

# Adsorption of hazardous dye crystal violet from industrial waste using low-cost adsorbent *Chenopodium album*

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# ABSTRACT

The present article describes the use of *Chenopodium album* ash (wildly growing weed) as effective adsorbent for the removal of a hazardous dye, crystal violet, from its aqueous solutions. This paper presents an experimental study and discussion of the adsorption characteristics of this dye on the plant ash. Two techniques, that is, batch and column operations have been used to explain the removal process. Column capacity is found to be lesser than the batch adsorption capacity. Batch adsorption studies were conducted as a function of adsorbent dose, equilibrium pH, contact time, initial dye concentration, kinetics and Freundlich isotherms. Extent of adsorption has been found to be greater at neutral pH. Kinetic studies indicate that the overall adsorption process is best described by pseudo-first-order kinetics. The adsorption data were fitted to linearly transformed Freundlich isotherm with  $R^2$  (correlation coefficient) 0.999. Values of Freundlich parameters *n* and *K<sub>j</sub>* have been found to be used as an effective and low-cost adsorbent for the treatment of wastewaters contaminated with organic dye crystal violet.

Keywords: Crystal Violet; Chenopodium album; Adsorption; Dye removal; SEM; Isotherm

#### 1. Introduction

The continuously increasing use of organic dyes in number of industries viz. textile, paper, plastics, rubber, leather treatment, etc. has led to release of these hazardous pollutants to water bodies. Most of the organic dyes do not degrade easily because of their complex aromatic structure [1]. Due to the foreseen water scarcity, wastewater treatment is the issue to be taken up on priority. It is need of time to carry out studies on removal of dyes from industrial waste. Various techniques viz. coagulation, chemical oxidation, membrane separation process, adsorption, electrochemical,

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and aerobic and anaerobic microbial degradation have been developed for treatment of dye contaminated water [2,3]. Adsorptive removal of pollutants is a cheap and convenient method for treatment of dye-contaminated wastewater as compared with other techniques such as coagulation, chemical oxidation, membrane separation and electrochemical method of treatment. A number of studies on applications of natural biosorbents as well as synthetic nanomaterials, and surface modified materials viz. bottom ash, deoiled soya, hen feather have been carried out for removal of organic dyes and other pollutants including heavy metal ions from effluents [4–13].

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The widely growing weed Chenopodium album (manure weed) is also known as pigweed and lambsquarters [14]. It is widely distributed throughout the world, mainly in Asia, Europe, North America and Africa. It grows in nitrogen-rich soils and some places it is consumed as leaf vegetable due to its high nutritious value. Manure weed is a polymorphous, erect herb, having height up to 3.5 m. Its stems are slender, angled, striped green, red or purple. Its leaves are 10-15 cm long, rhomboid, deltoid to lanceolate upper entire, lower toothed or irregularly lobed, petioles often as long as thick panicled, shinning black seeds, possessing sharp margins. In India, 21 species of C. album (manure weed) are found in Western Rajasthan, Kullu Valley and Shimla. Manure weed or C. album is traditionally used as antihelmintic, cardiotonic, carminative, digestive, diuretic and laxative. It is also useful in peptic ulcer, dyspepsia, flatulence, strangury, pharyngopathy, splenopathy, opthalmopathy and general debility [14-17]. It is widely available throughout the country. Removal of pollutants using agricultural waste as adsorbent is an economical and effective technique. Chenopodium album belonging to family Chenopodiaceae and genus Chenopodium has been reported to be used as adsorbent for removal of heavy metals from water bodies [15]. However, it has not been explored for adsorptive removal of organic dyes from industrial waste so far. So, the present study has been undertaken to explore the potential role of C. album ash for removal of crystal violet from wastewater. Crystal violet (CV), which is a cationic dye and belongs to the class of triarylmethane dyes, is mainly used in several industries such as dyeing and textile, paper, ball point pen, leather, additives, cosmetics and analytical chemistry/biochemistry. It is also used as pH indicator. It is also named as Basic Violet 3, gentian violet and methyl violet 10B. Its molecular formula is C<sub>25</sub>H<sub>30</sub>N<sub>3</sub>Cl and molecular weight is 407.98 g/mol. Structure of dye has been shown in Fig. 1. Toxicological investigations have revealed that CV has carcinogenic and mutagenic effects in rodents. It may cause eye irritation, skin irritation, digestive tract irritation, permanent injury to cornea and conjunctiva. In extreme condition, it may lead to respiratory and kidney failure and permanent blindness. Discharge of water containing this dye into water bodies can cause environmental degradation and the dye is reduced to leuco moiety, leucocrystal violet [18–21].

