FABRICATION SiO_2-NiO FOAM *via* REPLICATION TECHNIQUE FOR STEAM METHANE REFORMING CATALYST AT LOW TEMPERATURES

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UNIVERSITI TUN HUSSEIN ONN MALAYSIA

STATUS CONFIRMATION FOR THESIS DOCTOR OF PHILOSOPHY

FABRICATION SiO_2-NiO FOAM *via* REPLICATION TECHNIQUE FOR STEAM METHANE REFORMING CATALYST AT LOW TEMPERATURES

ACADEMIC SESSION: 2021/2022

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A thesis submitted in
fulfillment of the requirement for the award of the
Doctor of Philosophy in Mechanical Engineering

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MARCH 2021

I hereby declare that the work in this thesis is my own except for quotations and summaries which have been duly acknowledged

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To my precious Allah SWT,who gave me a new life, hope and purpose of life.

To my beloved Prophet Muhammad S.A.W who gave me guidance thru his sunnah and hadis.

To Abah, Mak and my in-laws, who giving all they have.

To my beloved husband, Mohd Firdaus bin Zulkifli, who supported me each step of the way,

And to my beloved daughters, Nur Raisha Sofia and Nur Rania Sakina who are my inspiration and motivation in completing this thesis

ACKNOWLEDGEMENT

Firstly, I thank Allah SWT for his grace and strength given to me so that this research could be made possible. Secondly, I would like to extend my gratitude to my supervisor, Associate Prof Dr Hariati Taib and co-supervisor, Associate Prof Dr Sufizar Ahmad and Associate Prof Dr Mas Fawzi Mohd Ali for their continuous understanding, encouragement and trust in me throughout the entire research. I am grateful for the endless construction comments and careful review of all the report.

I would also like to thank the following, who have helped me in various ways:

- Ministry of higher education Malaysia for research financial support under a FRGS Grant (1593).Moreover, for the MyBrain15 Scholarship (MyPhD) program for my studies scholarships.
- University Tun Hussein Onn Malaysia especially Research Management Centre (RMC) for their financial support for paper publication and conference fees.
- Faculty of Mechanical and Manufacturing Engineering and material engineering and design department members, for their understanding and the facilities support in accomplishing this resear

ABSTRACT

Porous ceramic is a type of material that has highly open and partially interconnected pores. It has a wide range of applications, which include catalyst support, filtration, adsorption and separation.The aim of this study is to fabricate Silica-Nickel Oxide (SiO_2-NiO) foams in the range 70 µm to 150µm open pore size, 75% to 90% of porosity, good physical and mechanical properties as a criteria catalyst in the Steam Methane Reforming (SMR) application. In this work, the porous foam fabricated with different compositions of SiO_2 as derived from Rice Husk Ash (RHA) (20% to 35%) and at different sintering temperatures (850°C to 1250°C) by using replication sponge method. Characterisation of SiO_2 and SiO_2-NiO foams included morphological analysis, porosity and density test, and compression test as criteria compatibility of SiO_2 and NiO as a catalyst in methane reforming. The morphology result showed open pores with size ranging from 15.13 µm to 76.06 µm. The lowest result for apparent porosity obtained was 65% and the highest was 81.74%, while the lowest and highest values for bulk density were 0.626 g/cm^3 and 1.070 g/cm^3, respectively. The result for compressive strength was within the range of 0.06 MPa to 0.47 MPa. Throughout the observations, the maximum performance shown the SiO_2-NiO foam produced with 35wt% SiO_2 and 5wt% NiO was found to have mechanical and physical properties much like those of a filter catalyst in SMR. The methane (CH_4) conversion using the SiO_2-NiO foam was shown the range of 34.72% to 42.6% at different low temperatures. The results proved that the foam from silica as derived from RHA and NiO is very suitable to be used as a catalyst in SMR due to achieved the minimum CH_4 conversion over than 21%.

ABSTRAK

Seramik berliang adalah sejenis bahan yang mempunyai liang yang sangat terbuka dan saling bergabung antara satu sama lain. Ia mempunyai pelbagai aplikasi termasuklah sebagai sokongan pemangkin, penapisan, penjerapan, dan pemisahan. Tujuan kajian ini dilakukan adalah untuk menghasilkan busa Silika-Nikel oksida dengan saiz busa antara 70µm hingga 150µm , keliangan 77% hingga 90% sebagai kriteria pemangkin di dalam pembaharuan wap methana (SMR). Kajian ini, busa SiO_2 dan busa SiO_2-NiO dihasilkan menggunakan teknik busa replikasi. Dalam kajian ini, busa SiO_2 dan busa SiO_2-NiO dihasilkan menggunakan teknik replikasi busa. Sampel yang dihasilkan juga menggunakan komposisi serbuk SiO_2 yang berbeza hasil daripada abu sekam padi (RHA) (20% hingga 35%) dan suhu sinter antara 850°C hingga 1250°C. Pencirian terhadap busa SiO_2 dan SiO_2-NiO telah dilakukan antaranya adalah ujian morfologi , keliangan , ketumpatan dan kekuatan busa bagi memenuhi kriteria sebagai pemangkin di dalam SMR. Morfologi menunjukkan saiz liang yang terbuka antara 15.13µm sehingga 76.06 µm dengan sangga bersambung dan liang yang rapat. Keputusan keliangan menunjukkan nilai paling rendah ialah 65% dan tertinggi adalah 81.74% manakala nilai ketumpatan terendah dan tertinggi masing-masing adalah 0.626 g/cm^3 dan 1.070 g/cm^3. Justeru itu, keputusan bagi kekuatan mampatan adalah dalam julat 0.06 MPa hingga 0.47MPa. Hasilnya, busa SiO_2-NiO (35 wt% SiO_2 dengan 5 wt% NiO) yang dihasilkan didapati mempunyai sifat mekanik dan fizikal yang lebih sesuai untuk pemangkin penapis dalam pembentukan metana wap (SMR). Keputusan penukaran metana (CH_4) menunjukkan busa SiO_2-NiO menghasilkan antara julat 34.72% hingga 42.6% dan hasilnya membuktikan bahawa busa silika daripada abu sekam padi dan NiO amat sesuai digunakan sebagai pemangkin di dalam aplikasi SMR dimana pencapaian CH_4 melebihi daripada 21%

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LIST OF SYMBOLS AND ABBREVIATIONS

SiO_2	- Silica/ silicon dioxide
RHA	- Rice Husk Ash
Kg	- kilogram
kg/m^3	- kilogram per cubic metre
K_2O	- Potassium oxide
Al_2O_3	- Aluminium oxide
CaO	- Calcium oxide
MgO	- Magnesium oxide
Na_2O	- Sodium oxide
Fe_2O_3	- Iron (III) oxide
g/cm^3	- gram per cubic centimetre
Ni	- Nickel
SMR	- Steam Methane Reforming
hr	- hour
mm	- millimetre
NiO	- Nickel Oxide
PVA	- Polyvinyl alcohol
SEM	- Scanning electron microscope
XRD	- X-Ray Diffraction
GC	- Gas Chromatography
POX	- Partial oxidation
CH_4	- Methane
H_2	- Hydrogen
CO	- Carbon monoxide
MgO	- Magnesium Oxide
nm	- Nanometer

