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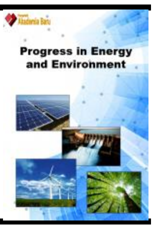

Adsorption of Methylene Blue and Congo Red Dye from Water onto Cassava Leaf Powder

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ABSTRACT

The suitability of Cassava leaf (CL) as low-cost adsorbent for dyes adsorption was evaluated in this study. Methylene blue (MB) and Congo red (CR) dye was chosen as adsorbate in this work as these two types of dyes are widely used in the industries. The impact of various parameters such as adsorbent dosage, pH, temperature and contact time for dye adsorption on CL were investigated. 99.9% removal of MB dye was achieved at pH of 11.0, temperature of 45°C, 0.5 g of CL under 60 minutes of contact time. Meanwhile, 99.7% removal of CR dye was obtained under similar contact time but at pH of 2.0, room temperature and smaller dosage of CL at 0.2 g. Based on the isotherms study, MB dye adsorption was best fitted in both Langmuir and Freundlich isotherms while Temkin isotherm was best fitted for CR dye adsorption on CL.

Keywords:
Cassava leaf, Methylene blue, Congo red.

1. Introduction

The fast pace of industrialization have caused pressure on the environment. It resulted in various environmental pollutions, especially water pollution through the effluent of the industrial wastes into water bodies. Industries such as textiles and dye manufacturing, food colouring, and cosmetics consume huge amounts of water for their industrial processing and subsequently resulted in generation of highly dye-contaminated effluents [1].

Due to wide applications of textile industry, the effluent discharged from textile industry are the main concerns. This is because the textile wet processing utilized more than 2000 different chemicals which can cause harmful effects to human and animal health [2]. From the lists of chemicals that presence in textile wastewater, dyes are the major pollutants among the other chemicals [3].

Based on to the volume of dye wastewater generated from various industries, azo dyes are the main group of dyes, with organic dyes generated worldwide accounts for 60–70% [4]. The example of azo dye is Congo red (CR) dye. As a benzidine-based anionic diazo dye, CR dye are commonly generated from various industries which includes dyeing, textiles, printing, paper, plastic, rubber industries, and etc [5]. With the presence of the aromatic amines in the azo dyes, it can increase the risk of skin irritation, permanent blindness, bladder cancer, contact dermatitis, chemosis, acute tubular necrosis

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supervene, lacrimation, vertigo, exophthalmos, rhabdomyolysis, edema of the face, neck, pharynx, tongue, and larynx along with respiratory suffering [6].

Apart from azo dyes, Methylene blue (MB) as a cationic dye is also commonly used by several industries, especially textile industry for a variety of purposes. MB, as basic dye, is a powerful colouring agent especially for acrylic fibres [7]. However, MB dye is toxic and have low biodegradability which could cause negative effects on health such as diarrhoea, vomiting, gastritis, headache, and chest pain [8-9]. Exposure to MB dye could potentially result in haematological toxicity, as it was found that there was development of Heinz bodies and haemolytic anaemia in treated rats and mice [10]. This is because MB dye would trigger the oxidation reaction of ferrous iron in haemoglobin into ferric state, which methaemoglobin will be produced when treated at high concentration.

The disposal of untreated coloured wastewaters into water bodies will cause negative impacts on natural aquatic life [11]. Specifically, in the presence of dyes, biochemical oxygen demand (BOD) of water would increase and this would cause reduction in the process of reoxygenation, influencing the growth of the photoautotrophic organisms [12]. In addition, it could disturb the biological cycles of the aquatic life by decreasing the light penetration through water, thus the photosynthetic activities would be affected [12].

There are many available technologies for colour removal application. This includes membrane separation, chemical oxidation, electrocoagulation, adsorption, and coagulation and flocculation [13]. However, each of the methods has its limitation in their application. For example, electrochemical oxidation is a cost intensive process where most commonly require controlled condition for reaction and high operating cost [12]. Therefore, adsorption method is currently preferable due to its simplicity in operation [11]. This promising prospect had encouraged researches to explore the dyes removal by using low cost adsorbent such as mandarin peels, grapefruit peels, peanut hull, peat, rice husk, wood sawdust, peanut husk, pine sawdust and banana peel [13].

