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ADSORPTION OF REMAZOL BRILLIANT BLUE DYE USING PALM OIL SHELL FLY ASH HCl ACTIVATED

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Abstract

Remazol Brilliant blue is an azo dye that is widely used in textile dyeing. The most appropriate handling method to be used in overcoming problems caused by textile dye waste is adsorption. Fly ash is an adsorbent that can be used to overcome this problem. Chemical activation was carried out using 1 M HCl solution. Besides that, physical activation was also carried out at 500°C for 1 hour. Research is needed to determine the optimum conditions for fly ash in dye adsorption to produce high adsorption efficiency. XRF characterization showed that palm shell fly ash was dominated by CaO of 71.064% and SiO_2 of 15.734%. Characterization using FTIR shows the presence of Si-O groups in Fly ash. To analyze the surface morphology of the fly ash adsorbent, an SEM test was carried out and it was known that the surface morphology of the fly ash adsorbent after chemical and physical activation showed pore formation. Characterization using Surface Area Analyzer showed a surface area of 13.6153 m^2/g. optimum absorption conditions at pH 6 with an adsorption capacity of 15.84 mg/g. optimum contact time of 60 minutes with an adsorption capacity of 15.54 mg/g. The optimum adsorbent mass is 0.5 g with an adsorption capacity of 9.54 mg/g and the optimum adsorbent concentration is at 200 ppm with an adsorption capacity of 69.08 mg/g. In this study the adsorption model used is the Freundlich isotherm. Based on the research result, it is known that fly ash is a suitable adsorbent for dye adsorption which is characterized by high color removal efficiency. Further characterization regarding the initial conditions of fly ash is needed as a comparison for fly ash after activation.

INTRODUCTION

The textile industry in Indonesia has developed quite rapidly from year to year. Of the several classes of synthetic dyes, azo dyes are the most frequently used dyes in the textile industry. The increasing use of textile dyes will increase the amount of dye waste which can cause environmental pollution, the textile industry is an industry that contributes quite a lot of dye waste. The characteristic of azo group compounds is that they have bonds (-N=N-) which are also called azo groups [1]. Until now, it is estimated that azo dyes in the world have been used for around 50-70% of the total use of dyes in all world industries. During the dying process, 10–15% of the textile dyes used will be wasted with the waste. Remazol Brilliant blue is an azo dye that is widely used in textile dyeing because it produces bright colors and does not fade easily. The most appropriate handling method to be used in overcoming problems caused by textile dye waste is adsorption. Fly ash is an adsorbent that can be used to overcome these problems.

Fly ash can be obtained from burning in the palm oil factory boiler. Indonesia is the largest palm oil producing country in the world. An increase in palm oil production will produce waste in the form of palm shells which can be used as fuel in palm oil factory boilers. Fly ash is classified as a Toxic Hazardous Material (B3). To overcome the problem of hazardous waste due to the accumulation of fly ash, fly ash can be used as an adsorbent to treat dye waste. Some of the components contained in palm fly ash are SiO_2 of 19.189%, Fe_2O_3 of 1.78%, Calcium oxide (CaO) of 68.894%. This component plays a role in the adsorption process [2].

Fly ash without activation has low adsorption capacity, chemical modification of fly ash can increase adsorption capacity [3]. The activation process is carried out so that Fly ash has a higher adsorption capacity, because the activation process functions to activate the pores and surface of the adsorbent. Fly ash adsorption ability can be increased by using HCL activator. This activation process can help remove impurities that cover the pores of fly ash [4]. The high composition of fly ash

components after activation such as SiO_2, Fe_2O_3, and CaO causes the high adsorption efficiency of remazol brilliant blue.

EXPERIMENTS

Material

Palm shell fly ash obtained from the Wuxi Boiler PT.Wilmar Nabati Indonesia Padang, distilled water, Merck's HCl 37%, Remazol Brilliant blue dye, Merck's HNO_3 and Merck's NaOH.

instrumentation

The tools used in this research are X-Ray Fluorescence (XRF) Panalirical Epsilon 3, Fourier Transform Infra-Red (FTIR) UATR PerkinElmer Frontier C90704 Spectrum IR Version 10.6.1, Scanning Electron Microscope (SEM) FEI Inspect-S50 and Surface Area Analyzer (SAA) Micromeritics Tristar II Plus.

