

## Biosorption of Malachite Green from aqueous solution using *Psidium Guajava*. L

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### ABSTRACT

The biosorption of Malachite green from aqueous solution by *Psidium guajava* L.f. was studied in a batch adsorption system as a function of contact time, pH, initial dye ion concentration, biosorbent dosage, biosorbent size and temperature. The Langmuir, Freundlich, Redlich-Peterson and Temkin adsorption models were applied to describe the isotherms and isotherm constants. Biosorption isothermal data were well interpreted by Freundlich model. The kinetic experimental data were properly fitted with pseudo-second-order kinetic model. Various thermodynamic parameters such as  $\Delta G$ ,  $\Delta H$  and  $\Delta S$  were calculated.

**Keywords:** Biosorption, Malachite green, *Psidium guajava* L.f., Isotherms, Kinetics, Thermodynamic parameters.

### INTRODUCTION

Pollution control is the prime concerns of society today. Untreated waste water in the natural ecosystem poses serious environmental problems today. Among all the industries dyeing industrial effluents are more toxic and may cause serious hazardous effect on the aquatic life. Dyes are the amazing coloring materials that add colors to a variety of items. People are attracted to use colours, as a result they consume large amount of colours. Many industries, such as dyestuffs, textile, paper and plastics use dyes in order to color their products and also consume substantial volumes of water. As a result, they generate a considerable amount of colored wastewater. It is recognized that public perception of water quality is greatly influenced by the color.

Color is the first contaminant to be recognized in wastewater [1]. The presence of very small amounts of dyes in water (less than 1 ppm for some dyes) is highly visible and undesirable [2]. Many of these dyes are also toxic and even carcinogenic and this poses a serious hazard to aquatic living organisms [3]. The major effect of dyes on humans is, they cause severe mutagenic and carcinogenic effects. Due to increasingly stringent restrictions on the organic content of industrial effluents, it is necessary to eliminate dyes from wastewater before it is discharged [4].

During the past three decades, several physical, chemical and biological decolorization methods have been reported. Few methods have been accepted by the paper and textile industries [5]. Amongst the numerous techniques of dye removal, adsorption is the procedure of choice and gives the best results as it can be used to remove different types of coloring materials [6]. In the present investigation untreated *Psidium guajava* L.f. leaves powder was examined in an attempt to remove Malachite green from its aqueous solution.

Malachite green is basic (cationic) dye. Basic dyes are the salts of the coloured organic bases containing amino and imino group and able to combined with a colourless acid, such as hydrochloric and sulphuric acid. Malachite green is 4-[(4-dimethyl amino phenyl)-N,N-dimethyl-aniline]<sup>[7]</sup>. It is a toxic chemical primarily used as dye.

**Formula** :  $C_{52}H_{52}N_4O_{12}$   
**Molecular weight** : 927.02 g/mol  
**Absorption maximum** : 554 nm

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Many researchers have done the research work on removal of dyes from aqueous solutions using different adsorbents [8-26], but no information was available for the removal of Malachite green from aqueous solution using *Psidium guajava* L.f. as adsorbent.

The aim of the present study is to remove Malachite green from aqueous solution using *Psidium guajava* L.f. as adsorbent material for various parameters like contact time, pH, initial dye ion concentration, biosorbent dosage, biosorbent size and temperature.

### MATERIALS AND METHODS

#### 1. Biosorbent:

Guava or *Psidium guajava* L.f. is of Myrtaceae family. It is a tropical and semi tropical plant. It is common in backyards and waste places. Its seeds and leaves possess medicinal value and are traditionally used to treat a number of human ailments.

#### 2. Preparation of biosorbent:

The green colored guava (*Psidium guajava*) leaves were collected from the mangalagiri rural areas, Guntur Dt. The collected Guava leaves were washed with deionized water several times to remove dirt particles. The washed leaves were then completely dried in sunlight for 20 days. The dried leaves were then cut into small pieces and powdered using domestic mixie. In the present study the powdered materials in the range of 75-300  $\mu$ m. average particle size were directly used as biosorbent without any pretreatment.

