

EFFECT OF COMPOSITION AND SINTERING TEMPERATURE ON  
SILICA-NICKEL OXIDE FOAMS BY SLURRY METHOD

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*“Special dedicated with much love and affection to my beloved father Baharom bin Mohammed, my beloved mother Rokiah binti Baharom, all my brothers, my supervisor Prof Madya Dr Sufizar binti Ahmad, my co-supervisor Prof Madya Dr Hariati binti Taib and also to all my fellow friends who always helped me and gave me an encouragement to complete my study in Degree Master of Mechanical Engineering.”*



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## ABSTRACT

It has been a long-standing effort to create materials with low density but high strength. Foam materials are very light, but compared with bulk materials, their strength is quite low because of their random structures. Natural lightweight materials such as bone, is a cellular solid with optimized structure. The aim of this study is to fabricate silica-nickel oxide (SiO<sub>2</sub>-NiO) foams with 20 μm to 800 μm open pore size, 50% to 80% of porosity, good physical and mechanical properties. In this research, silica (SiO<sub>2</sub>) powder and nickel oxide (NiO) powder with different compositions of 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.% and 10 wt.% were mixed together with binders which were polyethylene glycol (PEG) and carboxymethyl cellulose (CMC) to prepare slurry solution for impregnation of polyurethane (PU) sponge as the template. Silica-Nickel Oxide foams were fabricated by slurry method and sintered at 1000°C, 1100°C, 1200°C and 1300°C. Characterisation of SiO<sub>2</sub>-NiO foams included morphological analysis, porosity and density test, and compression test were done to determine the foam microstructure, apparent porosity, bulk density and compressive strength. The morphology of SiO<sub>2</sub>-NiO foam showed open pore with size ranging from 20 μm to 739 μm, interconnected cells by strut and close pore. The result of apparent porosity and bulk density of SiO<sub>2</sub>-NiO foams calculated according to Archimedes' principle. The lowest result for apparent porosity obtained was 53.03% and the highest was 66.21%, while the lowest and highest value for bulk density were 0.89 g/cm<sup>3</sup> and 1.22 g/cm<sup>3</sup>, respectively. The result for compressive strength of SiO<sub>2</sub>-NiO foam was within the range of 0.21 MPa to 1.86 MPa. The foam with good physical and mechanical properties obtained by 6 wt.% of SiO<sub>2</sub>-NiO foams sintered at 1300°C and will be used for further research in steam methane reforming application.



## ABSTRAK

Pelbagai usaha telah dilakukan untuk menghasilkan bahan yang mempunyai ketumpatan yang rendah dengan kekuatan yang tinggi. Bahan berbusa merupakan bahan yang sangat ringan, tetapi jika dibandingkan dengan bahan pukal, kekuatan bahan berbusa agak rendah kerana struktur rawaknya. Bahan semulajadi seperti tulang merupakan pepejal selular dengan struktur yang dioptimumkan. Tujuan penyelidikan ini adalah untuk menghasilkan silika-nikel oksida ( $\text{SiO}_2\text{-NiO}$ ) berbusa dengan liang bersaiz  $20\ \mu\text{m}$  hingga  $800\ \mu\text{m}$ , 50% hingga 80% jumlah keliangan, dengan sifat-sifat fizikal dan mekanikal yang baik. Dalam penyelidikan ini, silika ( $\text{SiO}_2$ ) dan nikel oksida ( $\text{NiO}$ ) dengan komposisi berbeza yang terdiri daripada 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.% dan 10 wt.% dicampurkan dengan pengikat iaitu politena glikol (PEG) dan karbosimetil selulosa (CMC) untuk menyediakan buburan bagi proses perendaman span poliuretena (PU) yang bertindak sebagai pemegang aram. Silika-Nikel Oksida berbusa dihasilkan melalui kaedah buburan dan disinter pada suhu  $1000^\circ\text{C}$ ,  $1100^\circ\text{C}$  dan  $1300^\circ\text{C}$   $1200^\circ\text{C}$ . Pencirian  $\text{SiO}_2\text{-NiO}$  berbusa termasuk analisis morfologi, ujian keliangan dan ketumpatan, dan ujian mampatan telah dilakukan untuk mengetahui mikrostruktur, keliangan terbuka, ketumpatan pukal dan kekuatan mampatan. Morfologi  $\text{SiO}_2\text{-NiO}$  berbusa menunjukkan liang terbuka dengan saiz dalam lingkungan  $20\ \mu\text{m}$  hingga  $739\ \mu\text{m}$ , saling dihubung oleh sangga dan liang tertutup. Hasil keliangan terbuka dan ketumpatan menyeluruh untuk  $\text{SiO}_2\text{-NiO}$  berbusa telah dikira berdasarkan prinsip Archimedes'. Jumlah keliangan terbuka paling rendah ialah 53.03% dan paling tinggi ialah 66.21%, manakala nilai paling rendah bagi ketumpatan pukal ialah  $0.89\ \text{g/cm}^3$  dan paling tinggi ialah  $1.22\ \text{g/cm}^3$ . Hasil kekuatan mampatan untuk  $\text{SiO}_2\text{-NiO}$  berbusa ialah dalam lingkungan 0.21 MPa hingga 1.86 MPa. Ciri-ciri fizikal dan mekanikal berbusa yang paling baik diperolehi oleh 6 wt.%  $\text{SiO}_2\text{-NiO}$  berbusa yang telah dibakar pada suhu  $1300^\circ\text{C}$  dan akan digunakan dalam kajian perubahan gas metana menggunakan wap.

