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Prediction of Adsorption Kinetic Rate Constant for Removal of Methylene Blue Using Teak Leaf Powder

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ABSTRACT

Teak Leaf Powder (TLP) was used as adsorbent for Methylene Blue (MB) adsorption and its kinetics was investigated. Effects of parameters like solution dye concentration, pH, solution temperature, amount of adsorbent and particle size on MB adsorption onto TLP were examined. Methylene blue adsorption onto TLP was favoured at pH greater than its pH_{zpc}, 7.2. Experimental data were found to follow the pseudo second order kinetics. Influence of Temperature (T) and the dimensionless ratio of initial concentration to adsorbent concentration (N) on kinetics of MB adsorption onto TLP were examined using multivariate regression analysis. A quadratic model was proposed to express the kinetic rate constant as a function of these variables. Statistical tools like Student's t-test, F-test and ANOVA were used to identify the statistical significance of coefficients included in the model. It was found that the main effect of T, main effect of N, the second order effect of T were statistically significant. The model predicted the rate constant with high R^2 value 95.3%. It was found that the rate constant decreased with increase in N and increased with increase in T.

Key words: Teak leaf powder, low cost adsorbent, methylene blue, kinetic constant, multivariate analysis

INTRODUCTION

Presence of dyes in industrial wastewater is highly objectionable even at very low concentrations (Pratibha *et al.*, 2010; Kumar *et al.*, 2013; Gunasekar *et al.*, 2013). Efficient removal of dyes from aquatic streams is, therefore, very much essential. Among many physico-chemical treatments employed to treat coloured effluents, adsorption was one of the more efficient (Ponnusami *et al.*, 2008a; Gunasekar and Ponnusami, 2013). Since, widely used commercial adsorbents like activated carbon are too costly, numerous researchers have analysed the possibility of using various low-cost alternate adsorbents for colour removal (Ponnusami *et al.*, 2008c, 2010; Gupta *et al.*, 2009; Hameed and Tan, 2010). In our previous study, we had shown that teak leaf powder possess excellent adsorption potential (Ponnusami and Srivastava, 2009). Isotherm and thermodynamic analysis of MB adsorption onto TLP revealed that the adsorption potential of TLP was in the range of 99 to 208 mg g^{-1} and the process was spontaneous chemisorption (Ponnusami and Srivastava, 2009). As much as equilibrium and thermodynamic parameters,

precise estimation of kinetic parameters is also highly essential for efficient design of any adsorption system. Thus, in this work we had studied the kinetics of MB adsorption onto TLP and investigated the influence of process variables on adsorption kinetics using multivariate analysis.

In the majority of previous works on adsorption of dyes/metals onto low cost adsorbents, kinetic data were investigated using pseudo second order kinetic model (Ponnusami *et al.*, 2008b). The major advantage of this model is its ability to fit the kinetic data over the entire time range of adsorption (Ho and McKay, 1998, 1999a, 2000). Results of previous researchers confirm that the process variables like initial solute concentration (C_0) (Ho and McKay, 1999a; Vadivelan and Kumar, 2005; Ho and Ofomaja, 2006; Gurses *et al.*, 2006; El-Khaiary, 2007; Ofomaja and Ho, 2008), adsorbent dosage (D) (Ho and McKay, 1999a), particle size (d_p) (Ho *et al.*, 2001) and solution Temperature (T) (Ho and McKay, 1999b; Ho *et al.*, 2001, 2004; Ho and Ofomaja, 2006; El-Khaiary, 2007; Ofomaja and Ho, 2008) influence the rate constant. However, these work employed conventional one to one analysis. But, in practice the process variables often interact with each other (Ponnusami *et al.*, 2007, 2009). Therefore, it is necessary to employ design of experiments to reveal such interactions between the process variables. Student's t-test, F-test and ANOVA can be employed to test the interaction between the each factors. In the present work, we have evaluated the influence of the parameters on kinetics of methylene blue adsorption onto TLP by multivariate analysis.

