Indonesian Journal of Science & Technology 4 (2) (2019) 229-240

Indonesian Journal of Science & Technology

Journal homepage: http://ejournal.upi.edu/index.php/ijost/

Removal of Methylene Blue by Adsorption onto Activated Carbon From Coconut Shell (Cocous Nucifera L.)

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ABSTRACT

This research has been conducted on process of production activated carbon from coconut shells, which are activated both physics and chemistry to improve the adsorption of methylene blue. The process of physical activation was done by burning the coconut shell using a furnace at a temperature of 700°C. The chemical activation was done using H_3PO_4 activator. The result of activated carbon physical activation (CAP) has a greater absorbency than activated carbon chemical activation (CAC) with each of the absorption of methylene blue at 99.42 and 98.64%. Analysis of surface morphology on the adsorbent was performed using a Scanning Electron Microscope (SEM). SEM results indicated that (CAP) has a surface morphology that is relatively similar to commercial activated carbon (CACm). Adsorption test was conducted on the determination of the optimum pH, adsorption rate, and isotherm adsorption of methylene blue. The results of the optimum pH on CAC, CAP, and CACm respectively obtained at pH 8 and the optimum contact time is obtained respectively at 40, 60, and 80 minutes. Adsorption kinetics data of methylene blue on CACm, CAP, and CAC tend to follow the pseudo second order kinetics with a correlation coefficient (R^2) is 0.937; 0.950; and 0.999, respectively. Adsorption isotherm of methylene blue on CACm, CAP, and CAC tend to follow the model of Freundlich isotherms.

ARTICLE INFO

Article History:

Submitted/Received 11 Nov 2018
First revised 18 Jan 2019
Accepted 17 Mar 2019
First available online 20 Mar 2017
Publication date 01 Sep 2019

Keyword:
Adsorption,
Isotherms,
Activation,
Activated carbon,
Coconut shell.

1. INTRODUCTION

Today environmental pollution is a serious problem in the world. Development in industrial sector and synthetic compounds use for plant cultivation and industrial purposes have a negative impact for environment (Anshar *et al.*, 2016). The pollutant substance can be severe metals, such as Pb, Cd, Cu, Ni, and Zn (Buhani *et al.*, 2012; Buhani *et al.*, 2013; Suharso and Buhani, 2011). These substances are usually present in the effluent water many industries, such as textiles, leather, paper, printing, and cosmetics. Dyes are colour organic compound which can colorize other substances (Nandiyanto *et al.*, 2016). These substances are usually present in the effluent water many industries, such as textiles, leather, paper, printing, and cosmetics. Over 10,000 dyes with a total yearly production over 7 x 105 MT are commercially available. It is estimated that approximately 15% of the dye stuffs are lost in industrial effluents during manufacturing and processing operations (Auto and Hameed, 2011; Tunc *et al.*, 2009; Gupta *et al.*, 2012). Highly colored wastes are not only esthetically unpleasant but also hinder light penetration and may disturb the river ecosystem. Moreover, dyes itself are toxic to some organism. Methylene blue (MB) is a cationic dye having various applications in chemistry, biology, medical science and dyeing industries. Its long term exposure can cause vomiting, nausea, anemia and hypertension (Foo and Hammeed, 2011; Hameed, 2009).

