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Adsorption of Crystal Violet Dye from Aqueous Solution onto the surface of Green Peas Shell (GPS)

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ABSTRACT

The adsorption of Crystal Violet (CV) cationic dye onto the surface of Green Peas Shell(GPS) was evaluated through batch adsorption experiment, in order to develop a low-cost, natural, eco-friendly and alternative adsorbent for the removal of organic pollutants. The effect of different parameters were studied such as pH (2-7), adsorbent mass (0.2 to 1.0 g), contact time (10 to 60 min), Initial dye concentration (25–50 mg L^{-1}), and temperature (293 K to 333K) on the adsorption of CV dye. The results Showed that CV adsorption on the Green Peas Shell depends on Contact time, pH, initial dye concentration adsorbent dose, and temperature. The adsorption equilibrium data were fitted in Langmuir and Freundlich adsorption isotherm models. The adsorption data obtained were well described by Langmuir isotherm model with correlation coefficients (R^2 =0.99). Maximum adsorption capacity of Green Peas Shell was found to be 96.974%. Thermodynamic study revealed that the adsorption of CV on Green Peas Shell is spontaneous physical adsorption process, exothermic and favorable. Experimental results indicate that the Green Peas Shell studied is a promising adsorbent for the removal of cationic dye molecules from aqueous solutions.

Keywords: GPS, CV, Adsorption, Batch experiment, Freundlich adsorption Isotherm.

INTRODUCTION

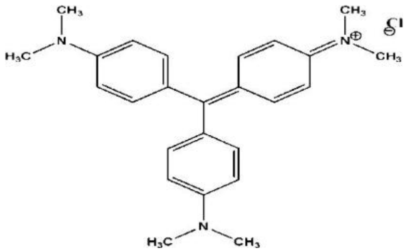
Industrial activities are an important source of pollution causing deterioration of the environment and health. The treatment becomes an important environmental issue to avoid environmental pollution and water contamination [1]. Indeed, currently, a great number of treatment technique of domestic or industrial wastewater, are used. They differ from each other and include, for example, adsorption, electrolysis, dissolution, precipitation, ion exchanges and the liquid-liquid extraction. Among these techniques, adsorption, on which rests this study, has shown great depollution capacities of wastewater. [2-10]. It is also very simple and effective for the removal of certain pollutants, particularly dyes. But its effectiveness depends greatly on the type of substrate used as adsorbent, particularly, its cost, availability and regeneration. Therefore, all these factors have stimulated a lot of research where the aim is the development or improvement of adsorption capacities of solid supports. This passes inevitably by understanding of the pollutant retention mechanism by the solid support. [1]

This study is within this perspective, using local biowaste as an adsorbent for the retention of some dyes such as Crystal Violet (CV). In deed recent years, biowaste GPS have attracted the attention of researchers because they are cheaper than activated carbon and are easily available. CV, cationic dye, is used as a model of pollutant for adsorption studies to measure adsorption capacity of biowaste GPS material.

In this work, chemical and physical characterization of material used was performed. The parameters that influence adsorption such as pollutant initial concentration, contact time, adsorbent mass, solution, pH and temperature were investigated. The adsorption equilibrium isotherm models (Langmuir, Freundlich) and thermodynamic parameters were also evaluated.

MATERIALS AND METHODS

Preparation of polluting solution: Crystal Violet (CV) [Cationic form, Chemical formula: $C_{25}H_{30}ClN_3$, Molar mass 407.979 g mol^{-1}) used in this work was purchased from S. D. Fine Chemicals Ltd. Mumbai and was used without further purification. The molecular structure of the dye is illustrated in figure 1. This dye is widely used in microbiology for gram stain, color indicator in assays reactions and fingerprint revelation. The dye stock solutions were prepared by dissolving accurately weighted dye in distilled water to the concentration of 1000 mgL^{-1}. The experimental solutions were obtained by diluting the dye stock [1, 11,12]



