai kalsium yang kaya dan sesuai dijadikan sebagai bioseramik seperti trikalsium fosfat (TCP) dan hidroksiapatit (HA) yang telah mendapat perhatian dari penyelidik dan ahli-ahli sains. Dalam kajian ini, terdapat dua projek ; sintesis apatit bioseramik dan rawatan haba kepada acuan gigi untuk dikitarsemula menjadi bahan mentah untuk membuat acuan gigi. Dalam projek sintesis apatit bioseramik, Pemprosesan Sisa Biji Kromit telah digunakan. Seterusnya, bahan telah larut dengan alkali kaustik pada 7 molar Natrium Hidroksida sekali dan dua kali pencucian kaustik sebelum dicampur dgn asid Fosforus. Hasil menggunakan 7 molar dua kali pencucian dapat mengurangkan kadar kromium. Untuk projek kedua, sisa batuan gigi telah dipanaskan pada suhu 130 °C untuk 2 jam dan 160 °C dalam masa 4 jam. Sisa bantuan gigi tersebut dapat digunakan sebagai POP.

OPTIMISATION OF APATITE BIOCERAMIC FORMATION FROM WASTE DENTAL MOULD

ABSTRACT

According to Advanced Medical And Dental Institute USM (AMDI), the institute disposed 50 kg of dental mould waste per month. Dental mould waste have possibility of calcium source which can form apatite bioceramics materials when react with phosphoric acid. Bioceramics materials synthesis by dental mould waste produced mainly Tricalcium Phosphate (TCP) or Hydroxyapatite (HA) that attracted researcher and scientist attention recently. In this study, it has two projects; synthesis apatite bioceramics from waste dental mould and heat treatment to waste dental mould for reuse it as raw material for making dental mould. For project of synthesis apatite bioceramics material, it used wet chemical precipitation with adopting Chromite Ore Processing Residue (COPR), following by caustic leaching 7M of Sodium Hydroxide (NaOH) solution for single and double time. The result of using 7M NaOH double leaching is able to reduce chromium content from leached residue, which form white color of apatite bioceramic material when react with Phosphoric acid. Main the phase of apatite material will form Tricalcium Phosphate (TCP) after sintered in 1100 °C. For 2nd project, dental mould waste undergone heat treatment with 130 °C in 2 hour and 160 °C in 4 hour times. The waste dental mould can be reuse as POP.

CHAPTER 1

INTRODUCTION

1.1 Research Background

Ceramic is derived from the Greek word “keramos”, which literally means ‘burnt stuff’, but has become to mean more specifically an inorganic material produced by burning or firing (Rama, 2013). A ceramic is an earthly material usually of silicate nature and may be defined as a combination of one or more metals with a non-metallic element usually oxygen. The American Ceramic Society had defined ceramics as inorganic, non-metallic materials, which are typically crystalline in nature, and are compounds formed between metallic and nonmetallic elements such as aluminum & oxygen (alumina - Al_2O_3), calcium & oxygen (calcia - CaO), silicon & nitrogen (nitride- Si_3N_4). Ceramics are characterized by their refractory nature, hardness, chemical inertness and susceptibility to brittle fracture (Babu et al., 2015).

However ceramics, used as biomaterials to fill defects in tooth and bone, to fix bone grafts, fractures, or prostheses to bone, and to replace diseased tissue, are called bioceramics. They must be highly biocompatible and anti thrombogenic, and should not be toxic, allergenic, carcinogenic, or teratogenic. Bioceramics can be classified into three groups; (1) bioinert ceramics, (2) bioactive ceramics, and (3) bioresorbable ceramics (Yamamuro, 2004). Ceramics for the reconstruction, fixation, and repair of the diseased or broken parts of the musculoskeletal system are known to possess properties such as bioinertness, for example, zirconia and alumina, bioresorption (e.g., β -tricalcium phosphate), bioactivity (e.g., hydroxyapatite, bioglass, and bioactive glass-

ceramics), or porosity for tissue ingrowth (hydroxyapatite-coated alumina). Compositions of such materials can be engineered to obtain desired phases and properties for various applications (Sooksaen et al., 2015).

Biomaterial is a material which can replace a part or substitute a defect component in human body. The biomaterials should be safe, economic, reliable and physiologically acceptable in the human body. It is mostly used in the treatment of injury caused by trauma and disease. It is a synthetic material used to replace the part of body which connects with living tissue.

