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Adsorption and Characterization of Activated Sugarcane Bagasse Using Sodium Hydroxide

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Abstract

Sugarcane Bagasse can be used as an adsorbent both under natural conditions and modified by chemical activation using sodium hydroxide (NaOH). Activation of sugarcane bagasse with NaOH was carried out at variations of 5:1, 10:1, and 20:1 (w/w). The absorption ability of bagasse adsorbent to methylene blue solution was carried out with the parameters of variation of contact time (60, 90, 120, 150, and 180 minutes), adsorbate concentration (20, 30, 40, 50, and 60 ppm) and temperature (30, 40, 50, and 60 ^{o}C). The adsorbent's characterization included determining the functional groups using FTIR, morphology, and mass of elements using SEM-EDX, and determining the surface area and volume of adsorbent pores using the BET methods. The highest adsorption percentage results were found in the $NASB_{10:1}$ adsorbent at 99.50%. The optimum conditions for the $NASB_{10:1}$ adsorbent are with a contact time of 120 minutes, an adsorbate concentration of 50 ppm and a temperature of 30 ^{o}C or 303 K. The $NASB_{10:1}$ adsorbent has the highest surface area compared to other adsorbents, namely 2.803 m^2/g so that it can perform the maximum absorption of methylene blue.

Keywords: Sugarcane bagasse, chemical activation, FTIR, methylene blue, characterization

INTRODUCTION

One type of waste that is widely found in the community is bagasse. The total production of dry bagasse in the world is around 54 million tons per year. In 2013, the total area of sugarcane plantations in Indonesia was 470.94 thousand hectares. Bagasse is biomass with great potential, especially in various currently developing studies (Badan Pusat Statistik, 2015). Bagasse is one of the by-products of agricultural waste, can be used both in pure and modified form (Renu et al., 2017). Bagasse has a lot of fiber, mainly found in the stem. The contents of bagasse include cellulose (45%), lignin (18%), and hemicellulose (28%) (Pehlivan et al., 2013).

Cellulose and lignin in bagasse are potentially used as an adsorbent (Abou-gamra and Medien, 2013). Adsorbent from bagasse, which contains lignin, can remove the dye in Rhodamine B with an optimum adsorption percentage value of 77.6%. Adsorbent from bagasse can also remove Fe2 + ion content in industrial wastewater and is very efficient in absorbing it. The absorption percentage of Fe2 + ions from the solution is 95.11% using pure bagasse adsorbent (Arkanni et al., 2019). Besides, bagasse can remove the Cu metal ions' content with an absorption percentage of 96% (Moubarik and Grimi, 2015). Esvandiar et al. (2014) conducted a study using pure bagasse and activated Carbon from bagasse, which resulted in 63% and 97% of Mn (II) absorption. The adsorbent from bagasse can be chemically activated using an acid or base solution. Bagasse modified with an acid solution (HCl) was able to absorb 98.75% Cu^{2+} ions. (Yam et al., 2020). Pure bagasse as a biosorbent is very useful for absorbing the Reactive Red 120 (RRI 120) dye solution with a maximum percentage of adsorption of 94.62% (Ahmad et al., 2018). In another study, Gusmau et al. (2012) used bagasse as an adsorbent to absorb the dyes of Methylene Blue (MB) and Gentian Violet (GV) with adsorption capacity values of 478.5 and 1273.2 mg/g, respectively.

The adsorption process is a phenomenon on the surface of a porous solid material with interactions involving chemical or physical bonds and occurs in liquid or gas conditions. The adsorption process depends on the type of adsorbent used. Adsorbent must have adequate adsorption capacity, sufficient kinetic adsorption, small pore size, large surface area, good porosity, high micropore volume, and extensive pore network to allow molecules to reach the inside of the adsorbent and make the adsorbent suitable for the adsorption process (Al-Ghouti and Da'ana, 2020). The adsorption process is also proven to be superior in dealing with dye waste because it is susceptible and straightforward to toxic materials. Although activated

Carbon is the most effective for dye adsorption, it has several disadvantages: high adsorbent costs, and regeneration is expensive and challenging to separate powdered activated Carbon from wastewater. Therefore, it is necessary to improve its capability and effectiveness in the entrapment process at an economical and commercial cost (Abou-gamra and Medien, 2013).