Preparation of adsorbent: The adsorbents selected for the present work were Green Peas Shell (Pisum sativum.) (GPS) which are locally available biowaste were collected in Aurangabad District of Maharashtra state (India).The biowaste waste GPS were identified and authenticated by Dr. Anil Bhuktar Associate Professor and Head of Botany Department Vivekanand College, Aurangabad (M.S.). The sample covering (Shell) of **Green Peas Shell (Pisum sativum.)** were dried in shadow, avoiding direct sunlight on them. The dried material of GPS were grinded into powder separately and were boiled in distilled water and filtered. The residue left of each was divided into five equal portions. One portion was considered as untreated where as Remaining four portions of each were treated with formaldehyde solution first and finally with very dilute solution of sulphuric acid, and sodium hydroxide respectively. All were then stirred for half an hour vigorously using mechanical stirrer at room temperature. Then all were filtered and washed with distilled water repeatedly to remove free acids and alkali. After chemical treatment all residues were dried first in air and finally in oven at 90-100^{0}C for 8-10 hours and powdered using electric grinder. The homogeneous powder of each was passed through mesh for desired particle size (125-250 µm). The adsorbents once prepared were used throughout the experimental work. The particle size of adsorbents selected for these experiments were on the basis of their settlement at the bottom of the system, so that the portion of the solution could be taken out conveniently from the supernatant liquid [12, 13].

Adsorption studies: Adsorption experiments were carried out by agitating 100 mg of adsorbent with 100 mL of dye solution, of the desired concentration and pH, at different temperatures (293,303,313,323 and 333) in a thermostated metrex water bath shaker with a shaking of 120 rpm. The samples were withdrawn from the shaker at predetermined time intervals and dye solutions were separated from the adsorbent using whatman filter paper [11, 12].

RESULTS AND DISCUSSION

Scanning Electronic Micrographic Studies (SEM): A scanning electron microscope (SEM) images is widely used to study the morphological features and surface characteristics of the adsorbent material. It also reveals the surface texture and porosity of adsorbent. It also plays an important role in determining the surface availability for the adsorption of dye on adsorbents [14]. Scanning electron microscope (SEM) study shows that the presence of macro-pores on the surface which were irregularly distributed pores which enhance the adsorption of CV dye molecules onto the surface of GPS adsorbent.

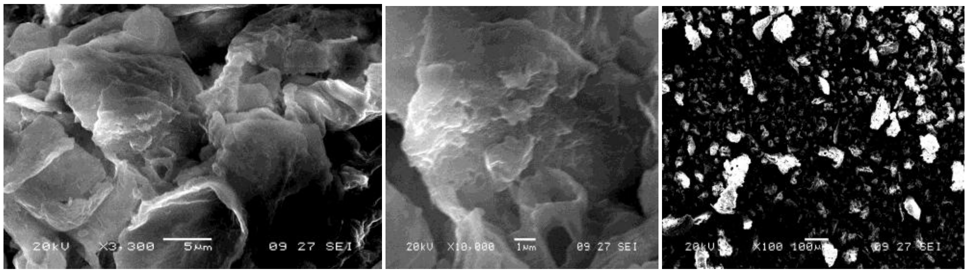


Figure 1: Scanning electron microscope (SEM) images of GPSC

Effect of Contact time: This study allowed determining the optimal time to reach equilibrium and kinetic model describing CV adsorption on the Green Peas Shell (GPS). It is carried out by introducing 1 g of adsorbent in 100 mL dye solution with concentration 20 mg L^{-1} at 293K and for a specified period. The obtained curve show that the adsorption capacity increases with contact time between adsorbate and adsorbent. The equilibrium was reached in about 60 min. adsorption initially was rapid which may be attributed to the presence of large number of active adsorption sites available on the surface of the adsorbent, compared to the number of adsorbate species in the solution. Our findings are in good agreement with Bharathi et.al [15]. The result is shown in figure. 2.