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**LIST OF ABBREVIATIONS**

SiO <sub>2</sub>	-	Silica
NiO	-	Nickel oxide
SiO <sub>2</sub> -NiO	-	Silica-Nickel oxide
CMC	-	Carboxymethyl cellulose
PEG	-	Polyethylene glycol
PU	-	Polyurethane
Al <sub>2</sub> O <sub>3</sub>	-	Aluminium oxide
Fe <sub>2</sub> O <sub>3</sub>	-	Iron oxide
Na <sub>2</sub> O	-	Sodium oxide
K <sub>2</sub> O	-	Potassium oxide
CaO	-	Calcium oxide
MgO	-	Magnesium oxide
P <sub>2</sub> O <sub>5</sub>	-	Phosphorus oxide
SO <sub>3</sub>	-	Sulphur oxide
H	-	Hydrogen
H <sub>2</sub>	-	Hydrogen gas
CH <sub>4</sub>	-	Methane
TGA	-	Thermal Gravimetric Analysis
XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscopy
EDS	-	Electron Dispersive X-ray Spectroscopy
XRF	-	X-Ray Fluorescence
XRD	-	Energy Dispersive Spectroscopy
UTM	-	Universal Testing Machine
MPa	-	Mega pascal
µm	-	Micrometer

nm	-	Nanometer
mm	-	Milimeter
wt. %	-	Weight percentage
$\sigma$	-	Compressive strength
$^{\circ}\text{C}$	-	Degree Celsius
$\text{g}/\text{cm}^3$	-	Gram/centimetre cubic (Density)
$W_d$	-	Weight of dry sample
$W_s$	-	Weight of the sample in water
$W_w$	-	Weight of the sample in the air after water fill up the pores



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**LIST OF APPENDICES**

<b>APPENDIX</b>	<b>TITLE</b>
A1	Morphology of polyurethane template
B1	Morphology of silica foam
C1	Element composition for silica powder
D1	Thermogravimetric analysis
E1	Proceeding and Journal Paper



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# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

Porous materials show a dramatic development nowadays instead of solid materials which have been used for the fabrication of variety applications. The applications include catalysis, filtration, thermal insulation, impact-absorbing structures, high specific strength materials, performs for metal-ceramic composites, biomedical implants and high-efficiency combustion burners (Dhara and Bhargava, 2003). For ceramic material, it offers higher temperature and environmental stability in comparison to conventional polymers or metals, and typical materials used for manufacturing cellular ceramics including crystalline inorganic materials such as silicon carbide, alumina, zirconia, cordierite, mullite, and also silicate glass and carbon as well as concrete (Colombo, 2006).