Procedure

Chemical Activation

Chemical activation was carried out using HCl. Fly ash was activated by adding 1 M HCl with a ratio of Fly ash and HCl (1:4) at a heating temperature of 90°C and stirring for 1 hour. The activated fly ash was then washed using distilled water. Fly ash is then dried in an oven at 110°C [4].

Physical Activation

Physical activation is done by heating at high temperatures. Fly ash that has been chemically activated using 1 M HCl is then heated in a furnace at 500 °C for 1 hour [5].

Adsorption Test

The adsorption test was carried out to determine the pH, contact time, mass of the adsorbent and the optimum adsorption concentration of remazol brilliant blue. Remazol brilliant blue 20 mL solution with a concentration of 25 ppm was added with 0.3 g of adsorbent. Adjusted the pH of solutions 1, 2, 3, 4, 5, 6, 7 and 8 by adding 0.1 M HNO_3 and 0.1 M NaOH. Then stirred for 30 minutes using a magnetic stirrer. Optimum contact time variations were set at 15, 30, 45, 60, 75 and 90 minutes with the solution conditions at optimum pH. The mass variation of the adsorbent is set at 0.05; 0.1; 0.2; 0.3; 0.4 and 0.5 g at optimum pH and contact time. For the optimum adsorbate concentration, various concentrations of 25, 50, 75, 100, 150 and 200 ppm were used at the optimum pH, contact time and adsorbent mass. After the adsorption process, the adsorbent was filtered using Whatman filter paper no. 4. The filtrate obtained was then analyzed using a uv-vis spectrophotometer. Adsorbed remazol brilliant blue was calculated using the following equation.

$$\% \text{ Adsorption efficiency} = \frac{(C_{awal} - C_{akhir})}{C_{awal}} \times 100\% \quad [3]$$

$$\text{Adsorption capacity} = \frac{(C_0 - C_e)v}{m} \quad [3]$$

The adsorption process that occurs at constant temperature can be determined using the adsorption isotherm. The Langmuir and Freundlich isotherm models are the most commonly used adsorption isotherm models. The following is the equation for the two adsorption isotherm models.

$$\text{Log Qe} = \log Kf + (1/n) \log Ce$$

(Freundlich isotherm) [4]

$$\frac{C_e}{Q_e} = + \frac{1}{Q_m} C_e \frac{1}{K_l Q_m}$$

(Lanmuir isotherm) [4]

RESULTS AND DISCUSSION

Chemical Activation

The activation process with HCl aims to remove impurities from the surface of the adsorbent so that the pores of the adsorbent will be more open and the surface area of the adsorbent will increase so that the adsorption capacity of the adsorbate will be even greater. the use of HCl as an activator aims to reduce the concentration of iron oxide and other oxides found in fly ash [6]. Fly ash which had been added with HCl was then stirred at 90°C for 1 hour using a magnetic stirrer. This process aims to increase the contact or interaction between Fly ash and HCl as an activator so that more impurities can dissolve as chloride. After 1 hour Fly ash was then rinsed using distilled water to clean the remaining activator. The dried fly ash is then followed by a physical activation process.

Physical Activation

This activation process aims to evaporate the water contained in the adsorbent until the adsorbent

is free from water content and to remove the organic components contained in the adsorbent. Heating at high temperatures can expand the surface of the adsorbent because the pores of the adsorbent are increasingly open so that it can increase the adsorption capacity of the dyes to be absorbed. The physical activation process carried out using an activation temperature of 500°C showed an adsorption capacity value of 0.476 mg/g [5].

pH Variation Adsorption Test

In this study, palm shell fly ash adsorbent was used to test the effect of pH on the adsorption of remazol brilliant blue. In this experiment, pH variations of 1, 2, 3, 4, 5, 6, 7 and 8 were used. The effect of pH on the adsorption process of remazol brilliant blue is shown in **Figure 1** and **Figure 2**.

