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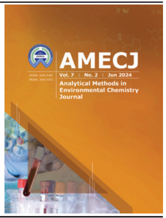

Adsorption of methylene blue into modified mesoporous silica from palm oil boiler ash

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ABSTRACT

The adsorption of methylene blue dye on modified mesoporous silica based on palm oil boiler ash (MS-POBA) as an adsorbent with a methyl ester sulfonate (MES) was investigated. MS-POBA and MES as a template improved the adsorption capacity of methylene blue by increasing the pore size of boiler ash silica. The characteristics of the material were determined using FTIR, XRD, BET, and SEM-EDX analyzers. The adsorption of methylene blue on the MS-POBA adsorbent was determined using a UV-Vis spectrophotometer. The MES as a template pasted to MS-POBA, could be increased surface area, pore diameter, and volume. In optimized conditions, pH, the adsorbent mass, the adsorption time, the methylene blue concentration, the adsorption capacity, and recovery were obtained at 7.0, 0.03 g, 45 minutes, 20 mg L^{-1}, 15.578 mg g^{-1}, and 96.9%, respectively. The adsorption of methylene blue on boiler ash silica with an MES template follows the pseudo-second-order kinetic model with a value of R^2 = 0.999 and Langmuir isotherm adsorption model.

1. Introduction

Indonesia has a large distribution of palm oil-producing factories that sustainably produce palm oil, including the Sumatra and Kalimantan provinces [1]. For this reason, it is necessary to utilize waste properly and optimally from palm oil mills as well as solid waste in the form of biomass, namely empty fruit bunches, fiber, and shells. Several potential uses exist for palm oil waste biomass in Indonesia [2]. The fibers and shells as solid waste are commonly applied as boiler fuel to create heat and mechanical power in palm oil processing. The problem that arises is the residual combustion in the boiler in the form of shell ash, which is disposed of as solid waste and causes disruption to the environment and health. Boiler ash contains 40.60% SiO_2, 19.60% CaO, and 3.71% Al_2O_3 [3]. The potential of boiler ash to produce silica (SiO_2) depends on the number of silica and the application for industry, such as catalysts, adsorbents, and filter media. Silica has different pore sizes, surface areas, particle sizes, and pore volumes. The addition of a template can modify the pore size of silica [4]. Mesoporous silica synthesis is carried out by adding an organic compound, one being a surfactant, and then the calcination process is carried out to remove the surfactant [5]. Mesoporous silica can be used in adsorption because it has high thermal stability, large pore volume, small pore diameter distribution, and high surface area [6]. Templates are structure-directing agents used to create hollow porous structures, where the gap in silica will be filled by the template, then after the template is removed, it allows for the formation of hollow

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particles with newly created pores [7]. MES anionic surfactant was added as a template because it has several advantages: economical use, low toxicity, and rapid biodegradability [8]. MES is an environmentally friendly green surfactant, comes from renewable materials, can be decomposed naturally, is made from vegetable oil, is relatively stable at reservoir temperatures, has relatively low adsorption on reservoir rocks, and can be produced economically [9]. The alkyl chain length, the degree of surfactant ionization, and the ratio of co-structure-directing agent (CSDA) and surfactant contribute to the measurement of silica pores. MES is an anionic surfactant with a long alkyl surfactant chain that is important in determining the size of micelles and mesopores [10]. Industries that produce wastewater, such as textiles, paper, plastic, printing, food, and cosmetics, can cause environmental problems. One of them, serious attention, is to be focused on the removal/separation of the coloring components from wastewater. The available methods for removing dyes from wastewater are adsorption, membrane separation, coagulation, electrochemical processes, reverse osmosis, chemical oxidation, and aerobic and anaerobic microbial degradation of dye components [11]. In this research, the adsorption method was chosen because adsorption effectively reduces negative environmental due to pollutants such as metals, dyes, and organic substances. It is environmentally friendly, simple, and relatively inexpensive [12]. Environmental impact, economic profit, and adsorption efficiency are the main parameters when selecting adsorbents. Various adsorbents derived from agricultural residues and industrial wastes have been used to remove various hazardous components from wastewater. Synthesis of modified adsorbent has been widely carried out to match the substance to be separated so that the adsorption process is more effective and efficient in its use [13,14,15]. However, the intensive power requirements, the evolution of toxic by-products, and the high cost make most methods less feasible in large-scale plants for removing dye components from wastewater [16]. Dye waste can cause harmful effects and can also endanger health if the waste is immediately disposed of without any prior processing. Methylene blue is widely found in wastewater. Methylene blue is a cationic dye with a heterocyclic aromatic chemical compound structure. Methylene blue dye influences the environment due to its highly toxic effects and is carcinogenic [17]. Methylene blue is allowed in the water at a 5–10 mg L^{-1} concentration. The Declare of the Environment Minister of the Republic of Indonesia (KEP-51/MENLH/10/1995) concerns the quality standards of industrial wastewater [18]. Also, nanotechnology methods were used to remove organic materials from different samples [19-21]. Methylene blue is a hazardous waste. One of the waste treatments in the aquatic environment is by adsorption using solid waste from palm oil mills, namely burning coconut shells, which produce boiler ash.