The characteristics of biomaterials are biocompatibility with tissue, nontoxic & nonallergenic to human body, chemically stable and inert when placed in human body, proper density and weight, easily available with suitable mechanical strength and longer fatigue life. (Park & Lakes, 2007) The application of biomaterials are in joint replacements, bone plates, bone cement, artificial ligaments and tendons, dental implants for tooth fixation, blood vessel prostheses, heart valves, skin repair devices, cochlear replacements and contact lenses (Tathe & All, 2010).

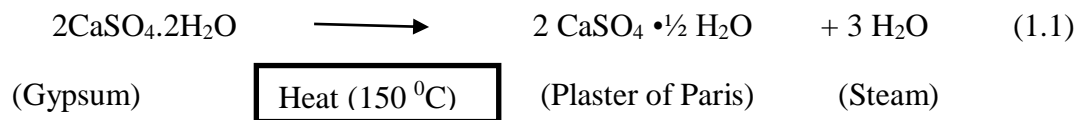
In addition, bioceramic materials are processed to obtain one of four general types of surfaces and associated mechanisms of tissue attachment. First, fully dense relatively inert crystalline ceramics that attach to tissue. Second, porous comparatively inert ceramics into which tissue ingrowth occurs and creating a mechanical attachment. Next, fully dense, surface-active ceramics that attach to tissue via a chemical bond. Lastly, resorbable ceramics that incorporated with tissue and eventually are replaced by host tissue (Adrezin 2004)

Generally, calcium phosphate based bioceramics have been studied as potential substitutes for hard tissue because of their superior bioactivity, excellent biocompatibility, and osteoconductivity (Sooksaen et al., 2015). Calcium phosphate (CaP) based materials are often used in conjunction with different biomolecules to provide surfaces for adsorption and catalysis of biochemical reactions. Adsorption of molecules to CaP surfaces is also dependent on the solubility of the system, in addition to the microstructures (surface area and porosity). Hydroxyapatite ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$) and β -tricalcium phosphate ($\text{Ca}_3(\text{PO}_4)_2$) and their derivatives and combinations are the most commonly used ceramics in orthopedics. Hydroxyapatite ceramic materials provide an osteoconductive matrix for bone in growth, but slow in vivo resorption can potentially limit their clinical applications. On the other hand, β -Tricalcium phosphate has been reported for biocompatibility, osteoconductivity, and higher resorbability but there has been limited data regarding long term outcome of its clinical use for bone tumors (Sooksaen et al., 2015)

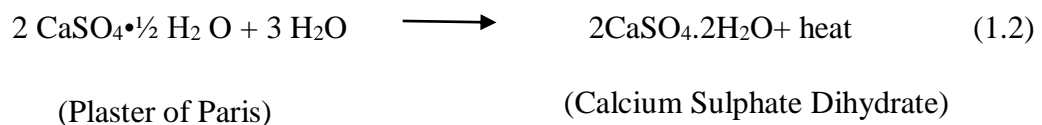
Tricalcium Phosphate (TCP) have 3 polymorphs, which is low-temperature β -TCP, and the high-temperature forms, α - and α' -TCP. However, α' -TCP is hard to exist because it only exist at temperatures more than 1430 °C and it reverts almost instantaneously to α -TCP on cooling below the transition temperature. β -TCP is stable at room temperature and it will transform into α -TCP at 1125°C, which is metastable. It will retain this form until room temperature during the cooling although rapid cooling is required. Theoretically, TCP will degrade when new tissue growth and replace it after implantation. Due to this reason, implantation of TCP does not have problem of interfacial stability in human body (Mirhadi et al., 2011).

Gypsum is one potential source of raw materials for the Calcium Phosphate biomaterials. It is a mineral that is found from earth crust. It can be use as the source of calcium for apatites as Gypsum is composed of calcium sulphate (CaSO₄) and water (H₂O) or Calcium Sulphate Dihydrate (CaSO₄.2H₂O). Plaster of Paris for mould making is produced from Gypsum. Generally, Gypsum will be heated around 150 °C and it will lose water to form Plaster of Paris.

