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The investigation of experimental condition and Kinetic study of Methylene Blue adsorption onto Neem (Azadirachta indica) leaf powd

Kornwipha Saengbutr, Nittaya Wangphon and Ratchaneekorn Wanchanthuek

The Center of Excellence for Innovation in Chemistry and department of Chemistry, Faculty of Science, Mahasarakham University, Kantarawichai District, Mahasarakham 44150, Thailand

Abstract- The methylene blue (MB) absorption onto neem leaf powder was investigated in term of both adsorption efficiency and kinetic study. The effects of operation parameters i.e., MB concentration, time of adsorption, adsorbent dosage, pH of solution and temperature were investigated. The results showed that all parameter could affect the adsorption efficiency. The adsorption isotherm and kinetics over various pH and temperature were also studied. It was found that the adsorption capacity was increased with pH and temperature increased; suggested the exothermic process. The experimental data showed that the MB adsorption was fitted with Langmuir isotherm and the kinetic model was the pseudo-second order.

Index Terms- Methylene blue, Adsorption, kinetic study, Neem leaf powder

I. INTRODUCTION

The environmental concerns including the sustainable energy, re-used materials as well as the environmental friendly operation are the often mentioned topics in every organization. Nowadays, the industrial technology has been rapidly developed to support the fast increasing of the population demands. However, these industrial could release the toxic effluent into the river and cause the environmental problem. The easy parameter to classify the waste water is the color, therefore the contaminated dye in waste water need to be removed before released to natural stream. The dye removal process which is the low cost of operation and occurred through the green reaction under the usage of the natural waste is the interested method. This is leaded to the de-colorizing of waste water by using the agriculture waste through the adsorption technique. The advantages of this technique among the others are the ease of operation, various source of adsorbent, economy favourable, environmental friendly and separated easily after process.

There are many agriculture wastes which proposed as adsorbents such as carbonized jackfruit peel [1], apple pomace, wheat straw [2], orange peel [3], water hyacinth roots [4], water hyacinth [5], raw and activated date pits [6], perlite [7], rice husk [8], peanut hull [9], palm fruit bunch [10], sawdust [11], coir pith [12] and neem leaf powder [13]. Neem tree or *Azadirachta indica* of family *Meliaceae* is the deciduous tree species and common find in Thailand. Its fresh or green leaves, bark, seed, flower and other parts have been used traditionally in medicinal, germicidal and insect pesticide [13]. During the autumn season, the mature leaves are defoliated and become the huge volume of waste during December to February. The fallen leaves normally left without any usage. The possible application of these leaves to increase the value is using as the adsorbent.

Therefore, the fallen leaves were collected to prepare as the adsorbent for MB removal in the present study. The MB was selected as the poisoned organic dye which could be presented in waste water. The experimental condition such as the initial concentration of MB, the adsorbent loading, pH and temperature was investigated. The amount of MB was followed by using the simple apparatus which was the spectrometer. Then, the adsorption isotherm (Langmuir and Freunlich isotherm) and the kinetic model were finally investigated.

II. EXPERIMENTAL

2.1 Neem leaf powder preparation

The fallen leaf of neem was collected and washed with distilled water for several times to remove the dust and dirt. The clean neem leaf was then left at room temperature for 6 hours to remove the excess water and then dried under the oven at 100 ºC for 24 hours. The sample was crushed in mortar and screened to separate the particle less than 250 μm using the sieve in order to achieve the powder. The resulted powder was boiled in water for 2 hours to remove the residual pigment and digested some of the leaf fiber. Then the mixture was filtered and dried at 100 ºC for 24 hours; resulted the neem leaf powder for further study.

2.2 Adsorption study

The stock solution of MB (80×10^{-5} M) was first prepared and then diluted (10×10^{-5} M). The batch experiments were carried out to study the effect of MB concentration, neem leaf powder loading, pH and temperature. The investigated parameter was varying and kept other parameters constant. In order to study the effect of initial MB concentration, the second stock solution was diluted into 0.8×10^{-5} - 6.8×10^{-5} M and adjusted pH to 5 by using either HCl or NaOH. The solution of MB was stirred and heated to desired temperature (60 ºC), then the neem leaf powder was added to the mixture (1.2 g/L). The mixture was kept stirring and heated for 70 min, then the mixture was subjected to centrifuge and the supernatant was measured the concentration of residual MB by Spectrophotometer at 665 nm.

2.3 Calculation

The percentage of dye adsorbed (%MB removal) and the amount of dye adsorbed on neem leaf powder (q_t) were calculated by equation (1) and (2), respectively

$$\%\ \text{MB removal} = \frac{C_{in} - C_{out}}{C_{in}} \times 100 \qquad (1)$$

where C_{in} and C_{out} denote to the initial and final concentration of MB (mol/L), respectively.

The MB removal per unit weight of adsorbent at time t, q_t (mg/g) were obtained from equation (2)

$$q_t = \left(\frac{C_{in} - C_{out}}{M} \right) V \qquad (2)$$

V is the volume of MB solution in adsorption process (L) and M is the mass of neem leaf powder (g).

In the kinetic study, the MB removal per weight of adsorbent at the equilibrium named q_e which obtained from equation (3)

$$q_t = \left(\frac{C_{in} - C_e}{M} \right) V \qquad (3)$$

where C_e is the concentratin of residual MB at the equilibrium. This could be found from the kinetic plot.

