Biosorption of Methylene Blue Dye Aqueous Solutions on *Delonix regia* (Flamboyant Tree) Pod Biosorbent.

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ABSTRACT

The biosorption of Methylene Blue (MB) dye solutions by *Delonix regia* (Flamboyant tree) pod has been studied using different parameters (initial concentrations, biosorbent dosage, pH, contact time, and temperature). The maximum optimum biosorption capacity/MB uptake (q_e) and removal efficiency of MB dye solutions by *Delonix regia* are 34.27215 and 97.9204, respectively. Biosorption is enhanced in the basic pH region, the highest and lowest biosorption were observed for MB dye biosorption at pH 10 and 3, respectively, while increases in the initial concentration of MB dye solution resulted in corresponding increases in q_e and MB removal efficiency. Dosage of the biosorbent also had an influence on the amount of MB dye solution adsorbed as increases in dosage resulted in increases in MB removal efficiency but decreases in q_e. Increases in temperature were found to enhance the biosorption as the biosorption of MB dye solutions and was highest at 50°C and lowest at 30°C for various concentrations. The kinetics of the biosorption process can best be described by the pseudo-second order model, and the thermodynamic parameters showed that the biosorption process is feasible (exothermic) with both Langmuir and Freundlich Isotherms well fitted for the biosorption.

(Keywords: biosorption, thermodynamics, kinetics, removal efficiency, *Delonix regia,* MB dye, isotherm)

INTRODUCTION

The problem of considerable pollution of the aqueous environment with organic pollutants still requires the development of quick and simple methods for the removal of these pollutants. Dyestuffs have been prominent organic pollutants that most of the industries use and discharge into surface and sub-surface water bodies. These compounds are troublesome pollutants which pose not only to toxicity and health hazards but also hamper the environmental treatment processes.

Disposal of effluents from the dyeing industry pose one of the major problem, because such effluents contain a number of pollutants including acids or bases, dissolved solids, surfactants, and dyes. Out of these, dyes are the most easily recognized contaminant because they are visible to the human eye. These dyes can cause harmful actions to various forms of aquatic-life. Dyestuff effluent is resistant to light and when discharged into the water bodies, it prevents the sunlight from penetrating through and reduces the aesthetic quality of water.

Several biological, physical, and chemical methods (chemical oxidation, froth flotation, coagulation, biosorption, etc.) have been used for the treatment of industrial textile wastewater including microbial biodegradation, membrane filtration, oxidation, and ozonation (Forgacs, *et al.,* 2004). However, many of these technologies are very uneconomical, especially when applied for treating large waste streams. Consequently, biosorption techniques seem to have high effectiveness in industrial dyestuff related wastewater treatment because of their proven efficiency/economic advantage as well as the stability and regeneration of the biosorbent in the removal of dyes and other organic and mineral pollutants (Robinson, *et al.,* 2002a, Garg, *et al.,* 2003, Abdel-Ghani, *et al*., 2007, Babel and Opiso, 2007).

The mechanism of the biosorption process has been reported to be very complex and possibly involves the combination of diffusion, biosorption, chelation, complexation, co-ordination, or micro-precipitation mechanisms depending on the specific biosorbent (Veglio and Beolchini, 1997). Therefore, biosorption can be affected by many chemical and physical variables such as pH, ionic strength, biomass dosage, and so on. Investigating these parameters will assist in having a sound understanding of the phenomenon.

The most widely used biosorbent for this purpose is activated carbon, but its expense (McKay, *et al.,* 1987, Low, *et al.,* 1995) has led to a search for cheaper alternative materials such as orange and banana peels (Annadurai, *et al.,* 2002), neem leafs (Bhattacharyya, *et al.,* 2003), agricultural residues (Robinson, *et al.,* 2002b), and peanut hulls (Gong, *et al.,* 2005). Several biosorbents have been reported for the removal of dyes from waste water (Poots, V. J. P., Mckay, G., Healy, J. J. 1976; Gupta , G. S., Prasad, G., Singh, V. N., 1988; Gupta, G. S., Srivastava, S. K., Mohan, D., 1997). The use of flamboyant tree pod as a biosorbent has been rarely reported and this provides a premise of its use for this research.

The objective of this study was to evaluate the effectiveness of *Delonix regia* (flamboyant tree) pod as biosorbent for the removal of Methylene Blue (MB), a cationic dye from aqueous solutions. Laboratory batch studies were conducted to estimate the biosorption capacity and dye removal efficiency using the biosorbent for different initial concentrations of the simulated waste water, and to assess the effect of biosorbent dosage, pH and temperature on the biosorption capacity of the biosorbent.

MATERIALS AND METHODS

Preparation and Characterization of Biosorbent: Samples of *Delonix regia* were collected and subjected to drying. Dried flamboyant tree pod was grinded and sieved. The grains obtained were characterized using FTIR. The grains were further used as biosorbent in the biosorption experiment.

MB Dye Solution Preparation: In this study, the methylene blue (MB) used was obtained from the laboratory. An accurate weighed quantity of the MB dye was dissolved in distilled water and made up to mark in appropriate standard volumetric flask to prepare different initial concentrations of the dye ranging from 10 to 70 mg/L at 10 mg/L intervals.