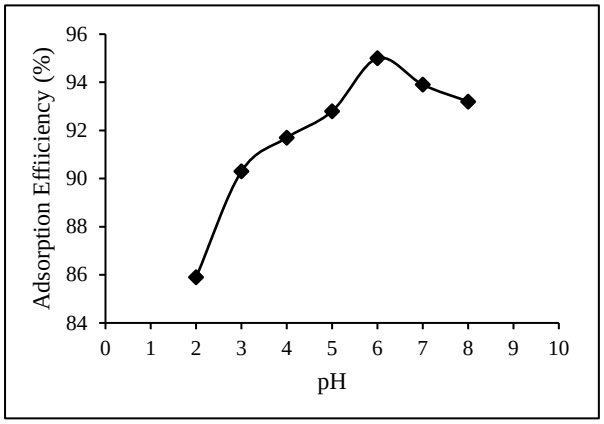

Figure 1. Graph of variations in pH and adsorption efficiency.

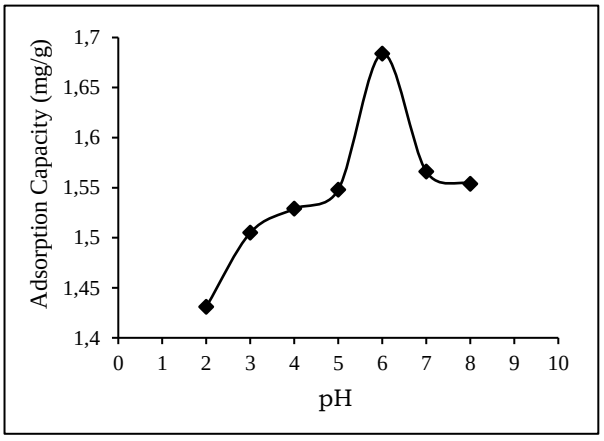

Figure 2. Graph of variations in pH and adsorption capacity.

Based on the graph, it can be seen that the optimum pH for the absorption of remazol brilliant blue is at pH 6. With an adsorption efficiency of 95%. The results of the absorption of pH 2 to 6 showed an increase in adsorption efficiency. When the pH is above 6 there is a decrease in adsorption efficiency because at that pH there are many OH^- ions in the solution which results in reduced adsorption efficiency. In research [7] the optimum pH conditions for the absorption of remazol brilliant blue at pH 6 were obtained with the highest adsorption efficiency of 82%. at pH 2 the adsorption capacity for remazol brilliant blue solution was 1.431 mg/g and continued to increase until the adsorption optimum pH was at pH 6 with an adsorption capacity of 1.584 mg/g.

Contact Time Variation Adsorption Test

Optimum adsorption contact time is the time required for the adsorbent to absorb the adsorbate optimally (**Figure 3** and **Figure 4**). The longer the contact time between the adsorbent and the adsorbate, the more adsorbate will be adsorbed due to the increasing number of interactions between the adsorbent and the adsorbate [8].

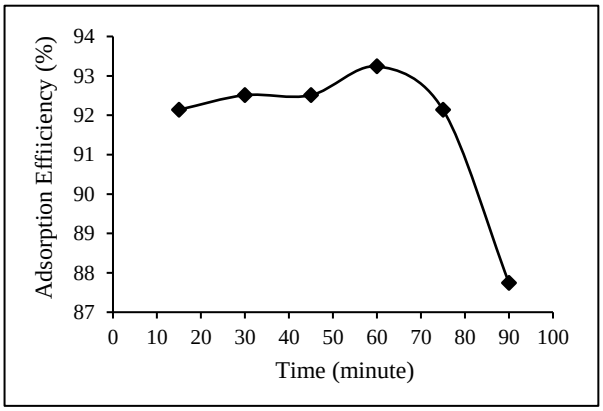

Figure 3. Graph of variations in contact time and adsorption efficiency.

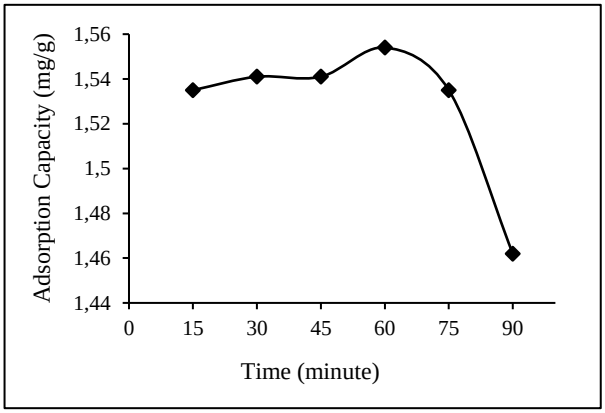

Figure 4. Graph of variations in contact time and adsorption capacity.