III. Results and Discussion

3.1 Effect of reaction condition

The effect of the initial MB concentration to the MB removal is shown in Figure 1. The MB initial concentration was varied between 1 x 10^{-6} – 7 x 10^{-6} M, the resulted % MB removal was slightly decreased from 96.5% to 93.5% which seem constant. The results exhibited that the neem leaf powder was one of the effective adsorbent because the % dye removal was almost 100% when using the adsorbent only 1.2 g/L at pH 10. Ponnusami and co-workers also studied the effect of initial dye concentration to MB adsorption over teak leaf power [14]. They varied the concentration of effluents between 10-50% (by vol.) and correlated the dye removal to the COD level. They found a similar result to ours that at high initial dye concentration less MB removal was observed. Moreover, various studied also found that at low concentration of initial dye concentration resulted in the high percentage of dye removal [15-17]. This indicated that the lack of active sites for dye adsorption at high dye concentration which resulted in decreasing percentage of dye removal at high dye concentration. Therefore, to increase the % of dye removal, the adsorbent contained high active site was required. However, the actual amount of dye absorbed per unit of adsorbent mass were increased with the increased of initial dye concentration [14-17] with in agreement with our results.

The percentage of MB removal was increased with the neem leaf powder increased and reached almost 100% dye removal when using adsorbent only 2 g/L. The dosage of neem leaf powder was varied between 0.5-2 g/L and the resulted removal power was 80-100% when adsorption study was run at pH 10 and MB concentration was 4 x 10^{-5} M. This finding could be due to the increasing of the surface area and the active sites for dye adsorption when increasing the dosage and the results were in the agreement with [17-18]. However, there was the optimum value because the initial dye concentration was fixed. The resulted in Figure 2 still suggested that the neem leaf powder was the good adsorbent for MB removal.

The adsorption of the target dye over the surface of adsorbent is the key reaction in MB removal. The MB is the cationic dye, therefore, the adsorption of MB is strongly depend on the surface charge of adsorbent. The consideration of the surface charge of adsorbent is related directly to the pH of solution. Thus, the effect of the pH to the dye removal efficiency was taken into account. Figure 3 showed the effect of pH which varied between 3-10. The percentage of MB removal was increased from 50 to 80% at the studied pH range. The results showed that the pH in basic range is favour for the MB adsorption over neem leaf powder. The similar findings were exhibited in Sharma and K. G. Bhattacharyya [19] and Gusmao et al. [20]. The Cd^{2+} removal by using neem leaf powder was studied [19]. The effect of pH was also investigated and showed that at acidic pH between 2-3.5 exhibited no Cd^{2+} removed. When pH changed to 4, about 9% of Cd^{2+} eliminated and pH 9.5 could remove about 94%. They explained that at low pH there was the competition between H^+ and Cd^{2+} to adsorb at the active site whereas the phenomena was reduced at alkaline pH and facilitated the Cd^{2+} adsorption by OH^-. Gusmao et al. [20] also studied the adsorption of cationic (MB) dye and anionic dye (gentian violet, GV) over sugarcane bagasse. The pH was varied between 2-10 and the results were showed that at acidic pH was suitable for the anionic dye and alkaline pH was favour for cationic dye adsorption. This was supported that the H^+ at low pH could decorate the surface of adsorbent and resulted in the decorated adsorbent suitable for anionic dye. On the other hand, the OH^- in alkaline pH was modified the adsorbent surface and facilitated the adsorption of cationic dye.

According to our finding, the possible adsorption of MB in alkaline region could expressed as;

Neem leaf powder + OH^- → Neem leaf powder-OH^- + MB^+ → Neem leaf powder-OH^- — MB^+

The temperature is also the important factor to interfere the adsorption efficiency. The various temperatures were operated in the MB adsorption onto the neem leaf powder and the results were expressed in Figure 4. The obvious results were found as the higher temperature was facilitated the adsorption efficiency; when the temperature increased from 30-70 °C the adsorption was growth from 70-90%. Therefore, it could be implied that the MB adsorption over neem powder was the exothermic process. The effect of temperature of the Brilliant green adsorption on the neem leaf powder was also studied and in agreement with our present study [21]. They suggested that high temperature the activation of the surface groups such as alcoholic, carboxylic and phenolic groups were found and these was raised the interaction of these surface groups and the OH^- in solution. Therefore, the higher negative surface charge of adsorbent was achieved, leaded to the higher adsorption capacity to MB. Moreover, the mass transfer coefficient was also increased at higher temperature which pushed more dye molecules to be closer to the adsorption site.

3.2 Adsorption isotherm

Langmuir and Freundlich isotherms were applied to determine the adsorption model of MB onto MgO. These isotherms have been selected to be a model for investigation the phenomena of MB adsorption over the neem leaf powder because the Langmuir isotherm is illustrated the monolayer adsorption. This model basically assumed that the adsorption occurred on homogeneous surface and the surface contained the identical adsorption sites. Moreover, each of adsorption site can adsorbed only 1 adsorbing molecule (related to monolayer adsorption) and there is no interaction between the adjacent of adsorbed molecule.

The Langmuir equation is shown in equation (3-4) [22]:

$$q_e = \frac{Q_0 K_L C_e}{1 + K_L C_e} \quad (3)$$

The reciprocal equation of equation (3) can be expressed as follows and illustrated in linear form:

$$\frac{C_e}{q_e} = \frac{1}{Q_0 K_L} + \frac{1}{Q_0} C_e \quad (4)$$

Q_0 (mg/g) is the maximum adsorption capacity of the dye (monolayer formation) per gram of adsorbent. K_L is a Langmuir constant related to the affinity of the binding sites (l/mg) or the free energy of adsorption. The plot of C_e/q_e vs. Ce was showed in Figure 6 (a).