The absorption ability of an adsorbent such as bagasse will increase when there is an activation treatment. The activation stage aims to enlarge the adsorbent pores. In general, there are two kinds of activation processes, namely chemical and physical activation, chemical activation is the process of breaking the carbon chain from organic compounds with the use of chemicals. Physical activation is the process of breaking carbon chains from organic compounds with the help of heat, steam and CO_2 gas (Latupeirissa et al., 2018).

Zaini et al. (2018) carried out the adsorption of manganese (II) in liquid waste using bagasse adsorbent, which was influenced by contact time and type of activator. The maximum adsorption capacity of the adsorbent activation with 0.5 N NaOH was 1.016 mg / g. Tchoumou et al. (2015) studied the adsorption capacity of bagasse modified with HCl and NaOH to remove Cu and Ni metals. Research carried out at room temperature included the influence of pH, contact time, and adsorbent weight. The results showed that the increase in pH, contact time, and adsorbent increased the adsorbed metal. Other observations obtained were that the bagasse modified with alkaline was more efficient than HCl, and the efficient adsorption results showed Cu (II) > Ni (II). Also, Nguyen et al. (2019) compared the adsorption capacity of modified bagasse with NaOH, citric acid, tartaric acid, and original bagasse (unmodified bagasse) with metal cadmium (Cd). The results showed an increase in the adsorption capacity after the chemical modification process. Modification with NaOH has an adsorption capacity of 29.41 mg/g. Unmodified bagasse has an adsorption capacity of only 18.8 mg/g. The confirmation of the Infrared spectrum for modified bagasse showed the addition of a carboxyl group's and an ester group's peak at the 1,738 cm^{-1}, thereby increasing the adsorption capacity.

Based on the description above, it seems that there have been many studies that show changes in the adsorbent's surface after undergoing a modification process to increase the ability of the adsorption capacity both the value of the adsorption capacity and the percentage of absorption. Therefore, this research will be activated bagasse with NaOH to determine the adsorption ability of them in the methylene blue solution and compare the surface morphology, functional groups, and surface area of bagasse before and after activation.

METHODOLOGY

Materials and Instrumentals

The instrument used in this study were 80 and 120 mesh sieves (WS Tyler Incorporated USA), magnetic stirrer (RSH-1DR), oven (Gallenkamp), FTIR (IRPrestige-21), SEM, analytical scales, desiccators, pH meters, and other glassware. The materials used include bagasse (derived from sugar cane traders in Riau University are), Sodium Hydroxide, distilled water, and Whatman filter paper 42.

Sample Preparation

The sample preparation of bagasse waste is carried out in several steps, including drying it under the sunlight, then cutting it into smaller sizes about 2 cm, then blending until smooth. The next stage is the sample is sieved using 80 and 120 mesh sieves in stages. The dimension of the used sample is a sample that is between 80 and 120 mesh (passes through the 80 mesh sieve and is stuck on the 120 mesh sieve).

The chemical activation process on bagasse uses NaOH solution. Bagasse and NaOH were mixed, stirring for 3 hours at a temperature of 85 ^{o}C with variations in the ratio of 5: 1, 10: 1, and 20: 1. The explanation of this comparison is 5, 10, and 20 grams of bagasse in 1 gram of NaOH at a volume of 100 mL. The next step is filtering the mixture and rinsing it with distilled water until the pH is neutral. The bagasse adsorbent that has been activated with NaOH is dried in an oven at 105 ^{o}C, then stored in a desiccator by providing a sample code: AT, $ANSB_{5:1}$, $ANSB_{10:1}$, and $ANSB_{20:1}$ (SB = Sugarcane bagasse, NASB = NaOH-Activated Sugarcane Bagasse).