MATERIALS AND METHODS

Adsorbent: Teak (*Tectona gandis*) is a famous timber tree. Teak is distributed from India to Thailand, Malaysia, Myanmar, Java and Philippines. Mature teak leaves were collected from the trees on the roadsides. Tap water was used to wash the collected leaves to remove dust and then distilled water was used to remove other impurities. These leaves were then dried at 70°C in hot air oven till they became crisp. Domestic mixer grinder (Smith kitchen machine) was used to powder the dried leaves. Ground leaves were screened in a rotary sieve shaker. Particles of different sizes (40, 125, 230, 548 μm) were collected separately and were given another wash with distilled water. Washed powders were dried in the hot air oven. Finally, the Teak Leaf Powder (TLP) was stored in air tight containers and used for analysis.

Adsorbate: The dye methylene blue (Chemical formula: $C_{16}H_{18}N_3SCl$; C.I. Classification Number: 52015, FW: 319.86 g mol^{-1}, class: thiazine, λ_{max} = 662 nm) was purchased from Himedia Limited (India) and used without further purification. Dye stock solution was prepared in a closed container by dissolving required amount of dye in double distilled water and stirred using magnetic bead overnight. The stock solution was further diluted to required concentrations.

Kinetic studies: Effects of process variables like solution temperature (T), dye concentration at initial (C_o), amount of adsorbent (D) and particle size (d_p) on adsorption of methylene blue onto TLP were studied in batch mode. For each experiment 100 cm^3 of methylene blue solution of known concentration was taken in a 250 mL conical flask. TLP in required quantity was added to that and agitated in an orbital shaker at a constant agitation speed and temperature. At regular time intervals samples were withdrawn from the flasks carefully using a syringe. Samples were subsequently centrifuged (Remi Research centrifuge) and clear solution was analyzed for residual methylene blue concentration by spectrophotometery (Ponnusami and Srivastava, 2009). Percentage dye removal at any time t was calculated using the Eq. 1:

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

where, C_0 is initial concentration of dye (mg L^{-1}) and C_t is concentration of dye (mg L^{-1}) at time t (min). Specific dye uptake (mg g^{-1}) was calculated by the following expression:

$$q_t = \frac{(C_0 - C_t)}{D} = \frac{R\ (\%) \times C_0}{100D} \quad (2)$$

where, D is the amount adsorbent added (g L^{-1}). The kinetic performance of the dye removal by adsorption process was investigated using pseudo-second-order kinetic model (Ho and McKay, 1998, 1999b, 2000). It is described by the following Eq. 3:

$$\frac{dq_t}{dt} = k_2 (q_e - q_t)^2 \quad (3)$$

where, q_t is dye concentraion on adsorbent at time t and q_e is dye concentration on adsorbent at equilibrium. Upon integration of this equation with suitable initial condition (at t = 0, q_t = 0) the following Eq. 4 is obtained:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (4)$$

Equation 4 was solved by linear regression using Excel 2003 to get the values of kinetic parameters k_2 and q_e.

RESULTS

Effect of pH: The effect of pH on the dye adsorption could be explained on the strength of pH zero point charge (pH_{zpc}) (Ponnusami *et al.*, 2008b). The value of pH_{zpc} was 7.3 for TLP. Adsorption of methylene blue onto TLP followed pseudo second order kinetic model (R^2>0.99). Ho's pseudo second order model was used to predict the maximum dye uptakes at different initial pH values and second order rate constants are presented in Table 1. Dye uptake was 15.02 and 45.79 mg g^{-1} when the solution pH was 2 and 9, respectively. It is evident from the Table 1 that adsorption was more when pH was greater than 7.3.

