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Original Research Article

Activated Carbon Produced from Rice Husk by NaOH and KOH Activation and its Adsorption in Methylene Blue

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Abstract: Sodium hydroxide (NaOH) and potassium hydroxide (KOH) rice husk activated carbon were prepared using a method of two-step comprising of carbonisation and activation processes. These adsorbent samples were used to remove methylene blue (MB) solution. Some chemical and physical characteristics of adsorbents were determined using proximate analysis, Brunauer–Emmett–Teller surface area (BET) and scanning electron microscope (SEM) before carrying out the MB adsorption experiments. The analyses of adsorption kinetics were performed at several conditions of pH, MB concentration and temperature to investigate the efficiency of adsorption of the samples on the MB solution. The analyses of adsorption kinetics related to the pseudo-second-order model. The capacity of adsorption of the KOH rice husk activated carbon (RHACK) on MB achieved a maximum of adsorption at 317.24 mg/g as compared to the NaOH rice husk activated carbon (RHACNa) at 210.90 mg/g. The model of intraparticle diffusion showed that the process of adsorption of RHACK and RHACNa against MB included the diffusion of MB molecules and external mass transfer to the surface of adsorbents.

Keywords: Rice husk, activated carbon, BET, adsorption, methylene blue

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1. Introduction

The sector of industry, human consumption and agriculture all need fresh water (Sharma & Bhattacharya 2017). Around 20% of the people in the world has been facing the depletion of fresh water (Sulyman *et al.* 2017). Fresh water resources in the world are polluted because of technological and industrial activities. The use of dyes has polluted water and is reducing the content of dissolved oxygen in the water. One of the aromatic heterocyclic compound called methylene blue (MB) has been used in the industry of textile which has negative impacts on water quality (Guo *et al*. 2014). Some technique has been introduced by researchers to treat the pollutants in the polluted water namely ozonation, flocculation, irradiation, coagulation, electrochemical destruction, membrane filtration, ion exchange, adsorption, electrokinetic treatment and photocatalytic oxidation (Danish *et al.* 2014). The process of adsorption using activated carbon (AC) is a technique that is mostly used due to it is feasible, easy to use, and inexpensive for the removal of colour of liquid (Sreńscek-Nazzal *et al*. 2013). Some researchers have investigated how to produce cheaper adsorbents due to the high cost of AC (Rafatullah *et al*. 2010; Crini 2006; Tan *et al*. 2008; El-Maghraby & El Deeb 2018). AC can be made from agriculture residue like paddy straw (Gao *et al.* 2011), coconut shell (Iqbaldin *et al.* 2013), corn cob (Song *et al*. 2013), peanut shell (Wu *et al*. 2013), palm kernel shell (Rugayah *et al*. 2014), and walnut shell (Yu *et al*. 2014).

AC can be produced either through chemical or physical (steam, air, or CO_2) activation. Chemical activation is preferred due to its high specific, high surface area and good pore enrichment of the AC produced (Patil & Kulkarni 2012). AC from KOH activation is better in microporous than NaOH (Okman *et al.* 2014). The usage of KOH also creates more surface area and OH groups formed on the carbon (Viswanathan *et al.* 2009).

There are some studies using rice husk (RH) AC in the adsorption. Rostamian *et al.* (2015) used RH activated with KOH. The adsorption of Na by RH AC had the highest adsorption capacity due to increased surface area and pore volume. Rahman *et al.* (2005) found that the adsorption efficiency of AC of RH has the highest malachite green adsorption due to the factor of high surface area. Lin *et al*. (2013) in their study said that the adsorption tests indicated that ACs derived from NaOH treated on RH had good adsorption capacity for MB in an aqueous solution because of surface area of AC has increased.