Adsorbent Characterization

The Identify of functional groups on pure and activated bagasse with NaOH using FTIR spectroscopy instruments. Microstructure analysis and surface morphology of an adsorbent can use Scanning Electron Microscopy (SEM-EDX). The surface area and pore volume were determined using the BET method.

Sugarcane Bagasse Adsorption Ability by Batch Adsorption Method.

Determining the adsorption capacity of bagasse was carried out on different parameters, namely variations in bagasse activation with NaOH, contact time, adsorbate concentration (methylene blue), and temperature.

Effect of Contact Time

In this process, 0.1 gram of sugarcane bagasse (SB) was mixed with 10 mL of methylene blue solution at a concentration of 10 ppm. The mixture is stirred using a water bath shaker at a speed of 120 rpm for 30 minutes at a temperature of 30 ^{o}C. Then centrifuged for 15 minutes at 2000 rpm. The filtrate from this treatment was analyzed using a UV-Vis spectrophotometer at a wavelength of 660 nm. The same treatment was also carried out for each adsorbent that was activated with NaOH ($NASB_{5:1}$, $NASB_{10:1}$, and $NASB_{20:1}$). Furthermore, the above treatment was carried out again on each of the adsorbent variations with different times, including 60, 90, 120, 150, and 180 minutes

Effect of Adsorbate Concentration

In this process, 0.1 gram of sugarcane bagasse (SB) was mixed with 10 mL of methylene blue solution at a concentration of 10 ppm. The mixture was stirred using a water bath shaker at 120 rpm at 30 ^{O}C and the previous treatment's optimum time. Then centrifuged for 15 minutes at 2000 rpm. The filtrate from this treatment was analyzed using a UV-Vis spectrophotometer at a wavelength of 660 nm. The same treatment was also carried out for each adsorbent that was activated with NaOH ($NASB_{5:1}$, $NASB_{10:1}$, and $NASB_{20:1}$). Furthermore, the above treatment was carried out again on each of the adsorbent variations with different MB concentrations, including 20, 30, 40, 50, and 60 ppm.

Effect of temperature

In this process, 0.1 gram of sugarcane bagasse (SB) was mixed with 10 mL of methylene blue solution at the optimum concentration from the previous treatment. The mixture is stirred using a shaker water bath with a speed of 120 rpm at 30 ^{o}C and with the optimum time in the last treatment. Then centrifuged for 15 minutes at 2000 rpm. The filtrate from this treatment was analyzed using a UV-Vis spectrophotometer at a wavelength of 660 nm. The same treatment was also carried out for each adsorbent that was activated with NaOH ($NASB_{5:1}$, $NASB_{10:1}$, and $NASB_{20:1}$). Furthermore, the above treatment was carried out again at each of the adsorbent variations with different temperatures, including 30, 40, 50, and 60 ^{o}C.

RESULTS AND DISCUSSION

The research results discussed in this journal include the characterization of bagasse, both pure and activated with NaOH. Besides, the ability of bagasse adsorption against methylene blue solution was also observed with variations in contact time, adsorbate concentration, and temperature. The description of the results and discussion in this study is as follows:

Determination of Sugarcane Bagasse Functional Groups by FTIR Analysis

The FTIR spectrum results on pure bagasse and those activated with NaOH can be seen in Figure 1. Based on the FTIR results, it can be seen that there are differences in several wavenumbers between pure bagasse and the activated. The change in wavenumber occurs at 3282.99 cm^{-1}, the absorption area of hydroxide (–OH) group, the activated bagasse has no visible peak in that area. However, at wavenumber 33747.85 cm$^{-1,}$ there is a broad peak at $ANSB_{5:1}$ and $NASB_{10:1}$, the O-H functional groups. Sunarsih et al. (2020), in the FTIR results of bagasse modified with KOH, showed that at a wavenumber between 3000-3800 cm^{-1}, there was a vibration of the O-H functional group. These results indicate that hydrogen bonds in O-H, both intra-molecular and inter-molecular. Therefore, this area describes the relationship of cellulose's crystallinity structure that remains the same even though it has been modified with NaOH.

