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PREPARATION AND CHARACTERIZATION OF ACTIVATED CHARCOAL FROM BAMBOO WASTE WITH PHOSPHORIC ACID AS A BIOSORBENT FOR RHEMAZOL BRILLIANT BLUE

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ABSTRACT

Bamboo waste can be converted into materials of high economic value when processed into activated charcoal which is useful as an adsorbent. This study aims to make activated charcoal from bamboo waste with 20%(w/v) phosphoric acid as an activator. The activated charcoal produced was used as an adsorbent for rhemazol brilliant blue dye. Characterization of activated and inactivated charcoal was carried out by referring to the Indonesian National Standard-1995. Contact time and pH were studied for optimizing the adsorption. The results showed that the optimum contact time was 60 minutes, and the pH of the solution was 2. Isotherm adsorption of both charcoals tends to follow Langmuir isotherm, and the adsorption capacity obtained was 12.2399 and 9.5697 mg/g for activated and non-activated charcoals respectively. Adsorption kinetic charcoals follow to pseudo-second order. Both charcoals are thought to contain functional groups of hydroxy, carbonyl, and aliphatic double bonds and have heterogeneous surfaces.

Keywords: Activated Charcoal, Adsorption, Bamboo, Biosorbent, Rhemazol.

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INTRODUCTION

Activated carbon is a multifunctional adsorbent and has been used intensively for various purposes such as in water purification, the pharmaceutical industry, wastewater treatment, and waste gas treatment. Commercial activated carbon is so expensive that recent attention has been directed to providing cheap carbon from renewable materials.[1,2] Activated carbon can be made from materials containing high concentrations of carbon, through a carbonization process at high temperatures under limited oxygen amounts.[3] Charcoal raw materials can be rice straw[2], wood[4], bamboo[5], coconut shell[6], apple waste[7], fruit stem[8], rapeseed, seaweed[9], and others.[10] Activation of charcoal can be done through physical or chemical methods. The physical activation is carried out by flowing aspirating N_2 gas, or water vapor at high temperatures. The chemical activation is through adding chemicals such as acids, bases, and salts such as HCl, H_2SO_4, H_3PO_4, KOH, NaOH, $ZnCl_2$, and K_2CO_3. Activation is intended to remove impurities that cover the pores in the form of tar, metal oxides, and volatile substances so that the charcoal's total volume and surface area increase. This will affect the adsorption capacity of the activated charcoal.[3,11,12] At the dyeing stage, the textile industry tends to use reactive dyes, acid dyes, dispersions, or bases. *Rhemazol brilliant blue* (Rbb) is a reactive dye, which has become a favorite choice as a basic dye because it is easily soluble in water and gives a bright color. However, the waste in the form of residual dye is toxic and carcinogenic to aquatic life. This negative impact can be minimized or even eliminated by the adsorption process using activated charcoal. Bamboo waste has a sufficient abundance and is easy to find in Bali, especially in Industrial Craft centers or building remains. This material can be processed into activated charcoal which has high economic value. The processing of bamboo into activated charcoal has been reported by previous researchers.[5,13] This research is focused again on the manufacture of activated charcoal from bamboo waste with an activator of 20%(w/v) phosphoric acid. The activated charcoal obtained was used as an adsorbent for Rbb.

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EXPERIMENTAL

Materials

The materials needed for this research are bamboo waste obtained from the handicraft industry center in Denpasar. The chemicals used in this study included H_3PO_4, distilled water, rhemazol brilliant blue dye powder, HCl, NaOH, I_2 solution, starch, methylene blue powder, and $Na_2S_2O_3$ 0.1N. All of the chemicals were of analytical quality provided by Merck & Co. Figure-1 shows the structure of rhemazol brilliant blue.