Increasing the pore size of boiler ash silica using the MES template was investigated. This research prepared mesoporous silica from palm oil boiler ash (MS-POBA) as an adsorbent through a simple method of microemulsion polymerization. Initially, MES as a templating agent was added during the synthesis of mesoporous silica, MES is kept in a silica framework and then calcinated to be removed. MS-POBA will provide an active site to improve the adsorption performance of methylene blue dye. Adsorption data will be explained based on the physicochemical properties of MS-POBA and methylene blue dye at equilibrium.

2. Material and Methods

2.1. Materials

The material used in this study was the boiler ash of the palm oil factory. Also, the reagents such as HCl (Merck, 6 M, CAS No.: 7647-01-0), NaOH (Merck, 6 M, CAS No.: 1310-73-2), Methyl Ester Sulfonate (MES, Sigma Aldrich, CAS No. 93348-22-2), 3-Aminopropyl Trimethoxysilane (APMS, Sigma Aldrich, CAS No.: 13822-56-5), and methylene blue dye (Merck, CAS No. 61-73-4) were prepared.

2.2. Instrument

The instruments include Fourier Transform Infrared Spectrophotometer (FTIR, 8201PC Shimadzu), X-Ray Diffraction (XRD, Bruker D8 Advance ECO), Brunaur, Emmet, and Teller (BET, Gemini VII Version 5.03 Serial 2037), Scanning Electron Microscopy-Energy Dispersive X-Ray (SEM-EDX, JEOL JSM-6510LA), and UV-Vis Spectrophotometer (Thermo Scientific Orion Aquamate 8100) were used. Also, a set of glassware, analytical balance, pH meter, filter paper and Whatman 42, 250 mesh sieve, porcelain cup, magnetic stirrer, hotplate, measuring flask, glass stirrer, thermometer, a set of tools for reflux, oven (Kirin KBO-190RAW), and furnace (Fisher Isotemp Muffle Furnace Model 184) were used.

2.3. Synthesis of Adsorbent

2.3.1.Boiler ash purification

Boiler ash from the palm oil factory was weighed, and 6 M HCl was added. The mixture was boiled at 110 °C for 3 hours and then was allowed to stand until it cooled down. The boiler ash was filtered using a vacuum filter and washed with distilled water until the neutral pH. The boiler ash was dried in an oven and then heated using a furnace. The boiler ash is cooled and weighed.

2.3.2.Extraction of Silica with NaOH

The purified ash was weighed at as much as 10 g and put into a beaker, and then 60 mL of NaOH 6 M was added. The mixture was heated while stirring, carried out with a stirrer until half of the initial volume evaporated. Then, it was allowed to stand at room temperature until it cooled down. The mixture was filtered with Whatman paper, taking a sodium silicate solution as a filtrate. The HCl (2 M) was added to the filtrate drop by drop until the pH was neutral and a white gel was formed. The sol-gel that has been formed is allowed to stand for 18 hours, or process aging was carried out so that the gel becomes mature and stiff. The solid precipitation product was washed with distilled water and filtered using a vacuum filter, then dried in an oven before the calcination process at a temperature of 500 °C.

2.3.3. Synthesis of MS-POBA

The synthesis of MS-POBA with a mesoporous structure was carried out by preparing solution A containing 0.98 g MES as a template, then adding 90 mL of distilled water and 9 mL of 0.1 M HCl. The solution was stirred at 40 °C for 1 hour. Solution B, which contained 2.4 g of silica, 5 mL of methanol, and 1.67 mL of APMS, was dissolved in 5 mL of methanol. Then, solution B was added to solution A, accompanied by stirring for 1 hour. Solutions A and B were refluxed at 70 °C for 5 hours. The solid precipitation product is washed with distilled water, then the solution is filtered, and the residue is rewashed with distilled water until the pH is neutral. After being neutral, the residue was dried in an oven at 90 °C for 12 hours. Then, it was calcined at 550, 750, and 950 °C to determine the optimum temperature.

2.4. Adsorption procedure

As Figure 1, the optimum pH condition was measured by preparing methylene blue solution with a concentration of 20 mg L^{-1} and then adjusting the pH solution to 4, 5, 6, 7, and 8. Each solution was added 0.05 g MS-POBA with and without the MES template, then stirred for 60 minutes. Then, it was filtered to separate the adsorbent and filtrate. The filtrate's absorbance was measured using a UV-Vis Spectrophotometer to determine the adsorption capacity of methylene blue into MS-POBA. The optimum mass of the adsorbent was carried out by adding methylene blue solution with a concentration of 20 mg L^{-1}, and the acidity level of the methylene blue solution was adjusted to the optimum pH. Then, MS-POBA with and without the MES template was added with masses of 0.01, 0.02, 0.03, 0.04, and 0.05 g. The stirring was carried out for 60 minutes, and then the mixture was filtered to separate the residue and the filtrate. The absorbance of the filtrate was measured using a UV-Vis Spectrophotometer and the adsorption capacity of methylene blue can be determined. Also, the adsorption contact time was determined by preparing 20 mg L^{-1} of methylene blue solution and MS-POBA with optimum mass by procedure.

