SYNTHESIS AND PERFORMANCE ORDERED SNO_2 USING SBA-16 FOR DETECTION OF DANGEROUS GASES

by

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LIST OF SYMBOLS

Symbol	Description	Unit
A	Term in Sensor Sensitivity Equation (Equation 12)	$Nm^{-1} K^{14}$
a_0	Pre-exponential constant	ppm^{-1}
a_o	XRD unit-cell parameter	nm
C_A	Gas concentration inside the film	ppm
$C_{A,S}$	Gas concentration outside the film	ppm
D	Average Pore Size	nm
D	Crystallite Size	nm
d_{110}	d-spacing for (1 1 0) reflection	nm
D_k	Knudsen diffusion constant	m2/s
E_a	Apparent Activation Energy	kJ/mol
E_k	Activation Energy of the 1st Order Reaction	kJ/mol
I	Current	A
k	Arrhenius Equation constant	-
k_0	Pre-exponential constant, from Term A	s^{-1}
L	Film thickness	nm
M	Molecular weight of dangerous gas (H_2)	g/mol
R	Resistance	Ω
R	Gas Constant	J/mol.K
r	Pore radius	nm
S_{BET}	BET Specific Surface Area	m^2/g
ß	Full-Width at Half-Minimum (FWHM)	nm
T	Sensor Operating Temperature	˚C, K

V	Voltage	V
V_T	Total Pore Volume	cm^3/g
W	Average pore-wall thickness	nm
θ	Bragg Diffraction Angle	°
λ	Constant Wavelength	nm

LIST OF ABBREVIATIONS

B1	Micron-Sized Spheres
B2	Randomly Shaped Aggregated Particles
B3	Completely Random Aggregated Particles
B4	Macro-Spheres
BET	Brunauer-Emmett-Teller
CEM	Conventional Evaporation Method
$Cu(NO_3)2x3\ H_2O$	Copper Nitrate
F127	Pluronic F127 $EO_{106}PO_{70}E_{106}$
H_2O	Water
HCl	Hydrochloric Acid
KIT-6	Korean Institute of Technology 6
MCM-41	Mobil Composition Matter no.41
MMO	Mesoporous Metal Oxide
MWD	Microwave Digestion
NaOH	Sodium Hydroxide
O_3	Ozone
OMC	Ordered Mesoporous Carbon
OMMOs	Ordered Mesoporous Metal Oxides
OMS	Ordered Mesoporous Silica
SBA-15	Santa Barbara Amorphous 15
SBA-16	Santa Barbara Amorphous 16
SEM	Scanning Electron Microscopy (SEM)
SLM	Solid Liquid Method

TEM	Transmission Electron Microscopy
TEOS	Tetraethylorthosilicate
VASEM	Vacuum-Assisted Solvent Evaporation Method

SINTESIS DAN PRESTASI SNO2 TERSUSUN MENGGUNAKAN SBA-16 UNTUK PENGESANAN GAS BERBAHAYA

ABSTRAK

Sensor berasaskan oksida logam (SnO_2) telah menarik minat ramai dalam bidang pengesanan gas berbahaya kerana sensitiviti yang baik, tindak balas pantas dan kestabilan jangka panjang. Walau bagaimanapun, kajian lanjut masih masih diperlukan bagi menambahbaik sensitiviti dan prestasi sensor berasaskan SnO_2. Kajian ini memberi tumpuan kepada sintesis dan prestasi oksida logam SnO_2 berliang meso tersusun tempa nano menggunakan SBA-16 untuk pengesanan gas berbahaya seperti gas hidrogen. Sintesis SBA-16 telah dijalankan menggunakan pluronik F-127, asid hidroklorik (HCl) dan tetraethylortosilicate (TEOS), diikuti dengan rawatan penuaan hidroterma pada suhu 80°C. Prestasi oksida logam (SnO_2) yang dihasilkan dibandingkan menggunakan dua jenis penyusupan yang berbeza iaitu Kaedah Sejatan Konvensional (CEM) dan Kaedah Pepejal-Cecair (SLM). Berdasarkan ciri-ciri struktur yang diperolehi daripada menggunakan analisis XRD, BET, dan SEM, Kaedah Penyejatan Konvensional (CEM) daripada hasil yang diperolehi menunjukkan struktur yang lebih baik berbanding Kaedah Pepejal-Cecair (SLM). Seterusnya, MATLAB telah digunakan untuk simulasi pengesanan gas berbahaya, berdasarkan ciri-ciri fizikal oksida logam SnO_2 yang telah dihasilkan. Keputusan eksperimen menunjukkan variasi sensitiviti terhadap kepekatan gas, memberikan graf lengkung yang berada dalam keadaan yang baik apabila kepekatan gas meningkatkan, sensitiviti gas juga menunjukkan peningkatan sehingga ia mencapai tahap tepu. Keputusan yang diperolehi menunjukkan bahawa lengkung berbentuk loceng yang serupa dengan data eksperimen daripada artikel penyelidikan boleh dilihat apabila membandingkan suhu operasi dengan sensitiviti sensor dengan perubahan kepekatan gas. Kesimpulannya, analisis sensitiviti bergantung kepada saiz liang (r) dan ketebalan filem (L) telah dijalankan dan ia membawa kepada keputusan di mana saiz liang (r) mempunyai hubungan secara langsung dengan kepekaan sensor gas dan sebaliknya untuk ketebalan filem (L).

SYNTHESIS AND PERFORMANCE ORDERED SNO_2 USING SBA-16 FOR DETECTION OF DANGEROUS GASES

ABSTRACT

Tin oxide (SnO_2) based sensor have attracted a lot of interest in the field of dangerous gas detection due to its sensitivity, fast response, and long-term stability. However, more study is still needed to improve the sensitivity and performance of SnO_2 based sensors. This study focuses on the synthesis and performance of nanocasted ordered mesoporous SnO_2 using the SBA-16 for the detection of harmful gases like hydrogen gas, H_2. Synthesis of the SBA-16 was carried out using pluronic F-127, hydrochloric acid (HCl) and tetraethylortosilicate (TEOS), followed by hydrothermal aging treatment at temperature 80°C. The produced ordered mesoporous SnO_2 was compared using two different types of infiltration namely, Conventional Evaporation Method (CEM) and Solid-Liquid Method (SLM). According to the structural characteristics discovered using XRD, BET, and SEM analysis, the Conventional Evaporation Method (CEM) demonstrates much better structure compared to Solid-Liquid Method (SLM). Following that, MATLAB was used to simulate the performance of the sensor in detecting dangerous gas, based on the obtained ordered mesoporous SnO_2's physical characteristics. The experimental result indicates that the variations of the sensor sensitivity against the gas concentration, which is shown as a well-resolved curve as concentration increases the sensitivity increases until it reach the saturation level. A bell-shaped curve that similar with the experimental data from the previous research was obtained when comparing the operating temperature against the sensitivity of the sensor with variable concentration. In conclusion, sensitivity analyses depending on pore size (r) and film thickness (L) were carried out and it comes into a result where the pore size (r) has directly relationship with gas sensor sensitivity and vice versa for film thickness (L).