In this study, Cassava leaf (CL) was chosen as adsorbent on dye removal application. This is because huge amounts of CL are currently disposed as wastes after roots harvesting activities [14]. Unlike other food based low-cost adsorbents, the presence of cyanogenic glucosides in CL has make it impracticable to consume in human diet [15]. Therefore, only small amounts of CL might be consumed either as supplements to non-ruminant diet or as condiments in human diet [14] and there is no conflict of food utilization. CL, with an all year-round availability, flexibility in planting and harvesting time, drought tolerance ability where it can easily grow and easy survival on low nutrients soils, is a good candidate for low cost and wide availability of natural adsorbent [16]. In this work, several parameters i.e. pH, adsorbent dosage, temperature and contact time was varied to investigate the effect of these parameters to the adsorption of MB and CR.

2. Materials and Methods

2.1 *Materials*

All chemical used in this works was analytical reagent grade and it was use without any further purification. In general, the main materials required in this research were the adsorbents, CL and dye powders (MB and CR). Both dyes used in this study was supplied by R&M Chemicals. For MB dye, its C.I. no. is 52015 and its wavelength, λ is 664 nm. Meanwhile, the C.I. no. of CR dye used in this research is 22120 and its wavelength, λ is 495 nm. The chemicals used to adjust the pH of the dye solutions for dye adsorption experiments were hydrochloric acid, HCl (0.1M) and sodium hydroxide, NaOH (0.1M) from Fisher Chemical.

2.2 *Preparation of Raw Material*

CL was purchased from Setapak Jaya market, Kuala Lumpur. The leaf was washed thoroughly to remove dust and impurities on the leaf surface. Then, it was cut into small pieces and underwent pre-treatment to avoid disturbances of its green pigments to dye measurement. In the pre-treatment steps, CL was boiled for 30 minutes using microwave oven (Model NN-CD997S, Panasonic, Japan). Thereafter, the leaves were soaked in 95% ethanol solution at constant temperature of 80˚C, controlled using water bath (Model Isotemp® GDP 28, Fisher Scientific, USA).

After that, the leaves were oven dried for 6 hours before being cut into smaller pieces of lesser that 1.0 mm using cutting mill (Model Pulverisette 19, Fritsch, Germany). The leaf powder was collected in air tight container and stored in air cooled chiller (Model DW-FL253, Remi, India) at the temperature of 1.0˚C.

2.3 *Characterization of Adsorbent*

Field Emission Scanning Electron Microscope (FESEM) (Model JSM-7800F, JEOL, Japan) was used to obtain visual image of the adsorbent before and after dye uptake. Fourier Transform Infrared Spectroscopy (FTIR) was conducted using Fourier Transform Infrared Spectrophotometer (Model IRTracer-100, Shimadzu, Japan) to investigate the functional group that contribute to the adsorption process.

2.4 *Adsorption Experiments*

The effect of pH on dye removal was investigated for pH 2, pH5, pH7, pH9, and pH 11. Dye solution was prepared at constant initial concentration of 100 mg/L. This level of initial concentration was selected in this study as an initial gauge on the ability of CL to adsorb the dyes. CL was added into the dye solution and agitated at a constant speed of 300 rpm for 1 hour at room temperature.

The effect of adsorbent dosage on MB and CR dye adsorption was investigated by varying different dosage of CL of 0.2 g, 0.4 g, 0.6 g, 0.8 g and 1.0 g into 100 mg/L of dye solutions at alkaline (pH=11.0) and acidic (pH=2.0) condition for MB and CR dye adsorption, respectively. The dye solutions was agitated using magnetic stirrer at constant speed of 300 rpm for 1 hour at room temperature.

To study the impact of temperature towards MB and CR dye adsorption, the temperature of dye solutions was varied at 25 ˚C, 45 ˚C, 55 ˚C, 65 ˚C and 75 ˚C. The solutions was then agitated with 0.5 g of CL at 300 rpm in alkaline condition (pH=11.0) of MB dye solution, while 0.2 g of CL in acidic condition (pH=2.0) of CR dye solution at 300 rpm for 1 hour.

A preliminary study conducted to trace the time needed for the batch adsorption process to reach equilibrium indicated that the concentration of dye stabilized within 120 minutes of contact time. Therefore, a total of 6 sets of 100 mg/L of dye solutions were prepared to study various contact time from 20 minutes to 120 minutes towards MB and CR dye adsorption on CL. The solution was then agitated with 0.5 g of CL at 300 rpm in alkaline condition (pH=11.0) of MB dye solution, while 0.2 g of CL in acidic condition (pH=2.0) of CR dye solution.

The experiment for each parameter was conducted in duplicate. The average dye removal percentage through the CL adsorption was calculated based on the following equation:

$$Removal\ percentage = \frac{(C_0 - C_e)}{C_0} \times 100\% \qquad (1)$$

where $C_o$is the initial dye concentration (mg/L) while C_e is the final dye concentration at equilibrium condition (mg/L).