The acidity level of the solution is adjusted at the optimum pH. The absorbance of the solution was measured with various contact times of 5, 15, 30, 45, 60, and 75 minutes. Furthermore, the adsorption capacity was obtained at each contact time. Determination of optimum Methylene Blue Concentration using the same procedure above at optimum pH, absorbent mass, and contact time was carried out. The initial Methylene Blue concentrations in the solution were 5, 10, 15, 20, 25, and 30 mg L^{-1}.

3. Result and Discussion

Due to the synthesis of the silica with the MES template, the boiler ash from the palm oil factory was purified using HCl to remove metal oxides and then extracted with NaOH. Using the sol-gel method, HCl was added to produce silica powder with Equation 1. Also, the silica structure is the interaction between sulfate ion, APMS, and silica which is shown in Figure 2. The interaction between MES, APMS, and silica sulfate ions shows that $–SO_3^-$ groups from MES and $–NH_3^+$ groups from APMS are involved in intermolecular interactions, determining the silica material's porous nature (Fig. 2).

$$SiO_{2(s)} + 2NaOH_{(aq)} \rightarrow Na_2SiO_{3\,(aq)} + H_2O_{(l)}$$
$$Na_2SiO_{3\,(aq)} + 2HCl_{(aq)} \rightarrow H_2SiO_{3(l)} + 2NaCl_{(aq)}$$
$$H_2SiO_{3(l)} \rightarrow SiO_2.H_2O_{(s)}$$

(Eq.1)

