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SHORT RESEARCH COMMUNICATION

Insight into biosorption equilibrium, kinetics and thermodynamics of crystal violet onto *Ananas comosus* (pineapple) leaf powder

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Abstract Biosorption performance of pineapple leaf powder (PLP) for removal of crystal violet (CV) from its aqueous solutions was investigated. To this end, the influence of operational parameters such as pH, biosorbent dose, initial dye concentration and temperature were studied employing a batch experimental setup. The biosorption process followed the Langmuir isotherm model with high correlation coefficients ($R^2 > 0.99$) at different temperatures. The maximum monolayer biosorption capacity was found to be 78.22 mg g^{-1} at 293 K. The kinetic data conformed to the pseudo-second-order kinetic model. The activation energy of the system was calculated as 58.96 kJ mol^{-1}, indicating chemisorption nature of the ongoing biosorption process. A thermodynamic study showed spontaneous and exothermic nature of the biosorption process. Owing to its low cost and high dye uptake capacity, PLP has potential for application as biosorbent for removal of CV from aqueous solutions.

Keywords Biosorption · Pineapple leaf powder · Crystal violet · Equilibrium · Kinetics · Thermodynamics

Introduction

The release of synthetic dye stuffs through the wastewater streams of industries such as textile, leather, rubber, paper, printing, paint, plastic, pigments, food and cosmetics is a serious global concern (Chowdhury et al. 2011a). This is mainly because of their negative ecotoxicological effects into the receiving water bodies and bioaccumulation in wildlife (Saha et al. 2010). Therefore, such dye effluent stream requires proper treatment prior to discharge.

In recent years, biosorption has been strongly recommended by researchers worldwide as an efficient and economically sustainable technology for the removal of synthetic dyes from industrial effluents (Farooq et al. 2010; Rafatullah et al. 2010; Demribas 2009; Gupta and Suhas 2009). A number of non-conventional low-cost materials, particularly agricultural waste/by-products such as rice husk, rice bran, wheat bran, orange peel, banana pith, banana peel, plum kernels, apple pomace, wheat straw, sawdust, coir pith, sugarcane bagasse, tea leaves, bamboo dust etc. have been proposed by several workers as effective biosorbents for the removal of dyes from their aqueous solutions (Gupta and Suhas 2009; Chowdhury et al. 2010).

Pineapple (*Ananas comosus*) is largely cultivated in tropical countries like India, China, Thailand, Indonesia and Taiwan and is consumed worldwide (Chowdhury et al. 2011b). Upon harvest, the leaves and stem cause potential disposal problems since they exist in enormous quantities and have no practical utility. Although direct open burning in fields is a common option for disposal, but this alternative causes serious air pollution. Thus the use of pineapple wastes as biosorbent is an attractive alternative from both economical and environmental point of view. Our previous study demonstrates that pineapple leaves in powdered form could be employed as an effective biosorbent for the removal of Basic Green 4 from aqueous solutions (Chowdhury et al. 2011b). Hence, a further attempt of the feasibility of applying pineapple leaf powder (PLP) for the removal of crystal violet (CV) dye from aqueous solution was explored in the present study. The study includes an evaluation of the effects of various

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operational parameters such as initial dye concentration, biosorbent dose, temperature and pH on the dye biosorption process employing a batch experimental setup. Biosorption isotherms and kinetics of the sorption process were studied. Also, thermodynamic and activation parameters were calculated in order to estimate the performance and predict the mechanism of the biosorption process.

Experimental

Biosorbent

Mature pineapple leaves were collected from the local farmlands in Durgapur, West Bengal, India. The leaves were first thoroughly washed with tap water to remove dust, dirt and any unwanted particles. The leaves were then sun dried and subsequently oven dried at 363 ± 1 K for 24 h. The dried leaves were ground to fine powder using a grinder and sieved to a constant size (100–125 μm) and used as biosorbent without any pretreatment for CV biosorption. The characterization of the biosorbent has been previously reported (Chowdhury et al. 2011b).

Dye

Crystal violet (CV) used in this study was of commercial quality (CI 42555, MF: $C_{25}H_{30}N_3Cl$, MW: 408, λ_{max}: 580 nm) and was used without further purification. Stock solution (1,000 mg L^{-1}) was prepared by dissolving accurately weighed quantity of the dye in double-distilled water. Experimental dye solution of different concentrations was prepared by diluting the stock solution with suitable volume of double-distilled water. The initial solution pH was adjusted using 0.1 M HCl and 0.1 M NaOH solutions.

