

Parametric & Kinetic Study for the Adsorption of Crystal Violet Dye by Using Carbonized Eucalyptus

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ABSTRACT: The adsorption of Crystal Violet (CV) dye was performed in this study on carbonized Eucalyptus wood. The wood charcoal was characterized using XRD and SEM-EDX. The batch experiments were performed to optimize the parameters for the maximum adsorption of the CV dye and the parameters included were - amount of adsorbent, initial dye concentration, contact time, pH of the dye solution and temperature. The maximum adsorption of 95.80% was obtained at 10 ppm initial dye concentration, 1 g/L adsorbent, pH 9 and at 40°C. The study of adsorption kinetics was carried out within framework of two most important models: pseudo-first order and pseudo-second order. It was found that the adsorption followed pseudo-second order kinetics. The experimental isotherm data was analyzed using Langmuir and Freundlich models. Both Langmuir and Freundlich isotherm models were found fit in this study, but Freundlich isotherm model gave better results than Langmuir isotherm model. Thus, this study indicates carbonized Eucalyptus wood as effective adsorbent for the removal of crystal violet dye from aqueous solutions.

KEYWORDS: Adsorption, Carbonized Eucalyptus, Crystal Violet, Dye removal, Isotherms, Wastewater Treatment

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I. INTRODUCTION

In the past few decades, water contamination has been a serious problem as a result of rapid industrialization. Waste water or sewerage effluent from various industries like food processing, textile, cosmetics, rubber and plastics consists of different types of synthetic dyes [1]. Due to their complex structure, they are stable to light and oxidizing agents [2]. Hence, removing off these coloured dyes from waste water is really a difficult task for the environmentalists and researchers. Among the different physical, chemical and biological treatment methods of dye removal from aqueous solutions, adsorption of dye molecules onto the adsorbent offers an appropriate as well as practical operation [3,4,5,6]. Most commercial systems directly practice zeolites, activated carbons, industrial by-products, agricultural wastes, clays, biomass together with polymeric items being adsorbents [7,8,9,10]. Among all these methods, activated carbon or charcoal is an efficient adsorbent in the interest of removal of dyes because of its high adsorption capability.

The existence of dye or other colored pigments (e.g. crystal violet) within a water body, even in just 1 ppm in concentration, is extremely visible and regularly disturbs the phenomenon of photosynthesis of water plants due to the blockage of the penetration of sunlight in the water [11]. Although crystal violet (CV) is being an invaluable and customary dye in several industries such as textiles, pharmaceuticals, dyeing as well as and so forth, it's far recognized an obstinate worsen owing to its non-biodegradability. CV has virulent, mutagenic moreover grim possessions in the interest of individual [12]. Accumulation of CV in body would lead to fatal diseases comparable to hypermobility, abdominal agony, outbreak and as a consequence respiratory failure [13].

Crystal Violet(CV) is a water-soluble cationic dye inherent in the category of triarylmethane dyes; they are used on a large scale in textile industries for dyeing cotton, acrylic, nylon, wool, leather, paper, plastics and silk [14]. The activated carbon is a largely used adsorbent because of the fact that it has a high adsorption capacity of organic materials [15,16]. Various other effective and less expensive adsorbents were also demonstrated e.g. hydroxyapatite, chitin-chitosan and clay minerals [15,17,18,19]. Recently, the application of pyrophyllite on wastewater treatment has interested the scientific community. Publications have reported the pyrophyllite potential for removal of heavy metal ions and dyes [20]. Adsorption using agricultural waste biomasses have been preferred for the dye adsorption as these are recyclable, easy to handle, less expensive, available throughout the year and more efficient.

In this work, our target is to study the basic dye Crystal Violet adsorption on Charcoal from Eucalyptus. Various experimental parameters analyzed in this study include amount of adsorbent, initial concentration of dye, contact time, pH of solution and temperature. The adsorption capacities were calculated using the Langmuir and Freundlich isotherms. The adsorption kinetics of the Crystal Violet dye was studied using the pseudo-first order and the pseudo-second order models.

