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By Ridhawati Ridhawati

Journal of Physics: Conference Series

PAPER • **OPEN ACCESS**

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To cite this article: Indah Raya *et al* 2021 *J. Phys.: Conf. Ser.* **2049** 012083

Prepare and Utilize Mesoporous Silica SBA-15 for Efficient Photocatalytic Adsorption of Methylene Blue and Copper(II)

Indah Raya[1], Nursiah La Nafie[1], Ridhawati Thahir[2,*], M. Yasser[2], Syarif Ismail[3]

[1]Department of Chemistry, Faculty of Mathematics and Natural Sciences, Hasanuddin University, Makassar, 90245, South Sulawesi, Indonesia.
[2]Department of Chemical Engineering, Politeknik Negeri Ujung Pandang, Makassar, 90245, South Sulawesi, Indonesia.
[3]Department of Agricultural, Politeknik Pertanian Negeri Pangkajene Kepulauan, Pangkep, 90655, South Sulawesi, Indonesia.

ridha331@poliupg.ac.id

Abstract. This work has provided a novel technique for preparation, characterization, and utilization of mesoporous silica SBA-15 in waste treatment to remove methylene blue as an organic pollutant and copper(II) as inorganic pollutant sampling with a photocatalytic adsorption system. To process of mesoporous silica SBA-15 was prepared by the sol-gel method for Pluronic as a surfactant template and following the hydrothermal process to high interaction between Pluronik and tetraethyl ortho silicate (TEOS) as precursor reagent. The proceed materials were characterized by the surface analyzer, X-ray diffraction, and Fourier transform infrared. Mesoporous silica SBA-15 was obtained with hexagonal structure having 72 percent amorphous content, high surface area, large pore-volume, approximately 948 m^2/g, and 1.3 cm^3/g. The second major finding was that mesoporous silica SBA-15 have a high photocatalytic adsorption capacity to remove methylene blue and copper(II). These present results suggest several courses of action in order to utilize SBA-15 samples in waste treatment.

1. Introduction

Mesoporous materials are a product of science and technology development that can produce produce new materials which different properties from simillar product of macroporous. One of the most significant current discussions in nanotechnology development is mesoporous materials, from silica family Santa Barbara Amorphous (SBA-15), SBA-16, mobil composition of matter No. 41 (MCM-41), and MCM-48. The mesoporous materials are important for a wide range of scientific and industrial processes. Mesoporous silica SBA-15 materials are among the most widely used for more applications, such as filters, catalysts, adsorbents [1], capacitors [2], and supported framework material [3]. Utilizing mesoporous silica SBA-15 as adsorbent, has long been a question of great interest in a wide range of fields [4,5]. The adsorption capacity of silica SBA-15 has been the subject of much systematic investigation.

Recently investigators have examined the effects of preparation variables of SBA-15. Previous research comparing the time and temperature of hydrothermal treatment has found high surface area analysis [6,7]. Determining the impacts of surface area on photocatalytic adsorption for the future to increase the application efficiency of silica SBA-15. It has been suggested that high surface area of silica SBA-15 materials can be applied as an adsorbent in environmental pollution removal systems such as methylene blue, azo color effluent [8,9], removal of copper(II) [10,11] and heavy metal of Pb(II) from water solution [12].

Methylene blue dye is a synthetic organic substance that is employed as a dye in the batik craft, paper industry, and cosmetics. Effluent industrial waste that still contains MB can pollute the environment. Based on Government Regulation No. 82 of 2001 concerning water quality criteria, the parameter of the organic matter content of MBAS (methylene blue active substance) in water is 200μg/L. Some waste treatment systems before being discharged into the environment can be carried out through precipitation methods using coagulant, ultrafiltration, photocatalytic degradation, and adsorption process [13]. The system for processing pollutants through the deposition process followed by coagulation can be applied if the pollutant content is more than 1000 mg/L [14]. The adsorption method is an alternative treatment system for pollutants to adsorb methylene blue dye [15]. Silica SBA-15 used as methylene blue and copper(II) adsorbents are based on catalytic properties, crystallinity, crystalline phase, and surface active side. Methylene blue dye and copper(II) photodegradation are a degradation processes employing visible light. In addition, silica SBA-15 materials are non-toxic oxides, have high thermal stability, and possess high oxidation capabilities. The photodegradation process is applied to remove pollutants from organic pollutants and copper(II) as inorganic pollutant sampling with photocatalytic adsorption system into compounds that are more environmentally friendly.

