FABRICATION AND CHARACTERIZATION OF POROUS ALUMINA

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FABRICATION AND CHARACTERIZATION OF POROUS ALUMINA

by

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LIST OF ABREVIATIONS

A	- Calculated area of the bearing surface of specimen
C	- Compressive strength
FESEM	- Field Emission Scanning Electron Microscope
TGA	-Thermogravimetric Analysis
L_A	- Length of green specimen
L_B	- Length of sintered specimen
P	- Total load on the specimen at failure
POP	- Plaster Of Paris
PPI	- Pore per inch
PU	- Polymeric Urethane
PVA	- Polyvinyl Alcohol
SEM	- Scanning Electron Microscope
TS	- Tapioca Starch
W_1	- Weight of dry samples
W_2	- Weight of suspended samples
W_3	- Weight of saturated samples
WS	- Wheat Starch
XRD	- X-ray Diffraction

LIST OF SYMBOLS

Al_2O_3	- Alumina or Aluminium Oxide
ºC	- Degree Celsius
d_{50}	- Average particle size
g	- gram
g/mole	- gram per mol
min	- Minute
mm	- Milimeter
MPa	- Mega Pascal
nm	- nanometre
s	- Second
t	- Time
µm	- Micron meter
λ	- Wavelength of radiation field

FABRIKASI DAN PENCIRIAN ALUMINA BERLIANG

ABSTRAK

Alumina berliang telah digunakan secara meluas di dalam aplikasi kejuruteraan. Di dalam kajian ini, alumina berliang telah difabrikasi melalui kaedah span polimer dan tuangan slip. Bagi kaedah span polimer (menggunakan span poliuretana (PU)), jenis bahan pengikat (polivinil alkohol (PVA), kanji gandum (WS) dan kanji ubi kayu (TS)), beban pepejal (50, 60 dan 70 peratus berat), suhu pensinteran (1200, 1300, 1400 dan 1500 ^{o}C) dan teknik salutan semula buburan telah dikaji. Bagi kaedah tuangan slip, PVA telah digunakan sebagai bahan pengikat dan pembentuk liang, kesan beban pepejal (50, 60, 70 dan 75 peratus berat) dan suhu pensinteran (1200, 1300, 1400 dan 1500 ^{o}C) telah dikaji. Keputusan menunjukkan alumina berliang dihasilkan melalui kaedah tuangan slip mengandungi liang tertutup, saiz liang pada julat 78-300 μm dan kekuatan mampatan lebih tinggi (1.22-42.79 MPa). Sementara itu, alumina berliang dihasilkan menggunakan kaedah span polimer mengandungi liang bersambung, saiz liang pada julat 800 μm-1.5 mm dan peratus keliangan yang lebih (77.66-95%). Bagaimanapun, menggunakan teknik salutan semula buburan, kekuatan mampatan sampel telah ditingkatkan. Kesimpulannya, alumina berliang dihasilkan menggunakan kaedah span polimer di dalam kajian ini berpotensi digunakan sebagai penapis gas panas, sementara alumina berliang melalui kaedah tuangan slip berpotensi digunakan sebagai pemangkin.

FABRICATION AND CHARACTERIZATION OF POROUS ALUMINA

ABSTRACT

Porous alumina have been widely used in engineering applications. In this study, porous alumina has been fabricated using polymeric sponge and slip casting methods. In polymeric sponge method (using polyurethane sponge (PU)), type of binders (polyvinyl alcohol (PVA), wheat starch (WS) and tapioca starch (TS)), solid loadings (50, 60, and 70 wt. %), sintering temperatures (1200, 1300, 1400 and 1500 ^{o}C) and slurry recoating technique were studied. In slip casting method, polyvinyl alcohol (PVA) has been used as a binder and pore former, and the effect of solid loadings (50, 60, 70 and 75 wt. %) and sintering temperatures (1200, 1300, 1400 and 1500 ^{o}C) were studied. Results showed that porous alumina produced by slip casting method consisted of closed pores, pore size in ther range of 78 to 300 μm and higher compressive strength (1.22- 42.79 MPa). Whereas, porous alumina produced by polymeric sponge consisted of interconnected pores, pore size in the range of 800 μm to 1.5 mm and higher percentage of porosity (77.66 to 95 %). However, using recoating technique, it showed the compressive strength of the samples was improved. In conclusion, porous alumina produced by polymeric sponge method in this study has a potential to be used as hot gas filtration, whereas; porous alumina produced by slip casting method has a potential use as a catalysis.

CHAPTER 1

INTRODUCTION

1.1 Background of research

According to Guzman (2003), materials with porosity over 30% are considered as porous materials. Porous materials are needed for various applications in daily necessities such as removing dusts for semiconductor production, purify drinking water, fabrication of sandwich panel and others (Ishizaki et al., 1998). Porosity of porous materials can be classified in three categories: moderate porosity (30-50%), high porosity (60-75%) and superhigh porosity (over 75%) (Guzman, 2003).

1.2 Porous ceramic

Over the last few years, there is an increase in interest in the fabrication and use of highly porous ceramic materials due to their multiple applications and utilizations in a variety of areas. In general, porous ceramics have been greatly investigated as structural materials for engineering applications involving filtration or fluid flow in high temperature applications due to its specific properties such as large surface area, high permeability, low density, stability at high temperature, low specific heat, and high thermal insulation (Scheffler and Colombo, 2005).