Several methods have been developed to reduce the dyes from waste water, such as adsorption, biosorption, coagulation and flocculation, advanced oxidation, ozonation, membrane filtration, and liquid–liquid extraction have been widely used for the treatment of dye-bearing wastewater. The advantages and disadvantages of every removal technique have been extensively reviewed (Hameed, 2009; Salleh *et al.*, 2011). According to Wang and Li (2013), the conventional method above is very complicated and expensive because some of these require additional chemicals so that they can produce toxic products. Among the method above, adsorption is a very effective separation technique in terms of initial cost, simplicity of design, ease of operation and insensitive to toxic substances (Patel and Suresh, 2008; Gupta and Bhattacharyya, 2006; Salleh *et al.*, 2011). Activated carbon (powdered or granular) is the most efficient adsorbent used for dye removal (Anshar *et al.*, 2016; Nandiyanto *et al.*, 2017; Nandiyanto *et al.*, 2018). However, commercially available activated carbons are still considered as expensive materials for many countries due to the use of non-renewable and relatively expensive starting material such as coal, which is unjustified in pollution control applications (Attia *et al.*, 2008). Therefore, in recent years, this has prompted a growing research interest in the production of activated carbons from renewable and cheaper precursors which are mainly industrial and agricultural by-products, such as apricot shell (Karagozuglu *et al.*, 2007), male flower of coconut tree (Senthilkumaar *et al.*, 2006), jute fiber (Senthilkumaar *et al.*, 2005), rubber wood sawdust (Kalavathy *et al.*, 2005), corncob (Tseng *et al.*, 2006), bamboo (Hameed *et al.*, 2007) and oil palm fibre (Tan *et al.*, 2007).

Coconut shell is one of the most common potential wastes in Indonesia. Coconut shell produced around 86 tons per year and only used for a household fuel. Though this coconut shell can be processed into products that have high economic value, namely as activated carbon. It has a lot of micropore, low ash content, high water solubility, high absorption, not harmful to the environment and high reactivity (Dhidan, 2012). Therefore, the main objective of this study was to evaluate the possibility of using coconut shell to develop a new low cost activated carbon and study its application to remove methylene blue dye from aqueous solution. Adsorption isotherm were determined, compared to Langmuir and Freundlich equation, and maximum capacities were calculated for different performance activated carbon (*e.g.*, Commercial, Physically, and Chemically). The kinetics study of removal MB allowed the calculation of parameters according to two different kinetic models.

2. MATERIALS AND METHODS

2.1. Materials.

Commercial activated carbon from Yinchuan Meikelin Activated Carbon Co. Ltd. Coconut shell waste from local home industries in Bandar Lampung, Lampung. Methylene blue from Solarbia. H_3PO_4 10%, HCl 0,1 M, NaOH 0,1 M were purchased from Alba Chemical. Purified water was produced in the laboratory by using a GS-590 water distillation system from Tokyo. All solutions used in this work were prepared using the purified water.

2.2. Suspendisse Preparation of activated carbon from Coconut Shell dapibus lorem pellen-tesque magna.

Coconut shell waste from the locally available from home industries in Bandar Lampung, Lampung, Indonesia. The waste was washed several times with water to remove adhered impurities from its surface. The waste was dried at 105 ºC for 24 hours. After that are burning on the kiln and the charcoal was milled and sieved to 60 µm particle size. Then the coconut shell powder is activated physically and chemically. For physical activation, accurately weighed powdered samples (50 mg) were put into the furnace at 700 ºC for 1 hour. While for chemical activation, 20 mg of the powder which are physically activated are soaked in 30 mL of H_3PO_4 10% solution for 24 hours to remove any acid, followed by washing with distilled water until pH 6 and dried in oven at 100 ºC for 1 h. The resulting activated carbon was preserved and used as an adsorbent and characterized by using a scanning electron microscope (SEM).

2.3. Experimental Protocol

The bath adsorption experiments were conducted in a set of 25 mL of erlenmeyer flask containing adsorbent and 20 mL of MB solution with various initial concentrations, respectively. The flasks were agitated in water-bath shaker at 120 rpm until the equilibrium is reached. After decantation and filtration, the equilibrium concentrations of dyes in the solution were measured at 664 nm using UV-Visible spectrophotometer. The pH of solution was adjusted with HCl 0.10 M and NaOH 0.10 M solutions. The amount of dyes adsorbed and percentage removal of MB were calculated using the following equation:

$$Q = (Co - Ce)V/W \tag{1}$$

where *Q* is the amount of dye in mg/g of adsorbent. *Co* and *Ce* represent the initial and the equilibrium concentration of MB (mg/L), respectively, respectively. *W* is the mass of adsorbent (g), and *V* is the volume of *MB* solution (L) (Buhani *et al*., 2015). The results are then plot as shown in **Figure 1**.

