# Basic Dye Removal from Synthetic Wastewater by Adsorption on Palm Ash

Abdulbari, A. Ahmad, Bassim H. Hameed[*] and N. Aziz

*School of Chemical Engineering, Engineering Campus, University Science Malaysia,*
*14300 Nibong Tebal, Penang, Malaysia*

## Abstract

Palm ash, an agriculture waste residue from palm oil industry in Malaysia, was investigated as a replacement for the current expensive activated carbon adsorbent for removing of basic blue 66 dye from synthetic wastewater. Adsorption equilibrium and kinetics of basic dye from aqueous solutions onto palm oil were studied in a batch system. Effects of the initial concentration (50 to 600 mg/l), contact time, pH (2-12) and temperature on adsorption rate were investigated. The experimental data were analyzed by the Langmuir, Freundlich and Redlich-Peterson models of adsorption. Equilibrium data fitted well Langmuir, Freundlich and Redlich-Peterson models in the range of 50-600 mg/l. The kinetic models pseudo first-order and pseudo-second-order were tested to investigate the adsorption mechanisms. It was shown that second-order kinetic equation could best describe the adsorption kinetics. The results indicate that the activated palm ash could be employed as a low cost alternative to commercial activated carbon.

*Keywords: Adsorption; Basic blue 66 dye; Palm ash; isotherms; Kinetic models*

## 1.0    Introduction

Dyes are used in large quantities in many industries including textile, leather, cosmetics, paper, printing, plastic, pharmaceuticals, food, etc. to color their products. Presently more than 9000 dyes are incorporated in color index belonging to various chemical application classes. The textile industry alone accounts for two thirds of the total dye stuff production, about 10–15% of the dyes used come out through the effluent [1]. Dyes and pigments represent one of the problematic groups; they are emitted into wastewaters from various industrial branches, mainly from the dye manufacturing and textile finishing. Dyes can be classified as anionic (direct, acid, and reactive dyes), cationic (basic dyes) and non-ionic (disperse dyes) [2]. Cationic dyes are applied to paper polyacrylonitrile, modified nylons, and modified polyesters.thier original use was for silk, wool, and tannin-mordanted cotton basic dyes are water-soluble and yield colored cations in solution. The principal chemical classes are diazahemicyanine, triarylmethane, cyanine, hemicyanine, thiazine, oxazine, and acridine. Some basic dyed show biological activity and are used in medicine as antiseptics [3]. The removal of textile dyes from wastewater is one of the most important environmental issues to be solved today. There are many methods to remove color effluents. Physical such as activated carbon adsorption, chemical methods, chemical oxidation, coagulation, flocculation, biological treatment, ozonation, electrolysis and photocatalysis have been developed for treating dye containing wastewater [4]. Activated carbon is the most widely used adsorbent for removal of color from textile effluents because it has a high capacity for

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[*] *Corresponding author: Fax: +604-594 1013, Email: Chbassim@eng.usm.my*

organic matter, extended surface area and micro pours structure but its use is limited due to its high cost. This cost problem has led to a search for the use of alternative cheap and efficient materials. Thus many researchers have tried to search for cheaper substitutes, which are relatively inexpensive, and at the same time with reasonable adsorptive capacity. These studies include use of coal [5], fly ash [6], activated clay [7], palm-fruit bunch [8], Bagasse pith [9], peat, bentonite, slag and fly ash [10], activated sludge [11], etc. Solid waste from palm oil industry is highly abundant in Malaysia because it is one of the largest palm oil producers in the world. This waste is usually used as fuel in palm oil mill factories. The ash is produced after the combustion of oil palm fibers and shell as boiler fuel for the steam generation for palm oil mill consumption. Therefore, Malaysia generates a lot of palm ash each year. Utilization of agriculture waste residues for wastewater treatment has the following advantages: 1) solid wastes are available abundantly at no or low cost; 2) disposal of the wastes is a series environmental problem in Malaysia which has extensive agricultural activities. Therefore, the objective of this work is to study the utilization of activated palm ash as adsorbent to remove basic blue 66 dye from aqueous solution.