II. MATERIALS AND METHODS

2.1 Materials

Eucalyptus wood was obtained from the campus of Shaheed Bhagat Singh State Technical Campus, Ferozepur (India). The dye, Crystal Violet (CV), was purchased from S D Fine Chemicals, India. The raw Eucalyptus wood was first cleaned with fresh water to remove the soil, sand and any other undesirable material and then, kept in sunlight for seven days. It was cut into small pieces, dried at 50°C in an air oven for 24 h. and further reduced to small size particles through grinding. These particles were paralyzed in a furnace at different temperatures for 4 hours. The produced char was grinded, sieved and stored in an air tight container and labeled as EWC.

2.2 Experimental Set-up and analysis of dye

The experiments were conducted in 250 mL Erlenmeyer flasks with the working volume of 100 mL of aqueous solution. The initial pH of the solution was adjusted to the desired value by adding 0.1 M NaOH or HCl. The required amount of adsorbent dose was added in the flasks. The flasks were shaken for the specified time period in a temperature controlled incubation shaker at 120 rpm. The flasks were withdrawn from the shaker after the desired time of operation. The supernatant and the spent adsorbent were separated by using the centrifugation at 5000 rpm for 15 minutes and operation (R24 REMI Centrifuge, Mumbai, India). The residual dye concentration in the supernatant was analyzed by measuring the solution absorbance at $\lambda = 588$ nm using a UV-visible spectrophotometer (Model - EI-2375, Electronics India). All experiments were carried out in triplicate and the average values are presented.

The amount of equilibrium absorption Q_e (mg/g) of the dye was calculated by:

$$Q_e = \frac{C_0 - C_e}{w} V \quad (1)$$

The percentage removal of dye was calculated as:

$$\% \text{ Dye removal} = \frac{C_0 - C_e}{C_0} \times 100 \quad (2)$$

where, C_0 and C_e (mg/L) are initial concentration and equilibrium concentration respectively, V is the volume of the solution (L) and w is the mass of the adsorbent(g).

The process parameters such as solution pH, adsorbent dose, initial dye concentration, time and temperature were chosen as the independent input variables and the efficiency of dye removal as the dependent output variable.

2.3 Characterization of adsorbent

The X-ray diffraction studies were done by XRD analyzer (Pan analytical X'pert Pro diffractometer, D/max rA) at 45 kV and 40 mA ($\text{Cu K}\alpha = 1.50406 \text{ \AA}$). Morphological structures of the adsorbent was drawn using scanning electron microscope (SEM) JSM-6510 LV, JEOL and elemental composition was performed by energy-dispersive X-ray spectroscopy (EDS) INCax-act, Oxford Instruments.

III. RESULTS AND DISCUSSION

3.1 Characterization of adsorbent

The XRD pattern of carbonized eucalyptus adsorbent is illustrated in Fig. 1. The main peak is at 2θ values of 29.35, two mid-size peaks at 14.92 & 24.37 and few smaller peaks at 23.03, 30.08, 35.95, 38.15, 39.40, 43.15, 47.32 & 48.44.

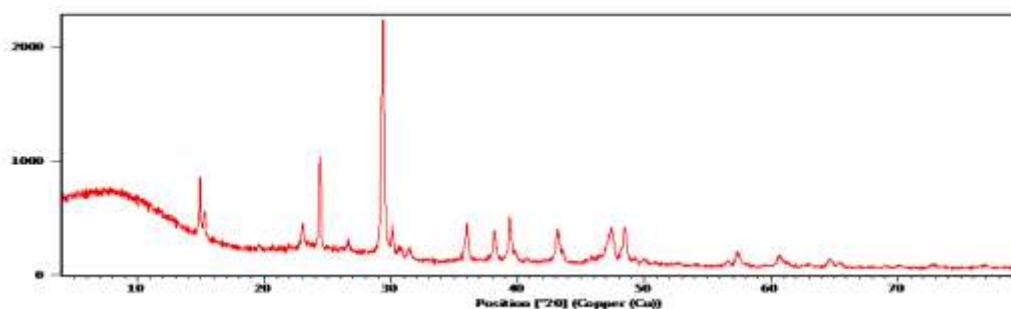


Fig.1 XRD analysis of Eucalyptus Wood Charcoal

The adsorbent samples were analyzed by scanning electron microscopy (SEM) coupled by energy dispersive X-ray spectroscopy (EDX). The application of these techniques made it possible to visualize the surface morphology of adsorbents. The image (2a) of microscopy SEM shows a porous aspect which facilitates materials adsorption.