In this study, the NaOH and KOH were activated with RH to produce AC. Additionally, various physical and chemical properties of AC were also studied. The experiments in adsorption were also performed on kinetic adsorption processes because there are fewer studies, especially using Malaysia RH AC. The study also focuses on the impact of the removal of MB on the parameters of temperature, pH, initial dye concentration, activation agents and time. The interpretation of the behaviour of the adsorption of MB on AC samples can be carried out by using kinetic models from the adsorption experiments data.

2. Materials and Methods

2.1 Preparation of RH

RH was taken from Sekinchan, Selangor, Malaysia. It was washed with water to remove dirt and oven dried at 105°C overnight.

2.2 Preparation of AC from RH

The RH was turned into rice husk charcoal (RHC) using the pyrolyzing system at 400°C for 4 h. Then, the RHC was immersed with the solution of KOH at 1:4 (RHC: KOH) for 24 h. It was filtered and oven dried overnight at 105°C. The process of activation was activated in a cylinder furnace with a nitrogen gas flow (100 mL per min) for 2 h at 850°C. Sample was washed with hydrochloric acid (HCl) solution (1 M) and deionised water until a pH of 6 to 7 in the rinse was obtained. The sample was called RHACK. The above procedure was repeated on the samples treated with NaOH and labelled as RHACNa.

2.3 Characterizations of the AC samples

The proximate analysis was analysed to obtain ash content, volatile matter and fixed carbon data. The RHC, RHACK, and RHACNa samples' surface area was studied using a surface analyser (Micromeritics ASAP, 2010). The samples were degassed before being analysed for 10 h under vacuum at 110°C. Surface properties of the samples were analysed using scanning electron microscopy (SEM) of the FEI model Quanta 400.

2.4 Adsorption Kinetics Study

The study of adsorption was performed at different MB concentrations, pH and temperatures. About 0.1 g AC was put into a flask with 100 mL of MB. The pH was adjusted at 3-9 using 0.1 M HCl and 0.1 M NaOH. Some experiments at temperatures (25-65°C) MB and concentrations (50-300 mg/L) were also done. The AC in the MB solution was mixed at 250 rpm. MB solutions (~0.1 mL) were measured its concentration using a UV-Vis Spectrophotometer (UV-vis, Jenway 7315) at different times. The MB adsorbed value, q_t (mg/g), was determined following equation 1:

$$q_t = (C_o - C_t)\, V/m \qquad (1)$$

where C_0 and C_t are the initial concentration of MB (mg/L) and the concentration of MB (mg/L) at different times, respectively. V is the solution volume (mL) and m is the weight of the adsorbent (g).

The model of kinetic to study the adsorption of MB was either under pseudo-first-order or pseudo-second-order models. Equation 2 of the pseudo-first-order model is as follows (Lagergren 1898):

$$\ln (q_e - q_t) = \ln q_e - k_1 t, \qquad (2)$$

where q_e and q_t are the amounts of adsorbed MB on the adsorbent (mg/g) at equilibrium and at time t, respectively. k_1 is the pseudo-first-order rate (min^{-1}).

Equation 3 of the pseudo-second-order is as follows (Ho & Mckay 1999):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \qquad (3)$$

where k_2 is the rate constant of the pseudo-second-order. It suggests that the rate-limiting step involves chemisorption of adsorbate on the adsorbent.

The model of intraparticle diffusion in equation 4 (Weber, 1963) was used to investigate the diffusion mechanism of the adsorbate-adsorbent:

$$q_{\mathrm{ref}} = k_{\mathrm{i}} t_{\mathrm{ref}}^{1/2} + C \tag{4}$$

where k_i is the intraparticle diffusion constant which reveals the presence of the intra-particle diffusion process calculated from the slope of a plot of q_t vs $t^{0.5}$ and C is the intercept (mg/g) (Weber, 1963). t_{ref} is the longest time taken in the adsorption process and q_{ref} is the adsorption capacity at t_{ref}. Intraparticle diffusion described the mechanism of the adsorbate diffused on the particles of adsorbent which is assumingly proposed by three steps (Wu *et al.* 2009):

i) The transport of adsorbate from solution to the outer surface layer of the adsorbent by molecular diffusion as external (film) diffusion.

ii) The transport of adsorbate from the particle surface adsorbent into interior sites as internal diffusion.

iii) The adsorption of the solute particles from active sites into the interior surface of the pores.

