



**Faculty of Mechanical Engineering**

**EFFECT OF PORE-FORMING AGENTS IN MACROPOROUS  
CERAMIC FABRICATION AS CARBON DIOXIDE ADSORPTION**

**Nurulfazielah binti Nasir**

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FABRICATION AS CARBON DIOXIDE ADSORPTION**

**Nurulfazielah binti Nasir**

**A thesis submitted  
in fulfilment of the requirements for the degree of Master of Science  
in Mechanical Engineering**

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**UNIVERSITI TEKNIKAL MALAYSIA MELAKA**

**2018**

## DECLARATION

I declare that this thesis entitled “Effect of Pore-forming Agents in Macroporous Ceramic Fabrication as Carbon Dioxide Adsorption” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : .....

Name : .....

Date : .....

## **APPROVAL**

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Mechanical Engineering.

Signature : .....

Supervisor name : .....

Date : .....

## **DEDICATION**

*“To my beloved husband, daughter, family and family in-law”*

## ABSTRACT

Currently, the excessive emissions of carbon dioxide in atmosphere which can cause the increasing of average temperature in atmosphere has become one of the most urgent environmental issues. This problem has triggered research for ways to reduce carbon dioxide emission. The aims of this research are to fabricate macroporous ceramic material by using mixture of aluminium powder with polymeric spheres, and yeast as pore-forming agent; determine the main properties of ceramic product such as porosity, pore size and mechanical strength; and analyse carbon dioxide adsorption on porous ceramic product. The preliminary experiment shows that yeast was found to give better results as a pore-forming agent compared to the mixture of aluminium powder and polymeric spheres. The average pore sizes by using yeast are closer to 200  $\mu\text{m}$  which is the optimal contact surface area with the gas flow and to ensure the uptake time of carbon dioxide gas in the order of seconds. The porous ceramic material was developed by the mixing of alumina, zeolite and calcium oxide as the main materials, yeast as the pore-forming agent and ethylene glycol as the binder. The yeast content varied from 0% up to 40% from the total weight of ceramic materials. Then, the slurry was cast into mould and allowed to dry under room temperature before being sintered at 1400  $^{\circ}\text{C}$  for two hours. Microstructural analysis and pores size measurement were performed to determine the effect of pore-forming agent on the ceramic and mechanical properties test has been carried out to determine the effect of density and porosity of sintered porous ceramic toward its mechanical strength. From the results obtained, the average apparent porosity and pore size increased with the increased weight percentage of yeast content from 35.46% to 46.54% and 49.814 $\mu\text{m}$  to 194.297 $\mu\text{m}$ , respectively. The increasing of porosity and pore size give an effect to the compression strength of sintered porous ceramic by decreasing it from 17.47 MPa to 10.66 MPa, which were inversely proportional to porosity and pore size. The phase determination by XRD, mapping and point ID spectrum at several points by SEM-EDX of the sintered ceramic indicates that zeolite particles remained after been sintered at 1400  $^{\circ}\text{C}$ . The increased average apparent porosity and pore size increased the volume of carbon dioxide adsorption. It was found that 20 wt.% of yeast content suitable to be applied as carbon dioxide filter.

## ABSTRAK

*Pada masa ini, pelepasan karbon dioksida secara berlebihan di atmosfera telah menjadi salah satu isu alam sekitar yang penting di mana ia boleh menyebabkan peningkatan purata suhu di atmosfera. Masalah ini telah mencetuskan pelbagai cara bagi mengurangi pelepasan karbon dioksida di atmosfera. Oleh itu, objektif kajian ini adalah untuk menghasilkan bahan seramik berliang dengan menggunakan campuran serbuk aluminium dengan sfera polimer dan yis sebagai agen liang-membentuk; menentukan sifat-sifat utama produk seramik seperti keporosan, saiz liang dan kekuatan mekanikal; dan menganalisis penyerapan karbon dioksida pada produk seramik berliang. Eksperimen awal menunjukkan bahawa yis didapati memberi hasil yang lebih baik sebagai agen liang-membentuk berbanding dengan campuran serbuk aluminium dan sfera polimer. Purata saiz liang dengan menggunakan yis lebih dekat kepada 200  $\mu\text{m}$  yang merupakan kawasan permukaan sentuhan yang optimum dengan aliran gas bagi menjamin masa pengambilan gas karbon dioksida dalam urutan detik. Bahan seramik berliang telah dihasilkan dengan mencampurkan alumina, zeolit dan kalsium oksida sebagai bahan utama, yis sebagai agen liang-membentuk dan etilena glikol sebagai pengikat. Kandungan yis yang digunakan adalah berbeza-beza dari 0% sehingga 40% daripada jumlah berat keseluruhan bahan seramik. Kemudian, buburan tersebut dibentuk dalam acuan dan dibiarkan kering di dalam suhu bilik sebelum dipanaskan pada suhu 1400 °C selama dua jam. Analisis mikrostruktur dan pengukuran saiz liang telah dijalankan untuk menentukan kesan ejen liang-membentuk pada seramik dan ujian mekanikal telah dijalankan untuk menentukan kesan ketumpatan dan keliangan seramik berliang ke arah kekuatan mekanikal. Daripada keputusan-keputusan yang diperolehi, purata keporosan permukaan dan saiz liang masing-masing meningkat dengan peratusan berat kandungan yis dari 35.46% kepada 46.54% dan dari 49.814  $\mu\text{m}$  kepada 194.297  $\mu\text{m}$ . Peningkatan keporosan permukaan dan saiz liang ini memberi kesan kepada kekuatan tekanan seramik berliang tersinter dengan mengurangkan kekuatan mekanikal bahan dari 17.47 MPa kepada 10.66 MPa, di mana ia berkadar songsang dengan keporosan permukaan dan saiz liang. Fasa penentuan oleh XRD, pemetaan dan titik spectrum ID di beberapa tempat oleh SEM-EDX menunjukkan bahawa partikal-partikal zeolite kekal selepas disinter pada suhu 1400 °C. Peningkatan purata keporosan permukaan dan saiz liang mempengaruhi isipadu penyerapan karbon dioksida. Didapati, 20 wt.% kandungan yis sesuai untuk diaplikasi sebagai penapis karbon dioksida.*