In addition, spectrum EDX (2b) shows the presence of various elements which were analyzed using EDX (Fig 3) as Calcium, Magnesium and Potassium.

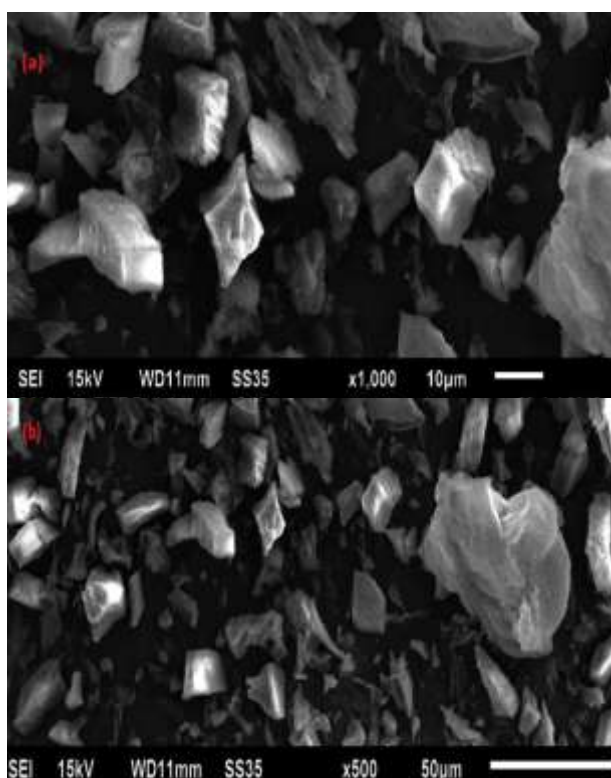


Fig 2 The SEM image of Eucalyptus Wood Charcoal carbonized at 450°C

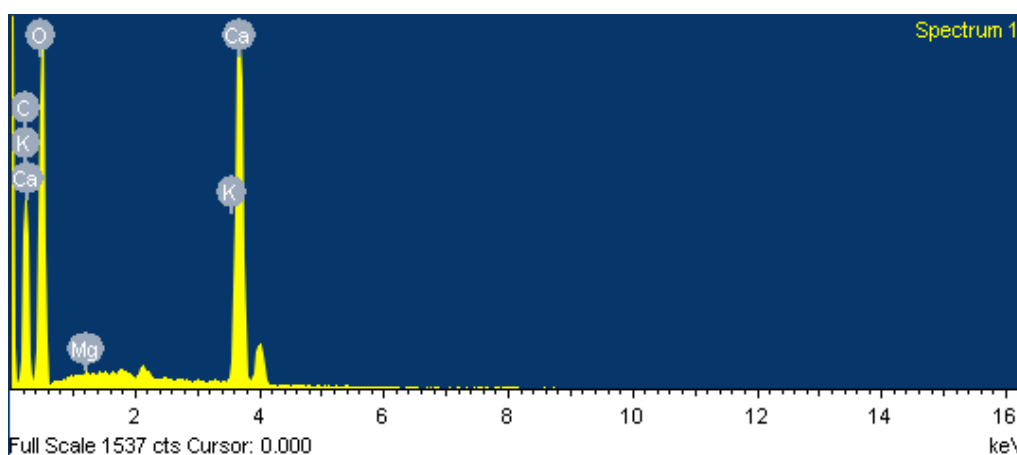


Fig 3 The EDX image of Eucalyptus Wood Charcoal carbonized at 450°C

3.2 Effect of carbonization temperature

The Eucalyptus Wood was carbonized for 4 hours at different temperatures ranging from 300°C and 500°C. The adsorption studies were performed for 1 hour using 1g/L of adsorbent and CV dye concentration of 10 ppm, the percent adsorption of CV dye was observed. As shown in Fig 4, the Eucalyptus Wood carbonized at 450°C gave the best adsorption efficiency.

