

# Synthesis of high surface area mesoporous silica SBA-15 by adjusting hydrothermal treatment time and the amount of polyvinyl alcohol

*By Ridhawati Ridhawati*

## Research Article

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Ridhawati Thahir\*, Abdul Wahid Wahab, Nursiah La Nafie, Indah Raya

# Synthesis of high surface area mesoporous silica SBA-15 by adjusting hydrothermal treatment time and the amount of polyvinyl alcohol

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**Abstract:** The high surface area of mesoporous silica SBA-15 has been synthesized successfully by hydrothermal treatment with direct addition of PVA, triblock copolymer (P123) as a direct structure agent and tetraethyl orthosilicate (TEOS) as a precursor. The mesoporous silica SBA-15 have been characterized with nitrogen physisorption, scanning electron microscopy, Fourier transformed infrared spectroscopy, and x-ray diffraction. Measurement of nitrogen sorption indicated that with the addition of PVA, the surface area is increased but the pore volume and pore diameter is not significantly. The short time of hydrothermal treatment (20 h) and using x-ray diffraction, showed that the morphological structure of silica SBA-15 can be changed to a orthorhombic crystal system. The result of the FTIR and SEM-EDX characteristic indicated the functional groups and morphology of the SBA-15 with a narrow pore size distribution. The BET method has exhibited the largest surface area 1726 m<sup>2</sup>/g, pore volume 1.4 cm<sup>3</sup>/g, and pore diameter 3.2 nm. It can be suggested that the silica mesoporous SBA-15 will have potential application prospect in catalysis, storage, and adsorbent.

**Keywords:** mesoporous silica; SBA-15; surface area; hydrothermal treatment.

\*Corresponding author: Ridhawati Thahir, Chemical Engineering Department, State Polytechnic of Ujung Pandang, Jl. Perintis Kemerdekaan Km. 10 Makassar, Indonesia, E-mail: ridha331@upg.ac.id

Abdul Wahid Wahab, Nursiah La Nafie, Indah Raya, Department of Chemistry, Faculty of Mathematics and Natural Science, Hasanuddin University, Makassar, Indonesia

## 1 Introduction

Porous materials are established as a solid have content of pores and the fraction of pore volume to the total volume 0.2-0.95 [1]. According to the IUPAC, porous materials have a classified base on pore size. They are macropores (pore diameter with more than 50 nm); mesopores (pore diameter between 2 nm and 50 nm) and micropores (pores with diameter no more than 2 nm) [2]. Santa Barbara Amorphous (SBA-15) is one of the typical mesoporous base silica that has well ordered hexagonal structure with uniform pore size up to 32 nm [3].

Previous studies have reported that mesoporous silica SBA-15 had advanced structure properties, such as high specific surface area to acquire many active sites in insufficient volume [4], uniformity of pore diameter to permit diffusion and adsorption of larger molecules [5–8], thick pore wall and exceptionally hydrothermal stability [9], and specify structure is still a considerable role for future material scientist [10]. The excellent mesoporous materials of SBA-15 can be used in an amount of application such as catalysis [11–13], water treatment [14,15], sensor [16–18], and supporting cell for composite materials [12,18–20]. The mesoporous materials, high surface area, and high thermal stability have widely used for more application in industry. In spite of properties of the SBA-15 in various application, it is still required to modify variable synthesis of SBA-15.

The changes experienced by the enhanced high surface area of SBA-15 had been studied by many researchers using the addition of PVA (polyvinyl alcohol). It was investigated that the surface area of SBA-15 is increased to 1248 m<sup>2</sup>/g while the structure and pore size stable [4]. This concept has recently been challenged by a decreased surface area of SBA-15, their studies explaining that added PVA during preparation can produce highly molecular sieve of SBA-15 with high pore and high hydrothermal stability. The result of the specific surface area by the adjunct of PVA is 687 m<sup>2</sup>/g. The role of PVA serves as a mild template to

produce better structural properties [9]. Therefore, one of the most significant currents investigates the effect of the amount PVA to explore the significantly high surface area, hydrothermal stability, and morphology of silica SBA-15