Adsorption kinetics and effect of process variables: The values of equilibrium dye uptake at various levels of C_o, D, d_p and T and adsorption rate constant were determined by linear regression

Table 1: Pseudo second order kinetics parameters for the MB adsorption onto TLP: Effect of initial solution pH

pH	k_2[a] (g mg^{-1} min^{-1})	$q_{m,pre}$[b] (mg g^{-1})	R^2[c]
2.00	0.00681	15.02	0.9980
4.00	0.00523	41.46	0.9993
6.00	0.00764	42.50	0.9999
8.00	0.00396	46.24	0.9999
9.00	0.00881	45.79	0.9999

[a]Pseudo second order kinetic rate constant $k_{2,pre}$ (g mg^{-1} min^{-1}), [b]Equilibrium dye uptakeq_e (mg g^{-1}), [c]Coefficient of determination

Table 2: Pseudo second order kinetic parameters for adsorption of MB onto TLP (temperature, T = 303, 313 or 323 K; particle size, d_p = 40, 125, 230 or 548 μm, adsorbent dose, D = 0.5, 1, 1.5 or 2 g dm^{-3}, initial dye concentration, C_0 = 50, 100, 150 or 200 mg dm^{-3}

T, K	d_p (μm)	D (g dm^{-3})	C_0 (mg dm^{-3})	$k_{2,pre}$[a] (g mg^{-1} min^{-1})	q_e[b] (mg g^{-1})	R^2[c]
303	125	0.2	50	0.0040	19.72	0.9914
303	125	0.2	100	0.0021	39.14	0.9970
303	125	0.2	150	0.0013	55.71	0.9967
303	125	0.2	200	0.0008	70.82	0.9982
313	125	0.2	50	0.0060	22.29	0.9993
313	125	0.2	100	0.0032	44.82	0.9994
313	125	0.2	150	0.0015	69.10	0.9990
313	125	0.2	200	0.0008	93.30	0.9995
323	125	0.2	50	0.0098	22.47	0.9981
323	125	0.2	100	0.0092	44.17	0.9998
323	125	0.2	150	0.0067	66.60	0.9996
323	125	0.2	200	0.0023	89.05	0.9993
303	125	0.5	100	0.0015	111.44	1.0000
303	125	1.0	100	0.0020	63.36	0.9900
303	125	1.5	100	0.0025	53.33	0.9990
303	40	0.2	200	0.0046	79.32	0.9997
303	125	0.2	200	0.0011	79.58	0.9971
303	230	0.2	200	0.0006	71.06	0.9896
303	548	0.2	200	0.0004	65.59	0.9768

[a]Pseudo second order kinetic rate constant $k_{2,pre}$ (g mg^{-1} min^{-1}), [b]Equilibrium dye uptakeq_e (mg g^{-1}), [c]Coefficient of determination

and the results are listed in Table 2 along with corresponding coefficients of determination. High values of R^2 confirmed that the kinetics of MB adsorption onto TLP followed pseudo second order model very well.

Multiple regression analysis: As mentioned earlier in introduction section, many researchers had shown that k_2 varied with C_o, D and T. In order to investigate the effects of these process variables multivariate regression analysis was employed. For the purpose of analysis initial concentration to adsorbent dosage was defined as a dimensionless variable N. Then, N and T^{-1} were included as independent variables. To predict the dependent variable k_2 the experimental data were fitted to a quadratic model:

$$Y = \beta_0 + \sum \beta_i x_i + \sum \beta_{ii} x_i^2 + \sum \beta_{ij} x_i x_j \tag{5}$$

where, β is the regression coefficient.

Student's t-test, F-tests and ANOVA were employed to elucidate the statistical significance of individual and combined effects of process parameters. The coded values of the variables were used for analysis. The variables X_i were coded as x_i according to the following Eq. 6:

$$x_i = \left(\frac{X_i - X_0}{\delta X} \right) \tag{6}$$

In this study the model coefficients were established using MINITAB 15. The result of multiple regression is shown in Table 3. Student's-t test was used to determine if calculated effects of these variables were significantly different from zero. From the Table 3 it is identified that the square