#### 3. Chemicals:

Malachite green was purchased from Universal Laboratories, Mumbai. Stock solutions of Malachite green were prepared by dissolving Malachite green in distilled water. The range of concentration of the prepared dye solutions varied between 20 and 100 mg/L. The pH of solution was adjusted with 0.1N HCl and NaOH. Blank experiments were conducted to ensure that no adsorption was taking place on the walls of the apparatus used.

#### 4. Biosorption Experiments:

Batch biosorption experiments were conducted in a rotary shaker at 180 rpm using 250 mL Erlenmeyer flasks containing 30 mL of different Malachite green concentrations. All the experiments were carried out at room temperature ( $30 \pm 1^\circ\text{C}$ ). Sample was taken at different timings upto 3 hours of contact time and the reaction mixture was centrifuged for 5 min and then filtered with whatman filter paper. After 60 min of contact (according to the preliminary sorption dynamics tests), with 0.1 g *Psidium guajava* L.f. an equilibrium was reached. The dye content in the supernatant

was determined using a UV-visible spectrophotometer (model-Chemito 2100, Australia) after filtering the adsorbent with whatman filter paper. The amount of dye adsorbed by *Psidium guajava* L.f. was calculated from the differences between dye quantity added to the biomass and dye content of the supernatant using the following equation:

$$q = (C_i - C_f) \frac{V}{M} \quad (1)$$

Where  $q$  is the dye uptake (mg/g);  $C_i$  and  $C_f$  are the initial and equilibrium dye concentrations in the solution (mg/L), respectively;  $V$  is the solution volume (L); and  $M$  is the mass of biosorbent (g).

The Langmuir [27] sorption model was chosen for the estimation of maximum Malachite green sorption by the biosorbent. The Langmuir isotherm can be expressed as,

$$q_{eq} = \frac{q_{max} bC_{eq}}{1 + bC_{eq}} \quad (2)$$

Where  $q_{max}$  indicates the monolayer adsorption capacity of adsorbent (mg/g) and the Langmuir constant  $b$  (L/mg) is related to the energy of adsorption. For fitting the experimental data, the Langmuir model was linearized as

$$\frac{C_{eq}}{q_{eq}} = \frac{1}{bq_{max}} + \frac{1}{q_{max}} C_{eq} \quad (3)$$

The Freundlich [28] model is represented by the equation,

$$q_{eq} = K_f C_{eq}^m \quad (4)$$

Where  $K_f$  (mg/g) is the Freundlich constant related to adsorption capacity of adsorbent and  $m$  is the Freundlich exponent related to adsorption intensity (dimensionless). For fitting the experimental data, the Freundlich model was linearized as follows,

$$\log q_{eq} = \log K_f + m \log C_{eq} \quad (5)$$

Jossens et al [29] modified the three parameter isotherm proposed by Redlich and Peterson [30] to incorporate features of both the Langmuir and Freundlich equations. The Redlich-Peterson model is represented as follows:

$$q_{eq} = \frac{AC_{eq}}{1 + BC_{eq}^g} \quad (6)$$

where  $A$ (L/g) and  $B$ (L/mg) are the Redlich-Peterson isotherm constants and  $g$  is the Redlich Peterson isotherm exponent, which lies between 0 and 1. The linearized form of equation is given by:

$$\ln \left( \frac{AC_{eq}}{q_{eq}} - 1 \right) = g \ln (C_{eq}) + \ln (B) \quad (7)$$

There are three unknown parameters  $A$ ,  $B$  and  $g$  in Redlich-Peterson isotherm equation. Therefore a minimization procedure is adopted to maximize the coefficient of determination, between the theoretical data for  $q_{eq}$  predicted from the linearized form of Redlich-Peterson isotherm equation and the experimental data.

The Temkin [31] isotherm has generally been applied in the following form,

$$q_{eq} = \frac{RT}{b_T} \ln (A_T C_{eq}) \quad (8)$$

where  $A_T$  (L/mg) and  $b_T$  are Temkin isotherm constants.