The methodological approach taken in this study is a combination of the amount of PVA and time adjusting for hydrothermal treatment. One of the most significant current studies in control of the pore size and mesoporous structure of the material is modified process variable during the preparation of typesetting Pluronic as a surfactant template and interaction process between surfactant and precursor silica source. Part of the purpose of this study was to investigate the modification variable synthesis to affect the physical sorption properties and mesoporous structure of the final product. The product silica SBA-15 was analyzed by nitrogen physisorption, scanning electron microscopy (SEM) with energy dispersive x-ray analysis (EDX), fourier transformed infrared spectroscopy (FTIR), and x-ray diffraction (XRD)

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## 2 Materials and methods

### 2.1 Materials

The chemical reagent used to prepare the mesoporous silica SBA-15 were Pluronics (P123, PEO<sub>20</sub>PPO<sub>70</sub>PEO<sub>20</sub>, average  $M_n \sim 5800$  Sigma-Aldrich, Singapore), tetraethyl orthosilicate (TEOS, 98% Sigma-Aldrich, Singapore), polyvinyl alcohol (PVA Sigma-Aldrich, Singapore), hydrochloric acid solution (HCl, 37% Merck), ammonium fluoride solution (NH<sub>4</sub>F), and heptane solution (C<sub>7</sub>H<sub>16</sub>) were received from J.T. Baker. All chemicals reagent which were analytical grade without further purification.

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### 2.2 Synthesis mesoporous silica SBA-15

SBA-15 was synthesized according to the improved procedure by Liang Chao et.al [4,5,9]. The previous study has assessed the properties of SBA-15 without co-surfactant PVA in the preparation process of Pluronic surfactant template [21]. A case study approach was near modified to synthesize SBA-15. For the first, 2.4 g of Pluronic and 0.027 g of NH<sub>4</sub>F were mixed in 1.3 M HCl solution as much 84 ml, followed by stirring at ambient temperature until the solution was clear. Then, the water-bath set at a temperature 10°C to prepare the Pluronic surfactant template for 1 h. In other solution, various of the amount PVA (1 g for the SBA\_1, SBA\_2, and SBA\_5; 2 g for SBA\_2

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Table 1: Summary of synthesis condition for SBA-15 samples.

SBA-15 samples	Time of hydrothermal treatment (h)	Amount of PVA (g)
SBA_1	20	1
SBA_2	20	2
SBA_3	24	1
SBA_4	24	2
SBA_5	96	1

and SBA\_4) were dissolved in 10 ml H<sub>2</sub>O at 60°C and mixed into the Pluronic surfactant template. In particular, 3.7 ml of TEOS precursor solution and 1.2 ml heptane were attached to the beaker. The Pluronic surfactant template and TEOS precursor solution were stirred to allow the solution to mix well for one day at room temperature in an open container. After that, the solution was transferred to the closed-teflon container for hydrothermal treatment at temperature of 100°C for certain time (20 h for SBA\_1 and SBA\_2; 24 h for SBA\_3 and SBA\_4; and 96 h for SBA\_5), it was then cooled to room temperature. The white gel products were precipitated by centrifuge to get the right product and washed it with deionized water until pH 7 was clear. Finally, to remove partial water, the right products were dried at 60°C for 24 h and calcined in the furnace at 550°C for 5 h to perfect the step process.

