

JAMUN SEED STONE ADSORBENT FOR THE REMOVAL OF CRYSTAL VIOLET DYE FROM AQUEOUS SOLUTIONS: AN RSM BASED APPROACH

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ABSTRACT. This study aims to apply Indian jamun seed stone (JSS) as an adsorbent for the removal of crystal violet (CV) dye from aqueous solution. The adsorbent was characterized with FTIR, SEM, and EDX techniques. Batch adsorption studies were performed for optimization of independent variables developed by CCD of RSM. The developed design matrix was found to be significant with a p-value < 0.0001, and the F-value was observed to be 125.4. The lack of fit was noted as non-significant, with a value being > 0.05, and the correlation coefficients, adjusted and predicted, were all close to one, making the model reliable and significant. The loading capacity of JSS was calculated to be 129.2 g mg⁻¹ towards CV ions from aqueous solutions. The isotherm analysis confirms the applicability of Langmuir and the Freundlich models to the present system, and the D-R isotherm confirms the physisorption mechanism for the removal of CV ions by JSS. The activation energy (E_a) was calculated from kinetic studies to be 32.46 kJ mol⁻¹, which supports the physisorption. The thermodynamics investigations suggest the spontaneous exothermic nature of the CV ions removal by JSS. The results conclude that JSS is an excellent adsorbent for the removal of CV ions from aqueous solutions.

KEY WORDS: Jamun seed stones, Crystal violet, Adsorption, RSM

INTRODUCTION

Environmental contamination has become a serious concern for humanity due to its adverse effects on ecosystems and life on Earth. Advances in technology and the use of specialty chemicals in finished products are ever-increasing, leading to their release into the environment through point and non-point sources [1]. The growing demand for aesthetic products has paved the way for the widespread use of colorful synthetic dyes, making the resulting dye-containing effluents a significant concern, particularly for aquatic ecosystems. Crystal violet (CV) is one of the most widely used synthetic dyes in industries, especially in dyeing [2]. Although the dye has various biomedical applications, such as antifungal, antimicrobial, and antiseptic uses, its primary application today is in the dyeing industry, which has led to the release of large quantities of the dye into the environment [3]. Direct contact with CV is reported to be carcinogenic, and several countries have banned its use in food and any form of adulteration [4]. Therefore, it is essential to remove CV from effluents or wastewater before they are discharged into aquatic systems.

Adsorption is one of the most commonly employed techniques for the treatment of wastewater and effluents to remove contaminants from aqueous solutions [5]. To make the adsorption process eco-friendlier and more cost-effective, agricultural wastes and their by-products have been

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explored as alternative adsorbents for the removal of metal ions and synthetic dyes [6]. In one study, watermelon rind was used as an adsorbent for the removal of crystal violet (CV) ions from aqueous solution, achieving a loading capacity of 104.7 mg g^{-1} [7]. This capacity was found to be significantly higher than that of several commercial adsorbents reported in the literature. In another study, surface-modified garlic peels were investigated as an adsorbent for the removal of CV ions in a continuous column mode, and the results demonstrated effective removal of CV ions under dynamic conditions [8]. Lime peels and pineapple leaves have been explored for the removal of CV, achieving removal efficiencies of 98% and 97%, respectively [9]. FTIR analysis revealed that carboxyl and carbonyl groups were involved in the removal process. Durian seed powder was investigated for CV removal, achieving a maximum removal of 93% with a loading capacity of 158 mg g^{-1} , where electrostatic forces, $n-\pi$ stacking, and hydrogen bonding interactions were found to dominate the binding mechanism [10]. Studies using *Ficus religiosa* leaves and *Daucus carota* pomace for CV removal reported loading capacities of 2.4 and 27 mg g^{-1} , respectively [11]. Although numerous studies have investigated the removal of CV ions from aqueous solutions using various agro-wastes, the reported loading capacities are often low, and removal efficiencies rarely reach 100%. Therefore, it is essential to identify a suitable agro-waste that can achieve 100% removal efficiency with high loading capacity. While fruit peels have been widely studied, research on the use of fruit stones for CV removal remains limited, and Jamun seed stones, in particular, have not yet been explored for this purpose.