Batch Biosorption Experiments: In each biosorption experiment, 50 ml of MB dye solution of known different initial concentrations (10 – 70 mg/L) at natural pH was added to 100 mg of the biosorbent in 250 ml flat bottom bottle at room temperature (28°C) and the mixture was stirred on an electric shaker at 200 rpm for 1 hr.

Further experiments were carried out at different initial pH values; initial pH was controlled by the addition of 0.1M sodium hydroxide (NaOH) or 0.1M hydrochloric acid (HCl). Here, 50 ml of MB dye solution of known initial concentration (20 ppm) at different pH (ranging from 3 to 10) was added to 100 mg of the biosorbent in 250 ml flat bottom bottle at room temperature (28°C) and the mixture was stirred on a electric shaker at 200 rpm for 1 hr.

A total of 50 ml of MB dye solution of known concentrations (20 mg/L) at natural pH was added to different dosage of the biosorbent (ranging from 20 mg to 140 mg at 20mg interval) in 250 ml flat bottom bottle at room temperature (28°C) and the mixture was stirred on a electric shaker at 200 rpm for 1 hr.

Experiments on the effects of temperature as well as thermodynamics studies on the biosorption of MB were done by varying the temperature between 30 and 50° C at 10° C intervals. 50 ml of varying concentrations (10– 40 mg/L) of MB dye solution at natural pH was added to 100 mg of the biosorbent in 250 ml flat bottom bottle at different temperature and the mixture was stirred on a electric shaker at 200 rpm for 1 hr. The thermodynamic parameters, ΔH^0, ΔS^0, and ΔG^0, for the biosorption process were calculated using the relationship:

$$\ln b = \Delta S^0/R - \Delta H^0/RT \qquad (1)$$

where b is the Langmuir constant related to energy. The linear plot of ln b versus 1/T yields a slope and intercept whose values correspond to ΔH^0 and ΔS^0, respectively. These values can then be used to compute ΔG^0 by applying the Gibbs relationship:

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \qquad (2)$$

In the biosorption kinetics experiment, 200 ml of MB dye solution of known different concentrations (10 – 40 mg/L at 10 mg/L interval) at natural pH was added to 400 mg of the biosorbent in 250 ml flat bottom bottle at room temperature and the mixture was stirred on a electric shaker at 200 rpm. Solutions of the MB dye were collected at 15 minutes interval over the period of 2 hours.

In all of the experiments, the supernatants were taken from every flask and filtered; the remaining MB concentration after biosorption process gives final (equilibrium) concentration. The initial (before biosorption experiment) and final/equilibrium (after biosorption experiment) concentrations were determined by using absorbance values at 668 nm with UV Visible Spectrometer.

The MB at equilibrium was calculated by:

$$q_e = V(C_o - C_e) / W \qquad \textbf{(3)}$$

where q_e is MB uptake, V is the volume of the solution and W is the amount of the biosorbent, C_o and Ce are initial and final (equilibrium) sorbate concentrations, respectively. The dye concentration was calculated from a calibration curve of absorbance versus concentration.

RESULTS AND DISCUSSIONS

Characterization of Biosorbent Using Infra Red Spectroscopy, IR: The prominent peak in IR spectra of the Biosorbent, *Delonix regia* (Flamboyant tree) pod is shown in Table 1.

Table 1: Characterization of Biosorbent (Prominent Peak in IR Spectra of the Biosorbent, *Delonix regia* (Flamboyant tree) pod.

Value (cm^{-1}) of Prominent Peaks	Functional Group Present
1747 - 1716	C=O groups
1651 - 1558	C = C
Strong 1045	C–O stretching
2924 – 2854	Saturated Alkyl H^+
3078 - 3032	Aromatic H^+
3645 – 3128	Active H^+ in ROH (Alcohol)

Effect of Initial Concentration on the Biosorption of MB: The MB removal efficiency for different initial concentrations (C_o) is above 97%. This suggests the occurrence of a strong attraction between the biosorbent surface and the MB dye solute. q_e increases as the initial concentration rises; this is so because particles of MB are available for surface interaction with the biosorbent (i.e., enhanced mass transfer of MB molecules to the surface of the biosorbent). This biosorption characteristic indicated that surface saturation was dependent on the initial metal ion concentrations.

At low concentrations, biosorption sites took up the available MB molecule more rapidly while when the concentrations increases to high concentration the rate of diffusion became slow. This is because MB particles needed to diffuse to the biosorbent surface by intraparticle diffusion and hydrolyzed MB ions will diffuse at a slower rate (Zafar et al., 2006). q_e showed a linear relationship with the initial concentration as shown in Equation 4.

The initial concentration imparts a significant force in overcoming mass transfer resistances of the dye between the aqueous and solid phases. Thus, initial concentration of MB dye solutions enhances the biosorption. (Aksu, 2001). The effects of initial concentrations of MB solution on the biosorption process are shown in Table 2 and Figure 1.

$$q_e = 0.4899C_o – 0.0381 \qquad \textbf{(4)}$$

Table 2: MB Removal Efficiency and q_e at Different Initial Concentrations.

C_o (mg/L)	C_e (mg/L)	MB Removal Efficiency	q_e
10	0.2551	97.4490	4.87245
20	0.5628	97.1860	9.71860
30	0.6978	97.6740	14.65110
40	0.7654	98.0865	19.61730
50	1.0430	97.9140	24.47850
60	1.4257	97.6238	29.28715
70	1.4557	97.9204	34.27215

Effect of Contact Time on the Biosorption of MB: The amount of MB dye solutions adsorbed at 40 mg/L is shown in Figure 2. The removal of MB dye solutions was rapid at the initial period of the contact time and decrease slightly until the equilibrium is reached. The sites for biosorption are bare at the initial contact time and this makes biosorption rate to be fast as a result of diffusion process from the bulk solution to the sites. In the latter contact time, the biosorption sites are less available leaving the process to be attachment controlled.