The Freunlich isotherm is exhibited the multilayer adsorption over the adsorption sites. This model is the extention of the assumptions in Langmuir isotherm for achieving the imperfection of the surface morphology by the factor of $\frac{1}{n}$. The assumption was mentioned in the surface roughness, inhomogeneity and adsorbate-adsorbate interactions which describing reversible adsorption. Thus, this could be implied that the adsorption could be the physical adsorption. The empirical Freundlich isotherms is also shown in equation (5) [23]

$$q_e = K_f C_e^{1/n} \quad (5)$$

The logarithmic form of this equation can be expressed as:

$$\ln q_e = \ln K_f + \frac{1}{n} \ln C_e \quad (6)$$

where K_f and n are the Freundlich constants which related to the adsorption capacity and the adsorption intensity of adsorbent. The relationship between $\ln q_e$ and $\ln C_e$ is a linear plot according to equation (6) shown in Figure 6 (b). The correlatin coefficient (R^2) could be used to indicate well fitted of experimental data.

The relationship between percentage of MB removal and reaction time known as kinetic plots were shown in Figure 5 (a) and (b); under the dependence of pHs and temperatures, respectively. The results confirmed that the alkaline pH was enhanced the MB removal and high temperature was the favor condition for the MB adsorption process over neem leaf powder. From these plots, the data was further analyzed to classify the type of adsorption isotherm and reaction order.

According to equation (4), the curve of C_e/q_e vs. C_e was plotted to examine the Langmuir isotherm which showed in Figure 6 (a). The level of well fitting to Langmuir isotherm was consider with respected to the correlation coefficient value (R^2) as the plotted relationship was the linear type: referred to equation (4). Figure 6 (c) showed linear equation and R^2 of Langmuir plot of the adsorption process operated at pH 10. Similarly, the curve in Figure 6 (b) was the Freunlich isotherm plot as refered to equation (6) and Figure 6 (d) also showed the linear fitted equation and R^2 of the reaction at pH 10. The comparison of R^2 in Figure 6 (c) and (d) showed that R^2 of Langmuir isotherm was higher than Freunlich isotherm; indicated that this process fitted well with Langmuir isotherm. The R^2 of adsorption isotherm investigation (in both the effect of pH and temperature) showed in Table 1 and 2. The results were in agreement that R^2 of Langmuir isotherm was higher than Freunlich isotherm, therefore, clearly confirmed that the MB removal by neem leaf powder was followed the Langmuir isotherm

From the slope of Langmuir plot, the maximum adsorption capacity (Q_0) of neem leaf powder could be calculated. The Q_0 was calculated from the optimum condition (at pH 10 and 70 °C) and showed Q_0 = 16.67 mol/g. Table 3 showed Q_0 of MB on various natural adsorbent. However, the Q_0 over neem leaf powder was studied before [13]. The comparison Q_0 in 2 studies were compared which showed clearly different; 2.35 x 10^{-5} mol/g [13] and 1.67 x 10 mol/g (this study). The different of these 2 adsorption processes was the reaction condition such as pH and temperature. Therefore, it suggested that the condition during the adsorption process played the vital rule in the removal capacity of this kind of adsorbent.

The similarly results were also found in the previous studies such as Han et al [12] and Awwad et al [29]. The adsorption isotherm of MB onto lotus leaf powder was examined [12] and Langmuir isotherm was found to be fitting well with the adsorption system by expressed high R^2 (0.9910). Moreover, The Cd^{2+} bioadsorption over loquat leave powder was also studied and the experimental data was related to the Langmuir adsorption at R^2 = 0.9999 whereas R^2 of Freunlich isotherm was only 0.9765.

3.3 Adsorption kinetic study

The Pseudo order approximation used to explain the reaction which contains at least one excess component and the adsorption rate of the excess component is constant. Pseudo-first-order kinetic model assume that the adsorbate uptake to the surface with time was directly proportional to difference in saturation concentration and the adsorbent amount [24]:

$$\frac{dq_t}{dt} = k_1 \left(q_e - q_t \right) \quad (7)$$

q_e and q_t are the amount of dye adsorbed (mg/g) at equilibrium and at time t, respectively. And k_1 is the rate constant of Pseudo-first-order (min^{-1}).

Equation (7) was integrated with the boundary condition at t=0, q_t = 0 and at t=t, q_t=q_t and rearranged the equation to obtain equation (8)

$$\log(q_e - q_t) = \log q_e^{-k_1 t / 2.303} \quad (8)$$

Similar with

$$q_t = q_e\left(1 - e^{-k_1 t}\right) \quad (9)$$

When the experimental data was plotted between q_t and t and the shape of the expressed relationship is in exponential curve as shown in equation 9. This indicated that the adsorption is followed the pseudo-first order. The resulted curved was shown in Figure 7 (a).

In term of the Pseudo-second-order model with proposed by McKay and Ho in 1999 [25] was:

$$\frac{dq_t}{dt} = k_2\left(q_e - q_t\right)^2 \quad (10)$$

where k_2 is the equilibrium rate constant of Pseudo-second order adsorption (g/mg min).