The wavenumber of sugarcane bagasse and NaOH-activated sugarcane are in the range C-H group's 2900.10 cm^{-1}. The wavenumber of sugarcane bagasse and NaOH-activated sugarcane ($NASB_{5:\ 1}$, and $NASB_{10:1}$) is 2925.17 cm^{-1} and 2906.85 cm^{-1}, respectively. The wavenumber for the carboxylate group (C=O) is in 1732.15 cm^{-1}, while the bagasse after activation ($NASB_{5:\ 1}$, and $NASB_{10:\ 1}$) are1703.22 cm^{-1} and 1734.08 cm^{-1}. The C-O group's wavenumber does not have the difference, shown in the wavenumber 2359.04 cm^{-1}. The wavenumbers for bagasse without activation and after activation ($ANSB_{5:1}$, and $ANSB_{10:1}$) are 666.43 cm^{-1}; 672.22 cm^{-1} and 655.83 cm^{-1} are –OCH_3 groups, which confirm the lignin structure in bagasse.

Microstructure and Surface Morphology Observation of Sugarcane Bagasse Adsorbent by Scanning Electron Microscopy (SEM-EDX)

The surface morphology of the pure and activated bagasse adsorbent can be seen in Figure 2. Based on observations, the four samples (SB, $NASB_{5:1}$, $NASB_{10:1}$, and $NASB_{20:1}$) have almost the same morphology.

The results of EDX observations on the surface of the adsorbent can be seen in Table 1. Based on the results of the EDX analysis on bagasse (SB) samples. there are elements of Carbon (C), Calcium (Ca), and Oxygen (O). The highest elemental content is C, with 55.27%, while the lowest element is Ca, about 0.48%. The NaOH-activated bagasse with 5:1 and 10:1 causes Na's appearance, which contains 2.85% and 2.13%, respectively. However, $NASB_{20:1}$ does not contain Na because there is too much bagasse mass used, about 20 gram. This condition causes Carbon in $NASB_{20:1}$ to be higher than other variations, about 56.14%.

Determination of surface area and pore volume using the BET method

The results of determining the surface area and pore volume of bagasse adsorbent before and after activated with NaOH can be seen in Table 2. The chemical activation using NaOH increase the adsorbent surface area. Based on Table 2, the NaOH-activated bagasse adsorbent with a variation of 10: 1 shows the largest surface area, which is 2.803 m^2/g. The total pore volume value of $NASB_{10:1}$ adsorbent also has the highest value: 8,455 x 10^{-3} cm^3/g. Therefore, activation of bagasse adsorbent with NaOH affected the adhesion's surface area and total pore volume.

