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REMOVAL OF CRYSTAL VIOLET FROM AQUEOUS SOLUTION USING DESERT DATE (*Balanites aegyptiaca*) SEED SHELL

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ABSTRACT

The feasibility of utilizing desert date seed shell (DDSS) as an alternative low-cost adsorbent for the removal of hazardous crystal violet (CV) dye from aqueous solution was investigated. The prepared adsorbent was characterized by Fourier transform infrared (FTIR) spectroscopy (Cary 630; Agilent Technologies) and scanning electron microscopy (PRO: X: Phenom World 800-07334). The influence of operational parameters such as contact time, adsorbent dosage, pH, and temperature were studied using batch mode technique. The obtained results showed that 60 min contact time, 0.4g adsorbent dose, solution pH of 8 and 60°C temperature resulted in maximum CV removal. The equilibrium data fitted very well to Freundlich model ($R^2 = 0.9983$) and the K_f and $1/n$ values were found to be $31.21 \text{ dm}^3 \text{ g}^{-1}$ and 0.828 respectively. The maximum adsorption capacity of DDSS for CV as determined by Langmuir isotherm is 294.12 mg g^{-1} at 303K. Kinetic analysis revealed that the pseudo second order was the best model ($R^2 = 0.9999$) for the description of CV uptake with the experimental value of q_e $20.3575 \text{ mg g}^{-1}$ being closer to the calculated value of the q_e $20.3651 \text{ mg g}^{-1}$. Thermodynamic studies revealed a negative value for ΔG° ($-11.07 \text{ kJ mol}^{-1}$) and a positive value for ΔH° ($33.75 \text{ kJ mol}^{-1}$) and ΔS° ($134.9 \text{ J mol}^{-1} \text{ K}^{-1}$). These indicated the spontaneous and endothermic nature of the adsorption process.

Keywords: Adsorption, Desert date seed shell, Crystal violet, Batch mode, Modeling

INTRODUCTION

Water is undoubtedly one of the most precious natural resource that exists on our planet. It is the lifeline of all living things on earth. Although, this fact is widely recognized, pollution of water is a common occurrence (Jodeh *et al.*, 2015). According to United Nations World Water Development Report (2012), 2,000,000 tons of wastes are discharged to receptor water bodies every day, including industrial wastes, dyes, pesticides, fertilizers and chemicals (Gupta and Ali, 2013). Most of these substances are resistant to biodegradation, persistent, difficult to remove and cause health problems when they exceed the tolerance limit in water (Seed and Sephr, 2011).

In the last few years, many different conventional technologies have been applied to the removal dyes from wastewater. These techniques include chemical precipitation, membrane filtration, ion-exchange, electrolysis, coagulation, solvent extraction, reverse osmosis, irradiation and adsorption (Sulyman *et al.*, 2017). Among all these mentioned techniques involving reclamation of industrial and municipal wastewater, adsorption is generally found to be a very promising one due to its high effectiveness and complete removal of pollutants even at low concentrations, and its low cost and

easy adaptation and sorbent material separation from the aqueous phase after finishing treatment (Babel and Kurniawan, 2003).

Many low-cost adsorbents have been used for the removal of CV such as *Lylisoma latisiqulum* seed powder (Karthik *et al.*, 2019), orange peel (Al-Azabi *et al.*, 2018), sawdust (Mashkooor *et al.*, 2018), water hyacinth (Kulkarni *et al.*, 2017), potato peel (Larini *et al.*, 2017), tea dust (Khan *et al.*, 2015), pineapple leaf (Neupane *et al.*, 2014), rice straw (Chowdhury *et al.*, 2013), groundnut shell and orange peel (Muhammad *et al.*, 2012), rice husk (Chakraborty *et al.*, 2011) and coniferous pinus bark powder (Ahmad, 2009). However, there is still need for adsorbents which are cheap, easily available and efficient.