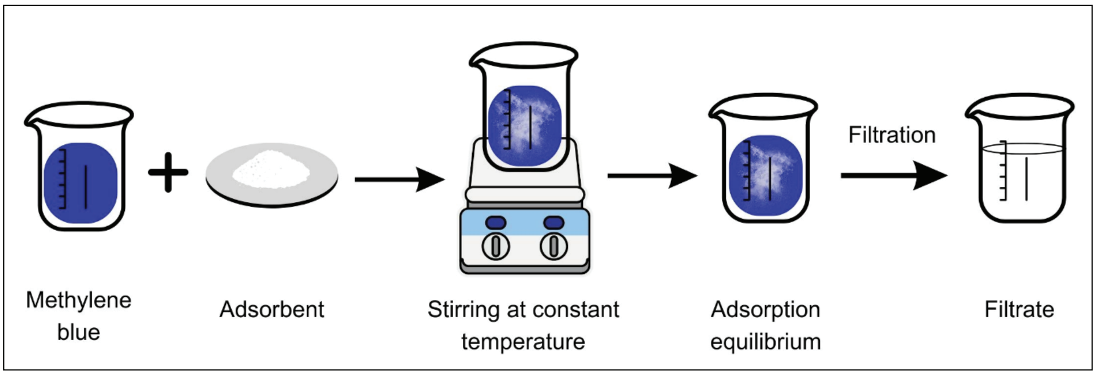


Fig. 1. Illustration of the adsorption mechanism

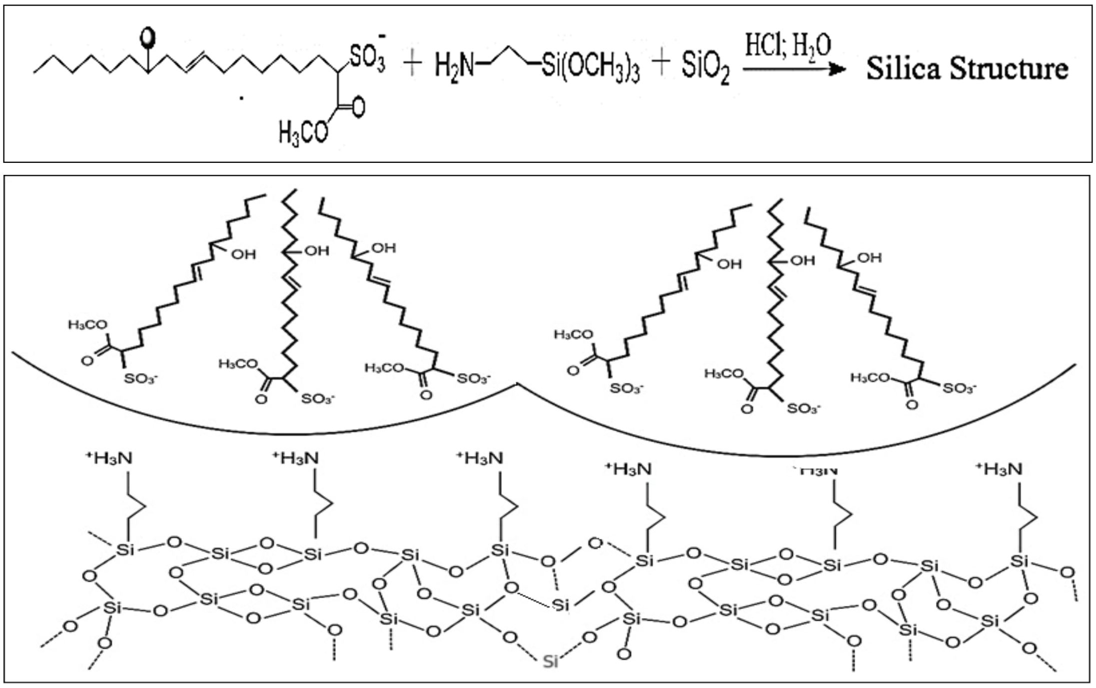


Fig. 2. Schematic of interaction between sulfate ion, APMS, and silica network

3.1. *FT-IR Data*

Fourier Transform Infra-Red (FT-IR) characterization aims to find out the functional groups of material. Infrared spectra of silica material without a template and modified silica with an MES template can be seen in Figure 3 and Table 1. MS-POBA has specific characters Si-OH and Si–O–Si, and MES have specific characters in the sulfonate group. The formation of a sulfonate group (S=O) is indicated by the peak of the wave number 1219–128 cm^{-1} [10]. Through the calcination process, MES is lost, and then modified mesoporous silica is formed. The absence of absorption at a wavenumber of 1226 cm^{-1} indicates the loss of sulfonate groups due to the calcination process.

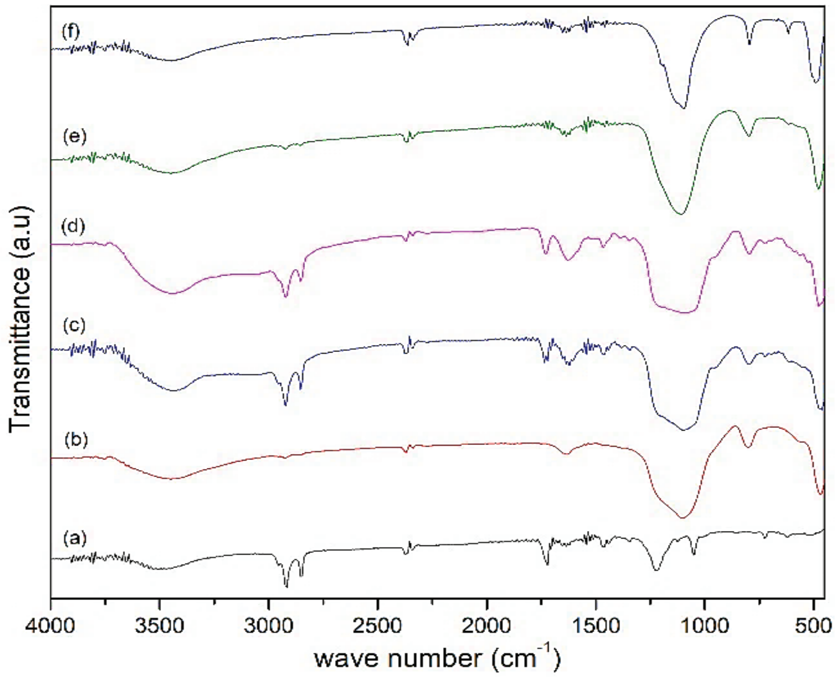


Fig. 3. FT-IR spectra of a) MES, b) MS-POBA, c) MS-POBA with MES template without calcination, d) MS-POBA with MES template (calcination temperature of 550 °C), e) MS-POBA with MES template (calcination temperature of 750 °C), f) MS-POBA with MES template (calcination temperature of 950 °C)

Table 1. Functional group and wave numbers by FTIR

Wave number	Functional groups
802	Si−O symmetric
1103	Si−O asymmetric
957	S=O symmetric
1211	S=O asymmetric
1620	N−H bending
2916−2854	C−H stretching
1620	O−H bending
3449−3749	O−H stretching

3.2. X-Ray Diffraction (XRD) Data

XRD was used to determine the phase on silica of the MS-POBA and MS-POBA with the MES template. Figure 4 (a) is a silica material without adding a template, showing the dominant form of a single phase in quartz (SiO_2) at 2θ = 23.08º, and the material is amorphous silica. Figure 4 (c) and (d) are mesoporous silica materials with the addition of MES and the calcination temperature of 750 ºC and 950 ºC. This mesoporous silica was tall and sharp, indicating that the structure was crystalline. Higher temperatures can result in the growth of larger crystals. The adsorption of methylene blue in this research used an amorphous material that calcinated at a temperature of 550 °C.

3.3. Brunaur, Emmet, and Teller (BET) Data

Figure 5 shows the graph of the nitrogen adsorption-desorption isotherm of MS-POBA and MS-POBA using a MES template. According to the IUPAC classification, the pattern shows a Type IV isotherm that has a hysteresis loop in the middle region. This indicated the type of adsorption from the mesoporous category. Table 2 shows the characteristics of the MS-POBA surface. The addition of an MES template affects porosity and causes an increase in surface area, pore diameter, and volume.

3.4. Optimized of adsorption parameters

3.4.1.Effect of pH

Figure 6 shows the optimal pH at pH 7, indicated by an increase in adsorption capacity to pH 7 and decreases after that. The adsorbent surfaces of MS-POBA with MES template can undergo protonation under acidic conditions, producing a charge more positive. The more positive charge can reduce the surface ability of the adsorbent to interact with cationic methylene blue. The electrostatic attraction between methylene blue and the adsorbent surface can be reduced, resulting in a lower adsorption capacity. The higher the pH, the more adsorption capacity increases because more adsorbents are deprotonated to be negatively charged, so methylene blue is more electrostatically adsorbed and optimally at pH 7.