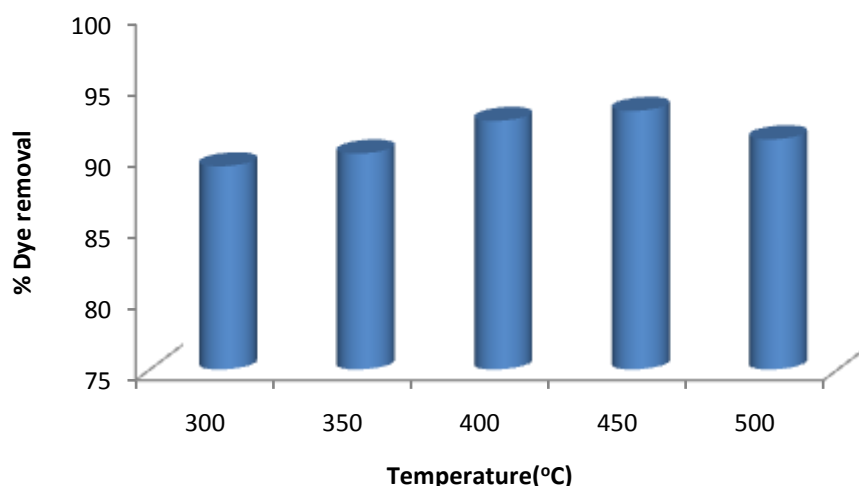


Fig 4 Effect of carbonization temperature for the Eucalyptus Wood adsorbent for the adsorption of CV dye

3.3 Effect of CV dye concentration and contact time

The CV dye concentration in the range of 10 to 50 ppm was studied for the evaluation of the effect of initial dye concentration on adsorption using EWC at 30°C and responses are shown in Fig. 5. It was observed that as the concentration of CV is increased from 10 ppm to 50 ppm, Q_e increased from 93.2 mg g⁻¹ to 413.9 mg g⁻¹ in 60 minutes when solution pH was 9 and adsorbent dose was 1g/L. The adsorbed quantity for EWC adsorbent increases by increase in initial concentration of Crystal Violet dye. This can be explained by the presence of a large number of molecules which will diffuse toward sites of the adsorbent surface and consequently partial adsorption depends on the initial concentration [21]. At higher concentrations, a high concentration gradient is established providing a thermodynamic driving force which overcomes the mass transfer resistance of CV molecules from aqueous solution on the solid phase. This gives the increase in the probability of collision of CV molecules with the binding sites of the adsorbent increases, resulting in higher adsorption capacity [22].

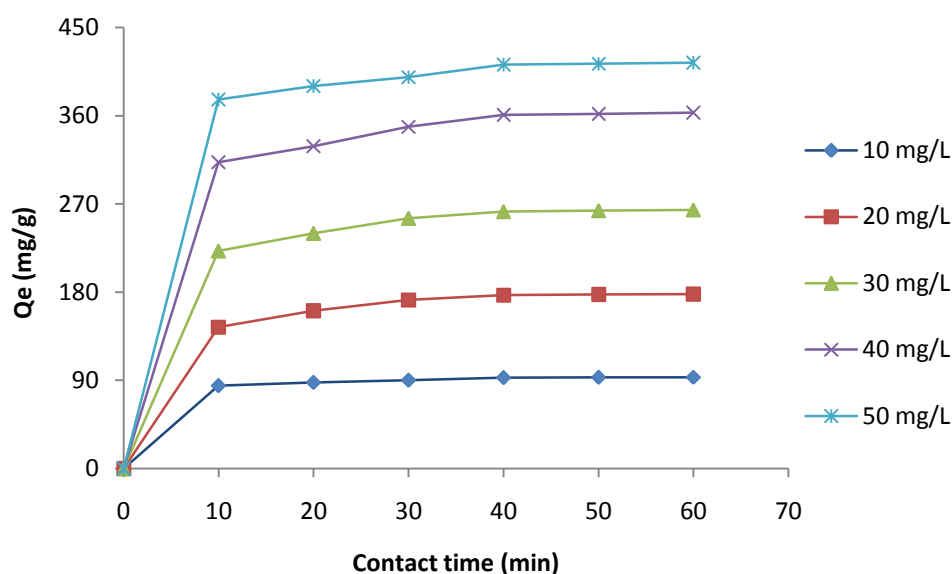


Fig 5 Effect of contact time and CV dye concentration. Interval of initial concentration (10–50 mg/L), adsorbent amount: W= 1.0 g/L, pH= 9.0, T= 30°C, and agitation time 60 min.

