DEVELOPMENT OF POROUS SINTERED ALUMINA BY A SPONGE SCAFFOLDING METHOD AND PORE FORMING AGENT USING NATURAL BIORESOURCES

by

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LIST OF ABBREVIATIONS

°C : Degree Centigrade

Å : Angstrom

AC : Alternating current

Al_2O_3: Aluminum Oxide

approx : Approximately

PVA : Polyvinyl alcohol

PFA: Pore-forming agent

SCP: Sugar cane waste

RHP: Rice husk powder

V: Volume

V_s: Seepage- velocity

A: Area

C : Compressive strength

ϕ: Porosity

F: Newton

PPI: Number of pore per inch

D : Dissipation Factor

DTA : Differential Thermal Analysis

ED : Electron Diffraction

ED : Electron Diffraction

EDAX : Energy-Dispersive X-Ray Spectroscopy

EDS : Energy Dispersive Spectroscopy

EDX : Energy Dispersive X-Ray

EMI : Electromagnetic Interference

FEG : Field Emission Gun

FWHM : Full Width at Half Maximum

g : gram

g/mol : gram per mole

Gd : Gadolinium

H_2O : Water

H_c : Coerceivity

HRTEM : High resolution transmission electron microscope

K : Kelvin

kg m^{-3} : kilogram per cubic meter

kJ/g : kilojoule per gram

LC : Inductance-Capacitance

LCR : Inductance Capacitance Resistance

LRTEM : Low Resolution Transmission Electron Microscope

ml : milliliter

ml/min : Milliliter per minute

mm : Millimeter

MPa : Megapascal

nm : nanometer

PPMS : Physical Properties Measurement System

Rp : Parallel Resistance

rpm : revolution per minute

SEM : Scanning Electron Microscopy

TEM : Tranmission Electron Microscopy

TG : Thermal Gravimetry

TG-DTA : Thermogravimetry- Differential Thermal Analysis

XRD : X-ray Diffraction

μm : Micrometer

Ωm : Ohm meter

PENGHASILAN ALUMINA BERLIANG TERSINTER DENGAN KAEDAH KERANGKA SPAN DAN AGEN PEMBENTUK LIANG MENGGUNAKAN SUMBER BIO SEMULAJADI

Kajian ini dijalankan untuk menghasilkan alumina berliang tersinter dengan kaedah kerangka span dan agen pembentuk liang menggunakan bahan sumber bio semulajadi. Alumina berliang telah dihasilkan daripada span polimer poliuretana yang direndam dalam buburan alumina. Yang dihasilkan daripada campuran serbuk alumina dan air suling dengan kepekatan yang berbeza, iaitu 54, 57 dan 63 peratus berat alumina (*wt.%*). Span alumina yang telah direndam dengan buburan dikeringkan pada suhu 80^{o}C di dalam ketuhar. Ia kemudiannya disinter pada suhu 1250^{o}C selama tiga jam. Pencirian terhadap sampel yang telah disinter dilakukan dengan menggunakan mikroskop imbasan elektron (FE-SEM) dan mikroskop optik untuk memerhatikan keadaan morfologi. Ujian mampatan juga dijalankan untuk menentukan kekuatan mekanik sampel tersebut. Kepekatan alumina yang berlainan memberikan nilai ketumpatan daripada 0.63 hingga 0.31 gcm^{-3} dengan keliangan relatif sebanyak 78.54 hingga 92.60 peratus, kadar aliran air 3.00 hingga 3.15 cm^{3}/s, kelajuan resap 0.120 hingga 0.138 cms^{-1} dan kekuatan mampatan 0.45 hingga 0.60 MPa. Mikrostruktur menunjukkan liang-liang yang kecil dan besar telah terbentuk dan saling berhubung. Agen pembentuk liang (PFA) yang digunakan untuk fabrikasi seramik berliang ini ialah hampas tebu (SC) dan serbuk sekam padi (RH). Selepas proses pembentukan dan pengeringan, jasad seramik tersebut juga disinter pada 1250^{o}C selama 30 minit. Keputusan yang diperiolehi menunjukkan bahawa penambahan SC memberikan nilai ketumpatan pukal dalam lingkungan 1.20 hingga

1.27 gcm^{-3}, keliangan pada julat 60.86 hingga 65.64 %, kadar aliran 0.10 hingga 0.13 cm^3/s, kelajuan resap jasad daripada 0.014 hingga 0.03 cm/s dan kekuatan mampatan pada julat 1.06 hingga 1.57 MPa. Penggunaan RH pula menyebabkan keliangan dan kadar aliran berkurangan dengan peningkatan RH. Nilai ketumpatan sampel meningkat daripada 1.27 kepada 1.57 gcm^{-3}. Hal ini menunjukkan bahawa hampas tebu (SC) dan sekam padi (RH) berpotensi untuk penghasilan bahan seramik alumina berliang. Walau bagaimanapun, berdasarkan keputusan yang diperolehi, SC didapati merupakan agen pembentuk liang (PFA) yang lebih baik berbanding RH.

