IARJSET
ISSN (Online) 2393-8021
ISSN (Print) 2394-1588
International Advanced Research Journal in Science, Engineering and Technology
ISO 3297:2007 Certified
Vol. 4, Issue 11, November 2017

Equilibrium and Kinetic Modelling of Methylene Blue Adsorption by Psidium Guajava

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Abstract: Adsorption is potentially an attractive technology for treatment of waste water for retaining dyes from dilute solutions. Studies carried through environmental biotechnology have shown that many adsorbents present in the nature have great capacity for removal of dyes. The Methylene Blue dye was adsorbed on an adsorbent prepared from matured leaves of the Psidium guajava. A batch adsorption study was carried out with variable dye concentration, adsorbent amount, pH, adsorbent size and temperature. 94.9% of Methylene Blue dye was removed by 0.1 g of Psidium guajava leaf powder from 30 ml of aqueous dye solution with a concentration of 20 ppm at 333K. The adsorption followed pseudo second order kinetics with a rate constant of 0.025 mg/min .The experimental data yielded an excellent fit with Langmuir, Freundlich and Tempkin isotherm models. Langmuir monolayer capacity had a mean value of 29.913 mg/g. The best fitted model to the experimental equilibrium data for Psidium guajava was Langmuir isotherm for methylene blue dye. Free energy of adsorption (ΔG), enthalpy change (ΔH) and entropy changes (ΔS) were calculated to predict the nature adsorption.

Keywords: Psidium guajava leaf powder, dye removal, batch adsorption, Kinetics, Modelling.

I. INTRODUCTION

Dyes are used in large quantities in many industries including textile, leather, cosmetics, paper, printing, plastic, pharmaceuticals, food etc. to colour their products. Today, there are more than 10000 varieties of dyes are available commercially [1], most of which are difficult to biodegrade due to their complex aromatic molecular structure and synthetic origin [2]. The discharge of waste water containing traces of dye into the environment is aesthetically displeasuring, impedes light penetration, damages the quality of the receiving streams, and may be toxic to food chain organisms and to aquatic life [3]. Hence, removal of dyes from waste water before discharging is a major environmental problem and complete dye removal is mandatory due to the fact that dyes are visible even at very low concentrations [4-5].Further, dyes can be classified as anionic (direct, acid and reactive dyes), cationic (basic dyes), and nonionic (disperse dyes) [6]. A number of processes like flocculation [7], chemical coagulation [7], precipitation [7], ozonation [8], and adsorption [9] have been employed for the treatment of dye bearing waste water. Although the above said physical and chemical methods have been widely used, they posses inherent limitations such as high cost, formation of hazardous byproducts and intensive energy requirements[10]. Biological processes such as adsorption [11], bioaccumulation [12, 13], and biodegradation [14, 15] have been proposed as potential methods for the removal of dyes from textile waste water. In the present study, common guava leaf powder (*Psidium guajava*) has been used as adsorbent for the removal of methylene blue dye from its aqueous solution. Methylene blue is selected as a model compound in order to evaluate the capacity of common guava leaf powder for the removal of methylene blue dye from its aqueous solution. Methylene blue dye has wide applications, which includes coloring paper, temporary hair colorant, dyeing cottons, wools, and coating for paper stock. Though methylene blue dye is not strongly hazardous, it can cause some harmful effects. Acute exposure to methylene blue dye will cause increased heart rate, vomiting, shock, Heinz body formation, cyanosis, jaundice, quadriplegia, and tissue necrosis in humans [16].

II. MATERIALS AND METHODS

2.1 Preparation of Adsorbent : Common guava leaves were collected from the College of Engineering, Andhra University, Visakhapatnam. The collected leaves were washed thoroughly with deionized water to remove dirt particles. The washing process was repeated till the wash water contains no dirt. The washed leaves were then completely dried in sunlight for 20 days. The dried leaves were crushed and powdered using domestic mixer & grinder. In the present study, two ranges of particle size (0-70 & 0-300 μm) were used.