The knowledge of contact time is essential for calculating the maximum adsorption capacity and for identification of the adsorption type which can occur into mono or multi-layer [23]. Results presented in Fig. 5 show that the adsorbed quantity of Crystal Violet (CV) increases quickly in the first 10 min and remains almost

constant after 10 min indicating a balance state. Indeed, some sites are difficult to occupy; that is due to the repulsion between Crystal Violet molecules adsorbed on the solid surface and those of the aqueous medium this leads to a saturation of adsorbent sites and therefore almost reach an equilibrium state after 10 min. It can also be noticed that the initial concentration of the CV does not have any significant effect over equilibrium time, but it is of paramount effect on the adsorption capacity of the support.

3.4 Effect of pH

Since pH of the solution influence the surface charge of the adsorbent and also the ionization of adsorbate and hence it is a very important parameter to examine the adsorption process. The effect of pH on the adsorbate elimination of crystal violet dye by carbonized Eucalyptus was observed in the pH range of 5-11 and shown in Fig. 6. It has been observed that the dye removal is increased from 78.3 mg/g of adsorbent to 93.2 mg/g on increasing the pH of the solution from 5 to 9. Afterward, there is a slight decrease in removal efficiency on further enhancement in pH of the medium. Thus, an optimum pH 9 is selected for further adsorption studies on CV dye. Crystal Violet is a basic dye and has a pKa value of 0.8 [24]. The pH of media can change the structure of the dye, it gets protonated in the acidic medium and deprotonated at higher pH and resultantly, CV dye changed since dye adsorption trends were different at different pH values for carbonized Eucalyptus adsorbent. The low adsorption of CV adsorption under acidic and highly basic pH conditions may be correlated with lyophobic behaviour between adsorbate and adsorbents and resultantly, the forces between adsorbate and adsorbent may change [25,26].

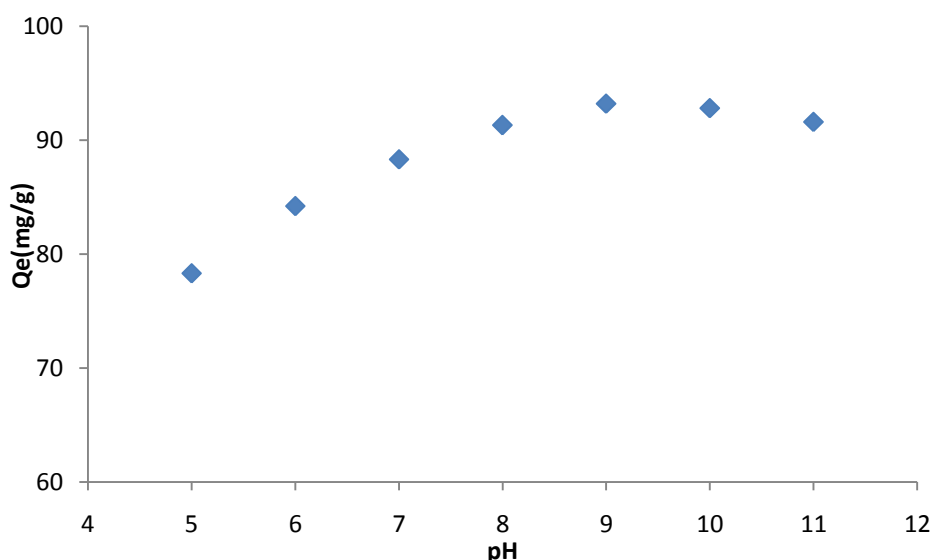


Fig 6 Effect of pH on adsorbed quantity of CV dye onto EWC. Initial Dye Conc.10 ppm, adsorbent dosage 1 g/L

3.5 Effect of adsorbent dosage

Since surface area available for dye ions has direct effect on adsorption, So for the effect of adsorbent dose in the range of 1 to 4 g/L was studied for 1 h contact time, 10 mg/L dye initial concentration, pH 9 at 30 °C. At low adsorbent dose, considerably higher CV adsorption was observed, which decreased linearly as the adsorbent dose increased (Fig.7). The influence of adsorbent dosage on the adsorbed quantity of Crystal Violet dye with time is represented in Fig. 7. The adsorption percentage increased with adsorbent's dosage rise, but the amount of adsorbed dye per adsorbent unit mass decreased with a rise in adsorbent amount from 1 to 4g/L. As the adsorbent amount increases, the number of active sites available for adsorption increases as well; thus, the percentage of removal also increases since all active sites may not be available during adsorption due to overlapping between active sites themselves and the adsorbed amount (mg/g) of adsorbent decreases. At higher adsorbent dose CV adsorption decreased, which may be due the decrease in surface area due to aggregation of adsorbent particles and resultantly, the availability of active sites was limited and dye binding efficiencies of adsorbents were decreased [26,27,28,29]. The increased adsorbent dose at constant dye concentration and volume lead to saturation of available active sites because most of the binding sites are blocked due to aggregates formation and low surface area [30,31].

