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Malachite green and crystal violet biosorption onto coco-peat: characterization and removal studies

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ABSTRACT

In this study, coco-peat was tested for its potential to sorb malachite green (MG) and crystal violet (CV) from aqueous solutions. The pH edge experimental results indicated that biosorption of two cationic dyes increased with increase in pH and maximum biosorption obtained at pH 7. The Fourier transform infrared spectrometry confirmed the involvement of negatively charged groups in the removal of cationic dyes. Furthermore, the scanning electron micrographs were used to examine the surface morphology of coco-peat. The equilibrium isotherms determined at different pH conditions were described using two-parameter (Langmuir and Freundlich) and three-parameter (Sips and Redlich–Peterson) models. According to the Langmuir model, maximum dye uptakes of 276.8 and 119.2 mg/g were obtained for MG and CV, respectively. Biosorption kinetics were modelled using pseudo-first- and pseudo-second-order models, with the former described experimental data with high correlation coefficients and low % error values. Various thermodynamic parameters such as changes in Gibbs free energy, enthalpy and entropy were calculated; and results indicated that the present system was spontaneous and endothermic process.

Keywords: Biosorption; Coco-peat; Wastewater treatment; Water quality; Bioremediation

1. Introduction

Cationic dyes have wide applications in industries such as paper, textile, printing, electroplating, food, cosmetic and pharmaceutical. Wastewaters emanating from these industries often comprise high concentrations of dye molecules. The presence of dyes in industrial discharges causes serious pollution problems because of their toxic nature to human and aquatic lives. Thus, it is necessary to remove such pollutants from the industrial effluents, in order to protect the environment. Many conventional methods have been used for dye removal such as precipitation, oxidation, flotation, adsorption and coagulation [1]. Among these, adsorption has been extensively employed for the removal of dye molecules from different types of water due to its unique advantages such as high efficiency, simplicity and practicability [2].

In recent years, there is a growing interest in using non-living biological materials for the adsorption of dyes [1,3]. Different biological materials termed

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biosorbents have been studied as alternative sorbents for the removal of cationic dyes from contaminated water. Most of the efficient biosorbents comes under the class of agricultural wastes [4], industrial wastes [5], bacteria [3], fungi [6] and fresh water algae [7]. Of these, considerable interest has been recently directed to usage of different agricultural wastes as biosorbents. Continuous availability, low-cost and waste utilization are some of the main factors motivated several researchers to explore the potential of different agricultural wastes for dye removal. Some of the important agricultural wastes employed in biosorption of cationic dyes include rice husk, bagasse, sawdust and fruit peels [1,4,8]. Other important agricultural wastes are those originate from coconut trees, in particular coconut husk and carbon derived from coir pith. These materials are well studied for the removal of different dyes from wastewaters [9–11]. However, less attention has been focused on coco-peat. Coco-peat is recently identified for preparation of soilless media for crop production [12,13] as well as lightweight substrate in green roofs, biofilters and vertical walls to support plant growth. It is also widely used as an oil absorbent for slippery floors. Apart from these, the application of coco-peat is limited considering its abundant availability.

Therefore, in the present study, coco-peat was used as a biosorbent for the removal of malachite green (MG) and crystal violet (CV) from aqueous solutions. Both MG and CV are cationic triphenylmethane dyes and have been widely used to colour silk, wool and leather, as well as effective biocides to resist fungal, parasitic and bacterial infections, especially in aquaculture [14–16]. However, they are highly toxic to mammalian cells and aquatic organisms even at trace concentrations [16,17]. Because of potential hazards on human health and environment, removal of these cationic dyes from wastewater has become a major environmental concern in recent years [19]. To account for various environmental related to wastewater treatment, biosorption experiments were conducted at different pH conditions, solute concentrations and contact time. The mechanism responsible for dye sorption was addressed as well.