Based on the graph, it can be seen that the optimum contact time is 60 minutes with an

adsorption efficiency of 93.24%. The absorption efficiency increases until the optimum contact time is reached, then decreases after the optimum contact time is reached because the adsorbent is in a saturated state [9]. At the beginning of the 15-minute adsorption time, the efficiency continued to increase because the entire pore surface of the adsorbent was still empty so that it would be filled with adsorbate molecules. at a contact time of 30 minutes, the adsorption capacity was 1.541 mg/g and continued to increase until the contact time was 60 minutes with an adsorption capacity of 1.554 mg/g.

Adsorption Test of Adsorbent Mass Variation

Determination of the optimum adsorbent mass aims to determine the adsorbent mass required to optimally adsorb remazol brilliant blue dye. Determination of the optimum adsorbent mass for the adsorption of remazol brilliant blue was carried out by varying the mass of the adsorbent 0.05; 0.1; 0.2; 0.3; 0.4 and 0.5 g. The results obtained can be seen in the **Figure 5** and **Figure 6**.

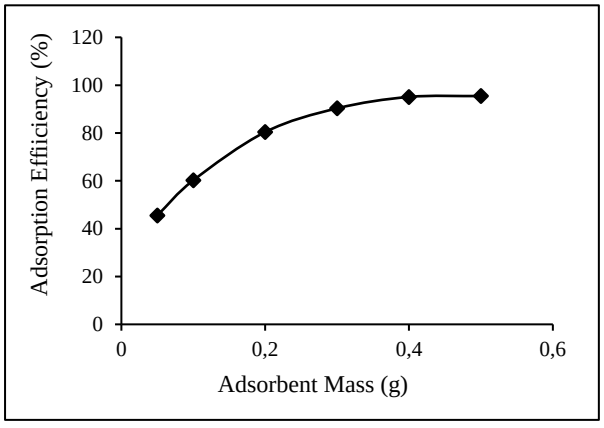

Figure 5. Graph of adsorbent mass variation and adsorption efficiency.

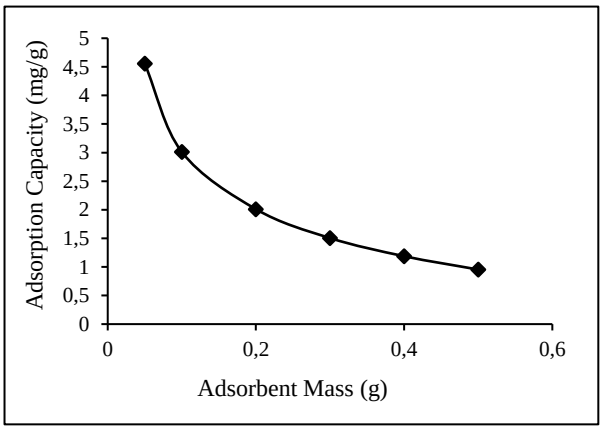

Figure 6. Graph of variations in adsorbent mass and adsorption capacity.

The adsorption efficiency of remazol brilliant blue increased with increasing mass of the adsorbent until the highest adsorption efficiency was obtained at 95.45% with 0.5 g of adsorbent. At the beginning of adding the adsorbent to the solution, there is an increase in surface area, causing more and more adsorbate to be absorbed by the adsorbent. That is, the more adsorbent used causes more adsorbate to be absorbed [10].

Based on research conducted by [11] it is known that the adsorption efficiency of dyes increases with increasing mass of the adsorbent which is associated with an increase in the surface area of the adsorbent and an increase in efficiency is obtained from 78% to 94%. The adsorption capacity of remazol brilliant blue decreased with increasing adsorbent mass. The decrease in adsorption capacity is due to the active side of the adsorbent which has not absorbed the adsorbent. The highest adsorption capacity was obtained at a mass variation of 0.5 g adsorbent with an adsorption capacity of 4.554 mg/g.