Cellular ceramics constitute a specific class of materials containing a high level of porosity (greater than 60 vol%) which are characterized by the presence of a recognizable 'cell', that is an enclosed empty space possessing faces and solid edges. The faces can either be fully solid or void, giving a closed cell or an open cell material, respectively (Colombo, 2006). Silica foam comes with close, fully open or partial interconnected porosity that can be produced from a broad range of ceramic materials in both oxide and non-oxide. Besides, it consists of open and closed cell that is commonly used for thermal insulation, fire protection, molten metal filtration, diesel engine and hot gas filtration. Most applications of porous microspheres are based on the porous structure such as porosities, pore sizes, and surface areas.



Silica foams are typically porous materials with the volume fraction of porosity ranging from 70% to 90%, and density varying from 0.3 to 0.6 g/cm<sup>3</sup>. Porosity plays an important role in determining the capacity of efficiency and release kinetics (Cai *et al.*, 2013). Porous ceramics offer interesting properties in comparison with dense ceramics such as permeability to fluids, high specific surface area, low density, low thermal conductivity, thermal stability, high corrosion and wear resistance (Sciamanna *et al.*, 2015). These properties depend on the composition and the microstructure of the porous ceramic. The microstructure features are highly influenced by the processing route used such as slurry method, compaction method, gel-casting and a few more that has been widely used in order to produce the porous material.

There are several methods used for the preparation of cellular ceramics such as foams, honeycomb structures and interconnected rods, fibres and hollow spheres as reviewed by Colombo (2006). The examples of fabrication method are replication of a sacrificial foam template or also known as slurry method, direct foaming and burn-out of fugitive pore formers. There are also other fabrication methods in order to produce porous silica foam such as gel-casting, powder metallurgy, freeze casting, gas entrapment and few more. Among those various type of fabrication methods, slurry method has been chosen to fabricate porous silica foam product. Slurry method has been founded and patented by Schwartzwalder and Somer (1963) state that cellular ceramic articles are produced in accordance with the present invention by immersing an open-celled porous element of synthetic polymer or natural organic material in a slurry of finely-divided ceramic powder plus ceramic binder so as to uniformly coat the inner cell defining walls of the element with a thin layer of the slurry. About 1 to 5 wt.% of binder composition is adding to the ceramic slurry to enhance the compressive strength of ceramic foam up to 1 to 2 MPa (Han *et al.*, 2002).

Ceramics with engineered porosity are promising materials for a number of functional and structural applications such as thermal insulation, filters, catalyst support, bio-scaffolds for tissue engineering, and performs for composite fabrication that has been enlightened by Hammel *et al.* (2014). In this research study, porous structure of SiO<sub>2</sub>-NiO foam will be used as a catalyst support in steam methane reforming technology.

Steam methane reforming technology is important to produce hydrogen gas ( $H_2$ ) which is the simplest and the most plentiful gas in the universe (Amin *et al.*, 2010). Reforming reactions of methane, which is the main component of natural gas, plays an important role in the production of synthesis gas and hydrogen from natural gas (Li *et al.*, 2011). The demand for hydrogen gas ( $H_2$ ) grows as it is widely used as raw material in chemical industry, food processing, hydrogenation process, production of ammonia and methanol, in the Fischer-Tropsch synthesis and in the pharmaceutical industry (Alves *et al.*, 2013). It has been investigated in biomass pyrolysis that nickel (Ni) is one of the best metal catalysts for tar elimination because it catalyses the cleavage of C-C, O-H, and C-H bonds, as well as the water-gas reaction (Widyaningrum *et al.*, 2012).

It is necessary to identify this slurry method which promises a foam with the morphology consists of 50% to 80% of porosity, open pore size within 20  $\mu\text{m}$  to 800  $\mu\text{m}$  and interconnected microstructure. The structure of foam which consists of large number of open pores and sinter at high temperature increase the reforming reaction compare to the material with dense structure. Materials containing high porosity exhibit special properties such as permeability to fluid, high specific surface area and low density that usually cannot be achieved by the conventional dense counterparts (Sciamanna *et al.*, 2015). It is also compulsory to fabricate  $\text{SiO}_2$ -NiO foams with good morphology, physical and mechanical properties to withstand stress during assembling and long-term operation, as they have to guarantee an adequate operation lifetime instead of reducing the production cost.