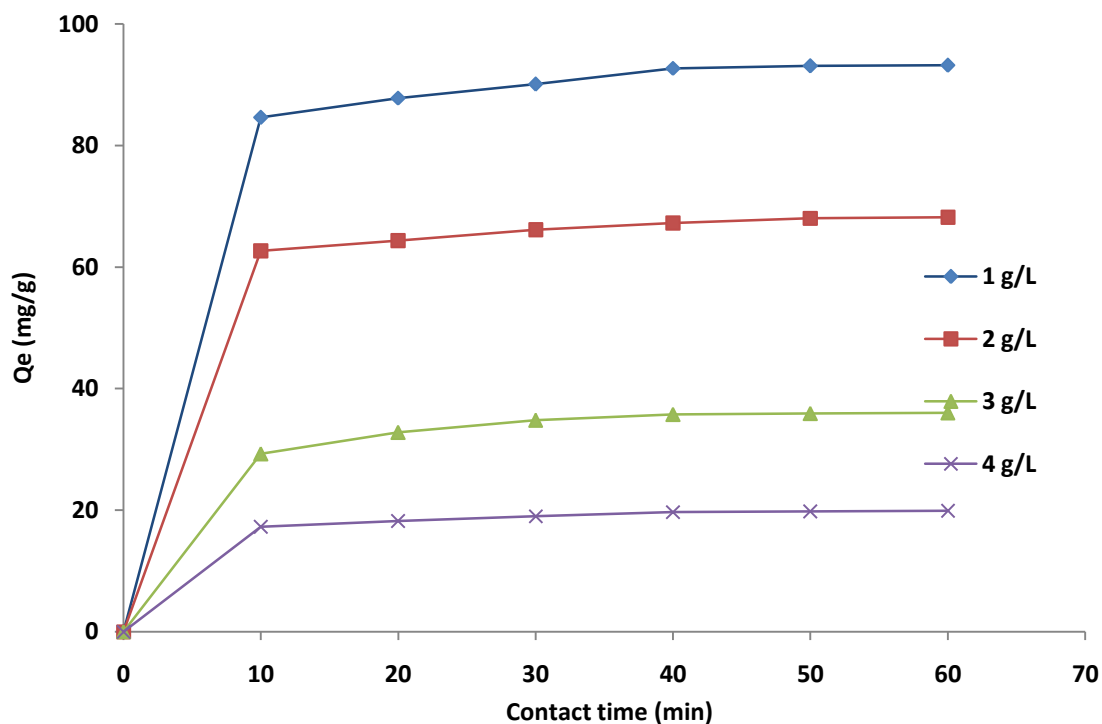


Fig.7 Effect of adsorbent amount on adsorption quantity, Initial dye concentration 10 ppm, pH =9, Temp =30 °C and agitation time 40 min.

3.6 Effect of Temperature

It is important to include the effect of temperature during the Crystal Violet dye adsorption. In this work, we have studied the effect of temperature on the Crystal Violet's dye adsorption in the range of 30–60°C (Fig. 8).