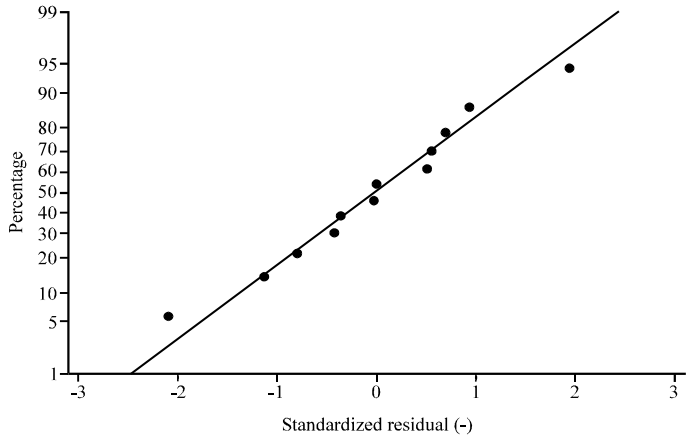

Fig. 1: Residual distribution for $\ln k_2$ for reduced model on adsorption of methylene blue onto teak leaf powder

Table 3: Multiple regression analysis for the adsorption of methyene blue onto TLP: Statistical parameters for full model and reduced model

	Full model[a]				Reduced model[b]			
Term	Coefficient	SE coef[c]	t[d]	p[e]	Coefficient	SE coef[c]	t[d]	p[e]
Constant	4.325	0.146	29.6	0.000	4.230	0.114	37.19	0.000
1/T	-0.634	0.084	-7.6	0.000	-0.634	0.080	-7.89	0.000
N	-0.846	0.092	-9.2	0.000	-0.846	0.088	-9.61	0.000
$1/T^2$	0.404	0.145	2.8	0.032	0.404	0.139	2.90	0.020
N^2	-0.171	0.154	-1.1	0.308				
1/T*N	-0.040	0.112	-0.4	0.731				

[a]R^2 = 96.2%, [b]R^2 = 95.3%, [c]Standard error of the estimated coefficient, [d]t -value: (Estimated coefficient)/(Standard error of the coefficient) [e]p-value: Probability of making a type I error

term N^2 and the interaction term N×(1/T) are not statistically significant at 95% confidence level (p-value>0.05). Therefore, these terms were eliminated from the regression model and again regression analysis was repeated and the result is given in Table 3. It can be seen that all the terms included in the model are statistically significant. However, to corroborate the adequacy of the model it is necessary to test the residual distribution of the data.

Residual distribution for ln k_2 is shown in Fig. 1. The uniform distribution of the residuals in the normal probability plot confirms the adequacy of the model to explain the variation in k_2 with N and T. The R^2 value for the reduced model was 0.953. That is, 95.3% of the variation in k_2 was explained by the regression while leaving only 4.7% to residuals. Therefore, the regression coefficients were substituted in the model equation. After rearrangement following empirical correlation was obtained:

$$k_2 = k' e^{-\frac{0.634}{T}} \tag{7}$$

Where:

$$k' = 65.497 e^{-\frac{0.846}{N} + \frac{0.446}{T^2}}$$

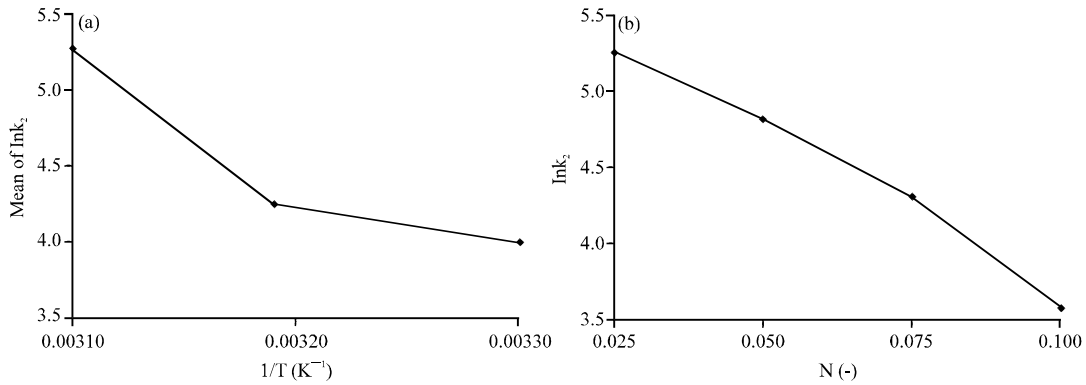

Fig. 2(a-b): Adsorption of methylene blue onto teak leaf powder: Main effects plot for (a) Mean of lnk_2 and (b) lnk_2

