

Synthesis of Adsorbent from Bagasse for Methylene Blue Adsorption

Nurhasni Nurhasni*, Sariana Harahap, Ahmad Fathoni, Hendrawati Hendrawati

Department of Chemistry, Faculty of Science and Technology, UIN Syarif Hidayatullah Jakarta
Jl. Ir. H. Juanda No. 95 Ciputat South Tangerang 15412, Indonesia

*Corresponding author: nurhasni@uinjkt.ac.id

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Abstract

The ability of bagasse adsorbents to adsorb methylene blue without activation using 0.5 M H₂SO₄ solution was examined. Methylene blue is widely used in the textile industry because it produces bright colors, and the dyeing process is fast and easy. This research aims to determine the optimum adsorption conditions, namely the variations in contact time, dye concentration, adsorbent mass, and pH effect on methylene blue, which were carried out using the batch method. Furthermore, the adsorbents were characterized by FT-IR and SEM. The optimum state of the bagasse adsorbent to adsorb methylene blue dye has a mass of 0.5 grams, a contact time of 30 minutes, a concentration of 50 ppm, and a pH of 5. The character of the adsorbent after activation with H₂SO₄ was better than without activation. The highest adsorption efficiency of methylene blue dye in the batch method was 99.67%. The FTIR spectrum of the bagasse adsorbent showed OH, C-H, C=O, C=C, and C-O functional groups. The adsorption isotherm model for methylene blue dye follows the Langmuir isotherm since the graph obtained is linear with the correlation coefficient (R^2) = 1, where the adsorbent has a homogeneous surface.

Keywords: Adsorbent, bagasse, adsorption isotherm, methylene blue.

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1. INTRODUCTION

Dyes are colored compounds widely used in textile, plastics, paper, paint, and other industries. The textile industry uses many dyes in the dyeing process, but without prior waste treatment, it can become a dangerous pollutant (Rao, 2013). The effluent contains hazardous and toxic materials that can prevent sunlight from entering the aquatic environment, disrupting biological processes and other adverse effects (Krim *et al.*, 2006; Daniel *et al.*, 2013).

One of the thiazine dyes, methylene blue, is widely used in industry because it is economical and readily available. Furthermore, it is commonly used in dyeing leather, mori cloth, cotton cloth, and tannin as a base dye. Unsaturated organic substances and azo groups content in methylene blue is difficult for microorganisms to degrade, which has an impact on the environment. Azo dyes under anaerobic conditions can also decompose into potentially carcinogenic aromatic amines,

therefore adversely affecting health such as skin irritation, cancer, and mutations in humans (Jeyajothi, 2014; Sarkar and Ghosh, 2012).

Coagulation, adsorption, ion exchange, and ozonation methods can be used to solve the dye waste problem. The adsorption method with adsorbent is efficient and economical (Sivakumar & Palanisamy, 2009). Adsorbents can be in the form of coconut shells (Pambayun *et al.*, 2013), corn on the cobs (Aminet *et al.*, 2016), rice husks (Nurhasni *et al.*, 2014), salak seeds (Aji & Kurniawan, 2012), peanut shells (Oktasari, 2018) and bagasse (Sari *et al.*, 2017).

Bagasse contains 37.65% cellulose (Asbhani, 2013), while cellulose contains functional groups such as hydroxyl, methyl, and carbonyl groups, which play a role in the absorption process of dyes in wastewater (Sulyman *et al.*, 2017). For example, the hydroxyl and carbonyl functional groups are the main functional groups on the surface of

the biosorbent involved in the biosorption of methyl red dye by rambutan seeds (Zein *et al.*, 2015).

Subsequently, to increase the adsorption capacity, activation was performed by reducing the impurities adhering to the surface and the pores of the adsorbent by physical or chemical means. In the activation process, the type of activator is either acid, HCl (Nurbaeti *et al.*, 2018; Asbhani, 2013), HNO₃ (Zein *et al.*, 2018), C₆H₈O₇-citrate, and H₂O₂ (Pham *et al.*, 2015) as well as base, NaOH (Sari *et al.*, 2017; Wulandari, 2018; Pham *et al.*, 2015), and KOH (Gao *et al.*, 2016). This research aims to determine the ability of bagasse to adsorb methylene blue dye after it has been activated with 0.5 M H₂SO₄. Therefore, bagasse can be used as an alternative adsorbent in the dye waste treatment.