### 2.3 Characterization

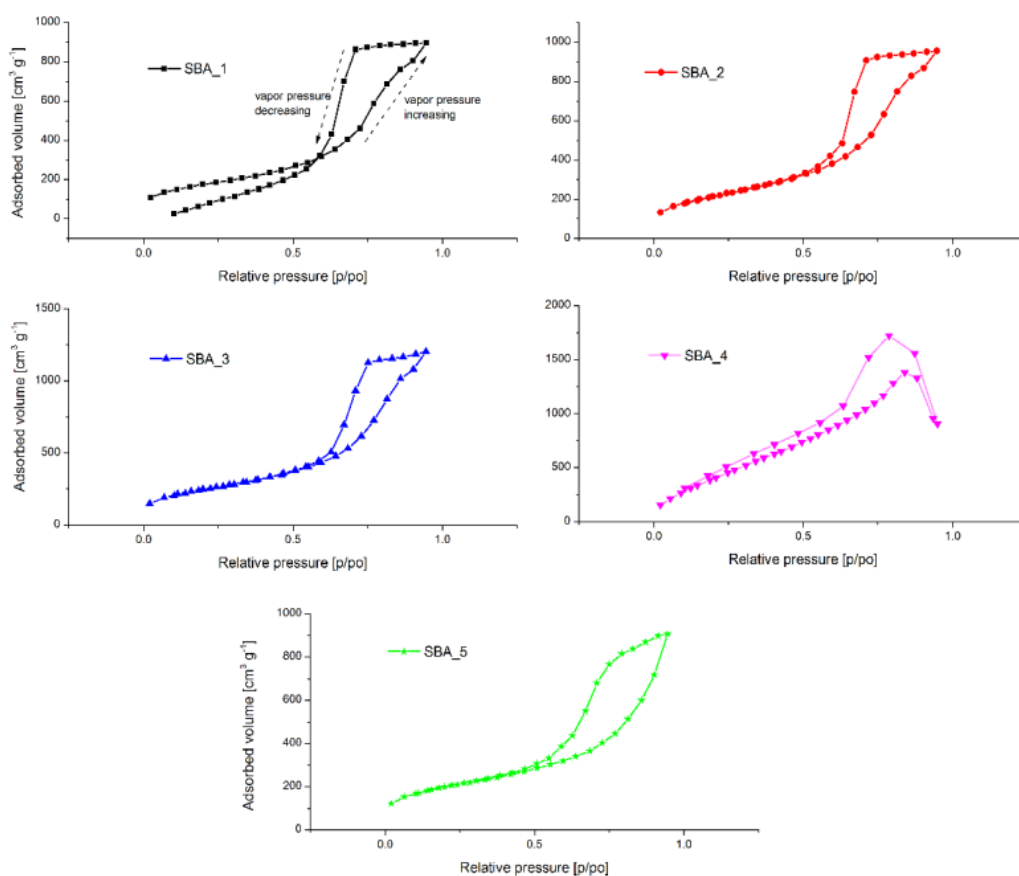
#### 2.3.1 Physisorption analysis

Physisorption analysis of SBA-15 samples were quantified at -196°C by using a Quantachroma NovaWin version 11.0 device. The physisorption analysis was measured at the purge process under vacuum at 300°C for 3 h. The amount of nitrogen adsorbed at a relative pressure  $[p/p_0] = 0.98$  was equivalent to analyzed the total pore volume of SBA-15 samples. The data from BJH pore size distribution desorption is required to determine exactly pore size distribution (PSD). The adsorption-desorption isotherm properties for analyzing specific surface area ( $S_{BET}$ ) was obtained using the multi-point BET data.

#### 2.3.2 Physicochemical properties

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Using the X-ray diffraction (XRD) and verifying at the actual pore structure properties, it was possible to identify the



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Figure 1: Nitrogen adsorption-desorption isotherm of SBA-15 samples.

crystal phase of SBA-15 samples. The instrument analysis XRD pattern was measured using Bruker D8 Phaser Diffractometer model with Cu  $\alpha$  radiation source with wavelength = 1.5406 Å,  $k\beta$  1.3922 Å, and run of the X-ray tube at 40 kV, 20 mA. The diffraction data were observed in the  $2\theta$  range from 5° to 90°, with a step size of 0.02° and the step time of 1 s. To analyze the surface morphology and the content of SBA-15 samples were characterized by using a scanning electron microscope (SEM with EDX) SU3500 model, running distance at 4940  $\mu$ m and 10 kV of electron voltage with the high energy electron beam. The content of Si and O spectrum were determined by SU3500 EDX type at 15 kV and lifetime 30 s. The instrument of 8400S SHIMADZU infrared spectroscopy was used to present data about chemical bond and functional groups of SBA-15 samples base on their characteristic absorption of infrared radiation in vibrational modes. For SBA-15 samples, we used scanned 450-4000  $\text{cm}^{-1}$  of the range

wavenumbers and ground together with KBr to the made pellet and placed into the DRIFT cell.

Ethical approval: The conducted research is not related to either human or animal use.