2. Materials and methods

2.1. Preparation of coco-peat and dye solutions

Coco-peat was purchased from a local nursery in Chennai, India. The samples were then dried under sunlight for three days and then further dried in an oven at 60˚C for 24 h. The samples were then grounded and subsequently sieved to obtain average particle sizes in the range of 0.75 mm. Two cationic dyes were used, viz. MG (molecular formula = $C_{52}H_{54}N_4O_{12}$, C.I. = 42,000 and molecular weight = 927.02) and CV (molecular formula = $C_{25}H_{30}ClN_3$, C.I. = 42,555 and molecular weight = 407.99).

2.2. Biosorption tests

Two types of equilibrium sorption (pH edge and isotherm) and one sorption dynamic (kinetic) experiments were conducted. For all biosorption experiments, in general, 0.2 g of coco-peat was agitated with 100 mL of desired dye concentration in a 250 mL Erlenmeyer flask. The flasks were then agitated on a rotary shaker at 160 rpm at desired temperature. The solution pH was adjusted to desired values with 0.1 M HCl or NaOH. When the sorption equilibrium was reached after 12 h, the suspension was centrifuged at 2,100 rpm for 15 min. The supernatant was diluted to desired ranges using DI water and analysed for dye concentration using a spectrophotometer (UV-1800, Shimadzu, Japan). For pH edge experiments, equilibrium pH values were maintained in the range of 2–9. Control experiments (without biosorbent) were also conducted at different pH conditions to check for precipitation. In order to obtain isotherm data, experiments were conducted at fixed pH condition with initial dye concentrations varied from 50 to 1,000 mg/L. For kinetic studies (temperature = 32˚C), at specific time periods (from 2 to 240 min), samples were withdrawn from flasks and analysed for dye concentration.

2.3. Coco-peat characterization

To elucidate the dye removal mechanism, samples of raw and dye-loaded coco-peat were analysed in Fourier transform IR spectrophotometer (Bruker-ATR IR (ACPHA), Germany) and scanning electron microscopy (Hitachi S4800, Japan).

2.4. Isotherm and kinetic models

Four equilibrium models were used to fit the dye–coco-peat biosorption isotherm experimental data as follows,

$$\text{Langmuir model:}\quad Q = \frac{Q_{\max} b_L C_{eq}}{1 + b_L C_{eq}} \tag{1}$$

$$\text{Freundlich model:}\quad Q = K_F {C_{eq}}^{1/n_F} \tag{2}$$

$$\text{Sips model:}\quad Q_e = \frac{K_S {C_{eq}}^{\beta_S}}{1 + a_S {C_{eq}}^{\beta_S}} \tag{3}$$

Redlich-Peterson model:
$$Q_{\mathrm{e}} = \frac{K_{\mathrm{RP}} C_{\mathrm{eq}}}{1 + a_{\mathrm{RP}} {C_{\mathrm{eq}}}^{\beta_{\mathrm{RP}}}} \tag{4}$$

where Q is the dye uptake (mg/g), C_{eq} is the equilibrium (final) dye concentration (mg/L), $Q_{\max}$ is the maximum dye uptake (mg/g), b_{L} is the Langmuir equilibrium coefficient (L/mg), K_{F} is the Freundlich coefficient (mg/g) $(\mathrm{L/mg})^{1/n}{}_{\mathrm{F}}$, n_{F} is the Freundlich exponent, K_{S} is the Sips model isotherm coefficient $(\mathrm{L/g})^{\beta}{}_{\mathrm{S}}$, a_{S} is the Sips model coefficient $(\mathrm{L/mg})^{\beta}{}_{\mathrm{S}}$, β_{S} is the Sips model exponent, K_{RP} is the Redlich–Peterson isotherm coefficient (L/g), a_{RP} is the Redlich–Peterson isotherm coefficient $(\mathrm{L/mg})^{\beta}{}_{\mathrm{RP}}$ and β_{RP} is the Redlich–Peterson model exponent.

