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Mangifera indica Leaves Powder for the Removal of Rhodamine B.: Isotherm and Kinetic Studies

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ABSTRACT

Adsorption of Rhodamine B (RB) dye onto a non-conventional plant biosorbent, *Mangifera indica* (mango tree) leaves powder (MLP), was investigated in batch adsorption experiments. Process parameters which include initial dye concentration, contact time and adsorbent dosage were varied in order to evaluate their influence on the adsorption process. Maximum adsorption efficiency (87.00%) was obtained at 75 min. The experimental data fitted well to pseudo-second-order kinetic model and the intra-particle diffusion model, however, diffusion was not only the rate-controlling step. The isotherm modeling suggested that Freundlich isotherm best described the process, with R^2 value of 0.972, which is an indication of presence of multi-layer adsorption. The n_f value (1.073) shows the adsorption process is favourable, indicating a physisorption. FT-IR results showed the shifting of peak down field and up field which depicts an interaction between the adsorbent and the dye molecules. The results showed that *Mangifera indica* leaves powder can be used as a low-cost adsorbent for the removal of RB from aqueous media.

Keywords: Adsorption, Isotherms, Kinetics, *Mangifera indica*, Rhodamine B.

INTRODUCTION

Many industries such as textile, paper, rubber, plastics, paints, printing, and leather discharge colored effluents indiscriminately, which cause pollution in receiving water bodies. The problem is more severe for textile industries because they are major consumers of dyes, most of which are toxic and non-biodegradable. The huge volume of generated wastewater, when released into the environment, causes adverse effects on aquatic ecosystem and human life. The coloured water is not only aesthetically objectionable but depletes sunlight penetration which reduces the photosynthetic activity in aquatic plants impeding their growth. Many dyes may cause allergic dermatitis, skin irritation, dysfunction of kidney, liver, brain, reproductive and central nervous system. Besides, some are suspected carcinogens and mutagens (Khan *et al.*, 2009).

Rhodamine B (RB) is used mostly in paper printing, textile dyeing, and leather industries. It is carcinogenic, and may cause irritation, redness and pain in eyes and skin. When inhaled, it causes irritation in respiratory tract with symptoms of coughing, sore throat, laboured breathing and chest pain. If swallowed, RB is likely to cause irritation to the gastrointestinal tract. Therefore, it is imperative that proper treatment of the dye effluents for colour removal is carried out before its discharge (Gupta *et al.*, 2003).

Numerous methods exist for the treatment of textile wastewater with varying degree of success (Mondal, 2008). Amongst these, the adsorption technique using low cost adsorbents derived from various natural, agricultural, and industrial wastes (Gurusamy *et al.*, 2002, Sepúlveda *et al.*, 2004, Allen and Koumanova, 2005, Crini, 2006, Mokhtar *et al.*, 2008, Papita, 2010, Ali, 2010, Ibrahim *et al.*, 2015, Sushmita *et al.*, 2015) are most widely employed in wastewater treatment. In this research, *Mangifera indica* (mango tree) leaves powder (MLP) was used as an adsorbent for the removal of Rhodamine B (RB).

MATERIALS AND METHODS

Adsorbent Preparation

The adsorbent for this work is *Mangifera indica* leaves (mango tree leaves, MLP) collected from local mango tree within premises of Bayero University, Kano. The leaves were washed severally with distilled water to remove dirty and dried at 80 °C in an oven for 4 hrs. The dried leaves were crushed into powder, and boiled with distilled water at 100 °C for 4 hrs to remove the colour. It was then filtered, dried at 80 °C in oven for 4hrs and sieved to different particle sizes (10 µm, 20 µm and 30 µm) and stored separately in air tight containers for further use (Jimoh and Ibrahim, 2017).

