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Elimination of Crystal Violet from Aqueous Media Using *Lagurus Ovatus* as a Natural Adsorbent

Saddam Mohammed Al-Mahmoud

Department of Chemistry, College of Education for Women, Tikrit University, Tikrit, Iraq

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Abstract

Dyes play an essential role in water contamination; however, more effort must be made to reduce this issue. This study utilised *Lagurus ovatus* as an adsorbent, which can be considered an inexpensive weed, to remove the crystal violet from aqueous media. Various factors were examined, and it was found that the highest dye removal efficiency was obtained at pH between 5–11, the adsorbent weight of 2 g/L, the contact time of 40 min, and at the temperature of 50 °C. At the same time, it decreases with increasing dye concentration to reach its lowest value of about 82% at 80 mg/L. The thermodynamic study denotes the endothermic behaviour of the removal system. Also, the kinetic investigation elucidates that the best fit of the removal data can be offered by applying the pseudo-second order kinetic equation. In addition, the study of the isotherms reveals that the removal system follows precisely the Langmuir isotherm. The proposed adsorption mechanism is the electrostatic attraction between the cationic dye and the biosorbent surface. These results suggest that *Lagurus ovatus*, a new and alternative natural adsorbent, can be utilised efficiently to eliminate crystal violet from aqueous media.

1. Introduction

The incidence of water contamination is increasing due to various human activities, which can pose substantial threats to the environment and human health [1]. Dyes are among the most common contributors to water pollution [2]. Various health risks are associated with dyes due to their high toxicity, as they may disrupt liver, kidney, and brain functions [3]. Effluents from the textile industry contain high concentrations of dyes, which can cause serious environmental impacts if not properly treated. Among the various dyes, crystal violet (CV) is widely used in different fields [4]. CV is a water-soluble cationic dye extensively employed in the textile industry for dyeing leather, wool, cotton, plastics, and paper [5].

Although there are various treatment methods, including, electrocoagulation [6], ozonation [7], Fenton process [8], photocatalysis [9], and ion exchange [10], their removal efficacy is limited due to the complicated operation and low efficiency. Adsorption is a commonly applied treatment modality for water decontamination, owing to its simplicity of operation, low relative cost, and absence of toxic by-products [11]. Different adsorbent materials, such as clays, activated carbon, agricultural materials, and metal oxides, are commonly used for water purification [12].

Nowadays, the growing demand for sustainable development promotes the utilisation of eco-friendly resources such as agricultural waste. Agricultural waste, available in abundance, can be a cost-effective alternative to other adsorbent substrates [13]. Many studies on water decontamination have been carried out using various agricultural materials such as pumpkin seed husk [14], fava bean peels [15], sawdust [16], papaya bark fibre [17], avocado seed waste [18], rice straw [19], moringa seed peels [20], and potato peels [21]. *Lagurus ovatus*, also called Hare tail grass or Bunny tail grass, can be classified as a winter weed used in dried flower manufacture to produce diverse outputs such as wall pictures, hangers, and flower planning [22]. Due to its con-

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*Corresponding author.
E-mail address: s_almahmoud@tu.edu.iq

siderable obtainability, *Lagurus ovatus* can be a remarkable alternative material in the adsorption technology.

The use of *Lagurus ovatus* in water treatment remains largely unexplored. This research focuses on investigating the potential of *Lagurus ovatus* as a new natural biosorbent for removing crystal violet dye from contaminated water

2. Experimental

2.1. Chemicals and Materials

The dye utilised in this research was crystal violet (Sigma Aldrich), and its chemical structure is presented in Fig. 1. Its chemical formula is $C_{25}H_{30}ClN_3$, and its molar mass is 407.979 g/mol (90% purity). *Lagurus ovatus* were gathered from the province of Mosul (Iraq). HCl (37%, Sigma Aldrich) and NaOH (98%, Sigma Aldrich) were used for pH adjustment.

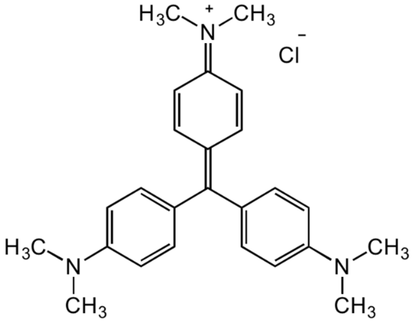

Fig. 1. Chemical structure of Crystal violet.