## 3 Result and discussion

### 3.1 Physisorption isotherm analysis

Surface area is a key property of mesoporous materials by physisorption methods [21]. The result obtained from the preliminary analysis of high surface area for SBA-15 is presented in Figure 1. Interestingly, the increased surface area of SBA-15 at higher  $p/p_0$  (0.7-0.9) is related to Type IV isotherm [22]. The nitrogen adsorption on samples occur at the beginning process  $P/P_0$  at the range of 0 to 0.5 was



**Table 2:** Physisorption analysis of SBA-15 samples with some variables.

SBA-15 sample	Conditions	$S_{BET}$ (m <sup>2</sup> /g)	Dp (nm)	Vp (cc/g)
1	PVA 1 g, 20 h	628	4.4	1.40
2	PVA 2 g, 20 h	780	3.8	1.48
3	PVA 1 g, 24 h	892	4.2	1.87
4	PVA 2 g, 24 h	1726	3.2	1.40
5	PVA 1 g, 96 h	699	4.0	1.40

adsorption in microporous initiated with the single layer of the material surface. On relative pressure of 0.6 to 0.9, there was parallel hysteresis loop adsorption-desorption, in which the adsorption of vapor pressure increasing is followed by desorption of vapor pressure decreasing simultaneously. It shows that the sample was mesoporous material and has uniform size pore

Figure 1 presents a typical isotherm, it is a relation of the amount of adsorbed volume and the relative pressure of adsorbed. A feature of the *Type IV* isotherm is typical for porous materials. At higher pressure, there is a hysteresis loop that can explain how the pore is shaped and identified with specific pore structure. All samples show a confirm step with a hysteresis loop corresponding type of H1 with narrow pore size distribution. Figure 1 provides some of the main characteristics of mesoporous materials. It is encouraging to compare this figure with that found by other authors [4–6].

The nitrogen sorption measurement by the Brunauer-Emmett-Teller (BET) method has the most widely used standard for the calculation of the surface area [22]. One unanticipated finding that the higher surface area of SBA\_4 sample of 1726 m<sup>2</sup>/g, the pore volume of 1.4 cc/g, and pore diameter of 3.2 nm. There are several possible explanations for this result. Change in the amount of PVA and the time for hydrothermal treatment were compared using physisorption analysis. The interaction of PVA with surfactant template during preparation process indicated that near significant effect on the micelles formed of P123. These results suggest that the diameter and pore volume for all samples are relatively stable. However, the interaction of PVA occurs on the surface area of silica. In the future investigation, it might be possible to use a different condition of time hydrothermal treatment and some variables synthesis of mesoporous materials. The correlation between the time of hydrothermal treatment and the amount of PVA by physisorption analysis is shown in Table 2.

Table 2 presents the experimental data based on the surface analysis, pore diameter (Dp, nm) and pore volume (Vp, cc/g). The results of the correlational analysis are presented in Table 2, the higher surface area of SBA\_4 generated the lower pore diameter. A positive correlation was found between surface area increases and the pore diameter decreases. The pore volume of the samples not significant, only in the range of 1.40 to 1.87 cc/g. The largest pore volume was SBA\_3 (PVA 1 g, 24 h), we can see that the amount of PVA did not effect expanding the pore volume. In the current study, comparing the role of PVA only affect to increase surface area, but no interactive with micelles formed from the surfactant template [4]. However, it is different for the time of hydrothermal treatment. The effect of time hydrothermal treatment and the amount PVA on the particle size distribution of mesoporous silica SBA-15 was explored, and the results are presented in Figure 2. The graph shows that there has been stable in pore volume and pore diameter for all the samples

shown in Figure 2(left), SBA\_3 (PVA=2 g, t=24 h) had a surface area of 892 m<sup>2</sup>/g and pore volume of 1.87 cc/g. However, the time of hydrothermal treatment increased the surface area and pore volume is decreased. This condition can be seen at SBA\_5 (PVA=2 g, t=96 h). In reviewing the literature, no data was found on the combination between the addition of PVA and adjusting of time hydrothermal treatment. The result of the correlation analysis is summarised in Figure 1. The addition of the amount PVA 2 g and time hydrothermal is raised from 20 h to 96 h, the surface area was increased but the pore volume and pore diameter were slightly more stable. It can be compared to Figure 1 for SBA\_1, can be explained that the time of TEOS as a precursor interacts with the surfactant template at the condensation step in teflon has not reached perfect time further work, this is an important issue, it requires the higher surface area, the larger pore volume and pore diameter we can do addition PVA 2 g by adjusting the time of hydrothermal treatment in the range 24 to 96 h.