DEVELOPMENT OF POROUS SINTERED ALUMINA BY A SPONGE SCAFFOLDING METHOD AND PORE FORMING AGENT USING NATURAL BIORESOURCES

ABSTRACT

Porous sintered alumina was developed using a sponge scaffolding method and pore-forming agents (PFA) using natural bioresources. The polymeric sponge method used a polyurethane foam soaked in an alumina slurry prepared by mixing alumina powder and distilled water with different concentrations of alumina, i.e. 54, 57 and 63 weight percent (wt.%) alumina. The soaked sponge was then dried in an oven at 80^{o}C. It was then sintered at 1250 $^{\circ}$C for 3 hours. A Field Emission Scanning Electron Microscope (FE-SEM) and an optical microscope were used to observe the morphology. A compression test was also carried out to determine the mechanical property. The different alumina concentrations produced densities ranging from 0.63 to 0.31 gcm^{-3} with relative porosities of 78.54 to 92.60 %, water flow rate 3.00 to 3.15 cm^{3}/s, seepage-velocity of 0.12 to 0.14 cm/s and the compressive strength 0.45 to 0.60 MPa. The microstructure showed that small and large pores were formed and these were largely interconnected. Pore-forming agents (PFA) used for the fabrication of porous ceramics were sugar cane wastes (SC) and rice husk powder (RH). After shaping and drying, the ceramic green bodies were also sintered at 1250 $^{\circ}$C for 3 hours. The results obtained showed that when SC was added the bulk density ranged from 1.20 to 1.27gcm^{-3}, porosity ranged from 60.86 to 65.64%, flow rate from 0.10 to 0.13 cm^{3}/s and seepage velocity from 0.014 to 0.029 cm/s whilst the compressive strengths ranged from 1.06 to 2.05 MPa. When RH was added, the porosity and flow rate results were reduced with increasing RH. The density of the samples increased from 1.27 to1.57 gcm^{-3}. It shows that both sugar cane wastes (SC) and rice husk (RH) are potentially capable to produce porous alumina. However, based on the results, SC was found to be a better pore-forming agent than RH.

CHAPTER 1

INTRODUCTION

1. Introduction

Over the last few years, there has been increasing interest in porous ceramic materials which are particularly important for industrials applications: such as in chemistry, mechanical engineering, biotechnology, aerospace and electronics. For most industrial applications of porous ceramics, an open pore is required for gas and liquid filtration, membrane support, purification, thermal insulation, lightweight porous structure, catalyst and biomaterials (Gregorova and Pabst, 2007a).

Porous ceramics materials for filters need to have a high fraction of open porosity. In open pores, penetrating pores are necessary for industrial applications such as in filters or for gas distribution. Closed porous ceramic materials are used mainly for sonic and thermal insulators or low specific-gravity structural components (Ishizaki et al., 1998).

Porous ceramic materials are important in many applications because of their high mechanical strength and uniform structural stability compared to polymers and metals. Porous alumina having a microstructure of continuous network of nanopores in its microstructure has been well studied for such application where there is need for high strength and high abrasion resistance.

Porous polymeric materials are used in soft tissue applications which include polyurethane, polyamide, polyester, etc. Porous reconstituted collagen has been used

as artificial skin whilst braided polypropylene has been used as artificial ligaments. As in the case of bone implants, the porosity encouraged tissue ingrowth (Edward 2003).

Introducing open pores in a material (producing open porous ceramics materials) changes the mechanical properties of materials. Two essential changes are a decrease in density and an increase in specific surface area. The changes generate useful properties (which are not observed in dense bodies) such as fluid permeability, filtration effects and thermal insulation capability. Narrow pore size distribution is important for porous filters, which allows for selective filtration. Materials with a bimodal pore size distribution are required for bioreactors, in which enzymes or bacteria are immobilized in the small pores and larges open pore are used as channels for transporting reactants products (Abe et al., 1992). A narrow pore size distribution for each pore mode is required for these applications.

Large specific surface area is necessary for catalysis. In many applications of porous materials, highly open porosity is desirable to increase the specific surface area or fluid permeability. However, an increase in porosity decreases the mechanical strength. Low mechanical strength limits the operating conditions for porous materials, and consequently, increases the required dimensions of porous materials. Both high open porosity and high mechanical strength may be required simultaneously in order to use porous materials under severe operating conditions. Different applications of porous materials require different pore size. For instance, pores of atomic scale are required for gas separation or catalysis (Ishizaki et al., 1998).

Therefore, the preparation of porous ceramics with controlled microstructure (porosity, pore size and pore shape) has been a subject of constant interest during the last decades and common processing techniques include dipping (sponge method) and slip casting techniques. The foams produced by dipping (sponge method) produce large pores, high porosity and uniform pore structure (Han et. al., 2003). The slip casting method produces pores in the nanometer size range and extremely high porosity. There are also other methods such as biomimetic processing (using pyrolized wood templates), ceramic hollow spheres process (e.g. alumina microball) and sacrificial (pyrolyzable) pore forming agents (PFA), i.e. synthetic organics or natural biopolymers which burn out during firing (Rice, 1998; Woyansky et al., 1992).

Although a great number of PFA has been proposed and used in ceramic technology, e.g. carbon (Lange et al., 1990; Balaszi et al., 2004), polyvinyl chloride, polystyrene, polyethylene (Corbin and Apte, 1999), wood flour (saw dust) and crushed nut shells (Rice, 1998; Woyansky et al., 1992), it seems that starch, a natural biopolymer consisting of amylase and amylopectin, has attained a prominent position as a PFA, including its recent application as a combined pore-forming and body-forming agent in starch consolidation casting. Moreover, as mentioned before, apart from its universal function as a PFA, starch can serve additionally as a body-forming agent in starch consolidation casting, due to its ability to swell in water at elevated temperatures, thus enabling ceramic green bodies to be fabricated by slip casting into nonporous molds of suspensions with starch. One example is porous alumina ceramics which had been reported to be produced using commercially available poppy seeds (Gregorova and Pabst, 2007a).