## 2.0 Materials and method

### 2.1 Adsorbent: palm ash

The palm ash was provided by United Oil Palm Mill, Penang, Malaysia. It was washed doubly with water and oven dried at $110^{o}$C overnight and sieved through sieve No 100 (150 $\mu$ m). Forty grams of palm ash were activated by refluxing with 250ml of 40 wt% $H_2SO_4$ at $60^{o}$C for 4h in a round-bottom flask. The suspension was cooled in air and filtered off and then washed several times with double-distilled water and dried in an oven at $110^{o}$C for 2h prior to use.

### 2.2 Adsorbate: Basic blue dye

Basic blue (BB66) dye, having molecular formula of $C_{22}H_{28}Cl\ N_5O_2S.0.5ZnCl_2$ (mol. wt. 530.16), was chosen as adsorbate. The dye was supplied by Sigma-Aldrich (M) Sdn Bhd, Malaysia.
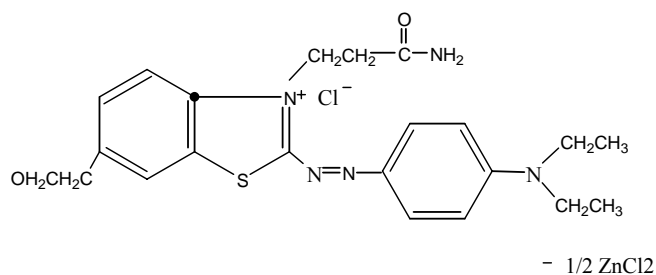


Figure 1        The chemical stricture of the dye

### 2.3 Characterization of adsorbent

Adsorption of $N_2$ at 77 K was performed to evaluate the adsorptive properties of the activated palm ash. The value of BET surface area and the average pore size measurement.is obtained from adsorption of $N_2$ at 77 K using Autosorb I, supplied by Quantachrome Corporation, USA. Prior to measurements, the powder samples were outgases under vacuum at $200^{o}$C for 3 hrs. The chemical composition of activated palm ash was determined using Rigaku RIX

3000 X-ray Fluorescence (XRF) spectrometer. The X-ray fluorescence was applied to identify the chemical composition and to determine the percentages of the chemical composition in the adsorbents.

## 2.4    Batch equilibrium studies

Adsorption isotherms were performed in a set of 43 Erlenmeyer flasks (250 ml), where solutions of dye (200 ml) with different initial concentrations (50-600 mg/l) were placed in these flasks. An equal mass of 0.3 g of particle size (150 $\mu$m) activated palm ash was added to dye solutions, and the mixture was kept in an isothermal shaker (30, 40 and 50°C) for 30, 40 hr to reach equilibrium as a solid-solution mixture. Similar procedure was followed for another set of Erlenmeyer flask containing the same dye concentration without activated palm ash to be used as a blank. The flasks were then removed from the shaker; and the final concentration of dye in the solution was measured at 615 nm using UV/Vis spectrophotometer (Shimadzu UV/Vis1601 Spectrophotometer, Japan). Each experiment was duplicated under identical conditions. The amount of dye on activated palm ash was calculated from the following equation

$$q_e = \frac{(C_o - C_e)V}{W} \tag{1}$$

where $q_e$ the amount of dye adsorbed at equilibrium in (mg/g), $C_o$ and $C_e$ (mg/l) are the liquid-phase concentrations of dye at initial and equilibrium, respectively. V is the volume of the solution (l), and W is the mass of dry adsorbent used (g).

The procedure of kinetic tests was basically identical to those of equilibrium tests. The aqueous samples were taken to preset time intervals and the concentrations of dye were similarly measured. The amount of adsorption at time t, $q_t$ (mg/g), was calculated by:

$$q_t = \frac{(C_o - C_t)V}{W} \tag{2}$$

where $q_t$ the amount of dye adsorbed at time t in (mg/g), $C_o$ and $C_t$ (mg/l) are the liquid-phase concentrations of dye at initial and any time t, respectively. V is the volume of the solution (l), and W is the mass of dry adsorbent used (g).