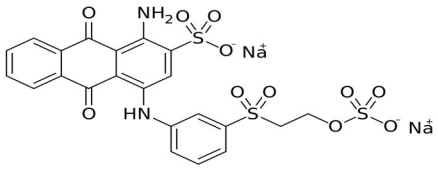


Fig.-1: Structure of Rhemazol Brilliant Blue

Carbonization and Activation

The preparation of activated charcoal follows the Indonesian National Standard (SNI) 06 3730-1995, and Manurung.[5,14] The bamboo waste was cut and chopped into small pieces up to ±1 cm, then washed with water. The samples were then oven-dried at 105°C to constant weight. A total of 1000 g of dry samples were carbonized in a kiln for 90 minutes at 600ºC. The sample that has become charcoal was cooled in a desiccator and weighed to determine the yield. Then the carbonized charcoal was ground until smooth and sieved through a 200-mesh sieve, divided into two portions, and labeled as B_A (activated) and B_0 (inactivated). A total of 100 g of B_A charcoal was soaked in 250 mL of 20% (w/v) H_3PO_4 solution for twenty-four hours. Then was filtered and washed with water until pH neutral. The charcoals were oven dried at 105ºC to constant weight. Then characterized according to the SNI-1995 procedure, including water, volatile matter, total ash, carbon content, Iodine, and methylene blue number.

Preparation of Rbb Dye Solution and Determination of Maximum Wavelength

One gram of the Rbb dye powder was carefully weighed, dissolved with distilled water in a 1000 mL volumetric flask, then diluted to the mark. From this solution, 1.0 mL was diluted into 100 mL with distilled water to obtain a 10 mg/L Rbb solution. The absorbance of the solution was measured using a UV-Vis Spectrophotometer at a 400 – 700 nm range.

Determination of Contact Time

The optimum contact time of B_A and B_0 charcoal against Rbb dyes was determined by stirring 25 mL of 30 mg/L Rbb dye with 0.5 g of each charcoal in 100 mL Erlenmeyers at 300 rpm for 15, 30, 45, and 60 minutes. The mixtures were then filtered, and the filtrates were measured for their absorbances using a UV-Vis at the Rbb maximum wavelength. The amount of dye adsorbed (mg/g) and the efficiency of adsorption (%) of the adsorbent were calculated using equations 1 and 2 respectively.[15,16]

$$Q_t = V\,(\,Co\text{-}Ct)/Wa \qquad (1)$$

$$E_f = \frac{Co-Ct}{Co} \times 100\ \% \qquad (2)$$

$$Qe = \frac{(Co-Ce)V}{Wa} \qquad (3)$$

Where:

Qt is the mass of dye adsorbed per gram adsorbent at time t (mg/g), Qe is the mass of dye adsorbed per gram adsorbent at equilibrium (mg/g), Co is the initial dye concentration (mg/L), Ce is the concentration of dye at equilibrium,(mg/L), Ct is the remaining dye concentration after adsorption at time t (mg/L), V is the volume of solution (L), Wa is the amount of adsorbent (g), and Ef is the efficiency of adsorption (%). The optimum contact time was obtained by making a curve between the contact time and the amount of Rbb dye adsorbed (mg/g).

Effect of pH on Adsorption Capacity

For each B_A and B_0, four 25 mL of 150 mg/L Rbb solution were prepared in 100 mL Erlenmeyers. The pH of the solutions was adjusted from 2, 4, 6, and 8 with the addition of 0.1 M HCl or 0.1 M NaOH solutions. Into each solution was added 0.5 g of charcoal (B_A or B_0). The mixtures were stirred at 300 rpm for the optimum time, then filtered and the filtrates were measured for their absorbances with a UV-Vis spectrophotometer at the maximum wavelength of Rbb. The amount of adsorbed dye (mg/g) of adsorbent (B_A) was determined using Equation 3.

The Adsorption Capacity

The determination of the adsorption capacity was carried out by stirring 25 mL of Rbb dye solution with various concentrations of 50, 100, 150, 200, 250, and 300 mg/L with 0.5 g of B_A or B_0 charcoal at a speed of 300 rpm under the optimum conditions, The mixtures were then filtered, and the filtrates were measured for their absorbances. The amount of dye adsorbed and the efficiency of adsorbent was calculated by equation 3.

Determination of Functional Groups and Surface Aspects

The functional group present in the B_A and Bo charcoal was determined by infrared spectroscopy (FTIR), using the KBr thin paste technique, and the topography surface was determined by SEM.