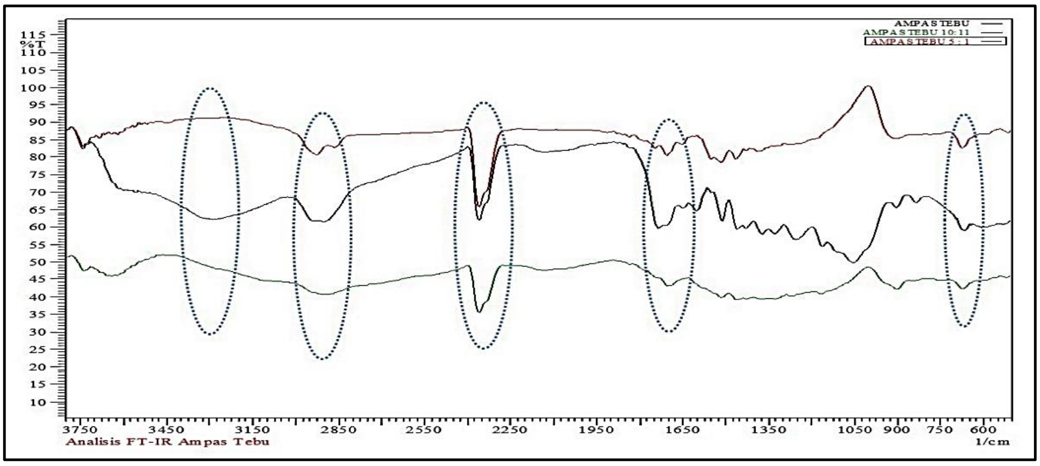

Figure 1. Sugarcane Bagasse (SB, $NASB_{5:1}$, and $NASB_{10:1}$) FTIR Spectrum

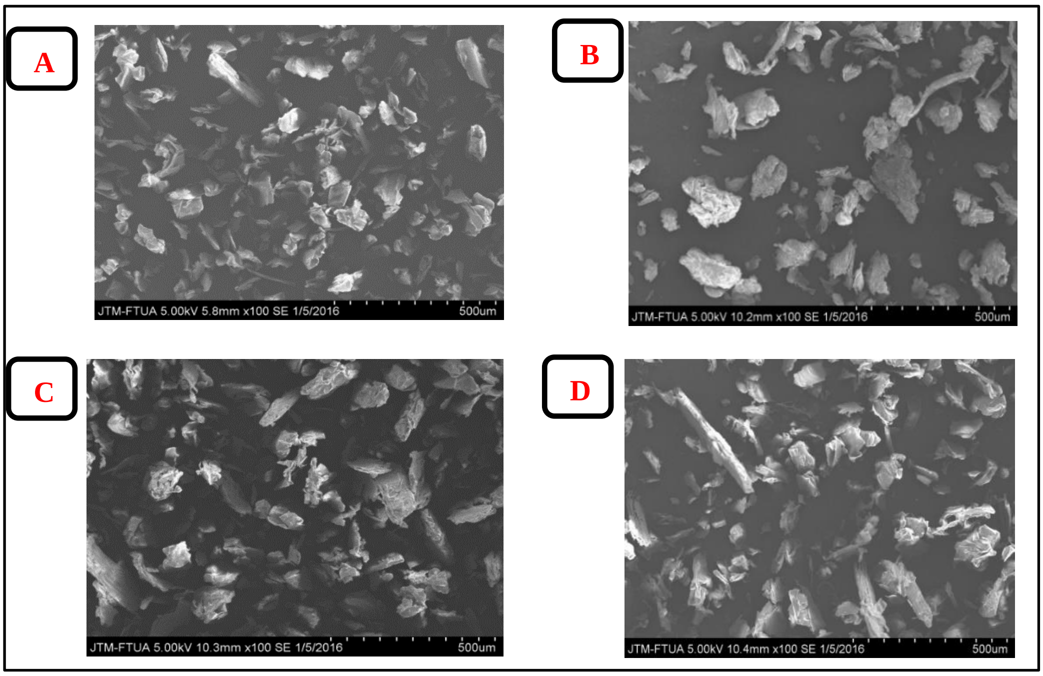

Figure 2. Form of surface morphology adsorbent of sugarcane bagasse (SB) (a), $NASB_{5:1}$ (b), $NASB_{10:1}$ (c), and $NASB_{20:1}$ (d).

Table 1. Elemental analysis on the adsorbent surface of sugarcane bagasse SB, $NASB_{5:1}$, $NASB_{10:1}$, and $NASB_{20:1}$.

Adsorbent	Elemental Analysis (wt%)			
	C	Ca	O	Na
SB	55.27	0.48	44.25	-
$NASB_{5:1}$	43.18	-	44.59	2.85
$NASB_{10:1}$	48.44	0.01	49.42	2.13
$NASB_{20:1}$	56.14	0.01	43.86	-

Note :
SB : sugarcane bagasse
$NASB_{5:1}$: sugarcane bagasse activated by NaOH 5:1
$NASB_{10:1}$: sugarcane bagasse activated by NaOH 10:1
$NASB_{20:1}$: sugarcane bagasse activated by NaOH 20:1

Table 2. Adsorbent texture properties of SB, $NASB_{5:1}$, $NASB_{10:1}$, and $NASB_{20:1}$.