2. MATERIALS AND METHODS

Tools and Materials

Blender, analytical balance, centrifuge, shaker, 180 m sieve, oven (Memmert), UV-Vis Spectrophotometer (Perkin Elmer Lambda 25), Fourier Transform Infrared Spectroscopy (FT-IR) (IRPrestige-21-Shimadzu), Scanning Electron Microscopy (SEM) (Carl Zeiss-EVO), and glassware.

The materials used are bagasse, methylene blue (Merck), sulfuric acid (H₂SO₄) (Merck), ethanol (C₂H₅OH) (Merck), citric acid (C₆H₈O₇) (Merck), sodium citrate (Na₃C₆H₅O₇·2H₂O)₂₈ (Merck), sodium phosphate monobasic (NaH₂PO₄·H₂O) (Merck), sodium phosphate dibasic (Na₂HPO₄) (Merck), borax (Na₂B₄O₇) (Merck).

Bagasse Preparation

The bagasse was washed thoroughly with running water and dried in the sun for one week. The dried bagasse was mashed using a blender and dried in an oven for ± 3 hours at 110 °C as well as sieved with a 180 m sieve, then coded (ATAKA). Meanwhile, bagasse without activation was coded (ATTA). Furthermore, the bagasse was activated by soaking in 0.5 M H₂SO₄ for 24 hours, filtered, and neutralized with distilled water. The ATTA and ATAKA adsorbents were then used to determine the optimum conditions.

Optimum Adsorption Time

Inactivated bagasse (ATTA) and chemically activated acidic bagasse (ATAKA) were weighed 0.5 g, poured into 50 ml of methylene blue dye solution with an initial concentration of 50 ppm, and then stirred with a stirrer at 180 rpm. Adsorption was carried out with variations in 30, 60, and 90 minutes (Raghuvanshi *et al.*, 2004). After completing the process, 10 mL is taken, then separated from the sediment by centrifuge at 4000 rpm for 10 minutes. The absorbance of the solution was measured using a UV-Vis spectrophotometer.

Test Solution Concentration

The adsorbents or ATTA and ATAKA weighed 0.5 g and were put into 50 mL of methylene blue dye with concentrations of 50, 100, and 150 ppm, stirred with a shaker at 180 rpm. The adsorption process was carried out with the optimum adsorption time. Once the process was complete, 10 mL was taken and separated from the precipitate by centrifuge at 4000 rpm for 10 minutes. The absorbance of the solution was measured using a UV-Vis spectrophotometer.

Optimum Adsorbent Mass

ATTA and ATAKA with the variation of the adsorbent mass 0.5: 1.0; and 1.5 g were placed in 50 ml of methylene blue dye solution with optimal concentration and stirred with a shaker at 180 rpm. The adsorption was carried out with the optimum adsorption time and concentration. After the process was complete, 10 mL was taken, separated from the precipitate by centrifuge at 4000 rpm for 10 minutes. The absorbance of the solution was measured using a UV-Vis spectrophotometer.

Optimum pH

ATTA and ATAKA were weighed to optimum mass, placed into 50 mL of methylene blue dye solution with an initial concentration of 50 ppm, and adjusted the pH with variations of pH 3, 5, 7, and 9 using a buffer (Basset & Denny, 1994). It was stirred with a shaker at a speed of 180 rpm. Furthermore, the adsorption was carried out with optimum adsorption conditions. After the process was complete, 10 mL was taken and separated from the sediment by centrifuge at 4000 rpm for 10 minutes. The absorbance of

the solution was measured using a UV-Vis spectrophotometer.