Porous ceramics can be divided into two types: reticulated ceramic and foam ceramic. Reticulated ceramic contains interconnected voids surrounded by webs of ceramic whilst foam ceramic is composed of closed voids within a continuous ceramic struts (Cao et al., 2004). Generally, reticulate porous ceramics are most commonly used for molten metal, diesel engine exhaust filters, and catalyst supports (Zhu et al., 2002b). Whereas, foam ceramic is used mostly for sonic and thermal insulation application, or low- density ratio structural components (Ishizaki et al., 1998).

1.3 Porous alumina

Alumina (Al_2O_3) is the common raw material used for making high temperature applications of porous ceramics (Surabhi, 2012). Poco et al. (2001) stated that the properties of porous alumina that are of interest to the ceramic industry are its high strength, good thermal conductivity, wear and abrasion resistance and the capability to withstand corrosive environment. For example, due to its special property of high resistance to molten aluminum attack, porous alumina ceramics are mainly used in application of molten aluminum filtration (Han et al., 2003).

Currently, various processing techniques have been used to fabricate porous alumina ceramics (Bai et al., 2011).The range of porosity, the pore morphology and pore size distribution of final samples are generally determined by fabrication method selected (Peng et al., 2000a). Therefore, the choice of processing route for the production of a specific porous ceramics is dependent on the desired properties and applications of final products.

Among those methods, the commonly used processing routes to fabricate porous alumina are polymeric sponge and slip casting methods (Zhu et al., 2002a; Dhara and Bhargava, 2003; Luyten et al., 2005; Tripkovic et al., 2006; Abd. Rahman and Guan, 2007). According to Han et al., (2003), the products produced by polymeric sponge offer large pores and high porosity whilst porous alumina ceramics with small pores, large surface area, and low porosity are obtained by using slip casting method.

1.4 Problem statement

Porous alumina is currently the focus of active research and development activities due to their high applications in industrial such as filtration of molten meals, high temperature thermal insulation, hot gas filtration, diesel engine exhaust filters and other range of applications (Svinka et al., 2009, Hamimah and Guan, 2008).

Many processing techniques are available to fabricate porous materials such as polymeric sponge, slip casting, sol-gel and others. Polymeric sponge is the most popular method, which can produce porous structure with controllable porosity and pore size, interconnected pores, and relatively high porosity (Studart et al., 2006, Scheffler and Colombo, 2005).

With slip casting method, a better homogeneous powder suspension can be achieved (Gauckler, 1998). Also, materials with a variety complex shape can be fabricated which could not be produced by other conventional methods. More than that, the fabrication of porous materials using slip casting method is relatively low cost since it is a conventional method and its mould is inexpensive (Lyckfeldt and Ferreira, 1998, Haris, 2009).

In fabrication of porous ceramic, binders play an important role in providing strength to green body (Lewis, 1997). Binders in powder form are mixed with ceramic materials and liquid to form viscous solution. They can be used to bind particles together, provide plasticity and help to control the flow properties of slurry (Mohanty, 2011). The types of binders that have been used for the fabrication of porous ceramic are polyvinyl alcohol (PVA) (Han et al., 2002, Hadi et al., 2011), potato, corn cassava (Talou et al., 2010), poppy seed (Gregorová and Pabst, 2007), and wheat (Prabhakaran et al., 2007). Different types of binder would give different properties of sintered porous ceramics.

Beside binders, other factors such as solid loading and sintering temperature would affect the final properties of porous ceramics (Lui, 2011). Sintering temperature and solid loadings are the factors affecting the densification that provide the strength of ceramic materials and other properties (Haris, 2009, Abd.Rahman and Guan, 2007, Khattab et al., 2012). Different solid loadings and sintering temperature are used, and different properties of final products are obtained. Consequently, the effect of sintering and sintering temperatures on the porous alumina were studied in this study in order to find out its optimum properties.

Usually, porous alumina ceramics fabricated via polymeric sponge method are of low strength and fracture toughness due to large porosity and thin webs with holes in their center (Brezny and Green, 1989, Zhu et al., 2001). Since the mechanical strength of porous materials by polymeric sponge is relatively low, several methods have been suggested to improve its strength characteristics. Attempts have been made for improving the neck growth among matrix grain and reducing crack formation during fabrication in order to ensure mechanical properties reliability (Ohji and Fukushima, 2012). For example, Yao et al., (2006) has successfully improved the strength of porous silicon carbide (SiC) ceramics by recoating process.

1.5 Objectives

This work is conducted to achieve the following objectives:

1. To fabricate porous alumina using two different methods: polymeric sponge and slip casting methods
2. To investigate the effect of different types of binder, solid loadings and sintering temperatures on the properties of sintered porous alumina
3. To improve the mechanical properties of porous alumina produced by sponge method and using recoating technique

1.6 Project overview

A brief flow chart of study is enclosed to explain the procedures adopted in the research project. In this research, porous alumina was manufactured by two different techniques: polymeric sponge and slip casting methods. In polymeric sponge, without recoating and with recoating techniques were involved. Polyvinyl alcohol (PVA) was used as a binder and pore forming agent in slip casting method.

Raw materials characterization was conducted in order to ensure their properties and raw materials condition before starting the next process of experiment. In this study, alumina (Al_2O_3) powder was characterized by using X-ray diffraction (XRD) to identify its phase purity. Raw materials morphology was observed by using field emission scanning electron microscope (FESEM). Particle size analysis was carried out to determine the average particle size and particle size distribution of raw materials used. The decomposition temperature of polymeric sponge and binders used were analysed by using thermogravity analysis (TGA). Fourier Transform Infrared Spectroscopy (FTIR) was conducted to identify the chemical bonding and functional group of polymeric sponge. Alumina slurries were prepared with different compositions and the samples were sintered at different temperatures.