2.4. Adsorption isotherm

2.4.1. Langmuir isotherm

The Langmuir sorption isotherm is applied to equilibrium sorption assuming monolayer sorption onto a surface with a finite number of identical sites. Adsorption process occurs due to chemical interaction between adsorbate and active side of existing adsorbent (Buhani *et al*., 2017). The Langmuir equation is written as (Langmuir, 1916):

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{k_L q_m C_e} \tag{2}$$

where *qe* is the amount of adsorbate on the adsorbent at equilibrium (mg/g), *Ce* (mg/L) is the equilibrium concentration of the adsorbate, *qm* (mg/g) is the maximum adsorption capacity, and *KL* (L/mg) is the Langmuir adsorption equilibrium constant. The values of *KL* and *qm* were calculated from the slope and intercept of a plot of 1/*qe* vs. 1/*Ce*, respectively.

2.4.2. Freundlich isotherm

The Freundlich equation for heterogeneous surface energy systems is given by Eq. (4) (Foo and Hammed, 2012; Nemr *et al.*, 2009).

$$ln\ q_e = ln\ k_F + \frac{1}{n} ln\ C_e \quad (3)$$

where Kf and n are Freundlich constants, determined from the plot of ln qe versus ln Ce. The parameters Kf and 1/n are related to sorption capacity and the sorption intensity of the system. The magnitude of the term (1/n) gives an indication of the favorability of the sorbent/adsorbate systems. In the Freundlich isotherm model, Kf indicates the adsorption capacity and n indicates the heterogeneity factor for which a favorable adsorption is larger than one (n>1). The values of Kf and n can be obtained from plotting log qe vs. log Ce (Buhani *et al.*, 2017).

3. RESULTS AND DISCUSSION

3.1. Characterization of Activated Carbon

Figure 2 shows SEM micrograph of (a) coconut shell before activating, (b) CACm, (c) CAP, and (d) CAC. Through this micrograph irregular and porous surface activated could be observed . Based on this fact, it can be concluded that CAP, and CAC present and adequate morphology for MB adsorption, but the best between both figure is CAK because it has a relatively similar surface morphology with CACm. In line with the adsorption results which showed that the capacity adsorption of MB with CAC was greater than the CAP, which were 99. 42 and 98.64 %, respectively.

3.2. Removal of MB

3.2.1. Effect of adsorbent dose

The effect of adsorbent dose on the adsorption process can be done by preparing adsorbent with different amount added to the adsorbates solution with a fixed concentration and stirred together for 1 hour (Salleh *et al.*, 2011). The adsorbent dose varied out from 0. 05 to 0.5 g. It is evidence from **Figure 1** that the amount of MB absorbed per unit weight (Q) of activated carbon decreases with increasing dose of adsorbent used . This is because at higher dose there is super-critical adsorption very quickly to the surface of the adsorbent which results in a lower solute concentration in solution than that when the adsorbent dose is lower. Thus, with increases in dosage of adsorbent, the amount of dye adsorbed per unit adsorbent mass decreases, so the Q value is decrease (Han *et al.*, 2007; Ragadhita *et al.*, 2019).

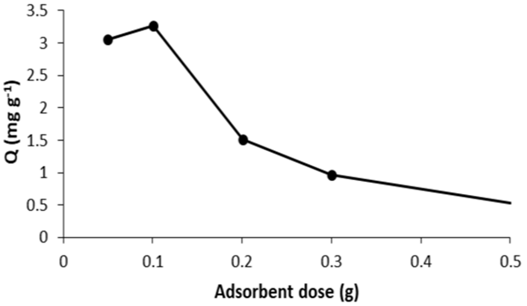

Figure 1. Effect of adsorbent dose on MB removal.