In this study, preparation of silica SBA-15 was carried out using Pluronic as a surfactant, and TEOS precursor as a source of silica in sol-gel method and following hydrothermal treatment. Characteristic analysis of silica SBA-15 includes determining particle size, phase, and crystal structure. The application of silica SBA-15 as adsorbent methylene blue dye and copper(II) was conducted to assess the extent to which adsorption efficient.

2. Methodology

2.1. Chemical reagents

The raw material of the chemical reagents which were analytical grade without further purification. The raw materials of mesoporous silica SBA-15 were synthesized using Pluronics (P123, Sigma-Aldrich, Singapore), tetraethyl orthosilicate (TEOS, 98% Sigma-Aldrich, Singapore), hydrochloric acid (HCl, 37% Merck), ammonium fluoride (NH_4F), and heptane were obtained from J.T. Baker.

2.2. Preparation of silica SBA-15 adsorbents

A variety of methods are used to prepare mesoporous silica SBA-15. Each has its advantages and drawbacks. Many research has prepared mesoporous materials to synthesis silica SBA-15. The procedure of this work was modified from Thahir et al, Liang Cao et al, and Emma et al [7,16,17]. The chemical reagents were substituted for TMOS as a precursor with TEOS and triisopropyl benzene as micelle expander with heptane. In order to understand how Pluronic as surfactant template regulates the mesoporous structure, a series of procedures was done. In one step, Pluronic (2.4 g) and ammonium fluoride (0.027 g) were added to 1.3 M of HCl solution (84 ml). Following this, the mixture was stirred until clear at ambient temperature. After that, prepare the initial temperature of the solution at 15^{o}C for Sample E and 10^{o}C for sample F and put it in a water bath for 1 h. In the other place, heptane (1.2 mL) was dissolved in TEOS (3.7 mL) as silica precursor and added to the surfactant solution. The samples were stirred overnight at the initial temperature. In this work, the interaction of the surfactant template and silica precursor with the hydrothermal process in a closed teflon. To prepare initial temperature and condition of hydrothermal treatment are set out in Table 1. The gel product was washed with deionized water until pH 5 was obtained. In the end, the products were dried at 60^{o}C overnight. The calcination process can be done to remove the surfactant template at 550^{o}C for 5 h.

Table 1. Preparation condition of silica SBA-15

No	Name of sample	Initial temperature, °C	Temperature of hydrothermal treatment, °C	Time of hydrothermal treatment, hour
1	SBA-15 E	15	120°C	48
2	SBA-15 F	10	100°C	96

2.3. Characterization
Using X-ray diffraction (XRD) and looking at the actual mesostructure of silica SBA-15 samples was obtained by Bruker D8 Phaser diffractometer system with Cu Kα radiation with 1.5406 Å, kβ 1.3922 Å, 20 mA, and run at 40 kV. The XRD patterns were analyzed with wide-angle in the range 5 to 90° of 2θ at any rate of 0.02° for the time step 1 s. To determine specific surface area and parameters of pore size, a question asking nitrogen adsorption-desorption isotherm with Quantachroma NovaWin instruments 11.0 at -196°C was used. To calculate pore size distribution, adsorption data with the BJH method was developed. The specific surface area was determined with the multi-point BET method [18,19]. The spectrum data was recorded using Prestige-21 Shimadzu infrared spectroscopy to identify various functional group and types of bonding of the samples in the range of wave numbers 500-4000 cm^{-1}.