Desert date is one of the abundant agro-based resources available in northern Nigeria. Traditionally, most of the seed shell waste is disposed because it has no practical utility. Utilization of this waste as adsorbent for the treatment of dye bearing wastewater will convert it into a useful material. Although very few studies have reported the use of desert date seed shell as adsorbent (Giwa *et al.*, 2018; Salim *et al.*, 2017), none (from the available literature) has reported its use for the removal of CV.

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Keeping in view the importance of treatment of CV contaminated water from industrial and other establishments, the present study seeks to explore the feasibility of CV removal from aqueous solution using desert date seed shell as adsorbent

MATERIALS AND METHODS

Chemicals and Glassware

All the chemicals used were of analytical grade and supplied by E. Merck (Mumbai, India). Distilled water was used throughout for the preparation of stock and experimental solutions. The glass wares and other plastics used were washed with detergent and thoroughly rinsed with water and finally dried before use.

Instrumentation

Fourier transformed infrared spectrophotometer (Cary 630; Agilent Technologies), Scanning electron microscope (PRO: X: Phenom World 800-07334), Perkin Elmer UV-Visible spectrophotometer (Labda 35), pH meter (Starter 2100), Analytical weighing balance (FA 2004), Muffle furnace (SXL-1008), Incubator shaker (Innova 4000; New Brunswick Scientific).

Sample Collection and Identification

Desert date seed shells were obtained from a local market in Gashua town, Yobe state. They were taken to the herbarium unit of the Department of Plant Biology, Bayero University

Kano for identification and taxonomic classification. The sample was identified as desert date (*Balanites aegyptiaca*) with herbarium number BUKHAN 359.

Adsorbent Preparation

The seed shells were first washed thoroughly with water to get rid of surface adhered impurities. They were subsequently air dried for 48 h and then in an oven at 105°C overnight. The dried shells were ground and screened to pass through a 1 mm sieve to remove larger particles. Finally, they were stored in an air-tight container and used as adsorbent without any further pretreatment. The characterization of the adsorbent was done using Fourier transform infrared (FTIR) spectroscopy and scanning electron microscopy (SEM).

Adsorbate Preparation

Crystal violet (CV) dye (chemical formula = $C_{25}H_{30}N_3Cl$, Molecular weight = 407 g.mol⁻¹, purity > 90%, solubility in water 16 g.dm⁻³ at 25°C) was used as model adsorbate. Stock solution was prepared by dissolving accurately weighed quantity of dye in 100 cm³ and made up to 1000 cm³ in a volumetric flask with distilled water. The working solutions of different concentrations were prepared by diluting the stock solution to give the appropriate concentrations. The initial solution pH was adjusted with 0.1 M HCl and 0.1 M NaOH.

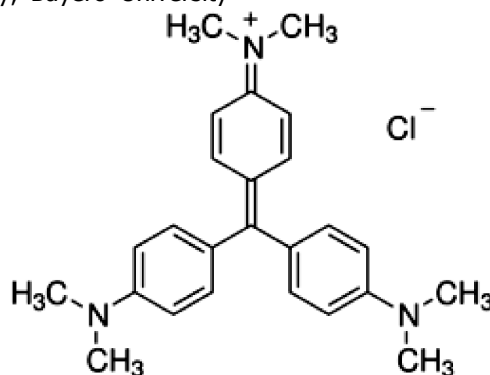


Figure 1: Chemical structure of crystal violet

Batch Adsorption Studies

One factor at a time optimization experiments were carried out to investigate the effect of the process parameters such as pH (2.0-10.0), contact time (5-120 min), adsorbent dosage (0.1-0.6 g) and temperature (303-313K) on the adsorption process. As a general methodology, a known amount of adsorbent was added in specified volume (50 cm³) of CV solution. One of the above parameters was varied with pre-defined values while the other parameters are kept constant. In each step of the study, one

parameter was optimized and used in the rest of the steps. After adsorption in each step, the solution contents were filtered and the residual concentration of the dye was determined from absorbance measurements of the filtrate at a pre-determined λ_{max} of 591nm. Control experiments were conducted without adsorbent to check for the retention of the dye at the glassware surface and other possible losses. No significant color loss at the glassware surface was observed.