1.2 Problem statements

Porous alumina ceramic are presently the focus of very active research and development activities. There are currently around 150 institutions working on ceramic foams worldwide, most of them focusing on their manufacture and characterization. Various companies are developing and producing these materials which are now being used in numerous industrial applications such as lightweight structures, biomedical implants, filters, electrodes, catalysts, and heat exchangers. (Lefebvre et al., 2008).

Alumina is a well known as typical specific material for use at high mechanical properties, high performance and high temperatures. Thus alumina has a wide range of applications such as in automobile, engine, aerospace, equipments, etc (Gregorova and Pabst, 2007b).

With growing demand for porous ceramics in industrial applications, a number of techniques have been developed for the fabrication of porous materials with high strength, catalytic activity, good erosion and corrosion resistance. These excellent properties of porous ceramic make it possible to be used in several operating conditions, compared to porous polymers, glasses and metal. In this study, porous alumina ceramics were fabricated using different processing for parameters comparison:

(1) In term of methods, dipping and casting were used: dipping using polymeric sponge and casting of slip casting into plaster molds.

(2) In term of materials, pore forming agents used were polyvinyl alcohol (PVA), rice husk (RH) and sugar cane waste (SC), apart from the alumina powder base.

All these parameters were optimized and characterized for related properties of the porous alumina ceramic material.

1.3 Objectives

1. To produce porous alumina using two different methods: dipping (a sponge scaffolding method) and casting method (using natural bio-resources: rice husks (RH) and sugar cane (SC), and PVA as the pore forming agents).
2. To evaluate and compare properties of the products prepared by dipping methods with three different types of alumina slurries (Al_2O_3; Al_2O_3+PVA; Al_2O_3+PVA+SC).
3. To evaluate and compare properties of the products prepared by casting methods with three different types of alumina slurries (Al_2O_3+PVA; Al_2O_3+PVA+SC; Al_2O_3+PVA+RH).

1.4 Research Scope

In general, the research work was divided into two parts which were described in detail as follows. The first part involved the use of a polymeric sponge method with three different types of slurries to produce porous ceramic alumina foams after sintering. The second part involved the use of a casting method (also three types of slurries) making full use of natural bioresources such as (rice husk and sugar cane

waste fibers). The porous products from both methods were then characterized for their physical and mechanical properties such as density, porosity, flow rate, velocity, compressive strength, and while their morphology using an optical microscope and a field emission scanning electron microscope (FESEM).

CHAPTER 2

LITERATURE REVIEW

2.1 Porous ceramics

Porous ceramic materials are defined as solids containing different types and percentages of pores. Generally porous materials have porosity of 20-95%, the percentage of pore volume to the total volume. Porous ceramics have been used in various applications from daily necessities, such as water filtration through porous ceramics, to uses in modern industries. Porous ceramics have been employed for dust and gas removal in high purity processes such as in semiconductor productions (Krasovitskii, 2007).

Pores are classified into two types: open pores which are connected to the outsides of the materials, and closed pores which are isolated from the outside. Penetrating pores are a kind of open pores which have at least two openings located on both sides of a porous ceramic material (Ishizaki et al., 1998).

In recent years, porous ceramics have become more important and are being developed for a variety of interesting applications such as thermal insulation, filter, lightweight materials, medical technology as well as electronics. For most industrial applications, materials with open pores are required. Porous ceramic materials such as filters and carriers for catalysts and bioreactors need to have a high fraction of open porosity. Penetrating pores are necessary for industrial applications such as filters or gas separation. Closed porous materials are used mainly for sonic and

thermal insulators, or low specific-gravity structural components (Ishizaki et al., 1998).

Introducing open pores in materials changes the material properties. Two essential changes are the decrease in density and the increase in surface area. The changes generate useful properties (which are not observed in dense bodies) such as fluid permeability, filtration effects and thermal insulation capability. Narrow pore size distribution is important for porous filters and allows selective filtration (Abe et al., 1992). Large specific surface area is necessary for catalysis. In many applications of porous materials, large open porosity is desirable to increase the specific surface area or fluid permeability. However, such an increase in porosity decreases the mechanical strength.

Porous ceramics have also been developed with high temperature stability, strength, catalytic activity, as well as erosion and corrosion resistance. These excellent properties of porous ceramics make them suitable to be used in several practical conditions, compared with porous polymers, glasses and metals. In spite of these excellent properties, the potential of porous ceramics has not been fully realized because of their well-known problems (Schaefer, 1994). These shortcomings include:

1. brittleness,
2. absence of an integrated material manufacturing system,
3. lack of pore size control,
4. lack of continuous processing methods,

5. use of processing/ sintering aids that limit toughness,
6. absence of technologies, and
7. absence of a model relating pore structure to mechanical properties.

Problems 1, 2, 4, 5 and 6 are also applicable to dense ceramics. To overcome these difficulties, researchers of porous ceramics have to approach the scientific and technological problems of materials, from powder production of raw materials to quality control of the final products (Schaefer, 1994).