Adsorbent	Surface area (m^2/g)	Total Pore Volume (cm^3/g)	Average Pore Size (Å)
SB	0.991	3.814×10^{-3}	1.53878×10^{2}
$NASB_{5:1}$	1.625	4.422×10^{-3}	1.08858×10^{2}
$NASB_{10:1}$	2.803	8.455×10^{-3}	1.20631×10^{2}
$NASB_{20:1}$	2.037	4.429×10^{-3}	8.69527×10^{2}

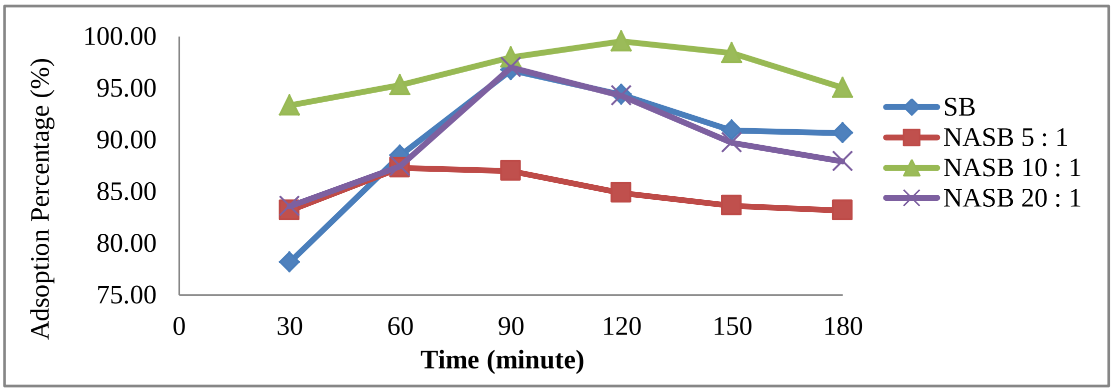

Figure 3. The graph of the effect of time variation on the percentage of adsorption on sugarcane and NaOH-activated sugarcane

Adsorption Process Based on Variations in Contact Time, Adsorbate Concentration and Temperature.

Adsorption of pure and activated bagasse NaOH on methylene blue was carried out with several variations, including contact time, adsorbate concentration, and temperature. Table 3 shows that the results of the treatment of the variations in the adsorbent's contact time show that the optimum time varies for each adsorbent used, which is between 60 to 120 minutes. Pure and $NASB_{20:1}$ occurs the maximum uptake with an adsorption percentage of about 96.73 and 97% at 90 minutes. The fast contact time occurred in activated bagasse adsorbent with $NaOH_{5:1}$, 60 minutes, so the adsorption percentage was 87.27%. The highest adsorption percentage value of all adsorbent variations was at $NASB_{10:1}$ at 99.50% with a longer contact time of 120 minutes. According to Ngapa and Ika (2020), in general, the longer the contact time between the adsorbent and the adsorbate, the more adsorbed the adsorbate will be. The contact between the adsorbent and the adsorbate increases the percentage of absorption power in each bagasse variation. When it reaches the optimum time, it will cause a slight decrease in absorption power percentage at the next time variation. This condition happens because all active sites of the adsorbent are saturated, so that the adsorption capacity has decreased. Besides, the bond between the ions in methylene blue and the active site of the bagasse adsorbent is weak, so that the ions will easily escape back into the solution (Talhutu et al., 2019).