Batch biosorption experiments

Batch biosorption experiments were carried out in 250 mL glass-stopperred, Erlenmeyer flasks with 100 mL of working volume, with a concentration of 50 mg L^{-1}. A weighed amount (2 g) of biosorbent was added to the solution. The flasks were agitated at a constant speed of 150 rpm for 3 h in an incubator shaker (Innova 42, New Brunswick Scientific, Canada) at 303 ± 1 K. The influence of pH (3.0–10.0), initial dye concentration (20–100 mg L^{-1}), biosorbent dose (0.5–5 g L^{-1}) and temperature (293–313 K) were evaluated during the present study. Samples were collected from the flasks at predetermined time intervals for analyzing the residual dye concentration in the solution. The residual amount of dye in each flask was investigated using UV/VIS spectrophotometer (U–2800, Hitachi, Japan). The amount of dye adsorbed per unit PLP (mg dye per g biosorbent) was calculated according to a mass balance on the dye concentration using Eq. (1):

$$q_e = \frac{(C_i - C_e)\,V}{m} \quad (1)$$

where C_i is the initial dye concentration (mg L^{-1}), C_e is the equilibrium dye concentration in solution (mg L^{-1}), V is the volume of the solution (L), and m is the mass of the biosorbent (g). The percent removal (%) of dye was calculated using the following equation:

$$\text{Removal}\ (\%) = \frac{C_i - C_e}{C_i} \times 100 \quad (2)$$

All the biosorption experiments were performed in triplicate. The results are the average of three independent measurements along with standard deviation (±SD) showing 95% confidence level with a precision in most cases being ±2%. Microsoft Excel program was employed for data processing. Linear regression analyses were used to determine slopes and intercepts of the linear plots and for statistical analyses of the data.

Results and discussion

Effect of pH

To investigate the effect of solution pH on the biosorption of CV by PLP, a series of batch biosorption experiments as described above were carried out over a pH range of 3–10. The results thus obtained are shown in Fig. 1. The biosorption capacity increased with increase in pH of the dye

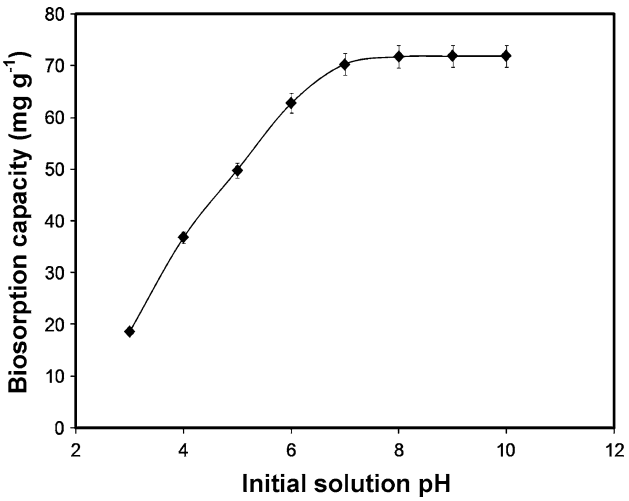

Fig. 1 Effect of pH on biosorption of CV by PLP (experimental conditions: initial dye concentration 50 mg L^{-1}, biosorbent dosage 2 g/0.1 L, agitation speed: 150 rpm, temperature 303 K, contact time 3 h)

solution, appreciably up to pH 8.0. With further increase in pH, no significant change in dye binding capacity was observed. Thus, all further experiments were carried out at pH 8.0. A quite similar result was previously reported for biosorption of CV from aqueous solution by treated ginger waste (Kumar and Ahmad 2011).

The pH of the aqueous solution affects both the surface charge of the biosorbent material as well as the degree of ionization of the dye molecule (Saha et al. 2010). PLP mainly contains –OH, –CH, $–NH_2$ and –C–O functional groups on its surface (Chowdhury et al. 2011b). Protonation of these functional groups at low pH values renders a net negative charge to the biosorbent surface while deprotonation of the functional groups at high pH values render it positively charged. The pK_a of CV is 0.8; it is completely ionized at pH greater than 0.8 and exists as cationic species (Saha et al. 2012). At low pH values, there exists a strong electronegative repulsion between the positively charged dye ions and the negatively charged biosorbent surface resulting in low dye binding capacity. However, as the pH of the dye solution increases, a considerable increase in dye binding capacity is observed due to strong electrostatic attraction between negatively charged sites on the biosorbent and the dye cations.