The chemical reaction after heat treatment is according to Equation 1.1[McCabe ,2008]:



When addition of water to Plaster of Paris, it becomes hardened due to water absorption, the rehydration reaction occurs like the following, to form a dehydrate (Equation 1.2):



In the case of dental plaster (Plaster of Paris), it is similarly produced from the heating process. The gypsum will be heated around 120 °C for drive off the water. If without control of the process, it will cause overheating (dead-burned) or would influence the setting characteristics of the final products.

In the case of dental stones, it may be produced from gypsum which is heated around 125°C under steam pressure condition. It will form α -hemihydrate, which produce a less porosity for hemihydrate materials. Most manufacturers will add some chemical component to dental stones which would easily allow people to differentiate that materials as dental stones which is white color (McCabe, 2008).

In this study, it is an attempt to produce apatite from waste dental stone. Apatite could be Hydroxyapatite (HAP) or Tricalcium Phosphate (TCP). Hydroxyapatite, $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ is a primary mineral content for human body's calcified cartilage and bone (Ravaglioli, 1992).

For developed country, such as Austria, Japan and Germany, these countries have efficient solid waste management. They were able toward sustainable waste management such as 'reduce, reuse and recycle' for their waste management (Moh, 2014). Recycle is a must in Malaysia because it can reduce the needs of materials to reduce the impact of environment. Hence, the recycling or reuse the waste dental mould is indeed an important project to ensure better waste management.

1.2 Problem Statements

Dental mould is a component that is use by the dentist for making model structures of human teeth for patient with tooth defect. Advanced Medical and Dental Institutes (AMDI) ,Universiti Sains Malaysia in Kepala Batas were reported to disposed more than 50 kg per month of these dental mould wastes. Thus, the total number of wastes contributed by all the hospital and dental institutes are expected to be much higher.

The mould waste is a possible calcium sources for forming apatite materials as it Calcium Sulfate, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. The dental mould that was collected from AMDI is green dental moulds consist of small amount of Chromium that have to be removed to be accepted as bioceramic for orthopedic purpose. Hence, there is a need to investigate the recycling or reuse the waste dental mould to avoid solid waste disposal by land refill, and therefore reduce environmental issue.

1.3 Research Objectives

The objectives of this research are listed as below:

- (i) To study the possibility of synthesizing apatite ceramics for the waste dental mould
- (ii) Investigate the possibility of dental mould waste to be recycled as raw material for the dental mould by heat treatment procedure.

1.4 Scope of works

In this study, the research had focused on synthesis of apatite ceramics from ground waste dental stone. The dental stone wastes were determined as gypsum, $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$ and thus suggesting as potential calcium source. The stages involved removal of chromium from the ground dental mould. This involved reaction with sodium carbonate, Na_2CO_3 and preheated at 865°C , before leaching with sodium hydroxide, NaOH . The leached powder was then pressed to form pellets and sintered at 1100°C . The sintered

pellets were then characterized. The processes of converting the gypsum to apatite bioceramics are conducted into 4 main sections:

- i. Characterization of raw ground dental stone.
- ii. Removing the chromium and characterization of apatite ceramic powders..
- iii. Synthesis and characterization of apatite ceramic powders
- iv. Sintering and characterization of apatite ceramic pellets.

Another project was to focus on converting the waste mould to gypsum by heating dental mould in 130 °C or 160 °C and therefore reuse as gypsum raw materials.

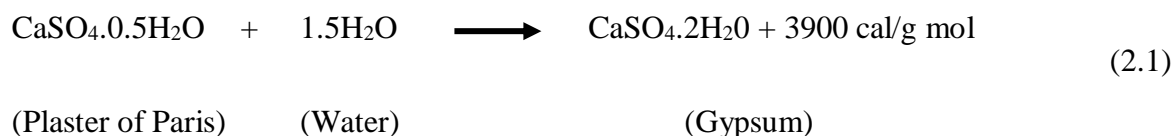
CHAPTER 2

LITERATURE REVIEW

2.1 Gypsum

Gypsum is a naturally occurring mineral that is made up of calcium sulfate and water ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) that is sometimes called hydrous calcium sulfate. Gypsum is a rock like mineral normally discovered inside the earth's crust, extracted, processed and utilized by man in construction or ornament within the form of plaster and alabaster on account that 9000 B.C. Plaster was discovered in Catal-Huyuk in Asia in an underground fresco and throughout the time of the Pharaohs, Gypsum become used as mortar within the production of the Cheops Pyramid (3000 B.C.). (Eurogypsum.org, 2018). In dentistry, gypsum is used in the form of Calcium sulphate hemihydrate. ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$).