The variety of characterization techniques are required to analyze and evaluate the properties of samples. The shrinkage test was carried out to find out the shrinkage of specimens before and after firing. Sintered samples were characterized by density and porosity test for its physical properties. The compressive strength test was conducted to determine the mechanical properties of sintered porous alumina. The morphology and microstructure analysis was carried out using scanning electron microscope (SEM), Hitachi TM3000. The details of each process were described in Chapter 3.

CHAPTER 2

LITERATURE REVIEW

2.1 Porous ceramics

In general sense, solids containing pores or holes are considered as porous materials. The porosity is the fraction of the pore volume to the total volume. Porosity can be found in almost every types of engineering materials such as metals, ceramics, polymers, composites, semiconductors and biomaterials. Porosity can affect the performance, properties, strength and density of materials. Those properties are dependent on the pore structure of that solid.

Porous ceramic offers irreplaceable and unique merits such as environmental stability and high temperature in comparison to the conventional polymers and metals (Colombo, 2006). Porous ceramic can be applied in many industries applications due to the good resistance to high temperature, chemical corrosion, high strength, porosity distribution, and long working life and easy to reproduce (Dong et al., 2008). Porous ceramic composed of specific properties such as high surface area, high permeability, high temperature stability, low weight and low thermal conductivity. Table 2.1 exhibits an overview of some relative properties of porous ceramic (Scheffler and Colombo, 2005).

Table 2.1 Some relative properties of porous ceramic (Scheffler and Colombo, 2005)

Low	High
Relative density	Specific strength
Thermal conductivity	Permeability
Dielectric constant	Thermal shock resistance
Thermal mass	Porosity
	Specific surface area
	Hardness/wear resistance
	Resistance to chemical corrosion

2.1.1 Structure of porous ceramic

Porous ceramic is also called as ceramic foam or cellular ceramics. It has been reported that porous ceramic has a three-dimensional network structure and a honeycomb structure. The structure of porous ceramics comprised of void surrounded by a web of ceramics. Pores inside the porous ceramic can be classified into open pores and closed pores. Open pores are connected to the outside of the material surface and can be penetrated by fluids, while closed pores are isolated holes. The component of porous ceramic can be divided into strut and vertex. In porous ceramic structure, the connection between pores or the wall separating the pores space is named as “strut”, and “vertex”, is where the struts join. Some of porous ceramic structure parameters for open and closed pores are shown in Figure 2.1. (Scheffler and Colombo, 2005)

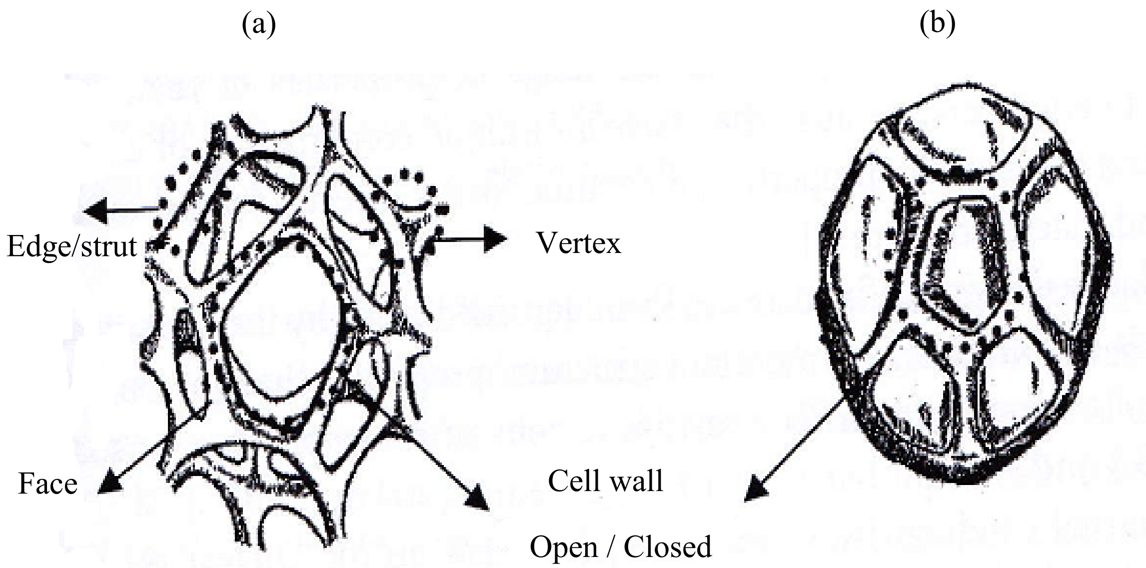

Figure 2.1 Some components of a cell unit of porous ceramic structure in a) open and b) closed pores (Scheffler and Colombo, 2005)

2.1.2 Classification of porous ceramic materials

Porous materials can be classified based on distinct criteria such as pores shape, pores size and fabrication techniques. Porous ceramic products can be categorized based on the following specifications (Guzman, 2003):

- ✓ Chemical compositions of raw material: silicate, aluminosilicate, oxide, non-oxide, oxygen-free and others
- ✓ Porosity: moderate (30-50%), high porosity (60-75%), and super-high porosity (> 75%)
- ✓ Physical rate of products: filling, continuous, piecewise
- ✓ Inner structure: granular, cellular, fibrous

- ✓ Refractoriness correlated to service temperatures: low-melting (below 1350 ºC), high-melting (1350-1580 ºC), refractory (1580-1770 ºC), high-refractory (1770-2000 ºC), super refractory (over 2000 ºC)
- ✓ Destination and application area: heat insulating (main parameter: thermal conductivity), heat shielding (main parameter: product of heat conductivity and apparent density value), and permeable (main parameters: porosity, pore size and permeability)

However, the International Union of Pure and Applied Chemistry (IUPAC) (Schaeffer, 1994) has defined porous materials depending on pores diameter such as:

- ✓ Microporous materials: pores diameter smaller than 2 nm ($d < 2$ nm),
- ✓ Mesoporous materials: pores diameter between 2 and 50 nm (2 nm< $d < 50$ nm), and
- ✓ Macroporous materials: pores diameter bigger than 50nm ($d> 50$ nm).