CHAPTER 1

INTRODUCTION

1.1 Mesoporous Metal Oxides (MMO)

Mesoporous metal oxides synthesis has become one of the famous industries to be explore deeper in the current situation. Mesoporous metal oxides, or MMO, such as SnO_2, CrO_2, MnO_2, Fe_2O_3, NiO, CeO_2, In_2O_3, and V_2O_5, are extremely helpful in a variety of applications. Their advantages include the ability to control pore size and shape, as well as stable thermal and chemical characteristics and a large surface area (Deng et al., 2016). There are wide of industries that are using mesoporous metal oxides as one of their devices such as atmospheric safety, medical diagnostics and security. Furthermore, the other industrial application that might be seen as a minor industry not only use of mesoporous metal oxides such as water remediation, adsorption, photonics and targeted drug delivery (Poonia et al., 2018). All in all, there are two types of semiconductors inside the mesoporous metal oxides (MMO) which are n-type semiconductor such as SnO_2, TiO_2, ZnO and WO_3 and p-type semiconductor such as CuO, PdO, TeO_2 and NiO (Zappa et al., 2018).

Normally, most commonly use of semiconductor is n-type of semiconductor compared to p-type of semiconductor because n-type of semiconductor is said to be more stable and higher selectivity. When it comes to the difference in quick reaction between n-type and p-type semiconductors, past research have indicated that the n-type semiconductor has a faster response than the p-type semiconductor. This can be prove by calculating the response between n-type and p-type semiconductor based on Equation 1 and Equation 2, respectively as shown below (Zhou et al., 2020)

$$Response = \frac{R_a}{R_g} \qquad Equation\ 1$$

$$Response = \frac{R_g}{R_a} \qquad Equation\ 2$$

where Ra is resistance in air

Rg is resistance in gas (targeted)

In fact, the differences between n-type and p-type of semiconductor is not only from their sensitivity and from fast response but also can be seen from the resistance gases perspective. Generally, n-type of semiconductor have higher resistance towards the oxidizing gases but lower resistance toward the reducing gas and vice versa for the p-type of semiconductor (Jusliha et al., 2018). Research has shown that the resistance of n-type of semiconductor is decreasing especially when exposed to the CO and H_2 whereas for p-type of semiconductor it is different because the resistance of the sensor will increase (Zhou et al., 2020). To prove the theory of resistance towards CO and H_2 gases can be shown as the Equation 3 below

$$Response = \frac{1 - R_g}{R_a} \qquad Equation\ 3$$

Moreover, it is widely known that the combination between n-type and p-type junction of semiconductor for example SnO_2-xCuO is bringing to us a good dimension of the mesoporous metal oxides (MMO). It is because the x value is plays major role in that combination for example if the value of x is lower than 2.78, it will become into n-type response and vice versa. This result can be shown as the Figure 1.1 below (Zhou et al., 2020)

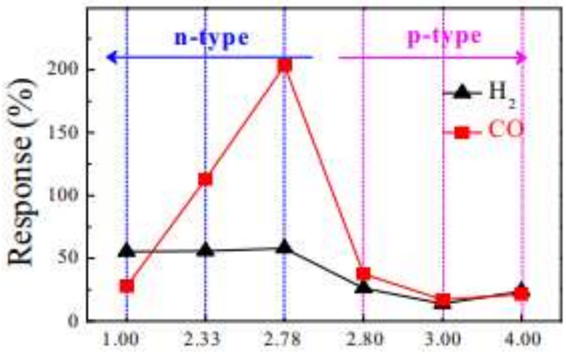

Figure 1.1: N-type and P-type response

Apart from that, the nanostructure of mesoporous metal oxides which come in many type for examples 2-dimensional (2-D) and 3-dimensional (3-D) is always good in term of sensitivity, response and stability due to their structure and properties. What is more interesting here is the part that it can offer to consumer where the process of adsorption to the targeted gas molecules such Volatile Organic Compounds (VOCs) and sources of resistance material can be done with higher efficiency (Amiri et al., 2020). One of the speciality of mesoporous metal oxide is it can be coated with a layer of sensor that can be act as insulator for the interdigitated electrodes.

Finally, the performance of mesoporous metal oxides is typically optimum at temperatures below 400°C; nevertheless, to employ mesoporous metal oxides for gas sensing, it is required to increase their thermal stability. This is due to the fact that normally for gas sensing application, it always carry out at the temperature above 500 ˚C and that is why it is already over the limit and can cause into the problem of sintering of the mesoporous silica (Jusliha et al., 2018). However, the other application for mesoporous metal oxides might be more reliable because it just use lower temperature for example used to deliver medicinal ingredients to sick cells as a medium due to their properties that are large surface area and high porosity (Jusliha et al., 2018). The mechanism of the mesoporous metal oxides is based on the template that will be used for the synthesis of it and the examples of the template are SBA-15, SBA-16, KIT-6, MCM-41 and MCM-46.

1.2 Problem Statement

SnO_2 is an n-type semiconductor and due to this property, it makes the SnO_2 one of the best mesoporous metal oxides for gas sensing application among the other. Normally, SnO_2 is used in the gas sensing application to detect the dangerous gases such as hydrogen gas, H_2 and ethanol. However, despite its great performance as gas sensing, the problems that normally need to face while using it are demand of higher sensitivity, only work at high temperature not at room temperature and the stability of the SnO_2 (Li et al., 2020). The selectivity of SnO_2 also changes with different value of air humidity (Gulevich et al., 2021). Not only that, by increasing the temperature of calcination during synthesizing of SnO2, it will change the structure of the SnO_2 amorphous into the crystalline structure due to dispersion state of amorphous structure (Chen et al., 2020).