2.4. Application of silica SBA-15 on adsorption experiments
To remove organic and inorganic pollutants, methylene blue dye (MBD) and Cu(II) were preferred as a sampling of pollution control by mesoporous silica SBA-15. The adsorption process was prepared according to the procedure used by Thahir et al. [20]. Two different stock solutions of 100 mg/L MBD and 500 mg/L of Cu(II) were provided to dissolved in a variety of requisite times. In order to investigate the adsorption efficiency of silica SBA-15 as adsorbent, specified the varieties of adsorption times are 10, 20, 30, 40, 60, 120, and 180 minutes. A batch adsorption system was established to evaluate the effect of silica SBA-15 adsorbent at apparent pH 7 [21] in the box with 150 Watt Hg lamp. Firstly, 100 mg of silica SBA-15 adsorbent was dissolved in 100 mL of MB or Cu(II) solution with continuous stirred at 200 rpm at room temperature. After that, centrifugation and filtration with Whatman paper filter to separate the solution. The filtrate was analyzed by spectrophotometer UV-vis double beam at the wavelength λ_{max} = for MB solution and λ_{max} = for Cu(II) solution.

Furthermore, to determine the percentage sorption (percent removal) of pollutant MB and Cu(II) on silica SBA-15 adsorbent were calculated as follows the equation:

$$\text{Adsorption efficiency, \%} = \frac{(C_i - C_o)}{C_i} \times 100$$

Where C_i and C_o are the initial and final pollutant concentration in the direct solution (mg L^{-1}), respectively.

3. Results and Discussion

3.1. Characteristic analysis of silica SBA-15
Fourier transform infrared (FTIR) analysis was applied to determine the bond spectrum that occurred in the functional groups of Si-O, Si-OH, -OH, and Si-O-Si. The spectrum data of the silica SBA-15 sample were used to analyse the vibration peak of the materials. Figure 1, shows the Si-O spectrum at a wave number of 900-970 cm^{-1}. The bond spectrum 800-810 cm^{-1} indicates Si-O-Si. While Si-OH is in the spectrum 3440-3500 cm^{-1} groups and water absorption on the surface of silica SBA-15 materials [22].

All spectrum data of functional group silica SBA-15 sample described as using some sort of hydrothermal treatment procedure were straight intensity in the analysis. One practical advantage of using a case study approach is that FTIR analysis of silica SBA-15 related for adsorption application.

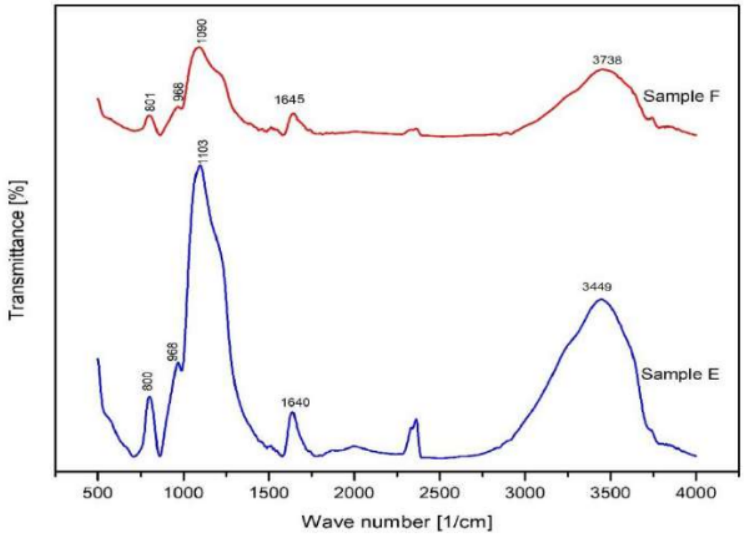

Figure 1. The spectrum FTIR analysis of SBA-15 samples

Analysis of phase characteristics and shape of the silica SBA-15 samples, the crystal structure was carried out using wide-angle X-ray diffraction. Figure 2, shows the peak shape of XRD diffraction results of SBA-15 F sample is sharper when compared to SBA-15 E samples.