2.5 *Adsorption Isotherm Study*

Adsorption isotherms are one of the important information to understand the mechanism of the adsorption. In this study, Langmuir, Freundlich and Temkin isotherms were chosen because they are most commonly used isotherm models which indicates the output obtained is dependable.

Langmuir isotherm can be represented by Eq. (2):

$$q_e = \frac{Q_0 K_L C_e}{1+K_L C_e} \tag{2}$$

where C_e is equilibrium concentration of adsorbate (mg.L^{-1}); q_e is the amount of adsorbate adsorbed per gram of the adsorbent at equilibrium (mg.g^{-1}); Q_0 is the maximum monolayer coverage capacity (mg.g^{-1}) and K_L denotes as Langmuir isotherm constant (L.g^{-1}). Eq. (2) can be transformed into linear form and expressed as:

$$\frac{1}{q_e} = \frac{1}{Q_0} + \frac{1}{Q_0 K_L C_e} \tag{3}$$

Freundlich isotherm is represented by Eq. (4):

$$q_e = K_f C_e^{1/n} \tag{4}$$

where K_f is Freundlich isotherm constant (mg.g^{-1}) and n is the adsorption intensity. Eq. (4) can be linearized via log function as in Eq. (5):

$$\log q_e = \log K_f + {}^{1}\!/_{n} \log C_e \tag{5}$$

Meanwhile, Temkin isotherm can be represented by Eq. (6) and Eq. (7):

$$q_e = B\ln(A_T) + B\ln(C_e) \tag{6}$$

$$B = \frac{RT}{b_T} \tag{7}$$

where A_T is Temkin isotherm equilibrium binding constant (L.mg^{-1}), b_T is Temkin isotherm constant R is the universal gas constant (8.314 J.mol^{-1}.K^{-1}), T is operating temperature at 298 K, B is constant related to heat of sorption (J.mol^{-1}).

3. Results and Discussion

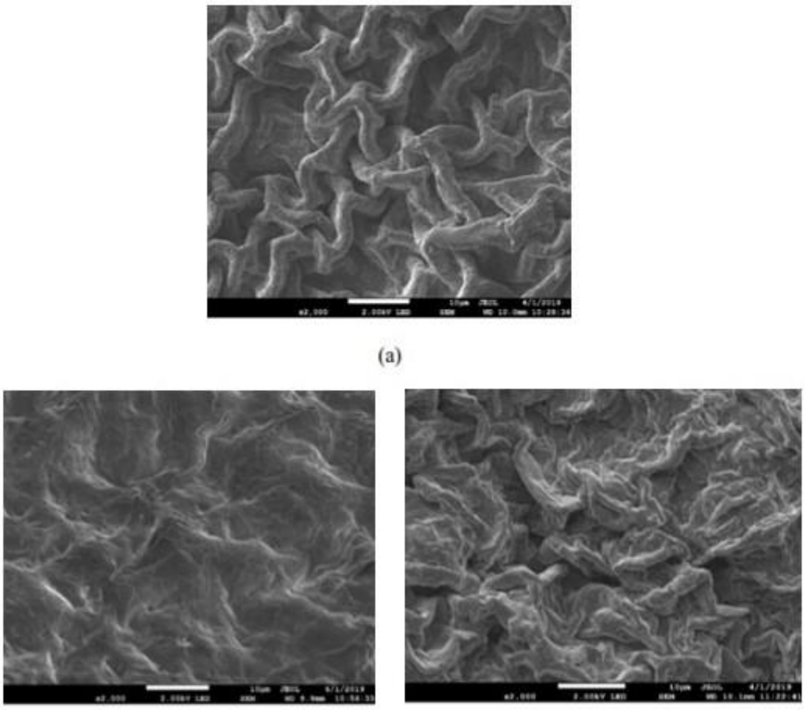
3.1 *Characterization of CL*

In order to comprehend the mechanisms of dye molecule binding on adsorbent surface, characterization of the visual and chemical properties of CL is essential. Fig. 3 shows FESEM images of CL, before and after loading with MB and CR dye.

Based on Fig. 3(a), CL appeared to have some shallow sites on its surface before dye adsorption. After binding with MB dye, the surface seems to have a smooth layer of dye attached on it as shown in Fig. 3(b). Comparatively, as shown in Fig. 3(c), the surface structure of CL after CR dye adsorption was uneven, indicating a probable different adsorption mechanism. This was irregular and rough surface characteristics was also observed by Adrian et al. [17] when the CL was tested for adsorption of cadmium (ii) ions from aqueous solution.