Table 4: ANOVA for lnk_2 for the adsorption of methyene blue onto TLP

Source	Degrees of freedom	Sequential sums of squares	Adjusted sums of squares	Mean squares	F[a]	P[b]
Regression	3	8.374	8.374	2.79	54.08	0.000
Linear	2	7.941	7.983	3.99	77.32	0.000
Square	1	0.434	0.434	0.43	8.40	0.020
Residual error	8	0.413	0.413	0.05		
Total	11	8.787				

[a]F: (MS of the regression term)/(MS of the error), [b]p: If calculated p-value is greater than 0.05, the term is statistically insignificant, at 95% confidence level, If it is less than 0.05, the term is statistically significant

Analysis of variance for lnk_2 is given in Table 4. Regression for lnk_2 included 2 linear and 1 square terms. All these terms were statistically significant (p-values<0.05) at 95% confidence level. Figure 2 and 3 show the main effects plot and contour plot, respectively. The main effects plot demonstrates that the rate constant increases with increase in T and decrease with increase in N. These trends were consistent with the earlier findings (Ponnusami *et al.*, 2008b). In addition the curvatures shown in main effects plot and the contour plots demonstrate the square effect of 1/T.

DISCUSSION

In the present study, powder addition method was used to determine the pH_{zpc} of the adsorbent as described by Kumar and Porkodi (2007). It (pH_{zpc}) was used to explain the influence of pH on adsorption pH_{zpc} was 7.3 and more adsorption was noted at pH greater than 7.3. The surface of the adsorbent particle gets more anionic charge when solution $pH>pH_{zpc}$ and favours binding of cationic dyes due to increased electrostatic force of attraction (Kumar and Porkodi, 2007; Ponnusami *et al.*, 2008c). Pseudo second order kinetic model was used to illustrate the kinetics of MB adsorption onto TLP. From Table 2 it was evident that the adsorption rate constant, k_2, decreases with increase in C_o and d_p. Meanwhile k_2 increases with increase in T and D. These trends were consistent with previous reports.

Many researchers had reported the variation of adsorption rate constant with initial solute concentration and solution temperature earlier. To correlate k_2 with initial solute concentration both linear and non-linear models had been used by researchers. Following forms of expressions had been widely used by the researchers in the past:

$$k_2 = a_1 C_0 - a_2 \tag{8}$$

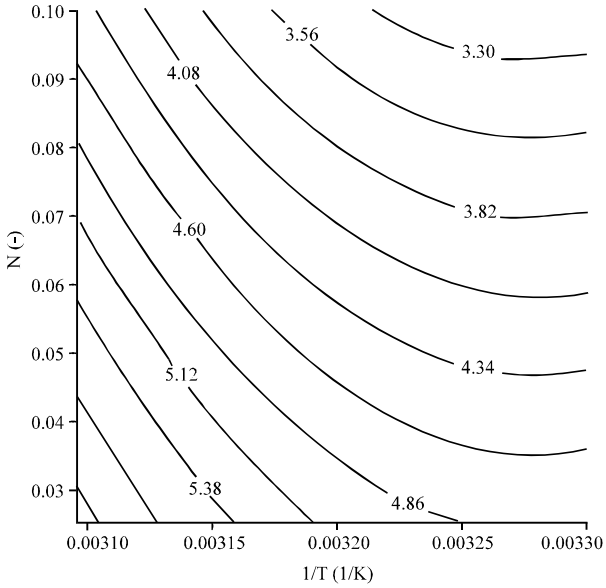

Fig. 3: Contour plot for $\ln k_2$ for adsorption of methylene blue onto teak leaf powder