In this study, we aim to explore the potential of Jamun seed stones for the removal of CV ions from aqueous solution. The adsorbent was prepared using a conventional method to ensure it is eco-friendly and effective for the adsorption of contaminants. The prepared adsorbent was characterized using various analytical techniques to understand its surface properties.

EXPERIMENTAL

Materials

Crystal violet was sourced from Sdfine chemicals, HCl, CH_3COOH , and NaOH were procured from Merck Limited.

Preparation of adsorbent from JSS

The Jamun seed stones were obtained from a local vendor and washed several times to remove dirt and dust from their surfaces. The washed stones were then dried in the sunlight for seven consecutive days to eliminate moisture. After sun-drying, the stones were deshelled, and the hard inner part was crushed into a powder using a conventional mixer. The crushed powder was stirred in distilled water to remove water-soluble natural dye molecules from the stones. The resulting mixture was filtered, and the solid residue was dried in an oven at 80°C for 4 hours before being used in batch adsorption experiments.

Box behnken design of RSM

Response surface methodology is one of the powerful tools to optimize the conditions for the adsorption experiments, and Box Behnken Design (BBD) is a useful design matrix that considers a second-order polynomial equation for designing an experimental run to identify the optimal conditions of the independent variable for maximum adsorption. In this study, BBD was adopted to optimize independent variables such as pH, contact time, and initial concentrations with minimum and maximum levels as shown in Table 1. The % removal and loading capacity of the JSS towards CV ions were calculated using the following equations

$$\% \text{ Removal} = \frac{C_0 - C_1}{C_0} \times 100 \quad (1)$$

$$q_e = \frac{C_0 - C_1}{m} \times V \quad (2)$$

Where q_e is the loading capacity of the JSS adsorbent in mg g^{-1} , C_0 and C_1 are the initial and final concentrations of CV solutions, V is the volume of the solution, and m is the mass of the JSS adsorbent.

Table 1. Levels and symbols of independent variables for optimization using BBD of RSM.

Variables	Symbol	Levels		
		-1	0	+1
pH	A	4	6	8
Contact time	B	30	75	120
Initial Concentration	C	50	100	150

RESULTS AND DISCUSSION

Characterization of Jamun seed stone

FTIR

FTIR analysis reveals the presence of various functional groups on the surface of the adsorbents, and in this study, the jamun seed stone adsorbent was analyzed with FTIR, as shown in Figure 1. The spectra reveal the presence of various functional groups in the adsorbent. A broad band at 3275 cm^{-1} confirms the presence of -OH groups, and 2927 cm^{-1} is due to the -CH groups of the biomolecules present in the stone adsorbent. A band at 1718 cm^{-1} corresponds to the Carbonyl group (C=O) of aldehydes or ketone groups, and 1614 cm^{-1} represents the asymmetric vibrations of hydroxyl groups. Bands between 1337 and 998 cm^{-1} correspond to the aromatic -C=C- and carboxyl groups. These observations suggest that the presence of hydroxyl, carbonyl, and carboxyl groups can effectively be involved in the capture of CV ions from aqueous solutions by JSS.

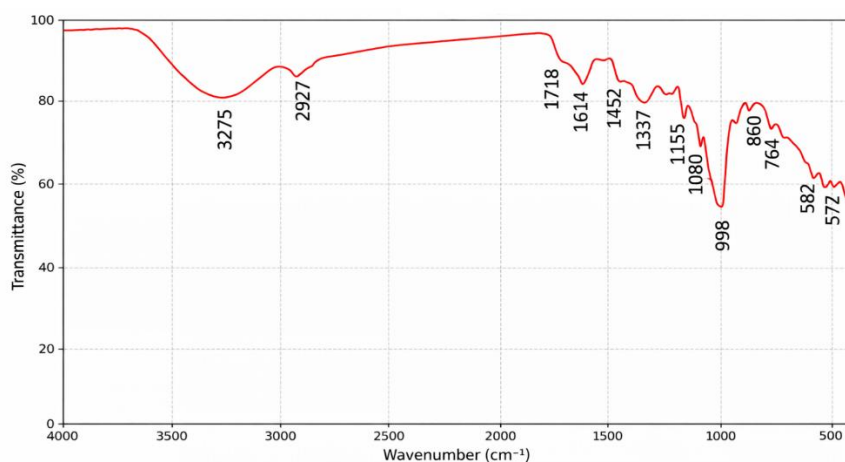


Figure 1. FTIR of jamun seed stone adsorbent.