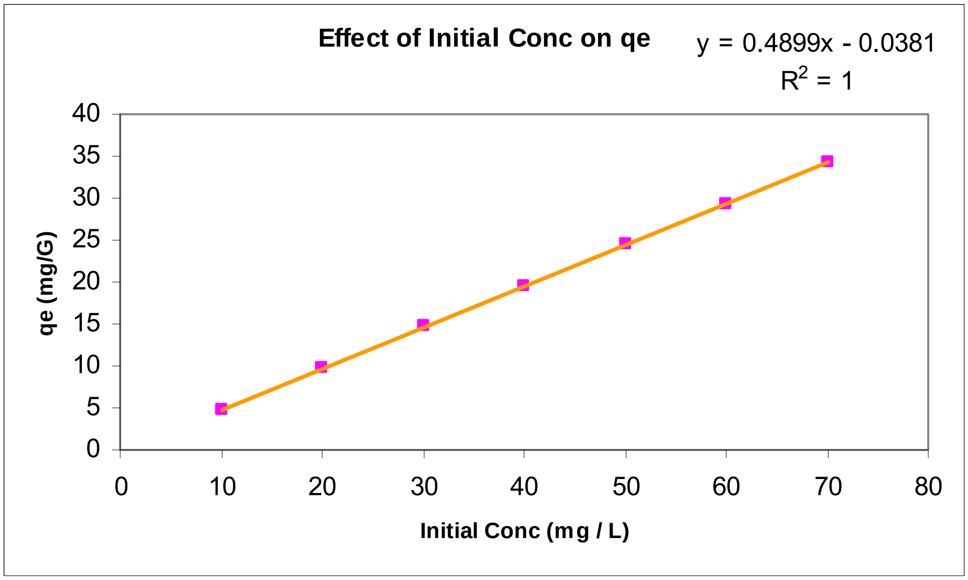

Figure 1: Effect of Initial Concentration on q_e.

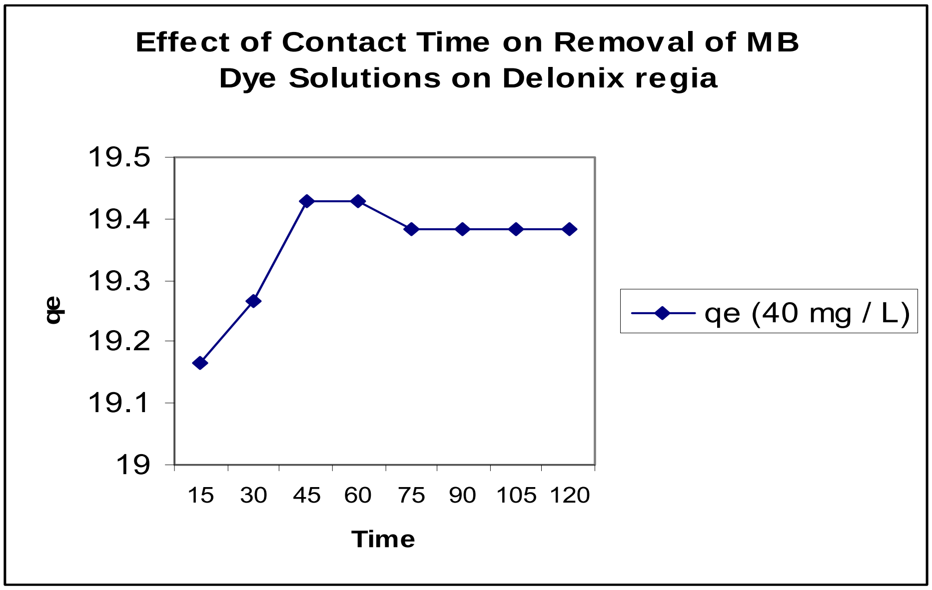

Figure 2: Effect of Contact Time on q_e.

The equilibrium required to attain equilibrium was 75 seconds. This pattern is the same for other concentrations (10, 20 and 30 mg / L). A similar trend has been reported in the biosorption of dyes using pine saw dust (Garg et al., 2003).

Effect of Biosorbent Dosage on the Biosorption of MB: MB removal efficiency increases with the increase in biosorbent dosage with least value of 94.97% obtained with 0.02g and highest of 97.60% with 0.14g of the biosorbent. This is due to the increase in surface area and the availability of biosorption sites. The decrease in q_e could be as a result of splitting effect of the concentration gradient between the MB dye solution and biosorbent with increasing biosorbent dosage bringing about a decrease in the amount of MB adsorbed per unit weight of biosorbent. This phenomenon has been reported in previous research (Donmez et al., 1999; Vansanth Kumar et al, 2006, Sun Xue-Fei et al., 2008). These results are shown in Figure 3.