Gusmao, et al. (2012) states that the amount of dye adsorbed at equilibrium shows the dye's maximum adsorption. The uptake of the dye on the activated bagasse occurs rapidly at an early stage (starting from the lowest time), and after that, it slows down until it reaches equilibrium. This condition is because many available adsorbent surface sites are still empty at the initial stage. They begin to be filled with adsorbate ions until all of the adsorbent surface sites are entirely filled. Therefore, after reaching equilibrium, the dye's absorption decreases due to the absence of any more empty surface sites for adsorbate to fill. Following the results obtained on the variation of the adsorption contact time for the $NASB_{10:1}$ adsorbent, it can be seen that the adsorption percentage increases starting at 30 to 120 minutes, namely 99.50%. When the contact time was increased to 180 minutes, the adsorption percentage decreased to 95.04%.

The data of table 4 shows the adsorption percentage value obtained by varying the adsorbate concentration (Methylene Blue). NaOH-activated bagasse with variations in the ratio of 5:1 and 20:1 has a high adsorption percentage at a concentration of 40 ppm. The ability to absorb the power, respectively, was 89.19% and 90.44%. However, the optimum concentration for pure bagasse and 10:1 NaOH-activated was 50 ppm with adsorption percentages of 89.48% and 90.98%, respectively. Based on the above results, it can be concluded that the optimum concentration-time, which has the largest adsorption percentage, is $NASB_{10:1}$ adsorbent with a concentration value of 50 ppm. Increasing the concentration of adsorbate, it will cause the ability to absorb it will be even higher. However, when it reaches the optimum concentration, the absorption power percentage has decreased in all types of adsorbents used. This condition reveals that the adsorption capacity of an adsorbent has gradually reduced.

Table 4. The Adsorption Sugarcane bagasse and NaOH-activated sugarcane bagasse in Methylene Blue with various adsorbate concentrations
($T = 30\ ^oC$, $m = 0.1$ g, $C_o = 10.3198$ mgL^{-1}, $v = 120$ rpm, $V = 0.01$ L).

Number	Adsorbate Concentration (ppm)	Adsorption Percentage (%)			
		SB	$NASB_{5:1}$	$NASB_{10:1}$	$NASB_{20:1}$
1	10	80.36	79.82	86.35	77.65
2	20	81.92	80.20	87.05	78.48
3	30	84.13	86.37	89.41	88.28
4	40	86.92	89.19	89.56	90.44
5	50	89.48	88.07	90.98	89.71
6	60	86.39	79.12	84.93	80.09

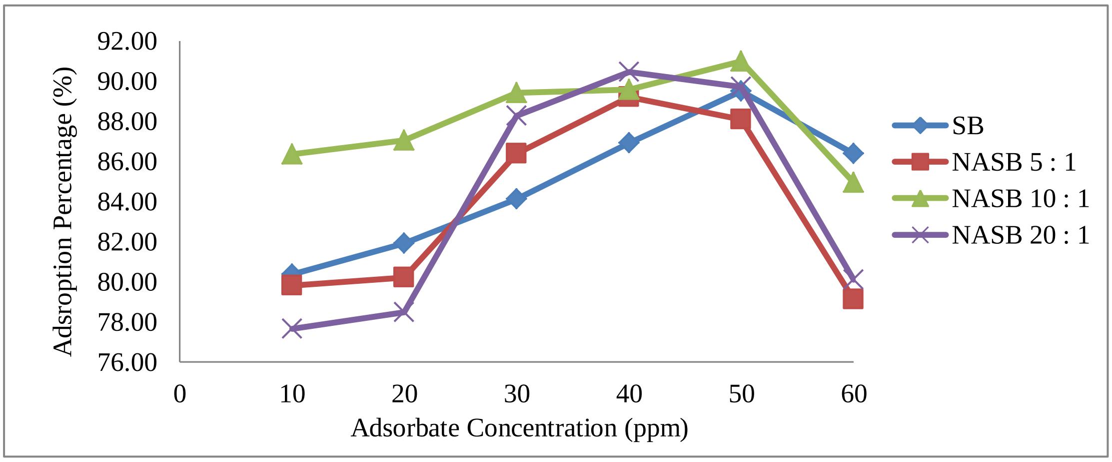

Figure 4. The graph of effect of adsorbate concentration variation on the percentage of adsorption on sugarcane and NaOH-activated sugarcane.