2.2 Preparation of Adsorbate: Methylene Blue dye (82% purity) was obtained from Ranbaxy Laboratories Ltd. (India). A stock solution of 1000 ppm Methylene Blue dye was prepared by dissolving 1.219 g of dye in 1000 ml of double distilled water which was later diluted to required concentrations. All the solutions were prepared using double distilled water. The structure of the Methylene Blue dye and its related information is given below.

2.3 Procedure: Batch adsorption experiments were carried out by varying, contact time (t, min), pH of the solution, Initial concentration of dye (C_I, ppm), particle size of adsorbent (d_p μm), Adsorbent dosage (w, gm) and Temperature

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(T ^{o}, K).In each experiment known amount of accurately weighed common guava leaf powder was added to 30 ml of dye solution of different concentrations in a 250 ml conical flask. The mixture was agitated at 200 rpm in a shaker for a specified time at room temperature. Samples were withdrawn at regular time intervals and centrifuged in centrifuge (Remi Research Centrifuge). The mixture was centrifuged and Methylene Blue remaining unadsorbed (λ_{max} 663-667 nm) was determined spectrophotometrically using spectro photometer (Hitachi 3210). Calibration curves were established using standard Methylene Blue dye solutions. The amount of dye adsorbed on adsorbent was calculated from the differences between dye quantity added to the biomass and dye quantity in the supernatant using the following equation:

$$q_t = (C_i - C_f)V/w \qquad (1)$$

Where q_e is the dye uptake (mg g^{-1}), C_i is the initial dye concentration (ppm) and C_f is dye concentration at equilibrium (ppm), V the volume of aqueous solution (L) and w is the amount of adsorbent (g).

III. RESULTS AND DISCUSSION

The removal of Methylene blue dye using *Psidium guajava* as adsorbent was investigated as a function of contact time (t, min), pH of the solution, Initial concentration of dye (C_i, ppm), Particle size of adsorbent (d_p µm), Adsorbent dosage (w, gm) and Temperature (T ^{o}, K). Results were given in q (mg/g) and q_{eq}(mg/g), amount of dye removed per unit mass of adsorbent at any time, t and at equilibrium time.

3.1 Effect of contact time (t):

Fig 1 shows the time course profiles for the adsorption of Methylene Blue dye from a solution of concentrations of 20, 40, 60 80 and 100 ppm. From the data, it is observed that the percent dye removal is increased with contact time at a uniform rate upto contact time of 130 minutes and no further increase is observed in percent dye removal for all concentrations of Methylene Blue dye solution. Therefore, the percent of dye removal (Ceq, ppm) and the amount of dye uptake (q_{eq}, mg/g) at the end of 130 minutes was given as the equilibrium value. For further studies of adsorption, the equilibrium time of 130 minutes has been considered.

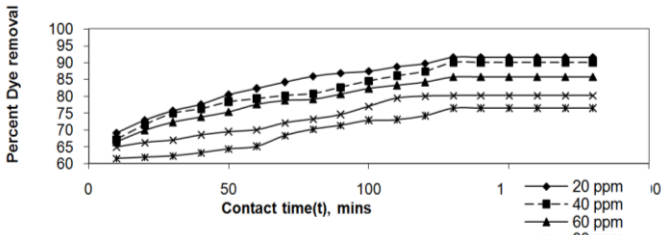

Fig 1- Effect of contact time on % removal

3.2 Effect of pH:

Fig 2 shows the effect of initial pH on the adsorption of Methylene Blue dye onto psidium guajava at room temperature, adsorbent concentration of 0.1g in 30ml of dye solution and adsorbent size of 0-70µm. With the increase in pH from 2 to 9, it is observed that the percent of dye removal is also increased from 54.45% to 91.62% and dye uptake was increased from 3.267 to 5.496 mg/ g. Equilibrium value for pH is observed at pH of 8. At lower pH, both the hydrogen ions as well as Methylene Blue ions compete together resulting in less adsorption. As the pH increases,the amount of OH^- ions increases which leads to the electrostatic attraction of the positive Methylene Blue ions with that of negative ions on the adsorbent there by % of adsorption increases.