There are numerous ways to generate SnO_2, making a more thorough understanding of its synthesis important to improve SnO_2's performance in gas sensing applications in the real world. Previous study that have been made by other researcher has shown that synthesis of SnO_2 by several methods such as conventional sol-gel method, utilization of amphiphilic structure directors (soft templating) and structure replication (hard templating) (Jusliha et al., 2017). All of these methods have their own advantages and disadvantages, which then will give several choices that can be taken before select the best of it.

The utilisation of hard templates such as SBA-15, SBA-16 and KIT-6 has gained a lot of interest in order to produce ordered SnO_2 with better physical properties such as crystallite size, surface area. The improved physical properties greatly improved the sensor sensitivity by enhancing the diffusion and chemisorption of the harmful gas. The work will explore the utilisation of SBA-16 as a hard template for the synthesis of ordered SnO_2 to be used for dangerous gas sensor

1.3 Research Objectives

For this research, below are the main objectives to achieve by completing this research

1. To synthesis ordered mesoporous SnO_2 by using SBA-16 hard template via nanocasting technique
2. To investigate the different parameters impact towards properties of the SnO_2 nanocasted mesoporous metal oxide (MMO) in term of structure and appearances.
3. To study the performance of SnO_2 nanocasted mesoporous metal oxide (MMO) as a sensor for the dangerous gas sensing.

CHAPTER 2

LITERATURE REVIEW

2.1 Hard Templating Method

For the synthesis of mesoporous metal oxides (MMO) or even known as ordered mesoporous oxide can be divided into three different method. The first one is the conventional method which is by using sol gel method and the second method that can be use is as soft templating method and the last one is hard templating method. Due to the special properties of mesoporous, metal oxides such as high surface area and coming in different pores structures and size make it valuable for the gas sensing application. The synthesis of mesoporous metal oxides play major role to indicate that the synthesis is reliable and the conditions are in good properties (Zhou et al., 2017). Therefore, one of the best method for the synthesis of mesoporous metal oxides is by using hard templating method. In addition, in order to clearly see the differences between the hard templating method and soft templating method, it is illustrated as in the Table 2.1 below (Zhang et al., 2019)

Table 2.1: Differences between Hard Templating Method and Soft Templating Method

Hard Templating Method	Soft Templating Method
Advantages 1. Most suitable for the all types of crystalline oxides 2. Chemical structure is stronger and durable	Advantages 1. Simpler process and not complicated 2. Easily to control the pore structures and size
Disadvantages 1. More complicated process which lead to higher cost 2. Lower yield 3. Hard to control the pore structures and size	Disadvantages 1. During the crystallization process, the porous structure can disintegrate 2. Less stable in term of thermal stability

Hard templating method or also called as nanocasting or structure replication is booming right now especially for certain industries such as gas sensing industry and medical industry. Hard templating method is use to produce a rigid defined porous structures by filling up suitable precursor into a MMO such as SnO_2. Mainly, there are three steps for the synthesis of MMO by using hard templating method as mentioned below (Zhang et al., 2019) as shown in Figure 2.1

i) synthesizing of mesoporous replicas

ii) infiltration of metal precursors

iii) removal of hard template

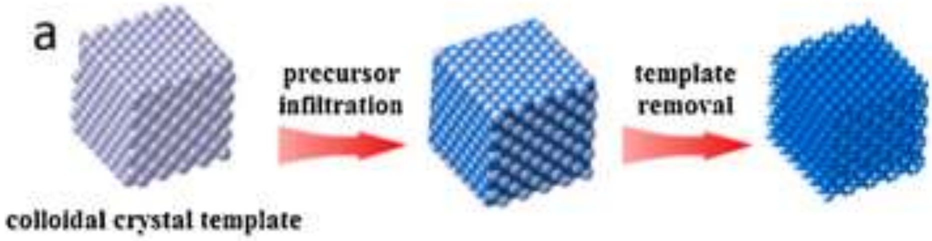

Figure 2.1: Hard templating method process synthesis (Zhou et al., 2017)

It is widely known that by implementing the hard templating method, the mesoporous metal oxide is formed with a consistent pore size distribution which then lead into a more stable nanostructure (Jusliha et al., 2018). Not only that, the template produced from hard templating method also can be used at higher temperature compare to by using soft templating method. The reason behind this advantage is due to hard templating method has used higher calcination temperature during the wet impregnation method As mentioned previously, the second step is involving infiltration of metal precursors by using the wet impregnation method and normally need to be done few times. There are two types of impregnation method that can be used, which are wet impregnation and dry impregnation, but the best between these two is the wet impregnation method. Research has shown that the precursor infiltration is done repeatedly for the sake of fulfilment the template pores (Zhou et al., 2017).

Therefore, below is the Table 2.2 from (Jusliha et al., 2018) for the normal product that can have in the market that were produced from the synthesis of ordered mesoporous metal oxides (OMMOs) by hard templating method

Table 2.2: Synthesis of Ordered Mesoporous Metal Oxides (Ommos) By Hard Templating Method for Several Types of Metal Oxides

Metal Oxides	**Template (by hard templating)**	**Structure of the Template**	**Properties of the Metal Oxides**
SnO_2	KIT-6	*Ia3d*	S_{BET} = 160 m^2/g D = 3.8 nm V_T = 0.34 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 80 m^2/g
	SBA-15	*p6mm*	S_{BET} = 84 m^2/g D = 2.8 nm V_T = 0.22 cm^3/g
	SBA-16	*Im3m*	S_{BET} = 121 m^2/g D = 4.1 nm V_T = 0.28 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 114 m^2/g D = 3.4 or 18.0 nm V_T = 0.32 cm^3/g
	SMS	Disordered	S_{BET} = 117 m^2/g D = 7.1 nm V_T = 0.35 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 80 m^2/g D = 4.4 nm V_T = 0.34 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 75 m^2/g D = 3.8 nm V_T = 0.08 cm^3/g
ZrO_2	SBA-15	*p6mm*	S_{BET} = 220 m^2/g D = 2.9 or 27 nm V_T = 0.57 cm^3/g
Al_2O_3	FDU-15	*p6mm*	S_{BET} = 141-357 m^2/g D = 4.1-5.6 nm V_T = 0.16-0.61 cm^3/g

Table 2: (Continued)