In the present investigation, *C. album* ash was prepared and explored for its potential in removing crystal violet dye from aqueous solution. Effect of various parameters viz. pH, initial dye concentration, dose, temperature and contact



Fig. 1. Structure of crystal violet.

time was evaluated. The adsorption process was analyzed according to kinetics and adsorption equilibrium data. Two techniques namely batch and column processes have been applied to study dye removal by *C. album* ash in the present investigation.

#### 2. Experimental section

#### 2.1. Development of adsorbent

In the present investigation, ash of *Chenopodium album* has been explored for adsorptive removal of crystal violet. Aerial parts of *Chenopodium album* were collected, washed and dried under shade at  $25^{\circ}$ C ±  $3^{\circ}$ C for 15 d. The dried material was ground to a fine powder to pass through a 300 mesh size sieve. The fine powder is then converted into ash by keeping it in a muffle furnace at 823 K for 3 h. After cooling to room temperature, the fine ash powder was collected and used without any chemical treatment.

#### 2.2. Materials and methods

Crystal violet (CV), a blue-violet colored nitrogen containing basic dye, is purchased from Merck (India) and used without further purification. 100 mg/L stock solution of crystal violet dye was prepared. For adsorption study, aliquots of dye stock solution were diluted to make subsequent dye solutions. Study of effect of various parameters on dye removal was carried out using 10 mL dye solutions and after removal of dye concentration of remaining dye was determined by recording absorbance at the wavelength  $(\lambda_{max})$  of 589 nm. UV–Vis spectra of the dye solution were obtained using a Shimadzu UV 1800 spectrophotometer with a quartz cuvette of 10 mm length. SEM images of the bioadsorbent before and after adsorption were captured on MerlinVP Compact (Carl ZEISS Germany make) having an air lock chamber. Surface area and pore volume of the adsorbent manure weed ash were determined using BET method (Sorptomatic 1990) after degassing at 383 K under vacuum, using nitrogen adsorption at 77 K and desorption while allowing the temperature to rise to ambient.

#### 2.3. Dye adsorption study

Adsorption study of crystal violet was carried out using a batch process of mixing 5 mg of plant ash in a test tube with 10 mL of crystal violet solutions of concentration ranging from 2 to 6 mg/L. Change in concentration of dye solution was measured by recording absorbance of dye solutions at a wavelength ( $\lambda_{max}$ ) of 589 nm.

The percentage removal efficiency of *C. album* plant ash and the amount of dye on plant ash at equilibrium  $(Q_{e})$  and the amount of dye on plant ash at time  $t(Q_{i})$  were calculated using earlier reported method [7].

Effect of various parameters viz. pH, temperature, adsorbent dose, dye concentration and contact time were carried out.

#### 2.4. Column operation study

For column operation study on dye removal, a weighed sample of ash of manure weed was filled in a glass column of total length 18 cm and internal diameter 1 cm. The dye containing influent with initial concentration of 5 mg/L was allowed to pass through the bed of adsorbent in the down flow method at 1.3 mL/min and the effluent was collected in a 6.5 mL samples at a regular time interval and analyzed without previous filtration. The column operation was stopped after about 80% exhaustion because of column getting choked. The breakthrough curve [21] is plotted in terms of  $C_t/C_0$  vs. time, where  $C_t$  is the concentration of effluent at time t and  $C_0$  is the initial concentration influent dye solution. Breakthrough capacity (BC), exhaustion capacity (EC) and degree of column (DOC) utilization are the important features of a breakthrough curve. Breakthrough capacity is defined as the amount of dye eliminated by the adsorbent at breakthrough concentration. Exhaustion capacity is the amount of dye adsorbed by the unit weight of the manure weed ash at the point of saturation. DOC utilization is the ratio of amount of dye adsorbed at breakthrough to the amount of dye adsorbed at complete saturation [22].