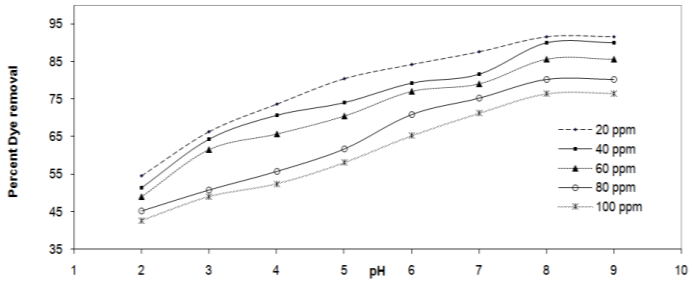

Fig 2-Effect of pH on % of dye removal

3.3 Effect of initial dye concentration (C_i):

The effect of initial dye concentration is shown in ***Fig 3***. To determine the effect of initial dye concentration, the initial concentration of the dye was varied from 20 ppm to 100 ppm. At higher concentrations, lower percentage of dye removal was observed because of the saturation of the adsorption sites. With increase in initial dye concentration from 20 ppm to 100 ppm, percent of dye removal decreased from 91.62% to 76.08% and dye uptake was increased from 5.496mg/g to 23.724 mg/g.

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$$\% \text{ Adsorption} = \frac{(C_i - C_f)}{C_i} \times 100 \qquad (2)$$

The initial concentration C_i (ppm) and final metal concentration C_f (ppm) at any time were determined and the dye uptake q_{eq} (mg dye adsorbed /g adsorbent) was calculated from the mass balance as follows:

$$q_{eq} = \frac{V(C_i - C_{eq})}{1000w} \qquad (3)$$

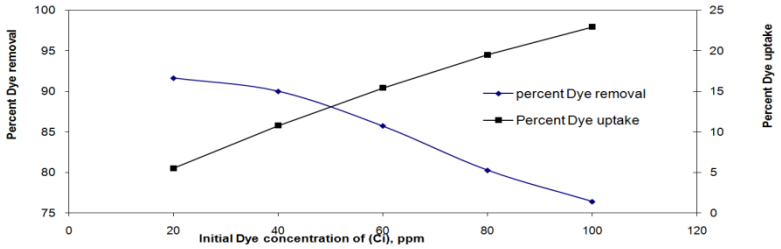

Fig 4 - Effect of Adsorbent dosage on percent dye removal and dye uptake

3.4 Effect of adsorbent dosage (w):

To study the effect of adsorbent dosage on removal of Methylene Blue dye, adsorbent dosage is varied from 0.02 g to 0.1 g, fixing other parameters like initial concentration at 20 ppm, pH 8 and particle size 0-70µm. The equilibrium contact time was taken as 130 minutes. From ***Fig 4***, it is observed that, with increase in adsorbent dosage, % dye removal is increased. The % dye removal is increased from 78.56% to 91.62% with an increase of dosage rate from 0.02 to 0.1 g and the amount of dye uptake decreased from 23.724 mg/g to 5.720 mg/g. The increase in % removal of the dye with adsorbent dosage can be attributed to the increased available surface area of the adsorbent and hence higher number of sites available for adsorption. However, the decrease in dye uptake capacity q_e value may be due to the splitting effect of flux (concentration gradient) between sorbate and sorbent with increase in biomass concentration causing a decrease in amount of adsorbed onto unit weight of biomass.