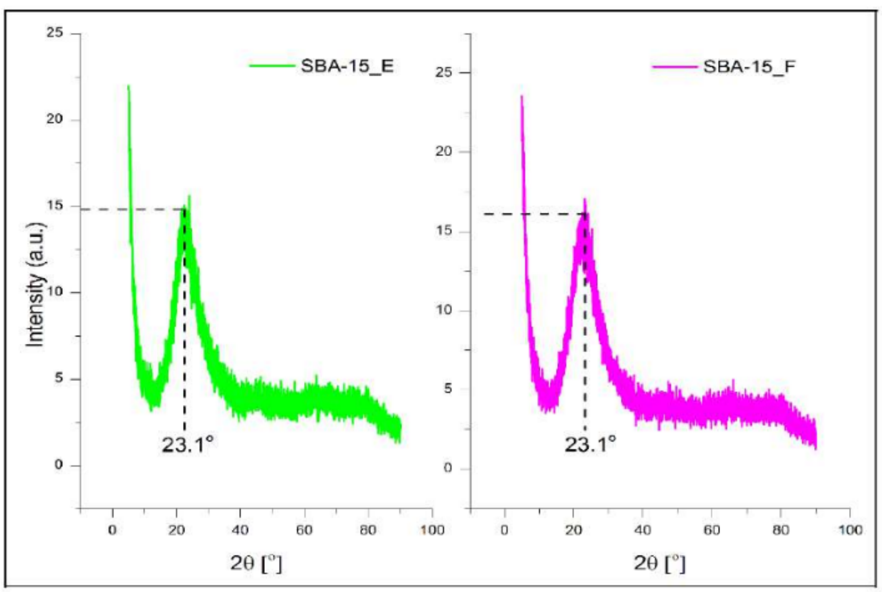

Figure 2. The diffraction of XRD analysis of SBA-15 samples

This is consistent with research conducted by Morsi et al. 2018. Heating at higher temperatures will produce more perfect crystals [23]. The crystal structure of the silica SBA-15 E and SBA-15 F samples has the same hexagonal shape and amorphous phase. In addition, the hydrothermal process in closed teflon can produce small nucleation. Particle size control can be conducted from precursor and surfactant preparations.

The one peak diffraction of the SBA-15 sample in Figure 2 occurred at 2θ: 23.1^{o} which was correlated with the intensity of the planar (100). The X-ray diffraction analysis D8-Phase Bruker have amorphous composition materials around 70.9% for the SBA-15 E and 70.2% for the SBA-15 F. The mesostructure analysis of silica SBA-15 samples is not significantly [6].

3.2. Compare multi-point BET analysis of silica SBA-15 as adsorbent application

The first set of questions objective analysis the surface area of silica SBA-15. Adsorption-desorption isotherm method for measuring pore size, pore volume, and specified surface area by multi-point BET. Figure 3 shows the result obtained from the adsorption-desorption isotherm preliminary analysis of silica SBA-15. In Figure 3, there is a clear trend of decreasing surface area for SBA-15 E sample. Taken together, these results provide important insights into the preparation condition of silica SBA-15. Comparison of the findings with those of other studies confirms for pore volume of silica SBA-15 samples [7]. Consistent with the kinds of literature, all SBA-15 samples have reported of hysteresis loop model of H1 and isotherm curve are IV type [24].