Similarly to pseudo-first-order, equation 10 was integrated with similar condition boundary to obtain equation (11):

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (11)$$

The substantiation of the Pseudo-second order kinetic model was the plot of $\frac{t}{q_t}$ and t. If the trend is followed the linear relationship of equation 11, it suggested that the adsorption process is the pseudo- second order.

The kinetic model was investigated by using the curves in Figure 7 (a) and (b). The relationship of q_t and t (Figure 7 (a) and (c)) was the pseudo-1^{st} order plot and the obtained plot was not followed the exponential as type of equation (9). This could be implied that the adsorption process was not the pseudo-1^{st} order reaction. In other hand, the plots of $\frac{t}{q_t}$ and t (in Figure 7 (b) and (d)) were the pseudo-2^{nd} order plot which was the linear relationship. Similarly, the well fitting was considered by R^2. The obtained R^2 of the pseudo-2^{nd} order which studied the effect of pH and temperature effect were reported in Table 1 and 2. They were all in agreement and found that R^2 was closely to 1 (0.987-0.997). This was indicated that adsorption was fitted well with the pseudo-2^{nd} order.

Refer to equation 11, the interception of pseudo-2^{nd} order plot could be calculated to obtained k_2 and it showed $k_2 = 1.19 \times 10^5$ g/mg min. The obtained k_2 could be suggested that the adsorption is favor occurred.

IV. Conclusions

The optimum condition of MB removal using neem leaf powder was investigated. The results found that the removal was favour at low MB concentration, higher pH and higher temperature. The optimum loading of adsorbent was 1.2 g/L. The experimental data was fitted with Langmuir isotherm better than Freulich isotherm in both pH and temperature dependences. This was suggested that the adsorption was monolayer adsorption with the chemisorptions. Moreover, the kinetic analysis in both the pH and temperature effect was in agreement; showing that the pseudo second-order process was fitted with the data.

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AUTHORS

First Author – Kornwipha Saengbutr , The Center of Excellence for Innovation in Chemistry and department of Chemistry, Faculty of Science, Mahasarakham University, Kantarawichai District, Mahasarakham 44150, Thailand

Second Author – Nittaya Wangphon, The Center of Excellence for Innovation in Chemistry and department of Chemistry, Faculty of Science, Mahasarakham University, Kantarawichai District, Mahasarakham 44150, Thailand

Third Author – Ratchaneekorn Wanchanthuek, The Center of Excellence for Innovation in Chemistry and department of Chemistry, Faculty of Science, Mahasarakham University, Kantarawichai District, Mahasarakham 44150, Thailand, Tel./ Fax. +66 43 754 246, E-mail address: ratchaneekorn.p@msu.ac.th

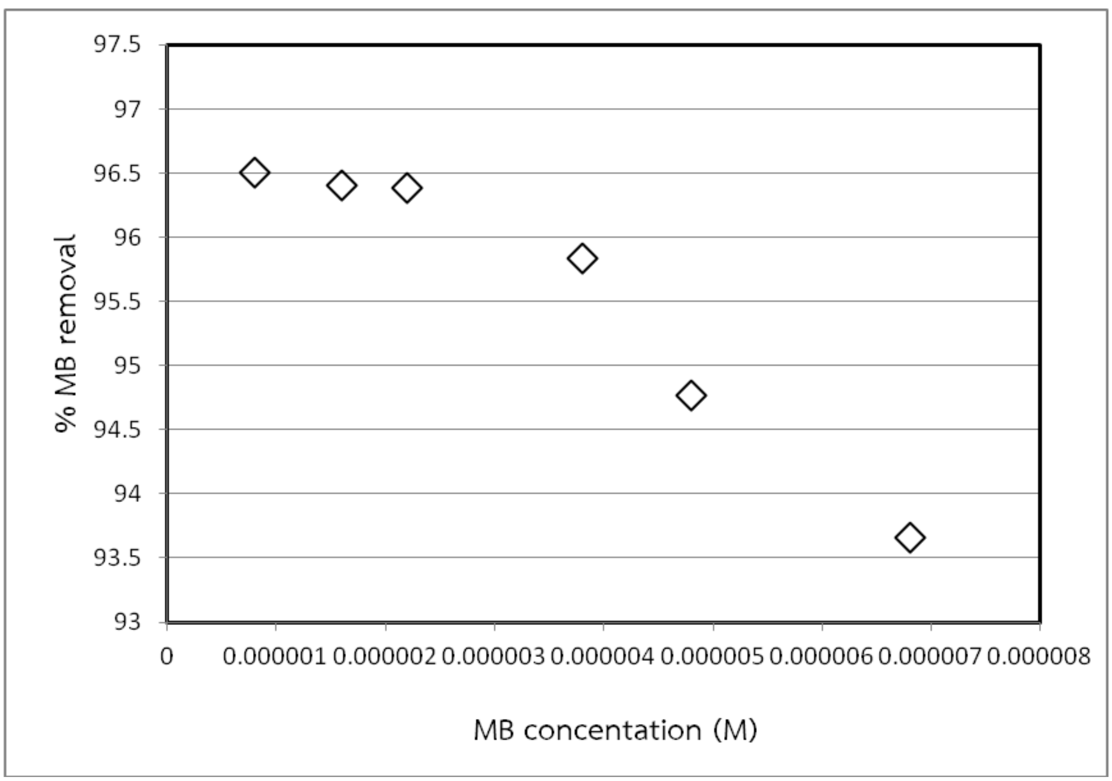


Figure 1 Effect of the initial MB concentration in MB adsorption onto neem leaf powder