## 1.2 Problem Statement

Morphology of ceramic foam is a study to observe the microstructure which consists of open pore, closed pore and interconnected cells by strut. The size of open pore obtains from replication method within the range of 1  $\mu\text{m}$  up to 2 mm which cannot be obtained by other method. The variation of strut sizes which connect the pores increase the strength of the foam. Ceramic foam with high porosity approximately 90%, low bulk density under 1.0  $\text{g}/\text{cm}^3$  and good compressive strength is fabricated by selecting the proper composition of raw materials to prepare the slurry solution for

replication process. This is because the quality of ceramic coating on the polymeric sponge is highly dependent on the viscosity of the slurry instead of density of polymeric sponge (Nor *et al.*, 2009). Proper selection of SiO<sub>2</sub> powder, NiO powder and binders will determine the slurry concentration and affect final properties of sintered foam which includes the morphology of the foam.

Apart of selecting the composition of raw material, processing temperature is also one of the challenges for material's scientists to select the suitable temperature for foam fabrication. A research on several sintering temperatures are compulsory to obtain a porous SiO<sub>2</sub>-NiO foams with morphology which consisted of 50% to 80% of porosity, and also has good mechanical and physical properties. In firing stage, the green body undergoes numerous endothermic and exothermic processes, including organic removal like binder or space holder, dehydration, decomposition and phase formation (Gupta *et al.*, 2010). The common problem occurs in the production of porous ceramic product is associate with low strength. Defects can be minimized and the sintered body would not crack with proper heating and atmosphere control such as low heating and cooling rate which is 2°C/min for SiO<sub>2</sub>-NiO foams fabrication.

There are several factors such as material composition, size of raw material, fabrication method, type of binder and its distribution that affect the morphology, physical and mechanical properties of porous ceramic foam. Porosity is one of the crucial factors that controlling the performance, properties, strength, and density of the ceramic foam. Materials containing 50% up to 80% of porosity is preferable compared to dense ceramics because it offers interesting properties such as permeability to fluids, low density, high corrosion and wear resistance. It is suitable for steam methane reforming application because the foam with open pore structure let the methane gasses pass through the foam easily for gas conversion process. The conversion of gas from methane (CH<sub>4</sub>) to hydrogen gas (H<sub>2</sub>) occurs at temperature of 400°C to 900°C (Amin *et al.*, 2011).

### 1.3 Research Objectives

The objectives below are the solution to the problem statements in the fabrication of porous materials. The objectives of this research are:

- 1) To investigate the composition of SiO<sub>2</sub> powder based on the SiO<sub>2</sub> foams morphology to fabricate SiO<sub>2</sub>-NiO foams.
- 2) To identify the suitable composition and sintering temperature for SiO<sub>2</sub>-NiO foams fabrication process.
- 3) To characterise physical properties and mechanical properties of SiO<sub>2</sub>-NiO foams after sintering process.

### 1.4 Scope of Study

Several scopes and criteria have been studied and determined to carry out laboratory works for foam fabrication. The scope in this research study can be divided into several points which are material usage, fabrication and finally the characterisation of SiO<sub>2</sub> foams and SiO<sub>2</sub>-NiO foams. The scopes of this research study are summarized as below:

- i. Raw materials used in this study are SiO<sub>2</sub> powder, NiO powder, polyurethane (PU) sponge as the template, polyethylene glycol (PEG) and carboxymethyl cellulose (CMC) as binder and distilled water.
- ii. Various compositions of SiO<sub>2</sub> foams from 45 wt.%, 50 wt.%, 55 wt.%, 60 wt.% and 65 wt.% are studied with 2.5 wt.% of CMC and 2.5 wt.% PEG fabricated by slurry method.
- iii. Method to fabricate SiO<sub>2</sub>-NiO foams is slurry method with 55 wt.% of SiO<sub>2</sub> and variation of NiO compositions consisting of 2 wt.%, 4 wt.%, 6 wt.%, 8 wt.% and 10 wt.%. The composition for binders which are PEG and CMC is 2.5 wt.%, respectively.
- iv. Two stages of sintering profile are set up with sintering temperatures of 1000°C, 1100°C, 1200°C, and 1300°C.

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