2.2. Preparation of the adsorbent

Lagurus ovatus was collected from the local area in Tikrit, Iraq. The feathery seed heads were separated from other plant parts, washed many times with distilled water and then dried in a drying oven (LabTech, Republic of Korea) for about 14 h at 85 °C. It was then milled using an electric grinder and sieved using a molecular sieve to achieve an average particle size of no more than 100 µm. The field emission scanning electron microscope (FESEM) (Mira 3, TESCAN) was applied to examine the resulting dried powder of *Lagurus ovatus*, which was stored in a plastic container for adsorption experiments.

2.3. Adsorption experiments

The batch adsorption experiments were performed using 500 mg/L of CV dye as a stock solution. The effects of the main operating parameters, including pH (2.5–11), starting CV concentration (20–80 mg/L), removal time (5–80 min), *Lagurus ovatus* weight between (0.04–4 g/L), and temperature (20–50 °C), were investigated utilising 100 mL conical flasks that contained 25 mL of the crystal violet solutions. A controlled shaker water bath (GFL, Germany) was used to shake the solutions at 150 rpm. The centrifuge (Gallenkamp, England) with a rate of 2500 rpm was employed for about 7 min to gain the supernatant that contain the residual CV dye. A UV-visible spectrophotometer (UV-1800, Shimadzu) was employed to detect the absorbance of the residual dye at 591 nm. Then, the amount of CV adsorbed was calculated from the concentration difference.

The adsorption capacity at equilibrium (q_e) and the adsorption efficiency (%Adsorption), were calculated using Eqs. (1) and (2), respectively.

$$q_e = \frac{(C_0 - C_e)V}{m} \tag{1}$$

$$\%Adsorption = \frac{(C_0 - C_e)}{C_0} \times 100 \tag{2}$$

where C_0 – initial dye concentration, mg/L; C_e – dye concentration at equilibrium, mg/L; m – adsorbent weight, g and V – dye solution volume, L.

The resulting removal data from adsorption experiments were further analysed by applying various adsorption equations. Three kinetic models were utilised for kinetics investigation: pseudo-first order, pseudo-second order, and intra-particle diffusion equations. For the isotherm study, Langmuir and Freundlich isotherms were employed. The linearized forms of these removal models are presented in Table 1.

3. Results and discussion

3.1. Lagurus ovatus surface characterization

The surface features for the *Lagurus ovatus* powder were determined using the FESEM technique, and the resulting image is presented in Fig. 2. A rough surface containing irregular folds and grooves can be observed. In addition, the distance between these folds is about 39 nm (see insert), and their depth can be estimated of about 50 nm. It can be concluded that these irregular folds can enhance the surface area to allow better contact between CV molecules and the *Lagurus ovatus* surface.

Table 1. The linearized forms of the different removal models.

Adsorption model	Equation	Plot
	Kinetic Models	
Pseudo-first order	$\ln(q_e - q_t) = \ln q_e - k_1 t$	$\ln(q_e - q_t)\ vs.t$
Pseudo-second order	$\frac{t}{q_t} = \left(\frac{1}{k_2 q_e^2}\right) + \left(\frac{1}{q_e}\right) t$	$t/q_t\ vs.t$
Intra-particle diffusion	$q_t = k_{int} t^{1/2} + C$	$q_t\ vs.t^{1/2}$
	Isotherm models	
Langmuir	$\frac{1}{q_e} = \frac{1}{q_m K_L C_e} + \frac{1}{q_m}$	$t/q_e\ vs.t/q_m$
Freundlich	$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$	$\ln q_e\ vs.\ln C_e$

Here q_m is the maximum adsorption capacity, k_L is the Langmuir constant, k_F and n are the Freundlich constants.