#### 3. Result and discussion

# 3.1. Characterization of adsorbent

The surface textural and morphological properties of the developed adsorbent were carried out using SEM imaging. Scanning electron micrographs of *C. album* ash after adsorption exhibited the coverage of ash surface by crystal violet dye confirm adsorption at the active sites of ash surface. Chemical constitution of *C. album* ash before and after adsorption was studied by EDX analysis. The percentage composition of carbon was increased after adsorption of dye molecules from 8.1% to 11.9%, which further confirms adsorption of dye at surface of binding sites of plant ash.

Some other important physical properties of the material, such as pore volume and surface area, are also determined by standard procedures. The values of pore volume and surface area are found to be 0.018 cm<sup>3</sup>/g and 9.14 m<sup>2</sup>/g, respectively.

# 3.2. Adsorption of crystal violet onto C. album ash

#### 3.2.1. Effect of initial dye concentration

Removal of dye by plant ash was studied at several different initial dye concentrations ranging from 2 to 6 mg/L. The results are presented in Fig. 2. It has been observed that with increase in the initial dye concentration, dye removal percentage decreased from 94.4% to 89.5% after 24 h of adsorption process. The result indicates saturation of binding sites on the surface of adsorbent as the concentration of crystal violet increased [23].

#### 3.2.2. Effect of adsorbent dose

To study the effect of sorbent dose on adsorption, 5–25 mg of plant ash were added to dye solutions having concentrations of 3 ppm (Fig. 3). Results reveal that the removal of the dye increased as the amount of ash increased due to increase in the surface area and adsorption sites of the adsorbent [24].



Fig. 2. Effect of initial dye concentration on percentage adsorption of dye on adsorbent.



Fig. 3. Effect of dose of ash on adsorption.

#### 3.2.3. Effect of temperature

Effect of temperature on adsorption of dye was studied at different temperatures viz. 303, 308, 313, 318 and 323 K. The results of these experiments are presented in Fig. 4. The removal of dye from its aqueous solution increases with increasing temperature, revealing the endothermic nature of adsorption process and increase in mobility of dye molecules with increase in temperature [25,26].

#### 3.2.4. Effect of contact time

In order to study the effect of contact time on adsorption process, the experiments were carried out for different contact time with constant amount of adsorbent (5 mg). The absorbance of remaining dye was recorded at different time intervals. The result obtained (Fig. 5) reveal increase in removal of crystal violet dye with increasing contact time. Initially, the plant ash reflects rapid removal of dye, later



Fig. 4. Effect of temperature on adsorption of crystal violet on manure weed ash.



Fig. 5. Effect of contact time.

the rate of adsorption decreases gradually. This is possibly due to the presence of a large number of vacant sites at the surface initially and after a period of time the dye molecules get adsorbed at the surface of ash and it becomes difficult to occupy vacant sites due to repulsive forces between CV adsorbed on the surface of manure weed ash and the CV dye present in solution phase [27].

### 3.2.5. Effect of initial pH of dye solution

The impact of pH of dye solution on adsorption process is shown in Fig. 6. It is clear that removal of Crystal violet dye is higher at pH 7. In addition removal of the dye is slightly higher in pH range of 6.5–8.5 after attaining equilibrium (24 h). As the pH is increased, the surface charge density on the ash changes and the adsorbent becomes negatively charged resulting in an enhanced attraction between the positively charged dye molecule and the adsorbent surface. A similar trend is reported for basic dye removal [28].

### 3.3. Adsorption isotherm study

Relationship between amount of dye adsorbed by unit mass of adsorbent and the remaining amount of dye in the solution after adsorption can be explained by the use of adsorption isotherms. In the present work, three adsorption isotherms Freundlich, Langmuir and Temkin have been explored.