Most gypsum products are obtained from natural gypsum rock. Because gypsum is the dihydrate form of calcium sulfate ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$), on heating, it loses 1.5 g mol of its 2 g mol of H_2O and is converted to calcium sulfate hemihydrate ($\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$). When calcium sulfate hemihydrate is mixed with water, the reverse reaction takes place, and the calcium sulfate hemihydrate is converted back to calcium sulfate dihydrate (equation 2.1).



2.1.1 Application of Gypsum

Gypsum is non-toxic mineral, so it can be helpful to humans, animals, plant life, and the environment. Typically gypsum are used in production of gypsum blocks, plasterboards, plaster and gypsum fibreboards. Gypsum is also used as a generic name for many types of sheet products made of a non-combustible core with a paper surfacing that adds strength. These include drywall, ceiling tiles, partitions, whose strength is directly related to its thickness and a few trace materials. Gypsum used in Portland cement and special cement products for set and expansion control. Gypsums are added into glass to fabricate large, lightweight architectural decorations (Eurogypsum.org, 2018).

Gypsum is use widely in medical applications and plays important roles in surgical and dental work. Gypsum has been used in dentistry application such as impression plaster and mounting the casts to the articulation, beside that it also can form casts and dies. Gypsum can be used as a binder for silica and as a mold for processing dental polymers (Uobabylon.edu.iq, 2018).

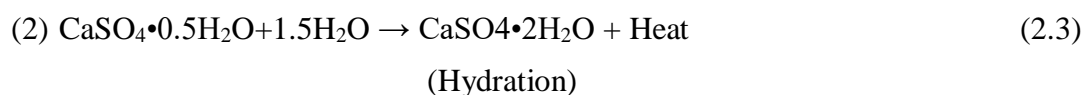
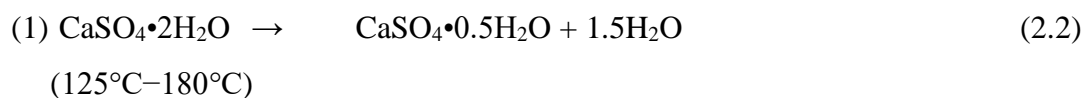
Gypsum can control the pH value of soil and neutralise the acidity of soil. These properties make gypsum very useful in agriculture. One of gypsum's main advantages is its ability to reduce aluminum toxicity, which often accompanies soil acidity, particularly in subsoil. Plus, it also improves soil structure and water infiltration as Gypsum improves the ability of soil to drain and not become waterlogged (CropLife, 2018)

Gypsum is recognized as acceptable for human consumption by the U.S. Food and Drug Administration for use as a dietary source of calcium, to circumstance water utilized in brewing beer, to govern the tartness and readability of wine,, and as an

ingredient in canned vegetables, flour, white bread, ice cream, blue cheese, and other foods(Gypsum Association, 2018).

2.2 Gypsum Recycling

Gypsum recycling is the process of turning gypsum waste into recycled gypsum hereby generating a raw material that can replace virgin gypsum raw materials in the manufacturing of new products. Gypsum should be recycle as when mixed with bio-degradable waste can produce hydrogen sulphide gas in landfill which is both toxic and odorous. Gypsum plaster is considered a sustainable binder, produced by the calcination of gypsum (calcium sulfate dihydrate: $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$) in a dry process at temperatures that range from 125 °C to 180 °C By controlling thermal treatment, calcium sulfate dihydrate loses 75% of its combined water, and it is produced the calcium sulfate hemihydrate ($\text{CaSO}_4 \cdot 0.5\text{H}_2\text{O}$ (Geraldo et al., 2017). The reaction is reversible and once in contact with water the hemihydrate hydrates (equation 2.2) and is transformed to the dihydrated form (equation 2.3)



The hemihydrate can be found in two different crystalline structures: α , when it is prepared from gypsum by the wet process, in a water vapor saturated ambient; and β , when it is prepared by the dry process, in which, water vapor is eliminated as it is

formed. The β -hemihydrate is the so called “Plaster of Paris” and is employed as a construction material (Geraldo et al., 2017).