RESULTS AND DISCUSSION
Fourier Transform Infrared (FTIR)
Characterization

The FTIR spectrum reveals the complex nature of the adsorbent as evidenced by the presence of large number of peaks Figures 2 and 3. The spectroscopic analysis indicated broad band at 3309.97 cm^{-1} , representing the stretching frequency of bonded -OH groups corresponding to the aliphatic moieties in polysaccharides (cellulose and hemicellulose) and lignin (Chowdury *et al.*, 2013). The peaks at 2921.96 cm^{-1} and 2851.97 cm^{-1} could be assigned due to the stretching frequency of C-H bond of methyl and methylene groups. The absorption

frequency observed at 1741.97 cm^{-1} was assigned due to the carbonyl group (C=O) of unionized carboxylate stretching of carboxylic acid or ester while the peak at 1640.97 cm^{-1} was attributed due to the C=O stretching of carboxylic acid with intermolecular hydrogen bonding form (Khan *et al.*, 2015). The peaks at 1525.84 cm^{-1} and 1424.97 cm^{-1} are associated with aromatic ring stretching of lignin and the large peak at 1022.92 cm^{-1} to 1238.96 cm^{-1} is associated with the C-O bond bending of cellulose (Werkneh *et al.*, 2014). FTIR spectra after adsorption show low transmittance intensity and the shifted peaks at different locations are due to CV adsorption.