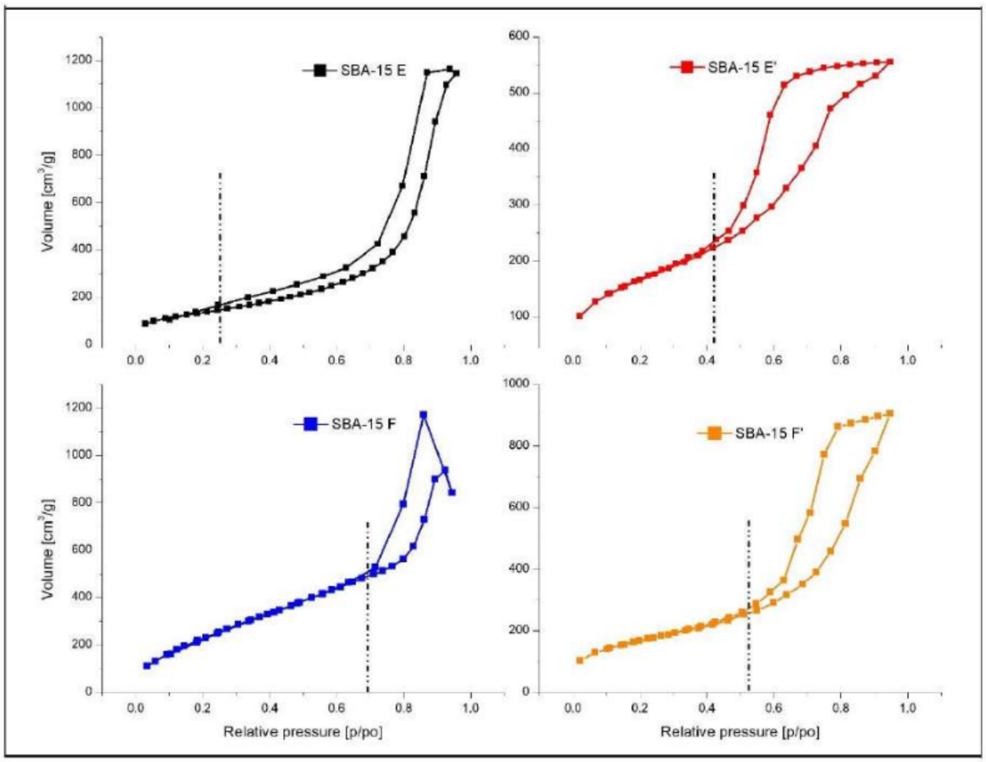

Figure 3. The adsorption-desorption isoterm curve of SBA-15 samples

What is surprising is that the pore diameter of SBA-15 E samples are 14.33 nm. This result may be explained by the same fact of the interaction of the Pluronic surfactant template and TEOS precursor at hydrothermal treatment condition [7]. This is a particularly useful finding that the large pore diameter can be applied as an adsorbent to remove organic and inorganic pollutant.

Table 2. Multi-point BET analysis of silica SBA-15 sample

No	Name of sample	Multi-point BET analysis	Before adsorption application	After adsorption application (SBA-15 E' and F')	Confirm of BET analysis
1	SBA-15 E (adsorption of MBD)	S_{BET}, m^2/g	494	493	stable
		Pore volume, cm^3/g	1.70	0.86	decreased
		Pore diameter, nm	14.32	2.41	decreased
2	SBA-15 F (adsorption of Cu)	S_{BET}, m^2/g	948	594	decreased
		Pore volume, cm^3/g	1.30	1.30	stable
		Pore diameter, nm	5.50	4.69	stable

The adsorption-desorption isotherm process was carried out to reduce the concentration of MBD and Cu(II) in the solution. Determination of initial and final concentration was employed using a uv-vis spectrophotometer.

Table 3. Preparation condition of silica SBA-15

Pollutant model	Name of sample	Adsorption times, minute	Initial concentration, ppm	Final concentration, ppm	Efficiency of the adsorption, %
MBD	SBA-15 E	10	100	25.11	74.89
		20		14.34	85.66
		30		12.26	87.74
		40		4.63	95.37
		60		3.85	96.15
		120		3.85	96.15
		180		3.73	96.27
	SBA-F	10	100	18.21	81.79
		20		14.00	86.00
		30		15.01	84.99
		40		11.98	88.02
		60		11.47	88.53
		120		11.47	88.53
		180		10.45	89.55
Cu(II)	SBA-15 E	10	500	13.57	97.23
		20		12.67	97.45
		30		12.52	97.50
		60		8.19	98.36
		120		7.45	98.51
		180		6.70	98.66
	SBA-F	10	500	5.21	98.96
		20		4.46	99.11
		30		2.67	99.47
		60		1.48	99.70
		120		0.43	99.91
		180		0.43	99.91

The application of silica SBA-15 as adsorbent to trapt effluent industrial waste. The efficient photocatalytic adsorption used to identify the multi-point BET method involved surface area, pore diameter, and pore volume of silica SBA-15. Table 3 compares the intercorrelation among the two different stock solutions of 100 mg/L of MBD and 500 mg/L of Cu(II).