Two kinetic models were used to describe dye–coco-peat biosorption kinetics experimental data as follows,

Pseudo-first order model:
$$Q_{\mathrm{t}} = Q_{\mathrm{e}}(1 - \exp(-k_1 t)) \tag{5}$$

Pseudo-second order model:
$$Q_t = \frac{Q_e^2 k_2 t}{1 + Q_e k_2 t} \tag{6}$$

where Q_{e} is the amount of dye sorbed at equilibrium (mg/g), Q_{t} is the amount of dye sorbed at time t (mg/g), k_1 is the pseudo-first-order rate constant (1/min) and k_2 is the pseudo-second-order rate constant (g/mg min). All model parameters were evaluated by non-linear regression using Sigma Plot (version 4.0, SPSS, USA) software. The average percentage error between the experimental and predicted values is calculated using:

$$\varepsilon\,(\%) = \frac{\sum_{\mathrm{i}=1}^{N} (Q_{\mathrm{exp,i}} - Q_{\mathrm{cal,i}}/Q_{\mathrm{exp,i}})}{N} \times 100 \tag{7}$$

where Q_{exp} and Q_{cal} represents experimental and calculated dye uptake values, respectively, and N is the number of measurements.

3. Results and discussion

3.1. Effect of pH on adsorption of cationic dyes and removal mechanism

The process of biosorption is strongly pH dependent [20]. The solution pH influences the solute speciation as well as sorbent surface properties [3]. Considering this, experiments were conducted at wide range of pH (2–9) conditions (Fig. 1). As can be seen from Fig. 1, cationic dye biosorption by coco-peat was favoured at pH values greater than 5. Percentage dye removal values of 16.6 and 15.8% recorded for MG and CV, respectively, at pH 2 increased to 84.3 and 43.4%, respectively, at pH 7. It should also be noted that the extent and magnitude of removal were different for each dye, with CV more sensitive to pH particularly in acid pH ranges (Fig. 1). Lower uptake of cationic dyes by coco-peat at strong acidic conditions (pH 2) may be due to presence of excess H$^+$ ions in the solution. Hydrogen ion acts as a bridging ligand between the biosorbent cell wall and the dye molecule. At these conditions, the functional groups on the surface of coco-peat will be protonated by H$^+$ ions and thus, the overall charge of the biomass will be positive. On the other hand, basic dyes release coloured positively charged dye ions in solution. Since the overall charge of the biomass at strong acidic conditions is positive, the electrostatic attraction of cationic dyes is less and hence relatively lower uptake was observed. As pH increases (above pH 5), the functional groups are deprotonated and the overall charge of biosorbent become negative which enhance the electrostatic attraction between cationic dye and the anionic reactive groups of coco-peat. Comparing the extent of removal, coco-peat sorbed more MG (210 mg/g) compared to CV (107.8 mg/g). It should be noted that the uptake values presented in Fig. 1 are on a weight basis (mg/g), but on a molar basis (mmol/g), coco-peat sorbed more CV (0.264 mmol/g) than MG (0.226 mmol/g). Adsorption is a process in which several parameters can influence the extent of solute uptake, and they depend on either the sorbent characteristics (specific surface area, pore size distribution, etc.) or the sorbate structural features (molecular weight, shape, molar volume, flexibility, branching,

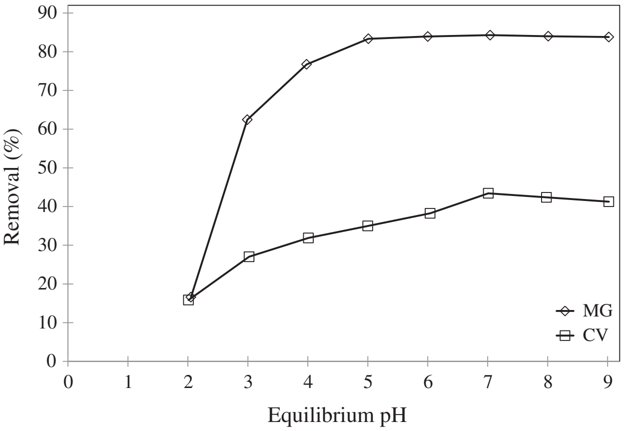

Fig. 1. Effect of equilibrium pH on the removal of MG and CV by coco-peat (initial dye concentration = 500 ± 3 mg/L; temperature = 32˚C).