### 5. Biosorption Kinetics:

The kinetics studies were carried out by conducting batch biosorption experiments with different initial Malachite green concentrations. Samples were taken at different time periods and analyzed for their Malachite green concentration.

## RESULTS AND DISCUSSION

### 1. Effect of contact time:

Time course profiles for the biosorption of Malachite green for solutions of 20 mg/L, 40 mg/L, 60 mg/L, 80 mg/L and 100 mg/L are shown in Fig-1. The data showed that a contact time of 75 min for Malachite green was required to achieve an optimum

biosorption and there was no significant change in concentration of the dye solution with further increase in contact time. Therefore, the uptake and unadsorbed Malachite green concentration at the end of 75 min are given as the equilibrium values,  $q_{eq}$  (mg/g) and  $C_{eq}$  (mg/L). For further studies of biosorption with other variable parameters the optimum time of 75 min has been chosen for contact period.

### 2. Effect of pH:

It is well known that the pH of the medium affects the solubility of dye and the concentration of the counter ions, on the functional groups of the biomass cell walls. Thus pH is an important parameter on biosorption of dye ions from aqueous solutions. *Psidium guajava* L.f. presents a high content of ionizable groups (carboxyl groups) on the cell wall polysaccharides, which makes it very liable to the influence of the pH. As shown in Figs-3 and 4, the biosorption was decreased with increase in the pH from 1 to 8 and the maximum % biosorption was found at pH = 1. The maximum uptake (23.67) was found with 100 mg/L at pH = 1 as shown in Fig-4. Two possible mechanisms of biosorption of Malachite green on *Psidium guajava* L.f. may be considered: (a) electrostatic interaction between the protonated groups of carbon and acidic dye and (b) the chemical reaction between the adsorbate and the adsorbent. At pH 1.0 a significantly high electrostatic attraction exists between the positively charged surface of the adsorbent and anionic dye. As the pH of the system increases, the number of negatively charged sites increases and the number of positively charged sites decreases. A negatively charged surface site on the adsorbent does not favour the biosorption of dye anions due to the electrostatic repulsion. Also, lower biosorption of Malachite green at alkaline pH is due to the presence of excess OH<sup>-</sup> ions competing with the dye anions for the biosorption sites. At alkaline pH significant biosorption of the anionic dye on the adsorbent still occurred. This suggests that the second mechanism, i.e. chemisorption, might be operative. A similar trend was observed for the removal of Malachite green from water by adsorption onto activated carbon prepared from coir pith [23], Acid Brilliant Blue [32], and Acid Violet [33] on biogas residual slurry and biosorption of Malachite green on waste orange peel [24] and banana pith [34] and biosorption of Acid Violet on coir pith carbon [35].

### 3. Effect of initial dye concentration:

Experiments were undertaken to study the effect of the initial dye concentration on Malachite green removal kinetics from the solution. The results obtained are shown in Fig-5. The obtained curves showed that the dye uptake increases with increase in initial concentration of Malachite green while the percentage removal of Malachite green decreases with an increase in initial Malachite green concentration. The increase of dye uptake is a result of the increase in the driving force i.e. concentration gradient, with an increase in the initial dye concentrations (from 20 to 100 mg/L). However, the percentage biosorption of Malachite green on *Psidium guajava* was decreased from 92.7 to 78.9%. Though an increase in dye uptake (5.562 to 23.67) was observed, the decrease in percentage biosorption may be attributed to lack of sufficient surface area to accommodate much more dye available in the solution. The percentage biosorption at higher concentration levels shows a decreasing trend whereas the equilibrium uptake of dye displays an opposite trend. At lower concentrations, all Malachite green present in solution could interact with the binding sites and thus the percentage biosorption was higher than those at higher initial Malachite green concentrations. At higher concentrations, lower biosorption yield is due to the saturation of biosorption sites. As a result, diluting the wastewaters containing high dye concentrations can increase the purification yield.

