Indian Journal of Chemical Technology
Vol. 29, January 2022, pp. 54-61

Sunan candlenut shells activated carbon: Preparation, characterization, and application adsorption of Rhodamine B

Rahmi[1], Intan Lestari[1], Lenny Marlinda*[,1], Y W Mirzayanti[2] & Muhammad Al Muttaqii*[,3]

[1]Department of Chemistry, Faculty of Sains and Technology, Universitas Jambi, 36361 Indonesia

[2]Department of Chemical Engineering, Adhi Tama Institute of Technology, Surabaya, 60111, Indonesia

[3]Research Center for Chemistry, National Research and Innovation Agency (BRIN-Indonesia), South Tangerang, 15314, Indonesia

E-mail: marlindalenny@unja.ac.id; almuttaqiimuhammad@gmail.com

Received 16 March 2021; Accepted 10 August 2021

The activated carbon has been produced from Sunan candlenut shells wastes produced by chemical activation using potassium hydroxide as an activating agent. The activated carbon has been characterized by Brunauer-Emmett-Teller (BET), X-Ray Diffraction (XRD), Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX), Fourier-Transform Infrared (FT-IR), and the results compared with those of the Sunan candlenut shell used as raw material. The activated carbon produced used as an adsorbent in the Rhodamine B adsorption process. Several examined factors that can affect the adsorption process, such as *p*H, contact time, and adsorbate concentration, have been studied. The yield of activated carbon from Sunan candlenut shells obtained is 74.05%. The activated carbon with the chemical process has characteristic adsorption-desorption isotherms of type IV with mesoporous pore size. The surface area of activated carbon of 355.563 m^2/g and the pore size of 2.1258 nm. The functional groups from FTIR contain C≡C and C=O at a wave number of 2161.55 cm^{-1} and 1980-1994 cm^{-1}, respectively. The results also show the percent adsorption of rhodamine B is 98.75% and the initial maximum adsorbate concentration of 30 ppm at *p*H 4 for 60 minutes.

Keywords: Activated carbon, Adsorption, Sunan candlenut, Rhodamine B

Sunan candlenut is a plant producing vegetable oil as non-food material. It comes from Philippine, spreads into Indonesia, especially West Java. It can produce 300–500 kg of dry seeds per tree per year with an oil concentration of 50–56%. Thus, it has excellent potency. In 1 hectare can be planted 100 trees and produces 50 tons of dry seeds that equal to 15–25 tons oil that higher than palm oil[1]. The production process of vegetable oil from Sunan candlenut seeds produces a side product of seed shells. The seed shell weight is around 35% of the weight of the Sunan candlenut seeds. Based on these data, the number of Sunan candlenut shells from one hectare is around 273-322 kg/ha[2]. Sunan candlenut shells are a by-product that is one of the problems in the environmental field, so there is a need for processing to reduce the waste. A side product of seed shells has potential as organic fertilizer and biogas. In addition, seed shells also have the potential to be used as activated carbon[3].

In another study, candlenut shell waste was used as activated carbon in the purification of used frying oil[4]. This allows the Sunan candlenut shell waste to be used as activated carbon. Activated carbon is also known as activated charcoal or activated coal because the constituent components of this material are charcoal. Activated carbon is widely applied as an adsorbent in the field of. In industrial processes, activated carbon is used for water purification and gold absorption[6] to overcome environmental problems used to remove dyes from water[7] and absorption of methylene blue[8].

Activated carbon is an economical adsorbent with unique textural properties and good adsorption capacity. In traditional applications, microporous activated carbon (pore width <2 nm) is usually employed. However, the development of highly mesoporous activated carbon (2 nm < pore width < 50 nm) has gained considerable attention due to its wide range of applications[9]. Based on research by Hendra et. al., Sunan candlenut shells can be used as activated carbon by chemically activating using H_3PO_4[10]. Carbon activation functions to enlarge the surface area of the carbon material[11]. In addition to using acids, the chemical activation process can also use alkaline activators. One of the basic activated carbon agents is Potassium Hydroxide (KOH).