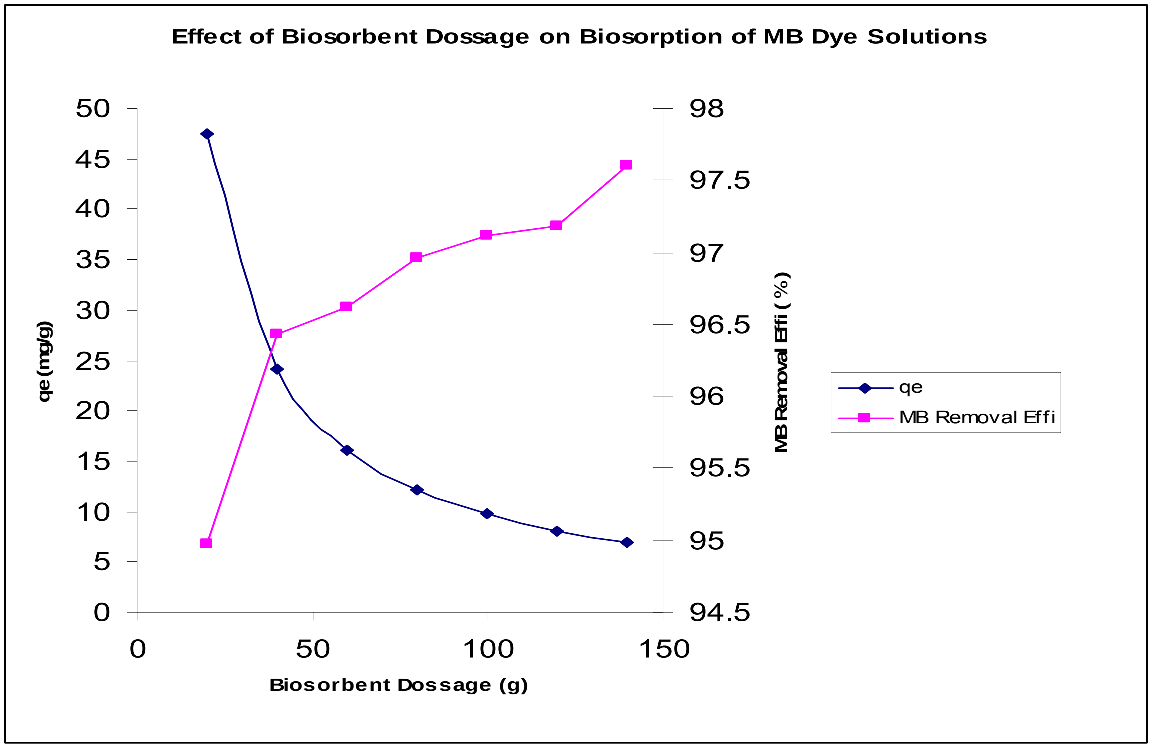

Figure 3: Effect of Biosorbent on MB Removal (q_e and MB removal Efficiency).

Effect of pH on the Biosorption of MB: The result obtained on the effect of pH on MB removal efficiency and q_e showed that both increases as the pH varies from 3 to 10 (with both values being least at pH = 3 and highest at pH = 10). This implies that there is a strong interaction between the biosorbent and the MB particles in solution that H^+ and OH^- could affect both MB removal efficiency and q_e.

As the pH increases, the number of the positively charged available biosorption sites decreases while the number of the negatively charged sites increases, the surface of the sites gets negatively charged thus enhances a stronger electrostatic force of attraction for the positively charged MB dye solution The lower biosorption capacity at highly acidic pH reveals the likelihood of the development of positive charge on the biosorbent which slows down the biosorption of MB over it. Also, the low biosorption of MB at acidic pH is as a result of the presence of excess H^+ competing with MB cations for the biosorption sites (Vansanth Kumar et al, 2006; Sun, Xue-Fei et al., 2008).

Effect of Temperature on Biosorption of MB: The MB removal efficiency and q_e increase with increase in temperature for all initial concentrations. In each concentration, both values are highest at 50°C and least at 30°C. Therefore, it denotes that biosorption equilibrium is a thermo-dependent process. This effect may be due to the fact that at higher temperatures, an increase in the movement of the solute occurs. Similar results have been reported by other researchers (Chakraborty, et al., 2005; Ncibi, M. C. et al., 2007).

Thermodynamic Studies on Biosorption of MB: The increase in Q^o (Langmuir constant related to biosorption capacity) with increase in temperature shows that the biosorption capacity of the MB on the biosorbent is enhanced by rise in temperature; this is so because of the increase in the surface activity and kinetic energy of the MB particles. On the other hand, b (Langmuir constant related to energy of biosorption) reduces as the temperature rises.

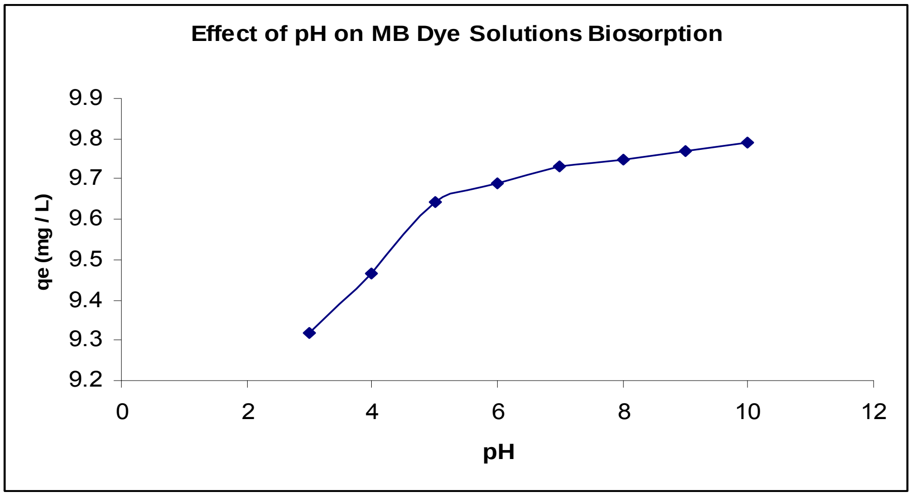

Figure 4a: Effect of pH on q_e.