Figure 2.2 shows classification along with typical applications and fabrication processes specific to the pore diameters.

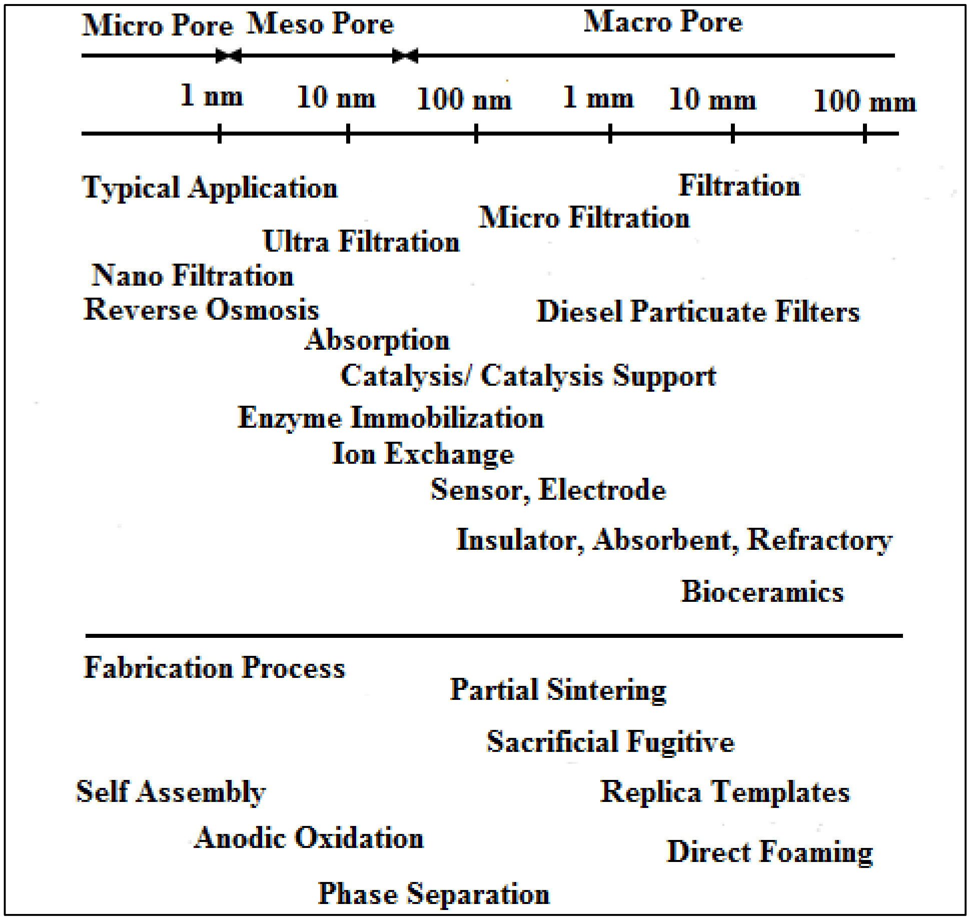

Figure 2.2 Classification of porous materials by pore size and corresponding typical application and fabrication process (Ohji and Fukushima, 2012)

Generally, porous ceramics can be classified into two categories: reticulate ceramics and foam ceramics (Cosentino et al., 2003). A reticulate ceramic contains interconnected voids surrounded by a web of ceramic struts. This type of materials is considered highly porous ceramic which composed of porosity from 70 to 95%, and three dimensional network structure (Mouazer et al., 2005). It is fabricated by infiltration and replication of a polymeric sponge perform (Cao et al., 2004). Reticulate porous ceramics are commonly employed for those applications such as molten metal filtration, diesel engine exhaust filters, catalyst supports and industrial hot-gas filters (Peng et al., 2000b).

A ceramic foam consists of closed and open within a continuous ceramic matrix. This is usually produced by forming processes applying forming agents or by powder consolidation using fugitive organic additives as pore formers (Surabhi, 2012). Porosity, size and shape of pores and permeability of both types of porous ceramics are different. The permeability is high in reticulate ceramics compared to foam ceramics which the permeability is low due to larger pores size and open pores structure.

2.1.3 Applications of porous ceramic

Porous ceramics have numerous industrial applications. Hong et al., (2012) reported that porous ceramics are often utilized in high temperature environment. Most applications of porous ceramic materials require some specific requirements such as microstructure and porosity and mechanical properties (Kritikaki and Tsetsekou, 2009; Ishizaki et al., 1998).

The required properties of porous ceramics are dependent on its applications. Different applications of porous materials require different pore sizes, which can range from the atomic scale to millimetre scale. A summary of typical porous properties of various porous materials and the relationship between pore size and its applications are shown in Figure 2.3 and Figure 2.4, respectively (Okada et al., 2011; Ishizaki et al., 1998).