Potassium hydroxide (KOH) has been widely used as an activator for preparing activated carbon. Despite producing well-developed porosity and a high specific surface area, the activated carbon yield is usually low[9]. The other studies show that the yield of charcoal products from tamarind seed prepared by carbonization at 500 °C is 40.14 wt %. The percent yield of activated carbon prepared from tamarind seed with KOH activation ranges from 54.09 to 82.03 wt% using impregnation ratios of 0.5:1 – 1.5:1 and activation temperatures of 500–700 °C[12]. Activated carbon/gelatin adsorbent provides an efficient and eco-friendly bio-absorbent for the removal and treatment of Rhodamine B in aqueoussolution[13].

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The aim of this study is to study the adsorption process with activated carbon from the pecan shell that will be used as an adsorbent in the Rhodamine B dye. Different factors that can affect the adsorption process such as *p*H, contact time, adsorbate concentration and the adsorption isotherm model were investigated.

Experimental Section

Materials

The Sunan candlenut was obtained from West Java, Indonesia. Rhodamine B, HCl, and KOH were purchased from Merck.

Preparation of activated carbon

Activated carbon prepared from the Sunan candlenut shell by KOH chemical activation[9, 12, 16]. The initial stage of the Sunan candlenut shell has been prepared is carbonized at 600 °C for 2 h then mashed using mortar and pestle. After that, the Sunan candlenut shell carbon is chemically activated. The activator used in the activation process is Potassium Hydroxide (KOH) which is dissolved with distilled water as much as 100 mL. Activated carbon powder was added to the KOH solution with a mass ratio of KOH: Carbon (1: 3). The activation process was carried out by stirring (100 rpm) for 1 hour at 120 °C. After that, the activated carbon obtained is filtered and washed with hot water until the neutral *p*H then dried at 65°C.

Characterization of activated carbon

The activated carbon products were analyzed by Brunauer-Emmett-Teller (BET) to determine surface area and the total pore volume. X-Ray Diffraction (XRD) was used to determine the phase structure of the activated carbon. Scanning Electron Microscope-Energy Dispersive X-Ray (SEM-EDX) was used to determine the morphology and composition of the carbonized and activated products. Fourier Transform infrared spectroscopy (FT-IR) method was applied to analyze surface functional groups[12, 16].

Application of activated carbon as an adsorbent

The adsorbent (0.1 g) was added to solution and then stirred for 1 h. The solution consists of 50 mL of Rhodamine B 20 ppm with variations in *p*H 2, 4, 6, and 8 were prepared. Then, the variation of stirring time of 15, 30, 45, 60, and 75 min The variations concentrations of the adsorbate of 15, 20, 25, and 30 ppm, which have been adjusted at optimum *p*H then stirred during optimum contact time. After finished, the solution was filtered, and the filtrate was analyzed using a UV-vis spectrophotometer at a wavelength of 554.8 nm. Some factors such as *p*H, contact time, and adsorbate concentration, were studied.

Result and Discussion

Activated carbon of the Sunan candlenut shell

The activated carbon produced has a powder texture. The color of activated carbon is darker than carbon before activated. During the activated process stirring and heating so that carbon was impregnation with KOH and the water content in the KOH solution evaporates. This causes KOH and carbon to react so that pores form and increase the carbon surface area. The chemical activation method has advantages over physical activation methods because it is a faster process with a lower activation temperature. KOH has been widely used in the manufacture of low cost activated carbon. Besides, KOH also enhances the specific surface area and the formation of -OH functional groups on the carbon surface[9]. Alkali chemical reagents, such as KOH often used as chemical activating agents due to their suppression of production of tar, consequently improving the yield of activated carbon. In the KOH activation process, a considerable amount of K_2CO_3 formed due to the reaction between KOH and carbon, making the pore structure developed. The use of KOH as an activating agent can provide a high surface area and produce carbonate compounds[16]. A comparison of activated carbon from the carbon activation process can be seen in Table 1. It showed the carbon activation process obtained an average yield of activated carbon of 74.05 %.

Table 2 showed the surface area of carbon with activated physical and chemical processes. Based on Table 2, the surface area of activated carbon with chemical activation has the highest from the others of 355.563 m^2/g. Carbonization and carbon activation treatment aim to enlarge the surface area of carbon material. It is because the carbonization process and the activation using KOH molecules can easily come into contact with the char's outer surface. The produced char is more susceptible to KOH's chemical reaction, which leads to the formation of a higher degree of mesopores and micropores. During carbonization, KOH acts as a dehydrating agent, to eliminate the presence of water in the precursor, which thereafter would cause the formation of tar that could clog the pores[9, 16].