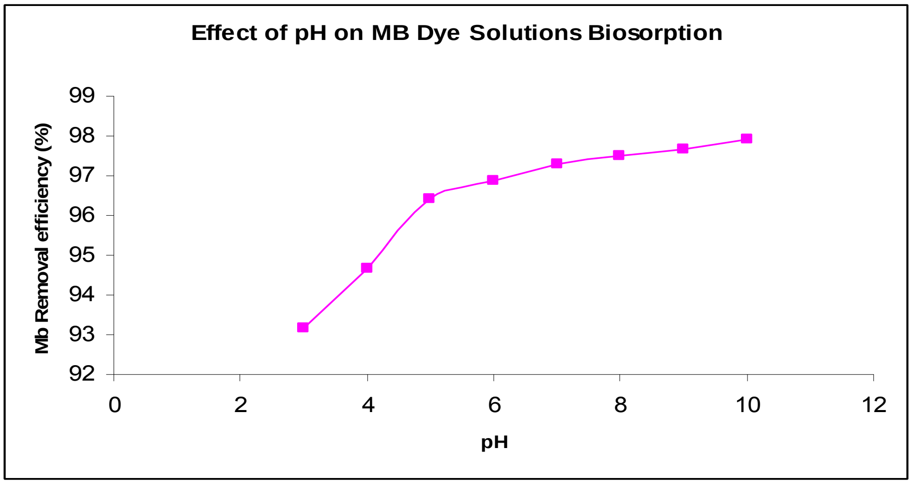

Figure 4b: Effect of pH on MB Removal.

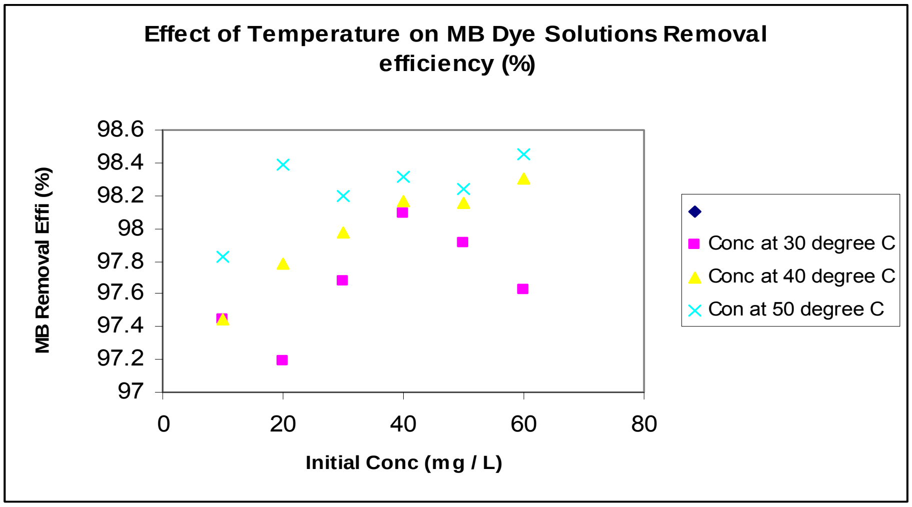

Figure 5: Effect of Temperature on MB Removal.

To ascertain if the biosorption phenomenon is favorable or unfavorable, for Langmuir type biosorption process, isotherms can be classified by R_L, a dimensionless constant separator factor (Stephen et al, 2006) stated as:

$$R_L = 1 / (1+ bC_o) \qquad \textbf{(5)}$$

According to Stephen et al., 2006; using mathematical calculation that R_L indicates the shape of isotherm to either be favourable ($R_L > 1$), linear ($R_L = 1$), irreversible ($R_L = 0$) or favourable ($0 < R_L < 1$). From the experiment, R_L varies from 0.027 to 0.138 for different MB dye concentrations (10 – 60 mg / L at different temperatures. These values ranged between 0 and 1, thus indicating a favorable biosorption.

The values of n (Freundlich exponent) were greater than 1, indicating that MB was adsorbed favorably by the biosorbent at all temperatures studied (Juang et al., 2006). The increase in the value of n with the increase in temperature shows that biosorption intensity increase as temperature increases. Also, value of n indicates better biosorption mechanism and formation of relatively stronger bond between adsorbate and biosorbent.