Freundlich adsorption isotherm is based upon the heterogeneous adsorption of adsorbate molecules on the adsorbent surface having unequal binding sites with different adsorption energies [29]. Linear form of Freundlich isotherm is given by the equation.

$$\ln Q_e = \ln K_f + \frac{1}{n} \ln C_e \tag{1}$$

Here,  $Q_e$  (mg/g) is the amount of crystal violet adsorbed by the ash at equilibrium,  $C_e$  (mg/L) is the equilibrium concentration of crystal violet solution,  $K_f$  and n are the Freundlich constants which are related to adsorption capacity and adsorption intensity, respectively.

A plot of  $\ln Q_e$  vs.  $\ln C_e$  gives a straight line whose slope and intercept give the value of n and  $K_{j'}$  respectively. Value of 1/n is useful to describe the removal ability of the adsorbent used over the concentration range of the dye solution. The adsorbent can adsorb the dye only from its high concentration solution if the value of 1/n is higher than 1. On the other hand, value of 1/n < 1 suggests the applicability of the adsorbent for entire range of dye solution [30]. Fig. 7 shows a linear plot for Freundlich adsorption isotherm for the present work. Related parameters are listed in Table 1.

According to Langmuir adsorption isotherm, there is a fixed number of homogeneous active binding sites on the surface of any adsorbent material and adsorption of adsorbate material stops as these active binding sites get saturated.



Fig. 6. Effect of initial pH of dye solution.

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Fig. 7. Linear plot for Freundlich adsorption isotherm.



Fig. 8. Linear plot for Langmuir adsorption isotherm.

Table 1 Isotherm parameters of crystal violet adsorption

S. No.	Adsorption isotherms	Parameters	$R^2$
1	Freundlich isotherm	n = 1.642, 1/n = 0.609	0.999
		$K_f = 14.253$	
2	Langmuir isotherm	Q <sub>L</sub> = 18.518	0.963
		$K_{L} = 2.077$	
3	Temkin isotherm	$b_T = 617.386$	0.964
		$K_{T} = 20.510$	

This isotherm explains monolayer adsorption process [24,29]. Langmuir isotherm is expressed as follows:

$$\frac{C_e}{Q_e} = \frac{1}{K_L Q_L} + \frac{C_e}{Q_L}$$
(2)

where  $Q_e$  (mg/g) is the amount of Crystal violet adsorbed by the ash at equilibrium,  $C_e$  (mg/L) is the equilibrium concentration of crystal violet solution,  $K_L$  and  $Q_L$  refers to Langmuir isotherm constant and maximum monolayer coverage capacity, respectively. Fig. 8 shows a linear plot for Langmuir adsorption isotherm for the present work and related parameters are listed in Table 1.

Temkin adsorption isotherm indicates the adsorption of adsorbate on energetic and non-equivalent adsorption sites on the surface of adsorbent and the adsorption takes place on the more energetic adsorption sites at first [31]. There is a linear increase in the heat of adsorption with the coverage of adsorbate molecules over the adsorbent surface [29]. Linear form of Temkin isotherm is expressed as:

$$Q_e = \frac{RT}{b_T} \ln K_T + \frac{RT}{b_T} \ln C_e$$
(3)

where  $Q_e$  (mg/g) is the amount of Crystal violet adsorbed by the ash at equilibrium,  $C_e$  (mg/L) is the equilibrium concentration of crystal violet solution,  $b_T$  and  $K_T$  are Temkin isotherm constant and Temkin isotherm equilibrium binding constant, respectively. *R* is gas constant (8.314 J/mol K) and *T* is temperature (K). Fig. 9 shows linear plot for Temkin adsorption isotherm for present work and related parameters are listed in Table 1.

For the present study, the results showed that the Freundlich isotherm is best fitted with  $R^2 = 0.999$  (Table 1), indicating that the Freundlich adsorption isotherm is applicable for the entire adsorption process. The linear plot for Freundlich isotherm provides  $K_f$  (=14.253) and 1/n (=0.609). The value of 1/n is found to be less than 1 indicating that the plant ash is useful for removal of entire range of concentration of crystal violet dye [30].