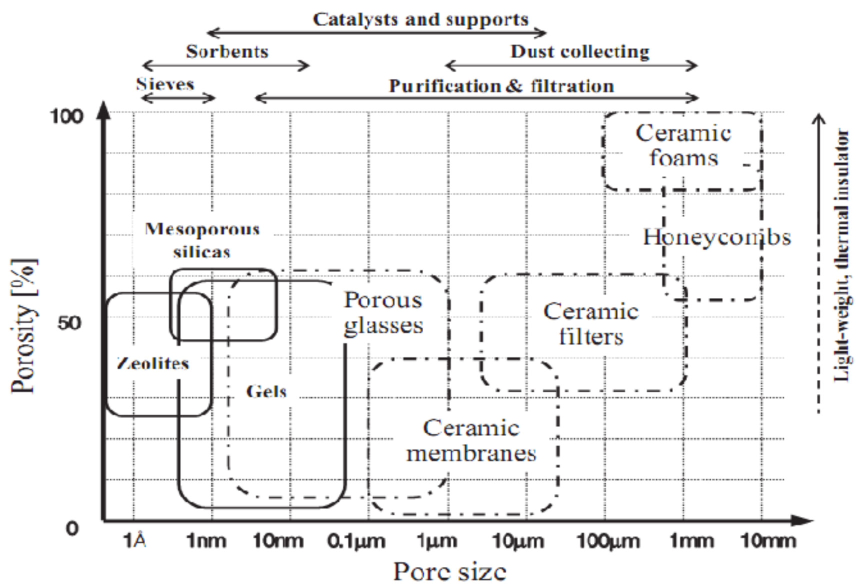

Figure 2.3 Pore size and porosity of typical porous substances and ceramics (Okada et al., 2011)

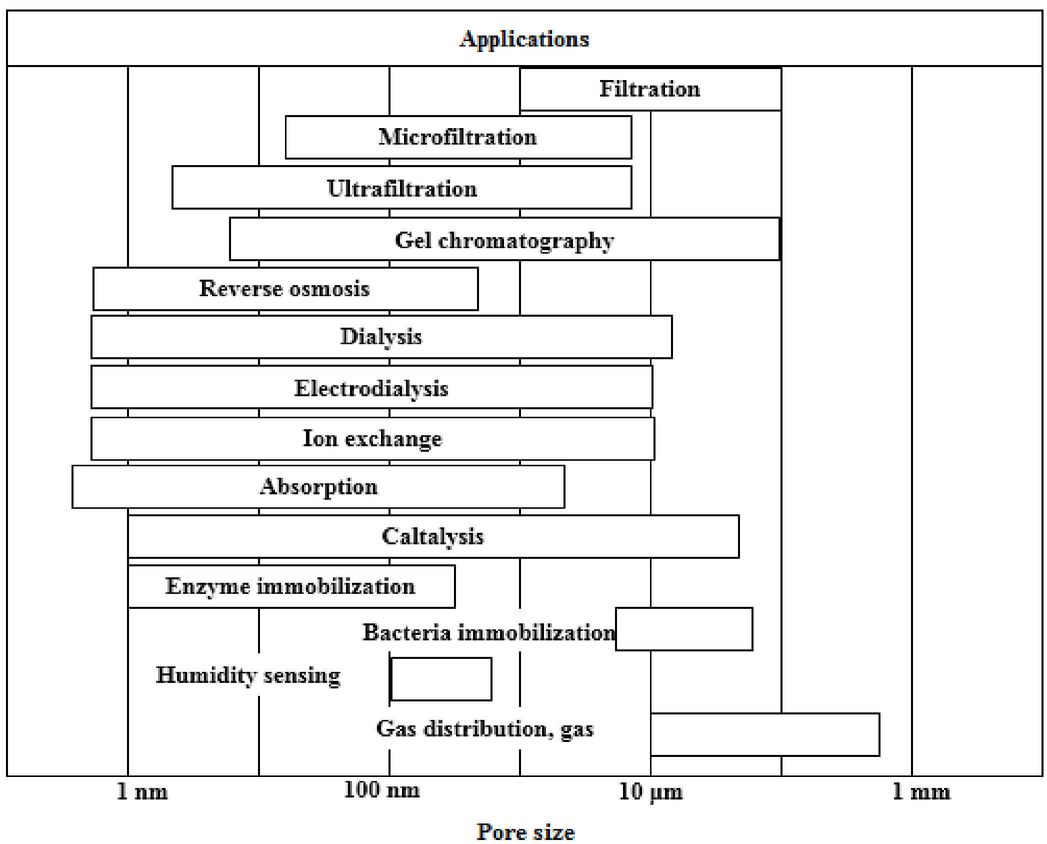

Figure 2.4 The relationship between pore size and applications of porous materials (Ishizaki et al., 1998)

Various applications of porous ceramics have been used including molten metal and hot gas filtration, catalyst support, as well as refractory insulation of furnace (Gonzenbach et al., 2007, Gregorová and Pabst, 2007).

2.1.3.1 Molten metal filtration

Porous ceramics have been used as filtration. During filtration, the impurity particulates get attached to the filtration medium and the liquid passes through a porous ceramic to make it pure. Commonly, porous ceramic is used for filtering molten metal. The material for filtration must be able to retain its strength at high temperature, withstand thermo-mechanical stress and must not react chemically (Bhaduri, 1994). The option of selecting material for this filtration is based on what material is to be filtered. Materials that have been used for high temperature filtration are alumina, aluminum silicate, cordierite, zirconia and steatite.