3.2.2. Effect of initial pH solution

The pH of a dye solution is very important for influencing factor in the adsorption of MB onto activated carbon. **Figure 3** shows that the amount of MB adsorbed is in following the increase in pH solution. The capacities of MB has a significant increament from 3.78 to 6.89 mg/g with the increase of pH value in the range 2-12. And, the MB removal can reach up to 6.10; 6.74; and 6.89 mg/g for CACm, CAP, and CACm, respectively, when the initial pH solution is 8. Thus, in acidic medium, the positively charged surface of sorbent tends to oppose the adsorption of the cationic adsorbate.

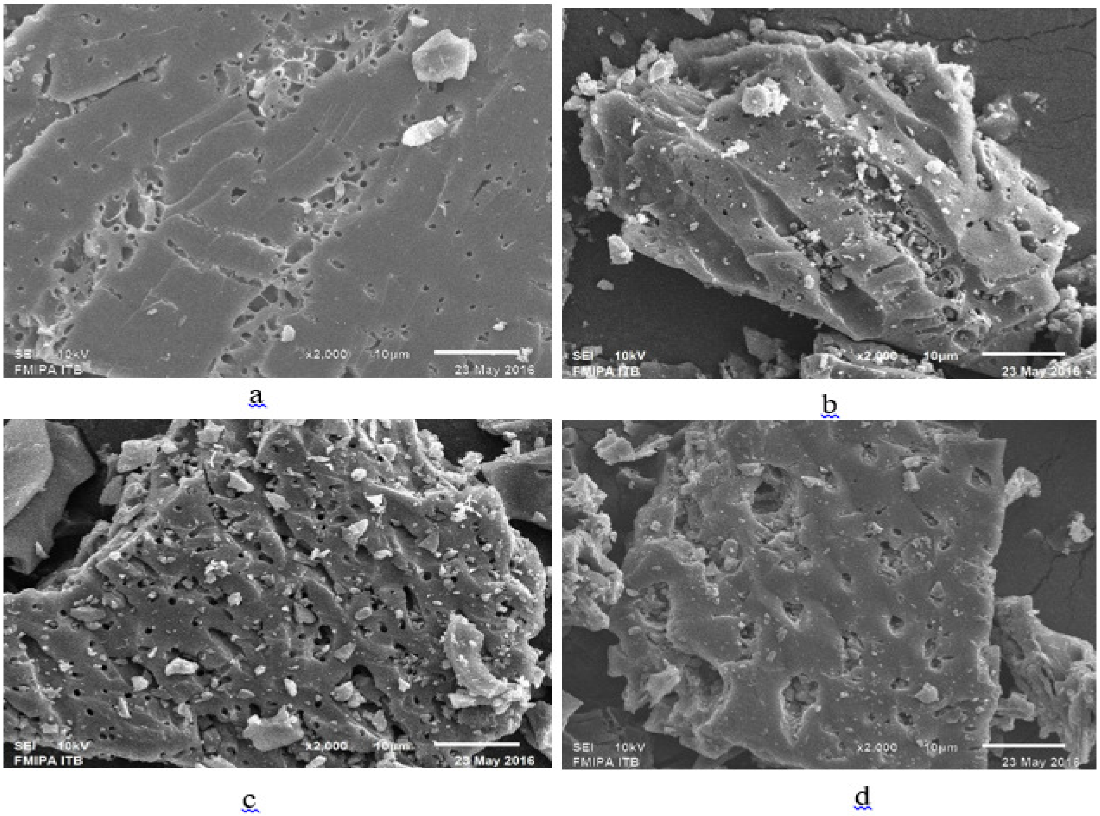

Figure 2. SEM micrograph images of (a) coconut shell before activating, (b) CACm, (c) CAP, and (d) CAC at 2000 x magnification.

Table 1. Comparison of pseudo-first and second order kinetic model parameters for different activated carbon.