The N_2 gas adsorption-desorption isotherm on carbon before activation and after activation can be seen in Fig. 1. Desorption of nitrogen gas adsorption isotherm in activated carbon of Sunan candlenut nut shell has a hysterical loop in the middle region which indicates the isotherm curve tends to follow type IV absorption volume. According to the IUPAC classification, this type of absorption indicates that the activated carbon obtained is a mesoporous solid. The porosity of activated carbon was formed during the carbonization process.

On activated carbon there are 3 pore sizes, namely micropore (< 2 nm), mesopore (2 nm - 50 nm), and macropore (> 50 nm)[17]. The pore size can be seen from the desorption of nitrogen gas branching using the BJH model (Barrett-Joyner-Halenda). The results of the pore analysis of carbon and activated carbon of the Sunan candlenut shell can be seen in Table 3. It shows the average pore diameter of the activated carbon of the Sunan candlenut shell, which is 2.12582 nm.

The XRD pattern of activated carbon from Sunan candlenutis presented in Fig. 2. It shows the microstructure of activated carbon, which is an

Table 1 — Comparison of carbon mass before and after activation

Carbon mass (g)	Mass of activated carbon (g)	Lost mass (g)	Lost mass (%)	Yield of activated carbon (%)
12	8.95	3.05	25.42	74.58
12	9.03	2.97	24.75	75.25
12	8.68	3.32	27.67	72.33

Table2 — Properties of Sunan candlenut with physical and chemical processes

Sample	Surface area (m^2/g)
Sunan candlenut shell	4.915
Carbon of the shell of the Sunan candlenut is activated in physics	278.627
Carbon of the shell of the Sunan candlenut is chemically activated	355.563

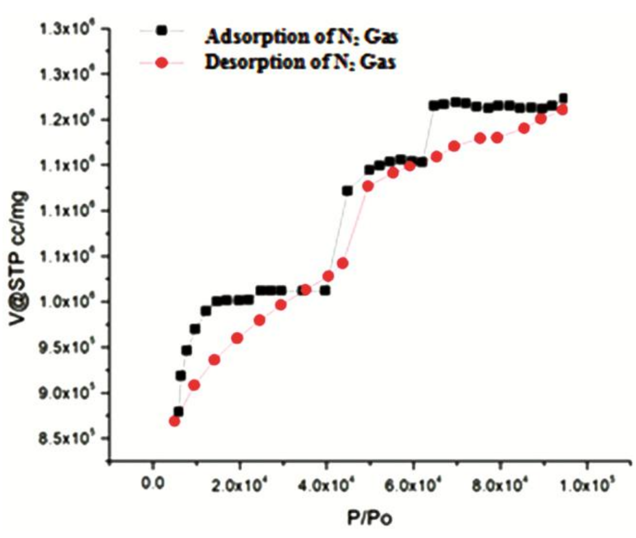

Fig. 1 — Isotherm of adsorption-desorption of activated carbon N_2 gas shells of Sunan candlenut seeds

Table 3 — Results of pore carbon analysis of seed shells and seed shell activated carbon

Pore volume (BJH) (cc/g)	Pore diameter (BJH)(nm)	Average pore diameter (nm)
0.041	3.799	2.50435
0.054	3.748	2.12582