SEM

The surface morphology of the JSS was analyzed with SEM, and the surface was found to be irregular with pores (Figure 2a). The irregular and porous surface can entrap the CV ions effectively on the surface, which can lead to enhanced removal from aqueous solutions. The elemental analysis of the JSS was performed with EDX analysis, and the pattern is presented in Figure 2b. It is noticed that the EDX patterns contain elements of C, O, and K, and the presence of K can facilitate the ion-exchange process with CV ions. These observations conclude that JSS is a suitable adsorbent for the removal of cations from aqueous solution.

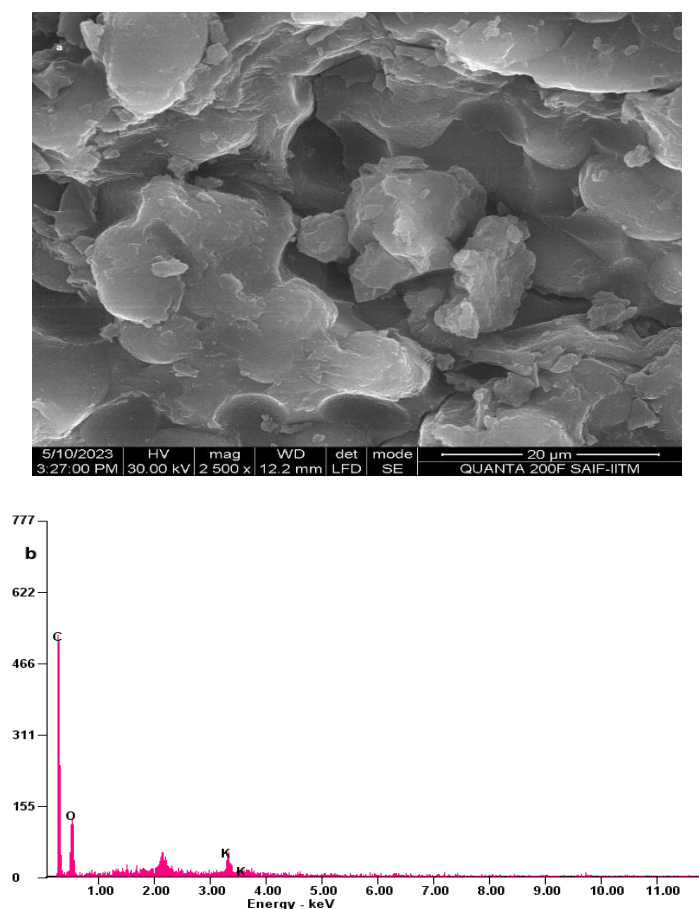


Figure 2. a) SEM image b) EDX pattern of Jamun seed stone adsorbent.

BBD – RSM

Box Behnken Design of RSM was adopted to optimize the independent variables, and a total of 17 runs with a combination of independent variables were performed as reported in Table 2. The experimental values obtained at various combinations are in good agreement with the predicted values that were developed from the BBD. A quadratic second-order polynomial equation

obtained for the three independent variables used in this study is presented in Eq. 3. The equation indicates a high baseline % removal when all the factors are at their center. The positive values of A and B indicate the rise in removal efficiency with an increase in value, and the negative value of C indicates that the decrease in removal occurs with a rise in initial concentration.

$$\% \text{ Removal} = +76.86 + 6.81 A + 6.41 B - 4.90 C + 3.50 AB - 9.88 AC - 0.0250 BC - 41.78 A^2 - 4.13 B^2 + 0.5950 C^2 \quad (3)$$

The ANOVA data of the present model are summarized in Table 3. A F-value of 125.4 indicates the applicability and reliability of the model, and it is further supported by the p-value being < 0.0001 , which is found to be significant. The high F-value indicates the suitability of the model, and there is only 0.01 % of chance of obtaining such a high value due to errors. The lack of fit obtained in this study was found to be 9.51 with a p-value of 0.504, which is found to be insignificant. Further, the pure error observed in this study is small, suggesting a good consistency of the model, and the observed small value might be due to random noise and not from the experimental flaws of this study. An adequate precision value of 29.51 suggests the highly reliable optimization of the model with a moderate std. deviation of 2.80. The coefficient of variation was found to be 5.05, suggesting the high precision of the model. The ANOVA data highlights the precision and applicability of the model for the effective removal of CV ions by JSS.