2.3 **Dental Gypsum**

Dental impression making is the process of creating a negative form of the teeth and oral tissues, into which gypsum or other die materials can be processed to create working analogues (Punj, Bompolaki and Garaicoa, 2018). Impression materials can be classified according to their composition, setting reaction, and setting properties, but a commonly used system is based on the properties after the material has set (Punj, Bompolaki and Garaicoa, 2018).

The properties of ideal model material for the (gypsum products) are the dimensional stability, which means no expansion or contraction during or after setting and high compressive strength to withstand the force applied on it. The model must be able to produce a smooth surface and have a reasonable setting time.

2.4 **Calcium Phosphates Family**

Calcium phosphates are the largest group of artificial bone graft substitutes. This is mainly due to their close resemblance to the mineral components of bone. Calcium phosphate biomaterials are well known in the medical materials and devices world, in particular, for their use in the treatment of osseous defects and as coatings on metal alloy prostheses and fixing devices (Zhang and Cresswell, 2016).

The geometrical properties of calcium phosphate ceramics are mainly the porosity and the interconnectivity of the pores (Blokhuis, 2014). The chemical composition of the calcium phosphates has changed significantly over the past decades. Initially the calcium phosphates consisted of hydroxyapatite (HA) ($\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$), an inert material which is not resorbed over time. With the introduction of β -tricalcium phosphate (β -TCP) ($3\text{-Ca}_3(\text{PO}_4)_2$) as well as other compositions such as α -TCP, β -octocalcium phosphate, dicalcium phosphate, and others, the field of calcium phosphates has changed significantly. Generally speaking HA is not resorbable, whereas the other compositions are resorbed quickly. By combining HA and TCP, resulting in biphasic calcium phosphates (BCP) the rate of resorption of a material can be influenced, and the other calcium phosphate compositions all aim to establish different resorption rates (Blokhuis, 2014).

Table 2.1: Different types of calcium phosphate apatite ceramics

Type of CaP	Chemical formula	Ca/P ratio	Crystal structure	Density
Amorphous apatite	$\text{Ca}_3(\text{PO}_4)_2 \cdot n\text{H}_2\text{O}$	1.50	Amorphous	3.14
Biphasic calcium phosphate	$(1-x) \text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2 + 3x \text{Ca}_3(\text{PO}_4)_2$	Dependent on ratio of HA and β -TCP	Hexagonal and rhombohedral	3.07 – 3.21
Calcium deficient hydroxyapatite	$\text{Ca}_{10-x}(\text{PO}_4)_6-x(\text{HPO}_4)_x(\text{OH})_{2-x}$ [x is between 0 to 1]	1.50 - 1.67	Hexagonal	3.16
Carbonated hydroxyapatite	$\text{Ca}_{10}(\text{PO}_4, \text{CO}_3)_6(\text{OH})_2$	Non-stoichiometric	Hexagonal	2.80
Chlorapatite	$\text{Ca}_{10}(\text{PO}_4)_6\text{Cl}_2$	1.67	Hexagonal	3.17 – 3.18
Dicalcium phosphate dihydrate	$\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$	1.00	Monoclinic	2.31
Fluorapatite	$\text{Ca}_{10}(\text{PO}_4)_6\text{F}_2$	1.67	Hexagonal	3.10 – 3.20
Hydroxyapatite	$\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$	1.67	Hexagonal	3.14 – 3.21
Monocalcium phosphate monohydrate	$\text{Ca}(\text{H}_2\text{PO}_4)_2 \cdot \text{H}_2\text{O}$	0.50	Triclinic	2.58
Octacalcium phosphate	$\text{Ca}_8\text{H}_2(\text{PO}_4)_6 \cdot 5\text{H}_2\text{O}$	1.33	Monoclinic	2.61
α - Tricalcium phosphate	$\text{Ca}_3(\text{PO}_4)_2$	1.50	Monoclinic	2.87
α' - Tricalcium phosphate	$\text{Ca}_3(\text{PO}_4)_2$	1.50	Hexagonal	2.70
β - Tricalcium phosphate	$\text{Ca}_3(\text{PO}_4)_2$	1.50	Rhombohedral	3.07
Tetracalcium phosphate	$\text{Ca}_4\text{P}_2\text{O}_9$	2.00	Monoclinic	3.06

2.4.1 Hydroxyapatite

Zhang and Cresswell, 2016 stated that Calcium hydroxyapatite (HA), $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$, is an important inorganic material in biology and chemistry. Biological apatites, which are the inorganic constituents of bone, tooth enamel, and dentin, are typically very variable in their composition and morphology, and typically contain different impurities. In general, these impure biological apatites are designated as calcium deficient or non-stoichiometric apatites. Synthetic HAs are frequently used as reference materials in biomineralization and biomaterial studies. The composition, physicochemical properties, crystal size, and morphology of synthetic apatites are extremely sensitive to preparation conditions. For control and reference purposes, it is important to have available pure and stoichiometric HA, or nearly stoichiometric HA, characterized in detail with respect to its chemical composition and numerous other important properties.