## 3.0    Results and Discussion

### 3.1    Characterization of palm ash

The BET specific surface area and average pore diameter of activated palm ash were found to be 5.356 m²/g and 2.263 nm, respectively. The chemical composition of activated palm ash was determined using Rigaku RIX 3000 X-ray Fluorescence (XRF) spectrometer. It was found to have the composition: $SiO_2$ 40%, $K_2O$ 12.1%, $CaO$ 10.0%, $Al_2O_3$ 6.1%, $MgO$ 6.4%, $P_2O_5$ 8.32%, C 5.4%, others 4.38% and its ignition loss 7.3%.The high oxides contents in activated palm ash give its structure credibility as a good adsorbent.

## 3.2    *Effects of initial concentration and contact time*

The adsorption of dye by activated palm ash increases as the initial dyes concentration increased as shown in Fig.2. The basic blue dye is adsorbed to achieve adsorption equilibrium in about 24 h. The time required to attain this state of equilibrium is termed equilibrium time, and the amount of dye adsorbed at the equilibrium time reflects the maximum adsorption capacity of the adsorbent under those operation conditions. At the beginning, the dye ions were adsorbed by the exterior surface of activated palm ash, the adsorption rate was fast.
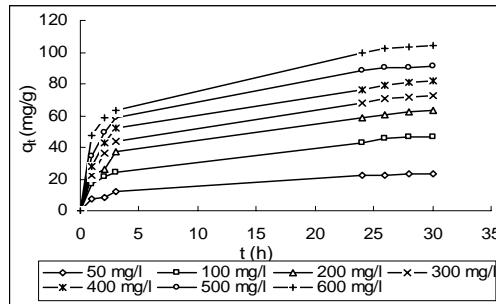


Figure 2    Adsorption of basic blue dye on activated palm ash vs. contact time at
different initial concentration at 30$^{o}$C

When the adsorption of the exterior surface reached saturation, the dye ions entered into the activated palm ash particle by the pore within the particle and were adsorbed by the interior surface of the particle. This phenomenon takes relatively long contact time. It can was observed for the adsorption of Basic Yellow 24 using dried activated sludge biomass. They reported that the uptake of the dye increased from 18 to 90 mg/g with increasing dye concentration from 50 to 300 mg/l [12].

## 3.3    *Effect of pH*

Fig.3. the removal of a basic blue dye by activated palm ash increase with pH increased from 2.0 to 12.0. It was suggested that the increase in sorption depended on the properties of the adsorbent surface and the dye structure. At higher pH more protons will be available and increasing electrostatic attractions between positive charged dyes and negatively charged adsorbent sites and causing an increase in dye adsorption. It is likely that positive charge develops on the surface of an adsorbent in an acidic medium, resulting in a higher adsorption at higher pH. If this is the case, the sorption of the cationic dye should decrease at a lower pH.
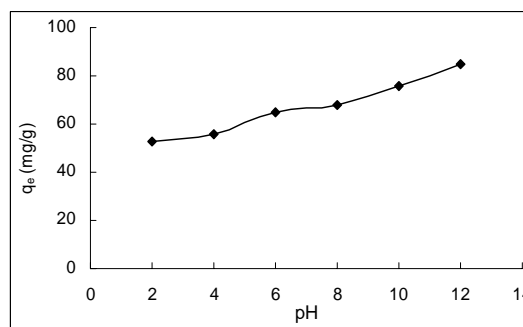


Figure 3        Effect of pH on the adsorption of basic blue dye onto activated palm ash

The amount adsorbed in the present work remained nearly constant in the pH range of 6.0 to 10.0, and therefore, the adsorption of basic blue on activated palm ash might be attributed to weak electrostatic interactions between the dye molecules and the solid surface. Similar results reported by [13] on adsorption of methyl violet onto perlite.

## 3.4    Effect of temperature

The adsorption studies were carried out at three different temperatures 30, 40 and 50°C, and the results of these experiments are presented in Fig.4 for basic blue. The adsorption capacity increases with the increasing temperature, indicating that the adsorption is an endothermic process Table 1.
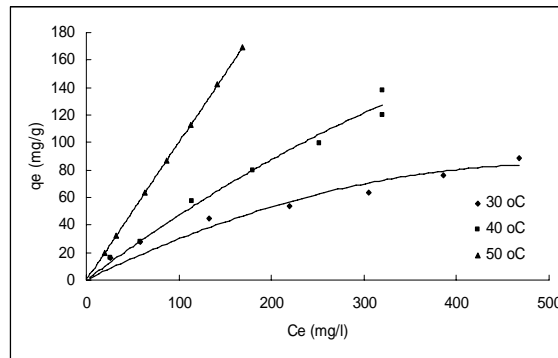