2.2 Applications of porous ceramics

2.2.1 Filters

There are many techniques for fluid separation. Filtration by passing a liquid or gas through a porous material to make it pure is a common separation method. Filtration is used for many applications requiring a particular particle size or pore size. Porous filters have been applied in many ways from dairy applications to high-tech processing. Many materials have been used as porous filters (Ishizaki et al., 1998).

2.2.2 Dental implants

Recently, a new type of porous alumina dental implant has been designed. A biological seal is provided by a single-crystal alumina on its cervical portion and fixation capability in bone is provided by a porous alumina layer with an average interconnecting pore size of approximately 130 μm. These alumina porous implants

were expected to become closely attached to the gingival tissue and prevent contamination because they have a smooth and biocompatible surface in the cervical portion. With suitable pore sizes for bone in-growth this implant was expected to become fixed to the alveolar bone rapidly (Spector et al., 1988). As a porous material is implanted in the bone, the pore become filled first with blood which clots, then with osteoprogenitor mensenchymal cell, then, after 4 weeks, bony trabeculae. The in-growth created then becomes remodelled in response to mechanical stress (Edward et al., 2003)

2.2.3 Molten metal filtration

For the production of metal casting, foam ceramic filters help to improve quality and productivity by removing non-metallic inclusion; the filters must be able to resist attack at high temperatures by a variety of molten metal which can contain such reactive elements as aluminium, titanium, hafnium and carbon. Thermal shock behaviour is obviously also important, which was found to be strongly dependent on unit cell size (increasing with increasing cell size) and weakly dependent on density (increasing with increasing density) (Montanaro and Jorand, 1998). The material selected depends on the material to be filtered and usually metallic oxides of various compositions as shown in Table 2.1.

Table 2.1 Some characteristics of molten metal filters (Sheppard, 1993; Montanaro and Jorand, 1998).

Trade name	Composition	Application	Benefits
Coring	77% Al_2O_3, 23% SiO_2	Carbon low alloy, stainless steel	Pouring temperature up to 1750^{o}C.
Cerapor	Alumina, SiC, Cordierite, ZrO_2	Aluminium, iron, copper, bronze, steel zinc	Laminated duplex and triplex construction.
Udicell	Alumina, mullite, ZTA, PSZ	Superalloys, low- car bon Stainless steel	Large volume up to 120 tons
Alucel	92% alumina with mullite phase	Nonferrous alloys	Improved thermal shock resistance, smaller filters required.
Selee	Alumina, PSZ	Aluminum, iron, steel	High flow rates

2.2.4 Hot gas cleanup

Hot gas cleanup is an application of high- performance, high-temperature particulate control ceramic filters which are expected to be beneficial not only to the advanced fossil– fuel processing technologies, waste incineration processes but also to diesel soot filtration. Development and utilization of hot gas filters depend on the creative design and use of new high-temperature materials (Montanaro and Jorand, 1998). Such filters must withstand variation in the effluent gas stream chemistry, variation in the nature and loadings of the entrained fines and oscillations in the effluent gas stream temperature and possible pressure, while still maintaining high particulate removal efficiencies with high flow capacity, relatively low pressure drop flow characteristic. During operation the filter must also withstand a variety of mechanical, vibrational, and thermal stresses. The

principal materials for these applications include alumina, mullite, cordierite, silicon nitride and silicon carbide. Both alumina/mullite and cordierite have been demonstrated to have certain advantages over non-oxide materials (Tutko et al., 1984; Gabathuler et al., 1990).

2.2.5 Thermal insulators

A principal application of these porous ceramics is in the fabrication of thermal insulators due to their specific characteristics, such as thermal stability, low thermal conductivity, low density, resistance to thermal cycling, thermal shock resistance and low gas adsorption (Montanaro and Jorand, 1998). The net conductivity is affected by pore shape and interconnectivity.

2.2.6 Catalysts

Porous alumina ceramics are used as catalyst or as their carriers. High specific surface area is required for catalyst applications in order to increase the surface contact with reactants. Chemical stability is also necessary under corrosive conditions. At high temperatures, high specific surface area required for catalysts would decrease due to surface diffusion or evaporation – condensation. High thermal stability is an important property for high temperature applications, for example, catalyst for automobile exhaust gases. In this case of catalytic beds, it had been reported that the accommodation capacity for catalytic materials is an important property (Ishizaki et al., 1998).

2.3 Aluminium oxide (Al_2O_3)

Alumina or aluminium oxide is the most widely used oxide ceramic material. The raw materials of this high performance technical grade ceramic are readily available and reasonably priced, resulting in good value for the cost in fabricated alumina shapes. With an excellent combination of properties and an attractive price, it is not surprising that fine grain technical grade alumina has a very wide range of applications. Figure 2.1 shows the crystal structure image of alumina.