3. Results

3.1 Proximate Analysis

Table 1 shows the results of the proximate analysis. It shows that the carbonisation process has increased the fixed carbon value of AC and reduced the volatile material content. The study of Mopoung *et al.* (2015) found that rice charcoal ash content was higher after the carbonisation process because ash is not a volatile material in which volatile material will decrease after the carbonisation process. It was also reported that rice charcoal fixed carbon increased fivefold after the carbonisation process. It was further stated that the value of ash content and volatile material for AC was reduced possibly due to the effect of degradation by activation agents during the activation process. The high content of RHC ash compared to AC results in its low surface area value is as shown in Table 2. Yeganeh *et al.* (2006) stated that the high ash content was due to inorganic material content results in low sample surface area, mechanical strength and adsorption capacity.

Table 1. Proximate analysis results.

Type of sample	Ash of content (%)	Volatile matter (%)	Fixed Carbon (%)
RH	15.34 ± 0.033	63.32 ± 0.72	21.34 ± 0.69
RHC	19.11 ± 1.29	14.06 ± 0.53	66.83 ± 1.82
RHACK	14.84 ± 2.91	10.23 ± 1.11	74.93 ± 1.80
RHACNa	13.90 ± 4.72	7.47 ± 0.0013	78.63 ± 4.72

3.2 Surface Area Analysis

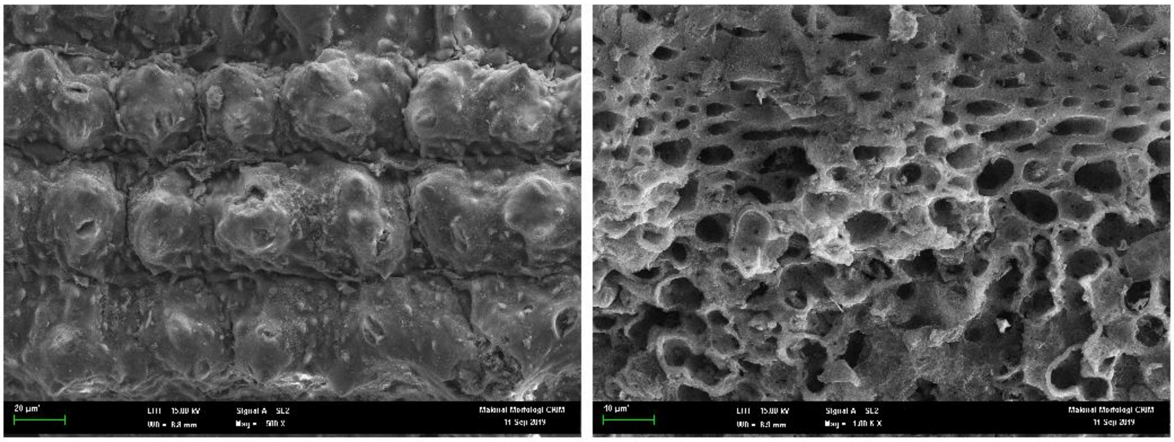
Surface area value of RHACK is 1131.73 m^2/g compared to RHACNa 429.82 m^2/g. It was found that KOH activated better than NaOH. It causes the creation of pores and volume of pores and in turn, produces higher surface area porous carbon (Lillo-Ródenas *et al.* 2001). These results are equivalent to the study performed by Guo *et al.* (2003). Increased porosity was found to be related to alkaline cation activation agents in the following order: Li <Na <K <Rb <Cs, in this case, KOH has more alkaline cations than NaOH, therefore KOH reacts more with carbon to produce pores (Guo *et al.* 2003). The average RHACK and RHACNa pore sizes were in the range of 2.67–2.69 nm as displayed in Table 2. It shows that the majority of pores are between the sizes of 2–50 nm, which is a meso-sized hole. According to IUPAC 1972, pore sizes have three types namely micro pores (below 2 nm), meso pores (2-50 nm) and macro pores (over 50 nm) (Lu *et al.* 2015).