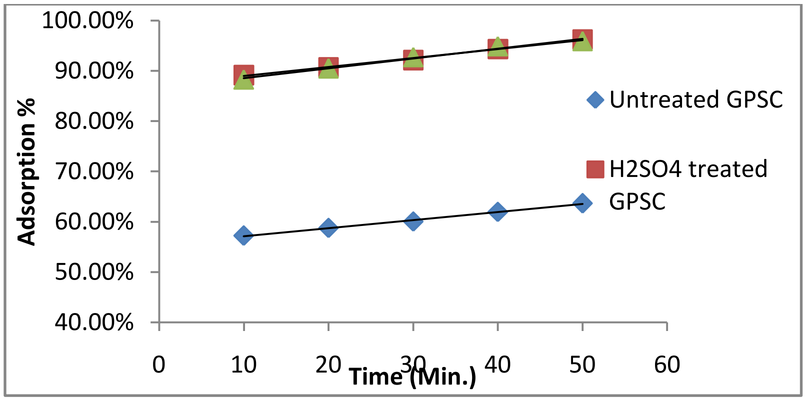


Figure 2: Effect of Contact Time

It was observed that the adsorption kinetic of CV on to the surface of Green Peas Shell (GPS) involves two distinct steps: The first step is rapid where CV dye was adsorbed by the exterior surface of Green Peas Shell (GPS) [16]. The second is slow, due to the diffusion of CV molecules into the adsorbent pores, with a higher resistance which makes slower process [17]. In the present study Minimum Removal of Crystal Violet (57.217%) on to the surface of untreated GPS where as Maximum of 96.254**%** onto the surface of H_2SO_4 treated GPS surface.

Effect of Adsorbent dose: This study is carried out by varying the mass of the adsorbent from 0.2 to 1 g. The concentration, temperature and adsorbate were maintained at 20 mg L^{-1}, 303 ±0.5 K and 100mL. The result indicates that an increase of the mass of adsorbent leads to an increase of CV adsorption performance. Our findings are in good agreement with the work reported by Ubale et.al [18]. The result is shown in figure.3.

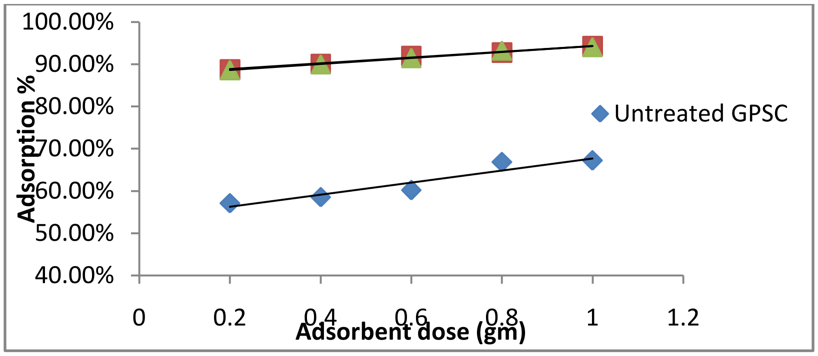


Figure 3: Effect of Adsorbent dose

It is clear that equilibrium is reached after 0.6 g. Beyond 0.8g a slight decrease adsorption performance is observed, which can be explained by desorption phenomenon or by agglomeration of the adsorbent particles leading a decrease in the specific surface of the Green Peas Shell (GPS) which is in good agreement with Tharcila et.al [1] The increase in adsorbent dose from 0.2 to 1 gm increases removal of CV which can be attributed to increased adsorbent surface area and availability of more adsorption sites resulting from the increase dose of the adsorbent.[19] The Minimum Removal of CV was 57.102 % onto the surface of Untreated GPS and Maximum of 94.282% onto the surface of H_2SO_4 treated GPS.

Effect of Initial Concentration: The effect of initial CV concentration under equilibrium conditions was investigated. The experiments were carried out at a fixed adsorbent dose (1 g in 100 mL of Crystal Violet solution) and at different initial CV concentrations (25ppm to 45ppm) and initial pH at 303 ±0.5 K. Our findings show that adsorption capacity of untreated Green Peas Shell (GPS) 57.907% and that of treated GPS found to be 95.931 %. The result is shown in figure 4.