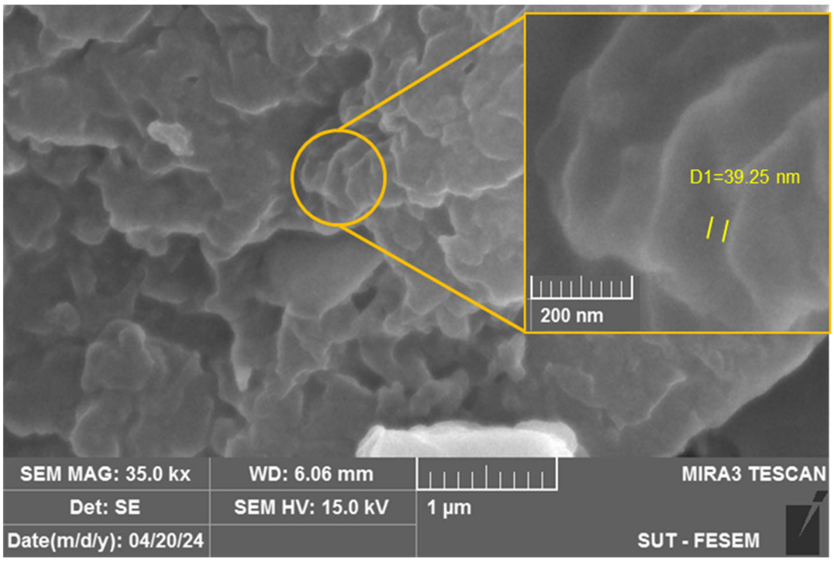

Fig. 2. FESEM image of *Lagurus ovatus* surface.

3.2. Adsorption study

The effect of pH is an intrinsic factor that leverages the removal process. It was tested by changing the pH between (2.5–11) using a 2 g/L *Lagurus ovatus* dosage, 20 mg/L CV concentration, 40 min adsorption time, at 20 °C. It can be seen from Fig. 3 that there is a decrease in the adsorption efficiency with decreasing pH values, which can be associated with the competition of the H^+ liberated inside the acidic medium with the positively charged dye ions. On the other hand, increasing the pH values above 5 can raise the negative charge on the *Lagurus ovatus* surface that permits a significant electrostatic attraction between the negatively charged *Lagurus ovatus* surface and positively charged CV cationic dye, which delivers about 94% of the adsorption efficiency. This suggests that the highly acidic medium is not preferred for the elimination of CV onto the *Lagurus ovatus* surface, and it is necessary to conduct the experiments within neutral (pH = 7.3) or basic medium environments to achieve higher adsorption removal.

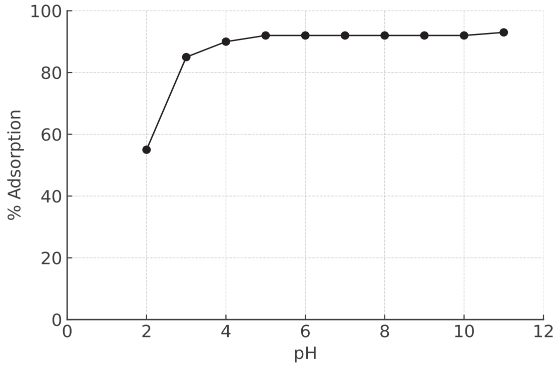

Fig. 3. pH impact on the CV removal onto *Lagurus ovatus* surface at 40 min.

The impact of the dye initial concentration was studied using diverse crystal violet concentrations (20–80 mg/L), 2 g/L *Lagurus ovatus* dosage, pH of 7.3, 40 min adsorption time, and at 20 °C. Figure 4 shows the highest adsorption efficiency of 94%, which can be noticed at 20 mg/L, and as the starting CV concentration increases, the adsorption percentage decreases. This can be explained considering that for higher initial dye concentration, the residual dye concentration in the solution at equilibrium is also higher due to the lower availability of the free adsorption sites, which are required to adsorb the CV molecules [11]. On the other hand, a gradual increase in the adsorption capacity was noticed as the initial CV concentration increased. A specific adsorbent dose will supply a limited number of active adsorption sites on the adsorbent surface. As the initial CV concentration increases, a considerable number of CV molecules will compete for the limited number of obtainable active sites. This means that the number of occupied sites increases at the expense of free sites, leading to increased adsorption capacity.