Table 5. Adsorption of pure and NaOH-activated bagasse in Methylene Blue with temperature variations (m = 0.1 g, v = 120 rpm, V = 0.01 L).

Number	Temperature (K)	Adsorption Percentage (%)			
		SB	$NASB_{5:1}$	$NASB_{10:1}$	$NASB_{20:1}$
1	303	87.05	88.82	90.75	89.56
2	313	85.09	85.01	87.05	82.36
3	323	79.31	74.88	82.55	77.08
4	333	68.92	67.54	78.85	70.47

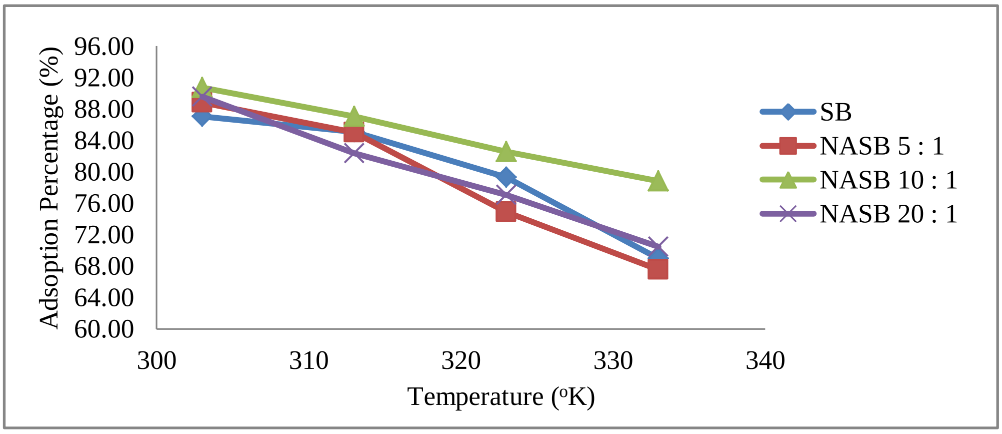

Figure 5. The graph of the effect of temperature on the percentage of adsorption on sugarcane and NaOH-activated sugarcane.

Adsorption of pure and NaOH-activated bagasse in Methylene Blue by varying the temperature can be seen in table 5. The optimum adsorption of all adsorbents is obtained at 30 °C (303 K). The highest adsorption percentage is 90.75% ($NASB_{20:1}$), and the lowest is 87.05% (SB). Based on the data in table 5, it can be concluded that the increasing temperature in the adsorption process causes the adsorption percentage value to decrease. Based on the data obtained, it shows that the temperature is inversely related to the absorption power. This condition indicates that the adsorption process is exothermic, possibly because the higher temperature causes methylene blue's solubility to increase. The attraction between the solution and the solvent is stronger than the solution adsorbent. As a result, the solute is more challenging to absorb.

CONCLUSION

Bagasse can be used as an adsorbent by carrying out chemical activation using NaOH with variations 5:1 ($NASB_{5:1}$), 10:1 ($NASB_{10:1}$), and 20:1 ($NASB_{20:1}$). The adsorbent surface area's characterization results obtained the highest value for the $NASB_{10:1}$ type of adsorbent than other adsorbents, namely 2.803 m^2/g. $NASB_{10:1}$ adsorbent can perform adsorption maximally in the methylene blue solution with an adsorption percentage of about 99.50%. The optimum conditions for $NASB_{10:1}$ adsorbent are contact time of 120 minutes, adsorbate concentration of 50 ppm, and temperature 30 °C or 303 K. Bagasse adsorbents, pure and NaOH-activated, are effectively in absorbing methylene blue dye. The adsorption percentage obtained is very high for all absorbents that have been activated with NaOH, and the variation in parameters is about 70.0% - 99.0%.

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