NiO/ZnO.	- Nickel oxide/Zinc oxide
µm	- micrometer
PU	- Polyurethane
TGA	- Thermal gravity analysis
MPa	- Megapascal
°C/min	- Celsius per minute
kN	- Kilonewton
ASTM	- *American Society for Testing and Materials*
Si_3N_4	- Silicon nitride
SS 316L	- 316L stainless steel
Al	- Aluminum
Mg	- Magnesium
Ni_2SiO_4	- Nickel silicate
2Θ	-2 theta
FESEM	- Field Emission Scanning Electron Microscope
JCPDS	- The Joint Committee on Powder Diffraction Standards
rpm	- revolution per minute
g	- gram
XRF	- X-Ray Fluorescence
ml	- Milliliter
TPR	- Temperature Programmed Reduction

CHAPTER 1

INTRODUCTION

1.1 Background of the study

The Green Technology Master Plan (GTMP) is an outcome of the Eleventh Malaysia Plan (2016–2020), which has earmarked green growth as one of six game-changers to alter the trajectory of the nation's growth. The National Green Technology Policy is built on four pillars, which are Energy, Environment, Economy and Social. Green technology is aimed to be the key driver in accelerating the national economy and promoting sustainable development in Malaysia (Ministry of Energy, Green Technology and Water, 2017). There have been numerous innovations of technology being utilised in daily life. Examples of technologies that are widely used are cars, buses and motorcycles in the field transportation and electrical insulators and tiles in buildings and the aerospace industry, as well as innovations in the medical field. Among the most significant innovations are the materials used to build these products, such as metals, ceramics, plastics, polymers and many other materials. Different materials have their own properties and specialities to make them better. In this research study, ceramic is the primary material chosen instead of metal for the steam methane reforming applications.

In the fabrication of ceramic foams, a majority of the materials used are costly and not sustainable. In this study, in order to address this issue, the raw materials used were Silica (SiO_2) derived from Rice Husk Ash (RHA) and Nickel Oxide (NiO). Rice husks are among the variety of agricultural wastes or biomasses available. In addition, silica from rice husk ash is a renewable resource and therefore generates low production costs for the foams (Hossain *et al.*, 2018).

Rice Husk Ash is an agricultural waste material that are produced approximately 100 million tons annually. Approximately 20 kg of rice husks could be produced from 100 kg of rice. RHA has been widely used in the construction industry owing to its high availability, low bulk density (90–150 kg/m^3), durability, abrasive character, weather resistance and unique composition (Taufik *et al*., 2013)

Rice Husk Ash contains 75%–90% organic matters, such as cellulose and lignin, and the rest are mineral components such as silica, alkalis and trace elements. Other constituents of RHA, such as K_2O, Al_2O_3, CaO, MgO, Na_2O, Fe_2O_3, are available at less than 1%. Moreover, RHA has a bulk density of 96–160 kg/m^3 and consists of 31%–37% oxygen, 0.2%–0.32% nitrogen and 0.04%–0.08% sulphur (Kumar *et al*., 2013). The main components in RHA are silica, cellulose and lignin.

Silicon dioxide or silica (SiO_2) is an oxide of the silicon element, which is the second-most abundant element found on earth. Silica has been identified in some way in all natural water resources. In addition, many plants, such as strawberries, avocados, onions, root vegetables, wheat and oats, contain silica and it can also be found in nature as sand, sandstone, quartz, flint, agate or granite (Hossain *et al*., 2018). Silica has three phases, which are amorphous, crystalline and tridymite. Most silicas exist in the amorphous form, while quartz is the purest form of silica. Other than that, sand and rock are natural sources of silica that are less refined. The three most common polymorphs of crystalline silica are quartz, tridymite and cristobalite (Carcinogen, 2006). Silica-based materials have attracted considerable attention because of their high surface area (>200 m^{2}g^{-1}), ordered pore distribution, narrow pore size, high thermal stability and natural regeneration and reusability in comparison with several soils and soil components. Due to these properties, they are ideal base materials as catalysts, catalyst supports and adsorbents, and as a template for other materials. Silica is attractive as a catalyst support since it has strong structural robustness, is stable even at elevated temperature and is chemically inert (Majewski *et al*., 2013). Furthermore, a combination of SiO_2 with NiO is a promising solution as a catalyst in the steam methane reforming industry (Bej *et al*., 2013).

In ceramic applications, silica foams typically show fully open pores, closed pores and partially interconnected porosity. Most applications of porous microspheres centre on the porous structure, such as porosity, pore size and surface area. Silica foams typically have 70% to 90% porosity, with density varying from 0.4 to 0.7 g/cm^3 (Nazaruddin *et al.,* 2017)

Many methods are used in the manufacturing of ceramic foam, such as the replication method, also known as the slurry process, as well as a direct foaming. Among these various types of fabrication method, the slurry method has been chosen to fabricate the ceramic foam-like silica foam. The slurry method was developed by Schwartzwalder and Somer (1963), and presently the ceramic fabrication is by using an open-cell porous product of a synthetic polymer or a natural organic material in a slurry, which is divided into ceramic powder and ceramic binders, to uniformly coat the inner cells forming the walls of an element. The binder composition usually used is about 1% to 5% in order to enhance the compressive strength of the ceramic foam up to 1 to 2 MPa (Han *et al*., 2002).

For the catalyst process, it is necessary to have high mass, a high heat transfer rate and a wide contact surface in order to achieve excellent catalytic reaction performance. Nickel (Ni) has emerged as a new metal with catalytic power towards oxygen evolution, and great efforts have been put to develop Ni-based catalysts, including oxides, hydroxides, sulphides and nitrides in alkaline solution (Liang *et al*., 2015). A Ni-based catalyst is often considered as a catalyst for steam reforming due to its high tar destruction activity. It is evident that the selection of the support material is considered as one of the particularly crucial issues in tar steam reforming for hydrogen production. The most commonly used supports are minerals and alumina or modified alumina (Gao *et al*., 2015) which the high cost of material.

Nickel is considered to be one of the best non-noble catalyst materials for hydrogen evolution reaction (HER). This is mainly due to the fact that Ni is primarily resistant to corrosion at high pH values and also due to the particular importance of nickel's structure having a large specific surface area (large roughness) (Pierozynski *et al*., 2014). Ni has comparable activities to noble-metal catalysts, and that a higher loading amount of Ni is feasible in terms of catalyst cost in order to increase the activity per catalyst volume. Although Ni is inferior to noble metals in regards to the resistance to carbon deposition and catalyst oxidation, the high resistance to coke formation and catalyst oxidation can be realised by the addition of minimal amounts of noble metals (Wu *et al*., 2013).

In this study, nickel oxide was preferred as the silica foam support because of the lower price as it is derived from the RHA foam. This would lead to the high ability of the silica itself in the absorption of substances and to obtaining good strength and hardness properties in the ceramic products because firing rice husks at high temperature generates high strength (Zainal *et al*., 2019). As the field of technical ceramics continues to expand, the use of ceramic materials as catalyst support is opening several opportunities for the review of applications, preparation and stability of porous ceramic materials. Ceramic foams have been used as catalyst supports in the areas of ammonia oxidation, catalytic combustion, partial oxidation ,steam reforming and exhaust catalysis. More works have since appeared on foam-supported catalysts for methane or propane combustions. Steam reforming of methane from biogas at a small scale could potentially provide a source of hydrogen for applications such as electricity generation via fuel cells. The efficiency of the reforming process is dependent upon an active catalyst, and thus this present work aimed to produce a highly active catalyst for methane reforming that is resistant to deactivation (Majewski *et al*., 2013).