2.1.3.2 Exhaust gas of diesel engine cleanup

Bhaduri (1994) stated that porous ceramics have been used for the removal of soot particles from the exhaust diesel engine. Ceramic foams are the preferred structure for collecting the soot particles from the exhaust diesel engine at high temperature due to its honeycomb structure (rough surface). The exhaust can be cleaned by being flowed through the ceramic foam. The length of the flow of the exhaust gases through a ceramic foam is between 3 and 15 cm (Mizrah et al., 1990). The filter must also withstand a variety of mechanical, vibrational, and thermal stresses. The materials used for these applications include alumina, mullite, cordierite, silicon nitride and silicon carbide (Mizrah et al., 1990).

2.1.3.3 Catalysts support

Another application of porous ceramic is catalyst support. For catalytic applications, high specific surface area and chemical stability are needed for the increasing of the surface area in contact with reactant and for the corrosive conditions respectively (Ishizaki et al., 1998). For the application of catalysts in high temperature, the automobile exhaust gases and property of high thermal stability are necessary.

2.1.3.4 Refractory insulation for furnace

Refractory bricks that have high thermal shock resistance as well as low thermal conductivity due to the presence of many pores are one of the oldest applications of porous ceramic. The applications of refractory bricks can be found in kilns and furnace in many industrial fields. A part of that, many bricks are being utilized in steel making. Furnace for steel making namely the blast furnace and converter are conducted from refractory bricks. On the other hand, metal working such as heat treating process (quenching and annealing) and rolling are carried out in special type of furnaces that are made from refractory bricks (Ishizaki et al., 1998).

The materials that have been used to produce refractory bricks including alumina, mullite, magnesia, calcia, zirconia, zircon, silicon and carbide and their compounds. Normally, for high applications below 1000 ^{o}C, unrefined alumina-silicate based ceramics can be used. As for high temperature applications, such as 1600 ^{o}C, high purified alumina bricks or magnesia bricks were used (Ishizaki et al., 1998).

2.2 Alumina (Al_2O_3)

Alumina or aluminum oxide (Al_2O_3) is one of the most studied ceramics in engineering ceramic materials due to ease of manufacture, properties of resistance to corrosion and high-temperature refractory nature (McColm, 1983). Based on Insaco Inc. (2006), alumina grades can be classified as 90%-97% purity of alumina and 98%-99.95% purity of alumina. Alumina with purity range of 90%-97% is best suited for metalizing (metal deposition which allows brazing) due to large grain structure. The common range for isostatically pressed grades, with extruded shapes and available at low cost is alumina with purity range of 98%-99.95%.

Alumina has a number of phases such as alpha (α) phase, gamma (ɣ) phase and beta (β) phase. Among the aluminum oxide, α-alumina is the most stable phase thermally (Sifontes et al., 2010). While other phases of alumina change at certain temperature. For example, at temperature of 1000 ^{o}C, ɣ- alumina phase will convert to α-alumina.

McColm (1983) reported that α-alumina occurs in corundum structure. It has a hexagonal structure and two alumina molecules per unit as presented in Figure 2.5. Three oxygens form an equilateral triangle with aluminums above and below the center of triangle. One of these group is placed at each corner of a cube while another is placed at the center of the cube. α-Al_2O_3 is produced by Bayer process using bauxite as a starting material (Munro, 1997) .

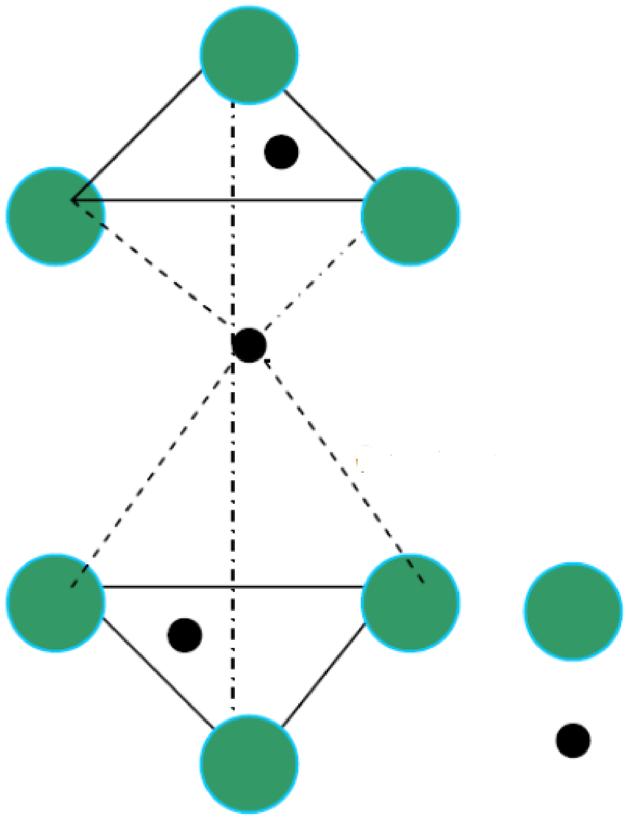

Figure 2.5 Schematic demonstration of oxygen arrangement around Al^{3+} ion in alumina (McColm, 1983)

Alumina is especially used for high- temperature applications (Svinka et al., 2009). Creeping and sintering of alpha-alumina begins at 1750°C and it will melt at temperature of 2040°C (Harper, 2001). High-density, high-purity (>99.5%) Al_2O_3 (α-alumina) was the first bioceramic widely used clinically (Hench, 1998). Properties of alumina are shown in Table 2.2.