Metal Oxides	Template (by hard templating)	Structure of the Template	Properties of the Metal Oxides
TiO_2	KIT-6	*Ia3d*	S_{BET} = 90-207 m^2/g D = 3.0-4.3 nm V_T = 0.06-0.17 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 258 m^2/g D = 2.7 nm V_T = 0.30 cm^3/g
	MSU-H	*p6mm*	S_{BET} = 150 m^2/g D = 5.1 nm V_T = 0.44 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 220 m^2/g D = 4.9 - 7.2 nm V_T = 0.14 – 0.25 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 103 - 346 m^2/g D = 4.9-7.2 nm V_T = 0.14 – 0.25 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 30-317 m^2/g D = 3.3-7.6 nm V_T = 0.09-0.33 cm^3/g
	Chiral Silica Film	Chiral	S_{BET} = 150-230 m^2/g D = 2.5-7.9 nm V_T = 0.23-0.31 cm^3/g
NiO	KIT-6	*Ia3d*	S_{BET} = 96 m^2/g D = 3.48 or 21 nm
	KIT-6	*Ia3d*	S_{BET} = 81-118 m^2/g D = 3.3-3.7 nm V_T = 0.23-0.51 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 82-109 m^2/g D = 3.1-33 nm
	SBA-15	*p6mm*	S_{BET} = 47 m^2/g D = 4.0 nm

Table 2: (Continued)

Metal Oxides	Template (by hard templating)	Structure of the Template	Properties of the Metal Oxides
Cr_2O_3	KIT-6	*Ia3d*	S_{BET} = 72-92 m^2/g D = 2.5 or 11.0 nm V_T = 0.18-0.24 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 48-82 m^2/g D = 3.1-12.1 nm V_T = 0.13-0.22 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 86 m^2/g D = 3.1 or 12.3 nm V_T = 0.61 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 88 m^2/g D = 3.3 nm V_T = 0.23 cm^3/g
	MWD-SBA-15	*p6mm*	S_{BET} = 65 m^2/g D = 3.6 nm V_T = 0.34 cm^3/g
	Amine-SBA-15	*p6mm*	S_{BET} = 58 m^2/g D = 3.4 nm V_T = 0.14 cm^3/g
CoO	KIT-6	*Ia3d*	S_{BET} = 55 m^2/g D = 4.0 nm V_T = 0.14 cm^3/g
RuO_2	KIT-6	$I4_132$	S_{BET} = 131 m^2/g D = 3, 15-20 nm V_T = 0.42 cm^3/g
CeO_2	KIT-6	*Ia3d*	S_{BET} = 112 m^2/g D = 6.7 nm V_T = 0.18 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 123.9 m^2/g D = 3.4 nm V_T = 0.26 cm^3/g

Table 2: (Continued)

Metal Oxides	Template (by hard templating)	Structure of the Template	Properties of the Metal Oxides
	KIT-6 SBA-15	*Ia3d* *p6mm*	S_{BET} = 198 m^2/g D = 3.5 nm V_T = 0.24 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 192-224.7 m^2/g D = 3.3-3.8 nm V_T = 0.23-0.37 cm^3/g
In_2O_3	KIT-6 SBA-15	*Ia3d* *p6mm*	S_{BET} = 80-90 m^2/g D = 7.8-9.1 nm V_T = 0.13-0.27 cm^3/g S_{BET} = 52-87 m^2/g D = 9.2-9.6 nm V_T = 0.14-0.26 cm^3/g
	KIT-6 SBA-15	*Ia3d* *p6mm*	S_{BET} = 170 m^2/g D = 2.7 nm V_T = 0.45 cm^3/g S_{BET} = 153 m^2/g D = 2.6 nm V_T = 0.43 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 56.05 m^2/g D = 6.1 nm
	FDU-5	*Ia3d*, $I4_132$	S_{BET} => 70 m^2/g V_T = 0.15 cm^3/g
Co_3O_4	KIT-6	*Ia3d*	S_{BET} = 13 m^2/g D = 4.6 nm
	KIT-6	*Ia3d* or $I4_132$	S_{BET} = 83-152 m^2/g D = 4-10 nm V_T = 0.1-0.47 cm^3/g
	SBA-15	*p6mm*	S_{BET} = 101-122 m^2/g D = 3.4-5.1 nm V_T = 0.14-0.22 cm^3/g

Table 2: (Continued)

Metal Oxides	Template (by hard templating)	Structure of the Template	Properties of the Metal Oxides
	MWD-SBA-15	*p6mm*	S_{BET} = 82 m^2/g D = 3.4 nm V_T = 0.37 cm^3/g
	MWD-SBA-16	*Im3m*	S_{BET} = 92 m^2/g D = 6.5 nm V_T = 0.33 cm^3/g
	Vinyl-KIT-6	*Ia3d*	S_{BET} = 122 m^2/g D = 3.8 nm V_T = 0.22 cm^3/g
α-Fe_2O_3	KIT-6	*Ia3d*	S_{BET} = 108 m^2/g D = 4.0 nm
	KIT-6	*Ia3d*	S_{BET} = 139 m^2/g D = 3.9 nm
	KIT-6	*Ia3d*	S_{BET} = 205.4 m^2/g D = 2.2-15.7 nm
	MWD-SBA-15	*p6mm*	S_{BET} = 137 m^2/g D = 4.0 nm V_T = 0.43 cm^3/g
ß-MnO_2	KIT-6	*Ia3d*	S_{BET} = 87-137 m^2/g D = 3.3-3.7 nm V_T = 0.23-0.48 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 84 m^2/g D = 4.9, 18.2 nm V_T = 0.38 cm^3/g
	KIT-6	*Ia3d*	S_{BET} = 87 m^2/g D = 3.6, 30 nm V_T = 0.55 cm^3/g

2.2 SBA-16 as Template for Ti (IV) Oxide (SnO_2) synthesis

SBA-16 or known as Santa Barbara Amorphous is one of the templates that can be used for synthesizing of Ti (IV) Oxide (SnO_2) and another family of SBA-15. The main difference between both two is where SBA-16 is coming in 3-dimensional (3-D) structure whereas SBA-15 is in the 2-dimensional (2-D) structure. On the other hand, the other template that is useful to synthesize SnO_2 for example KIT-6, MCM-41, MCM-48, FDU-1 and FDU-12. All of these templates have different types of nanostructure where indirectly it will bring into different properties and function. From the previous discussion, it is undeniable that normally SBA-16 is mainly used for gas sensing application. However, the other usage of SBA-16 can be seen in different industries for example in medical industry (i.e. detection of exhaled acetone) and safety (i.e. detection of air pollutants, volatile organic compounds (VOCs), flammable gases and regulation of industrial exhaust).