Fourier Transform Infra-Red (FTIR) spectrometry was conducted to get an insight on the functional group information of CL before and after MB and CR dye adsorption. As shown in Fig. 4, FTIR spectra showed that there are chemical interactions between the functional groups in the CL which consists of ether (-C-O-C), carbonyl (-C=C), nitrile (-C≡N), amine (-N-H) and hydroxyl (-OH).

The figure shows a wide range of absorption peaks appear where typical peaks at wavenumber range of 3300-3500 cm^{-1} represents the O-H stretching of CL. Other sharp peak at wavenumber 1640 cm-1 signifies the presence of -C=O group. Meanwhile, the peak at 2200-2250 cm^{-1} indicated the existence of -C≡N group. Both FTIR spectra after MB and CR dye adsorption showed a lower transmittance compared to before dye uptake. These low transmittance indicated that there was a high population of bonds due to vibrational energies corresponding to the incident light [18]. Hence, these slight changes can confirm the adsorption process took place in CL.


(a)

(b)

(c)

Fig. 3. FESEM images of CL at 2000x magnification (a) before dye adsorption (b) after MB dye adsorption (c) after CR dye adsorption

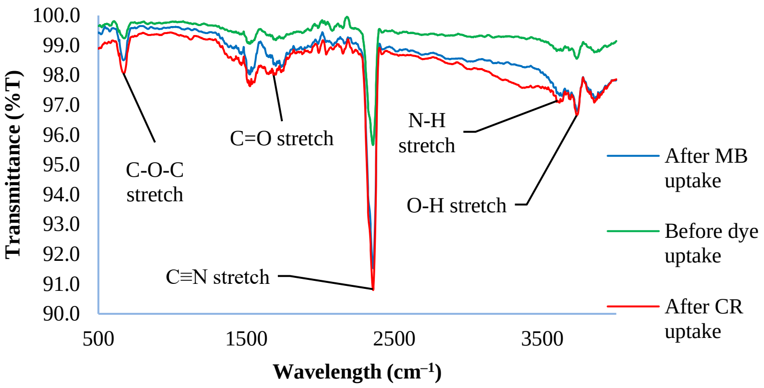


Fig. 4. FTIR spectra of CL before and after MB and CR dye adsorption

3.2 *Effect of pH on Dye Adsorption*

At fixed contact time, adsorbent dosage and temperature, the impact of pH on the MB and CR dye removal rate is presented in Fig. 5.

Based on the graph trend in Fig. 5, the removal of MB dye appeared to be better when the solution was at pH 5 onwards. The removal percentage of MB dye at pH 11.0 was 99.94% as compared to 94.3% of removal when the pH was at 2.0.

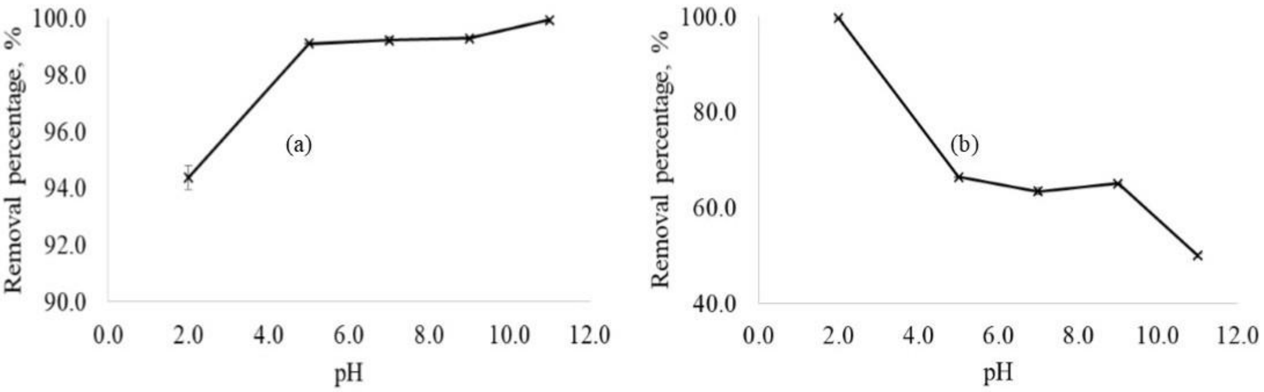


Fig. 5. Removal percentage of (a) MB and (b) CR dye at varied pH condition (Temperature = 25˚C; contact time = 60 min; adsorbent dosage for MB dye = 0.5 g; adsorbent dosage for CR dye = 0.2 g)

In contrast, pH 2.0 appeared to be the favourable condition for removal of CR dye. 99.67% of CR removal was obtained at pH 2.0 and a significant decrement of removal rate was observed as the pH was increased. At pH 11.0, only 49.96% of CR removal was obtained.