Statistical Analysis

The absorbance value obtained from the UV-Vis spectrophotometer was entered into a linear regression equation to obtain the concentration. Then, the final concentration of each test solution was used to calculate the adsorption efficiency (E) and the adsorption capacity (Q) using Equations 1 and 2.

$$E (\%) = \frac{(C_0 - C_t)}{C_0} \times 100\% \quad (1)$$

$$Q = \frac{(C_0 - C_t)}{w} \times V \quad (2)$$

Description:

- E = Adsorption efficiency (%)
- Q = Adsorption capacity (mg/g)
- C₀ = Initial concentration of solution (ppm)
- C_t = Final concentration of solution (ppm)
- w = Adsorbent mass (g)
- V = Volume of solution (L)

3. RESULTS AND DISCUSSION

Bagasse Adsorbent Activation

The activation of acid can increase the volume of the carbon cavity or pore system and dissolve impurities such as inorganic minerals (Nurhasni *et al.*, 2012). The activation of the acid solution can release impurities or metal ions such as Ca²⁺, K⁺, and Mg²⁺, which partially cover the adsorbent pores; therefore, the adsorbent pores have a clean and wide surface, as well as the active groups contained in the adsorbent are more reactive when binding dyes (Purnamawati *et al.*, 2014). The activation of the adsorbent using H₂SO₄ concentration ≥ 0.1 M can undergo a dehydration reaction; therefore, water is released and can open and expand the carbon pores and increase the absorption efficiency. Utomo (2019) stated that calcined H₂SO₄ 0.5 M bagasse at 400 °C obtained an adsorption efficiency of 83.93% against Naphthol Yellow S dye.

Sulfuric acid can cause dehydration of tertiary alcohol through the process of protonation of the hydroxyl group and forms a carbocation, which is characterized by the release of a water molecule. Furthermore, the hydroxyl group-containing the proton is removed to form an alkene. The dehydration

process of the hydroxyl group is shown in Figure 1.

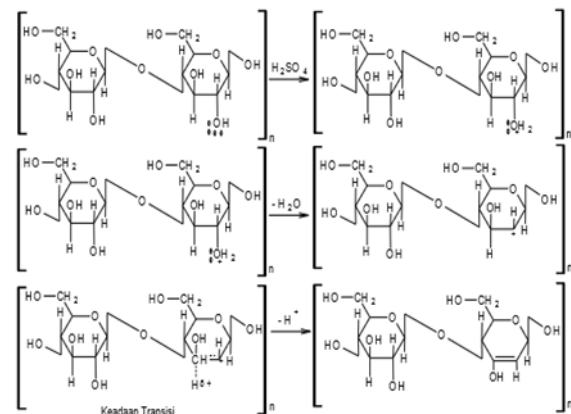


Figure 1. Dehydration reaction of hydroxyl groups on cellulose (Rajawane, 2008).

Optimum Adsorption Time

The determination of the optimum adsorption time is 30, 60, and 90 minutes. The constant parameters are a concentration of 50 ppm, a volume of 50 ml, and a mass of 0.5 g. The results of the absorption efficiency of methylene blue dye can be seen in Figure 2.

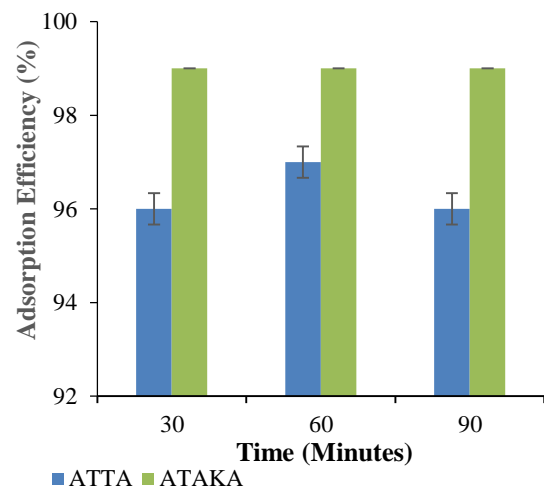


Figure 2. Effect of adsorption time on methylene blue dye.