Furthermore, steam methane reforming (SMR) can be catalysed by several metal catalysts. For instance, cobalt, platinum, palladium, iridium, ruthenium, rhodium and nickel have all been cited as usable for this purpose. Even in the light of this, as one of the relatively lower-activity metals, nickel has become the industry-standard catalytic metal used in steam methane reforming because of its relatively low cost, wide availability and sufficient catalytic activity, all combining to make it the most cost-efficient choice (Meloni *et al*., 2020).

This study focuses on the compatibility of RHA-derived silica with the addition of nickel oxide for the fabrication of ceramic foams as the steam methane reforming catalyst.

1.2 Problem Statement

Rice husks (RH) are bulky agricultural wastes with an annual world production of approximately 100 million tons. Most of the husks produced from rice processing are currently either burned or dumped as waste thus causing damage to the land and environmental pollution (Azmi *et al*., 2016). Besides that, it has been reported that at 600 to 1000°C, amorphous silica is formed . Therefore,to solve energy problem, the burning temperature at 450°C was used in producing of silica as a catalyst.

Furthermore, in catalyst, most of researchers used a metal as a catalyst carrier. Mostly a metal are limited high cost and rusty when reaction with the steam (Wang *et.al*., 2020). The improvement, this study are used the ceramic material (SiO_2) due to low cost and rust resistant.

In previous studies, there are several factors, such as the type of raw material, material composition, size of the raw material, fabrication method, type of binder and binder distribution, that affect the morphological, physical and mechanical properties of porous ceramic foams (Baharom, 2014). However, choosing the appropriate manufacturing process is very important since the pore distribution, size, shape and volume porosity of the porous material produced depends on the type of fabrication method used. It is known that, the pore structure of the cancellous bone is open and interconnected. Therefore, fabrication methods that can produce such open and interconnected pore structure need to be identified. Some of researchers used the compaction method to fabricate ceramic foams. This method consists of five major steps: powder selection, mixing, compacting, sintering and spacer removal (Ahmad & Kassim, 2011). However, this method has some problems, such as the pressing speed and the releasing speed of the punch usually leading to the crack formation of the compacted parts. Too high of a punch speed will result in a higher density at the contacting surface, which is susceptible to cracks. Meanwhile, too high of a releasing speed will discharge the internal pressure too quickly and lead to cracks (Baharom, 2014). Foam replication is a very economical powder metallurgical method for producing porous materials with open and interconnected pores, and also with a unique combination of properties that is suitable for various applications. It is possible to fully transform the open and interconnected pores of PU foam into open and interconnected pores of the porous ceramic.To date, there has been limited comprehensive research that studies the parameters involved in each processing step for the fabrication of porous SiO_2 and NiO with open and interconnected pores using foam replication methods especially for applications as catalyst in SMR.

In addition, one of the challenges in selecting the appropriate temperature for foam manufacturing is to avoid fabricating porous ceramics with low strength. Hence, the current study can be reduced using adequate heating and regulation of the environment, such as a low heating and cooling rate of 2°C/min, which would result in a sintered body that does not crack. Beside that, the temperature used in fabrication of ceramic foam required the high temperature as 1450°C and its should be more energy to handle it. So, to reduce of the high energy the low temperature below to 1250°C were choose in this research.

The catalytic process affects the chemical reaction and production of the synthesis gas (hydrogen). Common noble-metal catalysts used include ruthenium, rhodium and platinum. Noble metal sources are limited, expensive and resistant to chemical reactions (Wang *et al*., 2020). A solution to the noble metal problem is using another type of catalyst, which is the nickel-based catalyst. This low-cost catalyst has a high propensity to carbon deposition through the steam methane reforming (SMR) cycle (Zhang *et al*., 2015).

1.3 Objectives

The objectives of this study are as follows:

1) To elucidate the suitable composition of SiO_2 and NiO for the SiO_2-NiO foam fabrication.
2) To examine the physical and mechanical properties of the SiO_2 and SiO_2-NiO foams fabricated with different compositions and at different sintering temperatures.
3) To examine the capability of the SiO_2 and SiO_2-NiO foams as a catalyst in the steam methane reforming process.

1.4 Scope of Study

The scope of the study was designed so that the objectives are met and to avoid deviating the results of the study from the desired objectives. The scope of study is as follows:

1) Producing of SiO_2 as derived from Rice Husk Ash (RHA) at temperature 450°C for 8 hrs with a heating rate of 10°C/min.
2) Composition of the SiO_2-NiO foam:
    a) SiO_2 compositions used were 20 wt%, 25 wt%, 30 wt%, 35 wt%, 40 wt%, 45 wt%, 50 wt% and 55 wt%.
    b) Nickel oxide's compositions: 0 wt%, 5 wt%, 10 wt%, 15 wt%, 20 wt% and 25 wt%.

c) PVA was used as a binder at 5 wt%

3) Phase compatibility studies were conducted on the SiO_2-NiO foam at sintering temperatures of 850°C, 950°C, 1050°C, 1150°C and 1250°C.

4) The characterisations of the raw materials include:
   a) Morphology analysis – Scanning Electron Microscopy (SEM)
   b) Phase analysis – X-Ray Diffraction (XRD)
   c) Elemental analysis – X-Ray Fluorescence (XRF)
   d) Thermal analysis – Thermogravimetric analysis (TGA)

5) The characterisation tests of the SiO_2-NiO ceramic foam include:
   a) Morphology analysis – Scanning Electron Microscopy (SEM)
   b) Phase analysis – X-Ray Diffraction (XRD)
   c) Physical properties such as density, porosity and shrinkage were examined.
   d) A ccompressive test was conducted to identify the strength properties of the SiO_2-NiO foam.

6) Steam methane reforming test of the SiO_2-NiO foam was conducted using Gas Chromatography (GC) to identify the catalytic capability of the SiO_2-NiO foam. The sample used for the catalytic process in SMR was 35wt% SiO_2 with 5wt% NiO and the catalyst reaction temperature are 500,550 and 600°C.

1.5 Novelty and Contribution

This section describes the significant findings and the novelty contributed by this study.

The derivation of SiO_2 from RHA was conducted by burning the rice husks at 450°C. Based on the review of the latest literature, the derivation of SiO_2 are burning at 600°C and most silicas used in the fabrication of ceramic foams are commercial silica .Thus far, the literature on the study regarding SiO_2 from RHA in the fabrication of ceramic foams is limited.

In addition, the combination of RHA-derived SiO_2 and NiO in the fabrication of ceramic foams is yet to be investigated and reported, as confirmed by the literature review to date. Moreover, the properties of a foam using RHA-derived SiO_2 and NiO fabricated via the replica method has never been investigated, as evidenced by the review of previous studies.

In terms of catalyst application, there have been no studies reported in the literature on the analysis of methane conversion using the combination of RHA-derived SiO_2 and NiO and the reaction temperature in SMR at low temperature (500-600°C).