These trends can be explained by the electrostatic attraction exists between the charge of dye and the adsorbent surface [13]. In the case of MB which is a cationic dye, favourable adsorption capacity was achieved at pH of 11.0. This was due to the alkaline conditions which provided a greater amount of negative charges on the CL surface. Thus, high electrostatic force between negative charge on adsorbent surface and positive charge of MB dye resulted in favourable adsorption [13]. Meanwhile, CR, being an anionic dye, behaved as negative charge and preferred to associate with positive charge of adsorbent surface. Therefore, under acidic condition, it was able to achieve higher adsorption performance [13].

3.3 *Effect of Adsorbent Dosage on Dye Adsorption*

Fig. 6 show the impacts of adsorbent dosage on the removal percentage of MB and CR dye, respectively. Based on Fig. 6 (a), an increasing trend of removal was observed for MB dye (from 98.73% to 99.99%) when the dosage of CL was increased from 0.2 g to 1.0 g. This is because greater the adsorbent dose, more adsorption sites are available for adsorption process [19].

However, as shown in Fig. 6 (b), the removal rate of CR dye by CL decreased (from 99.67% to 98.84%) when the dosage of CL increased. This could probably due to the aggregation of adsorbent in excessive amount under acidic condition which hindered the adsorption sites of CL [21].

3.4 *Effect of Temperature on Dye Adsorption*

The impact of temperature on the removal rate of MB dye and CR dye are as shown in Fig. 7, respectively. Based on Fig. 7 (a), an increasing trend was observed for the removal rate of MB dye (from 99.45% to 99.91%) as the temperature was increased from 25˚C to 45˚C. However, a decreasing trend can be seen when the temperature was further increased to 50˚C and onwards with only 98.95% removal at 75˚C.

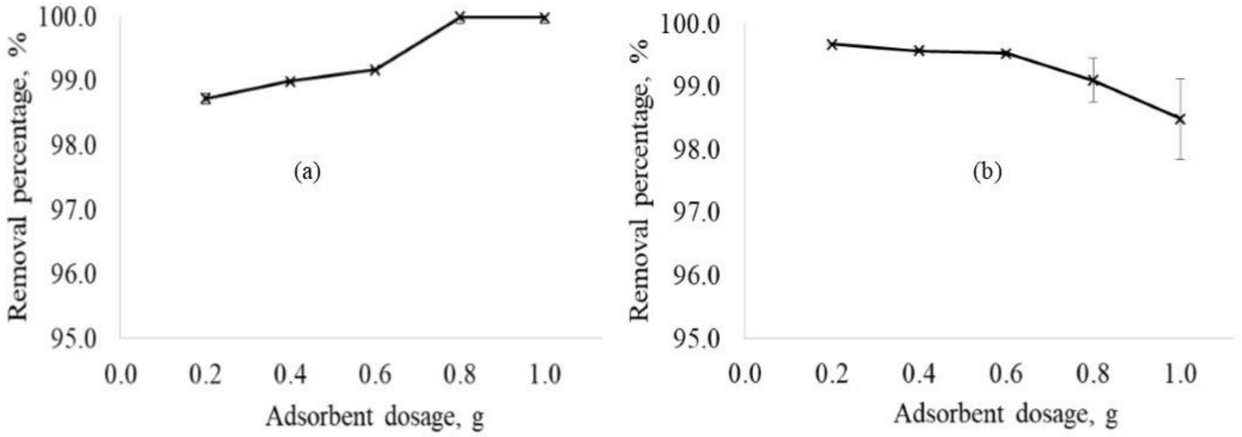


Fig. 6. Removal percentage of (a) MB and (b) CR dye at varied adsorbent dosage (Temperature = 25˚C; contact time = 60 min; pH for MB adsorption = 11.0; pH for CR adsorption = 2.0)

The frequency of collisions between the adsorbent and adsorbate (dye solution) increased at higher temperature which promote adsorption on the adsorbent surface [13]. Nevertheless, high temperature of dye solution might break the intermolecular hydrogen bonding between dyes and adsorbent, which act as the main contributors in the process of adsorption [20].

On the other hand, a decreasing trend was observed for the removal rate of CR dye (from 99.67% to 97.95%) as the temperature was increased from 25 to 75 ˚C as illustrated in Fig. 7 (b). This might be due to a more acidic pH at higher temperature [22]. At this elevated acidic condition, CR dye were prone to be more soluble in water and difficult to be removed from the solution [23].