Batch Adsorption Experiment

The influence of variables such as contact time, initial dye concentration and adsorbent dosage were all investigated in batch mode. In each experiment, 100 ml of RB solution in a 120 ml bottle was agitated and stirred at 300 rpm along with variation of adsorbent dose from 0.1-0.7 g at an initial dye concentration of 10-100 mg/L, initial pH of the dye and at constant temperature of 30 ± 2 °C. The mixture was then centrifuged and the residual concentrations of the dyes were determined from the supernatant using UV-Visible spectrophotometer (Model Hitachi 2800) at a predetermined λ_{max} of 546 nm. The experimental data obtained at various times (5-120 min) and concentrations were fitted to different models to evaluate and calculate the kinetic and isotherm parameters (Jimoh and Ibrahim, 2018).

The removal efficiency and adsorption capacity of the adsorbent were calculated from equations (1) and (2) respectively.

$$R(\%) = \frac{(C_0 - C_t)}{C_0} X\ 100 \quad (1)$$

$$q_e = \frac{(C_0 - C_t)V}{m} \quad (2)$$

Where C_0 and C_t (mg/L) are the initial and final concentrations, m (g) is the adsorbent dose, and V (L) is the volume of the dye solution (Jimoh and Ibrahim, 2018).

Adsorption Isotherm Studies

Three isotherm models namely Langmuir (Langmuir, 1918), Dubinin - Radushkevich (Vijayaraghavan, 2006) and Freundlich (Freundlich, 1906) were used to test the experimental data for the adsorption of RB onto the adsorbent at 298 K.

Adsorption Kinetics

In order to describe the adsorption kinetics for RB adsorption on MLP, pseudo first-order (Lagergren, 1898), pseudo second-order (Ho *et al.*, 2000), Elovich Model (Chien and Clayton, 1980), Intra-particle diffusion (Weber and Morris, 1963) and Bangham's model (Yakout and Elsherif, 2010) were tested with the experimental data (equations 3 -7).

Pseudo first –order equation:

$$\ln(q_e - q_t) = lnq_e - k_1 t \quad (3)$$

Pseudo second-order equation:

$$t/q_e = 1/k_2 q_e^2 + t/q_e \quad (4)$$

Elovich model equation:

$$q_t = 1/\beta \ln(\alpha\beta) + 1/\beta\, lnt \quad (5)$$

Where q_e = amount of adsorbate adsorbed at equilibrium (mg/g), k_1 = Adsorption rate constant for first-order (min^{-1}), k_2 = Adsorption rate constant for second-order (g. $mg^{-1}min^{-1}$), α = Initial adsorption rate (mg. g^{-1} min^{-1}), and β = desorption constant (g. mg^{-1}) which is related to the extent of surface coverage.

Intra-Particle Diffusion

$$q_t = k_i t^{1/2} + C \quad (6)$$

Where C = intercept and k_i is the intra-particle diffusion rate constant (mg g^{-1} $min^{-1/2}$).

Bangham's model

$$\log\left(log\left[\frac{C_i}{C_i - C_s q_t}\right]\right) = \log\left[\frac{k_b C_s}{2.303V}\right] + \propto logt \quad (7)$$

Where Cs is the weight of adsorbent used per liter of solution (g/L), V is the volume of the dye solution used, α (< 1) and kb (mg^{-1}) are Bangham's equation parameters.

RESULTS AND DISCUSSION

Effect of Contact Time

The influence of contact (agitation) time on the percentage removal of RB by MLP was investigated (Figure 1). It is seen that the amount of adsorption increased with increasing the contact time. Maximum adsorption was observed after 75 min, beyond which there was almost no further increase in the adsorption. This is obviously due to the fact that a large number of vacant adsorptive surface sites are available for adsorption during the initial stage, and after a lapse of time, the remaining vacant surface sites are difficult to be occupied due to repulsive forces between the solute molecules on the solid and bulk phases (Jimoh and Ibrahim, 2017). This time was therefore considered as the equilibrium contact time.