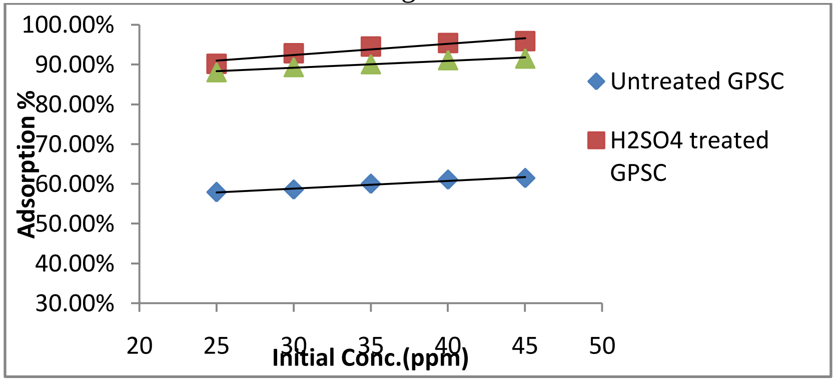


Figure 4: Effect of Initial Concentration

Adsorption phenomenon consists of diffusion of dye molecule from bulk solution to the surface, adsorption of the dye molecule on to the surface and layer formation on the surface of adsorbent. The adsorbed dye content gradually increases constantly upto certain time limit and finally attains the equilibrium indicating a saturated adsorption [20-21]. The time growth of uptake increase with concentration of dye solution. The increase adsorption of the adsorbate onto adsorbent may be due to increase in surface activity and due to micelle formation or the aggregation of dye molecule in the concentration range studied. Our results are in good agreement with the reported work by several workers. [22-23]

Effect of Temperature: The effect of temperature on adsorption has always been a very complex process and the net effects are likely to be determined by the adsorbent - adsorbate interaction [24]. The effect of temperature on adsorption shows that that the process is dominated by chemisorptive bond formation between the adsorbate and the adsorbent, but some physical adsorption cannot be entirely ruled out. The adsorption sites of the surface are expected to be heterogeneous, non-specific and non-uniform as in case of many other adsorbent [25-26]. In the present investigation effect of temperature on the CV adsorption experiment has been investigated at different temperatures (293, 303, 313, 323 and 333 K), by introducing 1 g of the Green Peas Shell (GPS) in 100 mL of the 20 ppm CV solution in a thermostatic bath. The result is shown in figure 5.

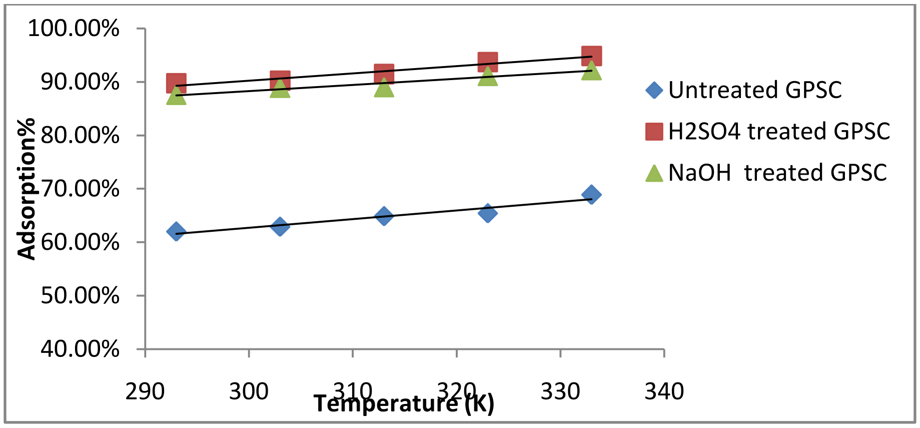


Figure 5: Effect of Temperature

Minimum Removal of CV dye was 61.987 % on to the surface of Untreated GPS adsorbent and Maximum of 94.846 % onto the surface of H_2SO_4 treated GPS adsorbent. The increase in the adsorbed mass with increase in temperature can be attributed to the reduced intermolecular forces between the water molecule which are adsorbed at the interface portion of the adsorbent which leads to the surface of the GPS adsorbent become more active, this facilitates diffusion of CV dye molecules towards the surface of GPS adsorbent easily which show the increased in adsorption with increase in temperature[27]. As the adsorption appears to be of physical type it is reasonable to consider that the involved intermolecular forces are strengthened at lower temperature which finally results in an increased adsorption. At lower temperature the possibility of agglomeration of dye molecule may not be ruled out as postulated by several workers in the case of the adsorption of several dyes [28-29]**.** Obviously in the agglomerated state the dye – molecule will be adsorbed to a greater extent.

Effect of pH: The pH is a paramount factor affecting adsorption of pollutant. The effect of pH on adsorption was carried out by adding 1 g of Green Peas Shell (GPS) to 100 mL of a series of dye solutions was prepared by adjusting the pH over a range of 3–10 (0.1 M) using HCl or NaOH solutions. The pH of the solution changes the intensity and mode of fixing the color to the Green Peas Shell (GPS) surface, therefore the pH influences not only the adsorbent properties, but also dye chemistry [30-31]**.** The result is shown in figure 6.