Adsorption Test of Adsorbate Concentration Variations

In this study, a variation of the initial concentration of the adsorbate was used to determine the optimum initial concentration for reducing the levels of dyes in the solution. Variation of the concentration used is 25; 50; 75; 100; 125 and 200 ppm. The adsorption process was carried out at the optimum pH, contact time and adsorbent mass of 0.5 g. The higher the initial concentration of an adsorbate, the higher the final concentration because many adsorbate molecules are not absorbed by the adsorbent [12]. The results obtained can be seen in the following **Figure 7** and **Figure 8**.

The graph shows that the higher the concentration, the adsorption efficiency will decrease. The highest adsorption efficiency was obtained at an initial concentration of 25 ppm adsorbate with an adsorption efficiency of 96.55% and the smallest adsorption efficiency was obtained at an initial concentration of 200 ppm adsorbate and an efficiency of 86.35%. This decrease in adsorption efficiency is due to the increasing amount of remaining adsorbate and can no longer be absorbed by the adsorbate thereby reducing adsorption efficiency. The higher the concentration of the adsorbate, the adsorption capacity will also be higher. The increase in adsorption capacity is due to the increasing number of adsorbate molecules that are able to be absorbed by each unit weight of the adsorbent.

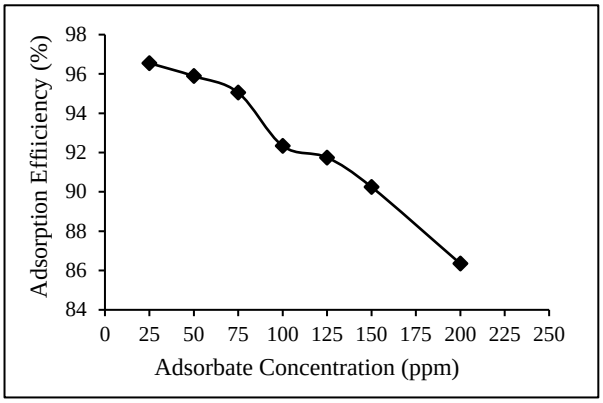

Figure 7. Graph of variations in adsorbate concentration and adsorption efficiency.

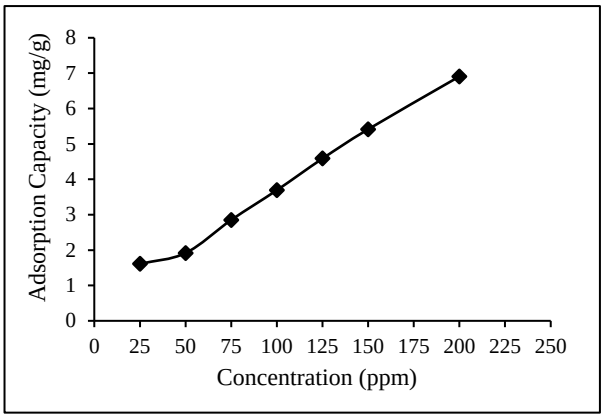

Figure 8. Graph of variations in concentration and adsorption capacity.

Adsorption Isotherm

Adsorption isotherms describe the relationship between the substance adsorbed by the adsorbent and the concentration at a constant temperature [13]. Determination of the adsorption isotherm pattern was carried out using a solution of remazol brilliant blue with several different concentrations, namely 25, 50, 75, 100, 125, 150 and 200 ppm. The mass of the adsorbent used is 0.5 g. Determination of the adsorption isotherm aims to determine the adsorption mechanism that occurs in the fly ash adsorbent in the adsorption of remazol brilliant blue. When the adsorption process tends to follow the Langmuir isotherm model, it means that the adsorption process takes place in a monolayer. Where the adsorbate molecule will be adsorbed only at certain sites. That is, each site accommodates only one adsorbate molecule [14]. Meanwhile, when the adsorption process tends to follow the Freundlich isotherm model, it means that the adsorption process takes place in a multilayer or multilayer layer formation occurs [15]. The results obtained can be seen in the following **Figure 9** and **Figure 10**.