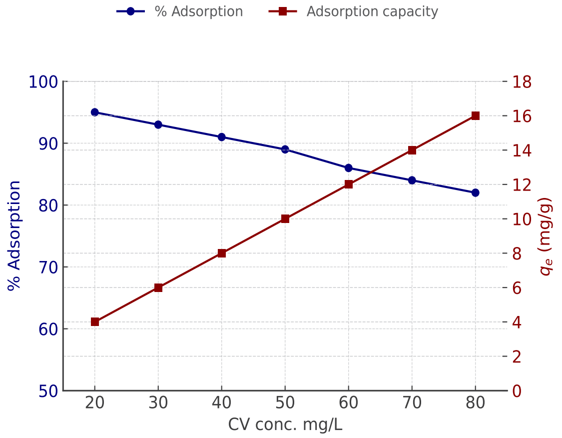

Fig. 4. Initial CV concentration impact on the removal process.

The effect of *Lagurus ovatus* dosage on crystal violet removal was also investigated. Figure 5 shows the results obtained with dosages ranging from 0.4 to 4 g/L at 20 mg/L CV concentration, pH 7.3, 40 min adsorption time, and 20 °C. At low dosages, the uptake level was relatively low; however, as the Lagurus ovatus dosage increased, the removal efficiency rose, reaching about 94% at 2 g/L. This improvement is attributed to the larger surface area and the greater number of vacant active sites available for adsorption. Beyond this dosage, further increases in adsorbent amount did not produce noticeable changes, indicating that equilibrium had been reached. In contrast, the adsorption capacity decreased with increasing *Lagurus ovatus* dosage. This behavior can be explained by the fact that adsorption capacity represents the ratio of occupied to vacant sites. At a constant CV concentration, increasing the adsorbent dosage provides more free active sites without a corresponding increase in occupied sites, thereby reducing the adsorption capacity.

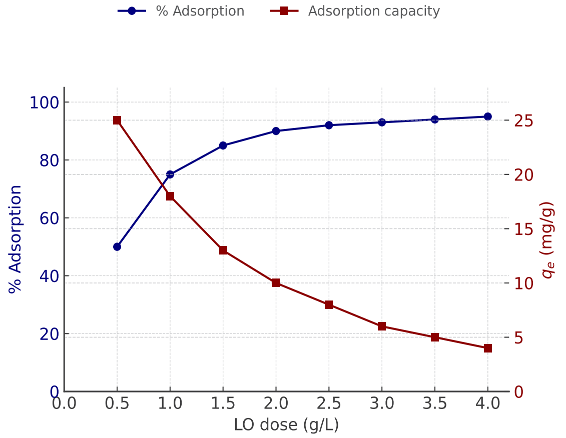

Fig. 5. *Lagurus ovatus* dose impact on the CV removal.

The adsorption time strongly influences the removal of crystal violet molecules onto the *Lagurus ovatus* surface. Figure 6 illustrates the effect of contact time on CV removal under different conditions (5–80 min, 2 g/L *Lagurus ovatus* dosage, 20 mg/L CV concentration, pH 7.3, at 20 °C). At a contact time of 5 min, about 56% of CV was adsorbed; thereafter, the adsorption efficiency increased rapidly during the first 30 minutes. This pronounced increase can be attributed to the large number of unoccupied active sites available on the *Lagurus ovatus* surface at the beginning of the adsorption process. Subsequently, the process slowed down due to the decrease in the number of unoccupied active sites, reaching a maximum removal of 94% at 40 min, which can be regarded as the equilibrium time.

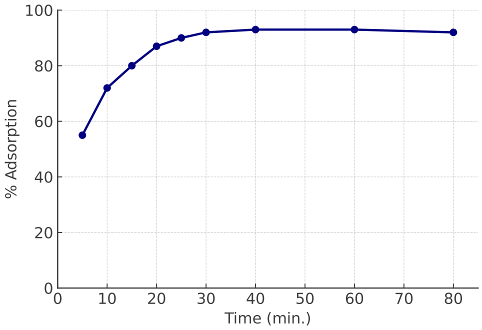

Fig. 6. Contact time impact on the removal process.