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction to Catalytic Reforming Technologies

Recently, hydrogen has become a promising energy carrier since it has high energy density and generates no pollutants during its combustion, which reduces the impact on the environment significantly. Future applications of hydrogen as an energy carrier will provide effective, clean and recyclable energy, and its combustion releases a remarkable amount of energy per unit weight. Hydrogen can be used in a variety of applications, for example, as fuel for fuel cells and internal combustion engines and as a reactant in the hydrogenation process. In industrial production, the preparation of hydrogen is widely studied, and it is usually produced via steam reforming of traditional fossil resources. Currently, due to the abundant natural gas reserves, methane is widely used in hydrogen production. Among the different production methods of hydrogen, the steam reforming of methane is one of the most widely used (Wang *et al*., 2020). A hydrogen economy has become an interest in the world (Belhadi *et al*., 2016). Steam reforming reactions will play a key role in new applications of synthesis gas and in the future hydrogen economy.

As explained by Madon *et al*. (2018), the world's energy sources are being threatened by the increasing demand for energy consumption. Hydrogen as a potential alternative energy source and energy carrier has been explored to address such needs. This is because hydrogen is widely accepted as the cleanest, efficient and pollution-free energy source. The production of hydrogen from various hydrocarbons, especially methane, mainly comes from the chemical reaction processes of the catalytic reforming technologies, such as steam reforming, partial oxidation and autothermal reforming. Among these, steam methane reforming (SMR) has the advantages of relatively low reaction temperature and high hydrogen content in the reforming products (Madon *et al*., 2016; Wang *et al*., 2013; Matsumura *et al*., 2004). Ttypes of catalytic reforming are graphically illustrated in Figure 2.1.

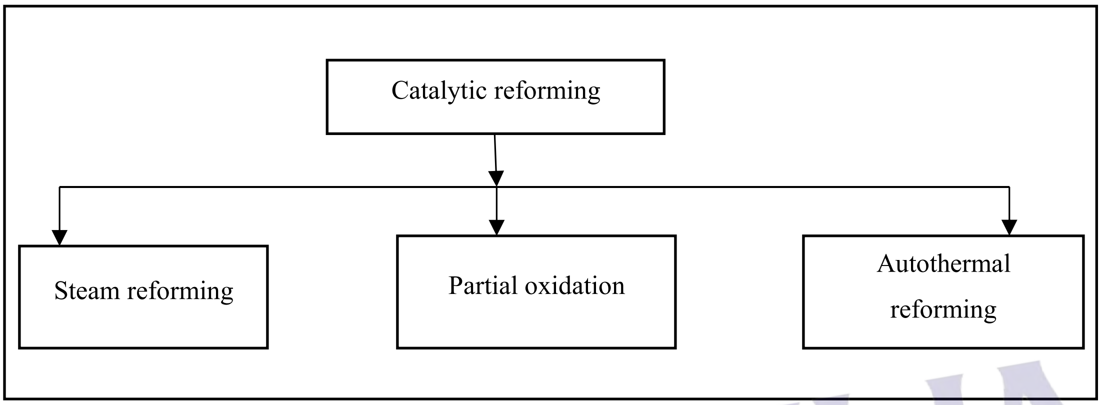

Figure 2.1 :Type of catalytic reforming (Madon *et al*., 2018)

Steam reforming uses an external source of hot gas to heat tubes in which the catalytic reaction takes place, which converts steam and lighter hydrocarbons such as methane, biogas or refinery feedstock into hydrogen (H_2) and carbon monoxide (CO) (syngas). Syngas reacts further to give more hydrogen and carbon dioxide in the reactor.

Meanwhile, autothermal reforming is grouped under partial oxidation, which uses oxygen and carbon dioxide or steam in a reaction with methane to form synthesis gas. The reaction takes place in a single chamber where the methane is partially oxidised. The reaction is exothermic due to the oxidation. The main difference between autothermal reforming and steam methane reforming is that steam methane reforming does not use or require oxygen. The advantage of autothermal reforming is that H_2/CO can be varied, which is particularly useful for producing certain second-generation biofuels such as dimethyl ether synthesis (Speight, 2015). However, it is less efficient in producing hydrogen (Liu, 2006).

Partial oxidation (POX), on the other hand, is a type of chemical reaction. It occurs when a quantity of reactants of fuel-air mixture is partially combusted in a reformer, creating a hydrogen-rich syngas, which can then be put to further use. The oxidation causes the reaction to be exothermic (a chemical reaction that releases energy through light or heat). Catalytic partial oxidation does not involve operating at very elevated temperatures but involves the removal of sulphur, which poisons the catalyst. High-temperature partial oxidation can also manage much heavier oil fractions than the catalytic process and is therefore appealing for the processing of diesel, logistic fuels and remaining fractions. These fuels are removed in large-scale activities, but it is hard to scale and regulate the process. Nickel is one of the most widely used active phases in partial oxidation (Galvan *et al*., 2019). Partial oxidation, however, generates less hydrogen per methane molecule. This implies that partial oxidation is less effective than steam reforming for fuel cell applications.

2.2 Steam Methane Reforming (SMR)

Traditional steam methane reforming plants for hydrogen production not only consume large amounts of natural gas but also emit a lot of greenhouse gases (Li *et al*., 2020). Due to its clean and renewable nature, hydrogen has attracted considerable attention. The technical routes for hydrogen production to date include mainly water electrolysis and steam methane reforming (SMR). SMR has been the preferred technology for the industrial production of synthesis gas from methane to produce ammonia or methanol. This is an important technology pathway for the production of near-term hydrogen. Steam reforming of methane consists of three reversible reactions: the strongly endothermic reforming reactions 1 and 3, and the moderately exothermic water-gas shift (WGS) reaction 2 (Galvan *et al*., 2019).

$$CH_4 + H_2O \leftrightarrows CO + 3H_2 \ldots\ldots\ldots\ldots\ldots \quad (1)$$

$$CO + H_2O \leftrightarrows CO + H_2 \ldots\ldots\ldots\ldots\ldots\ldots \quad (2)$$

$$CH_4 + 2H_2O \leftrightarrows CO2 + 4H_2 \ldots\ldots\ldots\ldots \quad (3)$$

The basic reforming reaction for a generic hydrocarbon methane (CnHm) is as follows:

$$CnH_4 + nH_2O \leftrightarrows nCO + \left(\frac{m}{2} + n\right) H_2$$

Reactions 1 and 3 are reversible and are usually rounded by an active catalyst at high temperatures. The total product gas is a blend of carbon monoxide, carbon dioxide, hydrogen, unconverted methane and vapour. The temperature of the reactor, the working pressure, the structure of the feed gas and the amount of steam supplied to the reactor shall be determined by the product of the regulator. The quantity of carbon monoxide generated by steam reforming methane is quite large because the water gas shift reaction shown in Eq. (2) is thermodynamically favourable at higher temperatures. The amount of carbon monoxide in the final product of the steam methane reforming is determined by the thermodynamics and kinetics of the reaction within the reformer. This also determines the downstream procedures needed to decrease the CO concentration required by the proton-exchange membrane.