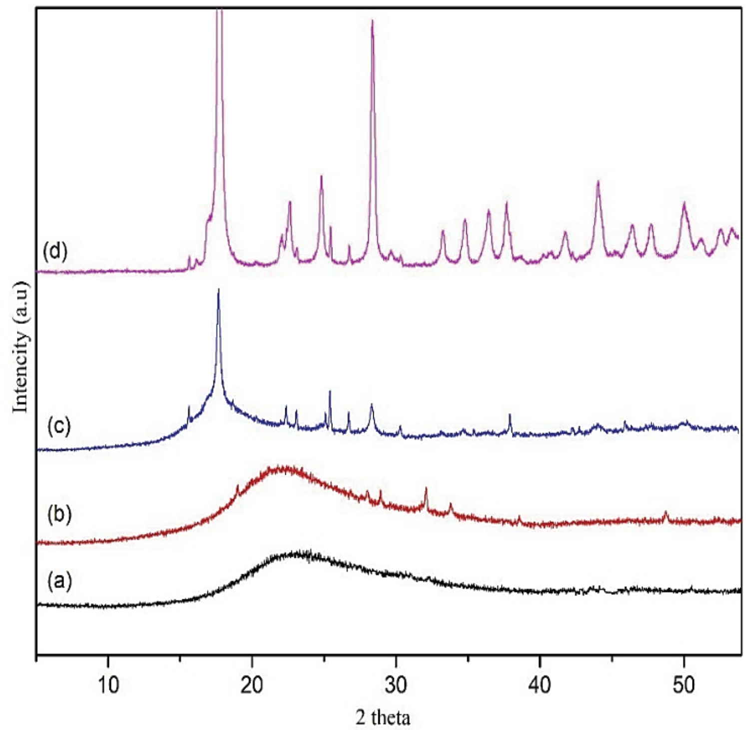
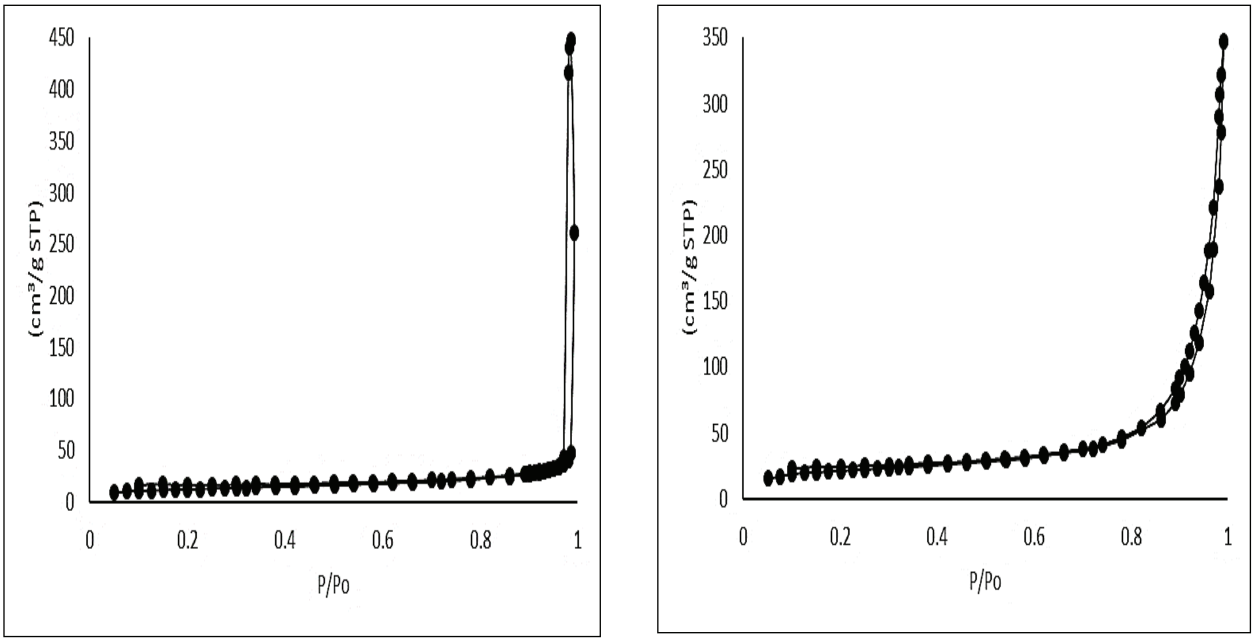


Fig. 4. The diffractogram of MS-POBA
a) without template, b) calcination temperature of 550 °C, c) 750 °C and d) 950 °C



(a (b

Fig. 5. Graph of Nitrogen Adsorption-Desorption Isotherm a) MS-POBA and b) MS-POBA with MES template

Table 2. The Surface Characters of MS-POBA

Sample	Surface Area ($m^2 g^{-1}$)	Pore Diameter (nm)	Pore Volume ($cc g^{-1}$)
MS-POBA	41.033	4.180	0.250
MS-POBA with MES	71.014	7.923	0.524