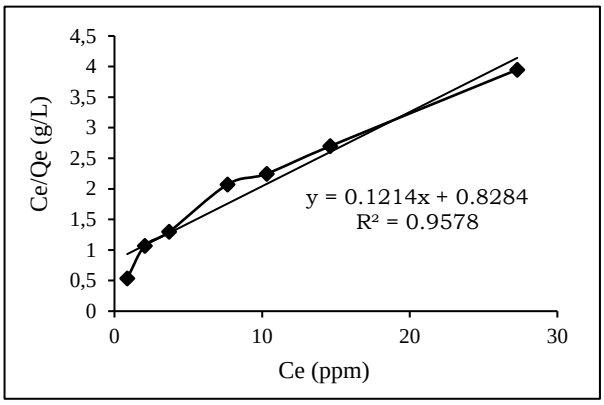

Figure 9. Langmuir isotherm graph.

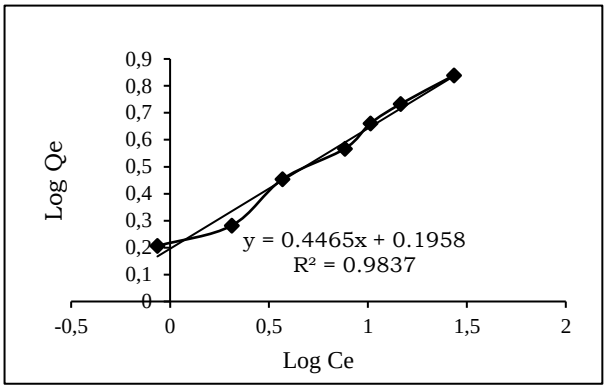

Figure 10. Freundlich isotherm graph.

Determination of the adsorption isotherm pattern was obtained from the data of variations in the concentration of the adsorbate and plotted into the curve. Based on the graph, it is known that the adsorption isotherm model that is suitable for this study is the Freundlich isotherm. This is because the data on the Freundlich isotherm curve has a larger linear regression value than the linear regression value on the Langmuir isotherm curve. The adsorption process that occurs can be seen in the following **Figure 11**.

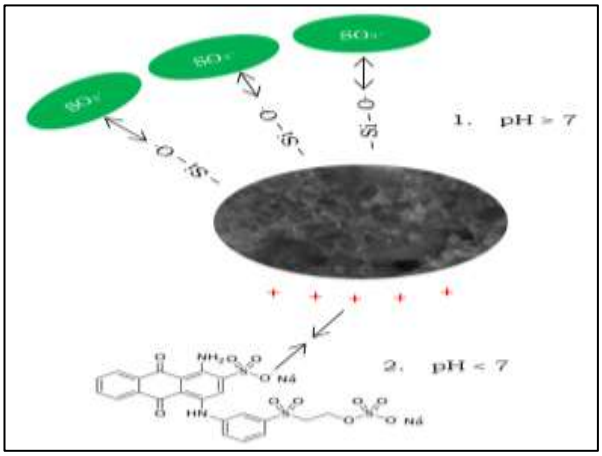

Figure 11. Adsorption mechanism of remazol brilliant blue on fly ash.

When the pH of the solution is below 6.58, the fly ash adsorbent will be positively charged, whereas when the pH of the solution is above 6.58, the fly ash adsorbent will be negatively charged [16]. At acidic pH, there is a difference in charge between the surface of the adsorbent and the sulfonate group (SO_3^-) of the adsorbate. This charge difference causes a pulling interaction between the adsorbate and the adsorbent so that the adsorbate can be attracted to the surface of the adsorbent. In the adsorption process of remazol brilliant blue by fly ash, the adsorption process that occurs on the surface forms a multilayer. The adsorbate will accumulate on the surface of the adsorbent to form the first layer, then the adsorbate will continue to be adsorbed on the surface of the adsorbent until it forms a multilayer [25].

Characterization

X-Ray Fluorescence (XRF)

The fly ash used in this study comes from industrial waste from PT. Wilmar Nabati Indonesia Padang. The XRF test carried out aims to determine the chemical composition contained in the fly ash. The XRF test results are shown in the following **Table 1**.

Table 1. Fly ash chemical composition.