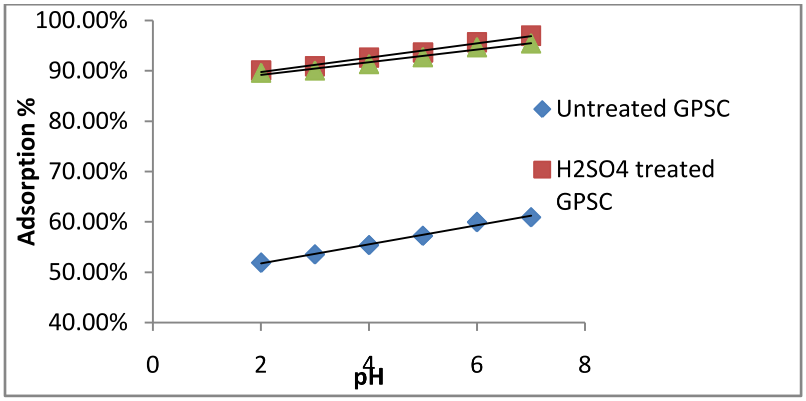


Figure 6: Effect of pH

Results show an increase in the adsorbed amount which increased from 51.871% (at pH 2) to 60.923% (at pH 7) onto to surface of untreated GPS, where as 90.076% (at pH 2) to 96.974 % (at pH 7) onto the surface of H_2SO_4 treated GPS and percentage removal of CV ranged from 88.615% (at pH 2) to 94.512% (at pH 7) onto the surface on NaOH treated GPS. It can be attributed to the addition of cations H^+ neutralizes the negative charges occurs in the Green Peas Shell (GPS) surface. Thereby the adsorption of the cationic colorant on the Green Peas Shell (GPS) is reduced due to the presence of an excess of H^+ ions in competition with CV dye cations for adsorption sites [17, 32].

Adsorption isotherms: Equilibrium isotherms and adsorption properties describe the interaction between adsorbate and adsorbent. Langmuir [33] and Freundlich [34] models were applied to describe the adsorption of CV on GPS. The adsorption isotherm was carried for different concentrations and different temperatures ranging from 20 ppm to 50 ppm and from 293 to 333K respectively. The mass of adsorbent is maintained in 1 g, for 1h. Langmuir model assumes monolayer adsorption onto adsorbent surface containing a finite number of identical adsorption sites [35]. It can be expressed by the following equation:

$$C_e /q_e = C_e /q_m + 1 /K_L q_m \qquad (1)$$

Where Q_m and K_L are the maximum adsorption capacity (mg g) and the Langmuir constant (L/mg) respectively. Dimensionless equilibrium parameter separation factor R_L [36] that characterizes adsorption is defined by the following equation:

$$R_L = \frac{1}{1 + K_L C_0} \qquad (2)$$

Adsorption is favorable if R_L values follows $0 < R_L < 1$, where as $R_L > 1$ (unfavorable), $R_L = 1$ (linear), $R_L = 0$ (irreversible). [1, 18]

In the Freundlich isotherm assumes heterogeneous surface energies and nonuniform distribution of the heat of adsorption over the adsorbent surface [21]. It is defined by (Eq.2):

$$\ln q_e = \ln K_F + \ln C_e/n \qquad (3)$$

Where K_F ((mg g^{-1}) (L mg^{-1}) $^{1/n}$) is the Freundlich isotherm constant and 1/n is the intensity of adsorption.

Table 1: Langmuir Adsorption Isotherm for Crystal violet

Adsorbate/ Adsorbent	--	**UT-GP**	**H_2SO_4-T- GP**	**NaOH-T-GP**
Crystal violet	**Slope**	0.0204	0.0097	0.0098
	Intercept	0.6201	0.1043	0.3051
	R^2	0.9338	0.9214	0.9842

Table 2: Freundlich Adsorption Isotherm for Crystal violet

Adsorbate/ Adsorbent	**--**	**UT-GPS**	**H_2SO_4-T-GPS**	**NaOH-T-GPS**
Crystal violet	**Slope**	0.6632	0.8148	0.6063
	Intercept	0.2687	0.7474	1.3351
	R^2	0.9950	0.9977	0.9927