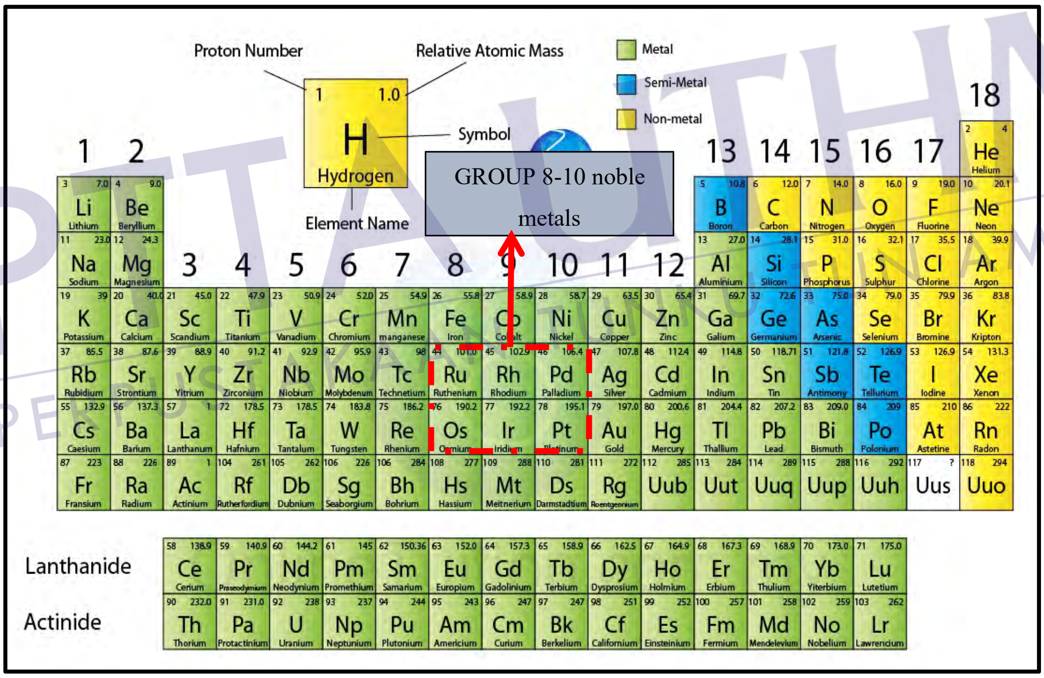

Figure 2.2: Group 8–10 of noble metals (Meloni *et al*., 2020)

Steam reforming is the most significant path for the large-scale production of ammonia, methanol and other petrochemical synthesis gas and for the production of hydrogen for refineries. In particular, the reforming responses are catalyse by Group 8–10 noble metals (Figure 2.2). However, iron is easily oxidizesed, cobalt is unable to withstand the partial steam pressure and precious metals (rhodium, ruthenium, platinum and palladium) are too expensive for industrial use (Meloni *et al*., 2020).

Ni-based catalysts have been intensively studied for methane reforming because of their excellent catalytic performance, wide availability and low cost (Chai *et al*., 2017; Meloni *et al*., 2020). Catalytic steam reforming of methane involves the reaction of methane with steam over a catalyst at elevated temperatures (400°C–900°C) and pressures (1–30 atm) (Bej *et al*., 2013). Ming *et al*. (2002) conducted a 300-h continuous test using a proprietary catalyst for steam reforming of is-octane at 800°C with a steam/carbon ratio of 3:6.

In the past, a great deal of attention has been paid to the preparation of catalysts and the assessment of the process and equipment, with little work being done on the kinetics and mechanism of the reaction. Consequently, there is a lack of kinetic information, and contradictory mechanisms have been suggested. Some early studies, such as by Temkin (1979), have worked on steam methane reforming by investigating the kinetics mainly with Ni catalysts. In the studies, the experiments were performed at temperatures ranging from 470°C–900°C.

The pproduction of synthesis gas from natural gas is one of the important technologies in the chemical industry because synthesis gas can be converted into liquid fuels and chemicals such as methanol and dimethyl ether. Hydrogen is practically produced from natural gas, and the produced hydrogen is mainly used for the ammonia synthesis. In particular, much attention has been paid to the hydrogen production relating to fuel cell technology. The general mechanism for the steam methane reforming is as follows (Beurden, 2004):

a) Water reacts with surface nickel atoms, yielding adsorbed oxygen and gaseous hydrogen.
a) The hydrogen formed is directly released into the gas phase, and/or the gaseous H_2 is in equilibrium with the adsorbed H and H_2.
b) Methane is adsorbed onto the surface nickel atoms. The adsorbed methane either reacts with the adsorbed oxygen or dissociates to form chemisorbed radicals, CH_x with x = 0–3.
c) The adsorbed oxygen and the carbon-containing radicals react to chemisorb formaldehyde (CH_2O), aldehyde (CHO), CO or CO_2.
d) CO and CO_2 are formed out of CHO and CH_2O species.

The production of synthetic gas is very significant in the chemical industry for reasons connected to rising oil prices, the depletion of oil reserves and the environmental issues with exhaust gases. Steam reforming of methane and other hydrocarbons has been an extremely important process to produce synthesis gas. The technology for steam reforming is of great interest because this part of the process represents a substantial portion of the investment costs. The reforming section costs about 60% to 80% of the total cost of the entire gas refining plant. Improvements and cost savings in the reforming section will therefore become very noticeable in the total plant cost (Speight, 2015).

2.3 Catalyst support

The function of the support oxide is not only to control the active metal dispersion but it often also has a significant impact on the catalytic reactions through the metal-supported interactions. A support may play a main role in regulating the size, shape, dispersion and structural stability of metal active locations during the response (Azancot *et al*., 2019). Noble metals, such as rhodium (Rh), ruthenium (Ru), palladium (Pd) and platinum (Pt), have been reported to offer excellent catalytic performance with higher activity, stability and resistance to carbon deposition for steam methane reforming. However, their high cost limits their use. Therefore, more attention has been focused on the utilisation of cheaper transition metal catalysts, such as nickel (Ni), copper (Cu) and iron (Fe) (Boudjeloud *et al*., 2019). Ni-based catalysts as low-cost materials are a promising alternative in reforming reactions since they present at high temperatures relatively good activity and high stability (Fang *et al*., 2016). A typical commercial catalyst uses between 12 wt% to 20 wt% of nickel applied to a refractory ceramic substrate (Dalgleish *et al*., 2007).

A support determines the dispersion of the catalytically active metal particles and the resistance of the catalyst to sintering. In particular, the function of a support is literally to provide surface assistance for the dispersion of active catalytic metal in order to achieve a stable and highly active metal surface. The most common supports for methane reforming are aluminium oxide (Al_2O_3), magnesium oxide (MgO), magnesium aluminate ($MgAl_2O_4$), silica (SiO_2), zirconium dioxide (ZrO_2) and titanium dioxide (TiO_2). These supports have a decent porosity, which allows for a more significant surface area. A support plays a crucial role since it determines the final particle size of the metal, its pore structure, its morphology and the phase transitions that it can undergo. Also, a support can have a chemical role as well, by activating one or more reaction steps.

According to Ozdemir *et al.,* (2010), it was found that Ni/MgO catalyst with 10% Ni content would be the best catalyst and would result in increased CH_4 conversion at temperatures from 500°C to 800°C. Al-Swai *et al*., (2017) investigated the 10wt% Ni-supported MgO catalyst for the dry reforming of methane reaction at different experiment conditions in a fixed bed reactor with reaction temperatures in the range from 700°C to 900°C, and the catalyst afforded as high as 93% CH_4 conversion at 900°C (Al-Swai *et al*., 2017)

Bej *et al*., (2013) reported the alumina-supported nano-NiO catalyst in silica synthesised using the sol-gel method. The NiO crystallite size was found to be 9–15 nm in the Ni loading range of 5%–15%. The catalyst containing 10% Ni was found to be the best for steam reforming reaction in terms of methane conversion. The reaction temperature range was 500°C–700°C and the best reaction condition was established at the temperature of 700°C with 95.7% conversion of methane.