Table 2. Results of surface characteristics of RHC, RHACK, and RHACNa.

	RHC	RHACK	RHACNa
S_{BET} (m^2/g)	0.23	1131.73	429.82
V_{micro} (cm^3/g)	0.0016	0.45	0.14
V_{total} (cm^3/g)	0.0026	0.76	0.29
Average pore size (nm)	4.42	2.67	2.69

3.3 Scanning Electron Micrograph

The SEM results of RHC and RHACK are shown in Figure 1 (a) and (b), respectively. There are no pores seen on the RHC surfaces, but many pores are found on the surface of RHACK samples which are due to the chemical activation process. It was the high BET surface area of RHACK which has resulted in the high number of pores as tabulated in Table 1. These studies are similar to results from previous researchers (Zhang *et al*. 2009; Oh & Park 2002); the KOH activation produced high pores and values of BET.



(a) (b)

Figure 1. SEM (a) RHC and (b) RHACK samples.

3.4 Initial pH

Figure 2 displays the adsorption of RHACK and RHACNa on the MB at different levels of pH at 25°C. The capacity adsorption rose from 92.0 mg/g to 97.4 mg/g for RHACK and 90.0 mg/g to 93.32 mg/g for RHACNa when the pH of the solution changed from 3 to 7. It could be due to the surface charge of the adsorbent that has been increased whenever the pH rose (Sajab *et al.* 2013). The acidic stage gave an increased adsorption process because the binding site of the adsorbent would closely be associated with the hydrogen ions which acted as bridging ligands between the adsorbent surface and dye molecules (Bazrafshan *et al*. 2014). The other factor that affects the decreases of the adsorption of MO dye onto PEI modified SB, especially at pH 9, which is the optimum pH because of the great amount of OH- competing with anionic MO molecules (Sajab *et al*. 2013).

The adsorption results showed a decrease at pH 9 for both samples. Related study has been discussed by other researchers regarding the adsorption of MB with other lignocellulose adsorbents (Pua *et al.* 2013; Batzias & Sidiras 2007). The researchers have done the SEM analysis under the adsorption of MB on the lignocellulosic adsorbents and found that the samples treated with activating agents produced pores on the surfaces.

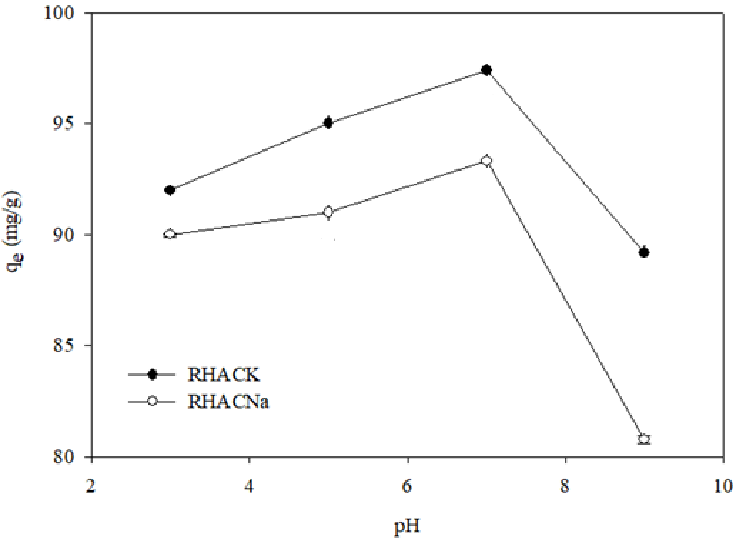


Figure 2. The effect of pH on the MB adsorption of RHACK and RHACNa (C_0: 100 mg/L; T: 25°C).