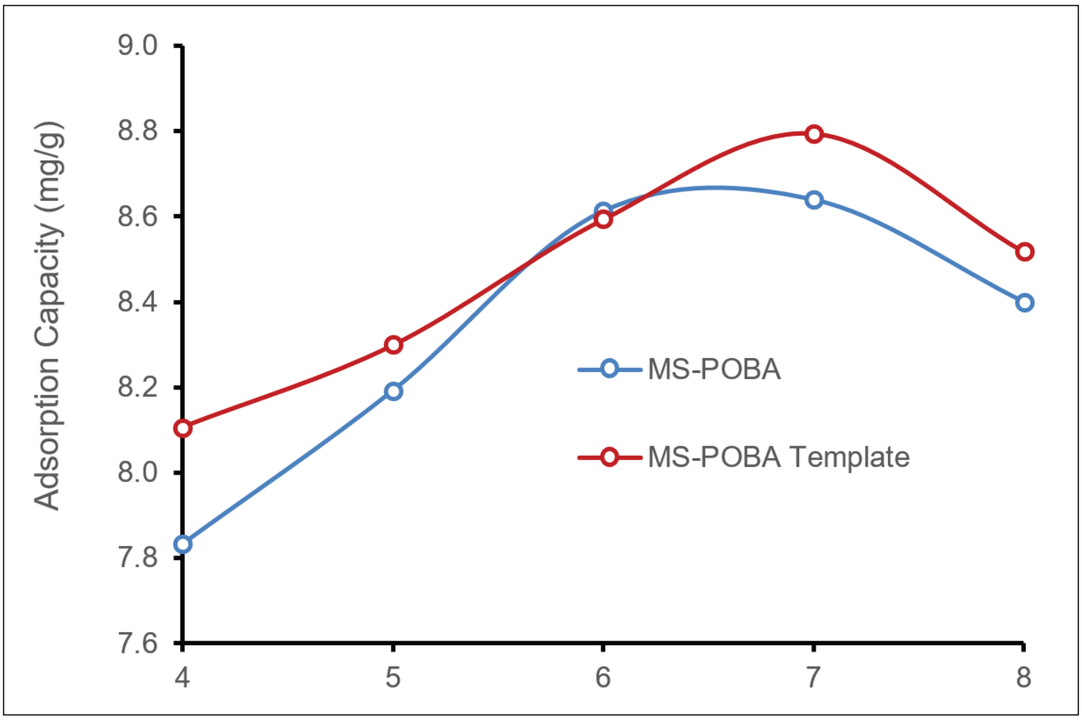


Fig. 6. Determination of optimum pH on methylene blue adsorption

3.4.2.Effect of adsorbent mass and contact time

Figure 7a shows an increased methylene blue mass that was adsorbed in MS-POBA and MS-POBA with the MES template and an increased adsorbent mass. The optimal mass of MS-POBA and MS-POBA with MES templates is 0.04 g and 0.03 g, respectively. However, the figure shows no significant difference in methylene blue adsorbed into MS-POBA and MS-POBA with the MES template. Figure 7b shows an increase in the adsorption capacity of methylene blue, along with an increase in contact time between adsorbent and adsorbate. Methylene blue adsorption capacity reached a contact time of 60 minutes on MS-POBA and 45 minutes on MS-POBA with the MES template. The equilibrium between the adsorbent and adsorbate will be reached when the number of active sites on the adsorbent has been exhausted. Tables 3 and 4 show the calculation results of the methylene blue adsorption kinetics model by MS-POBA and MS-POBA with the MES template. Both follow the pseudo-second-order adsorption kinetics model, which is indicated by the linearity value (R^2), which is close to 1, namely 0.998 of MS-POBA and 0.999 of MS-POBA with MES template.