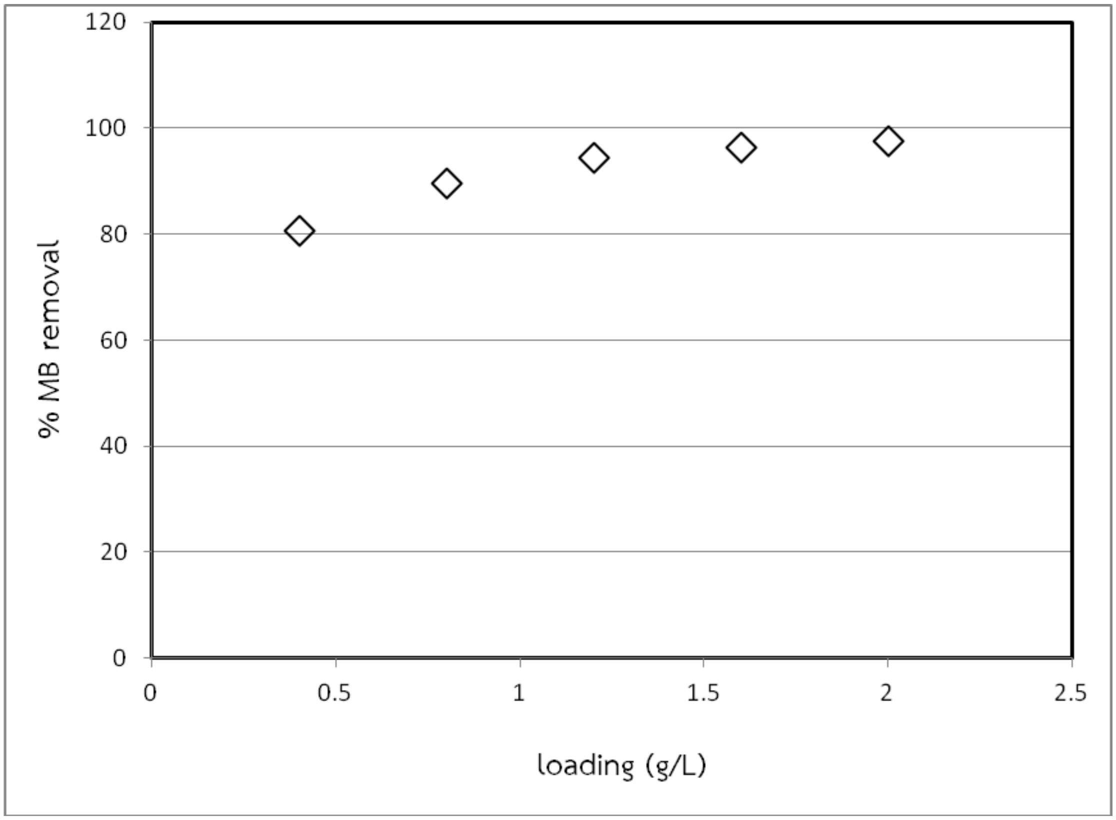


Figure 2. Effect of adsorbent content in MB adsorption onto neem leaf powder

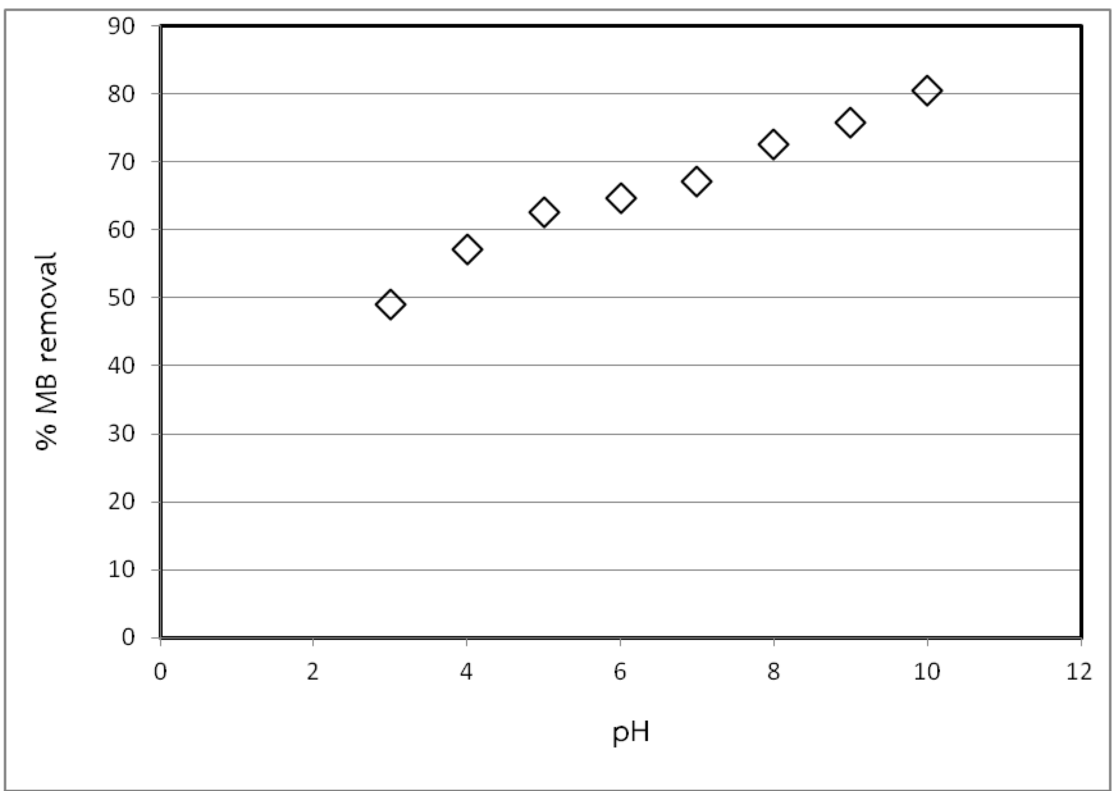