The adsorbent from acid-activated bagasse (ATAKA) can absorb methylene blue dye with an adsorption efficiency of 99% and an average adsorption capacity of 3.8 mg/g at 30, 60, and 90 minutes, while the unactivated adsorbent (ATTA) at 30 minutes produces 96% efficiency, the adsorption capacity of 3.7 mg/g, then at 60 minutes produces 97%

efficiency, the adsorption capacity of 3.75 mg/g. At 90 minutes, the efficiency decreased to 96% with the adsorption capacity of 3.73 mg/g. Over time, the absorbed dye can be rereleased through the mixing process in the batch method (Aji & Kurniawan, 2012). Therefore, the optimum time during which high adsorption efficiency is generated in the adsorbent's adsorbing process of methylene blue dye without activation and acid activation is 30 minutes. Sari et al. (2017) stated that on activated carbon of bagasse with 6% NaOH activation, the red portion adsorption efficiency was 69.04%, with a contact time of 90 minutes.

Dye Solution Concentration

The initial concentration of the solution to be tested for the adsorption of the methylene blue dye was 50, 100, and 150 ppm with a volume of 50 mL, the optimum time of 30 minutes with a mass of 0.5 g ATTA and ATAKA. The effect of concentration on the adsorption efficiency of methylene blue dye can be seen in Figure 3.

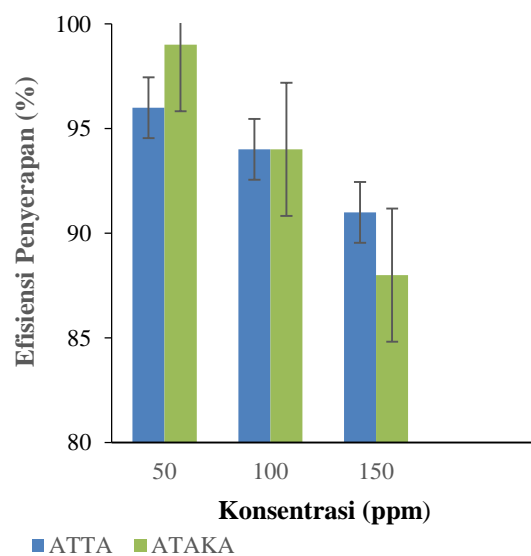


Figure 3. Effect of adsorbate concentration on methylene blue dye.

The optimum ATTA and ATAKA adsorbents have a dye concentration of 50 ppm. They could adsorb methylene blue dye with adsorption efficiencies of 96.36 and 99.79%, respectively, with adsorption capacities of 3.72 and 3.85 mg/g. At concentrations of 100 and 150 ppm, the adsorption efficiency of the adsorbent

decreases without activation and acid activation since the adsorbent has several active sites that will experience saturation at a certain concentration (Mousavi & Seyed, 2011). Moreover, Sari et al. (2017) reported that 6% bagasse-activated NaOH activated carbon achieved an adsorption efficiency of 76.3% on red procion from the liquid waste of the *songket* industry at 76.3%; with a concentration of 20 mg/L.

Optimum Adsorbent Mass

The mass of the adsorbent was 0.5, 1.0, and 1.5 g, with a methylene blue concentration of 50 ppm, a volume of 50 ml, and 30 minutes for the process of adsorbing the methylene blue dye. The effect of adsorbent mass on the adsorption efficiency of methylene blue can be seen in Figure 4.

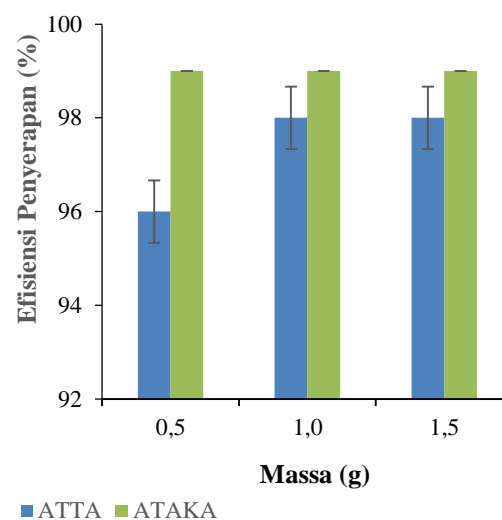


Figure 4. Effect of adsorbent mass on methylene blue dye.