Table 2. Experimental and predicted removal percentage with experimental runs for the removal of CV ions by JSS.

Std	Run	A:pH	B:Contact time	C:Initial Concentration	Removal %	
					Exp	Predicted
			min	mg/L		
14	1	6	75	100	76.5	76.86
13	2	6	75	100	76.9	76.86
1	3	4	30	100	22.1	21.22
7	4	4	75	150	31.8	33.84
5	5	4	75	50	26.5	23.89
6	6	8	75	50	59.3	57.26
16	7	6	75	100	77.2	76.86
12	8	6	120	150	78.3	74.81
17	9	6	75	100	76.8	76.86
11	10	6	30	150	63.2	62.04
15	11	6	75	100	76.9	76.86
2	12	8	30	100	29.3	27.85
9	13	6	30	50	68.3	71.79
10	14	6	120	50	83.5	84.66
4	15	8	120	100	46.8	47.68
8	16	8	75	150	25.1	27.71
3	17	4	120	100	25.6	27.05

Table 3. ANOVA for the removal of CV ions by JSS.

Source	Sum of Squares	df	Mean Square	F-value	p-value
Model	8866.47	9	985.16	125.45	< 0.0001
A-pH	371.28	1	371.28	47.28	0.0002
B-Contact time	328.96	1	328.96	41.89	0.0003
C-Initial Concentration	192.08	1	192.08	24.46	0.0017
AB	49.00	1	49.00	6.24	0.0411
AC	390.06	1	390.06	49.67	0.0002
BC	0.0025	1	0.0025	0.0003	0.9863
A ²	7349.76	1	7349.76	935.94	< 0.0001
B ²	71.82	1	71.82	9.15	0.0193
C ²	1.49	1	1.49	0.1898	0.6762
Residual	54.97	7	7.85		
Lack of Fit	54.72	3	18.24	9.51	
Pure Error	0.2520	4	0.0630		
Cor Total	8921.44	16			
R ²	Adjusted R ²	Predicted R ²	Adeq precision	Std, Dev	C.V.%
0.993	0.985	0.901	29.51	2.80	5.05

The 3D surface plots obtained in this study are represented in Figure 3. The interaction between pH and contact time (Figure 3a) indicates that the removal efficiency increases with pH and is found to be maximum at pH 6. Further increase in pH reduces the removal efficiency, and this observation is attributed to the PZC value of the JSS, which was found to be pH 5.7. Above pH 5.7, the surface of the JSS is negative, and it attracts the positively charged CV ions onto its surface for binding. This provides a positive environment for the surface binding. The decrease in efficiency beyond pH is due to the loss of cationic charge of the CV ions, which tend to repel from the surface, and further due to dye aggregation and change in solubility [12]. With an increase in contact time, the removal efficiency increased, leading to ample time for the CV ions to interact with the JSS surface for binding. However, with a rise in contact time, the removal efficiency decreased beyond pH 6. In the case of pH vs initial concentration, the removal efficiency was seen to be high at low concentrations and found to decrease with an increase in concentration (Figure 3b). The optimal pH was found to be pH 6 to obtain maximum efficiency at the initial concentrations investigated in this study. The interaction between contact time and initial concentration is presented in Figure 3c, and it is observed that at low initial concentrations and at higher contact time, maximum removal efficiency is obtained. These observations are consistent with the earlier reports [13]. The predicted and experimental values plot obtained in this study exhibits the uniform distribution of the points along the straight line, indicating the linearity and suitability of the model (Figure 3d).