3.5 MB Concentration

The initial MB concentration was studied with various MB concentrations, starting from 50 until 300 mg/L and the results are demonstrated in Figure 3. The increasing concentration of MB has increased the adsorption of the samples and required a longer time to reach the equilibrium of adsorption. It is believed that there is strong competition at high initial MB concentrations between MB molecules in aqueous conditions causing these molecules to concentrate on the surface of the adsorbent in the presence of a pore system (Lin *et al.* 2013). The KOH AC sample adsorbs more MB and achieves the adsorption balance faster than the NaOH AC. The higher surface area in RHACK samples may have caused more MB to be adsorbed onto the adsorbent pore.

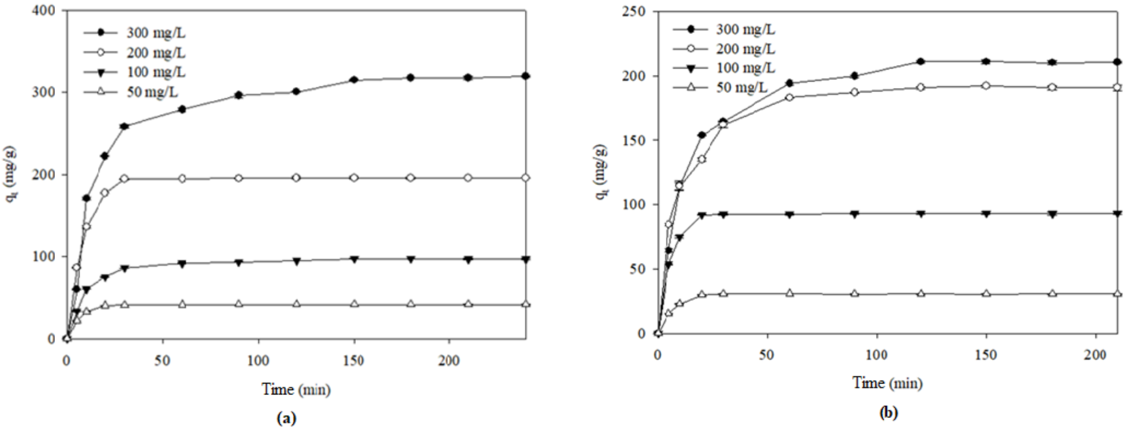


Figure 3. Results of MB adsorption kinetics of (a) RHACK and (b) RHACNa samples (pH: 7; T: 25°C).

3.6 Temperature

A study of the effect of temperature on MB adsorption was conducted from 25°C, 45°C, and 65°C at pH 7 and in MB solution at a concentration of 100 mg/L. Figure 4 shows the relationship between temperature and the amount of adsorbed MB on RHACK and RHACNa samples. It can be observed that the increase of MB adsorbed on the AC surface as the temperature was raised from 25°C to 65°C. The adsorption of MB in this experiment was influenced by temperature. The adsorption was increased along with the increased temperature may be due to the increased infiltration rate of the intraparticle ion of the adsorbed material into the pores where diffusion was in the endothermic process (Bharathi & Ramesh 2013). The results also found that RHACK samples were adsorbed more as the temperature increased compared to RHACNa. It could be because of the higher surface area of the AC sample with KOH.