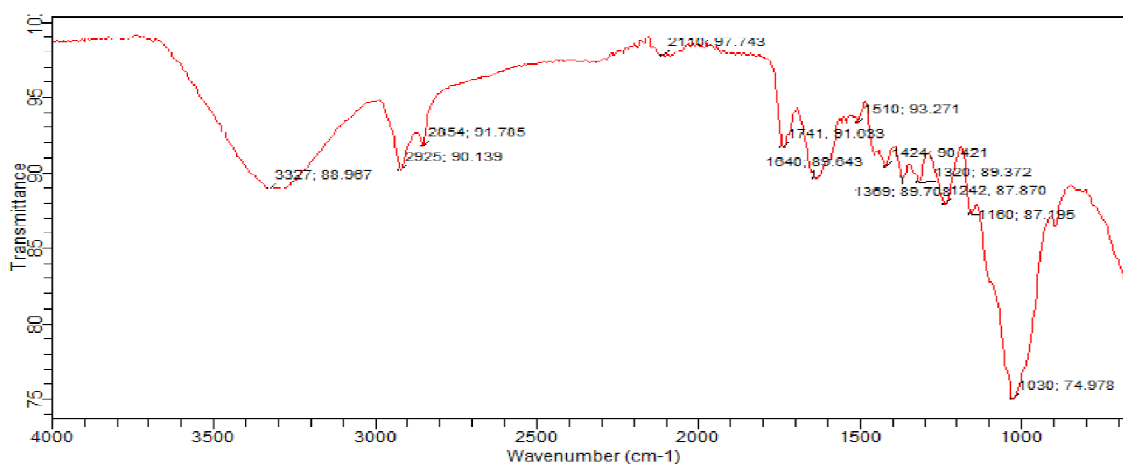


Figure 2: FTIR spectra of DDSS before adsorption

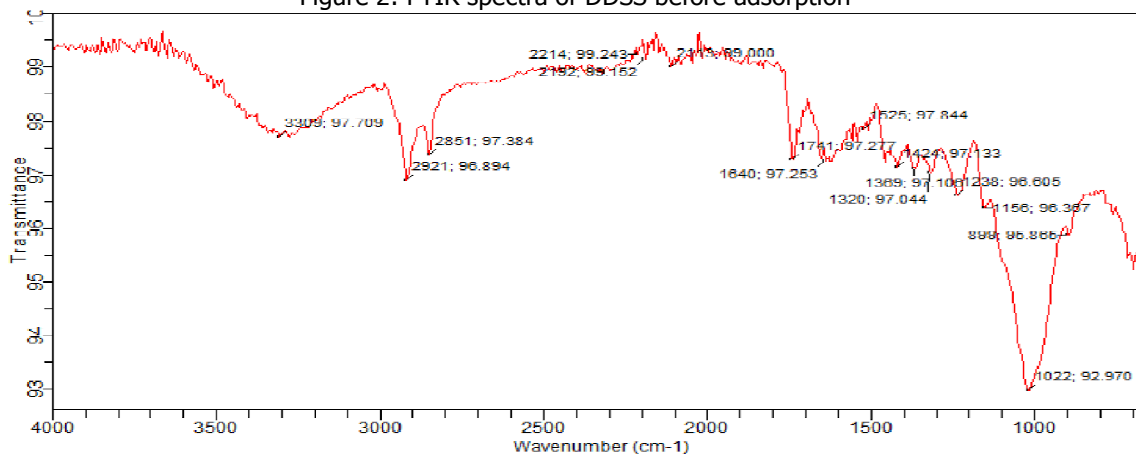


Figure 3: FTIR spectra of DDSS after adsorption

Scanning Electron Microscopy (SEM)

The morphological properties of the adsorbent were elucidated using scanning electron microscopy (SEM). The SEM micrograph of the adsorbent before and after adsorption are shown in Figures 4 and 5. It can be seen that

surface of the adsorbent is highly irregular with cracks, crevices and scattered pores. The SEM micrograph after adsorption show some distinguished dark spot with no observable pores which can be taken as a sign for effective adsorption.

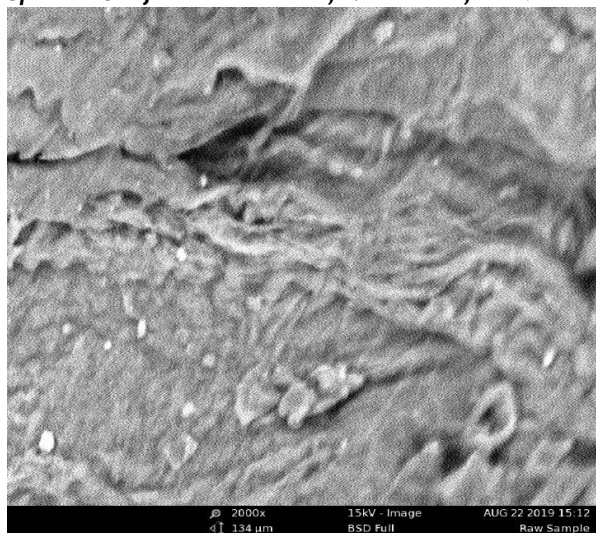


Figure 4: SEM Micrograph of DDSS Before Adsorption

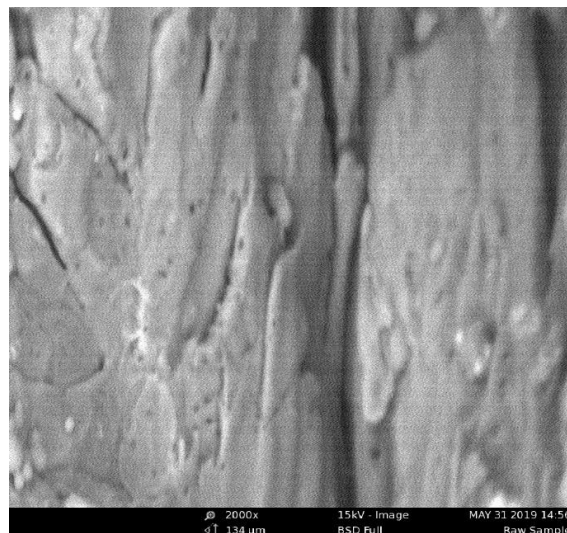


Figure 5: SEM Micrograph of DDSS After Adsorption

Effect of Contact Time

The effect of contact time on CV dye adsorption is presented in Figure 6. It is evident from the figure that the removal efficiency first rises rapidly in the early stage (0–20 minutes), gradually slows down after 30 minutes and lastly becomes almost constant after 60 min. After attaining the equilibrium, further adsorption was insignificant as a function of contact time at the employed conditions. Rapid increase in adsorption in the initial stage is attributed to the