RESULTS AND DISCUSSION

Carbonization and Activation

The carbonization process was carried out at a temperature of 600ºC, for 90 minutes to assist changes in the physical appearance of the sample before and after the heating process. Bamboo is made into small pieces to facilitate the carbonization process. Figure-2a shows the bamboo raw material that has been dried and is brown in color. Figure-2b shows bamboo that has undergone carbonization and turned into charcoal, black in color, slightly brittle, and not sticky.[3,5] The yield obtained was 16.2%. This result is lower, compared to the study of Manurung which is 18.92-22.13%.[5] This is because the sample used was a mixture of bamboo waste from various bamboo species. The high yield is generally influenced by the type and quality of raw materials, temperature, and the carbonization process.[3,9] Both activated and inactivated charcoals were heated at 900 ºC for 15 minutes, and the result is shown in Fig.-2c and 2d. Heating at high temperatures for 15 minutes is intended to remove impurities, and tar, and to open the pores of the charcoal, without destroying the crystal structure of activated carbon. Visually it can be seen that the appearance of activated (B_A) and inactivated (Bo) charcoal is different, for instance, B_A charcoal is dark black, and shiny (Fig.-2c) compared to Bo and opaque (Fig.-2d). This is because the 20%(w/v) H_3PO_4 activator is able to dissolve the impurities that cover the surface of the charcoal so that it looks shinier, as Manurung reported.[5] A short activation time was chosen to avoid damage to the carbon structure because heating at high temperatures for a long time can damage the structure of the carbon so that its adsorption capacity decreases.[1,6]

(a) (b) (c) (d)

Fig.-2: Raw Materials (a), Bamboo Charcoal (b), Activated Charcoal (c), Inactivated Charcoal (d)

Characterization

The quality of charcoal was characterized based on SNI-1995 for charcoal quality parameters listed in Table-1.

The results of the characterization show that the activation process with 20% (w/v) H_3PO_4 is able to dissolve impurities such as metal oxides, tar, and volatile substances from the pores and surface of the charcoal. Good adsorption of Iodine and methylene blue indicates that the charcoal has micropores and mesopores

with diameters respectively Ø < 2 nm and 2< Ø < 50 nm.[5,10] The maximum wavelength, λ_{max}, of the Rbb solution obtained was 593.3 nm.

Table-1: Characteristics of Charcoals

Parameters	BA Charcoal (%)	Bo Charcoal (%)	SNI (%)
Water Content	1.76	4.0	≤ 15
Volatile Substance	14.0	21.0	≤ 25
Ash Content	8.16	16.7	≤ 10
Carbon Content	76.08	59.0	≥ 65
Iodine ads (mg/g)	1015.30	736.0	≥ 750
Meth.blue Ads.(mg/g)	241.90	165.9	≥ 120

Effect of Contact Time and pH on Adsorption Capacity

The effect of contact time on the adsorption capacities is presented in Fig.- 3. These results show that the optimum contact time is different for B_A and B_0 charcoal, i.e. 60 minutes and 15 minutes respectively, indicated by the highest Rbb dye adsorbed. Nevertheless, subsequent experiments used 60 minutes for both charcoals. Figure-3 also shows that there are differences in the adsorption patterns of B_A and Bo. The adsorption capacity of B_A is greater than that of Bo charcoal. In terms of characteristics, Bo charcoal still contains levels of volatile substances and ash which are still high, and thus B_0 does not meet the SNI quality standards, while B_A charcoal is in accordance with SNI, for all parameters (Table-1). This fact also shows that the activation process has an effect on improving the quality of charcoal. These results are supported by reports from previous researchers.[8,12] The effect of pH on the adsorption capacity of activated charcoal (B_A) and (B_0), is presented in Fig.-4. It shows that pH affects the adsorption capacity of Rbb dye, and the optimum pH is 2 for both B_A and Bo charcoals. In acidic conditions, the surface of charcoals tends to be positively charged, because of the high hydrogen ions (H^+) content in the solution. As a result, it will facilitate the interaction between activated charcoal and Rbb which is negatively charged. The pH ranges of 4-8 tend not to be significantly different. Similar behavior is found in Bo charcoal, although the result is smaller.