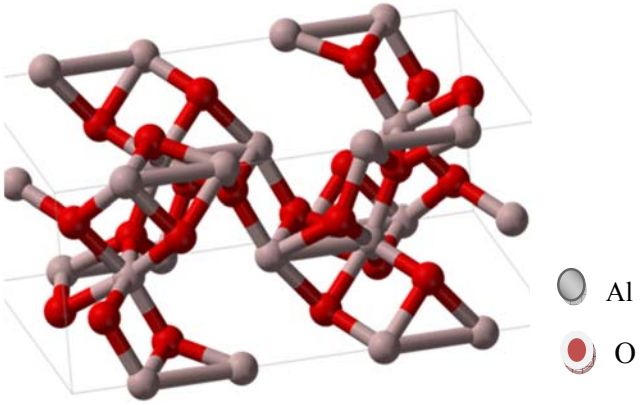

Figure 2.1 Alumina crystal structure [http://www.elantechnology.com/alumina]

Aluminium forms a range of hydroxides. Some of these are well characterized crystalline compounds, whilst others are ill-defined amorphous compounds. The most common trihydroxides are gibbsite, bayerite and nordstrandite, whilst the more common hydroxide forms are boehmite and diaspore (Dibyendu, 2009). Commercially the most important form is gibbsite, although bayerite and boehmite are also manufactured on an industrial scale. Aluminium hydroxide has a wide range of uses, such as flame retardants in plastics and rubber, paper fillers and extenders, toothpaste filler, titania coating and as a feedstock for the manufacture of aluminium chemicals. Figure 2.2 shows the production of alumina from bayerite, gibbsite and diaspore. Table 2.2 shows the physical properties of alpha alumina. Alumina exists in

many forms such as thermodynamically stable form at all the temperatures is alpha alumina.

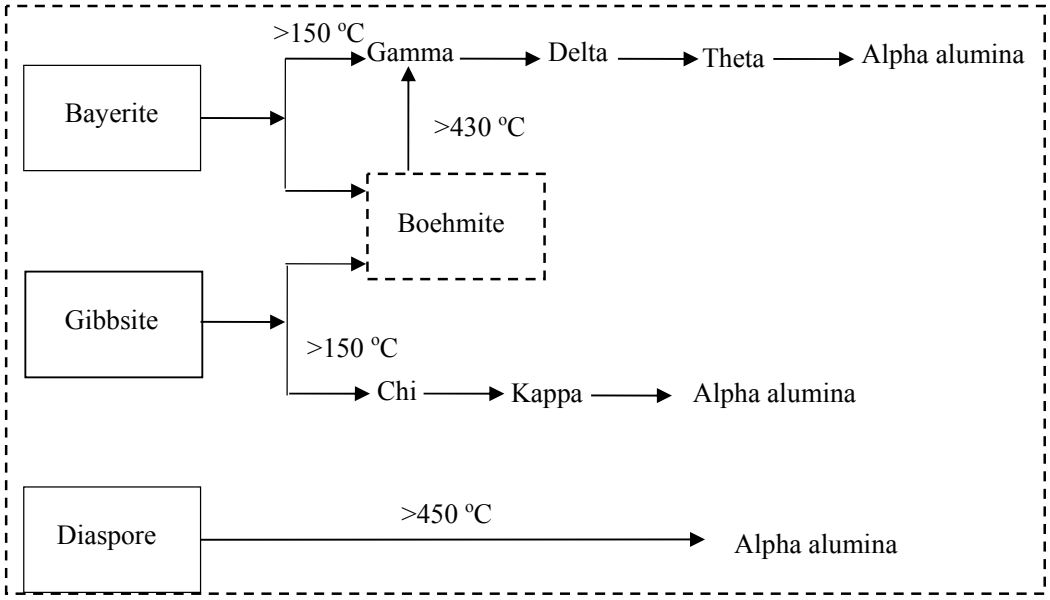

Figure 2.2 Production of alumina (Dibyendu, 2009)

Table 2.2 Physical properties of alpha alumina (George and Laurence, 2003).

Properties	**Value**
Chemical formula	α -Al_2O_3
Density	3.96g/cm^3
Melting point	2050˚C
Maximum operation temperature	< 2000˚C
Specific heat	0.300 cal/g K (1000˚C)
Thermal flow rate ability	0.1-0.4 W/cmK (25˚C)
Compression strength	2 GPa (25˚C)

2.4 Porous alumina

Porous alumina ceramics are widely used in many applications such as filter media, thermal insulator, lightweight porous structure and catalyst in biomaterials (Corbin et al., 1999; Toshihiro et al., 2006). Porous alumina ceramics can be fabricated via many techniques (Sheppard, 1993; Saggio et al., 1992; Mamata and Parag, 2008). These processes produced small pores in the micrometer range which can provide large surface areas. The preparation of porous ceramics with controlled microstructures as characterized by porosity, pore size and pore shape has been a subject of constant interest since the last decades (Liu and Miao, 2005; Gregorva and Pabst, 2007a). Amongst the processing techniques include slip-casting technique (nanometer size range and extremely high porosity), the use of polymeric foam templates (for large pores and extremely high porosity) and sacrificial pore forming agents (PFA) (Gregorva and Pabst, 2007b). The preparation and properties of porous alumina ceramics with high porosity and large pore size which have a high penetration rate greatly depend on the pore morphology, size and distribution of pores.

2.4.1 Sintering of porous alumina

Sintering for porous ceramic materials is generally carried out in the temperature range from 1000 to 1700^{o}C depending on the ceramic materials (Senguttuvan et al., 2001).

Some porous alumina foams have been sintered at a temperature range from 1400 to 1600^{o}C, and it was found that the strength of alumina foam increases with increasing sintering temperatures (Peng et al., 2000; Maiti et al., 1984; Han et al.,

2002 and 2003). The effect of sintering temperature on the foam strength had not been investigated in detail. In the production of porous materials by sintering, the powder characteristics, compaction techniques, sintering process and quality control evaluations are important. Most research on sintering focused on densification (Ishizaki et al., 1998). Simulations of sintering have also been developed to assess the mechanistic theories of sintering based on ceramics properties (Ken et al., 2001; Shinagawa et.,al 1996 and 1999). Some effects due to sintering temperature have been reported in the fabrication of porous alumina (Olevsky, 2000).