Furthermore, Belhadi *et al.,* (2016) reported the steam reforming of methane performed at 650°C, and the production of hydrogen was observed with the catalyst of 10% NiO/ZnO. The results showed that the catalytic activity in the steam methane reforming was strongly dependent on the percentage of nickel. These results appeared to depend not only on the interaction between nickel and the support but also on the state of the dispersion of nickel on the surface.

2.4 Characteristics and Properties of Porous Ceramic for Catalytic Performance

Porous ceramics, also known as cellular ceramics, was developed in the 1970s. They are made up of a heat-resistant porous material with many gaseous pores. Their pore size ranges mostly between angstrom and millimetre, their porosity normally ranges from 20% to 95% and their production temperature ranges from room temperature to 1600°C. Porous ceramics have several common characteristics (Liu & Chen, 2014):

a) **Good stability**

Choosing the appropriate material species and methods may create porous products suitable for various corrosive circumstances under which the products are supposed to operate.

b) **Great strength and stiffness**

The shape and size of the pores in porous ceramics will not be altered by gas pressure, liquid pressure and other stress loads.

c) **Good thermal stability**

Porous products of heat-resistant ceramics may filter molten steel or high-temperature gas. These outstanding features promise a wonderful future for porous ceramics in a broad range of applications and make these materials adaptable in many fields, including chemical engineering, environmental safety, power sources, metallurgy and the electronics industry.

Generally, porous metals with open-cell structures are necessary for functional applications, since a liquid or a gas medium is required to pass through the porous materials.

According to Twigg *et al*., (2002), the main characteristics of a ceramic foam for catalyst are sponge-like open structure with pore density of typically 0.02–0.23 g/cm^3 creating an interconnecting porosity in the range of 75%–90% or even higher. High porosity ceramics having open pores are considered suitable for catalyst supports because of the low mass-transfer resistance (Yokota *et al*., 2001).

In addition, Kim *et al*., (2009) stated that pore size, in particular, is one of the most important properties in determining the practical applications of a ceramic foam when the pore size is approximately 70–150 μm.

2.5 Porous Ceramic As Catalyst

In particular, porous ceramics can be categorised into two primary groups, as illustrated in Figure 2.3.

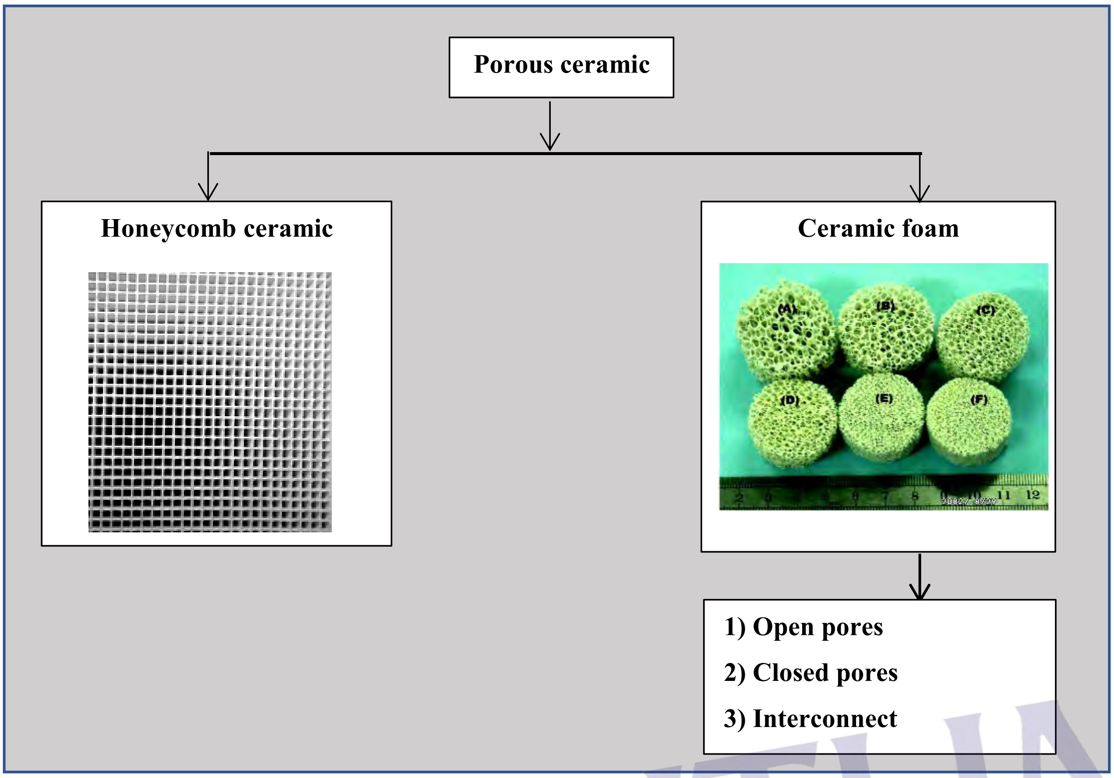

Figure 2.3: Type of porous ceramic (Al-Naib, 2018)

The common way to create ceramic honeycombs is extrusion molding, which involved mixing raw materials, extruding, drying, and firing to make the product.The ceramic honeycomb green body is obtained by extruding with the reticulated mold.After drying and sintering, porous ceramic honeycombs are produced with a regular shape.The green body has low strength, it is easily deformed, and surface dents and bubbles, cracks, and internal cracks may be found. Therefore, there are high requirements for compositions of the pug and property.

Solid open-cell foams have significantly higher mass and heat transfer coefficients compared with the monoliths and have a lower pressure drop relative to the packed bed. Ceramic foams are used as fused metal filters and thermal insulations at high temperatures (Gancarczyk *et al*., 2019). Matching the catalytic output is necessary to understand the ceramic structure regarding porosity, pore size and mechanical strength. Table 2.1 shows the structure of the ceramic foams in previous studies.

Table 2.1: Structure of ceramic foam

Type of foam	**Structure(s)**	**Reference**
Ceramic foam	• Interconnected	(Nasseh *et al*., 2019)
Ceramic foam	• Open cell • Closed cell • Interconnected	(Baharom *et al*., 2018)
Ceramic foam	• Fully open • Closed cell • Interconnected	(Rahim *et al*., 2017)

Based on the studies in Table 2.1, an open ceramic foam is generated by the reticulated structures, where the solid species constituting the foamed body is comprised only of pore struts. When the pores are separated by solid cell walls, a closed-cell ceramic foam is achieved. The three-dimensional images of open cells and closed cells are as shown in Figure 2.4.

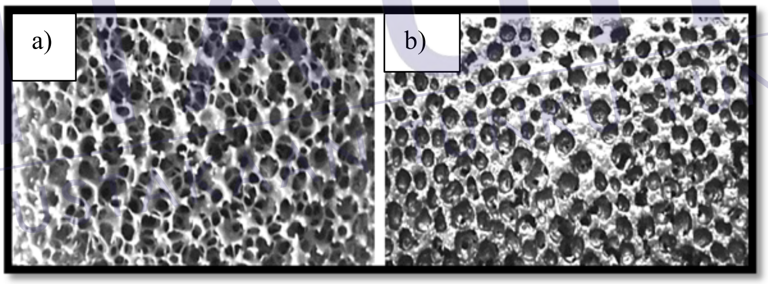

Figure 2.4: Three-dimensional images of ceramic foams: a) open pores and b) closed pores (Liu & Chen, 2014)

According to Wichianrat *et al*. (2012) and Ying *et al*. (2007), the open-cell foams refer to those with interpenetrating pores, while the closed-cell ones contain pores that are not interconnected. Typically, closed-cell foams exhibit relatively high compressive strength and therefore they are used in applications that require high strength and high energy absorption, such as automotive bumper and body armour applications. Figure 2.5 shows an example of a porous structure with some of the indicated features.