Table 2.2 Mechanical properties of alumina (Insaco Inc., 2006)

Property	Value	Units
Density	3.88	gm/cc
Hardness	82	Rockwell 45N
Tensile Strength	372.32	MPa
Flexural Strength	351.63	MPa
Poisson's Ratio	0.23	-
Compressive Strength	2537.27	MPa
Fracture Toughness	4.3	MPa $m^{1/2}$

2.3 Porous alumina

Materials such as alumina, zirconia, cordierite, mullite, silicon carbide, silicate glass, carbon, and concrete are materials typically used for manufacturing porous ceramics (Colombo, 2006). Among oxides utilized for the production of porous ceramics, alumina has a particular importance since most porous alumina or alumina-based ceramics exhibit relatively high strength along with improved thermal and chemical stability (Cao et al., 2004; Poco et al., 2001). Therefore, porous alumina ceramics play an important role in their high temperature applications (Svinka et al., 2009).

2.4 Fabrication techniques for porous alumina ceramics

Various technologies have been developed for the fabrication of porous ceramic materials in order to response to the growing demand for its industrial applications. There are many different methods used to produce porous alumina ceramics found in the literature review such as polymeric sponge method, gel casting, ice templating, slip casting, and freezing (Bai et al., 2011). Each method has its own advantages and drawbacks. The structure and properties of porous ceramics are controlled by the process applied. Therefore, the selection of the processing route should depend primarily on the final application. Those processing routes have been developed for specific applications and some specific requirements such as porosity, pore size and degree of interconnectivity.

In this study, polymeric sponge method and forming technique (slip casting) were used as a processing route to produce porous alumina.

2.4.1 Polymeric sponge method

Polymeric sponge method, which was invented by Schwartzwalder and Somers in 1963, is the most famous techniques used to fabricate reticulated porous ceramics (Zhu et al., 2002b). In this technique, the polymeric sponge (usually polyurethane (PU) sponge) is first immersed into the ceramic slurry (Colombo and Hellmann, 2002). Then, the excess slurry is removed by squeezing, and the ceramic coating over the struts of initial polymeric sponge is formed. The strength of porous ceramic body is provided after the ceramic-coated polymeric sponge is dried and sintered by heat treatment to remove the organics substances such as polymeric sponge template and binders. The basic principle for this technique is illustrated schematically in Figure 2.6.

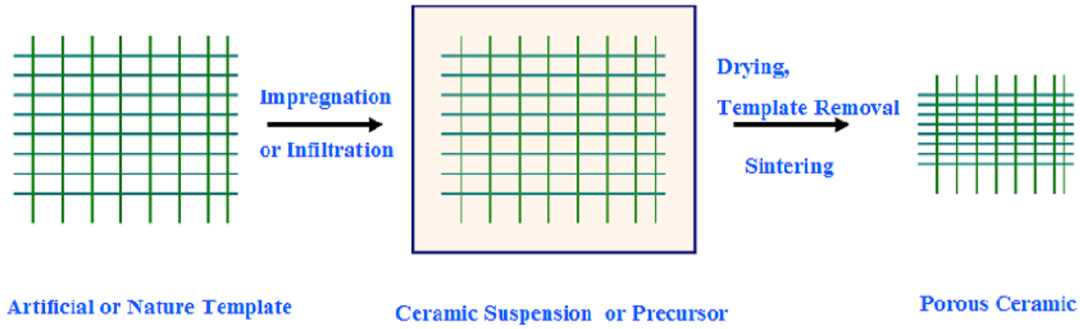

Figure 2.6 Schematic of polymeric sponge method utilized for the fabrication of porous ceramics (Studart et al., 2006)

Polymeric sponge method has been used to produce porous ceramics with different chemical compositions such as alumina, silicon carbide and hydroxyapatite. Several literatures that reported examples of porous ceramics fabricated by polymeric sponge method are listed in the following paragraph.

Sarkar et al. (2008) studied the production of porous TCP coated Al_2O_3 scaffold by polymeric sponge method. Polyurethane (PU) sponge was used as a template of TCP coated Al_2O_3 scaffold in that study. A porous structure was obtained after being sintered at temperature of 1500 ^oC. It was revealed that the scaffold has excellent interconnected porosity with pore sizes ranging from 300~700 μm in diameter.

Han et al. (2002a) successfully used polymeric sponge method to fabricate porous alumina. Polyurethane (PU) sponge was used as a template of porous alumina. It was observed that porous alumina with large pores and high porosity was obtained by using polymeric sponge technique.

Tripkovic et al. (2006) successfully fabricated porous alumina ceramic by using a polymeric sponge method. Samples were sintered at temperature of 1400 ^{o}C. The effect of viscosity on the characteristics of the final product produced by this technique was studied. It was revealed that the microstructure of porous ceramics is highly affected by the viscosity of the slurry. The samples prepared with more viscous slurry were stronger but less porous compared with the samples from a slurry having a lower viscosity.

By polymeric sponge method, Tripathi and Basu (2012) used PU sponge as a template to produce interconnected macroporous hydroapatite. Samples with pore size range of 100-300 μm and wall thickness of 50 μm could be achieved. The samples prepared with 60 wt.% HA loaded scaffold exhibited compressive strength of 1.3 MPa. It was confirmed that it is possible to produce porous scaffold with homogeneous pore distribution and wall thickness by using polymeric sponge method.

2.4.1.1 Template of porous alumina ceramic

Many synthetic and natural cellular structures were employed as the template of porous materials with various size and shape for the purpose of designing the specific applications. The templates for porous materials have to be burnt off during sintering process. After the template was removed, a matrix of particle with pore space that takes on the appearance of original template is left. Up to date, two different types of templates, artificial template (synthetic template) and natural template, can be used to fabricate porous ceramic through polymeric sponge method.