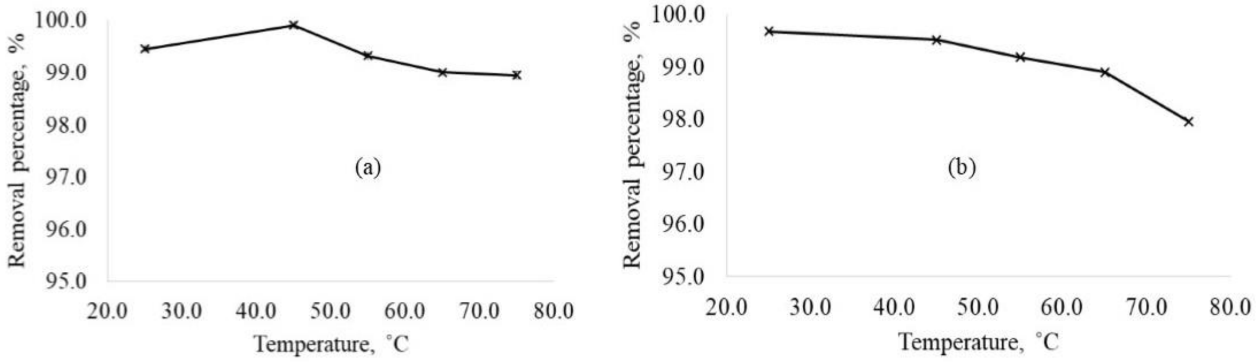


Fig. 7. Removal percentage of (a) MB and (b) CR dye at varied temperature (Contact time = 60 min; MB adsorption: adsorbent dosage = 0.5 g, pH = 11.0; CR adsorption: adsorbent dosage = 0.2 g; pH = 2.0)

3.5 *Effect of Contact Time on Dye Adsorption*

Based on the optimum condition obtained from each of the batch parameter study (pH, adsorbent dosage and temperature), the impact of contact time on the removal rate for MB and CR dye in CL is presented in Fig. 8, respectively.

Based on Fig. 8 (a), the removal rate of MB dye showed slight increase trend from 98.58% to 99.88% and achieved equilibrium after 60 minutes. This is because greater contact time allowed for more adsorbate-adsorbent interactions. Hence the dye adsorption increase until the adsorbent reached its maximum capacity because the adsorbent surface has achieved its equilibrium stage [24].

However, for CR dye removal, there were no significant increment since there was a rapid removal observed within 20 minutes, which was 99.49% and it continued to slightly increase to 99.67% then remained constant as shown in Fig. 8 (b). This condition can be further described as bulks of actives sites are available on the adsorbent surface at initial phase [25]. Nevertheless, in the course of time, the adsorbent sites remaining cannot be attached because of the presence of repulsive force between the adsorbent and adsorbate [26].

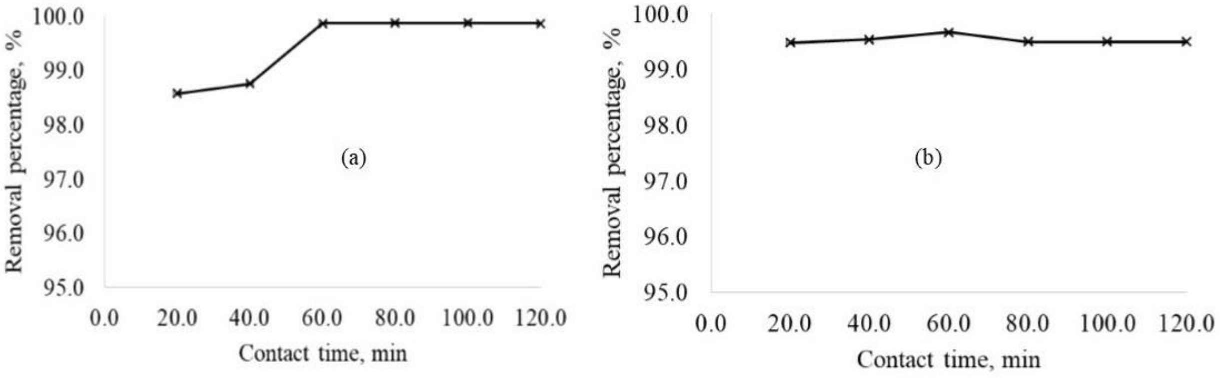


Fig. 8. Removal percentage of (a) MB and (b) CR dye at varied contact time (MB adsorption: temperature = 45˚C, adsorbent dosage = 0.5 g, pH = 11.0; CR adsorption: temperature = 25˚C; adsorbent dosage = 0.2 g; pH = 2.0)

3.6 *Adsorption Isotherm Study*

To ensure the design of the adsorption processes operating at optimum conditions, it is crucial to generate the most suitable correlation for the equilibrium curves. For this purpose, adsorption isotherms were developed to evaluate the mechanisms of the adsorption process where the interaction between adsorbent and adsorbate was clearly described. The isotherms plot is shown in Fig. 9 and Fig. 10 for MB and CR dye, respectively.