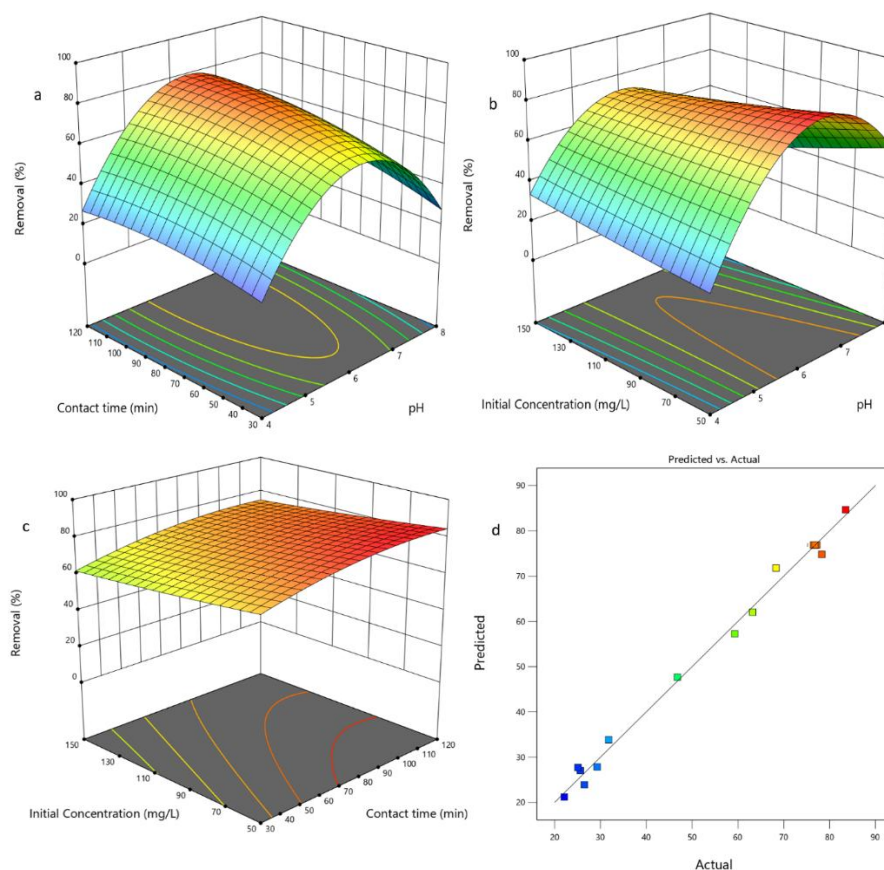


Figure 3. 3D Surface plots of a) pH vs Contact time, b) pH vs Initial concentration, c) contact time vs initial concentration, and d) Predicted vs Actual for the removal of crystal violet dye from aqueous solution by Jamun seed stone adsorbent.

Adsorption isotherms

The equilibrium data obtained in this study for the removal of CV ions by JSS were analyzed with three isotherm models, such as Langmuir, Freundlich, and D-R isotherm models, and their respective correlation coefficients and constants are shown in Figure 4a. The correlation coefficients obtained for the non-linear equations suggest that the Langmuir and the Freundlich isotherms are close to one, suggesting a better fit compared to the D-R model (Figure 4a). The maximum monolayer capacity Q_m obtained in this study was calculated to be 129.2 mg g^{-1} , and it is in close agreement with the experimental value that was calculated to be 123.5 mg g^{-1} , supporting the better applicability of the Langmuir model. A n value of 4.97 suggests the favorable adsorption of CV ions by JSS. Though the correlation coefficient was less for the D-R model, the mean potential energy (E) obtained from the model provides valuable information on the mechanism of adsorption. The E value obtained in this study was calculated to be 1.58 kJ mol^{-1} , suggesting the physisorption process for the removal of CV ions by JSS. Similar results were reported for the removal of brilliant green dye by modified jamun seeds [14].