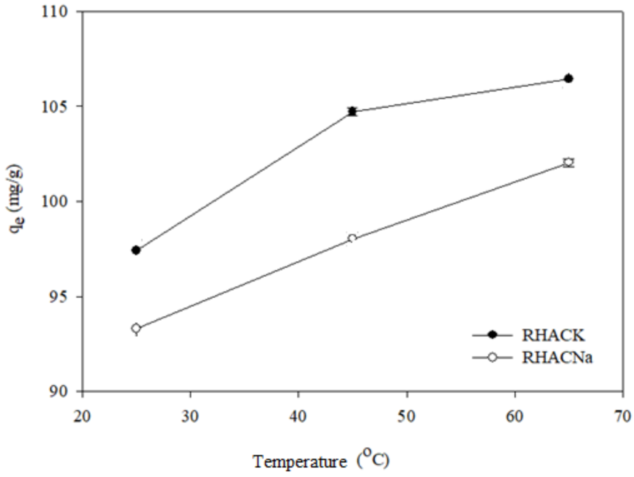


Figure 4. Results of MB adsorption kinetics of RHACK and RHACNa (C_0: 100 mg/L; pH: 7).

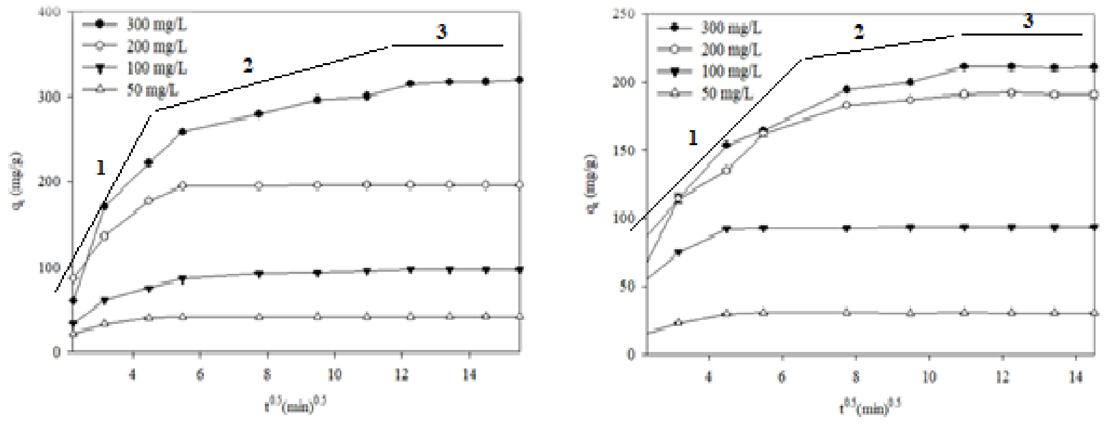
3.7. Adsorption Kinetics

The kinetic data calculated from pseudo-first-order and pseudo-second-order models using the experimental data was tabulated in Table 3. The results showed that the pseudo-second-order model fits the experimental data better compared to the pseudo-first-order. The orderly validity of the adsorption process depends on the values of r^2 and q_e of the experiment (Raoul *et al.* 2014). Table 3 shows the adsorption kinetic data complying with the pseudo-second-order model because its higher r^2 value and q_e count value for the pseudo-second-order model for all AC samples are almost the same as the experimental q_e compared to the pseudo-first-order model. This indicates that the pseudo-second-order model is best used to explain the reaction mechanism for all relevant AC samples. The feasibility of the application of the pseudo-second-order model indicates that the stage rate limit is chemical adsorption covering the forces resulting from the exchange of electrons between adsorbent and adsorbed material (Raoul *et al.* 2014). In this kind of adsorption, the chemical reaction seems significant in the rate-controlling step and the pseudo-second-order chemical reaction kinetics provide the best correlation of the experimental data and the adsorption's mechanism is chemically rate controlling and because of this, it is called chemisorption. In this mechanism, the kinetics of adsorption should correspond to a reversible second order reaction at low adsorbate/adsorbent ratios (first order at very low ratios), and two competitive reversible second order reactions at higher adsorbate/adsorbent ratios (Raoul *et al.* 2014).

Table 3. Results of kinetic data calculated from pseudo-first-order and pseudo-second-order models.