3.3. Thermodynamic study

The effect of temperature on the adsorption of crystal violet dye onto the surface of *Lagurus ovatus* was investigated at temperatures ranging from 20 to 50 °C, using a dosage of 2 g/L *Lagurus ovatus*, an initial CV concentration of 20 mg/L, a solution pH of 7.3, and an adsorption time of 40 minutes. The removal percentage increased with increasing temperature (Fig. 7), indicating the endothermic nature of the adsorption process.

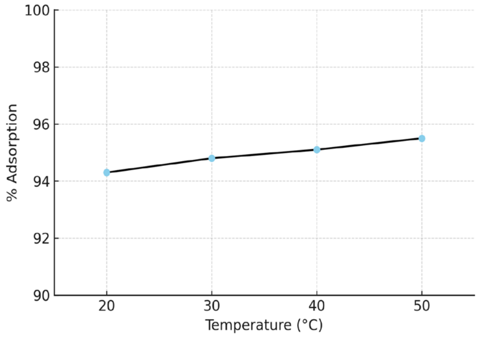

Fig. 7. Temperature impact on the removal process.

These experimental data were analysed further to calculate the thermodynamic values of the adsorption system. ΔG° was determined using Eq. (3). ΔH and ΔS were calculated using Eq. (4) from the slope and intercept of the plot between ln K against 1/T, respectively.

$$\Delta G^{\circ} = -RT \ln K \tag{3}$$

$$\ln K = \frac{\Delta S}{R} - \frac{\Delta H}{RT} \tag{4}$$

where R – the ideal gas constant, 8.314 J/mol K; T – the absolute temperature, K; and K – the adsorption equilibrium constant that can be determined using Eq. (5).

$$K = \frac{(C_0 - C_e)}{C_e} \tag{5}$$

The resulting thermodynamic values for the removal of crystal violet dye on *Lagurus ovatus* surface at varied temperatures are given in Table 2.

A positive enthalpy change ΔH indicates an endothermic behavior of the CV adsorption onto the *Lagurus ovatus* surface. Also, a positive amount of ΔS illustrates the growing randomness within the adsorption system. In addition, the Gibbs free energy change ΔG° gives a negative amount that elucidates the spontaneous behavior of the removal system, and the increase of its negative value with increasing temperature confirms that the CV adsorption onto the *Lagurus ovatus* surface is more favourable at elevated temperatures.

Table 2. Thermodynamic values for the CV removal on *Lagurus ovatus* surface

T (K)	ΔG° (kJ/mol)	ΔH (kJ/mol)	ΔS (J/mol K)	R^2
293	-6.853	5.256	41.43	0.977
303	-7.337	-	-	-
313	-7.725	-	-	-
323	-8.100	-	-	-

3.4. Kinetic study

Three kinetic models were applied to the data obtained from the adsorption time experiment: the pseudo-first-order, pseudo-second-order, and intra-particle diffusion models. Figure 8 shows the plots of these models, while the kinetic constants derived from them are presented in Table 3. All models exhibited high correlation coefficient values; however, the adsorption capacity calculated from the pseudo-second-order model was the closest to the experimental value. This indicates that CV adsorption onto the *Lagurus ovatus* surface is more likely to follow pseudo-second-order kinetics.

Table 3. Kinetic values calculated for the CV removal onto *Lagurus ovatus* surface

Kinetic model	Kinetic parameter	Value
-	q_e exp. (mg. g^{-1})	9.349
Pseudo-first order	q_e cal. (mg. g^{-1})	8.167
	k_1 (min^{-1})	0.141
	R^2	0.988
Pseudo-second order	q_e cal. (mg. g^{-1})	9.718
	k_2 (g. mg^{-1} min^{-1})	0.042
	R^2	0.999
Intra-particle diffusion	q_e cal. (mg. g^{-1})	11.24
	k_{int} (mg. g^{-1} min^{-1})	1.27
	R^2	0.993