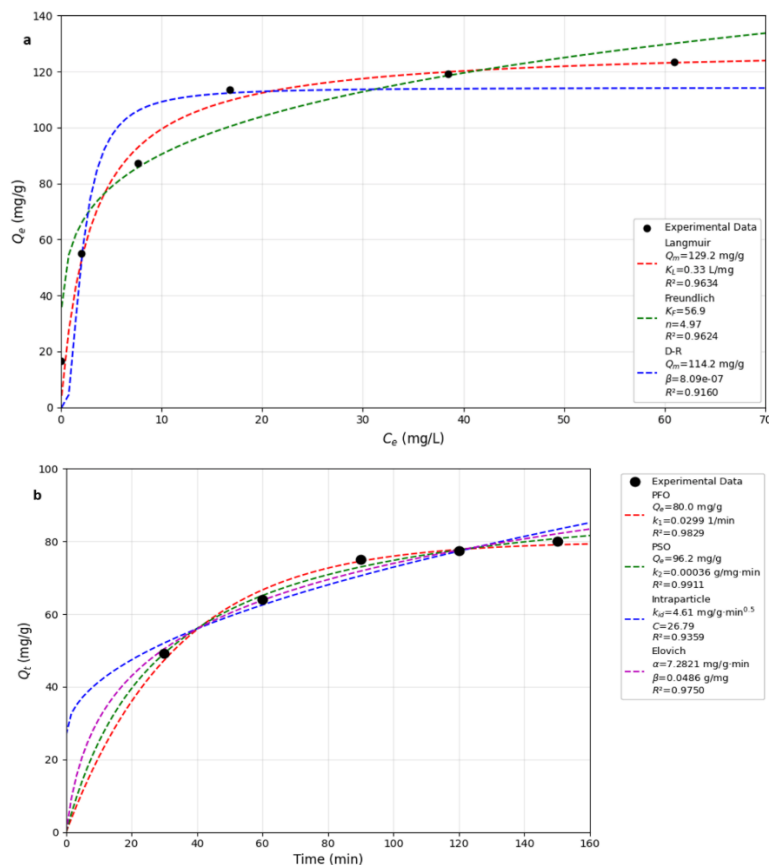


Figure 4. a) Isotherm and b) kinetic plots obtained for the removal of crystal violet dye from aqueous solution by Jamun seed stone adsorbent.

Kinetics of adsorption

The kinetic data were analyzed with four kinetic models, and their respective plots obtained for the non-linear equations are represented in Figure 4b. The correlation coefficients of Pseudo first order, second order, and Elovich were found to be high, and comparatively, the pseudo second order value is close to one, suggesting a better fit of the model. The activation energy (E_a) calculated from PSO was found to be $32.46 \text{ kJ mol}^{-1}$, which suggests the mechanism to be physisorption, and this is in good agreement with the findings from the D-R isotherm. The intraparticle diffusion was found to be the least fit model for the kinetic data with a diffusion rate of $4.61 \text{ mg g}^{-1} \text{ min}^{-0.5}$. An α value of $7.28 \text{ mg g}^{-1} \text{ min}^{-1}$ indicates a rapid initial adsorption, and this is due to the chemically diverse adsorbent surface of JSS [15]. A low β value of 0.048 g mg^{-1} indicates a stronger interaction between JSS and CV ions. This value aligns with the Freundlich isotherm describing the multilayer adsorption on chemically diverse surfaces [16].

Thermodynamics of adsorption

The thermodynamic investigations were performed for the removal of CV ions by JSS to understand the nature of adsorption, and the results are summarized in Table 4. The results indicate that the Gibbs free energy is negative, suggesting the spontaneous nature of CV ions removal by JSS, and with a rise in temperature, the spontaneity declines [17]. The enthalpy was noted to be negative, suggesting the exothermic nature of the process, and entropy was noted to be negative, suggesting the decrease in randomness at the interface of the CV and JSS. The decrease in free energy values is consistent with the physisorption process observed in the isotherm and kinetic studies [18].

Table 4. Thermodynamic parameters obtained for the removal of CV ions by JSS.

Temperature (K)	ΔG° (kJ mol ⁻¹)	ΔH° (kJ mol ⁻¹)	ΔS° (J mol.K ⁻¹)
303	-4.13	-10.24	-20.15
313	-3.93		
323	-3.73		

Desorption studies

It is essential to perform desorption investigations to make the adsorbent ready for consecutive cycles. In this study, desorption investigations were performed for the removal of CV ions from the JSS surface with four different desorbing agents, and the results are presented in Figure 5. It is noticed that the 0.01 M HCl exhibited higher removal efficiency compared to the other three adsorbents used in this study. This is due to strong proton competition onto the JSS. Acetic acid exhibited moderate efficiency due to weaker interactions. Similarly, the bases exhibited low efficiency, which is due to anion domination and weak ion exchange. The high desorption efficiency achieved highlights the reuse of the adsorbent for repeated cycles.