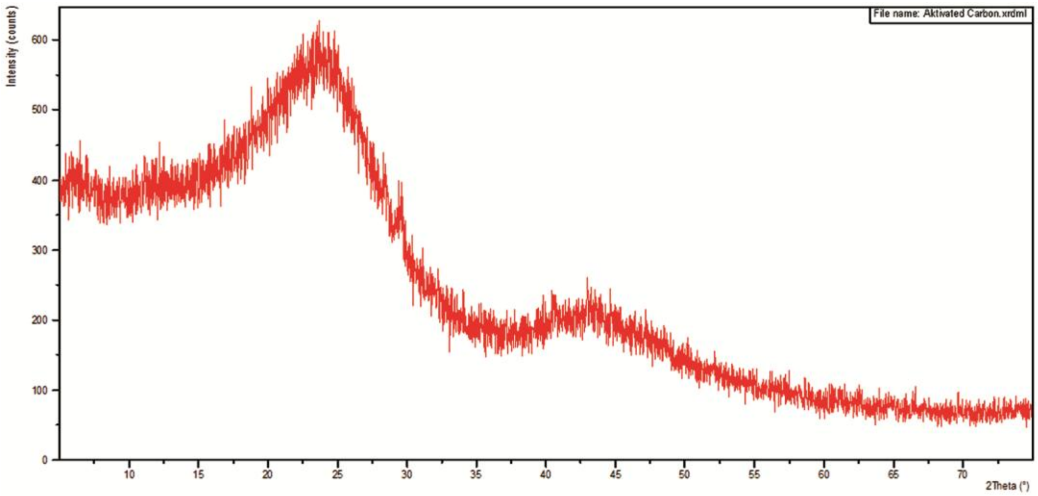

Fig. 2 — XRD spectra of activated carbon from Sunan candlenut

amorphous structure characterized by a peak at $2\theta = 23.69155^o$ with an intensity of 628. This value of 2θ approaches the peak originating from amorphous carbon, which is 24^o. The diffractogram of activated carbon in Sunan candlenut shells is almost the same as the activated carbon from the Cocoa shell. Analyzing the XRD spectrum of the cocoa shell, the band was observed around 18° and 24° and showed an amorphous phase from the adsorbent. Based on research, amorphous carbon from Cocoa shell can absorb amoxicillin[16]. So that the activated carbon from the Sunan candlenut shells which also has an amorphous structure, can absorb Rhodamine B quite well.

In Fig. 3, the difference between the morphology of the seed shell, and the activated carbon of the Sunan candlenut shell can be seen. Figure 3a, proves that the morphology of Sunan candlenut shells has not formed pores on its surface. Meanwhile, in Figure 3b, it can be seen that the surface morphology of activated carbon has formed pores marked by a red circle. From the EDX result found that the major elements contained in activated carbon is a carbon element with the highest percentage of 66.74%. The EDX results on the levels of carbon elements from activated carbon meet the minimum SNI quality standard requirements. The minimum standard quality requirements of carbon elements on activated carbon SNI are 65%.

The results are reinforced by surface area data obtained from BET. A large surface area produces a larger pore size, so it can absorb adsorbate. Meanwhile, the activation process of KOH has the function of oxidizing the inner surface of carbon and forming pores and increasing the adsorption power of Rhodamine B dyes. The EDX results of the shells of the Sunan candlenut seeds and activated carbon of Sunan candlenut shells can be seen in Table 4.

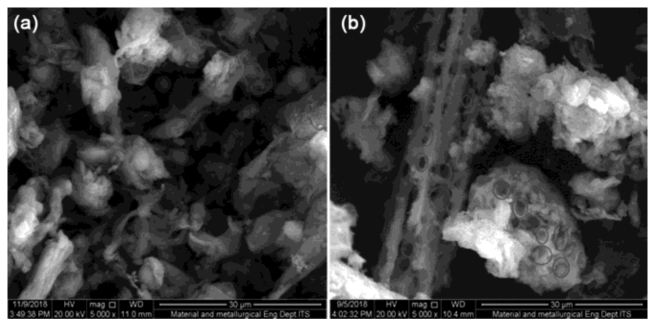

Fig. 3 — Scanning Electron Microscope images (a) shell; (b) shell activated carbon

Table 4 — EDX results of Sunan candlenut shells and activated carbon of Sunan candlenut shells

Sunan candlenut shell		Activated carbon Sunan candlenut shell	
Unsure	% atom	Unsure	% atom
C	48.14	C	76.41
N	02.98	O	18.74
O	43.04	Mg	01.44
Mg	01.31	Al	00.44
Al	00.69	P	00.65
Si	00.50	Cl	00.37
S	00.55	Ca	00.52
K	02.79	K	01.42

Based on Table 4, it is known that percent of carbon atoms increase from seed shell to activated carbon. The shell of seed has a percent of carbon atom 48.14%, while the activated carbon obtained has a major element in the form of carbon with a percent of carbon atoms of 76.41%. The increase in the percent of carbon atoms is due to the process of carbonization and activation in making activated carbon. Chemical activation is a single step process, as both carbonization and activation occur simultaneously at temperatures ranging between 400°C and 700°C. Potassium hydroxide activation can be achieved through either direct chemical activation or char-impregnated chemical activation[9].