Adsorbent	**Pseudo first order**		**Pseudo second order**	
	***k*₁ (menit⁻¹)**	***R*²**	***k*₂ (g mmol⁻¹ menit⁻¹)**	***R*²**
CACm	0.006	0.833	0.07	0.937
CAP	0.003	0.465	0.188	0.95
CAC	0.001	0.352	0.626	0.999

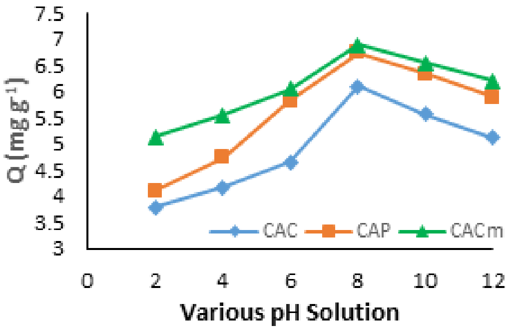

Figure 3. Effect of initial solution pH on MB.

When pH of the dye solution increases, the surface acquires a negatively charged. Thus, the amount of MB removal decreases. With increase in pH of dye solution, increases in the electrostatic attraction between positively charged dye and negatively charged adsorbent were obtained (Abd El- Latif and Ibrahim, 2009).

3.2.3. Effect of contact time

The effect of contact time on the removal of MB is shown **Figure 4**. This figure shows that the adsorption takes a relatively fast time at the initial stage on the outer surface of the adsorbent followed by a slower internal diffusion process which is in the stage of the rate determinant (Gialamouidis *et al*., 2010). And, after a certain time, removal of MB is decrease in number of vacant site of adsorbent and dye concentration. The decreased adsorption rate, particularly, toward the end of experiments, indicates the possible monolayer formation of MB on the adsorbent surface (Abd Abd El- Latif and Ibrahim, 2009; Nemr *et al*. 200).

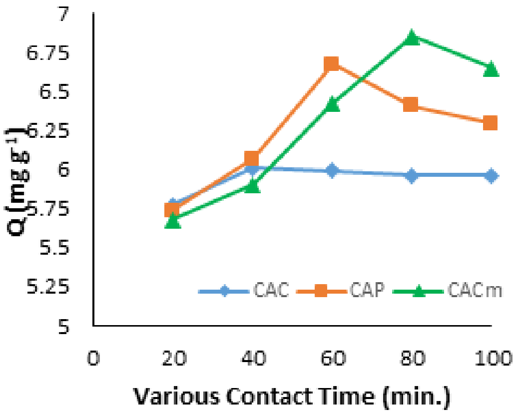

Figure 4. Effect of contact time on MB removal process.

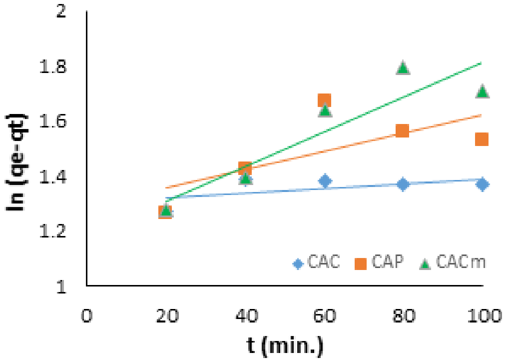

Figure 5. Pseudo-first order kinetic removal model parameters for adsorption of MB onto CACm, CAP, and CAC.

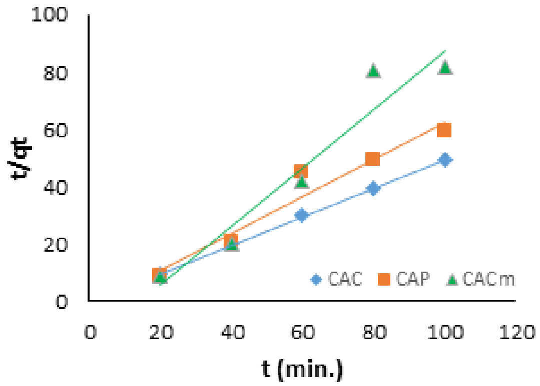

Figure 6. Pseudo-second order kinetic model parameters for adsorption of MB onto CACm, CAP, and CAC.