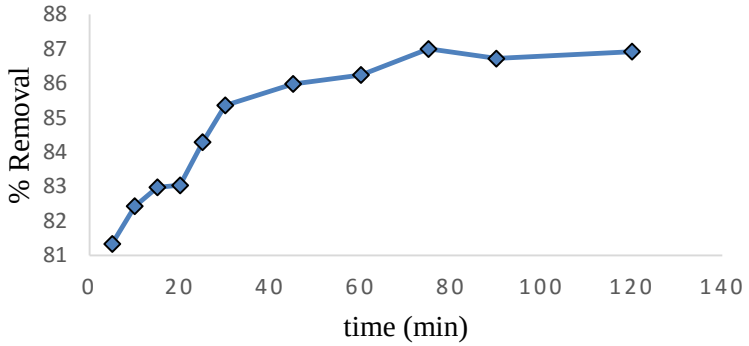

Figure 1: Effect of Contact Time on the Adsorption of RB by MLP

Effect of Adsorbent Dosage

The influence of MLP dosage on the adsorption capacity of RB is depicted in Figure. 2. As the adsorbent dosage increased from 0.1 to 0.7 g, the RB removal rate increased from 86.49% to 91.98%, this is because an increase in adsorbent dosage causes an increase in the available number of active binding sites on the surface of adsorbent (Israa and Hilal 2015). When the adsorbent dosage was 0.3 was 90.23%, an increase in adsorbent dosage result in a little difference in percentage removal, which indicates that adsorbent dosage had little influence on the adsorption of RB. Therefore, the adsorbent dosage, 0.4 g, was considered as the optimum dosage. Similar result was reported by Wang *et al* (2013) in their study of adsorption of RB from aqueous solution using heat-activated sepiolite.

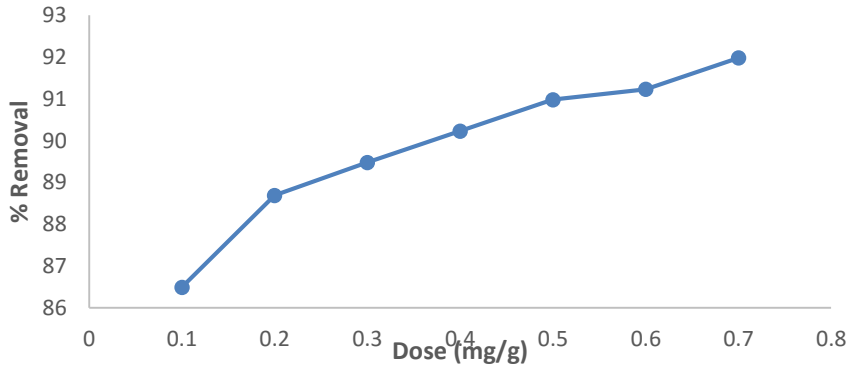

Figure. 2: Effect of Adsorbent Dosage on the Adsorption of RB by MLP

Adsorption Isotherm

In order to determine the adsorption capacity and potential for selecting the adsorbent for the removal of dye molecule, the study of adsorption isotherm is essential in selecting the adsorbent for the removal of dye molecules. The adsorption data obtained were analyzed in the light of Langmuir, Freundlich and Dubinin-Radushkevich isotherms.

Freundlich Isotherm

Batch isotherm data were fitted to the linear form of the Freundlich isotherm according to Eq. (8):

$$\log q_e = logK_L + \frac{1}{n} logC_e \qquad (8)$$

Where q_e is the amount of dye adsorbed per unit weight of the adsorbent (mg/g) and C_e is the equilibrium concentration (mg/L) of RB in solution. The fit of the data imply that the intercept K, is roughly an indication of the adsorption capacity and the slope 1/n, of the adsorption intensity, the constant incorporating all the factors affecting the adsorption process. Values of K and 1/n are calculated from the plot of $\log q_e$ against $\log C_e$ and the data are presented in Table 1. The linearity of the plot, R^2 = 0.972, shows the applicability of the Freundlich isotherm in describing the adsorption process. The value of n for the system was calculated and found to be 1.073 suggesting that the adsorption of RB by MLP may be governed by physisorption. The present findings are in good agreement with the finding of Treybal (1980) who mathematically evaluated values of n for a number of mass transfer operations of systems and reported that values of n between 1 and 10 would represent beneficial adsorption.