For synthetic template, many polymeric-sponge materials such as poly-urethane (PU), cellulose, polyviny1 chloride (PVC), poly (styrene) (PS), latex can be used as a template of porous ceramics (Montanaro et al., 1998). Among those polymeric sponges, polyurethane (PU) sponge is more widely used due to its low softening temperature and ease burning which can reduce the thermal stress that may fracture the ceramics (Hirschfeld et al., 1995, Bhaduri, 1994). Ramay and Zhang (2003) mentioned that sufficient time and slow heating rate are required in order to remove the PU foam. Otherwise, the final product will be a collapsed material. The soaking time is added in order to give sufficient time for the completed removal of PU foam during sintering process (Ramay and Zhang, 2003).

Beside the synthetic template, natural replica template which is available in nature also can be used as a template for the fabrication of porous ceramic. Natural template such as corals and wood (Cao et al., 2004) have been utilized as a template of the production of porous ceramic materials.

2.4.1.2 Advantages and disadvantages of polymeric sponge method

By using polymeric sponge method, porous ceramic structures with controllable pore size, interconnected pores (between 200µm to 3mm (Studart et al., 2006)) and desired geometry are obtained (Ramay and Zhang, 2003, Sifontes, et al., 2010). In addition to that, the porous ceramics manufactured by this technique generally have a wide range of porosity within the range of 40- 95% (Studart et al., 2006). Figure 2.7 shows the example of SEM of scaffold of porous ceramic prepared by polymeric sponge method.

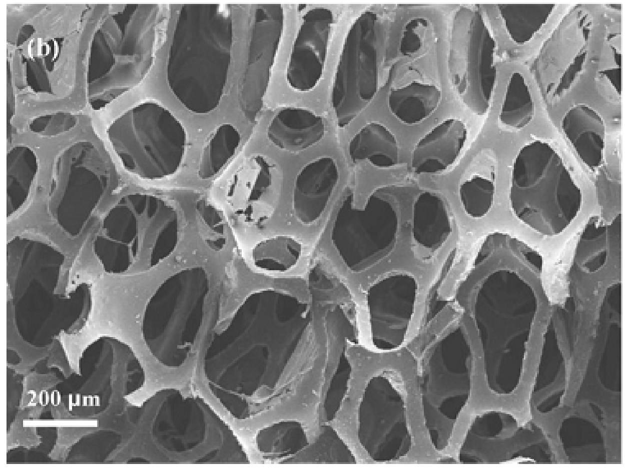

Figure 2.7 SEM image of porous ceramic produced via sponge method (Tripathi and Basu, 2012)

However, some defects might occur in the fabrication of porous ceramics via polymeric sponge method. The materials fabricated using this method consist of low strength, fracture toughness because of high porosity level, and the formation of hollow struts and residual cracks during processing of burning sponge template (Yu et al., 2008, Luyten et al., 2005). It has been observed that voids and cracks were found between the ceramic walls after the removal of organic substances during pyrolysis as shown in Figure 2.8. According to Han et al., (2002a), this can cause the porous ceramics produced using polymeric sponge techniques become sensitive to structural stress and limiting their applications.

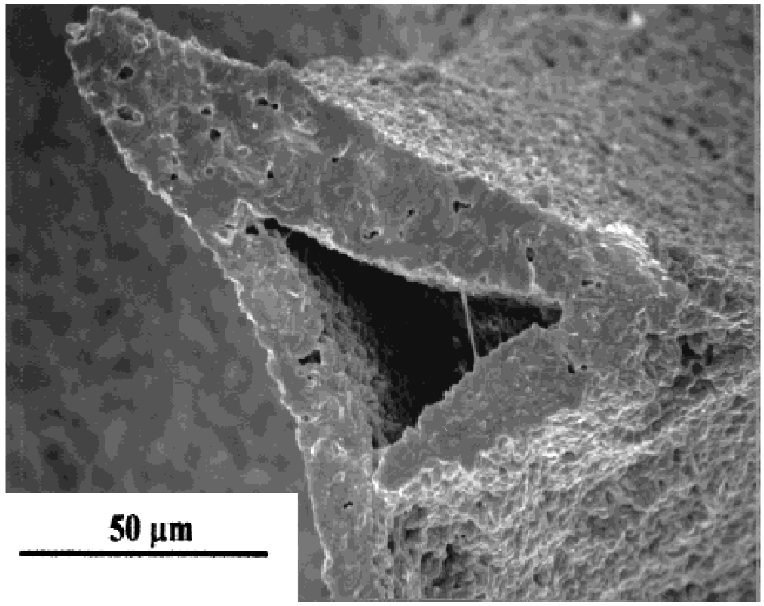

Figure 2.8 SEM image of some typical defects (triangular pores insides struts) of foam prepared using polymeric sponge method (Tulliani et al., 1999)

2.4.1.3 Recoating of slurry ceramic

Since very thin struts of ceramic structure are obtained after the organic sponge is burnt out, the ceramic materials produced by polymeric sponge normally have low strength and toughness. In order to overcome those drawbacks, several attempts have been made. Several techniques have been developed to thicken the ceramic coating on to improve the mechanical properties of porous ceramic materials. For example, the sponge vacuum infiltration of ceramic slurry and recoating of porous ceramic body were applied to reduce or eliminate the disadvantages of polymeric sponge technique (Zhu et al., 2001; Vogt et al., 2010).

Zhu et al. (2001) has been successfully fabricated reticulated porous ceramic with the improvement of ceramic body struts thickness, by using recoating technique. They