### 3.2 X-ray diffraction analysis

Powder X-ray diffraction method was applied to characterize the crystal phase and structure of SBA-15 samples. The X-ray diffraction pattern was at 2θ between 5° and 90° for five SBA-15 samples. Figure 3 shows that the SBA-15 has been successfully formed based on the amorphous peak approach at 2θ = 23°, corresponding just to the planar (100) with hexagonal planar symmetry (p6mm) [23]. All the samples are amorphous material around 82%.

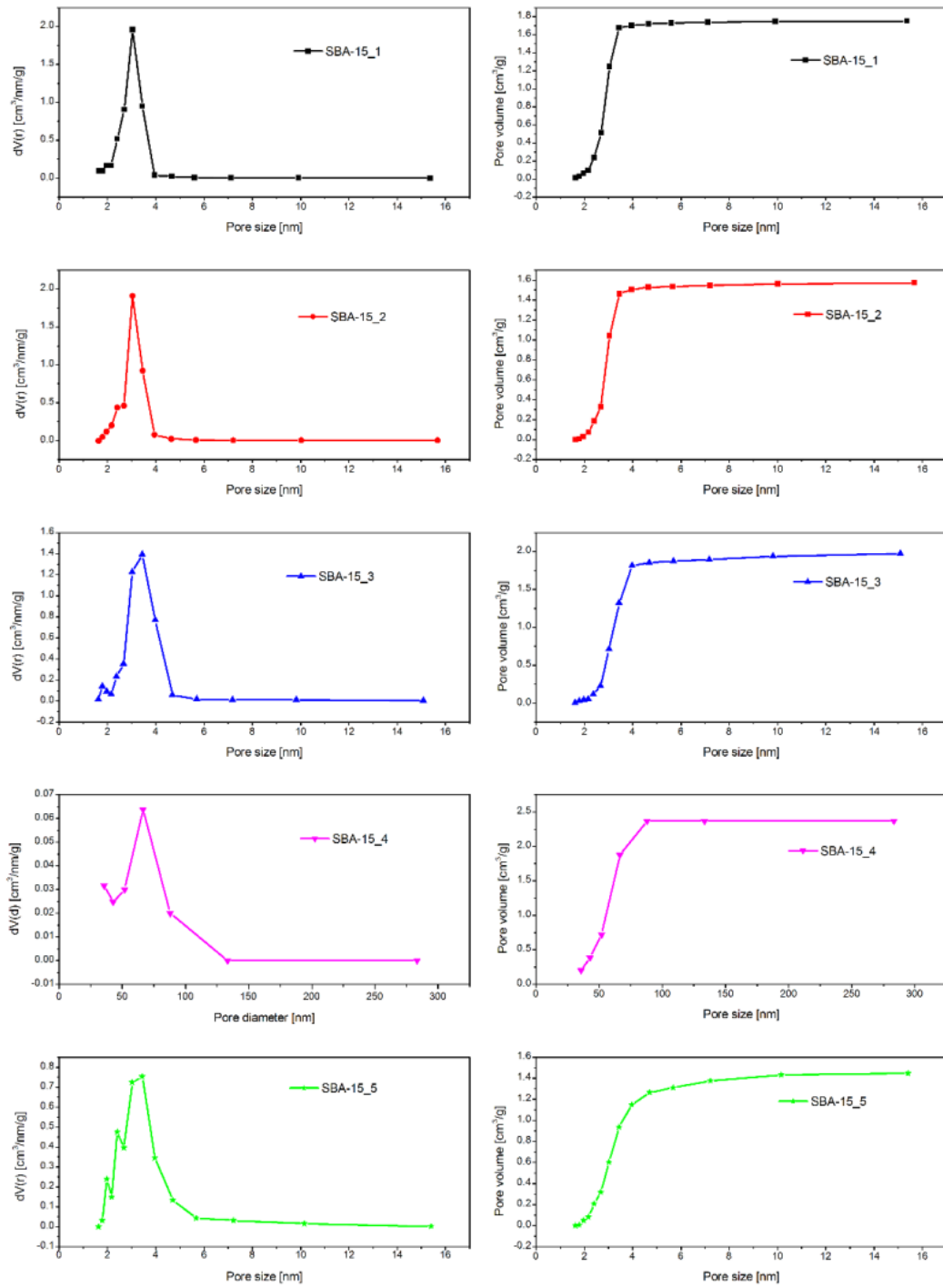
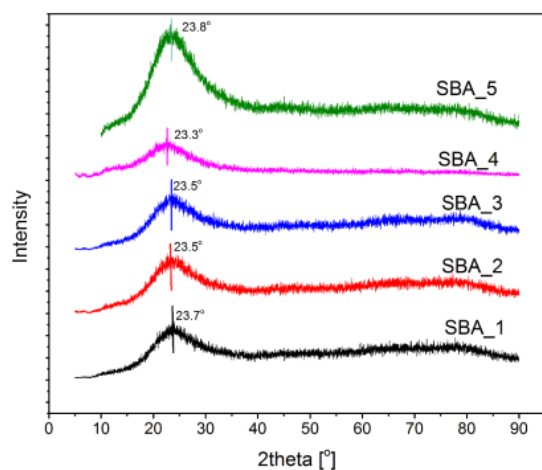