The results of FTIR characterization of seed shells, shell carbon, and activated carbon can be seen in Fig. 4. Two functional groups can be identified in the FTIR spectrum of seed shell activated carbon, namely the functional group C≡C at wave number 2161.55 cm^{-1}, C=O bond at wave number 1980-1994 cm^{-1} and one absorption peak in the fingerprint region. According to Niazi *et al.*[18], the Chestnut oak shell's infrared spectrum has a functional group CH, C=O, C=C, C=O, $C-H_3$, $O-CH_3$. This bond indicates carbonyl, lactone, aldehyde and carboxyl groups from Chestnut oak shells before becoming activated carbon[18].

Other results in carbon activation studies using KOH chemical activation, the peak located around 1.625 cm^{-1} were attributed to C=O stretching vibration, which decreased compared with raw lignin owing to the dehydration effect of KOH. Aromatic ring vibrations caused the peak centered around 1.400-1.600 cm^{-1}. The C-O bond at 1.000-1.250 cm^{-1} decreased significantly in activation process. The most significant changes caused by KOH activation was the reduction of –OH, implying the formation of irregular amorphous carbon structure[16]. In the fingerprint area, the number of absorption peaks decreases because of the components in the shell of the seed experience decomposition during the carbonization and activation process. Also, the presence of bond electrons π on functional groups of activated carbon can increase the adsorption process because bond electrons π easily bind to adsorbates (Rhodamine B).

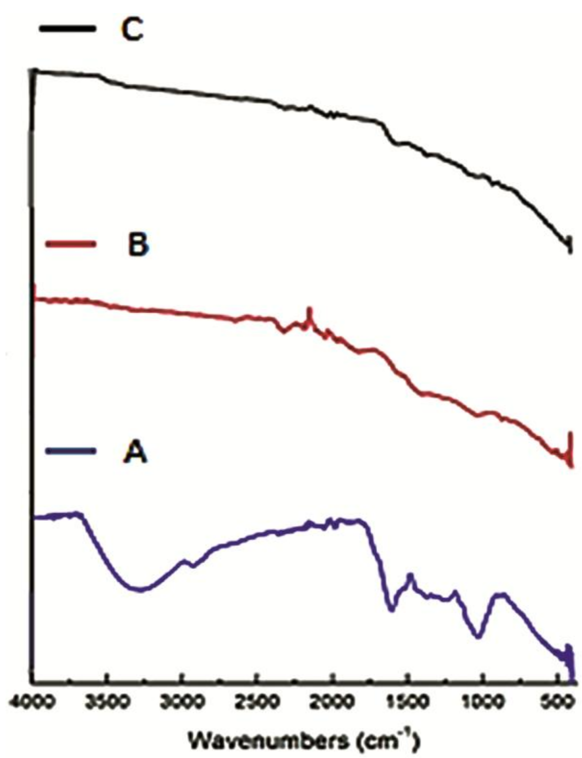

Fig. 4— FTIR Spectra (A) Sunan candle nutshell, (B) Sunan candle nutshell carbon and (C) Sunan candle nutshell activated carbon

Characteristics of rhodamine B adsorption with Sunan Pecan Seed Shell activated carbon as an adsorbent

Effect of *p*H

One important parameter that affects adsorption ability is *p*H. It can affect chemical equilibrium, both on the adsorbate and on the adsorbent, whose optimum conditions are determined based on the highest number of adsorptions.In this study, *p*H was adjusted by adding HCl or KOH dropwise until the target *p*H was reached. The results of determining the optimum pH conditions in Rhodamine B adsorption can be seen in Fig. 5.

Figure 5a illustrates the optimum *p*H condition of activated carbon in adsorbing Rhodamine B dye at *p*H 4 with a percent adsorption value of 98.75%. Meanwhile, at *p*H 8 the adsorption process is not easy because the Rhodamine B dye is an alkaline dyestuff. When the acidic conditions the adsorption process is easy, at the same time in alkaline conditions the adsorption process is difficult.

Effect of contact time

The contact time is a change in the concentration of the substance with time. The results at optimum contact time can be seen in Fig. 5b. The optimum contact time in the Rhodamine B adsorption process using activated carbon of the Sunan candlenut shell was 60 min with a percent adsorption value of 98.75%. The optimum contact time is influenced by the equilibrium between the adsorption and desorption rates and the adsorbent's surface area. Longer contact time could promote the further development of pores, leading to a greater surface area[9].