The optimum mass for acid activation adsorbent (ATAKA) in the methylene blue dye adsorption process was 0.5 g at a concentration of 50 ppm and 30 minutes with an efficiency of 99% and a capacity of 3.85 mg/g. An inactivated adsorbent (ATTA) has an optimum mass of 1.0 g, a concentration of 50 ppm, and a time of 30 minutes with an efficiency of 98.56% and an adsorption capacity of 3.72 mg/gram. Acid-activated bagasse adsorbents absorb methylene blue more effectively than non-activated ones. Subsequently, the volume of the cavity or pore system increases, thereby increasing the efficiency of bagasse adsorption (Irdhawati *et al.*, 2016). Sari et al. (2017) showed that the activated carbon of bagasse

activated with NaOH 6% obtained 76% red procion adsorption efficiency at pH 5.

Optimum Adsorption pH

In the adsorption test, the pH variations were pH 3, 5, 7, and 9 at the optimum adsorption concentration of the methylene blue dye. As a result, the adsorbent mass of ATTA was 1.0 g, and ATAKA was 0.5 g with an optimum time of 30 minutes. The effect of the pH of the methylene blue dye solution on the adsorption efficiency can be seen in Figure 5.

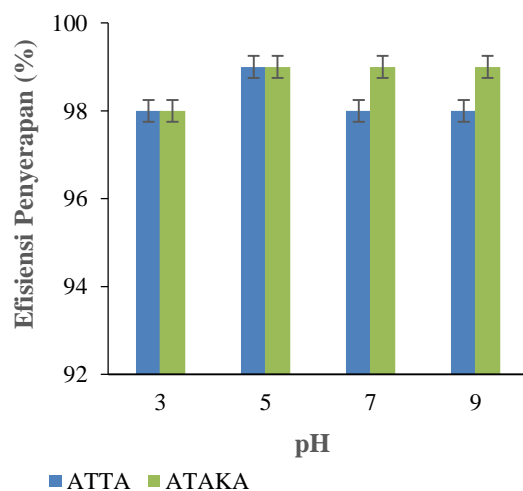


Figure 5. Pengaruh pH larutan terhadap zat warna *methylene blue*.

The optimum pH for the ATTA adsorbent on adsorption of methylene blue dye was pH 5 with a concentration of 50 ppm, 30 minutes, the adsorption efficiency of 98%, and a capacity of 1.08 mg/g. Meanwhile, for ATAKA adsorbent, the adsorption efficiency was 98%, with a capacity of 2.18 mg/g. Furthermore, Sari et al. (2017) reported that procion adsorbed red dye using bagasse activated carbon as the adsorbent and obtained optimum conditions at pH 5 with an adsorption efficiency of 76% and capacity 7.66 mg/g. The adsorption efficiency of the methylene blue dye at pH 9 for the adsorbent without activation decreased to 98% with a capacity of 1.08 mg/g. This is due to the alkaline pH conditions of methylene blue to form an ionized salt.

Adsorption Isotherm

The concentration variations were 50, 75, 100, and 150 ppm due to the optimum

activation of bagasse adsorbent, namely acid activation (ATAKA) with a volume of 25 mL, the adsorbent mass of 0.5 g, adsorption time of 30 minutes, and pH 5. Graph of Langmuir isotherm and isotherm Freundlich adsorption of methylene blue dye is shown in Figures 6 and 7.

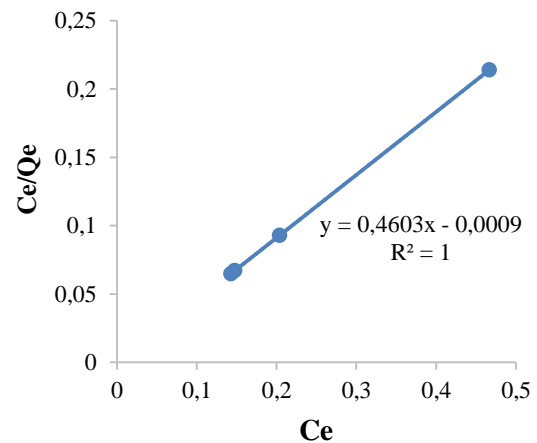


Figure 6. Langmuir isotherm methylene blue adsorption curve.