Mechanism of adsorption and superiority of JSS

The JSS has shown a superior adsorption capacity of 129.2 mg g⁻¹ towards CV ions from aqueous solutions. The superior adsorption of JSS is due to various factors that govern the adsorption of CV ions, and the primary factor is due to the electrostatic attraction of positively charged CV ions onto the negatively charged JSS surface at pH 6. Further, the functional groups present on the surface of the JSS, such as hydroxyl and carboxyl, evidenced from FTIR, effectively bind the positively charged CV ions. Additionally, the surface of the adsorbent will interact with the aromatic structures of the CV via π - π interaction for surface binding, and collectively these processes lead to the superior adsorption capacity. The physical adsorption is further favored by mean potential energy values being less than 8 kJ mol⁻¹ and activation energy, suggesting the physical adsorption nature of JSS. The superior adsorption capacity of JSS is further supported by comparison with other reported adsorbents in the literature (Table 5). The adsorption capacity of JSS is higher than several agro-waste-based adsorbents towards CV ions, highlighting the industrial applicability and real-time water treatments.

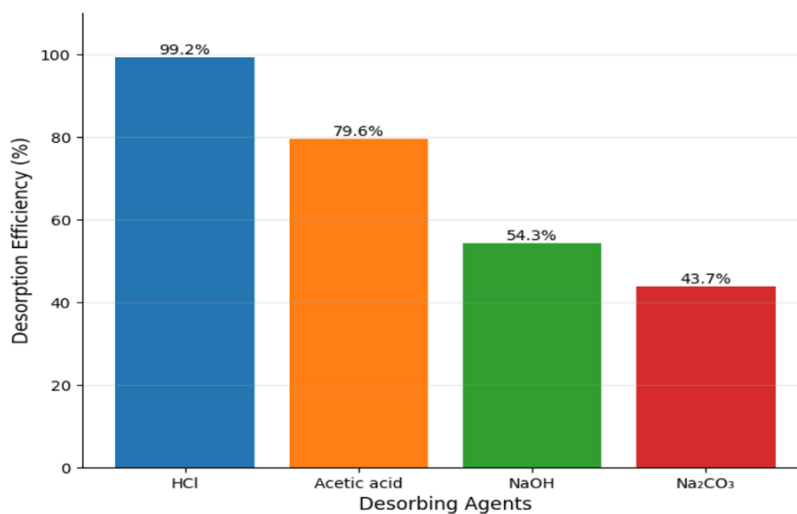


Figure 5. Desorption efficiency comparison chart for the desorption of CV ions from JSS.

Table 5. Comparison of the loading capacity of JSS with other reported adsorbents.

S.No	Adsorbent	Loading capacity (mg g ⁻¹)	Reference
1	Almond shell	12.2	19
2	Lemon peel	71.2	20
3	Peanut husk	20.9	21
4	Papaya peel	99.0	22
5	Rubber seed	23.8	23
6	Jamun seed stone	129.2	This study

CONCLUSION

This study conclusively establishes the Jamun seed stone (JSS) as a highly potent, novel, and sustainable biosorbent for the remediation of crystal violet (CV) dye from contaminated water. The strength of JSS lies in its remarkable loading capacity of 129.2 mg g⁻¹, a value that positions it as a competitive alternative to many conventional and commercial adsorbents. The adsorption process is governed by a spontaneous physisorption mechanism, which facilitates easy desorption and underpins the material's excellent reusability over multiple cycles, as confirmed by desorption studies. These findings underscore the significant potential of JSS for real-world industrial applications, particularly in the pretreatment of textile, paper, and leather processing effluents, where dye contamination is a critical concern. Its derivation from agricultural waste offers a cost-effective and eco-friendly solution for color removal, aligning with circular economy principles. Future research should focus on transitioning these promising batch-scale results to continuous-mode operations. Pilot-scale studies using fixed-bed columns and real industrial wastewater are essential to validate performance under practical conditions. Additionally, a direct comparative analysis with commercially available activated carbons will further quantify the economic and environmental advantages of JSS, solidifying its role in sustainable wastewater management.

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Conflicts of interest

Authors declare no conflicts of interest

Data availability

All the required data are available within the manuscript

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