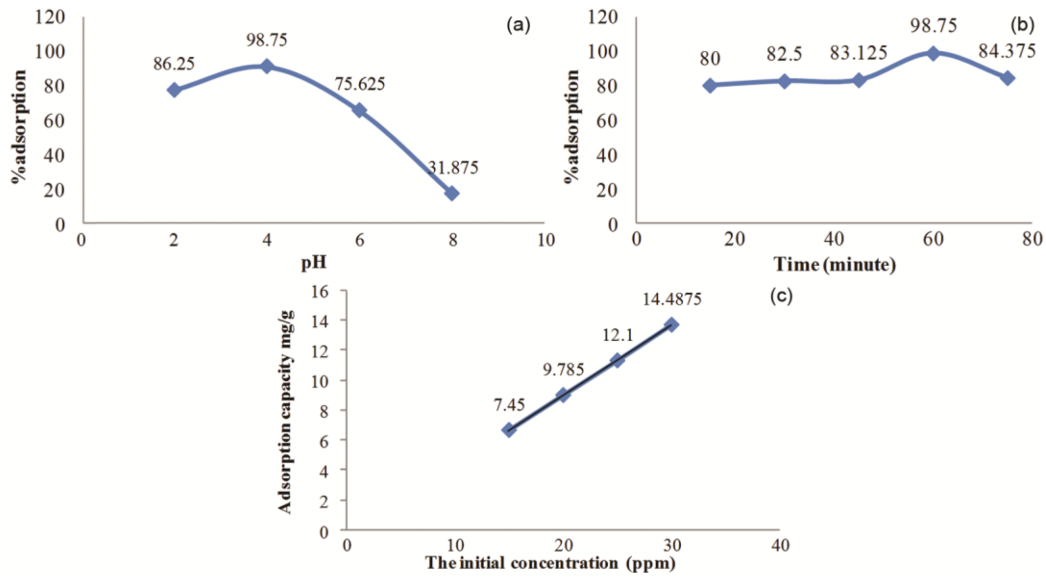

Fig. 5 — (a) Effect of *p*H (t=60 min, C_0 = 20ppm); (b) Effect of contact time (*p*H=4, C_0 = 20ppm) and (c) Effect of the initial concentration of the adsorbate (*p*H=4, t= 60 min)

Effect of the initial concentration of the adsorbate

The results of determining the initial concentration of the adsorbate can be seen in Fig. 5c. The graph shows that the higher the initial concentration, the absorption process increases. At a concentration of 15 ppm, the adsorption capacity was 7.45, while the highest initial adsorbate concentration was 30 ppm with an adsorption capacity of 14.4875. Thus, the greater the initial concentration the greater the adsorption capacity obtained. This is because the higher initial concentration provides the driving force to overcome all resistance from Rhodamine B between the liquid and solid phases, thereby increasing absorption. In addition, at low concentrations, the amount of Rhodamine B in the solution is small, so only a little Rhodamine B is absorbed by the activated carbon. According to Fransina *et. al*, the more bio-absorbate Rhodamine B dyes used, the maximum adsorbed concentration will increase[19]. In this study, the results obtained were the conditions of the maximum adsorbate's initial concentration because the optimum conditions had not been achieved, so the need to add a variable concentration above 30 ppm.

Adsorption isotherm

The isotherm condition is used to indicate the relationship between the concentration of the adsorbate and the absorption concentration can occur at the specified temperature[20]. Figure 6a shows the Freundlich adsorption isotherm pattern curve with a straight line equation y = 0.26 x + 1.137, which has a gradient of 1/n = 0.26. The intersects the log qe axis at 1.137 with R^2 = 0.967. The maximum adsorption capacity value obtained by the Freundlich isotherm (kF) model is 0.56/mg, and the adsorption intensity value (n) is 3.85.