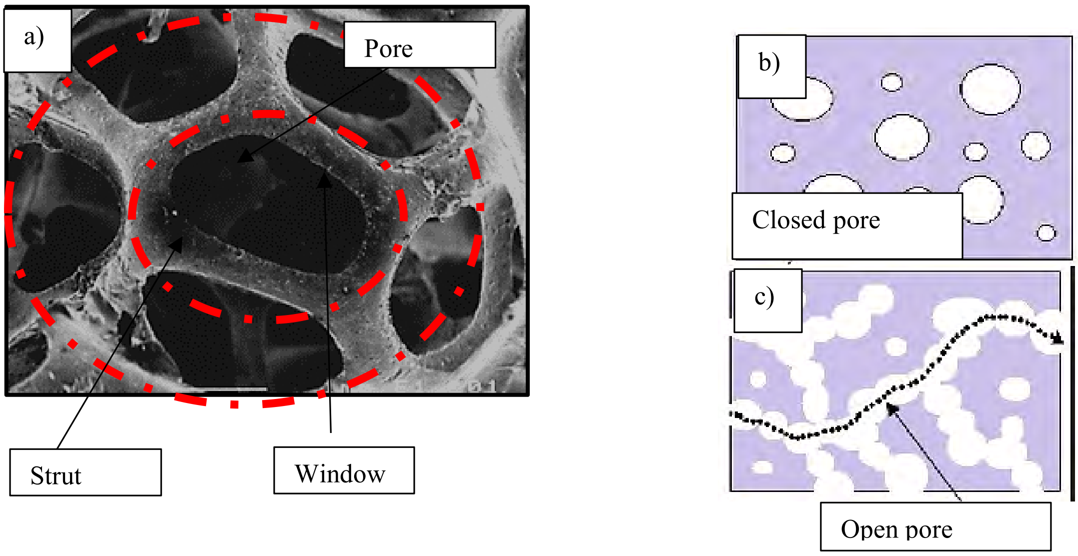

Figure 2.5: Examples of cells of ceramic foam: a) pore, window and strut, b) closed pores and c) open pores (Sampath *et al*., 2016)

Porous ceramics also can be categorised according to the size of their pores, as shown in Table 2.2.

Table 2.2: Type of pores in ceramic foam

Type of pore	Size of pore
Microporous	< 2 nm
Mesoporous	2–50 nm
Macroporous	> 50 nm

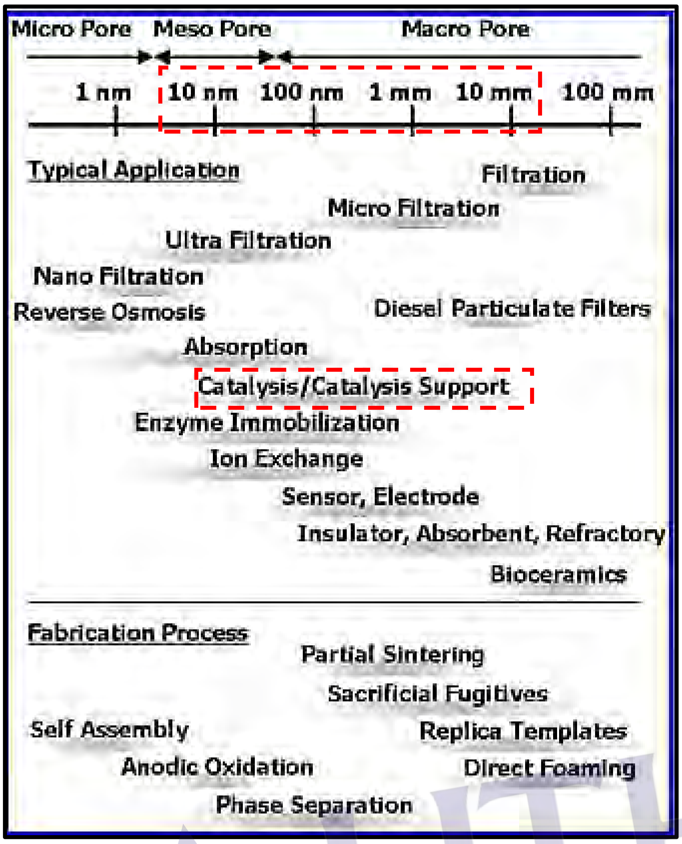

Figure 2.6: Schematic of classification of porous ceramics (Al-Naib, 2018)

Ahmad *et al*. (2014) studied porous materials and in their study, the selected starting ceramic powders were titanium dioxide (TiO_2), aluminium oxide (Al_2O_3) and silver nitrate ($AgNO_3$). In general, TiO_2 has lighter weight compared with other materials. Additionally, its brightness and refractive index are relatively high. On the other hand, $A1_2O_3$ is responsible for the resistance of metallic aluminium to weathering, while $AgNO_3$ plays an important role as antiseptics.

According to Buciuman & Czarnetzki (2003), ceramic foams can be extremely suitable catalytic carriers when a low-pressure drop is necessary. Compared with honeycomb monoliths, they offer additional advantages of radial mixing within the body and improved mass and heat transfer due to the turbulence of the flow.

2.6 Fabrication of Porous Material

Several processing methods have been investigated by researchers over the years to develop an open porous material that would have similar mechanical and physical properties to the gas catalyst. Importantly, the selection of the most suitable fabrication technique is based on the target application of the ceramic foam. This is due to the distinct properties of the ceramic foam produced by the manufacturing methods used and certain parameters involved, such as material selection, mixing, sintering temperature, sintering time and so on.

The fabrication technique can be classified according to the type of pores produced, which are open or closed pores, as illustrated in Figure 2.4. As previously mentioned, the selection of a suitable fabrication method is also based on the target application of the ceramic foam. In the present study, the structure of the ceramic foam as a gas catalyst (steam methane reforming) should consist of open and interconnected pores (Baharom *et al*., 2018).

Currently, a majority of researchers use the direct foaming method, the replication method and the compaction method to produce the porous ceramic. At present, powder foaming at high temperature is the most effective approach to manufacture ceramic foams, especially the production of closed-pore ceramic foams (Xu *et al*., 2010).

2.6.1 Foam replication method

The replication of polymer foams was one of the first manufacturing techniques developed for producing ceramics with controlled macroporosity, with the first patent acquired in 1963 by Schwartzwalder & Somers. Despite the modern era, it is still the most prevalent and commonly used method in the industry. In the replica technique, a cellular template is immersed or impregnated with a ceramic suspension/precursor solution to produce a porous ceramic having the same or different morphology as that of the original porous template. The replica method allows the fabrication of foams with larger porosity and pore size as well as being suitable for infrared heating applications (food processing) (Salleh *et al*., 2019).

In recent years, corals, wood structures, eggshell membranes and bacteria have been used as natural templates. Open-pore structures in the range of 10 to 300 µm have been formed with this technique at porosity concentrations between 25% and 95%. The formation of a ceramic microstructure with anisotropic mechanical strength is one of these templates' shortcomings.