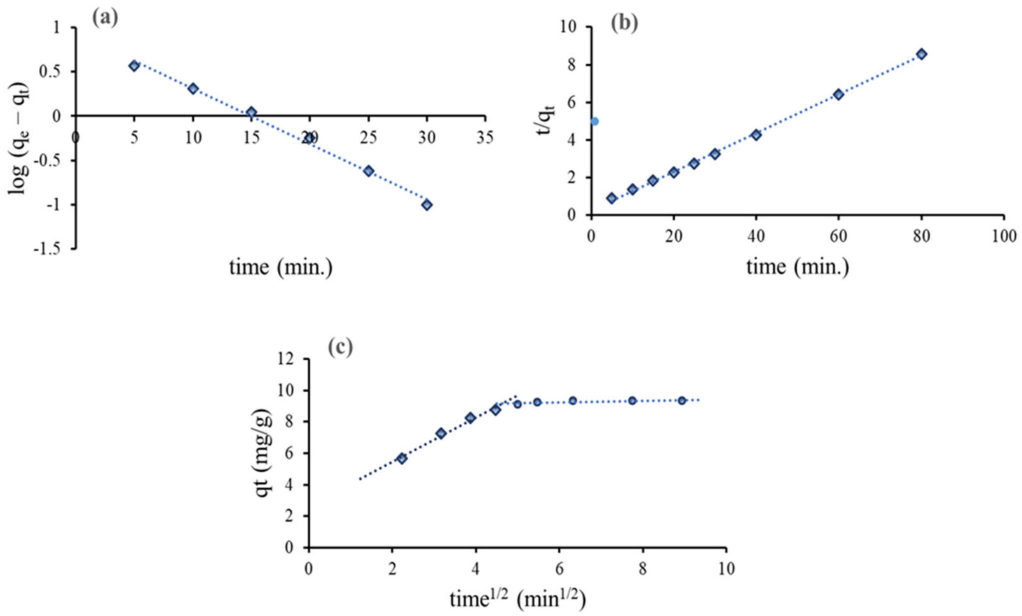

Fig. 8. The kinetic plots of (a) pseudo-first order, (b) pseudo-second order, and (c) intra-particle diffusion models.

3.5. Isotherm study

The adsorption isotherm is a substantial study that depicts the equilibrium accomplishment of the adsorbent that participated in the removal system at a constant temperature. The isotherms were applied to the data obtained from the initial dye concentration investigation at 20 °C. Two isotherm models were utilised: Langmuir isotherm, which supposes a monolayer adsorption with the energetically equivalent adsorption sites through the whole adsorbent surface, and the Freundlich isotherm assumes the heterogeneity for most adsorbent surfaces due to the irregular energy level varied on the adsorption sites [23]. The isotherm plots were presented in Fig. 9, and the isotherm parameters resulting from the analysis of the equilibrium data are summarized in Table 4. The dimensionless constant separation factor (R_L) is an essential feature in the Langmuir isotherm. It can be determined from Eq. (6). Its values suggest the type of isotherm, either unfavourable ($R_L > 1$), linear ($R_L = 1$), favourable ($0 > R_L > 1$), or irreversible ($R_L = 0$).

$$R_L = \frac{1}{(1 + k_L C_0)} \tag{6}$$

The separation factor (R_L) was calculated to be in the range of (0.127-0.035), and the n value was determined to be 2.175, both suggesting the favourability of the adsorption system. The highest correlation coefficient (R_2) value indicates that applying the Langmuir isotherm would give a better fit for the adsorption experimental data, which confirms that CV dye adsorption onto the *Lagurus ovatus* surface follows the Langmuir isotherm.

Table 4. Isotherm parameters for the CV removal onto *Lagurus ovatus* surface

Isotherm model	Isotherm parameter	Value
Langmuir	q_m (mg. g^{-1})	17.887
	k_L (L mg^{-1})	0.342
	R^2	0.988
	R_L	0.127-0.035
Freundlich	K_F (mg. g^{-1})	0.033
	n	2.175
	R^2	0.98

3.6. Adsorption mechanism

Agricultural materials are ordinarily rich in lignin, cellulose, and hemicellulose. Its surface contains various active groups such as hydroxyl (-OH) and carboxyl (-COOH), which can adsorb organic compounds through different mechanisms [24]. Many authors assumed that the electrostatic attraction between cationic dyes and the negatively charged surface sites is the dominant adsorption mechanism [25, 26, 27]. The effect of pH on the cationic dye removal onto the biosorbent surface is the lowest in