Effect of biosorbent dose

The influence of biosorbent dose on the CV biosorption capacity of PLP was investigated in the range of 0.5–5 g. The results obtained are summarized in Fig. 2. The percentage dye removal increases with increase of biosorbent dose from 0.5 to 2 g. It may be explained that increasing the biosorbent dose resulted in increased biosorbent surface area and availability of more binding sites (Saha et al. 2010). However, further increase in biosorbent dose did not significantly change the biosorption yield. This is due to the binding of almost all dye molecules to the biosorbent surface and the establishment of equilibrium between the dye molecules on the biosorbent and in the solution (Saha et al. 2010). These observations are in agreement with those reported previously by Saeed et al. (2010) for biosorption of CV by grapefruit peels.

Effect of initial dye concentration

Figure 3 shows the biosorption performance of PLP at different initial concentration of CV in the range of 20–100 mg L^{-1}. The adsorption capacity increased from 62.36 to 80.15 mg g^{-1} with increase in initial dye concentration from 20 to 100 mg L^{-1}. The increase in dye uptake capacity can be attributed to the fact that increasing concentration gradient provides an increasing driving force to overcome all mass transfer resistances of the dye molecules between the aqueous and solid phase, leading to an increased equilibrium uptake capacity until sorbent saturation is achieved (Chowdhury and Das 2011). On the contrary, the biosorption efficiency decreased with increase in initial dye concentration. This is mainly because all sorbents have a limited number of binding sites, which become saturated at a certain concentration (Chowdhury and Das 2011). Similar results have been reported for biosorption of CV by Sagaun sawdust (Khattri and Singh 2011).

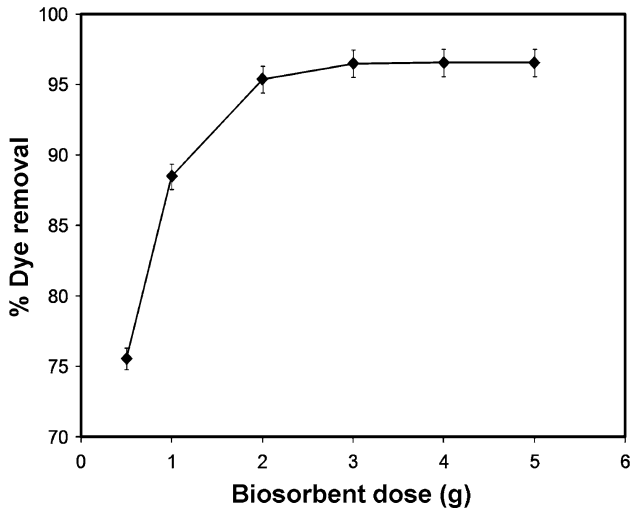

Fig. 2 Effect of biosorbent dose on biosorption of CV by PLP (experimental conditions: initial dye concentration: 50 mg L^{-1}, agitation speed 150 rpm, pH 8.0, temperature 303 K, contact time 3 h)

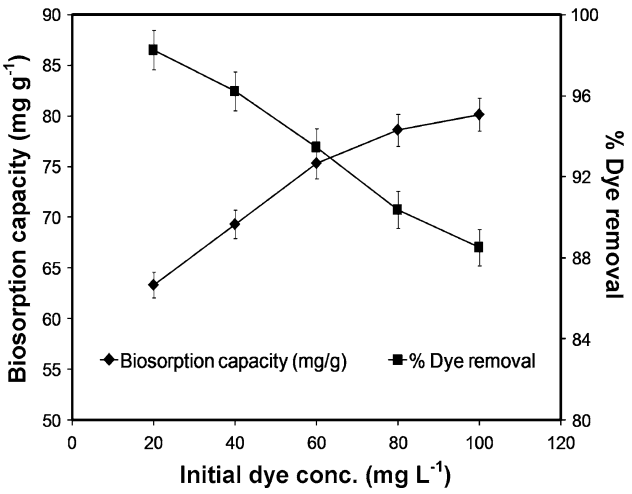

Fig. 3 Effect of initial dye concentration on biosorption of CV by PLP (experimental conditions: biosorbent dosage 2 g/0.1 L, agitation speed 150 rpm, pH 8.0, temperature 303 K, contact time 3 h)