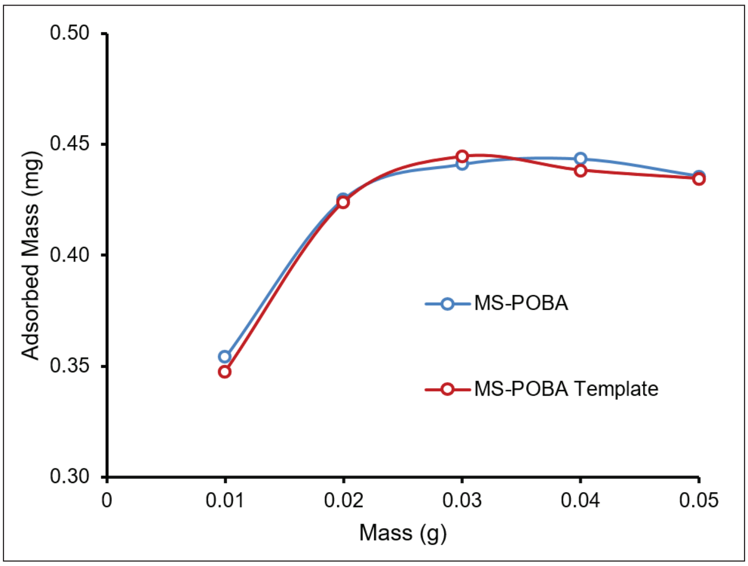


Fig.7a. Determination of optimum adsorbent mass

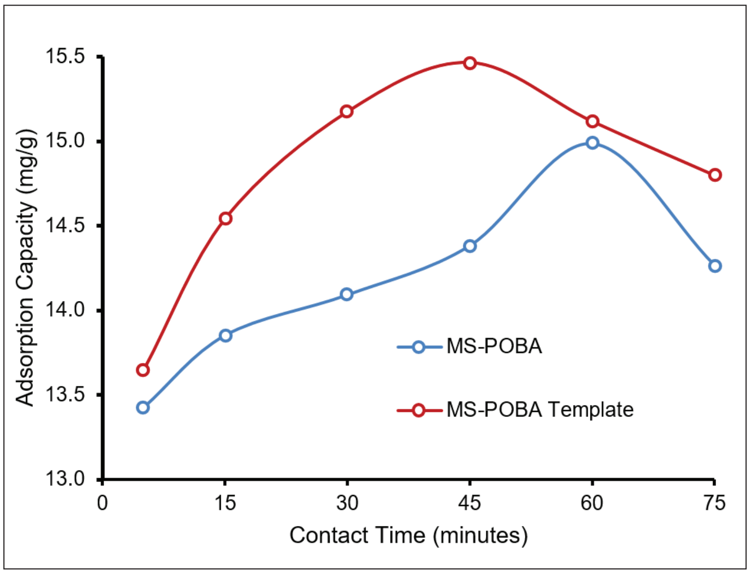


Fig.7b. Determination of optimum contact time

Table 3. The pseudo-first-order and pseudo-second-order kinetic model of methylene blue adsorption on MS-POBA

Model	Parameter	Result
Pseudo-first-order	R^2	0.209
	K (/minutes)	0.058
	q_e (mg g^{-1})	2.397
Pseudo-second-order	R^2	0.998
	K (g mg^{-1} min^{-1})	0.111
	q_e (mg g^{-1})	14.619

Table 4. The pseudo-first-order and pseudo-second-order kinetic model of methylene blue adsorption on MS-POBA with MES template

Model	Parameter	Result
Pseudo first-order	R^2	0.242
	K (/minutes)	0.012
	q_e (mg g^{-1})	1.130
Pseudo second-order	R^2	0.999
	K (g mg^{-1} min^{-1})	1.706
	q_e (mg g^{-1})	15.150

3.4.3. The optimum initial concentration of methylene blue

Figure 8 illustrates a direct correlation between the adsorption capacity and the concentration of methylene blue. As the concentration of the adsorbate in the solution rises, there is a proportional increase in the adsorption on the surface of the adsorbent, consequently leading to higher adsorption capacity. The optimum initial concentration of methylene blue obtained on the MS-POBA is 25 mg L^{-1}, and on the MS-POBA with MES template is 20 mg L^{-1}. After reaching equilibrium, the adsorption capacity is constant while the concentration of methylene blue in the solution increases. Based on Figure 8, the adsorption capacity is 15.396 mg g^{-1} on MS-POBA and 15.578 mg g^{-1} on MS-POBA / MES template, where it can be concluded that MS-POBA with the template has a slightly higher adsorption capacity compared to MS-POBA without templates. Tables 5 and 6 show the adsorption isotherm model of MS-POBA with and without template. The closest linearity to 1 is the Langmuir isotherm model with an R^2 value of 0.870 for MS-POBA and R^2 of 0.999 for MS-POBA /MES template, so it can be concluded that methylene blue adsorption in this study followed the Langmuir Isotherm model. The Langmuir Isotherm model is suitable for use in this study because the Langmuir Isotherm model assumes that no interaction between adsorbed molecules each other, and the adsorption occurs in a single layer of adsorbed molecules on the surface of the adsorbent.

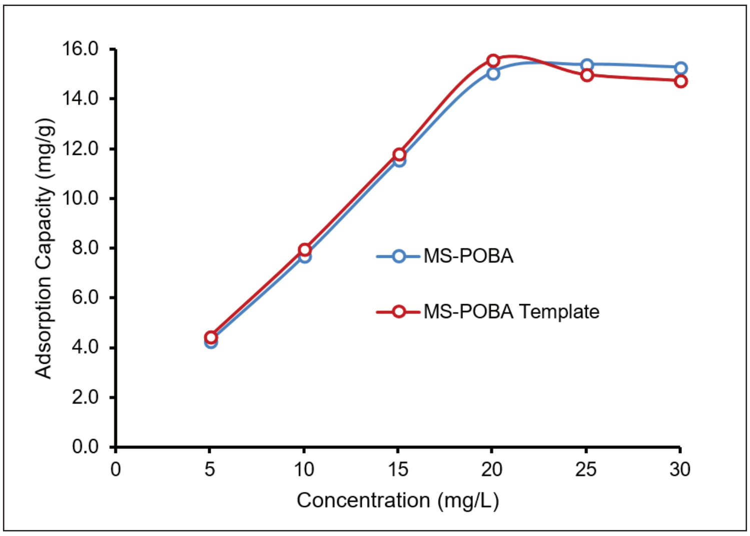


Fig. 8. Determination of optimum initial concentration of methylene blue

Table 5. The adsorption isotherm model of MS-POBA

Parameter	Results
Isotherm Langmuir	
R^2	0.870
K_L (L mol^{-1})	2.665
q_m (mg g^{-1})	17.211
Isotherm Freundlich	
R^2	0.653
K_f (L mol^{-1})	9.550
N	1.894