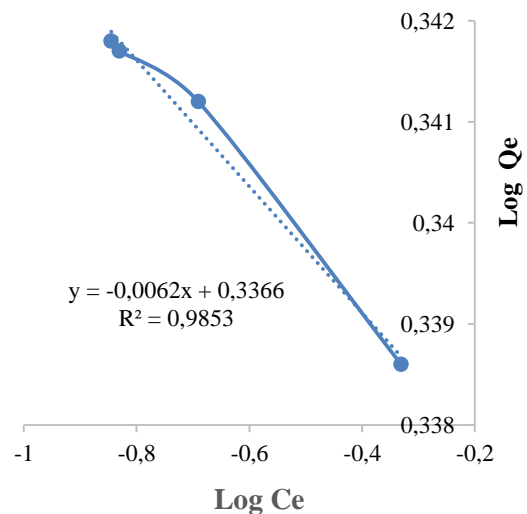


Figure 7. Freundlich isotherm methylene blue adsorption curve.

The adsorption model is a Langmuir isotherm because the graph obtained is linear with a correlation coefficient value (R^2) = 1. The adsorbent has a homogeneous surface and can only absorb one molecule for each adsorbent molecule. Therefore each surface area has the same bond energy. (Sari et al., 2017).

Characterization Results with Fourier Transform Infrared Spectroscopy (FTIR)

The adsorbent used was bagasse

before and after adsorption of methylene blue dye with optimum conditions, namely adsorbent mass of 0.5 g, the concentration of methylene blue 50 ppm, pH 5, and adsorption time of 30 minutes.

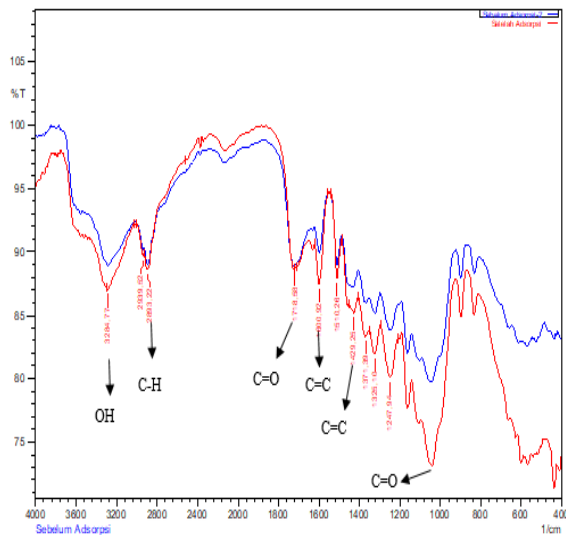


Figure 8. Spectrum characterization results using FTIR.

The FTIR spectrum shows a slight difference in the wavenumber between the adsorbent before and after adsorption (Figure 8). Changes in the wavenumber from 3280.92 cm^{-1} to 3284.77 cm^{-1} which is the absorption area for the $-\text{OH}$ group. The adsorbent also produces a sharper peak after adsorption than before adsorption. The wavenumbers for aliphatic C-H on the two adsorbents did not differ much at 2895.15 cm^{-1} and 2893.22 cm^{-1} . The C=O aldehyde group on both adsorbents at wavenumbers of 1726.29 cm^{-1} , for C=C before adsorption at wavenumbers of 1598.99 and 1433.11 cm^{-1} , after adsorption at wavenumbers of 1600.92 and 1429.25 cm^{-1} are the aromatic ring because it often appears in the form of a pair of absorption at 1600 and 1475 cm^{-1} (Sastrohamidjojo, 2013)