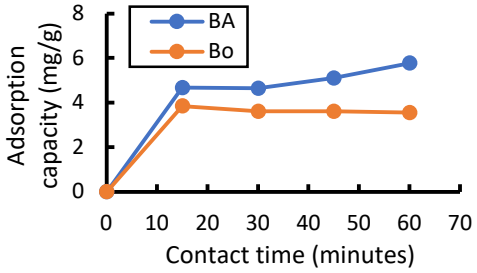


Fig.-3: Adsorption Capacity with Time

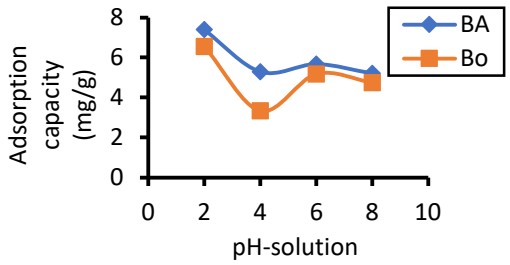


Fig.-4: Adsorption Capacity with pH

This finding is in accordance with the reports of Ahmad and Yusof (2014), which also obtained the highest adsorption capacity of activated carbon of Pinang[1] against Rbb dye at pH 2. Under alkaline conditions, namely pH = 8, it is possible that the surface of the charcoal prefers the hydroxyl ion (OH^-), whose molecular size is smaller than the Rbb anion. The surface of the charcoal is negatively charged so it rejects the Rbb dye which is also negatively charged. This opinion is also in accordance with the report by Ahmad and Hameed, (2010) on the adsorption of *malachite green*.[13,16]

Adsorption Capacity and Efficiency at Optimum Conditions

The adsorption capacity of B_A and Bo charcoal was determined at optimum conditions, with variations in Rbb concentrations of 100, 150, 200, 250, and 300 mg/L. The result is presented in Fig.-5. It shows that the initial concentration affects the adsorption capacity. The higher the initial concentration of Rbb in the solution, the higher the adsorption capacity. The high concentration causes the interaction and diffusion of Rbb ions to the surface of the charcoal to increase. The fact indicates that the saturation point for both types of charcoal has not been reached, because the adsorption trend continues to increase for concentrations up to 300 mg/L. At the concentration limit of 300 mg/L, the absorption capacity (Qexp) of B_A charcoal is

higher at 12.0943 mg/g compared to Bo at 10.7630 mg/g. This phenomenon is in accordance with reports of previous researchers stating that the initial concentration and contact time affect the adsorption capacity.[12] The activation process of charcoal with 20% phosphoric acid was able to increase the adsorption capacity of charcoal by 12.36%. The efficiency of adsorption on various concentrations of samples was determined by equation 2 and the result is shown in Fig.-6. At the sample concentrations of 100 mg/L, the percentage of adsorption efficiency is greater than 95% for both B_A and Bo charcoals. However, at higher concentrations, the adsorption efficiency tends to decrease, because of the limited charcoal surface capacity, and the amount of Rbb remaining in solution increases. At the concentration limit of 300 mg/L, there was a decrease in efficiency to 80 and 71%, respectively, for B_A and Bo charcoals (Fig.-6). At higher concentrations, the amount of adsorption increases but adsorption efficiency tends to decrease.

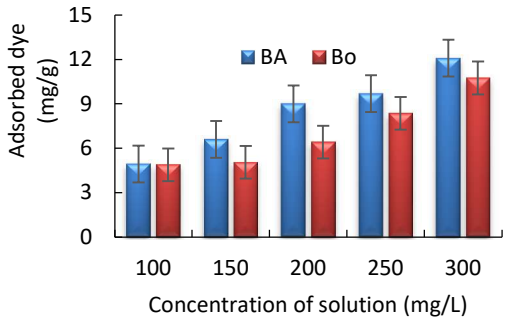


Fig.-5: Effect of Initial Concentration on Adsorption Capacity of B_A and Bo Charcoal

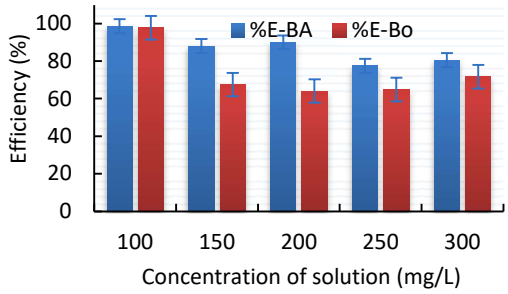


Fig.-6: Effect of Initial Concentration on Adsorption Efficiency of B_A and Bo Charcoal

Isotherm Pattern

The shape of the adsorption isotherm can be examined through two simple models, namely the Langmuir and Freundlich isotherms according to equations 4 and 5.[16,17]

$$Qe = \frac{Qm.Kl.Ce}{1+Kl\ Ce} \rightarrow \frac{Ce}{Qe} = \frac{1}{Qm.Kl} + \frac{Ce}{Qm} \quad (4)$$

$$Qe = K_f .Ce^{1/n} \rightarrow \ln Qe = \ln Kf + \frac{1}{n} \ln Ce \quad (5)$$

Plotting Ce/Qe vs Ce and lnQe vs ln Ce obtain linear lines and the slope equals Qm (maximum amount adsorbed in the saturated state (mg/g) and 1/n, then from the intercept the Langmuir and Freundlich constants K_L, K_F is obtained. The data are presented in Table-2.