Component	Percentage (%)
SiO_2	15,734
Al_2O_3	0.31
CaO	71,064
Fe_2O_3	3,074

XRF results on Fly ash show that the chemical composition of Fly ash originating from PT. Wilmar Nabati Indonesia Padang is dominated by CaO of 71.064% and SiO_2 of 16.734%. fly ash contains 47.778% SiO_2, 20.646% CaO, 4.304% Fe_2O_3 and 6.324% MgO [2]. Based on research [17], palm shell fly ash contains SiO_2 of 19.189%, Fe_2O_3 of 1.78%, Calcium oxide (CaO) of 68.894%.

Fourier Transform Infrared (FTIR)

The results of the characterization of the functional groups of Fly ash after activation and after adsorption of remazol brilliant blue can be seen in the following **Figure 12**.

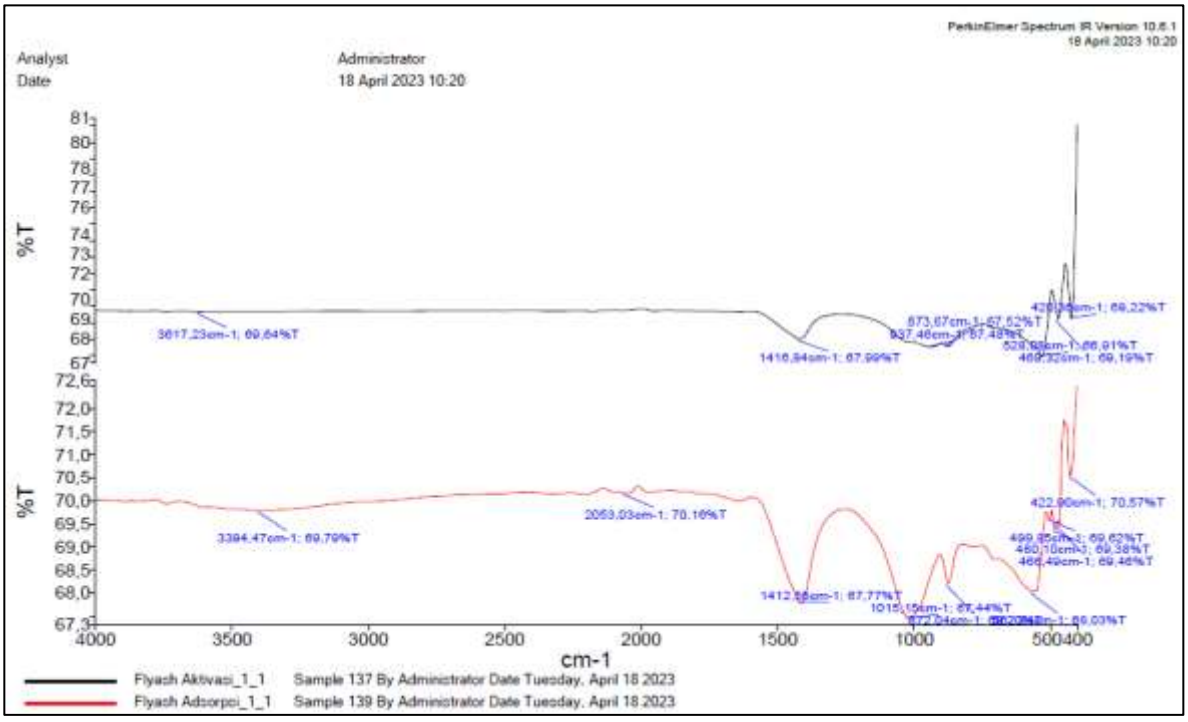

Figure 12. FTIR spectrum of fly ash.

Based on the figure it can be seen that there is absorption at wave numbers 469.32 cm^{-1} and 466.49 cm^{-1} which are bending vibrations of Si-O-Si [23]. In the region of wave numbers 550-330 cm^{-1} it shows absorption from Si-O bending vibrations. In the spectrum obtained, there are wave numbers 528.08 cm^{-1} [24]. Absorption at wave numbers 420.36 cm^{-1} and 422.90 cm^{-1} indicates the presence of Ca-O groups. Absorption at wave number 1412.15 cm^{-1} indicates the presence of S=O groups

associated with sulfonate groups. This sulfonate group is present in the structure of remazol brilliant blue which is the adsorbate in this study (**Table 2**).

Table 2. Wavenumber and functional groups of palm shell fly ash.