$$\% \text{ Biosorption} = \frac{(C_i - C_f)}{C_i} \times 100 \quad (9)$$

The dye uptake was calculated by the simple concentration difference method. The initial concentration  $C_i$  (mg/L) and the final dye concentration  $C_f$  (mg/L) at any time were determined and the dye uptake  $q_{eq}$  (mg dye adsorbed /g adsorbent) was calculated from the mass balance as follows:

$$q_{eq} = \frac{V (C_i - C_{eq})}{1000 w} \quad (10)$$

### 4. Effect of biosorbent dosage:

For studying the effect of biosorbent dosage on removal of Malachite green, the biosorbent dosage is changed from 0.02 to 0.1 g, fixing other parameters like initial concentration at 20 mg/L,

pH = 1 and biosorbent size 75µm. The contact time was 75 min for Malachite green as stated in the earlier article 4.1 in this chapter. The biosorption plot of Fig-6 shows an increase in % biosorption with an increase in biosorbent dosage. This is because of the availability of more binding sites for complexation of dye ions. The maximum uptake (24.49) for w = 0.1 g and minimum uptake (5.562) for w = 0.02 g were obtained.

**5. Effect of biosorbent average particle size**

Adjusting the initial concentration of aqueous solution at 20 mg/L, pH = 1, the fixed volume of 30 ml solution is allowed for 75 min for Malachite green biosorption with 0.1 g for biosorbent size 75 µm. Similar experimental runs were carried out for the remaining biosorbent sizes 150 µm, 212 µm and 300 µm. The %biosorption was decreased from 92.7 to 79.57 with increase in the biosorbent size. The dye uptake was decreased from 5.562 to 4.774 with increase in the biosorbent size as shown in Fig-7. This is due to less surface area available with increased particle size, thus reducing the biosorption.

**6. Effect of temperature:**

When the biosorption was carried out at four different temperatures from 303 to 333 K with an interval of 10 K for concentrations ranging from 20 mg/L -100 mg/L, the extent of biosorption is decreased steadily with an increase in biosorption temperature (Figs- 8 and 9). The Malachite green biosorption on the *Psidium guajava* L.f. was definitely exothermic in nature. When the biosorption is exothermic, an increase in temperature results in a decrease in  $K_L$ . This indicated a shift of the biosorption equilibrium to the biosorption direction. Increase in temperature is followed by a decrease in diffusivity of dye ion, and consequently by a decrease in the biosorption rate if diffusion is the rate controlling step. The amount of dye uptake was decreased from 23.67 - 18.54 mg/g for a temperature rise of 303°K-333°K. This decrease in binding could be due to decrease in surface activity and decreased kinetic energy of the dye molecules.

**Biosorption equilibrium:**

The equilibrium biosorption of Malachite green onto *Psidium guajava* L.f. as a function of initial dye ion concentration of Malachite green is shown in Fig-10. Different isotherms like Langmuir, Freundlich, Redlich-Peterson and Temkin models were used to describe the biosorption isotherms. The calculated results of each model were given in Table.1.

It was seen that the Freundlich adsorption isotherm is well fitted followed by Langmuir and Temkin with the concentration range studied. The Redlich-Peterson isotherm in not fitted with experimental data.

**Biosorption kinetics:**

The prediction of biosorption rate gives important information for designing batch biosorption systems. Information on the kinetics of solute uptake is required for selecting optimum operating conditions for full-scale batch process. Fig-2 shows the plot between dye uptake, q (mg/g) versus time, t (min) for initial solute concentration of 20 mg/L. From the figure it was observed that q value increased with increase in contact time. The kinetics of the biosorption 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. These models are explained as follows:

**1. Pseudo-first-order model:**

The possibility of biosorption data following Lagergren [36] pseudo-first order kinetics is given by:

$$\frac{dq}{dt} = K_1(q_{eq} - q) \tag{11}$$

Integrating Eq. (11) with respect to integration conditions q=0 to q=q at t=0 to t=t, the kinetic rate expression becomes:

$$\log(q_{eq} - q) = \log q_{eq} - \frac{K_1}{2.303} t \tag{12}$$

In order to obtain the rate constant, the straight-line plot (not shown) of time versus log (q<sub>eq</sub>-q) was made for *Psidium guajava* for initial Malachite green concentration, 20 mg/L. The intercept of the above plot should equal to log q<sub>eq</sub>. However, if q<sub>eq</sub> from intercept does not equal to the equilibrium Malachite green dye uptake then the reaction is not likely to be first order, even this

plot has high correlation coefficient with the experimental data. Correlation coefficients were found to be 0.955.