Table-2: Isoterm Patterns Data of B_A and Bo Charcoals

Adsorbents Charcoal	Langmuir			
	Qm(mg/g)	K_L(L/mg)	R^2	R_L
B_A	12.2399	0.1389	0.9577	0.01-0.03
Bo	9.5697	0. 0602	0.7062	0.03-0.07
	Feundlich			
	n	K_F	R^2	
B_A	4.8169	4.4749	0.8394	
Bo	7.2464	4.1508	0.4158	

The isotherm pattern of adsorption of Rbb dyes for both charcoal tends to follow the Langmuir with a correlation coefficient (R^2) of 0.9577 for B_A which is close to 1, K_L = 0.1389 L/mg, adsorption capacity (Qmax) theoretically is 12.2399 mg/g. The inactivated charcoal Bo has R^2 of 0.7062, an adsorption capacity (Qmax) of 9.5697 mg/g, and an equilibrium constant K_L of 0.0602 L/mg. This is also supported by the value of the equilibrium parameter (R_L), with a value of $0 < R_L < 1$ (Table-2), which means it is more in line with the Langmuir model. In the Freundlich isotherm pattern, B_A and Bo have correlation coefficients (R^2) of 0.8394 and 0.4158 respectively. When compared between the adsorption capacity of 300 mg/L, experimentally (Qe exp) for B_A and Bo of 12.0934 mg/g and 10.7630 mg/g respectively, while theoretically (Qmax) of 12.2399 mg/g and 9.5697 mg/g, there are differences of 1.21% and 11.09% for BA and Bo

charcoal, under one hour adsorption time. The adsorption process was controlled by the Langmuir model but was not completely homogeneous. The value of n >1 indicates the surface is heterogeneous and tends to be multilayer[1.13]

Adsorption Kinetics

The evaluation of the adsorption kinetics was carried out for charcoal BA and Bo using adsorption data from 25 mL of a 150 mg/L Rbb solution and 0.5 g of adsorbents from 15, 30, 45, 60, and 75 minutes. The data obtained was evaluated in pseudo-first and pseudo-second order according to equations 6 and 7.[16.18]

$$\ln(Qe-Qt) = \ln Qe - k1t \quad (6)$$

$$\frac{t}{Qt} = \frac{1}{Qe2.k2} + \frac{1}{Qe}\,t \quad (7)$$

k1, k2 pseudo-first, and pseudo-second-order rate constant respectively. Making a Figure between ln(Qe-Qt) vs t and t/Qt vs t in the form of a straight line, as in Fig.-7 and data prepared in Table-3.

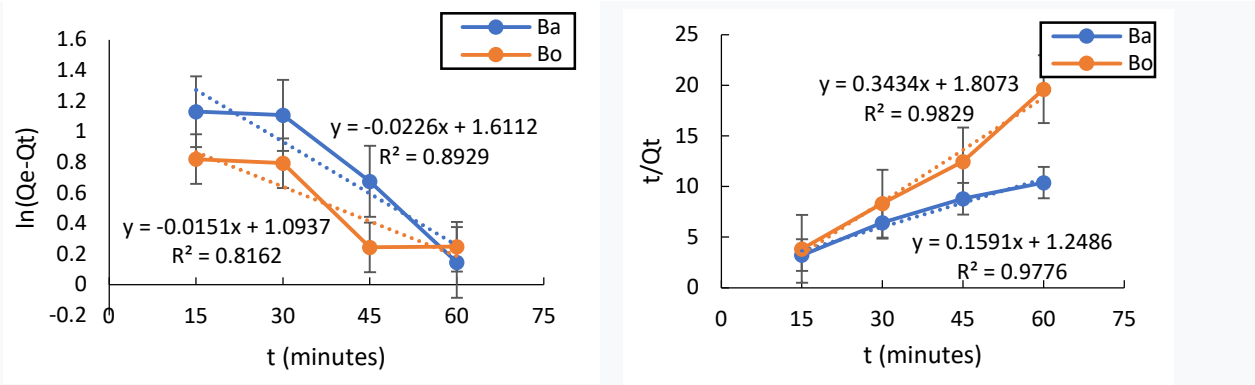


Fig.-7: Evaluation of the Adsorption Kinetics for BA and Bo Charcoal on 150 mg/L. Pseudo-First Order (a) and Pseudo-Second Order (b)