Figure 3. Effect of pH in MB adsorption onto neem leaf powder

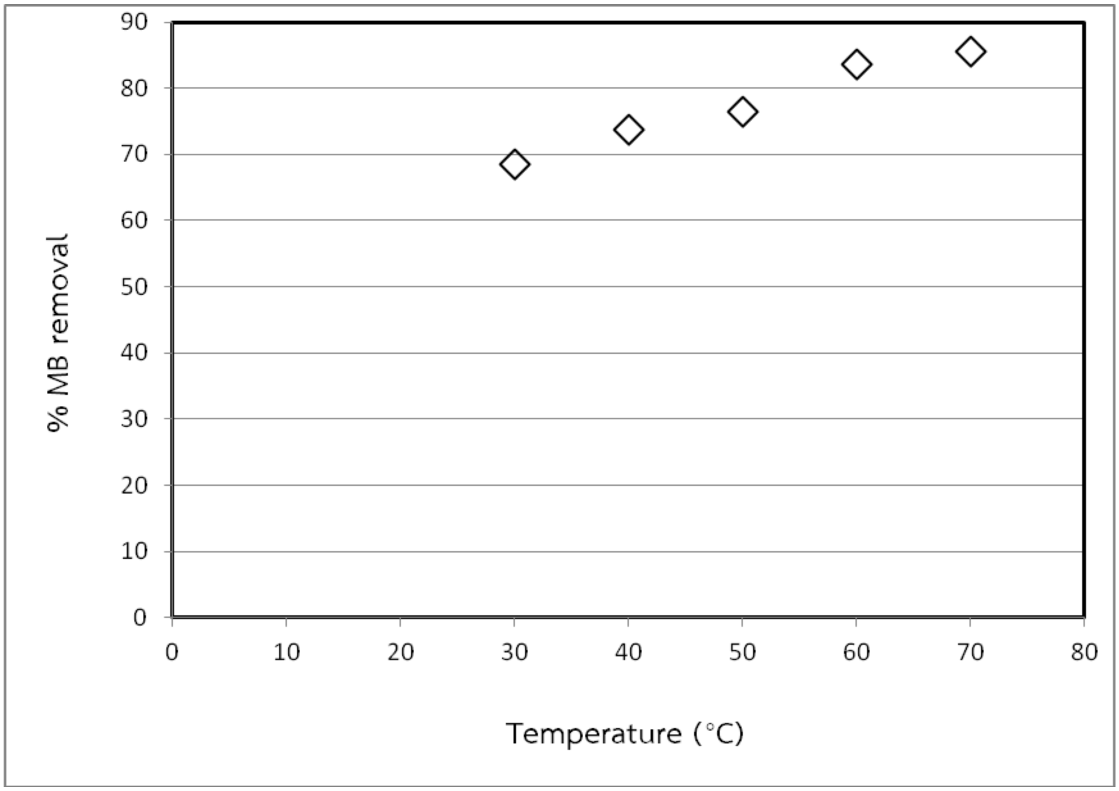
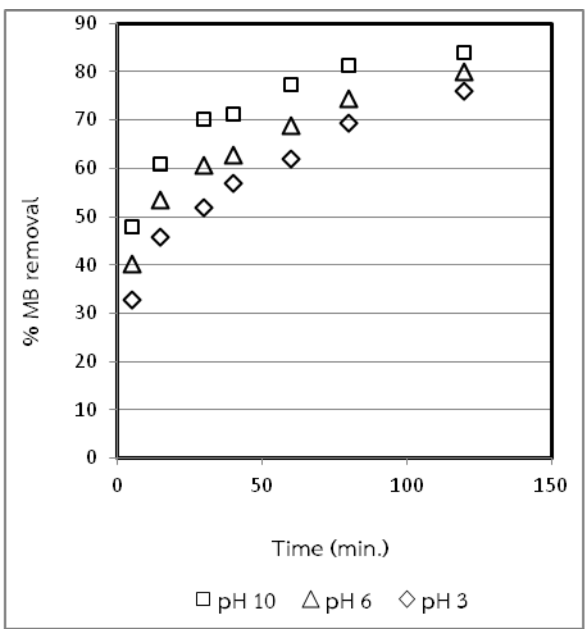
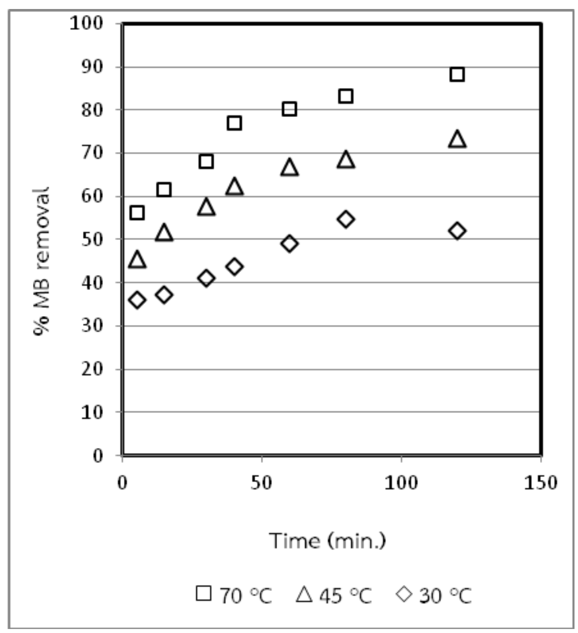