The data obtained presented in tables 1 and 2 for the Langmuir, Freundlich isotherms indicates that the Langmuir model best describes the adsorption of CV on Green Peas Shell (GPS), consequently dye molecules can be adsorbed in monolayer coverage, without any dye-dye interactions. The equilibrium experimental adsorption capacities of CV is in excellent agreement with adsorption capacities generated Langmuir isotherm, and confirming the best fit of Langmuir model with practical data. In contrast, Freundlich models do not describe the adsorption data. The values of the separation factor (R_L) for all temperature ranges (from 293 to 333 K) were $0 < R_L < 1$ indicating the favorable adsorption isotherm. Our findings are in good agreement with earlier reported work. [1, 36]

Thermodynamic study: Thermodynamic studies were carried out to determine the nature of adsorption process. Thermodynamic parameters such as change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) can be determined by tracing $\ln K_d = f(1/T)$ or were calculated using the following equation (4-5-6) [37-38].

$$K_d = C_{ads} / C_e \qquad (4)$$

$$\Delta G^\circ = - RT \ln K_d \qquad (5)$$

$$G^\circ = \ H^\circ - T \ \ S^\circ \qquad (6)$$

Where, K_d is the equilibrium constant. C_{ads} and C_e are the equilibrium concentrations (mg L^{-1}) of the dye on the adsorbent and in the solution respectively. T is the temperature in Kelvin and R is gas constant (8.314 J mol^{-1} K^{-1}). The results obtained are tabulated in table 3.

Table 3: Thermodynamic parameters

Adsorbate	**Temp (K)**	**Adsorbent**								
		UT-GPS			**H_2SO_4-T-GPS**			**NaOH-T-GPS**		
		ΔG (KJ)	ΔH (KJ)	ΔS (J)	ΔG (KJ)	ΔH (KJ)	ΔS (J)	ΔG (KJ)	ΔH (KJ)	ΔS (J)
Crystal violet	293	-3.639	-3.897	43.996	-11.133	-3.763	57.779	-14.484	-8.987	89.280
	303	-1.539			-12.351			-06.766		
	313	-0.255			-13.230			-08.688		
	323	-0.511			-14.936			-09.831		
	333	-2.448			-11.937			-12.695		

The negative values of ΔG° corresponding to temperatures 293 to 333K indicated the adsorption of CV on GPS is spontaneous and physiosorptive process. The negative value of the enthalpy ($\Delta H^{\circ)}$ showed exothermic nature of the adsorption and the positive value of ΔS° confirmed the increase in randomness at the adsorbate-adsorbent surface

In present study ΔG value was in the range of (-0.511 to -3.639 kj) where as the value of ΔH found to be -3.897 and ΔS was +43.996 for untreated GPS adsorbent surface, whereas ΔG value the ranged between (-11.133 to -14.936 (KJ)), the value of ΔH found to be -3.763 (KJ) and ΔS was + 57.779 J for H_2SO_4-Treated-GPS adsorbent surface. Also for NaOH -Treated-GPS adsorbent surface the value of ΔG ranged between (-6.766 to -14.484 (KJ)) that of ΔH found to be -8.987 (KJ) and ΔS was + 89.280. Our findings are well supported by earlier reported work. [37,38].

APPLICATIONS

Investigated the adsorption of crystal violet dye from aqueous solutions onto biowaste activated Green Peas Shell. This method is a low-cost, natural, eco-friendly, alternative adsorbent and applicable for the removal of organic pollutants.

CONCLUSIONS

The present study investigated the adsorption of crystal violet dye onto biowaste Green peas Shell collected and treated with H_2SO_4 and NaOH. The obtained results show that the adsorption may reach 94.66% of the total adsorption capacity in 60 min, removal of CV onto the biowaste Green peas Shell is best at basic pH and at high temperatures the adsorption capacity increased with increasing the mass of Green peas Shell (GPS) the adsorption Langmuir isotherm has been demonstrated to give the best correlation for the adsorption of crystal violet onto Green peas Shell (GPS). Thermodynamic results indicated the adsorption process is spontaneous, physiosorption and exothermic.

The various results obtained from this study indicated that the adsorbent chosen for the study was efficient and could be used for the removal of CV from the aqueous solution in a batch system. In addition the studied Green peas Shell is abundantly and locally available, it can be considered as an economical adsorbent for industrial dye effluents.

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