**2. Pseudo-second-order model:**

A pseudo-second order model proposed by Ho and McKay [37] was used to explain the sorption kinetics. This model is based on the assumption that the biosorption follows second order chemisorption. The pseudo-second order model can be expressed as:

$$\frac{dq}{dt} = K_2(q_{eq} - q)^2 \tag{13}$$

Separating the variables in Eq. (13) gives:

$$\frac{dq}{(q_{eq} - q)^2} = K_2 dt \tag{14}$$

Integrating Eq. (14) for the boundary conditions q=0 to q=q at t=0 to t=t, Eq. (14) simplifies to:

$$\frac{t}{q} = \frac{1}{K_2 q_{eq}^2} + \frac{1}{q_{eq}} t \tag{15}$$

Where  $t$  is the contact time (min),  $q_{eq}$  (mg/g) and  $q$  (mg/g) are the amounts of dye adsorbed at equilibrium and at any time, t. Correlation coefficients were found to be 0.999 for Malachite green, for initial concentration 20 mg/L. If second order kinetics is applicable, the plot (Fig-11) of time versus t/q of equation (15) should give a linear relationship from which the constants  $q_{eq}$  and  $K_2$  can be determined. The rate constants and the correlation coefficients of the dye and for both tested models have been calculated and summarized in Table-2.

A comparison of the maximum capacity  $q_{max}$  of *Psidium guajava* L.f. with those of some other adsorbents reported in literature is given in Table 3. Differences of dye uptake are due to the properties of each adsorbent such as structure, functional groups and surface area.

**Thermodynamic parameters:**

In environmental engineering practice, both energy and entropy factors must be considered in order to determine what processes will occur spontaneously. Gibb's free energy change ( $\Delta G^\circ$ ) is the fundamental criterion of spontaneity. Reaction occurs spontaneously at a given temperature if  $\Delta G^\circ$  is negative. The thermodynamic parameters such as Enthalpy change ( $\Delta H^\circ$ ), free energy change ( $\Delta G^\circ$ ) and entropy change ( $\Delta S^\circ$ ) can be estimated using equilibrium constants changing with temperature. These can be calculated by using the following equations:

$$\Delta G^\circ = -RT \ln K_a \tag{16}$$

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{17}$$

$$\ln K_a = \frac{-\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{18}$$

Eq (18) shows clearly that the biosorption process is composed of two contributions, enthalpic change and entropic, which characterize whether the reaction is spontaneous [38]. The free energy change for Malachite green dye ions on to *Psidium guajava* was determined using the equilibrium constant obtained from Langmuir isotherm model. The  $\Delta G^\circ$  and  $T\Delta S^\circ$  values of Malachite green dye ions at different temperatures 303K-333K and of pH (1.0) are given in table-4. However, the entropic contribution is even larger than the free energy of biosorption ( $\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ > 0$ ) it can be said that the biosorption of Malachite green on *Psidium guajava* is exothermically governed.

From the plot of 1/T versus ln  $K_a$  ( Fig-12), the standard enthalpy change  $\Delta H^\circ$  was obtained as -4.274 kJ /mol, while the standard entropy change  $\Delta S^\circ$  was determined as -0.191 kJ /mol K at pH 1.0. The value of  $\Delta H^\circ$  is negative, indicating that the biosorption reaction is exothermic in nature.

**Nomenclature:**

- $C$  : Concentration of solute in the solution.
- $q_t$  : Concentration of solute in the biosorbent at any time t.
- $C_i$  : Initial concentration of Malachite green solution mg/L.
- $C_f$  : Final concentration of Malachite green mg/L.
- $C_{eq}$  : Equilibrium concentration of Malachite green, mg/L.