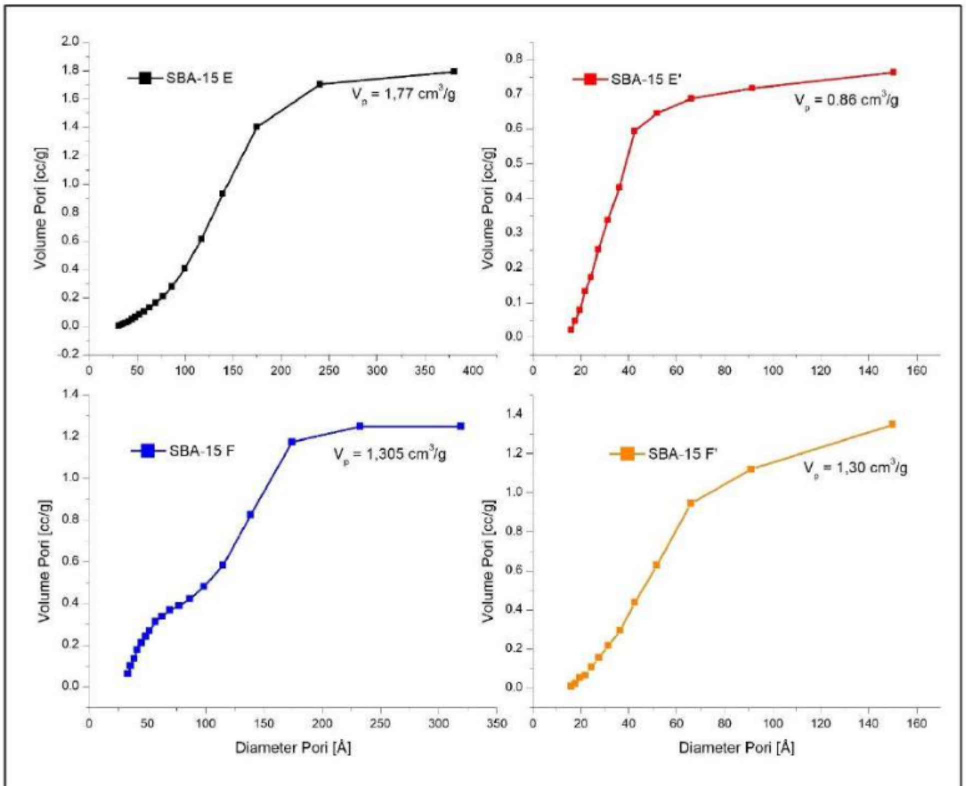

Figure 4. The adsorption-desorption isotherm curve of SBA-15 samples

The methylene blue dye and copper(II) adsorption process on the surface of silica SBA-15 occurs because the adsorbent material is mesoporous material which allows the adsorption of industrial waste into the silica SBA-15 pore. In addition, there is an interaction of Si-O and Si-O-Si groups as active groups which play a role in the MBD and Cu(II) adsorption process. The studied parameter of the adsorption process was the efficiency of adsorption of silica SBA-15 **[20,25]**.

Prior to analysing the interview data in Table 2 and Figure 4, the results were confirmed for a sampling of pollution control. On completion of the adsorption process, the multi-point BET of parameter estimation was carried out. When removing MBD as an organic pollutant model, it was important to large pore diameter and pore volume. Finally, questions were asked as to the role of the high surface area of SBA-15 can utilize to adsorb Cu(II) inorganic pollutant model. Overall, these results indicate that silica SBA-15 materials have more potential application in efficient photocatalytic adsorption of methylene blue dye and copper (II) solution. Further studies are required to establish the viability of adsorption capacity and morphology analysis of silica SBA-15.

4. Conclusion

The purpose of the current study was to determine multi-point BET, characteristic analysis of silica SBA-15, and evaluate adsorption-desorption isotherm curve for measuring pore size, pore volume, and specified surface area on photocatalytic adsorption removal of methylene blue dye and copper (II) as pollutant industrial model. The current data highlight the importance of large pore diameter and pore volume to trapt methylene blue dye and high surface area for active site to reduce copper (II).

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