Langmuir Isotherm

The linear form of the Langmuir isotherm was applied in the form of Eq. (9):

$$\frac{C_e}{q_e} = \frac{1}{qb} + \frac{C_e}{q} \qquad (9)$$

Where q and b are Langmuir constants indicating the adsorption capacity and energy of adsorption respectively. C_e and q_e have their usual meaning. The values of q and b are obtained from the plot of C_e/q_e versus C_e and presented in Table 1. The essential characteristics of the Langmuir isotherm can be expressed by a dimensionless constant called the separation factor R_L:

$$R_L = \frac{1}{1+bC_0} \quad (10)$$

R_L values indicate the adsorption to be unfavourable when $R_L > 1$, linear when $R_L = 1$, favourable when $0 < R_L < 1$, and irreversible when $R_L = 0$. From Table 1, it can be seen that the value of R_L is 0.019 which indicated that the adsorption process is favourable.

Dubinin - Radushkevich

The linear expression of Dubinin - Radushkevich isotherm model is given by equation 11:

$$\ln q_e = \ln q_m - \beta\varepsilon^2 \quad (11)$$

Where q_e: amount of dye adsorbed at equilibrium (mg/g), β: D-R isotherm constant (mol^2/kJ^2) associated with mean free adsorption energy of adsorbate molecules per mole as they are transferred from solution media onto the adsorbent surface. q_m: theoretical isotherm saturation capacity (mg/g), ε: Polanyi potential (kJ/mol). The values of β and q_m are gotten from the plot of $\log q_e$ against ε^2 and presented in Table 1. Expression of Polanyi potential is also given by:

$$\varepsilon = RT \ln\left(1 + {}^1/_{C_e}\right) \quad (12)$$

The constant β gives an idea about the mean free energy E (kJ/mol) of adsorption per molecule of the adsorbate when it is transferred to the surface of the solid from infinite distance in the solution and can be calculated using equation 13:

$$E = \frac{1}{(2\beta)^{1/2}} \quad (13)$$

Based on this mean free energy, the process can be classified has been physisorption or chemisorption. If the energy of activation is < 8 kJ/mol, the adsorption is physisorption and if the energy of activation is 8–16 kJ/mol, the adsorption is chemisorption in nature (Jimoh and Ibrahim, 2018). As seen in Table 1, the value of E is 0.912 kJ/mol, which also signified that the adsorption process may be governed by physisorption.

Table 1: Isotherm Parameter for the Adsorption of Rhodamine B by Mango Leaves Powder

Model	Parameter	Values
Langmuir		
	q (mg/g)	0.119
	K_L (L/mg)	4219.409
	R^2	0.069
Freundlich		
	n	1.009
	K_F ((mg/g)/(L/mg)$^{1/n}$	8.260
	R^2	0.972
Dubinin -Radushkevich		
	B (mol^2kJ^{-2})	6 x 10^{-7}
	q_m (mg/g)	6.138
	E ($kJmol^{-1}$)	0.912
	R^2	0.866

Kinetic Models

The studies of adsorption equilibrium are important in determining the effectiveness of adsorption; however, it is also necessary to identify the types of adsorption mechanism in a given system. In this study, four different models were used to predict the adsorption kinetics of RB adsorption on MLP (pseudo-first-order, pseudo-second-order, Elovich, Intra-particle diffusion and Bangham's models).

Pseudo-First-Order

In the pseudo-first-order rate expression, equation 3, the slope and intercept values of plot of log (q_e - q_t) against t, was used to determine pseudo first order rate constant (k_1) and theoretical amount of dye molecule adsorbed per unit mass of adsorbent $q_{e(the)}$, Table 2. R^2 values did not appears linear at initial stage of adsorption also $q_{e(exp)}$ values differ from the corresponding $q_{e(cal)}$ calculated from the plots showing that adsorption of RB on these adsorbent is not a first order kinetics.