$q_{eq}$  : Amount biosorbed per unit weight of biosorbent at equilibrium, mg/g.  
 $t$  : Time of contact, min.  
 $W$  : Weight of biosorbent, g.  
 $V$  : Volume of solution, ml.  
 $D_p$  : Biosorbent size,  $\mu\text{m}$ .  
 $T$  : Temperature of the dye solution, K.  
 $R$  : Correlation coefficient.  
 $q_{max} = q_{eq}$ : for a complete monolayer, mg/g.

$b$  : Sorption equilibrium constant, L/mg.  
 $k_f$  : Freundlich isotherm coefficient.  
 $m$  : intensity.  
 $A, B$  : Redlich-Peterson isotherm constants, L/mg.  
 $g$  : Redlich-Peterson isotherm exponent.  
 $A_T$  : Temkin isotherm constant, L/mg.  
 $b_T$  : Temkin isotherm constant.  
 $k_1$  : Pseudo first order constant, 1/min.  
 $k_2$  : Pseudo second order constant, g/mg min

Tables and Graphs:

Table No. 1: Langmuir, Freundlich and Redlich-Peterson models estimated from the fittings of experimental points of Malachite green Biosorption.

Langmuir model	Freundlich model	Redlich-Peterson model	Temkin model
$q_{max} = 32.148$	$m = 0.5513$	$g = -1.023$	$b_T = 367.11$
$b = 0.127$	$K_f = 4.708$	$B = 5.2858$	$A_T = 1.348$
$R^2 = 0.9922$	$R^2 = 0.9924$	$R^2 = 0.24$	$R^2 = 0.98306$

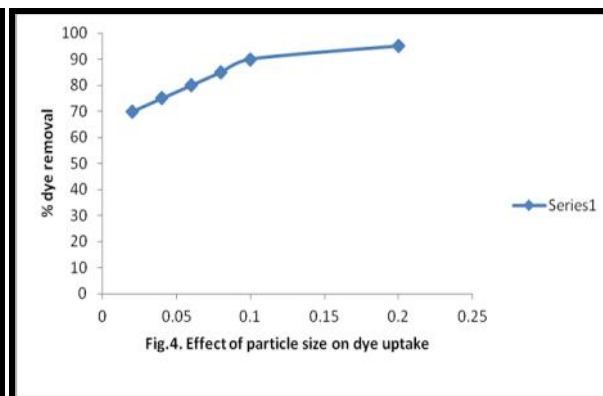
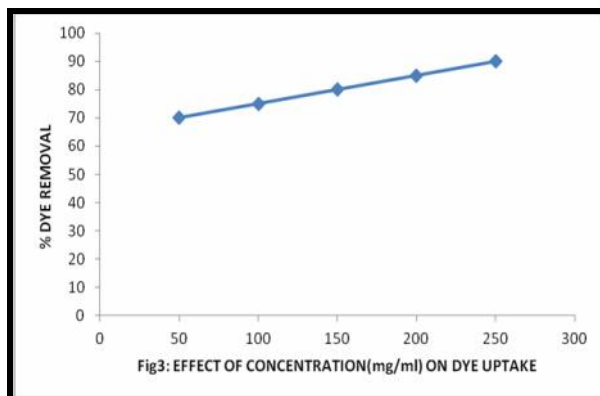
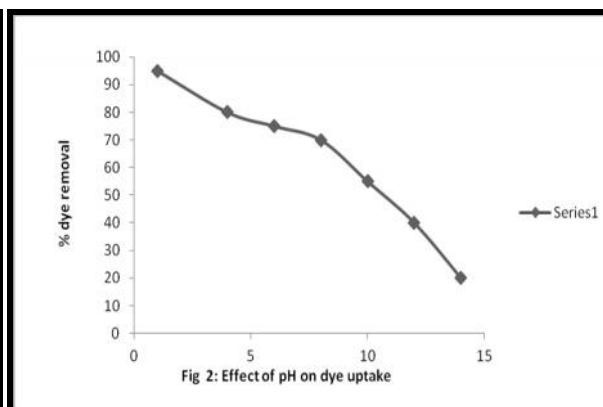
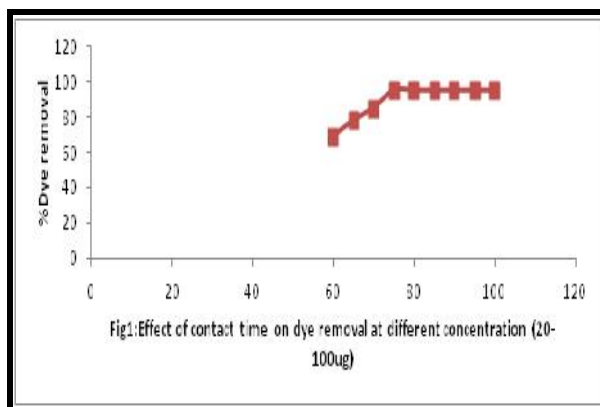
Table No. 2: Comparison of kinetic parameters