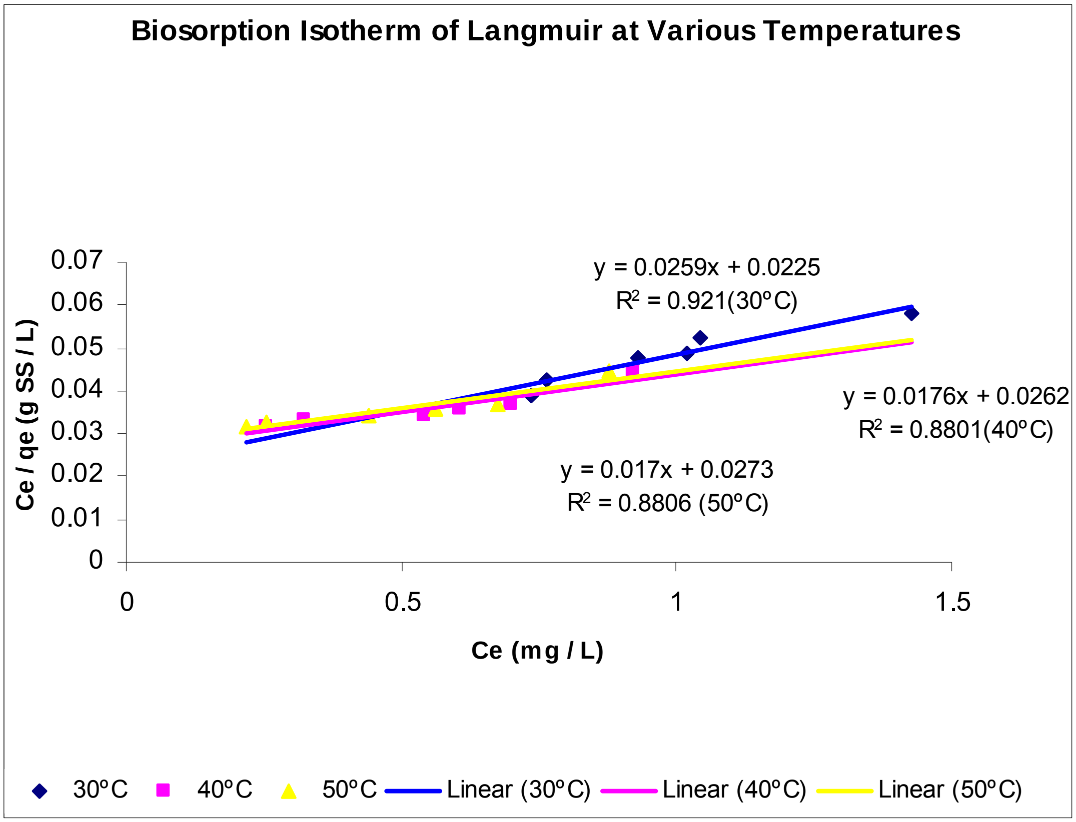

Figure 6: Linearized Biosorption Isotherms of Langmuir.

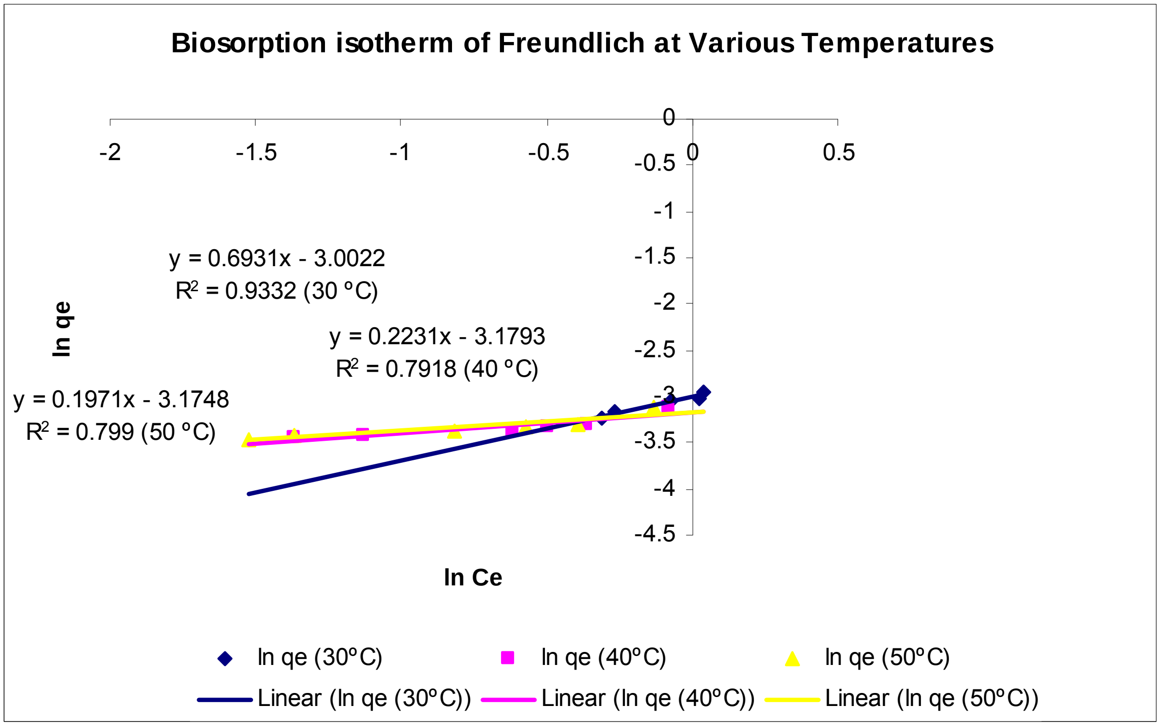

Figure 7: Linearized Biosorption Isotherms of Langmuir.