Figure 4     Adsorption isotherm for basic blue dye on activated palm ash at different
              temperatures

It was also observed that the maximum adsorption occurred at 50◦C. The order of the adsorption for basic blue was 30 < 40 < 50◦C. Since the adsorption is increasing at higher temperatures it is endothermic in nature. Similar unburned carbon as a low cost adsorbent for treatment of methylene blue containing wastewater [14].

## 3.5    Adsorption isotherms

### 3.5.1   Langmuir isotherm

The Lamngmuir equation, is given by

$$q_e = \frac{q_{max} K_L C_e}{\left(1 + K_L C_e\right)} \tag{3}$$

Where $q_e$ is the solid phase sorbate concentration in equilibrium (mg/g), $q_{max}$ is the maximum adsorption capacity corresponding to complete monolayer coverage on the surface (mg/g), $C_e$ is the concentration of adsorbate in solution (mg/l) and $K_L$ the Langmuir constant (l/mg). Eq. (3) can be rearranged to a linear form

$$\frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \frac{C_e}{q_{max}} \tag{4}$$

The constants can be evaluated from the intercept and the slope of the linear plot of experimental data of $C_e / q_e$ versus $C_e$ Fig.5. The fit is quite well for activated palm ash correlation coefficient $\geq 0.95$. Conformation of the experimental data into Langmuir isotherm model indicates the homogeneous nature of activated palm ash surface, i.e., each dye

molecule/ activated palm ash adsorption has equal adsorption activation energy and demonstrates the formation of monolayer coverage of dye molecule the outer surface of activated palm ash. Values of $q_{max}$ and were calculated and given in Table 1.
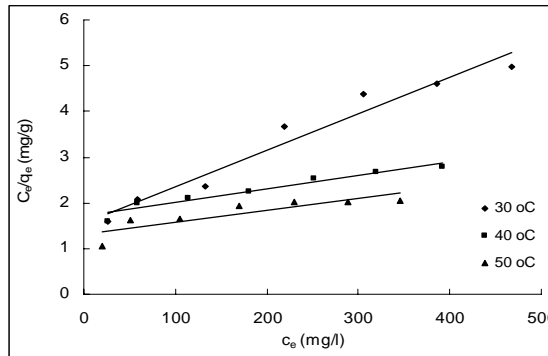


Figure 5    Linearized Langmuir isotherm for basic blue dye adsorption by activated palm ash

The essential characteristics of the Langmuir equation can be expressed in term of a dimensionless separation factor, $R_L$, defined as [15] and [16].

$$R_L = \frac{1}{\left(1 + K_L C_o\right)}$$  (5)

where, $C_o$ is the highest initial solute concentration and $k_L$ is the Longmuir's adsorption constant (l/mg). The values of $R_L$ were in the range of 0 to 1 (0.11-0.34) at different temperature confirm the favorable uptake of the basic blue process Table1.

### 3.5.2   Freundlich isotherm

The Freundlich equation is an empirical equation employed to describe heterogeneous systems, in which it is characterized by the heterogeneity factor 1/n. Hence, the empirical equation can be written

$$q_e = K_F C_e^{1/n}$$  (6)

Where $q_e$ solid phase sorbate concentration in equilibrium (mg/g), $C_e$ is liquid phase sorbet concentration in equilibrium (mg/l), $K_F$ Freundlich constant (mg/g) (l/mg) $^{1/n}$ and 1/n is heterogeneity factor. A linear form of the Freundlich expression can be obtained by taking logarithms of equation (6)

$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$$  (7)