Pseudo-Second-Order

Based on equilibrium adsorption, the pseudo-second-order kinetic equation, equation 4, slope and intercept values of plot of t/q_e against t were used to calculate k_2 and $q_{e(cal)}$ respectively. The values of model parameters (K_2 and q_e) at an initial concentration of 10mg/L, are given in Table 2, It can be seen that the kinetics of Rhodamine B

adsorption onto mango leaves powder follow this model with correlation coefficients of 0.999 and the equilibrium adsorption capacity (calculated), $q_{e(cal)}$ (8.695mg/g), is nearly equal to $q_{e(exp)}$ (8.7mg/g).

Table 2: Kinetic Parameter for the Adsorption of Rhodamine B by Mango Leaves Powder

Model	Parameter	Value
Pseudo-First-Order	$q_{e(exp)}$(mg/g)	8.70
	$q_{e(cal)}$(mg/g)	$1.364x10^{23}$
	k_1(min^{-1})	25.660
	R^2	0.314
Pseudo-Second-Order	$q_{e(cal)}$(mg/g)	8.77
	k_2(g/mg.min)	1.855
	R^2	0.999
Elovich	α(mg/g.min)	$2.755x10^{17}$
	β(mg/g)	5.348
	R^2	0.902
Intra-Particle Diffusion	k_i	0.068
	C	8.053
	R^2	0.884
Bangham's Model	k_b	0.081
	α	0.024
	R^2	0.948

Elovich Model:

Elovich equation is also used successfully to describe second order kinetic assuming that the actual solid surfaces are energetically heterogeneous, but the equation does not propose any definite mechanism for adsorbate–adsorbent (Yakout and Elsherif, 2010). The Elovich coefficients could be computed from the plots of q_t versus ln t from equation 5. The initial adsorption rate, α, and desorption constant, β, were calculated from the intercept and slope of the straight-line plots of q_t against ln t. Table 2 lists the kinetic constants obtained from the Elovich equation. It will be seen that applicability of the simple Elovich equation for the present kinetic data indicates that the Elovich equation was able to describe properly the kinetics of Rhodamine B adsorption on mango leaves powder.

Intra-Particle Diffusion

The possibility of intra-particle diffusion was explored by using Weber and Morris intra-particle diffusion resistance model (eq. 6) (Weber and Morris, 1963). The results indicated the possibility of the presence of intra-particle diffusion process as the rate determining step. The values of intercept (C) in the plot of q_t against $t^{1/2}$ (Figure 3) gives an idea about the boundary layer thickness, that is the larger the intercept, greater is the boundary layer effect (Pragnesh *et al.*, 2009). For this adsorption process, the value of C is 8.053. The first, sharp portion of the curve corresponds to the external surface adsorption stage or instantaneous adsorption stage. The second, less sloping linear portion, indicate the gradual adsorption stage and final portion represents the equilibrium stage. In this model, the MLP is treated as being surrounded by a boundary layer film through which the dye molecules must diffuse prior to adsorption on the MLP. The second portion of the figures indicated the intra-particle diffusion, where diffusive transport occurs through the internal pores of the MLP. As the bulk and surface metal concentrations start to decrease, the third section of the figure shows decrease in the rate of diffusion reaching a point of equilibrium.

The intra-particle diffusion, K_i, values were obtained from the slope of the straight-line portions of plot of q_t versus $t_{1/2}$. The correlation coefficient (R^2) for the intra-particle diffusion model for this adsorption process was found to be 0.884 (Table 2), and coupled with the fact that the curve did not pass through the origin, it indicate that the intra-particle diffusion is not the only rate-controlling step.

Bangham's Model

In the Bangham's model (eq. 7), log(log[Ci/Ci –Cs qt]) was plotted against log t and the values of the constants (α and k_b) are shown in Table 2. According to Pragnesh *et al.* (2009), if the experimental data fits this model, then the adsorption kinetics are limited by the pore diffusion. The double logarithmic plot did not yield satisfactory linear curves for the RB removal by the adsorbent (R^2 value 0.948). This shows that the diffusion of adsorbate into the pores of the sorbent was not the only rate-controlling step (Rajoriya *et al.*, 2007). Although, the film and pore diffusion were important to different extents in the removal process.