availability of the binding active sites of the adsorbent at the start. Whereas the slow CV adsorption at the transition stage was due to the saturation of binding sites and dye ions occupy the remaining vacant sites slowly due to repulsive forces between the dye ions and ions already adsorbed on the adsorbent. Similar behavior was observed in case of CV dye adsorption on clay and pineapple leaf (Colins *et al*/2019; Neupane *et al*/2014)

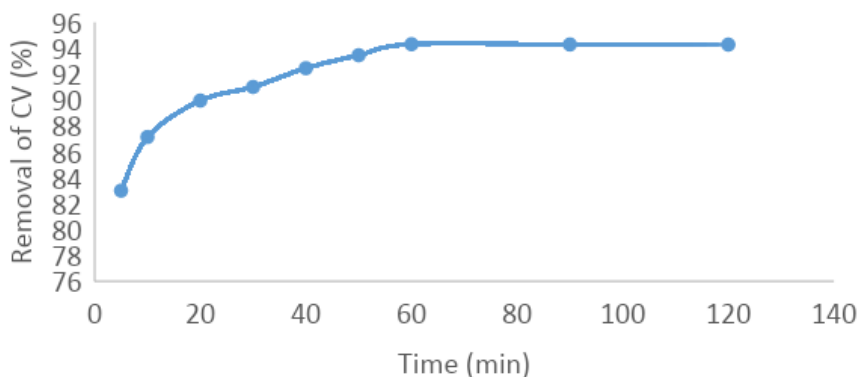


Figure 6: Effect of contact time on CV removal using DDSS

Effect of Adsorbent Dosage

The influence of adsorbent dose on CV adsorption by DDSS was investigated in the range of 0.1–0.6 g (Figure 7). The trend revealed that the adsorption efficiency increased from 91.5% to 94.7% as the adsorbent dose increased from 0.1 to 0.4 g. The increase in the percentage of dye removal with adsorbent dose could be attributed to an increase in the adsorbents’ number of adsorption sites available for adsorption. Further increase in the adsorbent

dose does not significantly change the adsorption efficiency which is mainly due to the binding of almost all dye ions to the adsorbent surface and the establishment of equilibrium between the dye on the adsorbent and in the solution. These observations are in agreement with those reported previously by other researchers for the adsorption of CV by different materials (Karthik *et al.*, 2019; Chowdhury *et al.*, 2013).

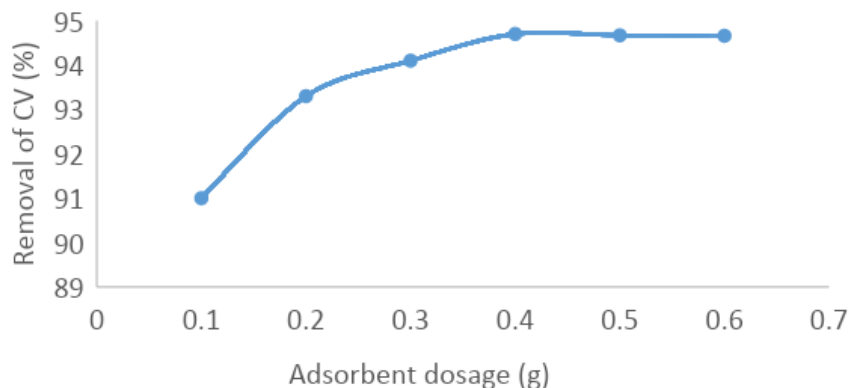


Figure 7: Effect of adsorbent dosage on CV removal using DDSS

Effect of pH

Basically, the pH of the solution affects the adsorbent surface charge through protonation/deprotonation. For the case of CV, the pH has a huge effect on the protonation of the amino groups located on the aromatic ring. It was observed that the dye solution had different colours as pH was changed from 1 to 12 which agreed with reported findings (Sulyman *et al.*, 2016). In neutral solutions, the fully conjugated molecule is brilliant violet colour with absorption maxima at 591nm. At a pH < 2 the dye is green with absorption maxima at 416nm and 617nm. Further addition of acid (pH < 0.5) creates HCV^{3+} ion which is yellow with an absorption maxima at 416nm. The different colours are a result of the different charged state of the molecule (Rammel *et al.*, 2011). It