Furthermore, SBA-16 is built up of spherical nanocavities linked by nanochannels that are very short (Yue et al., 2008) and have an *Im*3*m* space group, according to earlier study. For further information, the size of the openings to the cavities may be regulated in the range of 4–9 nm in diameter by varying temperatures between 100 and 140°C. In order to synthesise SBA-16 from the specified surfactants, few factors must be taken into consideration. The pH and temperature are two of the characteristics that are provided. To generate SBA-16, the pH must be acidic, although it is preferable if the pH is less than 2, because the synthesising process occurs only at low acidic conditions. As mentioned earlier, SBA-16 is coming in the 3-dimensional (3-D) structure and to be clear here, it is actually coming in cubic arrangement where it have eight openings at all side of the cages which then allow the access of another mesopores to join the network (Gonzalez et al., 2018).

Next, when it comes to the advantages of SBA-16 it is clearly stated that SBA-16 have few good properties such as large pore volumes, high surface-to-volume ratio, and customizable and consistent pore diameters are all desirable (Chen et al., 2020). Micropores in SBA-16 mesoporous materials cause intra-wall porosity, which connects the different types of mesoporous cavities (Choi et al., 2019). In addition, normally for the Santa Barbara Amorphous family either SBA-15 or SBA-16 both of them will use Pluronic F127 for process synthesis of them. Nevertheless, despite their provided advantages based on their structure and properties, SBA-16 also have their own drawbacks for example complicated method for synthesizing of SBA-16, low value in term of surface area and pore size and also less stable at higher temperature mostly above 500°C. Thus, the improvement and deeper investigation must be done to explore more especially about their abilities and performance

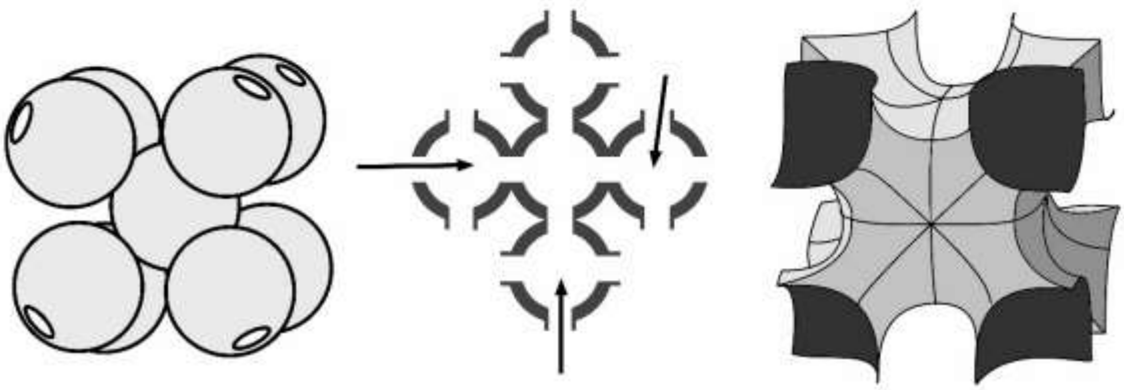

Figure 2.2: SBA-16 3-dimensional (3-D) structure (Retajczyk et al., 2020)

As in the Figure 2.2 above, the structure of SBA-16 is having 3-dimensional with 12 walls facing the corners of the cube. With these 12 wall channels, give huge advantages to the SBA-16 where it can lessen the possibility of pore blockage, which indirectly makes the mass transfer uncomplicated (Retajczyk et al., 2020). Not only have that, SBA-16 also had two different types of diameter, which are diameter at the cage and the entrance. For the first type diameter, which is diameter at the cage, it normally calculated based on the adsorption branch while for the second type diameter which diameter of the entrance, it can be calculated from the desorption branch. For an additional information, one of a researcher has mentioned that the diameter of the entrance between two mesopores is often significantly less than the diameter of mesopore at the cage diameter (Retajczyk et al., 2020).

The SBA-16 is synthesized by combining Pluronic F127 with TEOS, and it must be done in an acidic condition. To begin, 2.0 g Pluronic F127 is completely dissolved in 15 mL deionized water. Then, with constant stirring, 60 g of 2 M HCl solution is added to create a clear solution. Then, drop by drop, 2.5g of TEOS is added to the solution. To enhance silica hydrolysis and sol formation, the mixture was stirred at 37 °C for 24 hours. The resultant suspension is then put in a teflon-lined autoclave and held at 100 °C under constant conditions for 48 hours. The solids are next filtered and dried at room temperature. The dried solid is then calcined for 6 hours at 550°C in a tube furnace with dry airflow at a rate of 1°C/min. Finally, SBA-16 white powder is obtained.

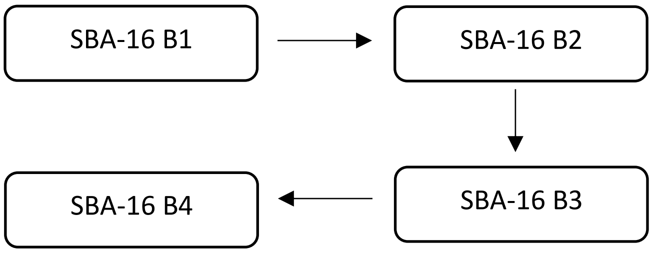

Figure 2.3: Route for synthesizing of SBA-16 (Stevens et al., 2006)

where B1 is Micron-Sized Spheres

B2 is Randomly Shaped Aggregated Particles

B3 is Completely Random Aggregated Particles

B4 is Macro-Spheres

The pH and temperature are two of the characteristics that must be taken into account when synthesising SBA-16. For the production of SBA-16, the pH must be acidic, and some of references said that it is preferable if the pH is less than 2, because the synthesising process occurs only at low acidic conditions. Furthermore, SBA-16 is built up of spherical nanocavities connected by nanochannels that are relatively short (Yue et al., 2008) and have an *Im*3*m* space group, according to earlier studies. Moreover, by varying the temperature between 100 and 140°C, the size of the cavity openings may be adjusted in the range of 4–9 nm in diameter.