Therefore, a plot of $\ln q_e$ versus $\ln C_e$ Fig. 6 enables the constant $K_F$ and exponent 1/n to be determined. The Freundlich isotherm describes reversible adsorption and is not restricted to the formation of the monolayer.
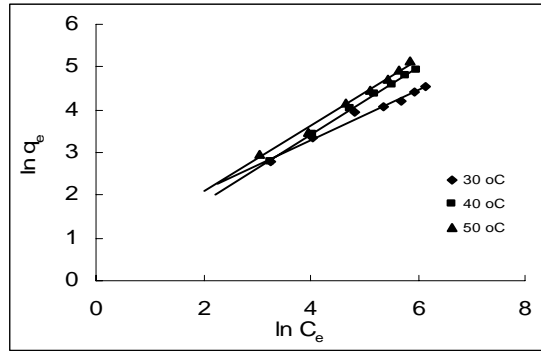
Figure 6    Linearized Freundlich isotherm for basic blue dye adsorption by activated palm ash

The Freundlich equation predicts that the dye concentration on the adsorbent will increase so long as there is an increased in the dye concentration in the liquid. It is clear from Table 1 that the value of the Freundlich exponent n was greater one (n >1) which represents favorable adsorption condition [17].

### 3.5.3   Redlich–Peterson (R–P) isotherm

The R–P equation incorporates three parameters into an empirical isotherm, and therefore, can be applied either in homogenous or heterogeneous systems due to its high versatility. The R–P equation is:

$$q_e = \frac{K_R C_e}{1 + a_R C_e^{\beta}} \tag{8}$$

where $K_R$ is R–P isotherm constant (l/g), $a_R$ is also a constant $(l/mg)^{1/\beta}$ and $\beta$ is the exponent which lies between 0 and 1. For $\beta = 1$, equation (8) reduces to Langmuir equation and for $\beta = 0$, it reduces to Henry's equation. Equation (8) can be converted to a linear form by taking logarithm

$$\ln(K_R \frac{C_e}{q_e} - 1) = \ln a_R + \beta \ln C_e \tag{9}$$

A plot of $\ln(K_R \frac{c_e}{q_e} - 1)$ versus $\ln c_e$ Fig.7 enables the constants $a_R$, $K_R$, $\beta$ and the correlation coefficients $R^2$, to be determined. Equilibrium data fitted well to Redlich–Peterson model ($R^2 > 0.95$). The R–P isotherm constants $a_R$, $K_R$ and $\beta$ and the correlation coefficients, $R^2$ are listed in Table 1.

The result shows from Table 1 the experimental data fitted well, Langmuir, Freundlich and Redlich Peterson models with high values of correlation coefficient $R^2 \geq 0.95$.
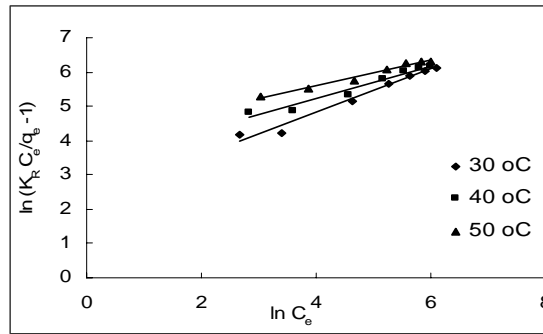
Figure 7    Linearized Redlich-Peterson isotherm for basic blue dye adsorption by
activated palm ash

Table 1    Langmuir, Freundlich nad Redlich–Peterson constants parameters for adsorption of
basic blue 66 dye on activated palm oil

| Temp. °C | Langumuir isotherm model | | | | Freundlich isotherm model | | | Redlich–Peterson model | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | $q_{max}$ (mg/g) | $K_L$ (l /mg) $\times 10^{-2}$ | $R^2$ | $R_L$ | $K_F$ (mg/g) (l/mg)$^{1/n}$ | n | $R^2$ | $K_R$ (l/g) | $a_R$ (l/mg) | β | $R^2$ |
| 30 | 108.7 | 0. 13 | 0.95 | 0.11 | 10.94 | 2.81 | 0.97 | 135.81 | 10.01 | 0.63 | 0.96 |
| 40 | 233.33 | 0. 83 | 0.96 | 0.18 | 5.61 | 1.91 | 0.96 | 281.76 | 27.85 | 0.47 | 0.95 |
| 50 | 284.61 | 0. 47 | 0.96 | 0.34 | 3.09 | 1.62 | 0.99 | 319.31 | 58.55 | 0.38 | 0.97 |

## 3.6    Adsorption kinetic study

To investigate the mechanism of basic blue dye adsorption on activated palm ash, the pseudo first-order and pseudo second-order equations were used to test the experimental data of initial concentration, pH 7 and 30°C.