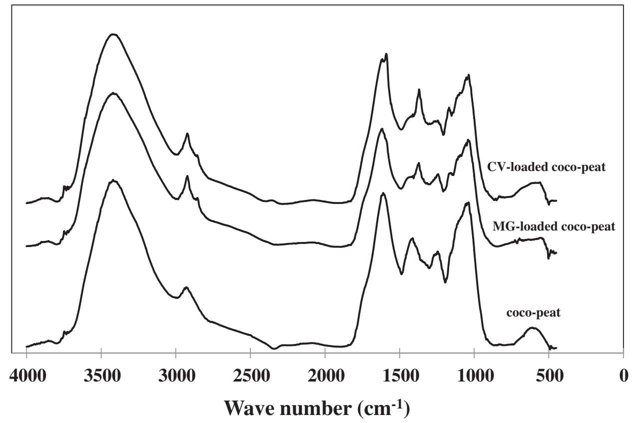

Fig. 2. FTIR spectra of coco-peat, MG-loaded coco-peat and CV-loaded coco-peat.

etc.) [21]. In several instances, adsorption can be correlated with the molecular size of the dye: a tendency might be that smaller the dye, faster and more extensively it will be sorbed [22,23]. Owing to low molecular weight and less complexity, coco-peat favoured biosorption of CV compared to MG.

To confirm the role of functional groups, the FTIR study was carried out. As shown in Fig. 2, the FTIR spectrum of the raw coco-peat displays a number of absorption peaks, indicating the complex nature of the sorbent. Despite this complexity, some characteristic peaks can be assigned (Table 1). The broad absorption peak around 3,420 cm^{-1} indicates the existence of bonded hydroxyl group [24]. The peak observed at 2,930 cm^{-1} can be assigned to the CH group. The spectrum also displays the absorption peak at 1,610 cm^{-1} corresponding to the asymmetric C=O stretch of COOH; whereas peak at 1,412 cm^{-1} attribute to symmetric C=O stretch [25]. The existence of C–O of carboxylic acid groups gave rise to the peak at 1,245 cm^{-1}. The phosphate group showed absorption peak around 1,037 cm^{-1} (P–H deformation) [26]. Significant changes in coco-peat functionalities were evident after exposure to two dyes (Fig. 2). This is due to the involvement of functional groups during interaction with two dyes and thus causing the observed wave number changes. In particular, major shifts were observed with asymmetric and symmetric C=O and C–O stretches in dye-loaded samples of coco-peat on comparison with raw coco-peat (Table 1). These results confirm the involvement of negative functional groups on the surface of coco-peat during biosorption of cationic dye molecules.

Fig. 3 shows SEM image (×200 magnification) of raw, MG-loaded and CV-loaded coco-peat samples. According to the images, coco-peat has complex, uneven and rough morphology. After biosorption, the surface of coco-peat was covered with dye molecules and appeared relatively smooth.

3.2. MG and CV isotherms

Experimental isotherm points for equilibrium dye biosorption on coco-peat at pH 5, 6 and 7 are plotted in Fig. 4. Each of these isotherms could be considered as L-shaped, i.e. the ratio between the dye concentration in the solution and that sorbed onto the biosorbent decreases with increase in dye concentration, providing a concave curve without a strict plateau [27]. However, comparing both dyes, it can clearly be seen that initial slope of MG isotherms was steeper than CV. This indicated that MG possesses high affinity towards coco-peat. The highest dye uptake values found at pH 7 were 261.7 and 119.3 mg/g for MG and CV, respectively.