3.5 Effect of adsorbent size (dp):

To study the effect of adsorbent particle size, two ranges of particle sizes (0-70 µm and 0-300 µm) are used in the current study, keeping the dosage rate at 0.1 g, initial concentration of solution at 20 ppm, pH8, volume of 30ml solution, contact time of 130.minutes. ***Fig 5*** shows that with increase in adsorbent particle size from 0-70 µm to 0-300 µm, % removal of dye decreases from 91.6 to 63.45. This is due to less surface area available with increased particle size, thus reducing the % of dye removal.

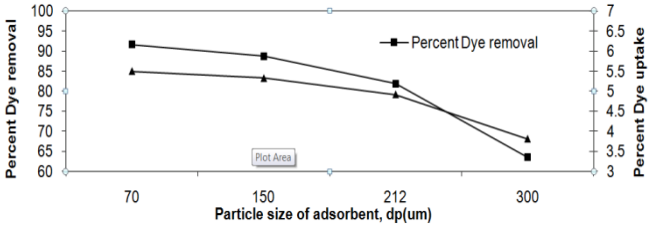

Fig 5 Effect of particle size of adsorbent, dp(um)

3.6 Effect of temperature:

When the adsorption was carried out at four different temperatures ranging from 303°K to 333°K for dye concentrations ranging from 20 to 100 ppm, the extent of adsorption for all the concentrations of the dye is improved steadily with an increase in adsorption temperature (Fig 6). Percent dye removal is increased from 91.62 to 94.90 and the amount of dye uptake is increased from 23.70 mg/ g to 24.30 mg/g for a temperature rise from 303-313 ^{0}K. This increase could be due to increase in surface activity and increased kinetic energy of the dye molecules.

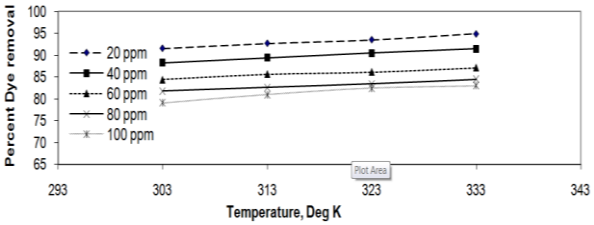

Fig:6 Effect of temperature on percent removal of dye

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3.7 Adsorption isotherms

3.7.1 Langmuir isotherm

The Langmuir sorption isotherm has been successfully applied to many pollutant adsorption processes and has been the most widely used isotherm. Langmuir theory is based on the assumption that the adsorption takes place at specific homogeneous sites within the adsorbent.

The Langmuir equation is given as:

$$\frac{C_{eq}}{q_{eq}} = \frac{1}{bq_{\max}} + \frac{1}{q_{\max}} C_{eq} \qquad (4)$$

Where, Ceq = concentration of the dye solution at equilibrium, (ppm)
q_{eq} = amount of dye adsorbed at equilibrium, (mg/g)
q_{max} = maximum sorption capacity, (mg/g)
b = Langmuir constant (L/mg)

Fig 7 shows the plot of the Langmuir equation (2), (C_{eq} / q_{eq} vs C_{eq}), which indicates a straight line with a slope of $1/q_{max}$ and an intercept of $1/bq_{max}$. From the plot of Langmuir equation, the maximum adsorption capacity (q_{max}) is found to be 29.94 mg/g and Langmuir constant (b) is found to be 0.132 L/mg.

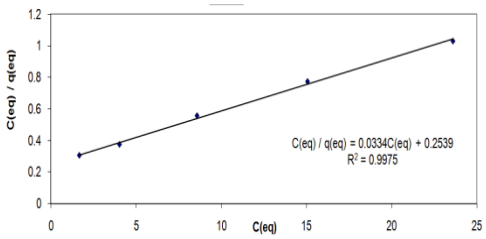

Fig 7-Langmuir sorption isotherm for methylene blue dye

3.7.2 Freundlich Isotherm

Freundlich isotherm states that, when the concentration of solute in the solution at equilibrium (Ceq) is raised to the power m, the amount of solute adsorbed being qeq, then Ceqm/qeq is a constant at a given temperature, which is expressed by the following equation:

$$\log (q_{eq}) = \log (K_F) + (1/n)^* \log (C_{eq}) \qquad (5)$$

Fig 8 shows the plot of log (q_{eq}) vs. log (C_{eq}). From the plot, the adsorption / distribution coefficient (K_F) is found to be 4.598 and the value of (n) is 1.879.