As shown in Fig. 9 (a) and Fig. 9 (b), a perfect linearity (R^2=1) can be observed from both Langmuir and Freundlich isotherm plot for MB dye adsorption. Based on the parameter obtained from Langmuir and Freundlich isotherms, the R_L value obtained from model was 0.818 which is within the range of 0 and 1, thus the adsorption is favorable and monolayer adsorption was expected [27]. As for Freundlich isotherm, the value of n is 1.027 where n > 1 which indicates the adsorption of MB dye onto CL is favorable and it is a physisorption process. The situation of n > 1 might reduce the interaction of adsorbent-adsorbate by increasing the density of surface [28]. From the comparison, Temkin isotherm has lower linearity (R^2=0.953) compared to Langmuir and Freundlich, thus it is not considered for the uptake mechanisms for MB dye [29].

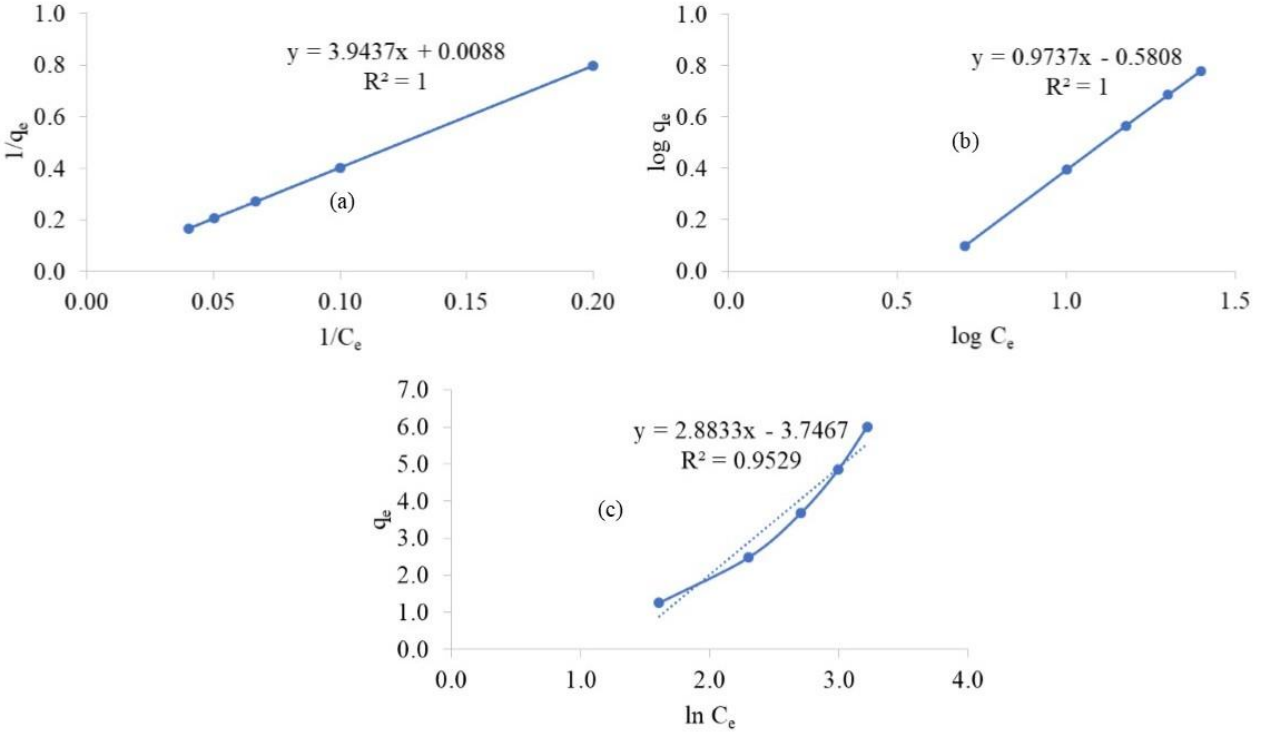


Fig. 9. (a) Langmuir isotherm (b) Freundlich isotherm (c) Temkin isotherm on MB dye adsorption onto CL

In contrast, the uptake mechanisms for CR dye on CL powder was best described in Temkin isotherm where the R^2 obtained from Temkin isotherm plot is 0.976 which indicates the bonding energies during adsorption of CR dye onto CL are distributed uniformly [30]. From the calculated parameters, the heat of adsorption is 2.03 $J.mol^{-1}$ where a positive value was obtained. Thus, the adsorption of CR dye onto CL was an endothermic process [27].