2.3 Ti (IV) Oxide (SnO_2) Properties and Applications

Despite the other metal oxides, the selection of SnO_2 in the sensing technologies as a medium to sense the dangerous gases such as hydrogen gas, H_2 and ethanol is booming right now. As shown in the Figure 2.4 (Asha et al., 2021), the percentage of using SnO_2 is the highest with big different compare to the other such as zinc oxide, aluminium oxide, indium oxide, iron oxide, copper oxide, tungsten oxide and mixed oxide. The characteristics of copper and iron impacted the pore volume, according to one study (Akti 2019). When copper oxides are used, pore volume increases, whereas when iron oxides are used, pore volume decreases. As a result, the selection for both is small since the buyer remains sceptical due to their inconsistency in detecting performance.

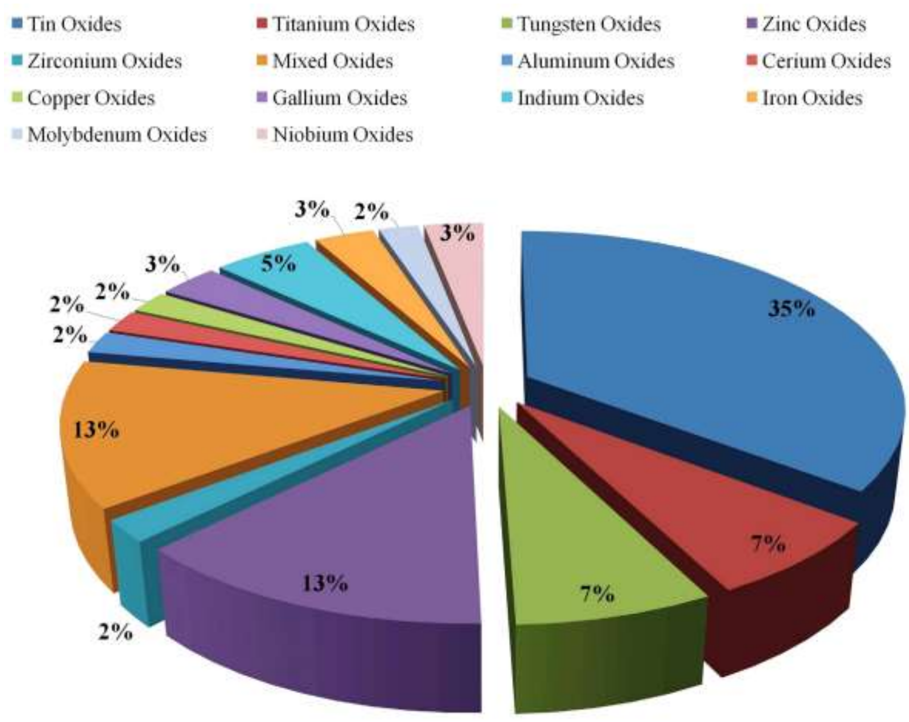

Figure 2.4: Trend of choices from various metal oxides as gas sensor (Asha et al., 2021)

As previously stated, the synthesis of mesoporous metal oxide for the manufacture of SnO_2 is advantageous to society and specialised businesses, particularly in the field of gas sensing. SnO_2 is a very dependable component in gas sensing applications owing to its qualities and structures, which include high efficiency and signal responsiveness, more stable for long-term use, improved selectivity, and resilience to any changes in operating conditions. As a result, SnO2 applications are separated into two groups, as shown below (Zhou et al., 2018).

1) Atmospheric Safety
   - detection of volatile organic compounds (VOCs)
   - detection of air pollutants
   - detection of dangerous gases (i.e. toxic, flammable and explosive)
   - regulation of industrial exhaust

2) Medical Diagnostics
   - detection of exhaled acetone

To begin with, the phrase "sustainability" is always associated with a brighter future and a cleaner environment. As far as the environment is concerned, this endeavour of synthesising mesoporous metal oxides employing SBA-16 in the hard templating technique for the synthesis of SnO_2 will not be harmful. This is owing to the fact that no harmful products were formed during the whole process, which may have resulted in contamination of any kind. Furthermore, no waste or by-product is produced because it has already been transformed into core product, SnO_2. As a result, with the applied conditions that are environmentally friendly, this method will be sustainable in the future. Moreover, the hard templating technique for producing SnO_2 by using the SBA-16 is said to be a sustainable process since it has provided significant benefits to society, particularly in allied areas such as safety, medicine, and sensors. Even though the synthesising process is difficult, the end result is extremely helpful and simple to utilise. One of the most significant advantages is that, despite the use of chemicals, it does not result in the production of greenhouse gases or the loss of the ozone layer.

In addition, it is a well-known fact that SnO_2 is one the best type of gas sensitive materials because the SnO_2 itself have several properties such as fast response, high sensitivity, small and light in term of size and weight. For that matter, SnO_2 also is the most suitable for gas sensing application because it can have shorter adsorption and desorption time despite in their economically term such as low cost due to they can save the energy process (Wang et al., 2018). Aside from that, despite SnO_2's advantages, it is must not disregard its difficulties because overcoming the weaknesses improves the overall qualities. Their high operating temperature and low selectivity are one of the hottest topics for the SnO_2 properties. Thus, many studies have proposed several methods regarding to that based on their experiences and knowledge. It is comes into a conclusion that hard templating method is becoming one of the best approach for synthesizing of mesoporous silica template, SBA-16 for the use of SnO_2 nanocasted mesoporous metal oxide for the application of dangerous gas sensing. Furthermore, in order to have a better understanding on how SnO2 is implemented, the mechanisms of the SnO2 as below (Wang et al., 2018)

1) When SnO_2 exposed to the air, the reaction between air and molecular surface occur
2) An electronic chemical adsorption oxygen bond is formed between the oxygen molecules and gas sensitive materials, reduced the electrons of SnO_2

2.4 Sustainability

There are definitely known facts that Volatile Organic Compounds or VOCs such as formaldehyde, benzene, acetone, ethylene, glycol and xylene is one of types of chemicals that are dangerous if they are released to the environment. Normally, VOCs also can be found in our daily and hence actually peoples are exposed towards it regularly. Some of these chemicals have properties such as transparent and not smelly, which make us did not notice when we are exposing to it in a long term. Exposure towards it in a long term can be bring into health issues (i.e. headache, vomiting, cancer and kidney damage) and lead to pollutions to the environment. Therefore, the development of mesoporous silica template or nanostructures is really important since they have their own abilities such as low cost, highly sensitive and selective and also fast response make it as one of the best option of sensors to detect common volatile organic compounds (VOCs) (Tomer et al., 2016)

SnO_2 is a one of the top example of ordered mesoporous silica and trustworthy component especially in gas sensing applications because of its qualities and structures, which include high efficiency and signal responsiveness, more stable for long-term use, improved selectivity, and resilience to any changes in operating conditions. As a matter of fact, SnO_2 itself becomes one of the best options to solve the environmental problems such as air and water pollution or even becomes a saviour to detect the drivers who may be under the influence of alcohol. Their durability against high operating condition (i.e.

high temperature, concentration and pressure) making it suitable and safer option in various applications. They also met the green technology requirements because they are not causing into environmental pollution, as the definition of green technology itself is the application of science and technology to limit human influences on the natural environment.