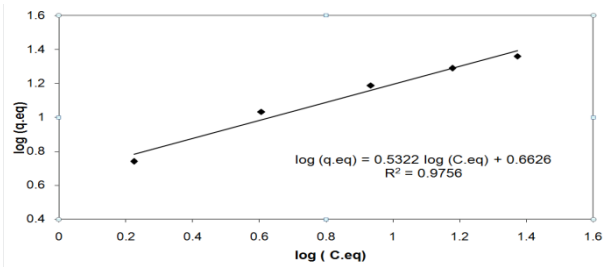

Fig 8- Freundlich sorption isotherm for methylene blue dye

3.7.3 Tempkin isotherm: This model takes into account the presence of indirect adsorbate / adsorbent interactions and suggests that because of these interactions the heat of adsorption of all molecules in the layer would decrease linearly with coverage. Tempkin isotherm is given as:

$$q_{eq} = \left(\frac{RT}{b_T}\right)\ln\left(C_{eq}\right) + \left(\frac{RT}{b_T}\right)\ln\left(A_T\right) + \qquad (6)$$

Where, b_T and A_T are Tempkin isotherm constants.

Fig 9 shows a plot of q_{eq} vs ln (c_{eq}), which is a straight line with slope of RT/b_T and intercept of RT/b_T * ln (A_T). From the plot, Tempkin constants are found as bT =384.54 and A_T = 1.316 L/mg with R-squared value of 0.9973.

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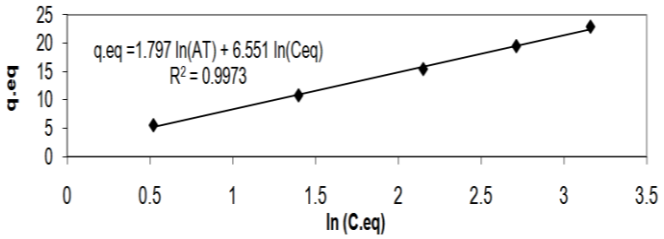

Fig 9 Tempkin sorption isotherm for methylene blue dye

The isotherm constants obtained from the above four isotherms are complied and listed in Table-2. The best fit equilibrium model is determined based on the linear regression coefficient (R^2). From the table, it is observed that the adsorption data are well represented by both Langmuir and Tempkin isotherms for Methylene Blue dye adsorption with higher R^2 of 0.997, followed by Freundlich isotherm with R-squared value of 0.975. The value of R^2 (0.323) for R-P isotherm model represents the non suitability of the model to the current adsorption system.

Table-2: Isotherm constants

Langmuir model	Freundlich model	Tempkin model
q_{max} = 29.94 mg/g	1/n = 0.532	b_T = 384.54
b = 0.132 L/g	K_f = 4.598	A_T = 1.316 L/mg
R^2 = 0.997	R^2 = 0.975	R^2 = 0.997

3.8 Kinetic studies

The kinetics of the adsorption data was analyzed using two kinetic models, pseudo-first order and pseudo-second order. These models correlate solute uptake, which are important in predicting the reactor volume.

3.8.1 Pseudo-first order model

The possibility of adsorption data following Lagergren pseudo-first order kinetics is given by:

$$\frac{dq}{dt} = K_{II}(q_{eq} - q) \qquad (7)$$

Where q is the amount of adsorbate at time t (mg/g), q_{eq} is the adsorption capacity at equilibrium (mg/g), K_I is the rate constant of the pseudo-first-order model (min^{-1}).