#### 3.4. Thermodynamic studies

Thermodynamic parameters viz. free energy (*G*), enthalpy (*H*) and entropy (*S*) for the adsorption of dye on *C. album* ash can be calculated using the following equations [32].

$$\ln\left(\frac{Q_e m}{C_e}\right) = \frac{\Delta S}{R} - \frac{\Delta H}{RT}$$
(4)

$$\Delta G = \Delta H - T \Delta S \tag{5}$$

where  $\Delta S$ ,  $\Delta H$  and  $\Delta G$  are the changes in entropy (J/mol/K), enthalpy (kJ/mol) and Gibb's free energy (kJ/mol), respectively. *m* is the adsorbent dose (g/L).  $C_e$  is the equilibrium concentration (mg/L) of dye solution, while  $Q_e$  is the amount of dye adsorbed at equilibrium (mg/g).

The plot of  $\ln(Q_em/C_e)$  vs. 1/T (Fig. 10) provides the values of  $\Delta H$  and  $\Delta S$  from its slope ( $\Delta H/R$ ) and intercept ( $\Delta S/R$ ). Values of  $\Delta G$  were calculated by Eq. (5). The values of thermodynamic parameters are reported in Table 2. The feasibility of the adsorption process and its spontaneous nature are confirmed by the negative values of free energy [33]. Further, on increasing temperature, the increase of absolute values of  $\Delta G$  suggests that the adsorption is favorable at higher temperatures [34]. Positive values of  $\Delta H$  and  $\Delta S$  describe that the adsorption process is endothermic and

random at the adsorbent–solution interface. Since the value of  $\Delta H$  is less than the 40 kJ/mol, the adsorption proceeds via physical adsorption [35].

# 3.5. Kinetic study

In order to explain the adsorption process, three kinetics models Lagergren's pseudo-first-order model, Ho's pseudo-second-order model, and Weber and Morris' intraparticle diffusion model were applied to interpret experimental data



Fig. 9. Linear plot for Temkin adsorption isotherm.



Fig. 10. Linear plot for thermodynamic parameters.



Fig. 11. Linear plot for pseudo-first-order model.

in the present study. The pseudo-first-order and pseudosecond-order kinetics equations can be expressed as,

$$\ln(Q_e - Q_t) = \ln Q_e - k_1 t \tag{6}$$

$$\frac{t}{Q_t} = \frac{t}{Q_e} + \frac{1}{k_2 Q_e^2}$$
(7)

where  $Q_e$  (mg/g) and  $Q_t$  (mg/g) refer to the amount of dye adsorbed at equilibrium and time *t*.  $k_1$  (min<sup>-1</sup>) and  $k_2$  (g/mg min) are the first order and second order rate constant, respectively.

Intraparticle diffusion kinetic model is related to the diffusion in the adsorption process. Its mathematical expression is given by,

$$Q_t = k_t t^{1/2} + C (8)$$

where  $k_i$  is the intraparticle diffusion rate constant and *C* is related to the boundary effect [30,32]. Three linear plots for the kinetic models are shown in Figs. 11–13 and their corresponding parameters are reported in Table 3. The correlation coefficients ( $R^2$ ) for pseudo-first-order kinetic model are close to unity. Therefore, it is applicable to control the overall steps involved in the adsorption process. Furthermore, calculated  $Q_e$  value computed from pseudo-first-order model equation showed good agreement with the experimental value.

Table 2 Thermodynamic parameters for adsorption of crystal violet on *C. album* ash

Dye	ΔΗ	$\Delta S$	$\Delta G$ (kJ/mol) at temperatures				
concentration	(kJ/mol)	(kJ/mol/K)	303 K	308 K	313 K	318 K	323 K
3	19.844	0.094	-8.650	-9.120	-9.591	-10.061	-10.531

# 4. Column operation for removal of CV dye

Batch adsorption study gives details of some important and useful data, but more practical results for real application can be obtained by continuous mode or column operation study [36]. The operation was performed using column (cross sectional area - 0.8 cm<sup>2</sup>, height - 1 cm, mass - 0.5 g) of manure weed ash at a flow rate of 1.3 mL/min for influent solution of CV dye. A 6.5 mL aliquot of effluent was collected and analyzed spectrophotometrically for



Fig. 12. Linear plot for pseudo-second-order model.