Table 6. The adsorption isotherm model of MS-POBA with MES template

Parameter	Result
Isotherm Langmuir	
R^2	0.242
K_L (L mol^{-1})	0.012
q_m (mg g^{-1})	1.130
Isotherm Freundlich	
R^2	0.999
K_f (L mol^{-1})	1.706
n	15.150

3.4.4.Scanning Electron Microscope–Energy Dispersive X-Ray (SEM-EDX) Data

The surface morphology and characteristics of the MS-POBA with MES template before and after methylene blue adsorption are shown in Figure 9. The adsorbent before adsorption shows an irregular particle distribution and irregular cavities between particles, hence having a significant number of pores that provide the place for the absorption of Methylene blue molecules. After the adsorption of the methylene blue process, Figure 9b illustrates morphological changes in the SEM image of silica

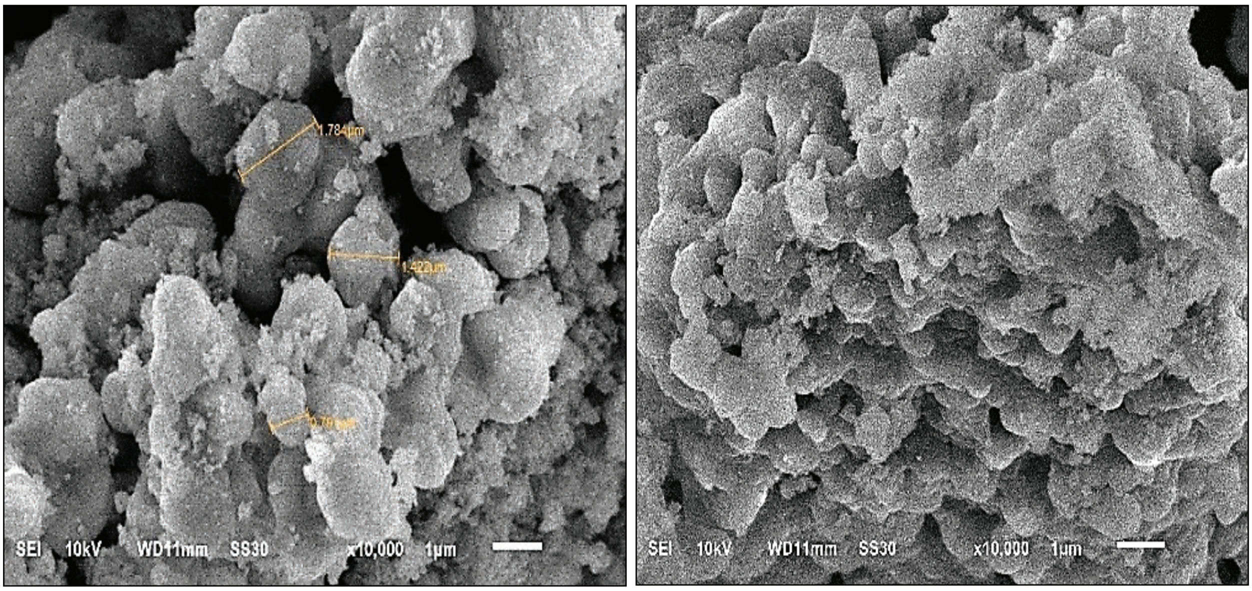


Fig. 9. SEM image of MS-POBA with MES template
(a: left) before adsorption and (b: right) after adsorption of methylene blue dye magnification 10.000 x

Table 7. Composition of MS-POBA with MES template before and after adsorption

Element	weight%	
	Before adsorption	After adsorption
C	6.71	4.34
O	51.91	45.48
Na	1.23	1.24
Al	1.16	1.20
Si	39.00	47.74

mesopores, showing that methylene blue molecules have partially filled the silica material's pores. This fact suggests that Methylene blue molecules are absorbed on the surface of the adsorbent.

As Table 7, the MS-POBA MES template contains several elements: C, O, Na, Al, and Si before adsorption. EDX is used to confirm the presence of Si and O in SiO_2. The adsorption process of methylene blue dye on the MS-POBA MES template changes the composition of the percentage elements contained.

4. Conclusion

In conclusion, this work demonstrated the synthesis of large pore mesoporous silica nanoparticles with an anionic surfactant as the template. The silica surface area, pore diameter, and pore volume of silica on MS-POBA with MES template are 71.014 $m^2 g^{-1}$, 7.923 nm, and 0.524 cc g^{-1}, respectively. The optimum conditions for adsorption of methylene blue dye by the MS-POBA adsorbent with MES template are obtained at pH 7, adsorbent mass 0.03 g, adsorption time of 45 minutes, and methylene blue concentration of 20 mg L^{-1} with adsorption capacity of 15.578 mg g^{-1}. The recovery (%) obtained in this study is 96.9%. The adsorption of methylene blue dye by MS-POBA with an MES template follows a pseudo-second-order kinetic model with a value of R^2 = 0.999 and a constant adsorption rate of 1.706 g mg^{-1} min^{-1}. The adsorption process follows the Langmuir Isotherm model.

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