Al-Sanabani, 2013 mentioned that Hydroxyapatite has unit cell lattice parameters $a = 0.94\text{nm}$ and $c = 0.68\text{ nm}$. It has a hexagonal symmetry; its unit cell can be considered to be arranged along the c-axis that make the lattice structure preferred orientation that gives rise to an oriented growth along the c-axis and causes it to have needle-like morphology. It has low fracture toughness and mechanical strength but it has strong biological fixation to bone tissues.

In term of mechanical properties, the behavior of HA is greatly differ depending on the sintering temperature and soaking time. In fact, HA is also known as a bioceramic material with low mechanical properties, in particular low tensile strength and fracture toughness (González Ocampo et al. 2016). The mechanical properties can be affected by the amount and size of porosity, impurity and test directions according to

the bone axis. Modulus of elasticity of HA in a range between 11.5 to 27 GPa and it shows that HA is a brittle material (Uysal et al. 2014). Biomedical application by using HA is limited to human body parts that subject to either compressive stress or reduce mechanical strain only (González Ocampo et al. 2016).

Various techniques have been used for preparation of HA powders. Variation of the method and conditions of synthesis for HA powders have influenced the chemical, structural and morphological properties of synthetic HA (Agrawal et al. 2011). Two main methods in preparation of HA powders which are wet methods and solid-state reactions.

2.5 Sintering

Sintering is densification of green body which either the powder or solid material involved on the heating process, normally at temperatures $> 0.5 T_m$ of that material (Marinescu et al. 2017). The sintering process is one of the important steps in manufacturing of the ceramic product. Sintering process might be effect the final properties of the product in various aspects such as mechanical and biological properties of that product (Mangkonsu et al. 2016). The sintered product was densified by bulk and grain boundary diffusion mechanisms in order to optimize and enhance the mechanical properties. During firing process, the porosity of the product will be reduced and it called as shrinkage. In another word, diffusion processes will be happened during sintering stage with driving force being a reduction in surface area (Marinescu et al. 2017). Thus, the higher sintering temperature will lead to more shrinkage (Kamardan et al. 2010). As mentioned earlier, this happens because sintering reduces the total surface

area and pore volume. In morphology aspects, the microstructure of the final product can be designed through the control of sintering variables (Mangkonsu et al. 2016).

2.6 Bioactivity

The main requirement for biomaterials is to be bioactive and capability interacts with tissue in human bone. To understand the biological of apatite material, various synthetic methods have been developed. In particular, simulated body fluid (SBF) has been used to mineralize biomaterial surfaces for studies of mineral growth and cell-mineral interactions (Choi et al. 2015). This method is useful to predict the in vivo bone bioactivity of a material as biomaterial (Kokubo & Takadama 2006).

Furthermore, the characteristic in term of capability to interact with tissue in human bone of a material is evaluated by examining the ability of apatite to form on the surface of material when soaked in a simulated body fluid (SBF) solution (Kokubo & Takadama 2006). SBF solution was prepared in according to chemical composition of human body fluid which ion concentrations nearly equal to those of the inorganic constituents of human blood plasma (Kovaleva et al. 2008). Basically, the material will be soaked in SBF solution for 7, 14 and 28 days and it has been described by many researchers. After several periods of soaking, the formation of the apatite layer become growth and thick due to increasing the soaking time in SBF solution (Saber-Samandari et al. 2016).