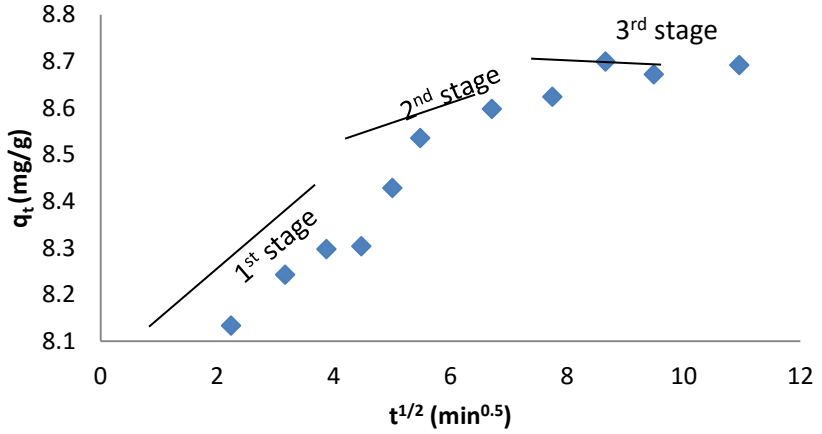

Figure 3: Plots of q_t versus $t^{0.5}$ showing the Three Diffusion Stages Predicted by the Diffusion Model for the Removal of RB by MLP.

Fourier Transform-Infrared (FT-IR) Characterization

The type and net charge of functional groups bonded to the adsorbent (mango leaves powder, MLP) surface are important in understanding the mechanism of adsorption of ionic adsorbates on the substrate. FT-IR spectra of the adsorbent (MLP) after adsorption of RB was taking and compared with the spectra before adsorption to obtain information on the nature of the possible absorbate-adsorbent interactions. The spectrum of the samples shows the presence of several functional groups (O-H, C=O, C-N, O-C=O) (Table 3). This demonstrates that after the adsorption, there is shifting of peak both up-field and down-field, indicating that there were binding processes taking place on the surface of the substrate. Presence of RB on the surface of the adsorbent was confirmed by comparing the FTIR of blank MLP with that of RB loaded MLP. It was observed that the peak at 3331 cm^{-1} for blank MLP shifted to a more intense peak at 3324 cm^{-1} for RB loaded MLP, with disappearance of peak at 1734 cm^{-1}. The peak at 3324 cm^{-1} could be due to O-H stretching vibration in carboxylic acid groups.

Table 3: Summary of FT-IR Results of MLP before and after Adsorption of Rhodamine B

S/N	Mango Leaves Powder wave number of absorption (cm^{-1})		Assignment
	Before adsorption	After adsorption	
1	3331	3324 +7	O-H stretching vibration in carboxylic acid groups (2500-3500 cm^{-1})
2	1734		C=O stretching in acid anhydrides (1765-1725 cm^{-1}) due to C=O stretching
3	1611	1592 +19	Benzene ring stretching in aromatic compounds (1615-1590 cm^{-1})
4	1320	1348 -28	COO- due to symmetric stretching (1400-1310 cm^{-1})
5	1037	1041 -4	C-N stretching vibration in amines (1030-1330 cm^{-1})
6	672	687 -15	O-C=O in carboxylic acid groups due to O-C=O bending (700-590 cm^{-1}); C-C-CHO in aldehydic compounds due to C-C-CHO bending (695-635 cm^{-1})

CONCLUSION

In this research work, *Mangifera indica* leaves powder was used as an effective adsorbent for the removal of Rhodamine B from aqueous solutions. The effects of variables, such as initial dye concentration, contact time and adsorbent dosage were studied on the adsorption. Isotherm modeling revealed that Freundlich model described the adsorption process. The dyes adsorption fitted well to pseudo-second-order kinetic model and also followed by intra-particle diffusion model, and diffusion is not only the rate-controlling step. FT-IR result showed shifting of peak both down field and up field which indicated that there is an interaction between the adsorbent and the dye molecules. In view of these results, it can be concluded that *Mangifera indica* leaves powder can be utilized as a low-cost and effective adsorbent in the removal of Rhodamine B from aqueous solutions.

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