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## LIST OF ABBREVIATIONS

ASTM	-	American society for testing and materials
AZPC	-	Alumina-zeolite porous ceramic filter
EDX	-	Energy dispersive X-ray
EPS	-	Expandable polymeric spheres
HC	-	Hydrocarbon
MOFs	-	Metal organic frameworks
SEM	-	Scanning electron microscopy
XRD	-	X-ray diffraction

## LIST OF SYMBOLS

$\text{\AA}$	-	Angstrom
$A$	-	Area
$B$	-	Bulk density
$C$	-	Compressive strength
$Cu\ K\alpha$	-	Radiation diffraction intensity
$D$	-	Dry mass
$d_{50}$	-	Average particle size
$l_g, l_s$	-	Diameter sample
$m$	-	Mass
$M$	-	Saturated mass
$P$	-	Total load
$S$	-	Suspended mass
$V$	-	Volume
$vol.\%$	-	Volume percentage
$wt.\%$	-	Weight percentage
$\rho, \rho_s$	-	Density

## LIST OF PUBLICATIONS

Nurulfazielah Nasir, Ridhwan Jumaidin, Mohd Zulkefli Selamat, Suhaila Salleh and Kok-Tee Lau, 2015, Effect of different types of pore-forming agent and sintering temperature on the macro pore size of ceramic. *Applied Mechanics and Materials*, Vol. 761, pp. 380-384, <http://dx.doi.org/10.4028/www.scientific.net/AMM.761.380>

Nurulfazielah Nasir, Ridhwan Jumaidin, Hady Efendy, Mohd Zulkefli Selamat, Goh Keat Beng and Muhammad Zulfattah Zakaria, 2014, Preparation of macroporous ceramic materials by using aluminium powder as foaming agent. *Applied Mechanics and Materials*, Vol. 699, pp. 336-341, <http://dx.doi.org/10.4028/www.scientific.net/AMM.699.336>

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background

Carbon dioxide ( $\text{CO}_2$ ) is a colourless and odourless gas found within our atmosphere that surrounding us in our daily life. Theoretically, the atmosphere contains about 78.053% of nitrogen gas, 21.014% of oxygen gas, 0.901% of argon gas, 0.030 % of carbon dioxide gas and 0.002% of other gases (Harrison, 1992). This means that the concentration of  $\text{CO}_2$  in the atmosphere is about 300 parts per million from these percentages.

However, from decades to decades, the concentration of  $\text{CO}_2$  keep on increasing at an accelerating rate from February 1959 until February 2017 as observed from Mauna Loa Observatory as shown in Figure 1.1. Since early February 1989 to date, the concentrations of atmospheric  $\text{CO}_2$  levels have continued to increase higher than 350 parts per million where the upper safety limits for concentration atmospheric  $\text{CO}_2$  is marked (McGee, 2007). Currently, the concentration of  $\text{CO}_2$  gas in the atmosphere already reached 400 parts per million. Muthiya *et. al.* (2014) stated that the concentration of carbon dioxide is predicted to rise above 750 parts per million by 2100.