2.7 Chromium Toxicity

Chromium is a cumulative poison with strong irritation and corrosiveness. It can invade the body through the respiratory tract, the gastrointestinal tract and the skin, which causes contact dermatitis and ulcers in the contact areas, chemical pneumonia, difficulty in breathing, liver damage, acute renal failure and other adverse reactions. The formation of the carbonate apatite layer decreases as the chromium ions concentrations increases in the samples (Sallam et al., 2012)

CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Introduction

In this study, synthesis of apatite ceramic (Hydroxyapatite (HA) or Tricalcium Phosphate) was performed by a wet chemical precipitation method. Initially chromium content present in dental mould waste which had resulted in green powder was removed and subsequently heat treated as well as reacting with NaOH alkali to form pure CaO. The CaO was then reacted with phosphoric acid to form apatite ceramic precipitate. Chromite ore process residue methods were used for this study to eliminate chromium content in bioceramic products. In project 1 stage 1, waste dental stone was crushed and milled to become ground powder. The ground waste was sieved and subsequently alkaline roasting with Sodium Carbonate according to chromium content from XRF result. After alkaline roasting, roasted samples were collected and ground by using agate mortar and pestle set were then caustic leaching with 7 M of Sodium Hydroxide and double leaching to discard the solute Sodium Chromate from the roasted sample. The leached residue was filtered and washed by deionised water. After filtering and washing, the leached residue reacted with Phosphoric acid to form apatite ceramic precipitates in project 2. Once apatite ceramic precipitates were collected, it was dried in oven and ground by using agate mortar and pestle set to form ground sample. The powder was pressed into pellets by hydraulic hand press. Apatite ceramic pellet was then undergone sintered and characterized. Besides that, another project was studied about recycling dental mould waste to form the raw materials for making dental mould.

Ground dental mould waste was heat treated with 130 °C and 160 °C in 2 hour and 4 hour. The heat treated products were casted into mould and undergone characterization. For characterised raw materials and sintered apatite ceramic products as well as the heat treated powder, it using the techniques included Visual Inspection, Fourier Transform (FTIR), X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM) and Energy Dispersive X-Ray (EDX), X-Ray Fluorescence (XRF), Shrinkage Analysis and Bulk Density Analysis, Porosity Analysis, Simulated Body Fluid test (SBF), Diametral Compression Test and Vickers Hardness Test.

3.2 Materials

The raw material that has been used in this study is waste dental mould which was collected from AMDI, Penang. Hardened dental stone waste is green color as shown in Figure 3.1.



Figure 3.1: Dental stone waste collected from AMDI.

For oxidation of chromium process, the raw material was alkaline roasting by used sodium carbonate then 7M concentration of sodium hydroxide solutions are prepared to leach out the chromium for leaching mechanism.

3.3 Work Flow Diagram of Experiment

The flow diagram below (Figure 3.2) briefly shows experimental work involved Project 1 : Synthesis of Apatite and Project 2 : Heat Treatment of Dental Mould.