Parameter	20 mg/L		40 mg/L		60 mg/L		80 mg/L		100 mg/L	
	P <sub>1</sub>	P <sub>2</sub>	P <sub>1</sub>	P <sub>2</sub>	P <sub>1</sub>	P <sub>2</sub>	P <sub>1</sub>	P <sub>2</sub>	P <sub>1</sub>	P <sub>2</sub>
Rate constant	0.087	0.161	0.067	0.099	0.079	0.044	0.071	0.033	0.052	0.03
$q_{calc}$	1.730	5.655	1.600	10.889	3.996	15.87	4.766	20.50	4.258	24.06
$q_{exp}$	5.562	5.562	10.752	10.752	15.54	15.54	20.08	20.08	23.67	23.67
$R^2$	0.955	0.999	0.951	0.999	0.944	0.999	0.935	0.999	0.952	0.999

P<sub>1</sub> - Pseudo 1<sup>st</sup> order kinetics; P<sub>2</sub> - Pseudo 2<sup>nd</sup> order kinetics

Table No. 3: Thermodynamic parameters

Temperature (K)	$k_a$ (L/mg)	$\Delta G^0$ (kJ/mol)	$-T\Delta S^0$ (kJ/mol)
303	0.127	1.542	57.87
313	0.073	1.733	59.78
323	0.072	1.925	61.69
333	0.070	2.117	63.60



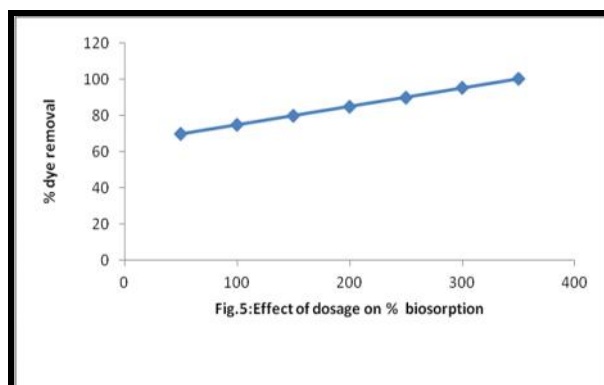


Fig.5:Effect of dosage on % biosorption

## CONCLUSIONS

The present study shows that *Psidium guajava* L.f. is an effective and cheap biosorbent for removal of Malachite green from aqueous solution. The biosorption performances are strongly affected by parameters such as pH, initial concentration, biosorbent dosage, biosorbent average particle size and temperature. The maximum biosorption takes place at pH 1. The dye uptake was increased with increasing the initial concentration of dye. The percentage biosorption increases with increase in the biosorbent dosage and decreases with increase in the average particle size of the biosorbent. The percentage dye removal of Malachite green decreases with increase in the temperature. Freundlich adsorption isotherm is well fitted followed by Langmuir and Temkin with the range studied. The kinetics of the biosorption of Malachite green was better described with Pseudo-second-order kinetics. The thermodynamic parameters such as  $\Delta G^0$ ,  $\Delta H^0$  and  $\Delta S^0$  were calculated and the adsorption process was exothermic.

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