$$k_2 = \frac{C_0}{a_1 C_0 + a_2} \tag{9}$$

$$k_2 = \frac{1}{a_1 C_0 + a_2} \tag{10}$$

$$k_2 = a C_0^b \tag{11}$$

Equation 8 was employed by Vadivelan and Kumar (2005). Equation 9 was applied by Gurses *et al.* (2006), Ho and McKay (1999b) and Ho *et al.* (2004). Equation 10 was used by El-Khaiary (2007). Equation 11 was employed by Ofomaja and Ho (2008). Comparison of k_2 values predicted by these Eq. 8 to 11 against those predicted by Eq. 7 is shown in Fig. 4.

It can be noted that the prediction by the quadratic model proposed was very close to the experimental data. Since the other models include only initial concentration as independent variable, these models need to be regressed for each temperature to get desirable result. As the present model includes both T and N as independent variables, it can be used for a wider range of data. Increasing trend of rate constant with increase in temperature had been shown by previous researchers for the following systems: Adsorption of copper onto palm kernel fibre (Ho and Ofomaja, 2006), methylene blue onto clay (Gurses *et al.*, 2006), adsorption of malachite green onto activated carbon (Onal *et al.*, 2007), adsorption of anionic dye onto sepiolite (Alkan *et al.*, 2007), sorption of lead ions onto tree ferns (Ho *et al.*, 2004), biosorption of Cadmium onto coconut copra meal (Ofomaja and Ho, 2008), adsorption of lead (II) onto peat (Kumar and Porkodi, 2007) etc. This trend can be very well explained by traditional Arhenius theory. The findings in the present study are consistent with this. Nevertheless, the constant k' depends on temperature and initial concentration. This observation can be attributed to the effects of external and internal diffusion

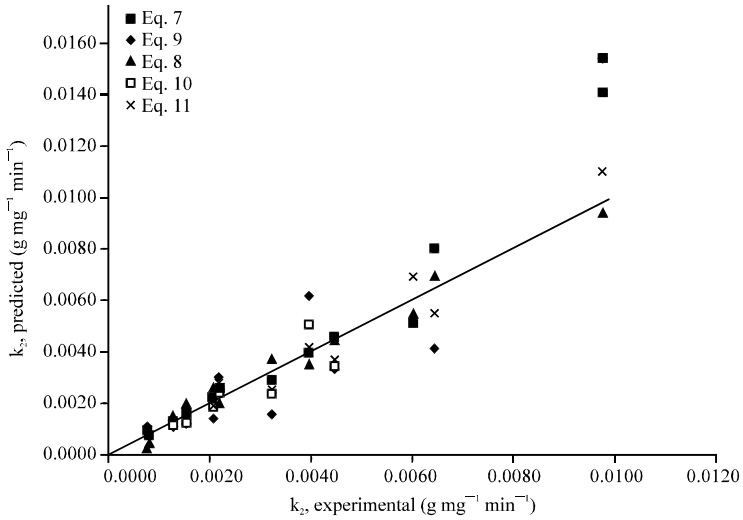

Fig. 4: Comparison of experimental and predicted k_2 values with other models proposed by previous researchers on prediction of k_2 from initial dye concentration