			Pseudo-first-order			**Pseudo-second-order**		
	C_o (mg/L)	**q_e exp (mg/g)**	**q_e cal (mg/g)**	**k_1 (1/min)**	**r^2**	**q_e cal (mg/g)**	**k_2 (g/mg min)**	**r^2**
RHACK	50	41.77	46.05	0.137	0.978	42.19	0.0113	0.999
	100	97.4	35.11	0.0309	0.878	100	0.00151	0.999
	200	195.82	35.78	0.0408	0.653	222.22	0.000439	0.988
	300	317.24	195.74	0.0261	0.929	333.33	0.000229	0.998
RHACNa	50	30.87	46.77	0.194	0.997	31.06	0.0154	0.999
	100	93.32	13.19	0.0499	0.665	94.34	0.00635	0.999
	200	190.72	112.88	0.0397	0.979	200	0.000729	0.999
	300	210.90	125.15	0.0291	0.947	222.22	0.000460	0.999

The results of intraparticle diffusion data analyses are presented in Table 4 and Figure 5. Two predictions can be made from the plot. Intraparticle diffusion is the rate-controlling step if the straight-line plot passes through the origin as shown in Figure 5. However, if not, the process of adsorption may involve other mechanisms along with intraparticle diffusion (Rubín *et al.* 2010). Figure 5 shows that the plots for (a) RHACK and (b) RHACNa do not go through the origin, but a three linear adsorption process was found. The first linear process was likely the external surface or boundary layer adsorption, the second was the adsorption where the intraparticle diffusion was rate controlled. The third was the equilibrium step where the intraparticle diffusion started to slow down because of the low adsorbate concentrations in the solution (Boyd *et al.* 1947; Kumar & Gaur 2011). The influence of mass transfer caused the shape of the q_t versus $t^{0.5}$ plot to be curved at a small time limit or in a very short time (Annadurai *et al.* 2002). This also proved that adsorption of the MB onto the adsorbent is a multi-step process, including adsorption on the external surface and diffusion into the interior. The values of *k*i and *C* (Table 4) are much higher with increasing initial MB concentration, resulting in more intraparticle diffusion and boundary layer effect or surface active sites, respectively (Chandrasekhar & Pramada, 2006). In addition, the *k*i and *C* values of RHACK are higher than for RHACNa, which suggests that the RHACK has more intraparticle diffusion and surface-active sites. The value of C is the thickness of the boundary layer. The higher the bypass value, the higher the boundary layer effect and intraparticle diffusion (Chandrasekhar & Pramada, 2006).



(a) (b)

Figure 5. Adsorption kinetics of MB on (a) RHACK and (b) RHACNa samples: intraparticle diffusion.

Table 4. Kinetic data calculated from intraparticle diffusion model for the adsorption of MB.

	Intraparticle Diffusion			
	C_0 (mg/L)	k_i (mg/g h^{½})	C (mg/g)	r^2
RHACK	50	7.98	5.31	0.959
	100	15.44	4.78	0.940
	200	33	21.91	0.955
	300	57.87	44.33	0.914
RHACNa	50	6.42	1.61	0.986
	100	16.91	17.97	0.974
	200	17.64	54.17	0.952
	300	22.03	36.39	0.889

4. **Conclusion**

RH AC activated with KOH and NaOH were made using the two-step method. RHACK and RHACNa produced less ash content and volatile matter but more carbon content than the RH and RHC in the proximate analysis results. The results of surface area analysis of RHACK and RHACNa were higher than RHC due to more pores as proved in SEM tests. The adsorption kinetics of RHACK and RHACNa followed pseudo-second-order which indicates that chemical adsorption existed during the adsorbent process between adsorbent and adsorbed material. The intraparticle diffusion of the adsorption of RHACK and RHACNa on the MB through the external mass transfer and diffusion of MB to the surface of adsorbents.

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