Fig. 13. Linear plot for intraparticle diffusion model.

dye content. This process was continued till the concentration of the dye in aliquot collected reached about 80% of the influent concentration, as the column gets chocked at this point. Breakthrough curve is plotted to define the efficiency of column operation. Breakthrough and exhaustion were defined at relative effluent concentration  $C_{\mu}/C_{0}$  of 0.015 and 0.79, respectively. The BC, EC and DOC utilization were calculated utilizing the areas (drawn by software OriginPro 8) in Fig. 14 and the obtained results are presented in Table 4. It is observed that breakthrough capacity is lesser than the batch capacity. This may be due to lesser time of contact of the dye solution with the manure weed ash [22].



Fig. 14. Breakthrough curve of Crystal violet on C. album ash.

Table 3	
Kinetic parameters for adsorption of crystal violet on C. album	ı ash

Kinetic models	Parameters	Crystal violet dye concentration	
		3 ppm	
Pseudo-first-order model	$R^2$	0.970	
	$k_1$	0.005	
	$Q_e^{ m cal}$	3.838	
	$Q_e^{ m exp}$	4.785	
Pseudo-second-order	$R^2$	0.944	
	<i>k</i> <sub>2</sub>	0.021	
	$Q_e^{\rm cal}$	5.319	
	$Q_e^{ m exp}$	4.785	
Intraparticle diffusion	$R^2$	0.954	
	$k_i$	0.199	
	С	0.545	

### Table 4 Breakthrough parameters of CV dye on C. album ash

Batch capacity (mg/g)	Breakthrough capacity (mg/g)	Exhaustion capacity (mg/g)	Degree of column utilization (%)
9.42	2.09	4.81	43.48

Table 5 Comparison with other adsorbents for the removal of CV dye

S. No.	Adsorbent	$Q_e(mg/g)$	Ref.
1	Chenopodium album ash	9.42	This work
2	Acacia nilotica leaves	33	[19]
3	Unexpanded perlite	3.305	[37]
4	Expanded perlite	1.142	[37]
5	Bagasse fly ash	26.233	[38]
6	Jute fiber carbon	27.743	[39]
7	MCM-22	48.957	[40]
8	H <sub>3</sub> PO <sub>4</sub> activated carbon of coconut male flowers	60.42	[20]
9	$H_2SO_4$ activated carbon of coconut male flowers	85.84	[20]
10	Uncalcined ball clay	48.957	[41]
11	Calcined ball clay	40.798	[41]

# 5. Comparison of adsorption of CV by manure weed ash with other adsorbents

Pore volume and surface area of *C. album* ash are found to be 0.018 cm<sup>3</sup>/g and 9.14 m<sup>2</sup>/g, respectively. It should be explored for its potential role for the adsorptive removal of pollutants. These values are comparable with those of other ash samples explored for adsorption studies. The adsorption capacity of the present work is compared with the other reported adsorbents as given in Table 5. It is clear that adsorption capacity of manure weed ash adsorbent is comparable with other adsorbents.

# 6. Conclusion

In this study, Chenopodium album plant ash was successfully used for the removal of crystal violet dye from its aqueous solution. Two techniques, that is, batch and column operations were used to explain the removal process. Column capacity is found to be lesser than the batch adsorption capacity. pH 7 was found to be optimum for the removal of CV by the plant ash. Freundlich adsorption isotherm revealed good applicability to describe the equilibrium of adsorption process. Thermodynamic parameters viz. change in enthalpy  $\Delta H$ , change in entropy  $\Delta S$  and change in free energy  $\Delta G$  were calculated. Negative free energy favors the spontaneity of adsorption process while positive enthalpy and entropy values indicate endothermic and random nature of adsorption. Since the value of  $\Delta H$  is less than the 40 kJ/mol, the adsorption proceeds via physical adsorption. The process of adsorption was best described by the pseudo-first-order and intraparticle diffusion kinetics models. The ash of C. album, which is a cheap and easily available weed throughout the country, can be used for the removal of crystal violet. Its potential as a good adsorbent can be explored for other toxic dyes as well as other hazardous pollutants.

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