4. Conclusion

In this study, CL was found to be an effective adsorbent for MB and CR dyes. MB was more effectively removed at alkaline condition which acidic condition was more preferable for CR dye removal. The removal of MB was increased with increase of adsorbent dosage, but a too high of dosage at acidic condition could deteriorate the efficiency of CR dye removal by CL. Meanwhile increasing the temperature after a certain threshold could also lower the dye removal efficiency by CL. Contact time of at least 60 min is preferable to allow the adsorption process to reach equilibrium. For the adsorption isotherm, the adsorption of MB dye on CL was deduced to be monolayer and heterogeneous surface while the adsorption of CR dye on CL was an endothermic process.

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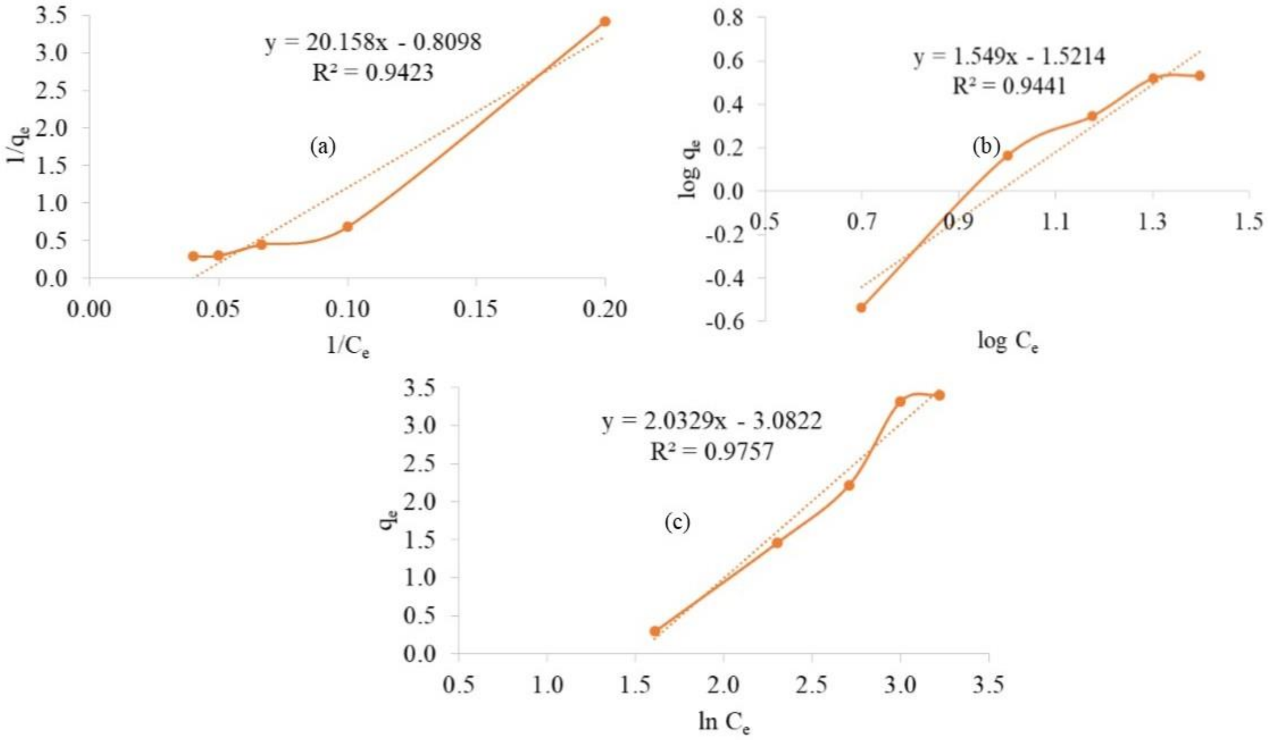

Fig. 10. (a) Langmuir isotherm (b) Freundlich isotherm (c) Temkin isotherm on CR dye adsorption onto CL

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