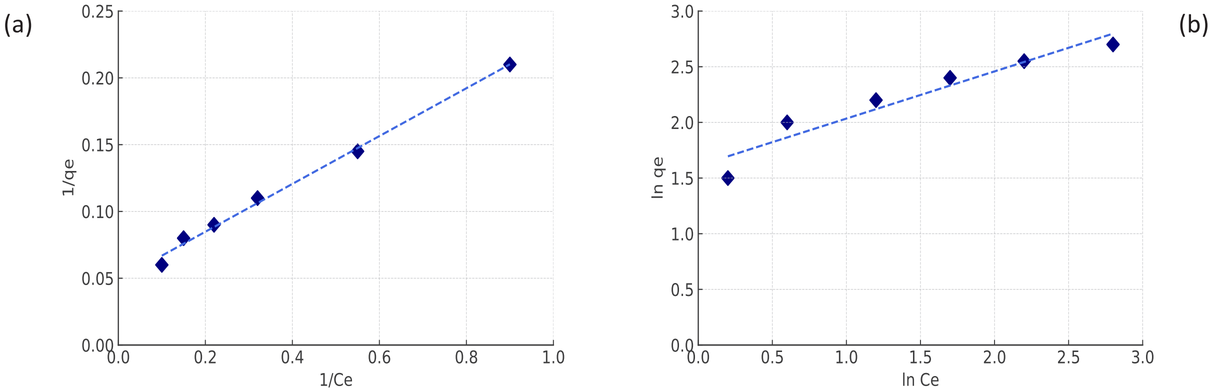

Fig. 9. The isotherm plots of (a) Langmuir, (b) Freundlich models.

the acidic medium (Fig. 3). On the other hand, at pH above 5, the functional groups on the biosorbent surface were deprotonated, thus the biosorbent surface would be negatively charged. This improves the interaction between the positively charged CV ions and the negatively charged active groups on the *Lagurus ovatus* surface. Therefore, the electrostatic attraction between CV dye ions and the negatively charged surface sites existing on the *Lagurus ovatus* surface can be suggested as the main adsorption mechanism. The suggested mechanism for CV adsorption onto *Lagurus ovatus* surface is illustrated in Fig. 10.

4. Conclusion

The capability of *Lagurus ovatus* to eliminate crystal violet from aqueous media was examined in this paper. The impact of the adsorption factors reveals that the acidic medium is unfavourable for the adsorption system. The optimal conditions for the removal process were found, which is the removal time was 40 min, the biosorbent dose was 2 g/L, and the starting crystal violet concentration was 20 mg/L, and the temperature was 50 °C. The thermodynamic investigation confirms the endothermic and spontaneous behaviour of the removal system. In addition, the kinetic removal data applied perfectly to the pseudo-second order equation. Moreover, a better fit for the removal data was achieved by Langmuir isotherm. Furthermore, the electrostatic attraction between the cationic dye and the biosorbent surface is the dominant adsorption mechanism. These findings suggest that *Lagurus ovatus* can represent an optimistic new and cost-effective biosorbent for eliminating crystal violet dye from aqueous media.

Acknowledgments

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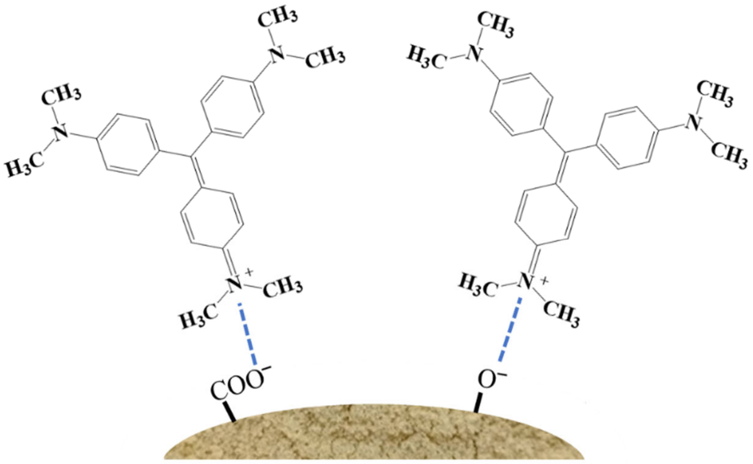

Fig. 10. Suggested mechanism of MO dye adsorption onto *Lagurus ovatus* surface.

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