Effect of temperature

Figure 4 presents the biosorption profile of CV by PLP at different temperatures. The dye removal efficiency decreased with increase in temperature over the range of 293–303 K. Increase in temperature results in weakening of the bonds between the dye molecules and the binding sites of the biosorbent leading to low dye removal efficiency (Chakraborty et al. 2011). Such a trend is indicative of the fact that biosorption of CV by PLP is kinetically controlled by an exothermic process. Similar phenomenon was also observed for biosorption of CV by NaOH-modified rice husk (Chakraborty et al. 2011).

Biosorption isotherms

The Langmuir and Freundlich isotherm models were used to describe the equilibrium biosorption data of CV onto PLP (Chowdhury and Saha 2010).

$$\text{Langmuir: } \frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad (3)$$

$$\text{Freundlich: } \log q_e = \log K_F + \left(\frac{1}{n}\right) \log C_e \quad (4)$$

where q_e (mg g^{-1}) and C_e (mg L^{-1}) are the solid phase concentration and the liquid phase concentration of adsorbate at equilibrium, respectively, q_m (mg g^{-1}) is the maximum adsorption capacity, K_L (L mg^{-1}) is the Langmuir adsorption equilibrium constant, K_F (mg g^{-1}) (L $g^{-1})^{1/n}$ is the Freundlich constant related to sorption capacity and n is the heterogeneity factor.

The parameters obtained from the Langmuir (C_e/q_e vs. C_e) and Freundlich (log q_e vs. log C_e) isotherm plots are listed in Table 1. To quantitatively compare the accuracy of the models, the correlation coefficients (R^2) were also calculated and are also listed in Table 1. Analysis of the R^2 values suggests that the Langmuir isotherm model provides best fit to the equilibrium biosorption data at all studied temperatures implying monolayer coverage of CV molecules onto the biosorbent surface. The maximum monolayer biosorption capacity (q_m) is 78.22 mg g^{-1} at 293 K. Table 2 summarizes the comparison of the maximum CV biosorption capacity of various sorbent materials including PLP. The comparison shows that PLP has higher biosorption capacity of CV than many of the other reported sorbent materials. Differences in dye uptake capacity are due to the differences in properties of each sorbent material such as structure, functional groups and surface area. The easy availability and cost effectiveness of PLP are some additional advantages, implying that PLP can be a better biosorbent for removal of CV from aqueous solutions.

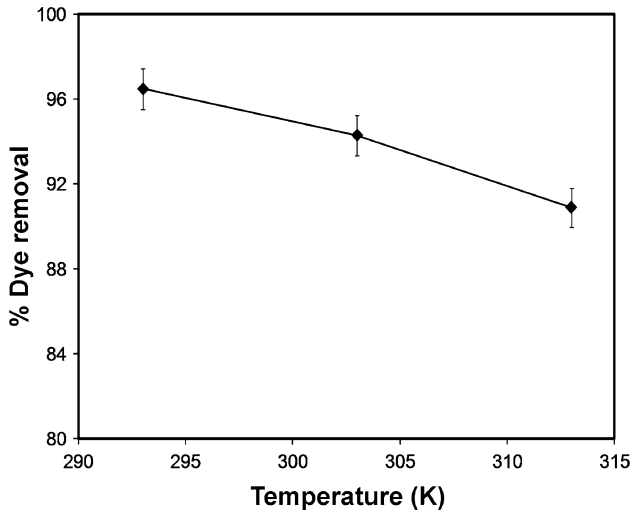

Fig. 4 Effect of temperature on biosorption of CV by PLP (experimental conditions: initial dye concentration 50 mg L^{-1}, biosorbent dosage 2 g/0.1 L, agitation speed 150 rpm, pH 8.0, contact time 3 h)

The magnitude of the Freundlich constant n gives a measure of favorability of biosorption. Values of n between 1 and 10 represent a favorable biosorption process (Chakraborty et al. 2011). For the present study, the value of n also presents the same trend at all the temperatures indicating favorable nature of biosorption of CV by PLP.