### 3.6.1    First-order kinetic model

A simple kinetic analysis of adsorption is the pseudo first-order equation in the form

$$\frac{dq_t}{dt} = k_1 (q_e - q_t) \tag{10}$$

Where $k_1$ is the rate constant of pseudo first-order adsorption and q represents adsorption capacity (i.e. the amount of adsorption corresponding to monolayer coverage. After definite integration by applying the initial conditions q = 0 at t = 0 and q = q, equation (10) becomes

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \tag{11}$$

where $q_e$ and $q_t$ are the amount of eosin adsorbed (mg/g) at equilibrium and at any time $t$, $k_1$ is the rate constant (min$^{-1}$). The plot of log ($q_e$–$q_t$) versus $t$ gives a straight line for the first-order adsorption kinetics as shown in Fig.8. The value of the first-order rate constant $k_1$ is obtained from the slope of the straight line.
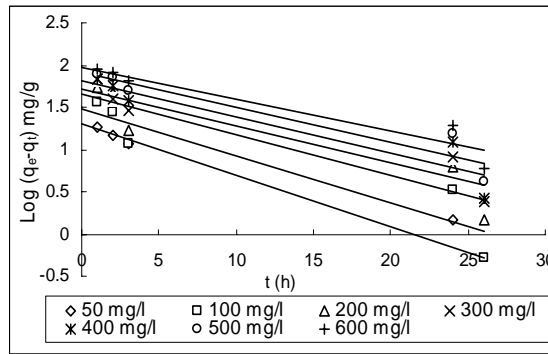
Figure 8        Pseudo-first-order kinetics for adsorption of basic blue dye on activated palm ash

The $k_1$ values, the correlation coefficients, $R^2$, and the predicted and experimental $q_e$ values for adsorbent /dye combinations are given in Table2.

### 3.6.2   Second-order kinetic model

The pseudo-second-order model can be represented in the following form

$$\frac{dq_t}{dt} = k_2 \left( q_e - q_t \right)^2 \tag{12}$$

where $k_2$ is the rate constant of pseudo second-order adsorption. Integration Eq. (12) and applying the initial conditions, we have

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^{\,2}} + \frac{1}{q_e} t \tag{13}$$

In Eq. (13) $k_2$ (h g/mg) is the rate constant for the pseudo second-order adsorption kinetics. The slope of the plot $t/q_t$ vs. t gives the value of $q_e$, and from the intercept $k_2$ can be calculated Fig.9.
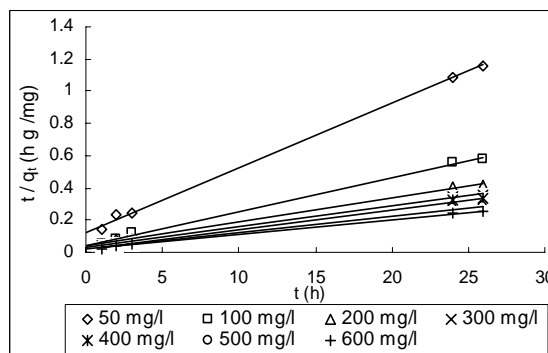


Figure 9        Pseudo-second-order kinetics for adsorption of basic blue on activated palm ash

Shows the pseudo-second order plot for basic onto activated palm ash. The pseudo-second order rate constant $k_2$, the calculated $q_e$ value and the corresponding linear regression correlation coefficient values $R^2$ are given in Table 2. At all initial dye concentrations, the linear regression correlation coefficient $R^2$ values were higher $R^2 \geq 0.95$. The higher $R^2$ values confirm that the sorption data are well represented by pseudo-second order kinetics and the supports the assumption behind the model that the sorption is due to chemisorption [18].

Similar kinetic results have also been reported for the adsorption of Congo red onto calcium-rich fly ash [4].