Table 3: Langmuir and Freundlich Isotherm Constants of MB Biosorption on Flamboyant Pod at Various Temperatures.

T (°K)	Langmuir			Freundlich		
	Q°	b	R^2	n	k_F	R^2
303	38.610	1.1511	0.9210	1.4427	0.0497	0.9332
313	56.818	0.6717	0.8801	4.4823	0.0416	0.7918
323	58.824	0.6227	0.8806	5.0736	0.0418	0.7990

Table 4: Isotherm Constants of MB Biosorption on Flamboyant Pod at Various Temperatures.

Thermodynamic Parameters			R^2
ΔG^0 (J/mol)	ΔH^0 (KJ/mol)	ΔS^0 (J/mol)	
-174.4578 (303°K)	-25.556	-82.556	0.8541
+651.1022 (313°K)			
+1476.6622 (323°K)			

The negative value of ΔH^0 indicates the exothermic nature of the biosorption process while the negative value of ΔS^0 implies that there was decreased randomness at solid/ solution interface during biosorption of MB on the biosorbent. The values of ΔG^0 obtained from thermodynamic analysis were found to be increasingly positive with increasing temperature; this indicates the presence of an energy barrier in the biosorption process. It has been suggested that a positive value for ΔG^0 is quite common when an ion-exchange mechanism applies in the biosorption of cationic sorbate because of the activated complex formed by the cationic sorbate with the biosorbent (Ozcan and Ozcan, 2004).

Kinetic Studies on the Biosorption of MB: In the kinetics study of the biosorption process, data from the kinetic studies were fitted into the pseudo-first-order and pseudo-second-order models. Kinetic biosorption of pollutants in wastewaters has been studied using predominantly pseudo-first-order (Langergren,

1898) and pseudo-second-order models (Ho and McKay, 2000). The linearized forms of pseudo-first-order and pseudo-second order models are giving by Equations 6 and 7, respectively.

$$\ln (qe-qt) = \ln qe - k_1 t \qquad \textbf{(6)}$$

$$(t / q_t) = 1 / (k_2 qe^2) + (t / q_e) \qquad \textbf{(7)}$$

where, k_1 is the rate constant of pseudo-first-order biosorption; q_e is the MB dye solutions uptake in mg/g at equilibrium; and q_t is the MB dye solutions uptake in mg/g at time, t.

A plot of ln (q_e-q_t) against t was made and values of k_1 and qe were obtained from the slope and intercept respectively. qe is pre-estimated by making a plot of qt against time, t , which gives a parabolic curve whose qt value plateau to qe at infinite time t. k_2 is the rate constant of pseudo-second order biosorption. A plot of (t/qt) against t gives (1/qe) as slope and $(1/ k_2 qe^2)$ as intercept from which k_2 can be obtained**.** Both models are tested for suitability using their correlation coefficient, R^2 (Ho and McKay, 2000).

As can be seen from Table 5, the calculated *qe* determined from the plot of the pseudo-first-order model for MB dye solutions at various concentrations differs from the experimental *qe*). This implies that the model is not very good in explaining the kinetics of the biosorption of the metals. On the other hand, the pseudo-second-order model as shown in Table 3 fits the kinetics better, as their correlation coefficient is close to 1 (i.e., 0.999 for MB dye solutions at various concentrations. The calculated qe are very close to the experimental qe. All these imply that second order kinetic best explain the observed rate, suggesting that biosorption is the rate-limiting step; and that biosorption of the MB dye solutions involves two species, in this case, the MB dye particles and the biosorbent particles (Wallace, et al. 2003).