Figure 4. Effect of temperature in MB adsorption onto neem leaf powder





(a) **(b)**

Figure 5. Kinetic plot of the MB removal over neem leaf powder as (a) pH and (b) temperature dependence.

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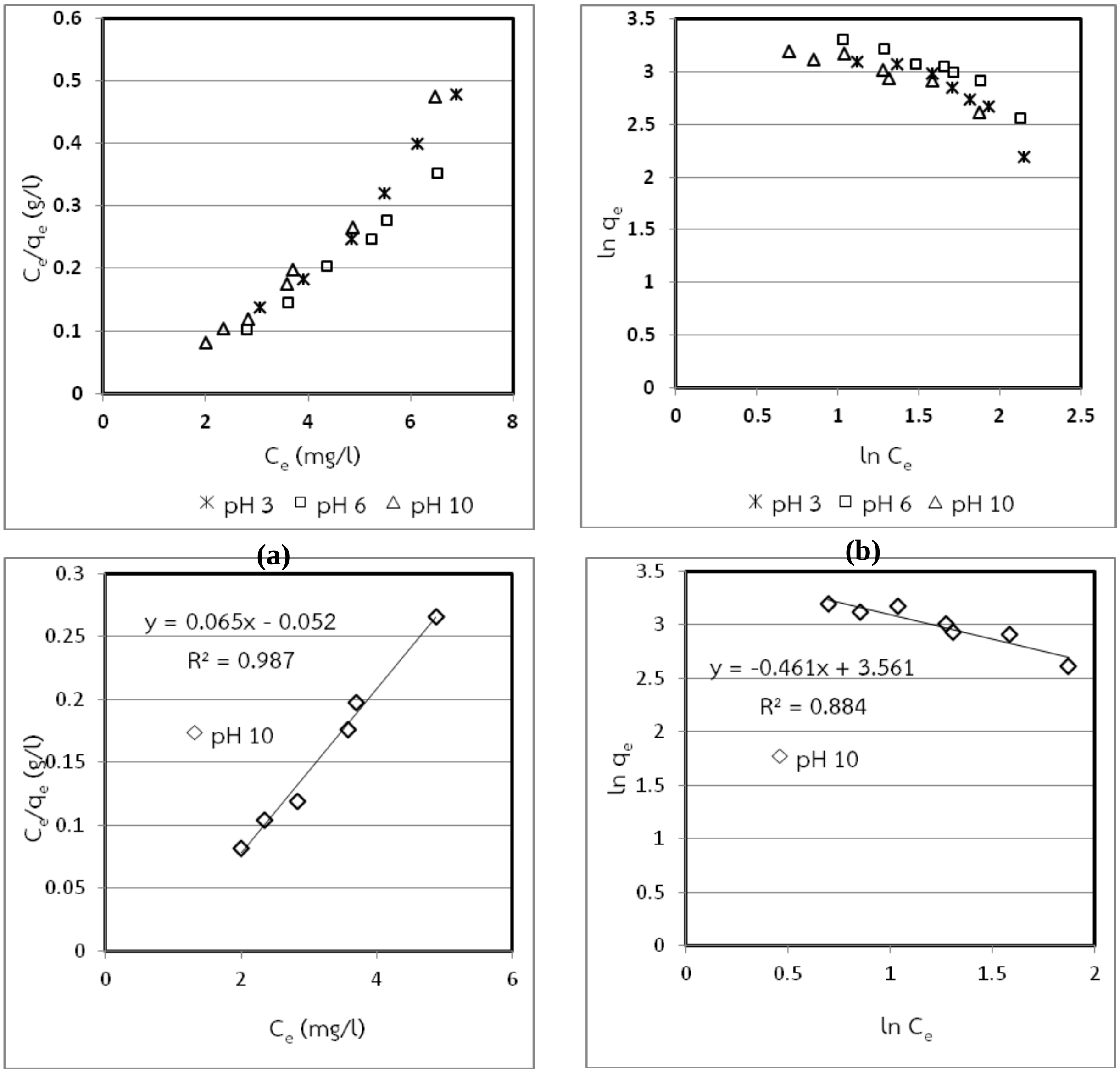

(c) (d)

Figure 6 The adsorption isotherm study of the MB removal, at MB concentration = 3.8 x 10^{-5} M and temperature 45 ˚C (a) Langmuir, (b) Freundlich adsorption isotherms over various pH, (c) Langmuir and (d) Freundlich adsorption isotherm at pH = 10.