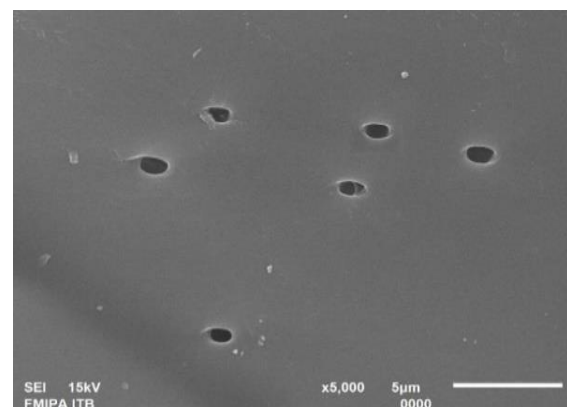
The adsorption for tertiary C-O alcohol before and after adsorption at 1163.08 cm^{-1} , and for primary C-O alcohol adsorption before adsorption was shown at 1047.35 cm^{-1} and after adsorption at 1041.56 cm^{-1} . Area $800\text{--}400\text{ cm}^{-1}$ is the fingerprint area (Wulandari, 2018). The process of bagasse adsorbent after adsorption showed an FTIR spectrum with the same band before adsorption but had a sharp spectrum and greater absorption intensity.

Results of Surface Morphological Characterization with Scanning Electron Microscopy (SEM)

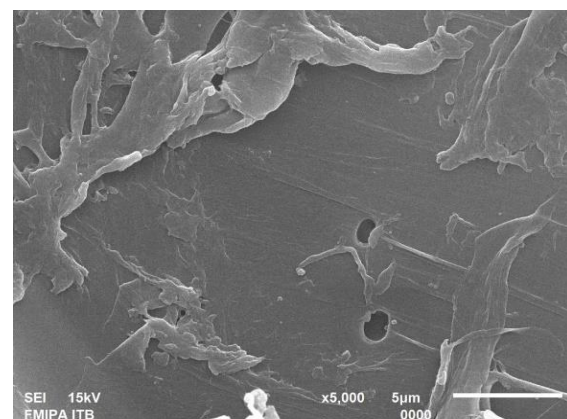
The bagasse adsorbent was characterized using SEM (Scanning Electron Microscopy) with a magnification of 5000x to observe the bagasse adsorbent's surface morphology and pore size before and after its adsorption. The choice of Acid Chemical Activation Bagasse (ATAKA) adsorbent, which adsorbs methylene blue, is due to the fact that it has the highest efficiency of 99.58%.

After adsorption, the SEM results showed a solid, hollow, and wavy morphology. This proves that the sample used to adsorb the dye produces a denser form because it is trapped in the adsorbent.

The SEM analysis result is shown in Figure 9.



(a)



(b)

Figure 9. The surface morphology of the adsorbent before (a) and after adsorption (b).

The bagasse adsorbent before adsorption at 5000x magnification appeared homogeneous, more regular, clean, and had pores that formed cavities. This number of

cavities affects the adsorption value of the bagasse adsorbent on dyes (Rahman *et al.*, 2016). A large number of cavities increases the adsorption capacity as this enables an absorption process in which the solution fills the surface of the adsorbent and fills the empty cavity; hence there is an interaction between the cell wall of the adsorbent and the solution (Hasfita, 2012). Meanwhile, Pham *et al.* (2015) showed a significant change in the surface morphology of the adsorbent. The adsorbent before adsorption has a regular structure texture and many pores, while it has an irregular morphology after adsorption.

4. CONCLUSION

Acid-activated bagasse has a better adsorption capacity to adsorb methylene blue dye. The optimum conditions for Acid Activated Bagasse (ATAKA) were 30 minutes, an adsorbent mass of 0.5 g, a concentration of 50 ppm, and pH 5. In comparison, non-Activated Bagasse (ATTA) lasted for 30 minutes, had a mass of 1.0 g, concentration of 50 ppm, and pH 7. The methylene blue dye adsorption pattern follows the Langmuir isotherm because the graph obtained is linear with the correlation coefficient (R^2) 1, where the adsorbent has a homogeneous surface.

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