Values of rate constant (K_I) and q_{eq} for adsorption of dye are determined from the plot of log (q_{eq}-q) vs. time (t), shown in ***Fig 10***. The intercept of the plot is equal to log q_{eq}. However, if q_{eq} obtained from the plot does not equal to the equilibrium Methylene Blue dye uptake, then the reaction is not likely to be first order, suggesting the insufficiency of Pseudo-first-order model to fit the kinetic data for all the initial dye concentrations.

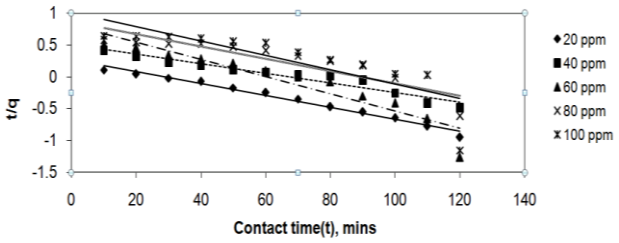

Fig 10- First order kinetics

3.8.2 Pseudo-second order model

A pseudo-second order model proposed by Ho and McKay was used to explain the sorption kinetics. This model is based on the assumption that the adsorption follows second order chemisorption. The pseudo-second order model can be expressed as

$$\frac{dq}{dt} = K_{II}(q_{eq} - q)^2 \qquad (8)$$

Separating the variables in Eq. (8) gives:

$$\frac{dq}{(q_{eq} - q)^2} = K_{II}\,dt \qquad (9)$$

Integrating Eq. (9) for the boundary conditions q=0 to q=q at t=0 to t=t, Eq. (9) simplifies to:

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$$\frac{t}{q} = \frac{1}{K_{II} q_{eq}^{\ 2}} + \frac{1}{q_{eq}} t \qquad (10)$$

Where t is the contact time (min), q_{eq} is the amount of dye adsorbed at equilibrium (mg/g) and q the amount of dye adsorbed at any time, t (mg/g). The correlation coefficients were found to be 0.9987 for initial concentrations 20 ppm to 100 ppm. If second order kinetics is applicable, the plot (***Fig 11***) of t/q versus time (t) of equation (10) should give a linear relationship from which the constants q_{eq} and K_{II} can be determined.

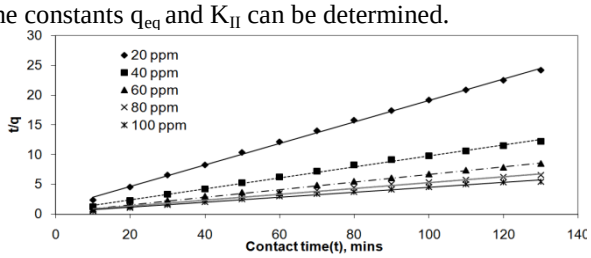

Fig-11 Second order kinetics

The rate constants and the correlation coefficients of the tested models (first order and second order) have been calculated and summarized in Table-3 and Table-4.

Table: 3-First order kinetic parameters for the adsorption of Methylene Blue dye .

Concentration, ppm	First order kinetics			
	K_I	R^2	q_{eq}(plot)	q_{eq}(expt)
20	0.02116	0.984	1.858	5.49
40	0.0017	0.939	3.217	10.79
60	0.0031	0.883	6.465	15.42
80	0.0223	0.813	9.384	19.47
100	0.0263	0.650	10.524	22.91

Table: 4 Second order kinetic parameters for the adsorption of Methylene Blue dye

Concentration, ppm	Second order kinetics			
	K_{II}	R^2	q_{eq}(plot)	q_{eq}(expt)
20	0.025	0.999	5.55	5.49
40	0.014	0.997	10.82	10.79
60	0.013	0.994	15.63	15.42
80	0.003	0.993	20.20	19.47
100	0.005	0.992	23.92	22.91

From Table-3 and Table-4, the kinetics of the adsorption of the Methylene Blue dye on Psidium guajava is best described withy second order kinetics rather than first order.