Figure 2: Pore size determination of SBA-15 samples. BJH desorption particle size distribution (left); the relation between pore size and pore volume (right).



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Figure 3: X-ray diffraction pattern on the SBA-15 samples.

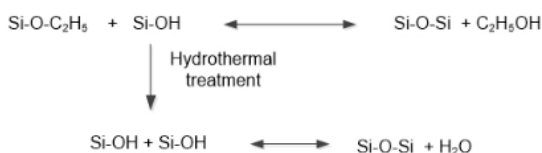


Figure 4: Reaction mechanism of silica.

Except for the sample of SBA\_1, it has been investigated that the crystal system is orthorhombic (not general properties of SBA-15) with space group  $pmmm$ ,  $d$  spacing of the silica is 20.5 Å corresponding to a unit cell parameter,  $a_0 = 20.57$  Å and  $b_0 = 9.74$  Å. This case can be explained that the amount of PVA 1 g for 20 h of the time hydrothermal treatment had not been reached to the hexagonal structure of SBA-15. This result diffraction pattern was corresponding to the match with COD-InorgREV204654. Although the exclusion of the crystal structure can reduce the effect of the amount PVA and the time of hydrothermal treatment, these result should be interpreted with caution [3,4]

In one well known recent experiment, limit on wide-angle X-ray diffraction (WAXRD) pattern was found to be diffusion straight peak of amorphous materials at  $2\theta = 23^\circ$  was reported [23]. As shown in Figure 3, the peak amorphous of SBA\_5 reported significantly more than the other samples. From the data in Figure 3, it is apparent that the length of time for hydrothermal treatment, the amorphous part can be increased. The amorphous part for the sample of SBA-15 were 81.4%; 83.4%; 82.6%; 82.4%, and 83.6% respectively. The value of amorphous composition suggests that a weak correlation may exist

between the amount PVA and the time for hydrothermal treatment. A further study with more focus on the amount PVA is therefore suggested.

Figure 5 presents the result obtained from the formation of the surfactant template. The template of mesostructure SBA-15 can be arranged during the preparation process of Pluronic. Micelle formation with Pluronic plays an important role in the structure of mesoporous materials. The critical micelle concentration (CMC) of the surfactant template can do in several varieties of pore size and mesostructure. The influence of addition PVA in the preparation process of mesoporous silica materials has increased the surface area [4]. The next interaction between TEOS as a silica precursor and Pluronic by hydrothermal treatment to silica stability materials [9]. Calcination process to remove the surfactant template. The end of the process, silica mesoporous SBA-15 was obtained with high surface area and high thermal stability. Interestingly, the various preparation surfactant template and addition PVA were observed to huge potential application in many industries.