The sources of carbon dioxide emission in the atmosphere come from both natural and human sources. Natural sources include decomposition of dead plants and animals, ocean release and respiration. Meanwhile, human sources come from the combustion of fossil fuel such as coal, natural gas and oil to generate electricity; transportation that uses gasoline and diesel to transport people and goods; and certain industrial processes through

chemical reactions such as cement and the production of metals (EPA, 2015). According to Albo *et. al.*, 2010, about 60% of global carbon dioxide emissions come from power and industrial sectors.

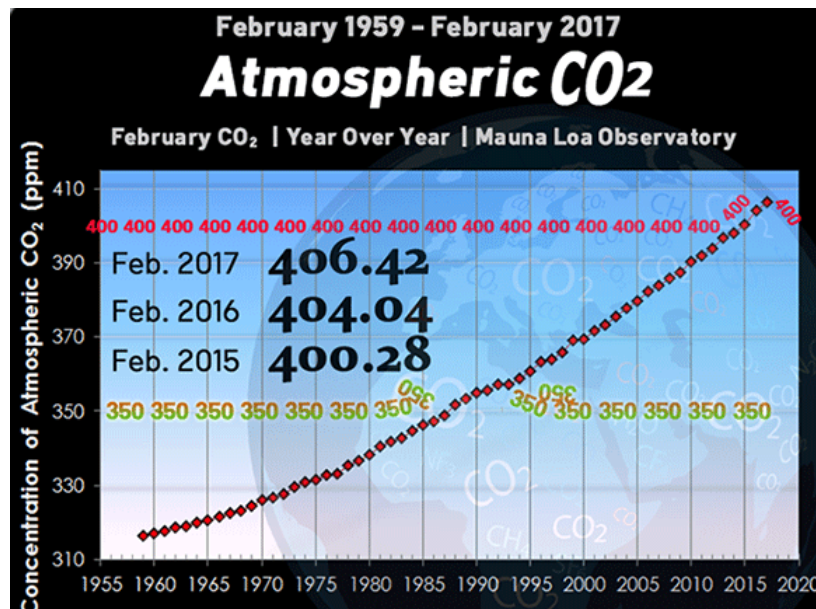


Figure 1.1 Concentration of atmospheric CO<sub>2</sub> February 1959 until February 2017 from Mauna Loa Observatory (McGee, 2007)

The excessive increasing in concentration of carbon dioxide in the atmosphere can give an impact on global climate change if no action is taken to overcome this current situation. The greenhouse effect is an example. The burning of fossil fuels is the largest single source of global greenhouse gas emission. Normally, greenhouse gases such as carbon dioxide, methane, nitrous oxide and water vapour make the earth surface warmer by absorbing and emitting heat energy from sunlight. However, the excessive of greenhouse gases in the atmosphere prevent the heat from escaping into the space. Some of the infrared radiation passes through the atmosphere and some is absorbed and re-emitted in all directions by greenhouse gas molecules due to the longer heat wavelength than the sunlight waves. Greenhouse gases act like a mirror which reflects some of the heat energy back to the earth. Thus, this increases the average temperature of the earth.

Recently, carbon dioxide capture and storage (CCS) becomes an issue in measuring the reduction of CO<sub>2</sub> gas emission substantially and rapidly. CCS is a technology that attempt to prevent the release of large quantities of CO<sub>2</sub> gas into atmosphere from large scale discharges sources such as factories and fossil fuel power plants. CCS securely pumps and stores CO<sub>2</sub> into underground or underwater (Isobe *et al.*, 2013). Another way for reducing carbon dioxide emission from energy to the atmosphere is by cross-cutting fossil fuel consumption. This applies to homes, businesses, industries and transportation. Consequently, cross-cutting fossil fuel consumption can lead to energy efficiency and conservation. This can be done through improving the insulation of buildings when reducing electricity demand and travelling in fuel-efficient vehicles by using fuels with lower carbon contents.

Additionally, other various technologies have been developed in order to reduce the emission of CO<sub>2</sub> gas such as absorption, adsorption and membrane separation. Between them, the adsorption process is widely been used process because of its relatively low operating and capital cost, as well as abundant selection of adsorbents (Zhang *et al.*, 2008; Yu *et al.*, 2013). According to Choi *et al.* (2009), the use of solid adsorbents is widely considered as an alternative technology that offers more energy and cost efficient separation as compared to the commonly used amine based liquid for CO<sub>2</sub> capture from flue gas. Generally, solid adsorbents used for CO<sub>2</sub> adsorption have high surface area. They can stands thousands of adsorption and desorption cycles (Hedin *et. al.*, 2013). Examples of solid adsorbents used are activated carbon, activated alumina, silica gel and molecular sieves such as zeolites and meso-porous silica.

Isobe *et al.* (2013) states that models for gas separation by using porous ceramic filter have long been proposed and the filters with micropores that separate gases by molecular sieve and surface diffusion are mainly adopted as research targets. Ceramics are