Table-3: The Kinetic Data B_A and Bo Charcoals Adsorption Test for Apparent First-Order and Pseudo-Second-Order for C = 150 mg/L

	Pseudo-first order				Pseudo-second order			
Adsorbent	R^2	k1 (mg/g)	Qe (mg/g)	Qexp	R^2	k2 (mg/g)	Qe (mg/g)	Qexp
B_A	0.8929	0.0226	4.3797	6.6069	0.9776	0.0609	6.5833	6.6069
Bo	0.8162	0.0151	2.9730	5.0675	0.9829	0.0950	2.9121	5.0675

Based on Fig.-7a and 7b as well as Table-3 data it is known that the $_{BA}$ and Bo charcoals follow the apparent pseudo-second order in terms of R^2 value, but based on the suitability value of Qe (theory) and Qe (exp), activated charcoal fitted the pseudo-second-order but Bo can be both. The difference in the value of Qe theory and Qe exp is 0.36%, and 42.53% for B_A and Bo respectively. The adsorption kinetic of the dye onto the solid adsorbent tends to follow the apparent pseudo-second-order, as stated by previous researchers.[1,18,19] The adsorption thermodynamic parameters were determined by determining the adsorption energy as a function of temperature. For this reason, the reaction temperature was varied from 30, 40, and 50 °C at optimum conditions. The adsorption equilibrium constant is considered to be the ratio between adsorbed dye and the remaining dye in the solution at equilibrium. Van Hoff (equation-8) and Gibbs (equation-9) determine the thermodynamic parameter values. The combination of equations 8 and 9 at equilibrium is obtained by equation 10. [15,18]

$$\Delta G = \Delta G^o + R.T \ln K \rightarrow \Delta G^o = - R.T \ln K \quad (8)$$

$$\Delta G^o = \Delta H^o - T.\Delta S^o \quad (9)$$

$$\ln K = \frac{-\Delta Ho}{R} x \frac{1}{T} + \frac{\Delta So}{R} \quad (10)$$

The thermodynamic aspect is obtained by drawing a curve between lnK and 1/T. Using equation 10 the enthalpy (ΔH^o), entropy(ΔSo), and Gibbs energy (ΔG^o) are therefore determined. The results obtained are shown in Fig.-8 and Table-4.

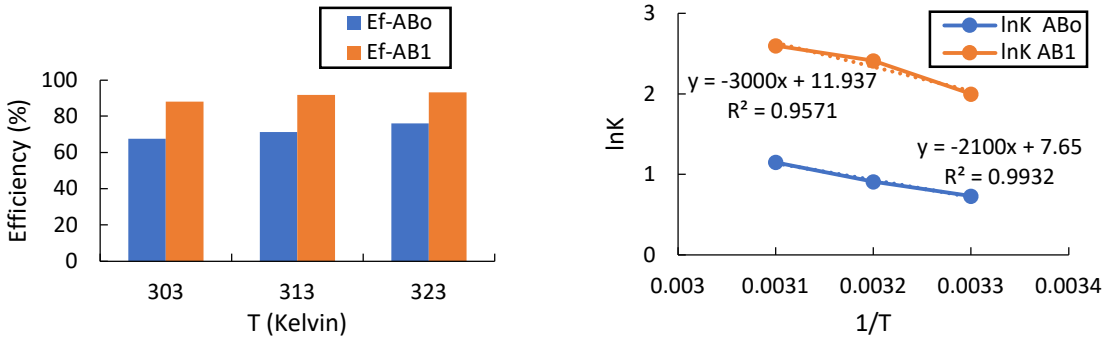


Fig.-8: Correlation Between Temperature and Adsorption Efficiency (a) and lnK to 1/T (b) for B_0 and B_A Charcoals

Table-4: B_0 and B_A Charcoals Adsorption Thermodynamic Data

Adsorbent	T (°K)	ΔH^o (KJ/mol)	ΔS^o (KJ/mol)	ΔG^o (KJ/mol)
Bo	303	17.4594	0.0636	-1.8114
	313			-0.7431
	323			-0.8425
B_A	303	24.9420	0.0993	-5.8696
	313			-3.9236
	323			-3.4315