2.4.2 Density and porosity of porous alumina

Density and porosity are important properties of porous ceramics. An increase in porosity leads to enhanced specific properties of porous ceramics such as gas permeability and absorption. The porosity of the porous ceramics ranges widely from 20 to 95%, depending on the production process (Zhang et al., 2007). Density and porosity are often measured by fluid displacement methods based on the Archimedean principle. Liquid such as water, toluene and alcohol are usually used with this method (Ishizaki et al., 1998).

2.4.3 Pore size of porous alumina

The pore size of porous ceramics ranges widely from atomic size to millimetre order. Different pore sizes are required for different application of porous ceramics materials but most porous ceramics materials do not have uniform pores. Hence, pore size distribution is also an important property. Narrow pore size

distribution, i.e. uniform pore size, is required in many applications, for instance filters (Toshihiro et al., 2006).

Methods of evaluating exact pore size are important for the manufacture and application of porous materials. There are many methods of evaluating pore size of porous ceramic materials. Mercury porosimetry is commonly used to measure pore size and pore size distribution. For evaluation of sintered porous ceramic materials and green bodies, mercury porosimetry is usually used because it can be applied to evaluate pore sizes from 1mm to nanometers in diameter, as well as pore size distribution (Mikijeli et al., 1991).

2.5 Fabrication of porous alumina

2.5.1 Polymeric sponge method

Polymeric sponges (foams) are porous materials with remarkably high open porosity (~90%). It could be used in the fabrication of ceramic foams (Woyansky et al., 1992; Senguttuvan et al., 2001). Using this fabrication method, the ceramic foam structure was obtained by coating ceramic slurry on polymeric foam. Ceramic foam filters are typically produced by impregnating an open cell, hydrophobic flexible foams material with an aqueous slurry containing a ceramic material, followed by squeezing the organic foam to remove excess slurry (Toshihiro et al., 2006). Ceramic foams with large pores of 50 μm-1 mm in diameter can be obtained by this method.

2.5.1.1 Polymeric sponge open cell foams

The use of open-cell alumina foams have been widely increasing given its diverse properties in various areas. Open-cell ceramic foams are useful for construction of light weight structures, as well as their use mainly in applications of fluid transport where the microstructure is needed. The insulation ability of this foam is related to the insulation value of the static air inside. One of the advantages that these lower density materials provide is a more economical yield (Angel, 2005).

Open cell foams of polymeric sponge are effective as a sound barrier, having about twice the sound resistance in normal frequency ranges as closed-cell foam. However, they are not recommended for hybrid applications. Characteristics of open-cell polyurethane foam include a softer appearance, as well as lower strength and rigidity than those of closed-cell foams (Lefebvre et al., 2008) are shown in Table 2.3.

Table 2.3 Some characteristics between of open-cell and closed-cell polyurethane foams (Angel, 2005)

Closed-Cell	Open-Cell
Highest insulating "R-Value" per inch (> 6.0)	Good insulation value (R = 3.5)
Low vapour permeability (low perm)	Higher vapour permeability, but controlled
Air barrier	Air barrier at full wall thickness
Increases wall strength	
Resists water (is a WRB "Water Resistive Barrier")	
Medium density (1.75 – 2.25 g/cm^3)	Low density (0.4 – 1.2 g/cm^3)
Absorbs sound, especially bass tones	Best sound absorption in normal noise frequency ranges
	Economical yield

2.5.1.2 Polymeric sponge closed cell foams

Polymeric sponge closed-cell foams (Figure 2.3) have varying degrees of hardness, depending on its density. Normally, the strength of closed-cell is strong enough to work on without major distortion. Most of the cells or bubbles in the foam are not broken but piled together in a compact configuration. This makes it strong or rigid because the bubbles are strong enough to take a lot of pressure, like the inflated tires that hold up an automobile. The cells are full of a special gas, selected to make the insulation value of the foam as high as possible (Lefebvre et al., 2008).

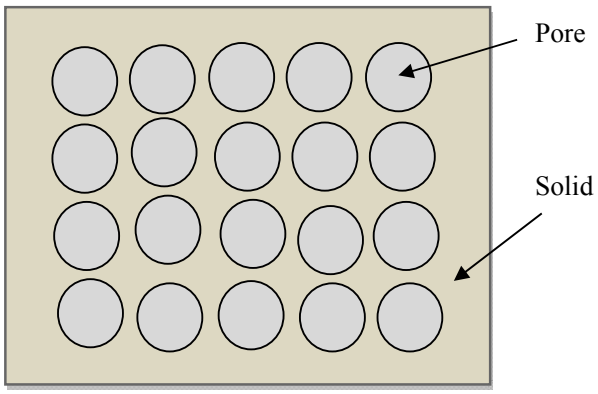

Figure 2.3 The closed cell pore structure

2.5.1.3 Advantages and disadvantages of foams

The advantages of the closed-cell foam compared to open-cell foams include their higher strength and greater resistance to the leakage of air (Lefebvre et al., 2008) or water vapor, high fluid flow, as well as open-cell foams have a poor mechanical property.