In order to describe MG and CV isotherm data, several two- and three-parameter models were used. The model constants, along with correlation coefficients (R^2) and percentage error (ε) values obtained from isotherm models are presented in Table 2. For both dyes, the Langmuir model described the experimental isotherm data reasonably well with high correlation coefficients and low % error values. Although

Table 1
Stretching frequencies observed in raw and dye-loaded coco-peat samples

	Wavenumber (cm^{-1})		
Assignment	Raw coco-peat	MG-loaded coco-peat	CV-loaded coco-peat
–OH stretching	3,421	3,421	3,428
C–H stretching	2,930	2,922	2,923
C=O stretching (asymmetric)	1,610	1,616	1,590
C=O stretching (symmetric)	1,412	1,371	1,370
C–O (COOH) stretching	1,245	1,241	1,244
P–H stretching	1,037	1,042	1,037

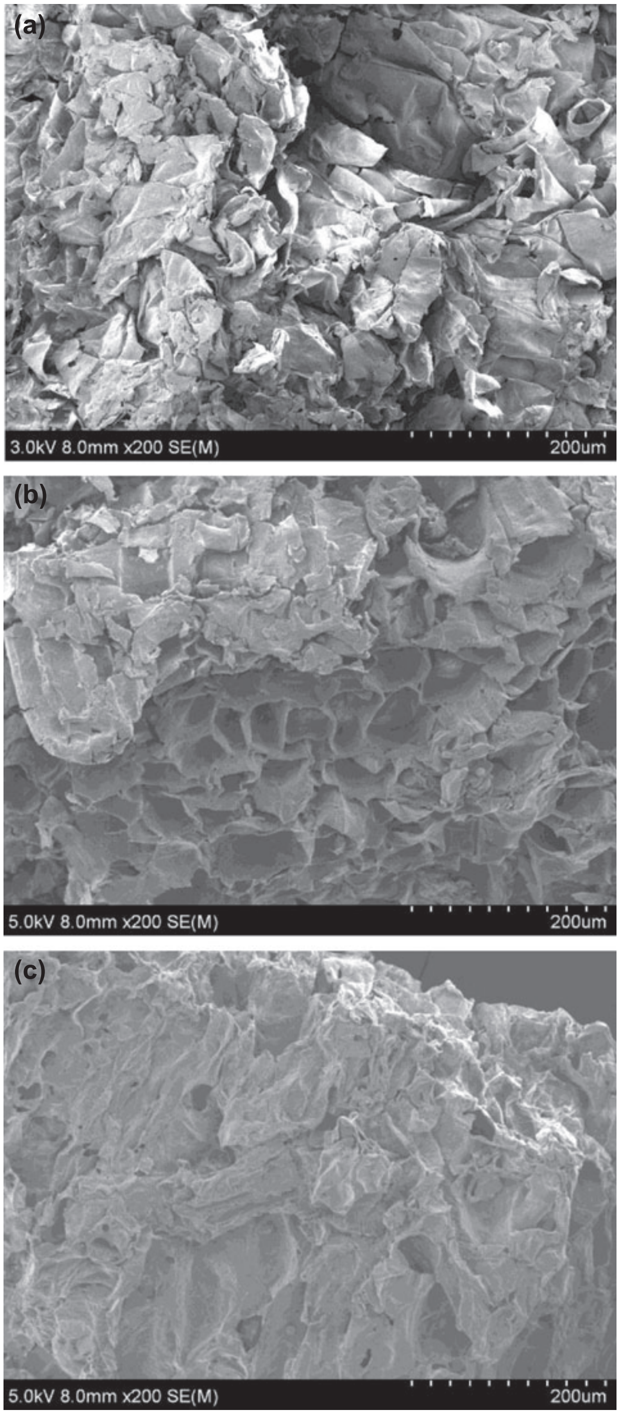

Fig. 3. Scanning electron micrographs of (a) coco-peat, (b) MG-loaded coco-peat, and (c) CV-loaded coco-peat.