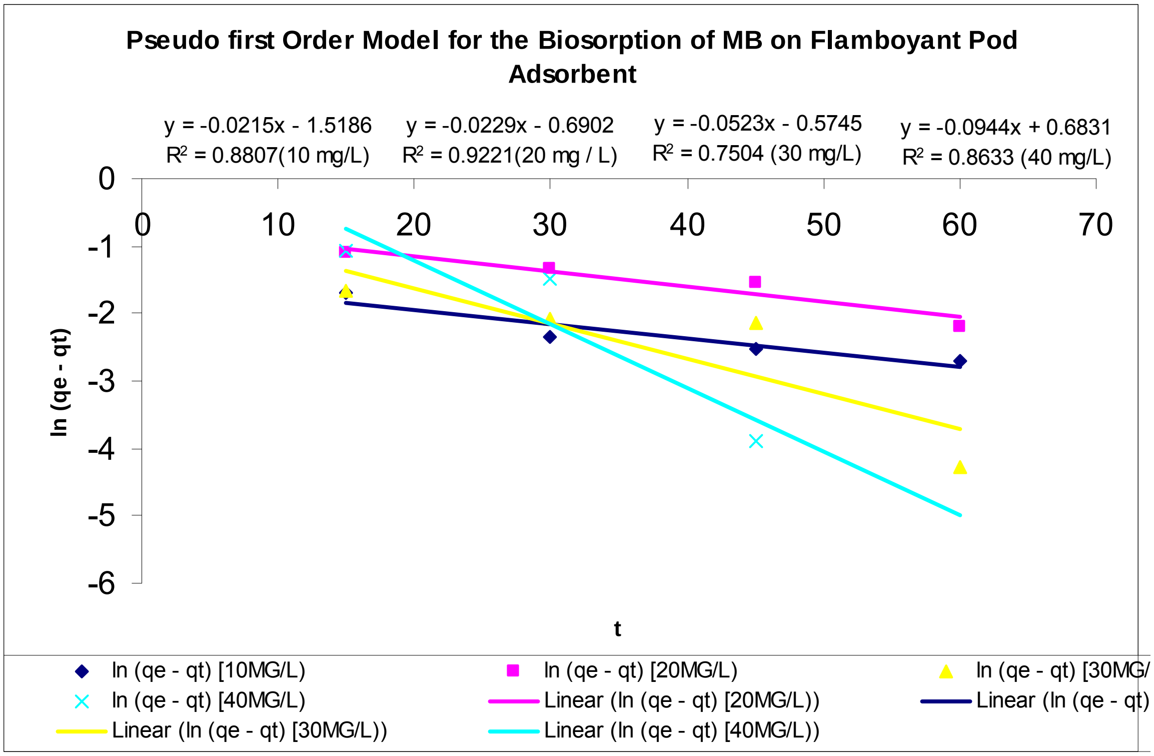

Figure 8: Linearized Pseudo-First Order Kinetic Model for Biosorption of MB Dye Solutions on *Delonix regia.*

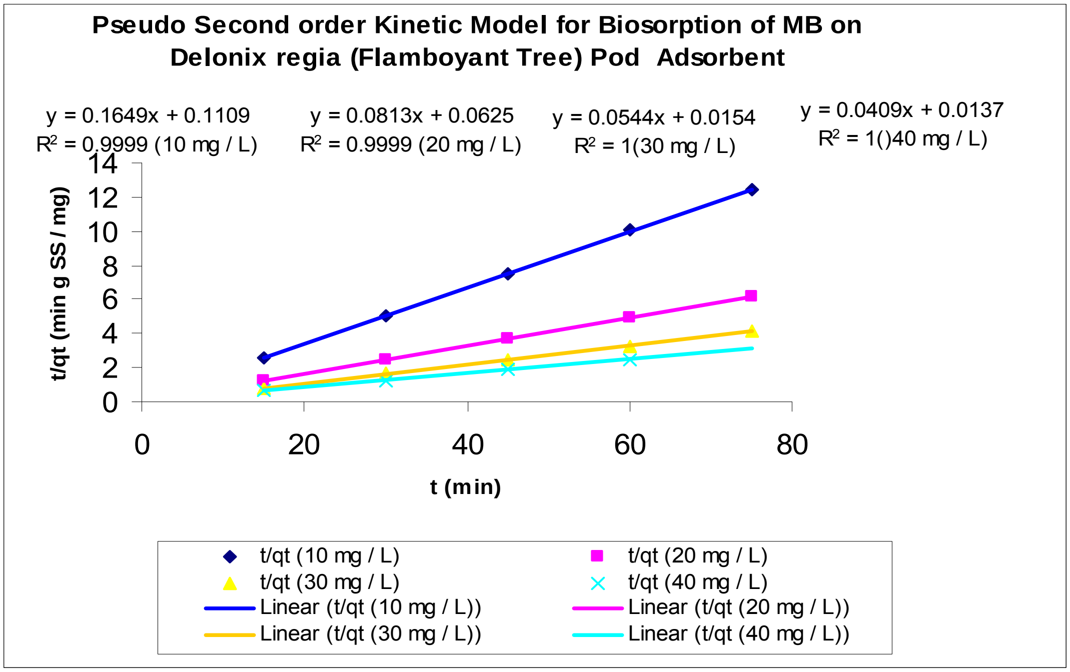

Figure 9: Linearized Pseudo-Second Order Kinetic Model for Biosorption of MB Dye Solutions on *Delonix regia.*

Table 5: Kinetics Parameters of MB Biosorption on Flamboyant Pod at Various Temperatures.

	Pseudo-first order			**Pseudo-second order**			**Observed**
C_o(mg / L	**K_1 (1/min)**	**q_e (mg/ L)**	**R^2**	**K_2(g/mg min)**	**q_e (mg/ L)**	**R^2**	**q_e**
10	0.16	0.219	0.88	0.25	6.06	0.99	6.03
20	0.08	0.501	0.92	0.11	12.30	0.99	12.21
30	0.05	0.563	0.75	0.19	18.38	1.00	18.32
40	0.04	1.980	0.86	0.12	24.39	1.00	24.28

CONCLUSION

This research entailed the equilibrium, kinetic and thermodynamic studies of the biosorption of MB dye solutions on *Delonix regia* (flamboyant tree) pod biosorbent. The results showed that *Delonix regia* pod biosorbent proved to be a very effective biosorbent in the removal of MB dye from waste waters.

- Biosorption of MB dye solutions was, to a large extent, dependent on initial concentration, biosorbent dosage, pH and temperature
- Biosorption of MB dye solutions obeyed Langmuir and Freundlich isotherms. R_L value from Langmuir and n from Freundlich isotherms shows that biosorption of MB dye solutions on *Delonix regia* is favorable. Maximum biosorption capacity of MB dye solutions on *Delonix regia* is 34.27215.
- Thermodynamic studies confirmed that the biosorption process was exothermic and spontaneous at room temperature and near room temperature.
- Batch kinetic studies carried out revealed that the kinetic data tended to fit well in second order kinetics, confirming the chemisorption of dye solutions on *Delonix regia.*

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