was observed that the removal efficiency of the adsorbate increase with the increase in pH of the dye solution, appreciably up to pH 8 (Figure 8). The percent removal of CV was found to increase from 66% at pH 2 to 96.7% at pH 8. At low pH values, the surface of the adsorbent becomes positively charge by the absorption of H^+ ions leading to repulsion between CV (a cationic dye) and positively charged adsorbent. . On the contrary, in basic medium, due to the presence of OH^- ions in excess, the surface of the adsorbent becomes negatively charged. This results in electrostatic attraction between a cationic dye and negatively charged adsorbent surface leading to enhanced adsorption in basic pH. A similar trend was reported for the adsorption of cationic CV by NaOH-modified rice husk (Chakraborty *et al.*, 2011).

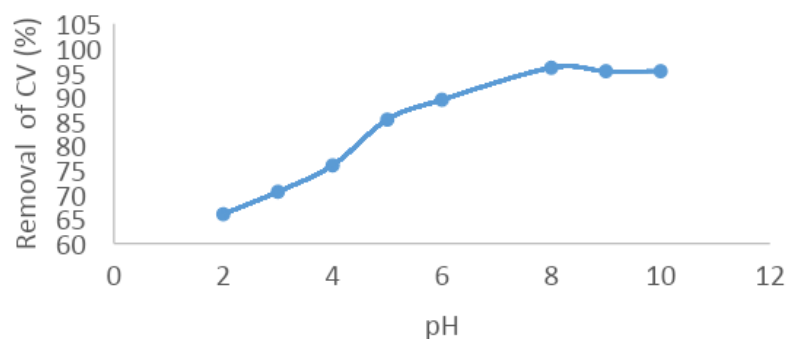


Figure 8: Effect of pH on CV adsorption onto DDSS

Effect of Temperature

The effect of temperature was investigated with different temperature ranges from 30 °C to 60 °C with the increment of 10 °C. The results obtained are shown in Figure 9. It is seen that the amount of CV adsorbed increases with temperature indicating that the adsorption of CV

is favored at high temperatures which is valid for an endothermic process. An increase in temperature generally improves the solubility of molecules and their mobility within the pores of the adsorbent. Similar phenomenon was also observed for adsorption of CV by clay (Collins *et al.*, 2019).

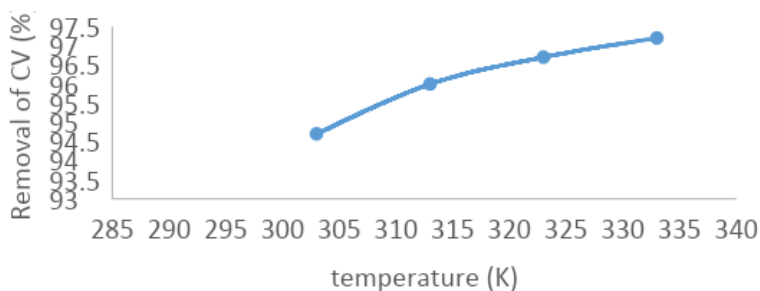


Figure 9: Effect of temperature on CV adsorption onto DDSS

ADSORPTION ISOTHERMS

The linear form of the Langmuir, Freundlich, Dubinin-Radushkevich and Temkin isotherm models are given by Eqs. (1) (2) (3) and (4) respectively:

$$\text{Langmuir equation: } 1qe = 1q_{\max}K_L C_e + 1q_{\max} \quad (1)$$

$$\text{Freundlich equation: } \ln qe = \ln K_f + 1/n \ln C_e \quad (2)$$

$$\text{D-R equation: } \ln qe = \ln q_m - \beta \epsilon^2 \quad (3)$$

$$\text{Temkin equation: } qe = B_T \ln C_e + B_T \ln K_T \quad (4)$$

The values of different isotherm constants for the adsorption of CV onto DDSS are listed in Table 1. In the light of relative values of correlation coefficients (R^2), the isotherm data can be best represented by Freundlich isotherm model. It is worth to mention that removal of CV by other adsorbents such as Lysiloma seed powder (Karthik *et al.*, 2019) and rice husk (Chakraborty *et al.*, 2011) has also been found to obey Freundlich isotherm. The values of the Freundlich constant, K_F and n are related to the adsorption capacity and adsorption intensity respectively. In general $n > 1$ illustrates that adsorbate is favorably adsorbed on an adsorbent, and the higher the n value the stronger the adsorption intensity. In particular, the value of n obtained is higher than unity

which strongly supported favourable adsorption by the adsorbent. In addition, the separation factor R_L from the Langmuir model was 0.6. This value is also designating favourable adsorption as it is within the range of 0-1. The D-R isotherm model constant gives an idea about the mean free energy of adsorption (E) and can be computed using the following equation

$$E = 1/\sqrt{2\beta} \quad (5)$$

However, the very low R^2 values (<0.90) suggest that D-R isotherm equation does not describe the dye removal process adequately. The mean free energy for the present study was found to be $< 8 \text{ kJ mol}^{-1}$ which implies that the adsorption of CV on DDSS may be considered a physical adsorption process.

Table 1: Isotherm parameters for adsorption of CV onto DDSS

Isotherm models	Parameters	Value
Langmuir	q_{\max} (mg g^{-1})	294.12
	K_L ($\text{dm}^3 \text{mg}^{-1}$)	0.07
	R_L	0.6
	R^2	0.9502
Freundlich	K_F ($\text{dm}^3 \text{g}^{-1}$)	31.21
	$1/n$	0.828
	n	1.2
	R^2	0.998
Temkin	K_T ($\text{dm}^3 \text{mg}^{-1}$)	3.24
	B_T (J mol^{-1})	35.97
	R^2	0.8895
	Dubinin- Radushkevich	q_m (mg g^{-1})
β		1×10^{-5}
E (kJ mol^{-1})		0.223
R^2		0.7338

Adsorption Kinetics

The kinetic data have been analysed in the light of different kinetic models, namely; pseudo-first order, pseudo-second order, Intra-particle diffusion and Elovich models by employing equation (6) (7) (8) and (9) respectively:

$$\text{Pseudo-first order model} \quad \ln(q_e - q_t) = \ln q_e - k_1 t \quad (6)$$

$$\text{Pseudo-second order model} \quad t q_t = 1/k_2 q_e^2 + t q_e \quad (7)$$

$$\text{Intra-particle diffusion model} \quad q_t = k_{id} t^{1/2} \quad (8)$$

$$\text{Elovich model} \quad q_t = \ln(a \times b) b + \ln(t) b \quad (9)$$

The kinetic parameters associated with these models so obtained are summarized in Table 2. On comparing the values of correlation coefficient, one can infer that the pseudo-second order kinetic model is the best one to represent the adsorption process. Furthermore, comparison of the calculated q_e values and the experimental q_e shows that the pseudo-second

order values are closer to the experimental values. This suggest that the rate limiting step may be the chemisorption involving valence forces between the adsorbent and the adsorbate. A similar phenomenon was also observed for clay (Collins *et al.*, 2019) and potato peel (Larini *et al.*, 2017).