### 2 3.3 FTIR spectroscopy analysis

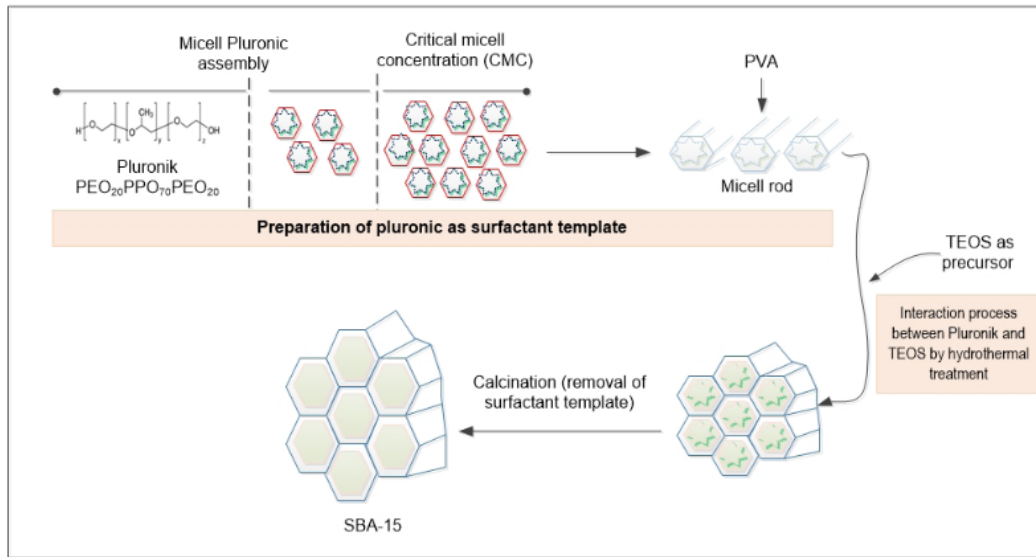
The function group and feature of the SBA-15 samples were identified base on FTIR analysis. The FTIR spectroscopy analysis of SBA-15 has verified that the band absorption of spectra from Si-O, Si-O-Si, Si-OH, and -OH were performed. The spectra of the samples can be compared for vibration peak of the characteristic silica SBA-15. The band absorption peaks from the Figure 4 can be seen 477  $\text{cm}^{-1}$ , 817  $\text{cm}^{-1}$ , 1215  $\text{cm}^{-1}$ , 1641  $\text{cm}^{-1}$ , and 3462  $\text{cm}^{-1}$  were characteristic peaks IR for SBA-15 materials

Spectra of all samples are shown in Figure 6, that the peaks at 816  $\text{cm}^{-1}$  with strong intensity by the presence of Si-O-H groups [23]. The bending of the O defined near at 478  $\text{cm}^{-1}$  with stretching vibration of the existence of silanol groups. The sharp peak at 1646  $\text{cm}^{-1}$  indicates that the formed -OH groups (H-O-H) [15,25]. The bands at the peak 3468  $\text{cm}^{-1}$  were defined to the band group H-O-H ( $\text{H}_2\text{O}$  molecules) [26]. In fact, the characterization of SBA-15 samples is identified with similar spectra in the mesostructure of the various synthesized of SBA-15.

### 3.4 Morphology analysis

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The scanning electron microscopy (SEM) image of the SBA-15 samples (SBA\_3 and SBA\_5) is shown in Figure 5





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Figure 5: Illustration of the formation mesostructure of SBA-15 [6,24].

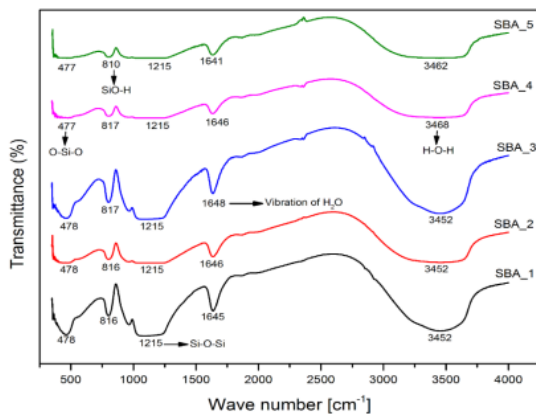


Figure 6: FTIR spectra of the SBA-15 samples.

successively. SEM analysis was incredible analysis for showing the surface morphologies of SBA-15 samples. Figures 5 shows that the mesoporous silica SBA-15 consists of the narrow pore with well ordered hexagonal arrays of mesoporous materials [25,27,28]

The comparison between Figure 7(a) and 7(b) reveals that the increased the time of the hydrothermal treatment can reduce the aggregate into well ordered porous materials. The SEM image of SBA\_5 gives an expression that the sample relative uniform. The longer hydrothermal treatment (96 hours) caused the more number of a particle formed with smaller size, it was caused by the interaction