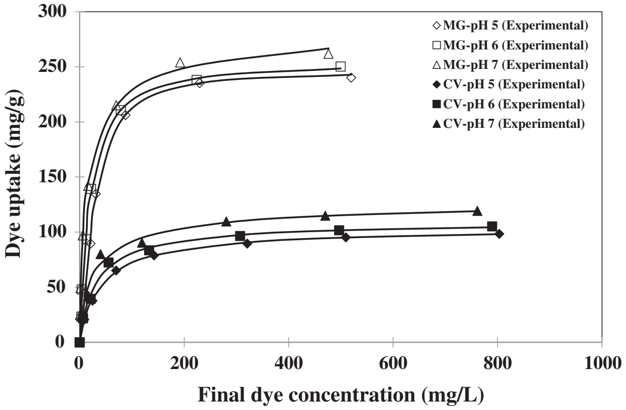

Fig. 4. Biosorption isotherms of MG and CV onto coco-peat. Curves predicted by the Sips model (temperature = 32°C).

the Langmuir model cannot provide any mechanistic understanding of the sorption phenomena, the equation maybe conveniently used to estimate the maximum uptake of dye molecules from experimental data. The constant $Q_{\max}$ corresponds to maximum dye uptake possible in the system, whereas constant b_L represents the affinity between the sorbent and sorbate. In accordance with experimental data, maximum $Q_{\max}$ and b_L values for both dyes were observed at pH 7.

The Freundlich model is an empirical equation assuming that the sorption process takes place on heterogeneous surfaces and the sorption capacity is related to the concentration of solute at equilibrium. Application of the Freundlich model to MG and CV isotherm data resulted in relatively low correlation coefficients and high % error values (Table 2). As the Freundlich equation (Eq. 2) is exponential, it can only be reasonably applied in the low to intermediate concentration ranges [28]. The model constants, K_F and n_F being indicative of the extent of the biosorption and the degree of non-linearity between solution concentration and biosorption, respectively, were recorded high at pH 7. For some cases, two parameters models are not competent enough [3]. Therefore, three-parameter models were used to improve curve fitting. The Sips model is postulated with the assumptions that surface active sites are of different strengths and that one molecule of adsorbate interacts with one active site. At low and high sorbate concentrations, the Sips model effectively reduces to the Freundlich and Langmuir equations, respectively. For both dyes, at all pH conditions, the Sips model exhibited high correlation coefficient (>0.989) and low % error (<2.52%) values, which produced a considerably better fit compared with the Langmuir and Freundlich models. In concurrence with experimental data, the Sips isotherm constant (K_S) recorded high for MG (40.6 L/g) than CV (10.9 L/g) at pH 7. Application of MG and CV experimental isotherm data to the Redlich–Peterson model also

Table 2
Isotherm model constants obtained during MG and CV biosorption onto coco-peat

		MG			CV		
Model		pH 5	pH 6	pH 7	pH 5	pH 6	pH 7
Langmuir	$Q_{\max}$ (mg/g)	269.0	271.2	276.8	102.2	107.6	119.2
	b_L (L/mg)	0.028	0.043	0.073	0.025	0.032	0.042
	R^2	0.987	0.994	0.985	0.999	0.995	0.982
	ε (%)	10.4	5.27	13.7	1.13	1.32	4.46
Freundlich	K_F (mg/g) $(\text{L/mg})^{1/n_F}$	37.8	48.1	64.5	19.0	22.5	27.8
	n_F	3.16	3.53	4.08	3.86	4.11	4.32
	R^2	0.885	0.909	0.947	0.951	0.945	0.953
	ε (%)	32.4	30.4	20.6	7.73	8.28	7.66
Sips	K_S $(\text{L/g})^{\beta_S}$	1.90	9.36	40.6	3.27	4.38	10.9
	a_S $(\text{L/mg})^{\beta_S}$	0.008	0.037	0.139	0.031	0.039	0.083
	β_S	1.45	1.11	0.706	0.924	0.919	0.724
	R^2	0.999	0.997	0.999	0.999	0.995	0.989
	ε (%)	2.17	2.52	1.95	0.178	0.178	0.165
Redlich–peterson	K_{RP} (L/g)	5.80	11.5	33.7	2.77	3.76	7.26
	a_{RP} $(\text{L/mg})^{\beta_{RP}}$	0.008	0.037	0.204	0.032	0.042	0.101
	β_{RP}	1.16	1.03	0.919	0.973	0.971	0.920
	R^2	0.996	0.996	0.995	0.999	0.996	0.987
	ε (%)	5.32	4.76	7.46	0.398	0.60	1.74