3.3. Adsorption Kinetics

The rate constant of adsorption is determined from the pseudo first-order equation given by Langergren and Svenska (1898):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (4)$$

where q_e and q_t are the amounts of MB adsorbed (mg g^{-1}) at equilibrium and at time t (s), respectively. k_1 is the rate constant adsorption (s^{-1}). Values of k_1 were calculated from the plot of *ln*(*qe*−*qt*) versus *t* (**Figure 5**) for different concentration of MB. Although the correlation coefficient values at high concentration are higher than 0.83; 0.46; and 0.35 for CACm; CAP; and CAC, respectively, the experimental *qe* values do not agree with the calculated ones. This can be obtained from the linear plots (see **Table 1**). This shows that the adsorption of MB onto activated carbon is not a first-order kinetic.

On the other hand, a pseudo second-order equation based on equilibrium adsorption (Ho and McKay, 1998) is expressed as:

$$\frac{t}{q_t} = \frac{1}{k_2 {q_e}^2} + \frac{t}{q_e} \quad (5)$$

where k_2 (g/mg s) is the rates constant of second-order adsorption. If second-order kinetics is applicable, the plot of *t*/*q* versus *t* should show a linear relationship. There is no need to know any parameter beforehand and *qe* and can be determined from the slope and intercept of the plot. Also, this procedure is more likely to predict the behavior over the whole range of adsorption. The linear plots of *t*/*q* versus *t* (**Figure 6**) show a good agreement between experimental and calculated *qe* values (**Table 1**).

The correlation coefficients for the second-order kinetic model are 0.92; 0.95; and 0.99 for CACm, CAP, and CAC, respectively, indicating the applicability of this kinetic equation and the second-order nature of the adsorption process of MB on activated carbon. These results indicated that the value in CAC is greater than CACm and CAP for adsorption of MB. The greater rate of adsorption with CAC is due to the chemical properties of the activator. Thus, it can accelerate the rate of MB adsorption with a lower viscosity value.

3.4. Adsorption Isotherms

The adsorption isotherm indicates how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process reaches an equilibrium state. The analysis of the isotherm data by fitting them to different isotherm models is an important step to find the suitable model that can be used for design purposes (El-Guindi, 1991). Adsorption isotherm is basically important to describe how solutes interact with adsorbents, and is critical in optimizing the use of adsorbents. Adsorption isotherm study is carried out on two well-known isotherms, Langmuir and Freundlich. The applicability of the isotherm equation is compared by judging the correlation coefficients (R^2).

Figure 7 shows the equilibrium adsorption isotherm of MB on the prepared activated carbon. The adsorption capacities of MB increase following with increasing the concentration of MB. Therefore, it can be concluded that the higher concentration of MB, the greater amount of MB which is adsorbed. From the data contained in **Figure 7**, it use and plotted the Langmuir adsorption isotherm models (**Eq. 1**) (**Figure 8**) and Freundlich (**Eq. 2**) (**Figure 9**) to produce the adsorption parameters shown in **Table 2**.

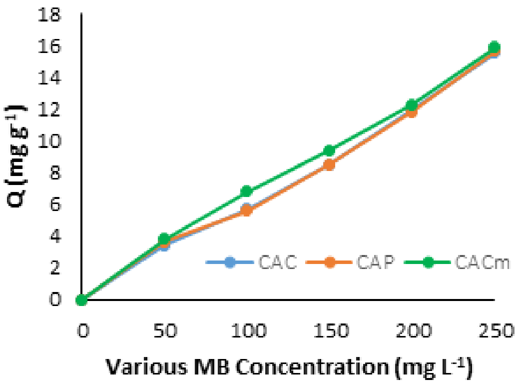

Figure 7. Equilibrium adsorption isotherm of MB onto activated carbon.