The replica method is one of the most significant manufacturing techniques for the fabrication of porous ceramics and is mainly used in the industry for the preparation of ceramic filters used in the filtration of liquid metals and other helpful applications. The sponge replica technique is a novel technique. The essential steps in this process include the adhesion of the suspension on the polymeric sponge and the rheology of the slurry. Due to high pore interconnectivity, the permeability of gasses and fluids is enhanced in these porous materials. Despite being a user-friendly process, cellular structures processed through this route have deficient mechanical strength due to the formation of cracked struts at the time of polymer sponge pyrolysis (Studart *et al*., 2006). However, by not involving any hazardous chemicals in the process, this method is the simplest and cheapest process to use (Ahmad *et al*., 2016)

Many distinct polymers may be used in this technique for foam precursors, including polyurethane (PU), polyvinyl chloride (PVC), polystyrene (PS) and cellulose. Selee Corp. in the USA, for instance, utilises an interconnected, open-cell polyurethane foam with a 97% vacuum volume as an organic precursor, the design of which consists of a complicated pattern of dodecahedra repeated in three dimensions. In addition, the PU foam used is also inexpensive, readily available and has a uniform microstructure (Tange *et al*., 2015). In this method, the PU foam is usually used as a sacrificial template due to its microstructure, which is typically similar to the microstructure of a filter catalyst, as shown in Figure 2.7. The replication method involves a three-step replication process, as shown in Figure 2.8.

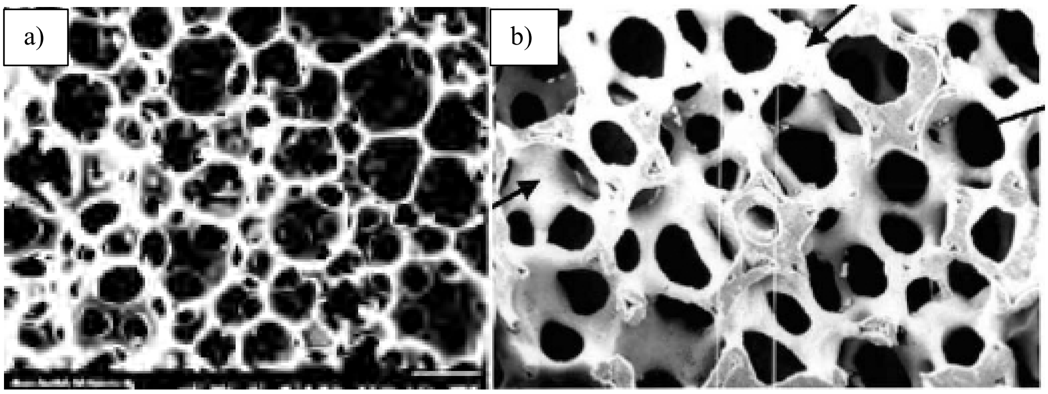

Figure 2.7: Microstructure of a) PU foam template (Asadi & Ohadi, 2015) and b) ceramic filter catalyst (Twigg & Richardson, 2002)

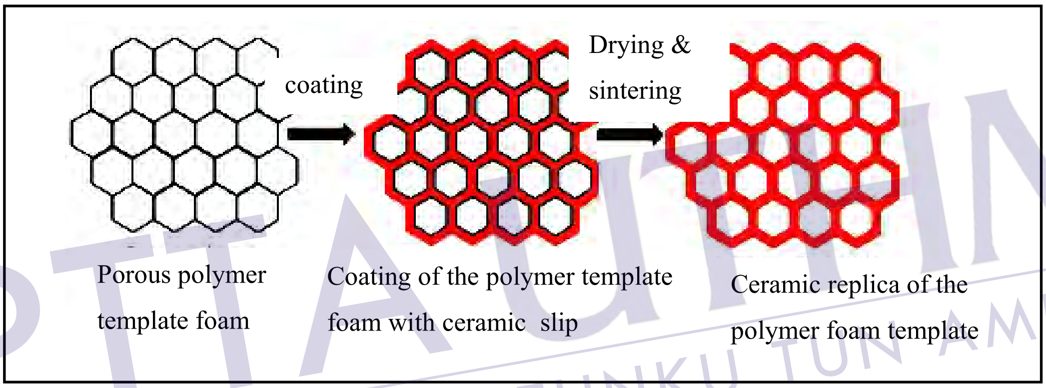

Figure 2.8: Schematic of porous material fabrication via the replication method (Bowen & Thomas, 2015)

Usually, the PU foam is initially immersed in a slurry containing metallic particles and suitable binders before subsequently being dried. After that, the PU template and the binders will be removed by pyrolysis, followed by sintering, which produces a reticulated open-cell foam (Ryan *et al*., 2006). Generally, pyrolysis is a thermal decomposition process to eliminate the binders and the PU foam template. The temperature for the pyrolysis process mainly depends on the type of binders and the PU foam used, since both have different decomposition temperatures. However, in the replica foam method, due to the cracking of the struts during pyrolysis, the mechanical properties of the reticulated foams are generally poor.

Fey *et al*. (2017) reported that it is crucial to determine the decomposition temperatures of the binders and the PU foam used. The decomposition temperatures can be identified by using thermogravimetric analysis (TGA). The samples' strength during and after the pyrolysis process is very fragile since the powder particles are only loosely connected and are not sintered yet. In addition, a lower heating rate is favoured to avoid thermal and mechanical stress generation due to the thermal expansion mismatch between the PU foam and the metal powder coating, which will cause defect formation or cracked struts (Studart *et al*., 2006). Usually, the pyrolysis process is carried out at about 1 hr of dwell time to allow sufficient time for the complete removal of the binders and the PU foam (Tange *et al*., 2015).

The replication method is done by the slurry method. According to a study by Mishra *et al*. (2009), the advantage of the slurry-based processing is the uniform distribution of the various phases through the co-dispersion in a solvent. Kovářík *et al*. (2017) used a ten-pore-per-inch polyurethane foam as a template, which was infiltrated with an aqueous potassium-based geopolymer slurry, obtaining a geopolymer/polyurethane porous composite after the drying step. Cellular geopolymers with a large amount of open porosity (~79 to ~88 vol%) and reasonably good compressive strength (~0.15 to ~0.85 MPa) were produced after sintering at 1100°C–1300°C for 4 hr.

Baharom *et al*. (2018.) reported that the replication method affects the size and distribution of the open and closed pores and the size of struts which connect the cell windows together. Mishra *et al*. (2009) reported that the results of the microstructural and mercury porosimetry studies of the composite foams showed a trimodal size distribution with small (4 nm to 8 μm), medium (40 μm to 200 μm) and large (≈1 mm or more) pores. The pores appeared spherical and interconnected, with the fibres embedded in the cell walls or struts.

As reported by Wen *et al*. (2008), the impregnation of polymeric sponge into slurries containing particles and appropriate binders was followed by drying and sintering. The porous ceramics produced by this method have open-cell structures. However, one of the more attractive features of this method lies in the easy control of the pore size by choosing different sponge templates, which is the main reason for the wide application of this method in the industry. The porous ceramics produced have open cells with porosity of around 74% to 78%.

Diverse types of porous materials have been successfully developed using the replication method, as summarised in Table 2.3 and Table 2.4. The scanning electron microscopy (SEM) images observed in Table 2.4 show that most porous materials produced by this method consist of open and interconnected pores. Indeed, porosity

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