The adsorption of rhemazol brilliant blue by B_0 and B_A charcoals from a solution is endothermic, but the overall reaction takes place spontaneously ($\Delta G^o < 0$) (Table-4). This is supported by experimental data that efficiency increases up to 50 °C. The low value of the reaction energy of about 20 KJ/mol indicates that the adsorption of rhemazol by B_0, and B_A is controlled by a mixture of chemical and physical adsorption.[19]

Spectroscopy BA and Bo Charcoals

Functional group analysis for B_o and B_A charcoals was determined by FTIR spectrophotometer, using the KBr technique. The result is shown in Fig.-9a and 9b.

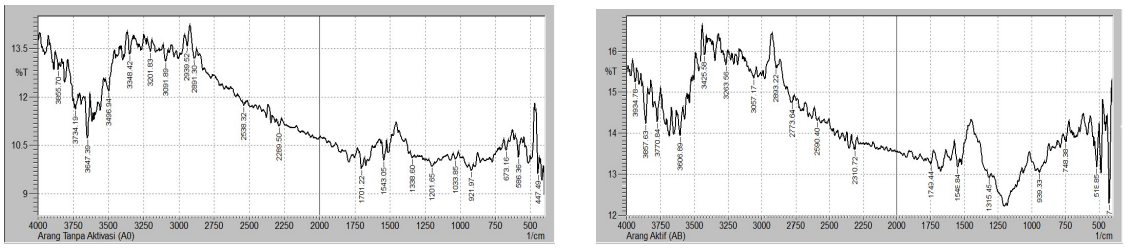
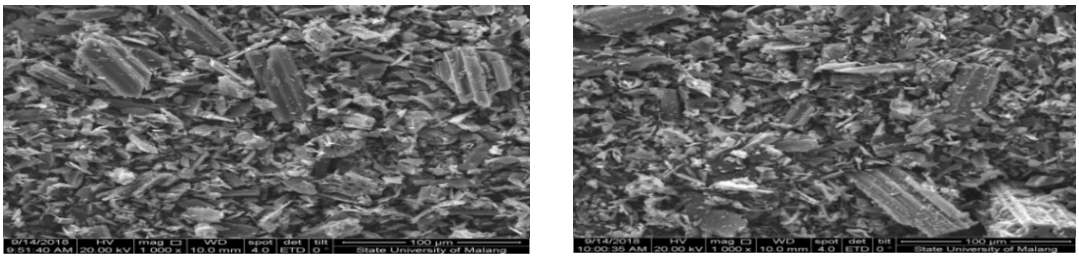


Fig.-9: FTIR Spectra of Bo (a) and B_A (b)

Both spectra show similarities, except that the absorption peak of the activated charcoal (B_A) was sharper. It was suspected that the charcoal contained an OH group (absorption peak in the 3700-3500 cm^{-1} region), a carbonyl group (C=O) peak absorption in the 1650-1700 cm^{-1} region, alkene group (-C=C-) of aromatic ring peak absorption in the 1600 $cm^{-1}$and also the CO ether group which gives an absorption around 1190 cm^{-1}, for the phosphoric acid activated charcoal. The topography of the surface of both charcoals taken with the SEM analysis shows that the surface appearance of B_o and B_A are heterogeneous and looks almost the same (Fig.-10). This suggests that the activation process carried out has not caused a change in the surface of the charcoal clearly.[20]



(a) (b)

Fig.-10: SEM Image of (a)Bo and (b)B_A

CONCLUSION

Based on the results of the study, it can be concluded that the charcoal preparation from bamboo waste was successfully carried out with a yield of 16.2%. Initial carbon content without activation of 59% does not meet SNI-1995. Activation of bamboo charcoal with phosphoric acid, H_3PO_4 20% can improve the charcoal quality to exceed the SNI, with 76.08% carbon content. The optimum pH is 2 and the optimum contact time is 60 minutes for B_A and Bo charcoals. Increasing the initial concentration up to 300 mg/L, the adsorption capacity for B_A and Bo charcoal was 12.2399 mg/g and 9.5697mg/g respectively. The activation process can improve adsorption capacity by 12.36%. The isotherm pattern for both charcoals follows Langmuir models and the adsorption kinetic tends to follow pseudo-second-order.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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