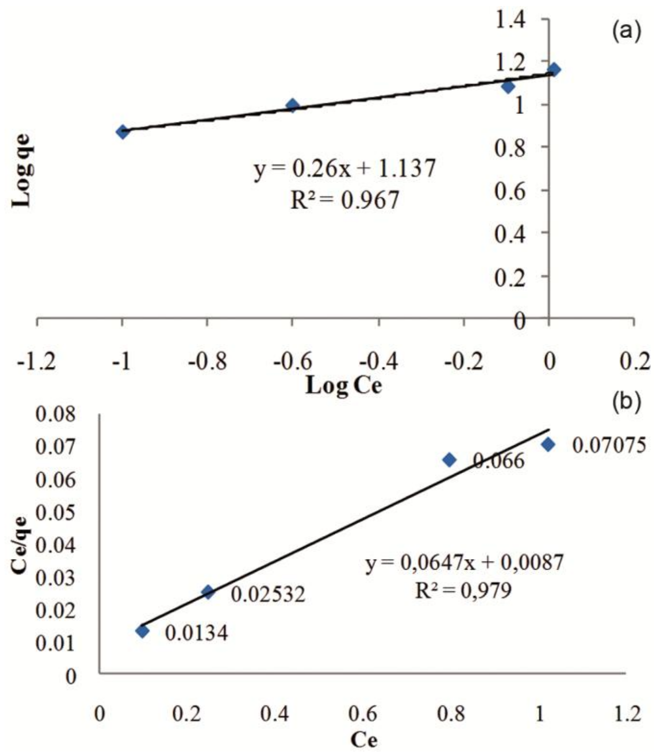

Fig. 6 — (a) Isotherm of Freundlich and (b) Isotherm of Langmuir

Langmuir adsorption isotherm defines that the maximum adsorption capacity occurs due to a

Table 5 — Value of determination of isotherm Langmuir and Freundlich at 30°C

Freundlich			Langmuir			
K_F	n	R^2	K_L	q_{max}	r	R^2
0.56	3.85	0.967	0.1345	15.45	0.27	0.979

monolayer adsorbate on the surface of the adsorbent, and all its surface sites are homogeneous because each active site can only adsorb one adsorbate molecule[21]. Figure 6b shows the Langmuir adsorption isotherm pattern curve with a straight-line equation y = 0.064x + 0.008 which has a gradient of $1/q_{max}kL$ = 0.064. At this line intersects the 1/qe axis at 0.008, resulting in a regression value (R^2) of 0.979 with a maximum adsorption capacity price Langmuir isotherm of (q_{max}) = 15.45 mg/g and the equilibrium constant (kL) of Langmuir isotherm is 0.1345/mg. The dimension value of the adsorption quantity (r) is obtained at 0.27.

The regression value (R^2) of the two-adsorption isotherm models has a value of R^2 that is almost the same, which is close to one. Adsorption isotherm serves to find out how much the absorption mechanism of the adsorbent in the data adsorption process, so that the equation of the adsorption isotherm is obtained. Adsorption isotherms are characterized by certain constant values that describe the adsorbent's surface characteristics, adsorbent affinity, and adsorption capacity. The graph obtained from the calculation of the adsorption isotherm. It shows the relationship between the number of adsorbents with the unit weight of the adsorbent and the number of adsorbates at the time of equilibrium. The isotherm equation used in this study is the Freundlich equation and the Langmuir equation. Freundlich assumes that adsorption occurs in a monolayer. The surface of the adsorbent is heterogeneous and no chemical events occur. In contrast, Langmuir assumes that chemical adsorption dominates, the arrangement of the adsorbate molecules on the surface of the adsorbent forms a single layer, all sites and surfaces are homogeneous. The data obtained were processed, so that the value of determining Isotherm Langmuir and Freundlich was obtained, as in Table 5.

Conclusion

The results of making activated carbon of Sunan candlenut shell have a yield of 74.05%. The activated carbon obtained has the characteristics of N2 type IV adsorption-desorption isotherms with mesoporous characteristics. A surface area of 355.563 m^2/g with a pore size of 2.12582 nm, amorphous structure, has a porous surface morphology and contains functional groups C≡C in numbers wave 2161.55 cm^{-1} and C=O 1980-1994 cm^{-1}. The characteristics of Rhodamine B adsorption using activated carbon from the shells of Sunan candlenut seeds obtained optimum *p*H conditions at *p*H 4, the optimum contact time of 60 min and the maximum initial concentration of adsorbate is 30 ppm. The model of rhodamine B adsorption isotherm dominated by Langmuir isotherm with R^2 value is 0.979.

Acknowledgment

This work was financially supported by the Subject and specially construction special fund of DIPA PNBP LPPM Faculty of Science and Technology-University of Jambi in Lecturer Research Scheme. The main contributor of this paper is Lenny Marlinda and Muhammad Al Muttaqii.

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