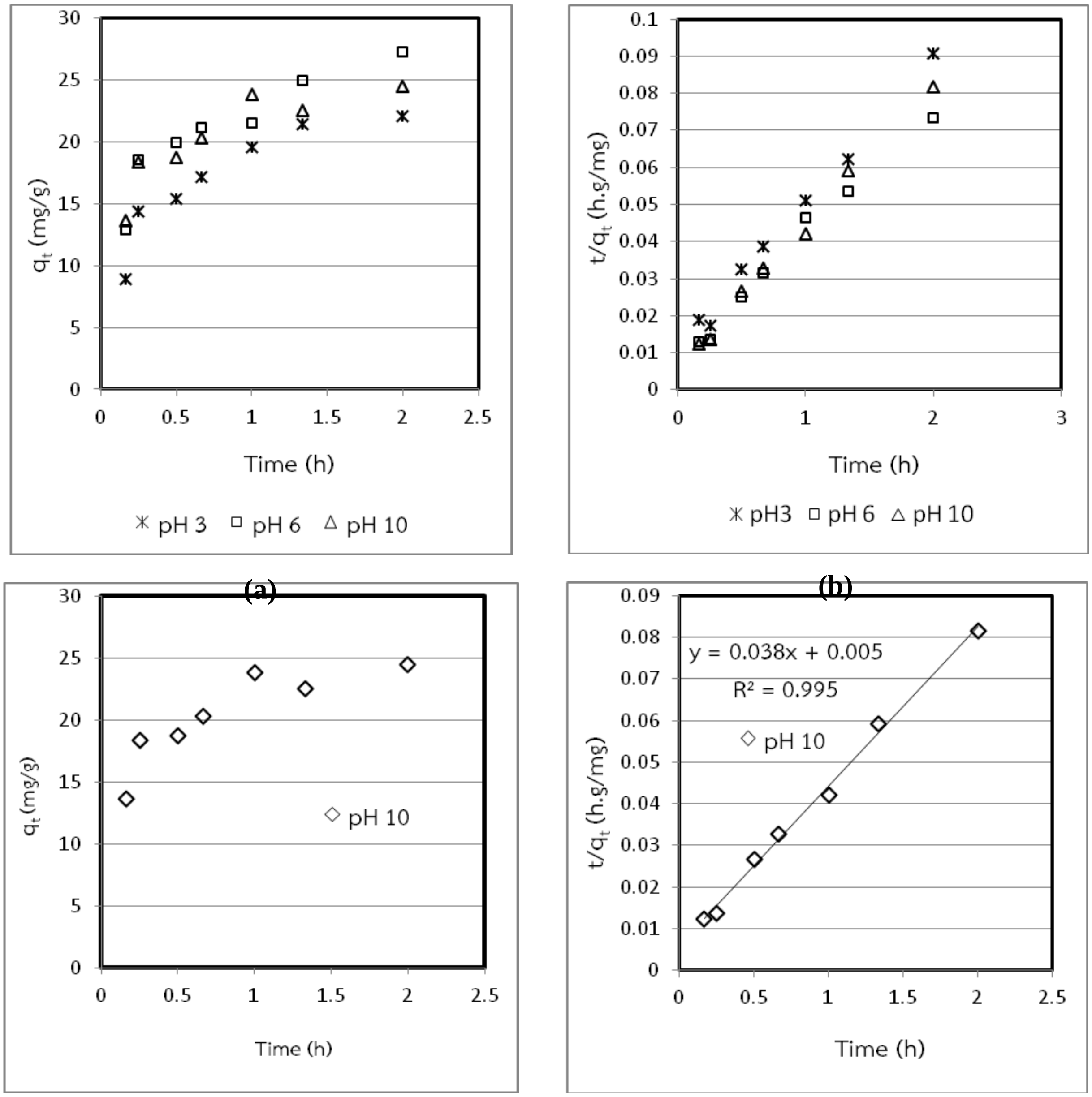

Figure 7 The kinetic study of the MB removal, at MB concentration = 3.8 x 10^{-5} M and temperature 45 °C (a) Pseudo-first order, (b) Pseudo-second order over various pH, (c) Pseudo-first order and (d) Pseudo-second order at pH = 10.

Table 1 Effect of pH in adsorption isotherm and kinetic model fitting

pH	R^2		R^2	
	Langmuir	Freundlich	Psuedo-1st order	Psuedo-2nd order
3	0.884	0.829	-	0.994
6	0.936	0.904	-	0.987
10	0.987	0.884	-	0.995

Table 2 Effect of Temperature in adsorption isotherm and kinetic model fitting

Temperature (°C)	R^2		R^2	
	Langmuir	Freundlich	Psuedo-1st order	Psuedo-2nd order
30	0.979	0.937	-	0.994
45	0.985	0.943	-	0.997
70	0.989	0.924	-	0.989

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Table 3 Comparison of adsorption capacity of MB on various natural adsorbent

Adsorbents	surface area (m^2/g)	pH	Temp. (°C)	Q_0 (mol/g)	Refs.
Pineapple leaf powder	5.236	7.5	24	8.88×10^{4}	[26]
Rice husk	NA	NA	20	8.36×10^{4}	[27]
Cotton waste	NA	NA	20	7.44×10^{4}	[27]
Peanut hull	72.35	5.0	20	1.82×10^{4}	[9]
Orange peel	22.1	7.2	30	5.51×10^{-5}	[28]
Neem leaf powder	NA	NA	27	2.35×10^{-5}	[13]
Neem leaf powder (yellow leaf)	NA	10	70	1.67 x 10	This study