3.9 Effect of Thermodynamic parameters:

The change in variation of adsorbed amount during adsorption can be explained by three main thermodynamic parameters. These parameters are entropy change (ΔS), enthalpy change (ΔH) and Gibb's free energy (ΔG). The negative value of ΔH indicates that the process is exothermic and the positive value of ΔH indicates that the process is endothermic.The thermodynamic parameters for the adsorption process were computed from the plot of log (q_{eq}/C_{eq}) vs. 1/T, shown in ***Fig 12***. The free energy change for methylene blue dye ions on to *psidium guajava* were determined using the equilibrium constant obtained from Langmuir isotherm model.

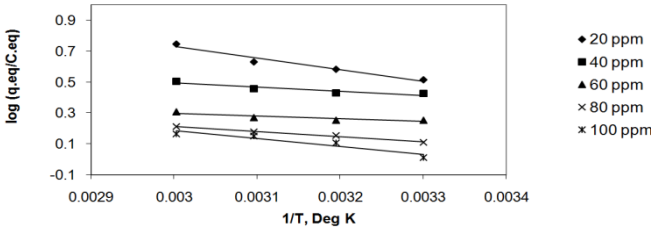

Fig 12 Effect of temperature on adsorption w.r.t concentration

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Table-5
Thermodynamics properties for adsorption of Methylene Blue dye for different concentrations

C_0 ppm	ΔH (KJ/mol)	ΔS (KJ/mol K)	ΔG (KJ/mol)			
			303 DegK	313 DegK	323 DegK	333 DegK
20	14.32	0.057	-2.931	-3.500	-4.070	-4.639
40	5.101	0.025	-2.399	-2.647	-2.894	-3.142
60	3.39	0.016	-1.418	-1.577	-1.735	-1.894
80	6.41	0.023	-0.660	-0.894	-1.127	-1.360
100	9.82	0.033	-0.179	-0.509	-0.839	-1.169

IV. CONCLUSIONS

➢ With increase in contact time (t), percent dye removal is increased and the equilibrium contact time is observed at 130 minutes and the percentage of dye removal is 91.62 at the equilibrium contact time.pH of the solution is varied from 2 to 9. With increase in pH from 2-9, it was observed that the percent dye removal is also increased from 54.5 to 91.62 and the percentage of dye up take was increased from 3.27 to 5.49 mg /g. Equilibrium value for pH is observed at pH of 8 with 91.62 percent dye removal.Dosage of the adsorbent (w) is varied from 0.02 to 0.1 g. It was observed that as the dosage rate increases, the percent dye removal is also increased. As the dosage of the adsorbent increased from 0.02 to 0.10 g, percent dye removal increased from 78.50 to 91.62.

➢ With increase in particle size of adsorbent (dp), it is observed that the percent dye removal is decreased. The percent dye removal is decreased from 91.6 to 63.45 as the particle size is increased from 0-70 μm to 0-300μm. The percent dye up take decreased from 5.496 to 3.807.With increase in the temperature from $303K^0$ to $333K^0$, it is observed that the percent dye removal is increased from 91.62 to 94.9.

➢ The adsorption data are well represented by both Langmuir, Tempkin isotherms for Methylene Blue dye adsorption, followed by Freundlich isotherm.The kinetics of the adsorption of the Methylene Blue dye on Psidium guajava is best described withy second order kinetics rather than first order.

➢ The present work helped in identifying a new parameter study of adsorbent for removal of dyes from effluent wastes containing low concentrations of Methylene Blue dye. The Langmuir, Freundlich and Tempkin isotherm models proved to be the best adjustment of the experimental data for the adsorbent, *Psidium guajava*. However, comparing the representative models of the adsorption between the adsorbents, a better adjustment of the equilibrium data is observed with the adsorbent *Psidium guajava*. The results obtained in these studies open perspectives with relation to the utilization of *Psidium guajava* in the removal of Methylene Blue dye ions, in treatment of wastewater provided from textile industrial effluents.

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