2.5.2 Fabrication of porous alumina by pore-forming agent (PFA)

To increase open porosity, pore forming agents are mixed with ceramic powders. These materials are evaporated or burnt out during sintering and as a result pores are formed. Various pore forming agents have been used including potato starch (Bonekamp et al., 1989), coal (Abe et al., 1992), spherical polymer (Hayashi et al., 1991), Iodine fluoride (Shapovalov, 1994), etc. Figure 2.4 illustrates this method. Pore forming agent is classified into two types: solid pore forming agent and liquid pore forming agent. The use of pore forming agents is effective in creating relatively large pores, in comparison with loosely packed samples. The pore shape created by pore forming agents can be controlled by the shape of the pore forming agent (Ishizaki et al., 1998).

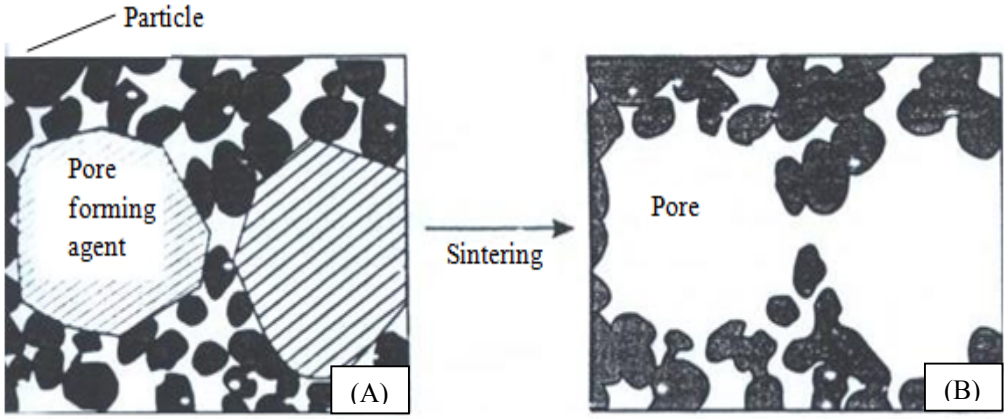

Figure 2.4 Formation of pore using pore -forming agent (A) before sintering and Figure (B) after sintering of (Ishizaki et al., 1998).

2.5.2.1 Solid pore forming agents (SPFA)

Carbon black, charcoal powders and salicylic acid, for example, have been used as solid pore forming agents. The degree of open porosity, pore shape, and the pore size of porous materials made by this method depend on the volume, particles shape and size of the mixed pore forming agent, respectively. This method

is, however, suitable for cost-effective preparation of porous materials with relatively large pores and high open porosity (Ishizaki et al., 1998).

2.5.2.2 Liquid pore forming agents (LPFA)

Liquid pore forming agent does not have the shortcomings of a solid pore forming agents in this respect. Porous alumina ceramics have been fabricated using (PFA) or slip casting method. They have been produced by using a bio-resource (poppy seed) in combination with a new ceramic shaping technique (Gregorova and Pabst, 2007a). In the processing of ceramic hollow spheres, sacrificial (pyrolyzable) (PFA), i.e. synthetic organics or natural biopolymers, were burned out during firing (Rice, 1998). Although a great number of PFAs have been proposed and used in ceramic technology, e.g. carbon (Lange et al., 1990; Corbin et al., 2001; Balaszi et al., 2004), polyvinylchloride, polystyrene, polymethylmetacrylate, polyethylene, wood flour (saw dust) and crushed nut shells, it seems that starch, a natural biopolymer consisting of amylase and amylopectin, has attained a prominent position including its recent application as a combined pore-forming and body-forming agent in starch consolidation casting (Lyckfeldt and Ferreira,1998; Bowden and Rippey, 2002). Among the main reasons for the success of starch as a PFA in ceramics are hygiene and ecological concerns, easy handling and processing (including defect-free burnout), the easy commercial availability in arbitrary amounts, low cost and with constant, controlled quality, the rounded shape with well defined aspect ratio (usually close to unity, without large scatter) and the well defined size distribution for each starch type (Gregorova and Pabst, 2007a).

2.5.2.3 Advantages and disadvantages of PFA

In addition, by varying the weight percentage of the pore-forming agents, the material properties of a porous ceramic can be altered to meet the necessities of various applications (Balaszi et al., 2004). The advantages of slip casting as a forming method are mainly due to the complex geometries that can be shaped, and good material homogeneity is generally achieved. Furthermore, the mould material is cheap. Slip casting is a method for powder-based shaping of ceramic components that has been used for a long time in the traditional ceramic industry for the manufacture of tableware and sanitary ware **(**Gregorova and Pabst, 2007b).

The disadvantages are that a large-scale production normally requires many moulds and large areas, coupled with the fact that the plaster moulds have a limited durability, as plaster of Paris erodes/corrodes in water processing. To get around these problems a method called pressure slip casting or pressure casting has been developed. Instead of plaster moulds, moulds of polymeric materials are used, and these have a porosity consisting of larger pores that do not give the same capillary forces but require an externally applied pressure to drive the filtration process (Lyckfeldt 1997).

2.5.3 Other method

2.5.3.1 Sol-gel processing

Sol-gel processing is the most widely used and developed among various synthetic powder preparation methods. Sol-gel processing starts with a solution (sol) which becomes a gel. The solution can be prepared from either inorganic salts or

organic compounds which is then hydrolyzed and condensed to make a sol or a gel. One can stop at the sol stage which refers to the dispersion of particles of colloidal dimensions in a liquid, or processed to the gel state which refers to a three-dimensionally-linked solid network with liquid filling the pore. These pores are interconnected in the wet gel making stage (Carter and Norton, 2007).