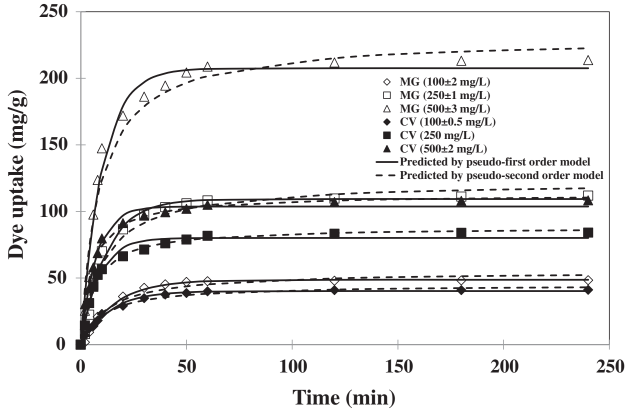

Fig. 5. Concentration–time profiles observed during biosorption of MG and CV onto coco-peat (pH 7; temperature = 32°C).

resulted in better fit. The Redlich–Peterson model combines elements from both the Langmuir and Freundlich equations, and the mechanism of adsorption is a hybrid and does not follow ideal monolayer. The model converges to Henry and Langmuir isotherm when β_{RP} values are equal to zero and one, respectively. Also, Redlich–Peterson model will represent a Freundlich isotherm when model parameters K_{RP} and $a_{RP} >> 1$ and $\beta_{RP} < 1$. It can be seen that β_{RP} are close to unity which means the isotherms are approaching the Langmuir, but not the Henry isotherm. Based on the R^2 and % error values, it can be noticed that the Sips model has the highest level of agreement with MG and CV experimental data (Table 2).

3.3. MG and CV Kinetics

The kinetics of dye sorption is an important parameter for designing sorption systems and is required for selecting optimum operating conditions for full-scale process. Therefore, kinetic experiments at different initial solute concentrations were conducted to understand the biosorption behaviour of coco-peat towards MG and CV (Fig. 5). It was identified that rates of MG and CV biosorption by coco-peat were high during early stages of the process and then declined to a relatively lower rate, leading to equilibrium. For instance, coco-peat took only 30 min to remove more than 90% of the total MG and CV uptake. This rapid phase was followed by slower phase until 60 min to reach equilibrium (Fig. 5). The higher sorption rate during the initial period is due to the accessibility of the free active sites on the surface of the sorbent and thereby a large concentration gradient that activates the process [29]. With increasing time, this concentration gradient was reduced due to the sorption of dye molecules onto the vacant sites, leading to decreased sorption during the later stage.

The kinetic pattern of MG and CV onto coco-peat has been examined using pseudo-first- and pseudo-second-order models. These models are based on the assumption that the rate of sorption is proportional to the number of free sites on the surface of the sorbent, respectively, to the first and second power. Application of pseudo-first-order model to both MG and CV kinetics data resulted in high correlation coefficients and low % error values (Table 3). Both constants (Q_e and k_1) increased with increase in initial dye concentration, which implies that high solute concentration favoured rate and extent of biosorption. Comparing both dyes, biosorption of MG onto coco-peat resulted in high Q_e and k_1 values. The greater similarity between the calculated and experimental Q_e values along with high values of R^2 and low values of ε showed the applicability of this kinetic model to both MG and CV. In the case of pseudo-second-order equation, the model produced relatively lower R^2 and higher ε values for both dyes. The equilibrium update predicted by pseudo-second-order model increases, whereas the rate constant decreases as the initial dye concentration increases. The kinetic curves as predicted by two models in comparison with experimental MG and CV kinetics data are presented in Fig. 5.