Wavenumber (cm^{-1})		
Fly ash activation	*Fly ash* adsorption	Functional groups
420.36	422.90	Ca-O
469.32	466.49	Si-O-Si
528.08	562.84	Si-O
-	1412.15	S=O
3617.23	3394.47	OH

Scanning Electron Microscope (SEM)

Characterization using SEM instruments aims to determine and analyze the surface morphology of the fly ash adsorbent (**Figure 13**).

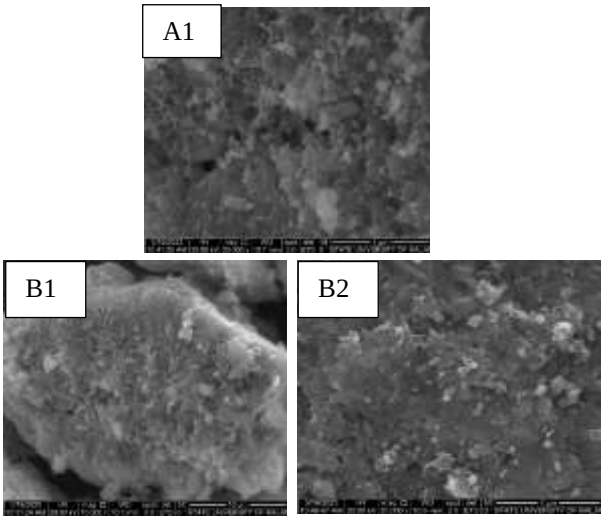

Figure 13. Fly ash SEM results; a) Fly ash after activation; b) Fly ash after adsorption of remazol brilliant blue.

The adsorption process that occurs is influenced by the surface morphology of an adsorbent. A material can be used as an adsorbent if the material has pores, where these pores play a role in the adsorbate absorption process [18]. Pictures A1 and B1 with a magnification of 10,000 times, and Pictures B2 with a magnification of 25,000 times. At 10,000 x magnification, it can be seen that the surface of the adsorbent is not smooth and there are not many pores visible. At a magnification of 25,000x (B2) you can see quite large pores in the adsorbent. The activation process also affects the surface morphology of the adsorbent, where the activation process can help open the pores on the adsorbent surface. Based on the SEM results for Fly ash after activation, it can be concluded that Fly ash can be used as an adsorbent material for adsorption of remazol brilliant blue because it has a porous surface. Figure B shows the surface morphology of Fly ash after adsorption on remazol brilliant blue. After the adsorption process, the pores on the surface of the adsorbent appear to be covered by the adsorbate, and some of the pores on the adsorbent have been filled with adsorbate molecules indicating that the adsorption process of remazol brilliant blue by the fly ash adsorbent has taken place.

Surface Area Analyzer (SAA)

This characterization was carried out to determine the surface area, pore volume and pore size of the Fly ash adsorbent. The data obtained is in the form of surface area, pore volume and pore diameter as follows (**Table 3**).

Table 3. Results of characterization of SAA fly ash.

Fly ash	
Surface Area	13.6153 m^2/g
Pore Volume	0.134344 cm^3/g
Pore Size	39.1864 nm

The greater the surface area of an adsorbent, the greater the adsorption capacity due to the large amount of adsorbate that can be absorbed by the adsorbent [19]. The pore size is related to the ability of the adsorbent to absorb the adsorbate. The greater the number and size of the pores, the greater the adsorption capacity. The larger the pore diameter, the larger the pore volume [20]. Based on the data table it is known that the fly ash adsorbent has a pore size of 39.1865 nm. Where the pore size is included in the mesoporous material which has a pore size range between 2-50 nm [21]. Fly ash adsorbent can be categorized as a good adsorbent because it has a large pore volume and surface area. The characteristics of a good adsorbent are that it has a large pore volume and surface area so that it can absorb more adsorbate [22].

CONCLUSION

Based on the research results, it is known that the optimum condition for the absorption of remazol brilliant blue by the palm shell fly ash

adsorbent is at pH 6 and the adsorption capacity is 1.584 mg/g. The optimum contact time is 60 minutes and the adsorption capacity is 1.554 mg/g. The optimum adsorbent mass is 0.5 g and the adsorption capacity is 4.554 mg/g. The optimum adsorbate concentration is 200 ppm with an adsorption capacity of 6.908 mg/g.

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