2.6 Flow rate through porous media

A porous medium means a material consisting of a solid matrix with an interconnected void, which supposes that the solid matrix is either rigid (the usual situation) or undergoes a small deformation. The interconnectivity of the pores (the void) allows the flow of one or more fluids through the materials (Chuanbin and Dong, 2010).

Pore connectivity is a key parameter for solute transport and the geometrical arrangement of pores and solid entities is a central issue in the computational of small-scale processes. Several methods have been applied in the computational reconstruction of porous media, including pore networks consisting of pores and connecting throats, random networks where connectivity is based on statistical (Meile and Tuncay 2006). For examples of the natural porous media are beach sand, sandstone, limestone, wood and the human lung in (Figure.2.5).

Filtration through a bed of granular media, usually sand, is a common method of potable water treatment and is also used for ‘cleaning’ of waste water effluent in situations where a high quality discharge is required. Although a superficially simple filtration, it is actually a highly complex process involving a number of

transport and attachment mechanisms. Theoretical consideration between the individual beds as it becomes clogged is thus highly complex (Boomsma et al., 2000).

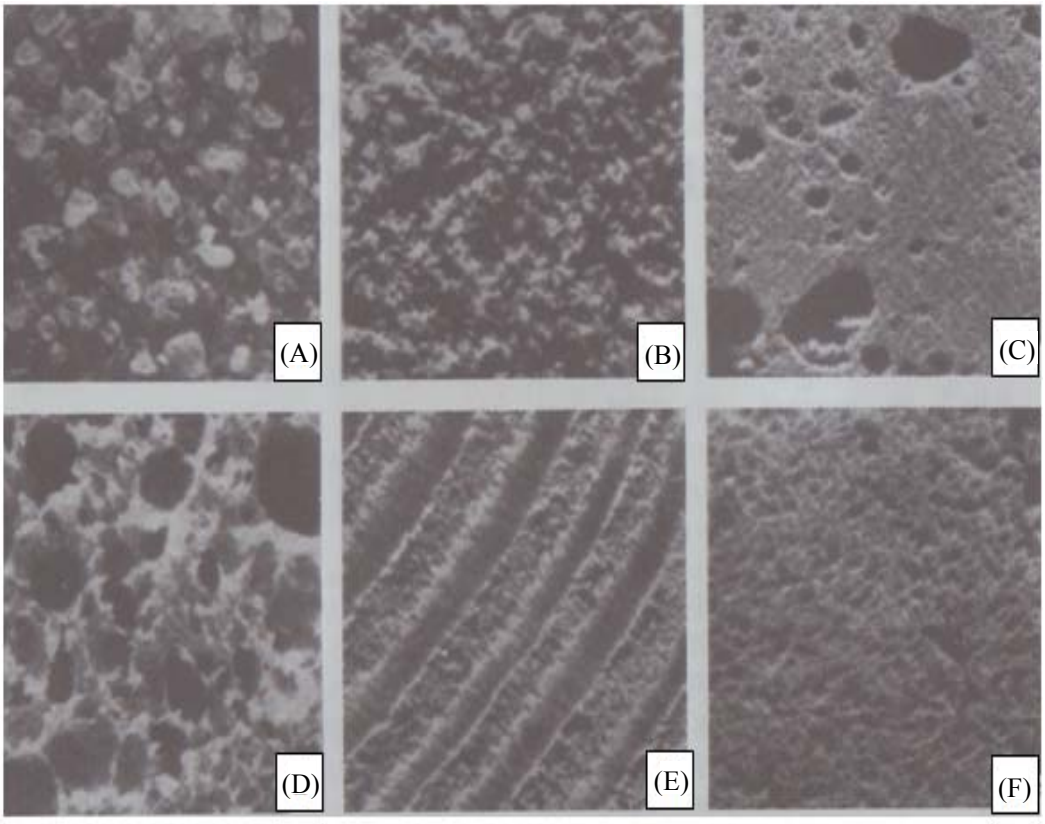

Figure 2.5 Examples of natural porous materials: (A) beach sand, (B) sandstone, (C) limestone, (D) rye bread, (E) wood, and (F) human lung (Boomsma et al., 2000).

CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Introduction

This chapter is composed of two parts. The first part introduced the experimental design to form suitable porous alumina (Al_2O_3) from the slurry. In this part, a polymeric sponge method was used by immersing the sponge into an alumina slurry. The second part used pore-forming agents from natural bioresources (PFANB) which was subsequently poured into a plaster mold. The alumina slurry was prepared using the same method as in the sponge method, followed by adding the natural bioresources. In both methods, the slurries were prepared by ball-milling the combination of materials to obtain a homogenous slurry or suspension. After shaping and drying, the samples were sintered and the sintered samples were characterised using various techniques such as FE-SEM, optical microscope, porosity, water flow rate.

3.2 Starting Raw Materials

The raw materials were carefully chosen because the ultimate properties of the finished products are very sensitive to purity of raw material compositions. In this work, high purity materials were used to prepare porous alumina ceramics, and the materials included: a polymeric sponge, alumina (Al_2O_3) powder, polyvinyl alcohol (PVA) and local natural bioresources (rice husk powder and sugar cane waste powder).