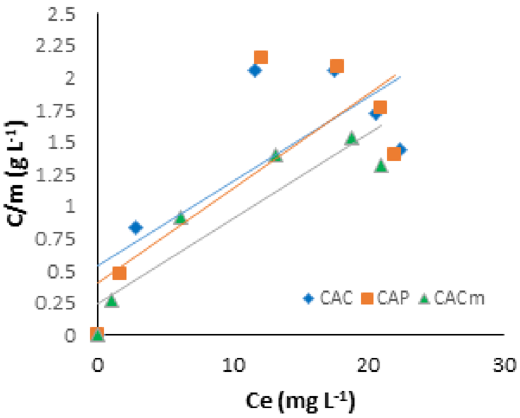

Figure 8. Langmuir adsorption isotherm of MB onto activated carbon.

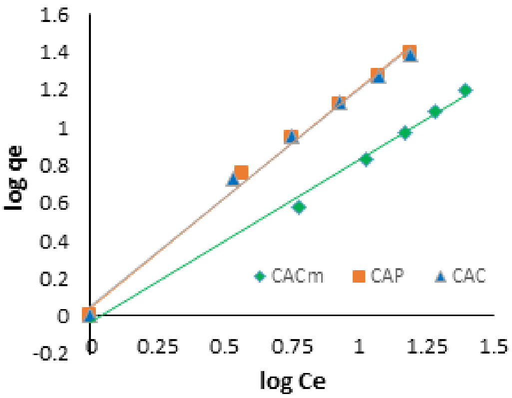

Figure 9. Freundlich adsorption isotherm of MB onto activated carbon.

Table 2 summarizes all the constants and correlation coefficients R^2 of the two isotherm models used. The Freundlich model yielded the best fit with R^2 values equal or higher than 0.99, as compared to Langmuir model. Freundlich adsorption isotherm explains that the adsorption process is heterogeneous. The meaning is that not all the adsorbent surface are able to experience adsorption, so the layer resulting from this adsorption model is multilayer because of the physical interaction between adsorbent and adsorbate (Buhani *et al.*, 2015). Thus, the adsorption capacity of MB cannot be determined but has a large amount of MB which is adorbed when the equilibrium is high as the concentration of MB is used, which is 15.775; 15.553; and 15.478 mg g^{-1} when the concentration of MB 250 mg L^{-1}.

4. CONCLUSION

The present investigation showed that activated carbon prepared from coconut shell was a promising adsorbent for the removal of methylene blue dye from aqueous solutions over a wide range of concentrations. The results of surface morphology analysis with SEM showed that the surface morphology in CAP was relatively same to CACm which is has a larger pore size than CAC. The capacity adsorption of MB followed second order kinetics with the value (k_2) are 1.0497; 0.2898; dan 1.2571 g/mmol.min for each CACm, CAP, and CAC, respectively. Equilibrium data of adsorption was in good agreement with Freundlich's isotherm, with the adsorption capacity around 15.775; 15.553; and 15.478 mg/g for each CACm, CAP, and CAC, respectively.

Table 2. Langmuir and Freundlich isotherm parameters and correlation coefficient for adsorption of MB on activated carbon.

Adsorbent	**Parameters adsorption**					
	Langmuir			**Freundlich**		
	b **(mg g^{-1})**	***K_L*** **(L mol^{-1})**	***R^2***	**q_e (mg g^{-1})**	***K_f*** **(L mol^{-1}**	***R^2***
CACm	15.243	0.262	0.849	15.775	0.934	0.993
CAP	13.679	0.18	0.404	15.553	0.925	0.992
CAC	15.223	0.123	0.533	15.478	0.921	0.992

5. ACKNOWLEDGEMENTS

The authors would like to thank to the Directorate of Research and Community Services, Directorate General of Higher Education (DIKTI), Ministry of Research, Technology and Higher Education of the Republic of Indonesia for financial support of this research.

6. AUTHORS' NOTE

The author(s) declare(s) that there is no conflict of interest regarding the publication of this article. Authors confirmed that the data and the paper are free of plagiarism.

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