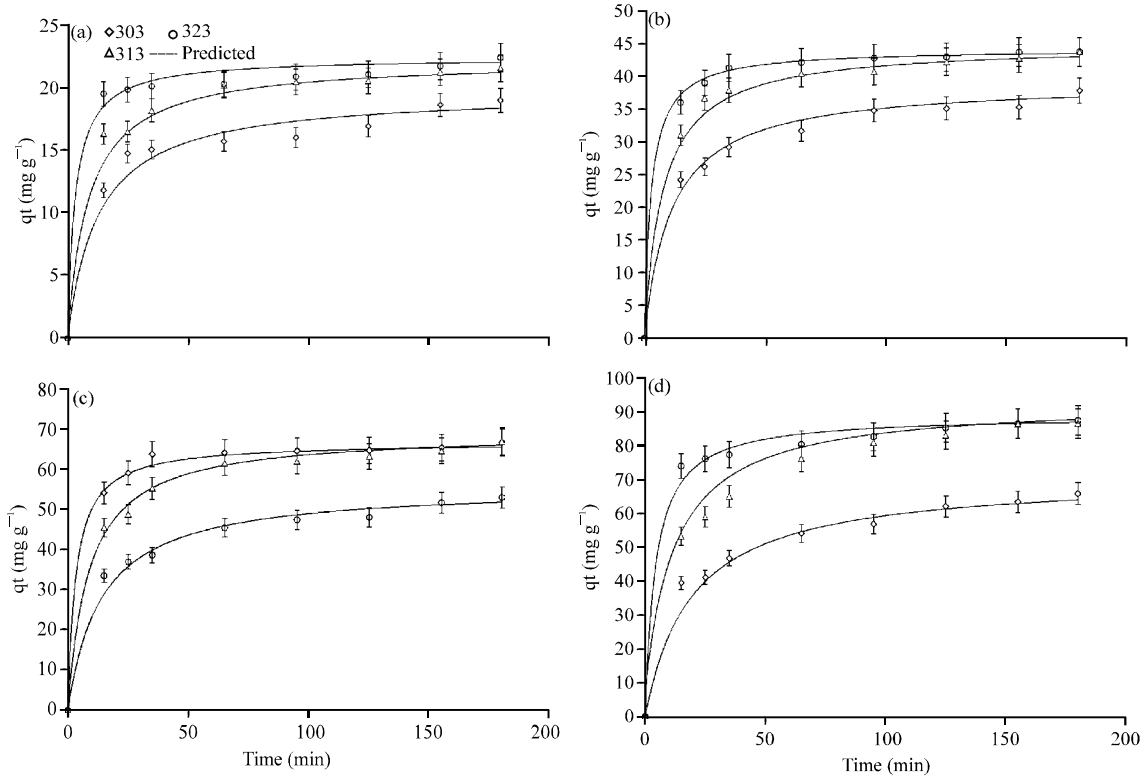

Fig. 5(a-d): (a) Comparison of predicted and experimental kinetic data (C_0 = 50 ppm, RPM = 200, dp =125×10^{-6} m, D = 2 g dm^{-3}), (b) Comparison of predicted and experimental kinetic data (C_0 =100 ppm, RPM = 200, dp = 125×10^{-6} m; D = 2 g dm^{-3}), (c) Comparison of predicted and experimental kinetic data (C_0 = 150 ppm, RPM = 200, dp=125×10^{-6} m, D = 2 g dm^{-3}) and (d) Comparison of predicted and experimental kinetic data (C_0 = 200 ppm, RPM = 200, dp = 125×10^{-6} m, D = 2 g dm^{-3})

resistances. Finally, using the k_2 values estimated using the Eq. 7, the kinetic data were predicted and the result was compared with the experimental data. The comparison is shown in Fig. 5a to d. These figures illustrate the efficiency of the proposed model in predicting the rate constant and concentration decay curve.

CONCLUSION

Adsorption of methylene blue onto Teak Leaf Powder (TLP) followed pseudo second order kinetics. In order to predict the rate constant as a function of most important process variables, namely solution temperature and the ratio initial concentration to adsorbent dosage a quadratic model was proposed in this study. The model predicted the rate constant with high R^2 value (0.953).

ABBREVIATIONS

R_t (%)	Percentage dye removal
C_0	Initial dye concentration (mg L^{-1})
C_t	Dye concentration (mg L^{-1})
t	Time (min)
T	Temperature (K)
q_t	Specific dye uptake (mg g^{-1})
D	Amount adsorbent added (g L^{-1})
q_e	Equilibrium dye uptake
pH_{zpc}	pH zero point charge
x	Process variable (coded units, -)
X	Process variable (un-coded units)
Y	Response variable (coded, -)
β_i, β_{ij}	Regression coefficients

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