Biosorption kinetics

The pseudo-first-order and pseudo-second-order kinetic models were used to study the biosorption kinetics of CV onto PLP (Chowdhury and Saha 2010).

$$\text{Psuedo} - \text{first} - \text{order} : \log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t \quad (5)$$

$$\text{Psuedo} - \text{second} - \text{order} : \frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t \quad (6)$$

where q_t and q_e are the amount of dye adsorbed at time t and at equilibrium (mg g^{-1}), k_1 (min^{-1}) is the pseudo-first-order rate constant and k_2 (g mg^{-1} min^{-1}) is the pseudo-second-order rate constant.

The values of the pseudo-first-order model constants, k_1 and q_e were calculated from the slope and intercept of the plots of log ($q_e - q_t$) versus t while the pseudo-second-order model constants, k_2 and q_e were calculated from the slope and intercept of the plots of t/q_t versus t. The calculated model parameters along with the correlation coefficient values (R^2) are listed in Table 1. As can be seen from Table 1, the low R^2 (<0.90) values for the pseudo-first-order model indicate that this model was not suitable for describing the biosorption kinetics of CV onto PLP. However, the relatively high R^2 (>0.99) values for the pseudo-second-order model suggest that the ongoing

Table 1 Isotherm constants and kinetic parameters for biosorption of CV by PLP at different temperatures

Model	Temperature (K)		
	293	303	313
Isotherm models			
Langmuir			
q_m (mg g^{-1})	78.227	75.176	72.391
K_L (L mg^{-1})	0.861	0.722	0.654
R^2	0.999	0.998	0.996
Freundlich			
K_F (mg g^{-1})(L $mg^{-1})^{1/n}$	30.779	25.178	14.343
n	4.137	3.572	2.419
R^2	0.967	0.955	0.959
Kinetic models			
Pseudo-first-order			
$q_{e,cal}$ (mg g^{-1})	56.855	53.278	50.576
k_1 (min^{-1})	8.39×10^{-2}	6.71×10^{-2}	4.19×10^{-2}
R^2	0.925	0.923	0.928
Pseudo-second-order			
$q_{e,cal}$ (mg g^{-1})	74.262	71.386	68.783
k_2 (g mg^{-1} min^{-1})	6.25×10^{-3}	3.65×10^{-3}	1.32×10^{-3}
R^2	0.992	0.996	0.994

Table 2 Comparison of CV biosorption capacity of PLP with other reported low-cost adsorbents

Sorbent	q_{max} (mg g^{-1})	Reference
Coir pith	2.56	Namasivayam et al. 2001
Sugarcane dust	3.8	Khattri and Singh 1999
Neem sawdust	3.8	Khattri and Singh 2009
Calotropis procera leaf	4.14	Ali and Muhammad 2008
Sagaun sawdust	4.25	Khattri and Singh 2011
Jalshakti® Polymer	12.9	Dhodapkar et al. 2007
Orange peel	14.3	Annadurai et al. 2004
Jute fiber carbon	27.99	Porkodi and Kumar 2007
Coniferous pinus bark powder	32.78	Ahmad 2009
Sawdust	37.83	Parab et al. 2009
Rice bran	42.25	Wang et al. 2008
Jackfruit leaf powder	43.39	Saha et al. 2012
NaOH-modified rice husk	44.87	Chakraborty et al. 2011
Japonica	82.83	Wang et al. 2008
Wheat bran	80.37	Wang et al. 2008
Pineapple leaf powder	78.22	This study

biosorption process obeys pseudo-second-order kinetics at all studied temperatures. The applicability of the pseudo-second-order kinetic model indicates that the biosorption process of CV onto PLP is chemisorption and the rate-determining step is probably surface biosorption. The pseudo-second-order rate constant, k_2 decreases with increase in temperature suggesting exothermic nature of the biosorption process.

Activation parameters

From the pseudo-second-order rate constant k_2 (Table 1), the activation energy E_a for biosorption of CV by PLP was determined using the Arrhenius equation (Chowdhury and Saha 2010):

$$\ln k = \ln A - \frac{E_a}{RT} \tag{7}$$

where k is the rate constant, A is the Arrhenius constant, E_a is the activation energy (kJ mol^{-1}), R is the gas constant (8.314 J mol^{-1} K^{-1}) and T is the temperature (K). By plotting ln k_2 versus $1/T$, E_a was obtained from the slope of the linear plot and was estimated to be 58.96 kJ mol^{-1}. According to literature, biosorption of CV by PLP follows chemisorption (Chowdhury et al. 2011b).