Lastly, I personally believed that by improving the study for ordered mesoporous structure would bring into bright future in this field. Even though in Malaysia, the study of ordered mesoporous structured is not one of the top field, but deeper research and exploration would bring it into another level as well as in the other countries. Nevertheless, the impact of this study not only beneficial to our society but also will bring us a greener circumstances.

2.5 Innovations

These gas sensors are made of mesoporous materials to reach out the demand for indoor pollution management in environmental monitoring systems. Hence, below are some of the products that we can use to have a sustainability environment with the use of ordered mesoporous structures

1) Alcohol Gas Sensors

Figure 2.5: Alcohol Gas Sensors

Research has shown that the properties of Ordered Mesoporous Structures (OMS) that provide us high sensitivity towards the dangerous gases such as hydrogen gas, H_2 and ethanol. Thus, this is the reason why OMS is used in the production of gas sensors to inspect the drivers that might be under alcohol influence. As shown in the Figure 2.5

above, this modern gas sensor is helpful especially to reduce possibility of accidents cause by drive under influence. Different types of mesoporous silica template such SBA-16, SBA-15, KIT-6 and MCM-12 will affect the structures and physical properties of SnO_2 nanocomposite, which then used as main component to build a gas sensor.

2) Water remediation

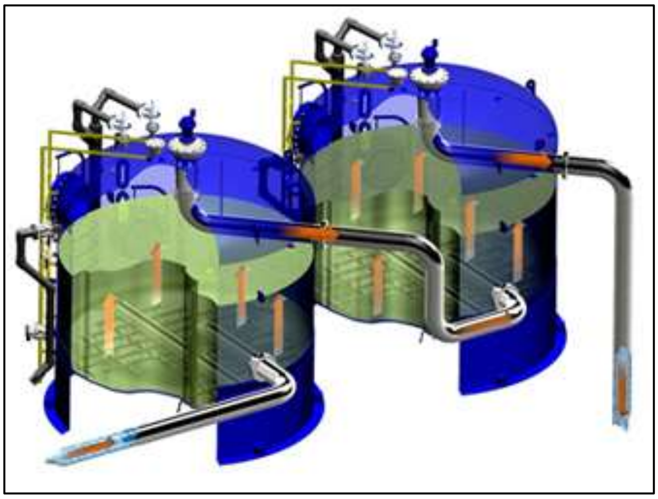

Figure 2.6: Water Remediation Technology

Currently, the issues of lack of water management is booming and becoming a worldwide problem that every countries are facing right now. Lack of new method and technologies of handling that water making it as a pollution when release to the environment. Filtration, membrane technology, chemical precipitation, reverse osmosis, ion exchange, solvent extraction, electrochemical treatment, evaporation recovery, photocatalysis and adsorption (Diagboya and Dikio 2018) have all been used to remove contaminants containing in the water to ensure that good water quality will be provided to the consumer. It is clear that the ordered mesoporous structures (OMS) such as SnO_2 that large pore size and surface area can provide us a good performance especially for detection of contaminants inside the water. By utilising the structure inside the OMS, highly porous adsorbents, which are in high demand in aqueous separations involving large molecules, can be synthesised from ordered mesoporous silica in order to include a wide variety of functionally active chemical components.

3) Catalysis

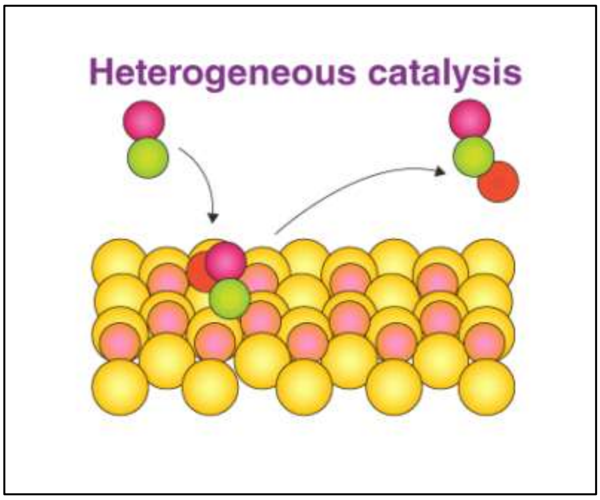

Figure 2.7: Heterogeneous Catalysis Molecule

Recently, the issues regarding to the homogenous catalyst for instance needs ligands that are somewhat large, as well as catalyst recovery and recycling always been debating especially for the industrial player. They mentioned that all these problems always lowering the homogeneous catalyst performance. Therefore, the development of heterogeneous catalyst has become a preferable choice nowadays compared to homogeneous catalyst since their abilities that have a photonic compound in high purity, their simplicity of handling and simple separation processes, as long as the catalytic performance is sufficient (Sahoo et al., 2011). Due to their distinctive pore structures, ordered mesoporous silica materials (OMS) such as SBA-15, SBA-16, KIT-6, and MCM-41 have gotten a lot of interest in the catalysis field inside the industry. As in many case study, all the researchers are agreed that OMS advantageous such as large pore volume and high surface area making it useful in the catalysis development to act as a support for them. Due to their durability in numerous chemical processes, heterogeneous catalysts based on ordered mesoporous silica have shown to be an effective alternative, particularly for the hydrogenation of ketones.

CHAPTER 3

METHODOLOGY

3.1 Research Flow

Figure 3.1 shows the overall process flowchart of the present work. Firstly, the synthesis of SBA-16 was carried out. The synthesised SBA-16 was characterised in order the ensure the desired structure was obtained. After that, mesoporous SnO2 was synthesised and characterised by using SEM, BET and XRD analyses. The performance of the sensor in detecting hydrogen gas was carried out using the improved MATLAB coding.