Table 2: Kinetic parameters for the adsorption of CV onto DDSS

Kinetic model	Parameters	Values
Pseudo-first order	k_1 (min^{-1})	0.0500
	$q_{e_{cal}}$ (mg g^{-1})	0.3400
	$q_{e_{exp}}$ (mg g^{-1})	2.3575
	R^2	0.9626
Pseudo-second order	k_2 ($\text{g mg}^{-1} \text{min}^{-1}$)	0.5000
	$q_{e_{cal}}$ (mg g^{-1})	2.3651
	$q_{e_{exp}}$ (mg g^{-1})	2.3575
	R^2	0.9999
Intraparticle diffusion	k_{id} ($\text{mg min}^{-1/2} \text{g}^{-1}$)	0.0590
	C	1.9450
	R^2	0.8194
Elovich	(g mg^{-1})	7.6860
	R^2	0.9062

Adsorption Thermodynamics

The thermodynamic parameters associated with CV-DDSS system were evaluated using equations (10) (11) and (12):

$$K_c = C_o - C_e C_e \quad (10)$$

$$\Delta G = -RT \ln K_c \quad (11)$$

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

The values of enthalpy change (ΔH°) and entropy change (ΔS°) were calculated from the intercept and slope of linear plot of ΔG° vs T as tabulated in Table 3. The negative values of ΔG° indicate that the adsorption process is spontaneous. The trend of free energy with temperature further indicates that the feasibility of the adsorption increases with increase of temperature. The positive value of ΔH° signifies

that the adsorption of CV onto DDSS is endothermic. Thus, the spontaneity of the adsorption is only due to entropy factor. The positive value of entropy change indicates an increase in randomness at the adsorbate-solution interface during adsorption process. Similar trend in thermodynamics have also been observed for the adsorption of CV onto activated carbon (Jayganes *et al.*, 2017).

Table 3: Thermodynamic parameters for the adsorption of CV onto DDSS

T(K)	ΔG° (kJ mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° ($\text{kJ mol}^{-1} \text{K}^{-1}$)	R^2
303	-7.05	33.75	0.1349	0.9948
313	-8.56			
323	-9.99			
333	-11.07			

Comparison of DDSS with other adsorbents

Table 4 summarizes the comparison of the maximum CV adsorption capacities of various adsorbents including DDSS. It is obvious from the table that DDSS has higher CV adsorption

capacity than some previously reported adsorbents in literature. The easy availability and low preparation cost are some additional advantages, suggesting DDSS to be a better adsorbent for treatment of dye bearing wastewaters.

Table 4: Comparison of CV adsorption capacity of DDSS with other reported low-cost adsorbents

Adsorbent	q_{\max} (mg g ⁻¹)	Reference
Clay	69.26	Collins <i>et al.</i> (2019)
L. latisiquum seed powder	14.41	Karthik <i>et al.</i> (2019)
Orange peel	27.17	Al-Azabi <i>et al.</i> (2018)
Water hyacinth	322.58	Kulkarni <i>et al.</i> (2017)
Tea dust	175.4	Khan <i>et al.</i> (2015)
Pineapple leaf	158.73	Neupane <i>et al.</i> (2014)
Rice straw	80.91	Chowdhury <i>et al.</i> (2013)
Egg shell	70.03	Chowdhury <i>et al.</i> (2012)
C. elegans alga	158.7	Rammel <i>et al.</i> (2011)
Rice husk	44.87	Chakraborty <i>et al.</i> (2011)
CP bark powder	32.78	Ahmad, 2009
Desert date seed shell	294.12	Present study

CONCLUSION

The present study shows that DDSS can be used as a low-cost adsorbent for the removal of CV dye from aqueous solutions. The operational parameters such as pH, contact time, temperature and adsorbent dosage, were found to have effect on the removal efficiency of the adsorbent. The results obtained showed that contact time of 60 min, adsorbent dose of 0.4 g, solution pH of 8.0 and temperature of 303 K are

the optimum conditions for the process with removal efficiency of 94.3, 94.7, 96.0 and 97.2%, respectively. Isotherm studies shows that Freundlich model is the best one to represent the equilibrium data. Kinetic modeling revealed that the adsorption of CV onto DDSS follows pseudo-second-order kinetics. Thermodynamic study indicated the spontaneous and endothermic nature of the adsorption process.

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