The Eyring equation was used to calculate the standard enthalpy ($\Delta H^{\#}$), and entropy of activation ($\Delta S^{\#}$) (Chowdhury et al. 2011a):

$$\frac{\ln k}{T} = \ln \frac{k_B}{h} + \frac{\Delta S^{\#}}{R} - \frac{\Delta H^{\#}}{RT} \tag{8}$$

where k is the rate constant, k_B is the Boltzman constant (1.3807×10^{-23} J K^{-1}), h is the Plank constant (6.6261×10^{-34} Js), R is the gas constant (8.314 J mol^{-1} K^{-1}) and T is the temperature (K). The values of $\Delta H^{\#}$ and $\Delta S^{\#}$ were calculated from the slope and intercept of the plot of

Table 3 Thermodynamic parameters for biosorption of CV onto PLP

ΔG^0 (kJ mol^{-1})			ΔH^0 (kJ mol^{-1})	ΔS^0 (J mol^{-1} K^{-1})
293 K	303 K	313 K		
−18.25	−16.62	−14.83	−68.47	−171.29

$\ln k_2/T$ versus $1/T$ and were found to be −0.146 kJ mol^{-1} for $\Delta H^{\#}$ and −198.18 J mol^{-1} K^{-1} for $\Delta S^{\#}$. The negative value of $\Delta H^{\#}$ (=−0.146 kJ mol^{-1}) indicates exothermic nature of the biosorption process. The negative value of $\Delta S^{\#}$ suggests that biosorption of CV onto PLP is an associative mechanism (Chowdhury et al. 2011a).

The values of $\Delta H^{\#}$ and $\Delta S^{\#}$ were used to compute the free energy of activation ($\Delta G^{\#}$) from the relation:

$$\Delta G^{\#} = \Delta H^{\#} - T\Delta S^{\#} \quad (9)$$

The values of $\Delta G^{\#}$ were found to be 58.21, 60.19, 62.17 kJ mol^{-1} at $T = 293, 303,$ and 313 K respectively. The large positive values of $\Delta G^{\#}$ suggest that energy was required in the biosorption reaction to convert reactants into products.

Thermodynamic parameters

The thermodynamic parameters–Gibbs free energy change (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0) for the biosorption process were calculated using the following equations for the temperature range 293–313 K (Chowdhury and Saha 2010):

$$\Delta G^0 = -RT \ln K_C \quad (10)$$

$$K_C = \frac{C_a}{C_e} \quad (11)$$

$$\Delta G^0 = \Delta H^0 - T\Delta S^0 \quad (12)$$

where K_c is the distribution coefficient for adsorption, C_a is the equilibrium dye concentration on the adsorbent (mg L^{-1}) and C_e is the equilibrium dye concentration in solution (mg L^{-1}).

The calculated Gibbs free energy (ΔG^0) values at different temperatures for biosorption of CV onto PLP are listed in Table 3. The values of ΔH^0 and ΔS^0 were determined from the slope and intercept of the plot of ΔG^0 versus T and are also listed in Table 3. The negative value of ΔG^0 at different temperatures indicates spontaneous nature of the biosorption process. The negative value of ΔH^0 indicates that the biosorption reaction is exothermic while the negative value of ΔS^0 suggests that the process is enthalpy driven.

Conclusion

Biosorption potential of PLP to remove CV from aqueous solutions was investigated. Batch experiments were carried out as function of solution pH, initial dye concentration, biosorbent dose and temperature. Both temperature and pH were found to have a strong influence on the biosorption process. The biosorption efficiency decreased with increase in initial dye concentration while it increased with increase in biosorbent dose up to a certain level. The Langmuir isotherm showed best fit to the equilibrium biosorption data with maximum monolayer biosorption capacity of 78.22 mg g^{-1} at 293 K. Kinetic studies showed that the biosorption process followed pseudo-second-order kinetics. The activation energy (E_a) determined using the Arrhenius equation confirmed that biosorption of CV by PLP involved chemical ion-exchange. Thermodynamic studies showed that the biosorption process was spontaneous and exothermic. Compared to various other sorbents reported in the literature, PLP appears to be a promising biosorbent for practical applicability due to its easy availability and high dye binding capacity. However, to apply this environmentally friendly and efficient biosorbent for the removal of contaminants from real industrial effluents, continuous column studies need to be performed.

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