Table 2      Pseudo-first, second-order and normalized deviation adsorption rate constants and the calculated and experimental $q_e$ values for adsorption of basic blue dye on activated palm ash

| $C_o$ (mg/l) | $q_{e\ exp.}$ (mg/g) | Pseudo first order kinetic model | | | | Pseudo second order kinetic model | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | $q_{e\ cal.}$ (mg/g) | $k_1$ (h)$^{-1}$ | SD (%) | $R^2$ | $q_{e\ cal}$ (mg/g) | $k_2$ (h g/mg) | SD (%) | $R^2$ |
| 50 | 23.62 | 16.13 | 0.09 | 7.94 | 0.91 | 25.64 | 0.011 | 3.49 | 0.98 |
| 100 | 46.67 | 42.85 | 0.046 | 3.34 | 0.65 | 50.7 | 0.0063 | 3.22 | 0.98 |
| 200 | 64.08 | 50.11 | 0.032 | 8.9 | 0.85 | 67.11 | 0.0053 | 1.93 | 0.98 |
| 300 | 60.83 | 58.88 | 0.029 | 3.31 | 0.89 | 74.62 | 0.0056 | 2.25 | 0.95 |
| 400 | 75.23 | 69.18 | 0.026 | 3.28 | 0.85 | 74.07 | 0.0067 | 0.63 | 0.99 |
| 500 | 91.86 | 79.43 | 0.021 | 4.15 | 0.92 | 91.74 | 0.0062 | 0.05 | 0.99 |
| 600 | 104.23 | 93.32 | 0.020 | 1.27 | 0.75 | 106.38 | 0.0043 | 0.84 | 0.97 |

### 3.6.3   Test of kinetic models

Normalized standard deviation, SD (%), is used to find the most applicable that model could describe the kinetic study of adsorption of basic blue dye on activated palm ash. The normalized standard deviation SD% was calculated using the following equation

$$SD(\%) = 100 \left\{ \sum \left[ (q_{e,\exp} - q_{e,cal}) / q_{e,\exp} \right]^2 / (N-1) \right\}^{1/2} \tag{14}$$

Where n is the number of data points, $q_{t,\exp}$ is the experimental values and $q_{e,cal}$ is the calculated value by both models. Based on the values of SD (%) given in Table 2, it is clear pseudo-second order equation was better in describing the adsorption kinetics of basic blue dye using activated palm ash.

## 4.0     Conclusion

The results show that activated palm ash had considerable potential for the removal of basic blue dye from aqueous solution. The adsorption capacity of activated palm ash was found at different temperature. The experimental data fitted well, Langmuir, Freundlich and Redlich Peterson models with high values of correlation coefficient $R^2 \geq 0.95$. The $R_L$ values showed that activated palm ash was favorable for the adsorption of basic dye. The pseudo-second-order kinetic model fits very well with the dynamical adsorption behavior of basic blue dye. It may be concluded that activated palm ash may be used for elimination of textile dyes from wastewater, activated palm ash is a low natural abundant adsorbent material and it may be alternative to more costly adsorbent materials.

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## Nomenclature

| | |
|---|---|
| $K_L$ | Adsorption energy constant of Langmuir adsorption isotherm, l/mg |
| $C_e$ | Equilibrium liquid phase concentration, mg/l |
| $C_o$ | Initial liquid phase concentration, mg/l |
| $q_{mon}$ | The maximum surface coverage (formation of monolayer) of sorbent, mg/g |
| $K_F$ | Freundlich isotherm constant related to adsorption capacity, $(m/g)(l/mg)^{1/n}$ |
| $k_1$ | Rate constant of first-order adsorption, per hour |
| $k_2$ | Rate constant of second-order adsorption, (h g/mg) |
| n | Freundlich isotherm constant related to adsorption intensity |
| $q_e$ | Equilibrium solid phase adsorbate concentration, mg/g |
| $q_t$ | Amount of adsorption at time t, mg/g |
| $K_R$ | R–P isotherm constant (l/g) |
| $a_R$ | Constant $(l/mg)^{1/\beta}$ |
| $\beta$ | Exponent which lies between 0 and 1 |
| $R^2$ | Correlation coefficient |
| $R_L$ | Dimensionless separation factor |
| V | Volume of solution, l |
| W | Mass of adsorbent, g |
| SD (%) | Normalized standard deviation |

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