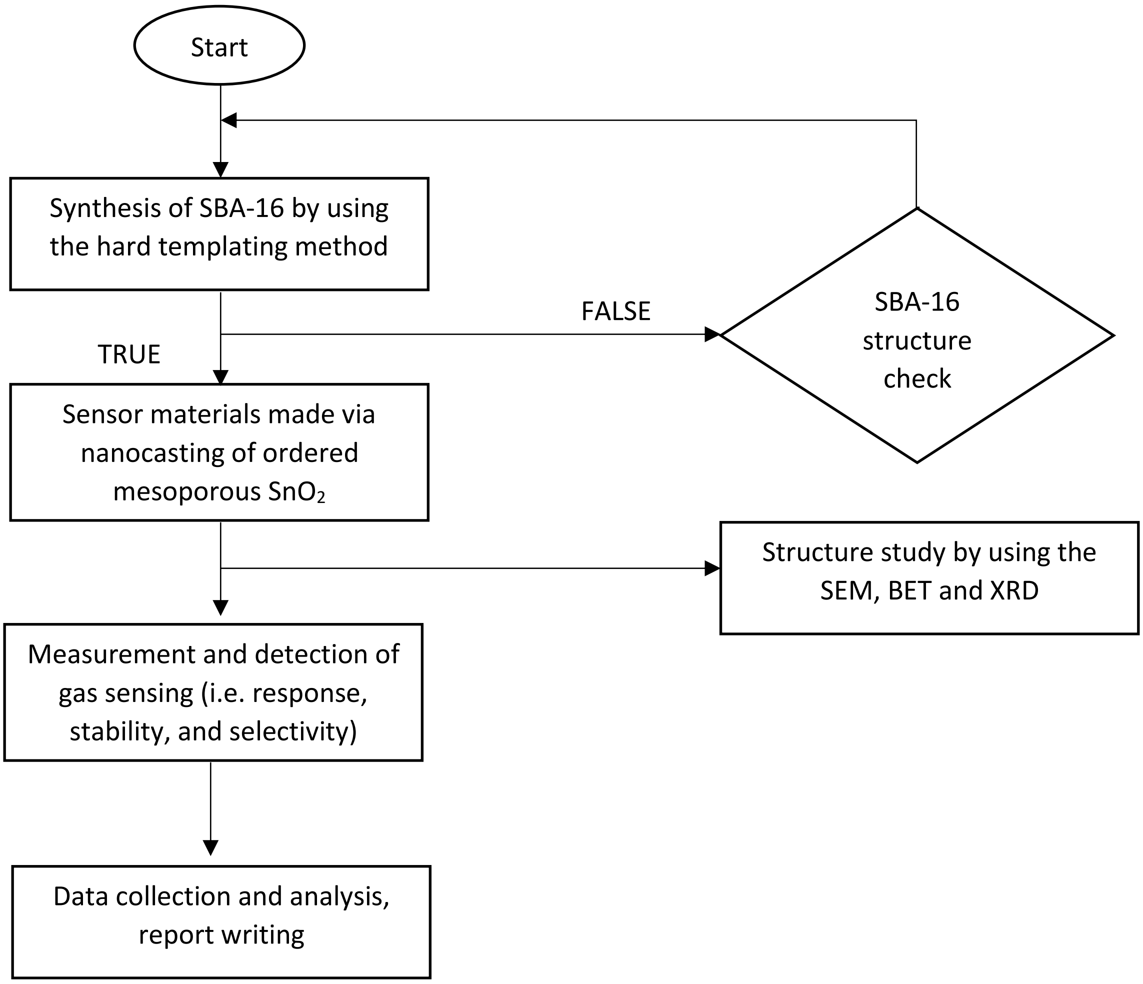

Figure 3.1: Overall Process Flowchart

3.2 Material Preparation

Table 3.1 and Table 3.2 gives the list of chemicals and equipment used in the present work for the syntheses of SBA-16 and ordered mesoporous SnO_2, respectively.

Table 3.1: Material Required for the Synthesis of SBA-16 Template and SnO_2

Materials	**Based**
Pluronic F127 ($EO_{106}PO_{70}E_{106}$) Tetraethylortosilicate (TEOS)	Template and Silica Source
Hydrochloric Acid (HCl)	Acid Source
Water (H_2O)	Solvent Source
Copper Nitrate ($Cu(NO_3)2$ x $3H_2O$)	Metal Source (Precursor)
Sodium Hydroxide (NaOH)	Removal Silica Template Source
Ethanol (C_2H_5OH)	Sensing Source

Table 3.2: Equipment Required for the Synthesis of SBA-16 Template and SnO_2

Equipment	**Purpose**
Transmission Electron Microscope (TEM)	By using electron energy to check the morphology and composition information (ordered SBA-16)
X-Ray Diffraction (XRD)	To examine the crystalline structure of the materials and to detect the SnO_2
Brunauer-Emmett-Teller (BET)	To check the structure especially the surface area and pore diameter of the SBA-16 template
Analytical Balance	To figure out how much powder and chemicals weight

Furnace	For sintering and annealing powder samples
Pestle and Mortar	To crush down the sample powder from the rock solid
Vacuum Oven	To dry up in vapour condition for the solvent that contain the sample powder
Hot Plate Stirrer	To heat up the sample prepared and stir or mix them together

3.3 Steps for the Production of SBA-16 Template

Jusliha et al., 2018's hard template approach was used for the synthesis of SBA-16. According to the previous section, the hard templating method is preferable to the soft templating method, especially in terms of benefits and effectiveness. Therefore, the procedures that must be followed in order to produce SBA-16 are listed below.

1. At a temperature of 28 °C (optimal temperature for synthesize of SBA-16 template), 3.7 g of pluronic F-127 was dissolved in 100 mL of a 0.2 M HCl solution.
2. 8.56 g of TEOS is added into the solution with steady stirring.
3. The solid product was collected by filtration and washed with ethanol
4. Then, it will be dried at room temperature
5. Next, it will be calcined at the temperature of 550 °C in air for 5 h at a heating rate of 2 °C/min
6. In accordance with the temperature of the hydrothermal treatment, the product is referred as SBA-16-80.

3.4 Steps for the Production of SnO_2 by using Conventional Evaporation Method

Wet impregnation approach was employed for the synthesis and manufacture of SnO_2 using the precursor Stannous Chloride Dehydrate, ($SnCl_2$ x $3H_2O$) (Guo et al., 2011). The processes for producing SnO_2 by removing the mesoporous silica template of SBA-16. Actually there are two method that used in this case study but at first it is involving the Conventional Evaporation Method (CEM) and steps of it is mentioned below