3.4. Thermodynamics of MG and CV biosorption process

The effect of temperature on MG and CV biosorption was carried out for a temperature range of 305–313 K by varying initial concentrations (50–1,000 mg/L) at pH 7. The Langmuir constants, Q_{max} and b_L increased with increase in temperature. These seem to imply that the MG and CV biosorption onto coco-peat was favourable at high temperature. The thermodynamic parameters such as the Gibbs free energy change (ΔG°), enthalpy change (ΔH°) and entropy change (ΔS°) for the biosorption process were calculated from the variation of Langmuir constant (b_L) with temperature (T) using well-known relations,

$$\Delta G^\circ = -RT \ln b_L \quad (8)$$

Table 3
Kinetic model constants obtained during MG and CV biosorption onto coco-peat

Model		MG			CV		
		100 ± 2 mg/L	250 ± 1 mg/L	500 ± 3 mg/L	100 ± 0.5 mg/L	250 mg/L	500 ± 2 mg/L
Pseudo-first order	Q_e (mg/g)	48.69	109.3	207.6	40.6	80.9	103.7
	k_1 (min^{-1})	0.064	0.083	0.099	0.075	0.119	0.137
	R^2	0.994	0.988	0.984	0.994	0.984	0.989
	ε (%)	19.7	10.1	5.02	2.50	0.822	0.986
Pseudo-second order	Q_e (mg/g)	55.06	122.4	230.6	44.9	88.2	112.8
	k_2 (g/(mg min))	0.0014	0.0008	0.0005	0.002	0.002	0.002
	R^2	0.969	0.974	0.975	0.991	0.982	0.988
	ε (%)	28.99	13.4	5.51	2.89	3.94	1.36

Table 4
Thermodynamic parameters during biosorption of MG and CV onto coco-peat

Dye	Temperature (K)	ΔG° (kJ/mol)	ΔS° (kJ/mol K)	ΔH° (kJ/mol)
MG	305	−28.9	0.368	83.2
	308	−30.2		
	313	−31.9		
CV	305	−25.2	0.402	97.4
	308	−26.6		
	313	−28.4		

where R is the gas constant (8.314 J/mol K) and T is the absolute temperature (K). The changes in the standard enthalpy and entropy were obtained from the plot of ln b_L vs. $1/T$; the equations are as follows:

$$\Delta G^\circ = \Delta H^\circ - T\Delta S^\circ \tag{9}$$

From Eqs. (8) and (9), we get,

$$\ln b_L = -\frac{\Delta H^\circ}{RT} + \frac{\Delta S^\circ}{R} \tag{10}$$

As shown in Table 4, ΔG° values were negative and increases with increase in temperature. This implies that MG and CV biosorption onto coco-peat was spontaneous and thermodynamically feasible process. The values of ΔH° were found to be positive for both MG and CV, which indicate that the process was endothermic. The positive ΔS° values correspond to an increase in randomness at the solid/liquid interface during the biosorption process

4. Conclusions

This work identified that coco-peat could be used as an efficient and low-cost biosorbent for the removal of MG and CV from aqueous solutions. The experimental factors such as pH (2–9), initial dye concentration (50–1,000 mg/L), contact time (2–240 min) and temperature (305–313 K) were investigated to study the dye biosorption process. Several isotherm and kinetic models were used to describe the experimental data. Based on R^2 and % error values, the Sips and pseudo-first-order models successfully described isotherm and kinetics experimental data, respectively. Based on ΔG°, ΔH° and ΔS°, biosorption of MG and CV onto coco-peat was feasible, spontaneous and endothermic process. Thus, our experiments indicated that coco-peat based biosorption process has advantages such as high dye removal efficiency, rapid kinetics and operation over wide range of pH conditions. In addition to the above, coco-peat is inexpensive, abundantly available and environmentally benign, which make this process practical for decontaminating MG and CV-bearing wastewaters.

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