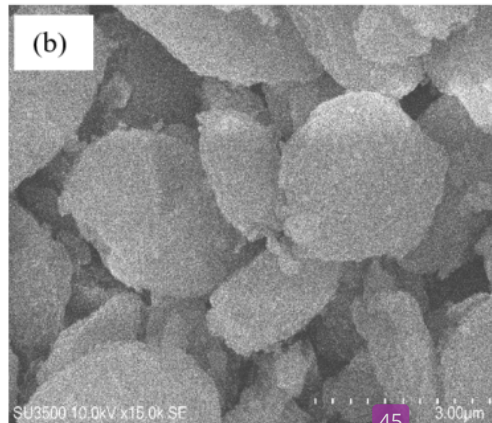
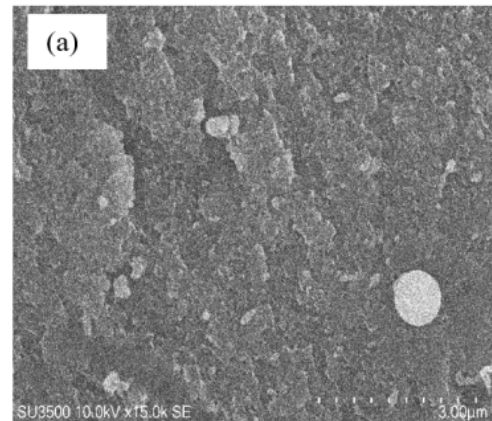


Figure 7: SEM image of the SBA-15 samples: (a) SBA\_3 and (b) SBA\_5.

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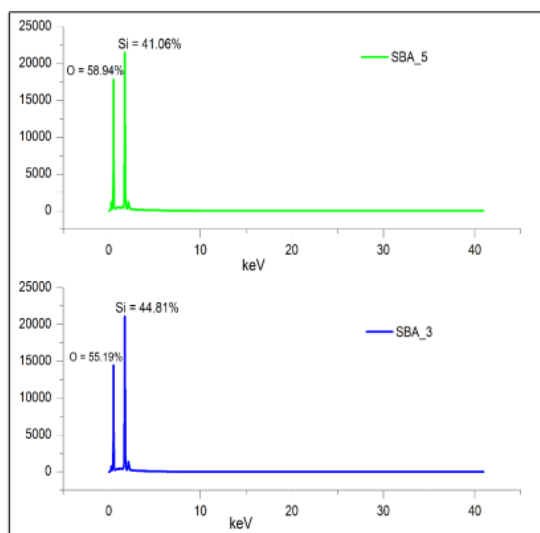


Figure 8: SEM EDX of the SBA-15 samples.

time between micelle surfactant and precursor formation during hydrothermal treatment. The resulting analysis of SEM has same correlation with XRD analysis, in which SBA\_5 (amorphous part = 83.6%) has a higher amorphous level compare to SBA\_3 (82.6%)

Compositional analysis of surface morphology and topology from SBA-15 is considered by SEM/EDX as shown in Figure 8. The EDX spectra of SBA\_3 and SBA\_5 indicated the presence of Si = 44.81% and oxygen = 55.19%, for SBA\_5 indicated the weight composition of Si = 41.06% and the composition of oxygen are 58.94%. The percentage of silica in the EDX analysis was identified that the synthesized silica material had been successful with uniform composition. This result can be proven by physical-sorption analysis, which the parallel process adsorption by desorption simultaneously

## 4 Conclusion

The high surface area of SBA-15 was synthesized base on an addition to the amount of PVA and adjusting the time of hydrothermal treatment. Physicochemical properties have been characterized by XRD, FTIR, and SEM/EDX. The BET method was found the higher surface area from the SBA\_4 by addition of 2 g PVA and 24 h for the time hydrothermal treatment. The addition of a little amount of PVA and a short time hydrothermal treatment (SBA\_1) can be changed to orthorhombic crystal structure and has

a lower surface area than all samples. It can be suggested that the silica mesoporous SBA-15 will have potential application prospect in catalysis, storage, and adsorbent.

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**Conflict of interest:** Authors declare no conflict of interest.

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