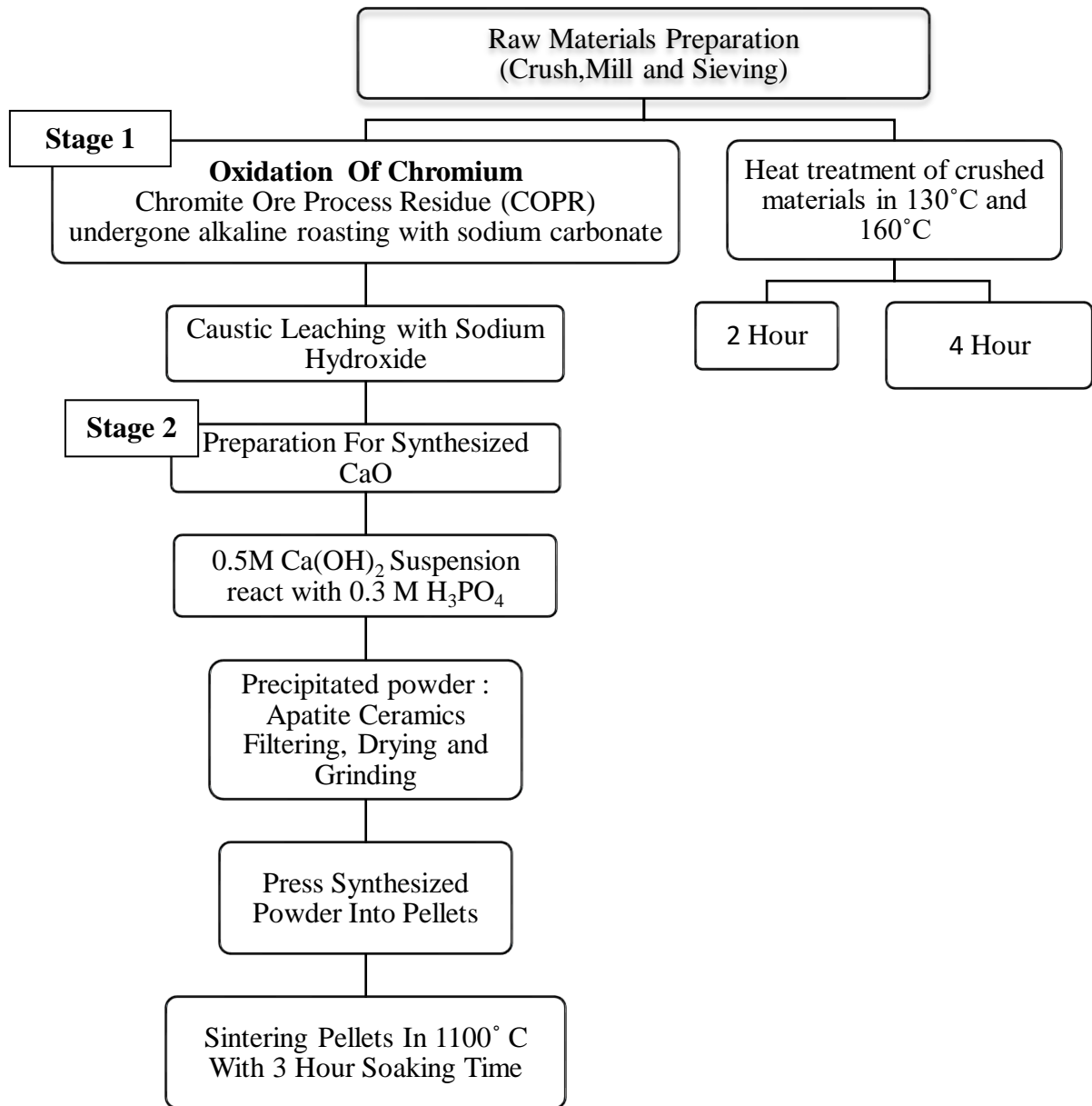


Figure 3.2: Flowchart Diagram of Experimental Work

3.3.1 Raw Material Preparation

Dental mould waste (Figure 3.1) was collected from AMDI. It was firstly crushed and to form powder. For dental mould waste become small particles, it required two level of crushing which is primary crushing and secondary crushing by using jaw crusher and cone crusher. Then, it undergoes planetary ball milling and then sieving using 200 μ m sieve.

3.3.2 Primary Crushing

Swing jaw crusher was chosen to crush dental mould waste into smaller form before further the milling to form fine powder. Pressurized air gun was used to clean the machine before crushing, to minimize the contamination of other material to waste dental stone. The crushed dental mould waste was collected from outlet tray from the machine. The crushed particle was still which bigger in size was further crushed using secondary crushing.



Figure 3.3: Swing Jar Crusher

3.3.3 Secondary Crushing

The crushed particles obtained from primary crushing were collected and then further undergo secondary crushing (cone crushing) and before using cone crusher, pressurized air gun was used to clean the machine to minimize the contamination of other material to dental mould waste. Brush and scoop was used for collected dental mould waste particles from cone crusher.



Figure 3.4: Cone Crusher

3.3.4 Milling

Milling is a technique for prepare the desired size of powder to undergo purification and synthesise process in mineralogy. Planetary ball milling was used to grind the waste dental stone particles.

To reduce moisture content in crushed samples, the samples were placed in dry oven for 30 mins at 60°C before being transferred into zirconia milling jar. The purposes of the crushed particles being dried out were to minimize the cavitation on the wall of the milling jar. 50g of crushed particles with 1:2 ratio to zirconia balls used (10

balls of 20mm in diameter) acted as milling media. The milling cycle was carried out at 320 rpm for 30minutes for forward, action 30 minutes for reverse and 15 minutes for rest time. The ground samples were collected by using a spatula. The ground samples were then proceed to synthesize apatite ceramic (section 3.5) and heat treatment of ground powder to form P.O.P (section 3.7)



Figure 3.5: Milling Jar and Zirconia Balls