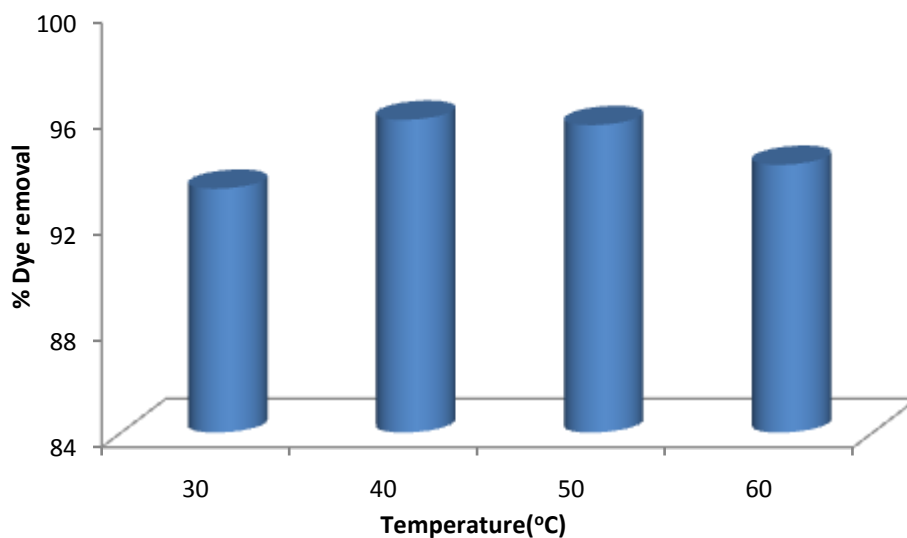


Fig. 8 Effect of Temperature on adsorption of CV dye on EWC

Generally the adsorption on any surface can be exothermic or endothermic owing to the nature of the dye (cationic or anionic dyes) in which the temperature of the medium is a noticeable point [32]. The number of binding sites for dye molecules on the adsorbent surface may be increased by the increased temperature [33]. This may be illustrated by the fact that the increased temperature adds power to the adsorbate molecule spread rate across the external limit layer and the internal pores of the adsorbent particles as a result of reduced

solution's viscosity [34]. On increasing the temperature the dye molecule mobility increases and hence it leads to the increase in absorption on the biosorption surface. As shown in Fig. 8 the absorption (%) increases up to 40°C and thereafter remains constant till 50°C. The adsorption decreased when the temperature was further increased to 60°C from 50°C

3.7 Adsorption kinetics

Various models can be used to express the controlling mechanism of adsorption process such as mass transfer and chemical reaction. The intra-particle diffusion, pseudo-first order and pseudo-second order models were most commonly used to test the fit of experimental data of dye adsorption on the kinetics equations proposed by various authors [35]. The kinetic adsorption of crystal violet dye onto EWC was studied at 30°C with an initial concentration of 10 ppm. The kinetics model of pseudo-first order is mostly used for the low concentrations of aqueous solution. This model is presented by the relation of Lagergren based on the adsorbed quantity. The pseudo-first order considers the adsorption rate of occupation sites to be proportional to the number of unoccupied sites. This model of pseudo-first order is represented by the following relation [36]:

$$\ln(Q_e - Q_t) = \ln Q_e - K_1 t \quad (3)$$

where K_1 is the constant speed of adsorption of the pseudo-first order model (min^{-1}), a straight line of $\ln(Q_e - Q_t)$ versus t indicates the application of pseudo-first order kinetics model.

Another equation, pseudo-second order equation [37] can be used for kinetics analysis and is based on the adsorption equilibrium capacity that may be expressed in the following form:

$$\frac{t}{Q_t} = \frac{1}{K_2 Q_e^2} + \frac{t}{Q_e} \quad (4)$$

where K_2 is the constant speed of adsorption model of pseudo-second order (g/mg min^{-1}); if this equation is checked, by tracing t/Q_t according to t , we must obtain a line of slope $1/Q_e$ and ordinate in the beginning equal to $\frac{1}{K_2 Q_e^2}$.

The adsorption kinetics modeling of CV on EWC adsorbent by pseudo-first order and pseudo-second order model is presented in Fig. 9 and Fig. 10 respectively. The parameters calculated by the two models are presented in Table 1, which also mentioned the correlation coefficients and the adsorbed quantity of the CV dye. From these results, it is clear that in case of pseudo-first order the actual quantity of CV dye adsorbed is different from that calculated using pseudo-first order kinetics. On the other hand, the actual adsorbed quantity is closer to the calculated value using the kinetics model of the pseudo-second order. This model is applied to systems in case of adsorbing/adsorbed studied considering the obtained values of R^2 coefficients, which are very close to the unity. Hence it is concluded that the pseudo-second order model correlates the kinetic data better ($R^2 > 0.999$) than pseudo-first order.

Table 1 Comparison between the pseudo-first order and pseudo-second order adsorption constant rate, calculated and experimental Q_e values for different initial CV concentrations.

Dye (mg/L)	Conc.	$(Q_e)_{\text{exp}}$	Pseudo-first order			Pseudo-second order		
			$(Q_e)_{\text{cal}}$	K_1	R^2	$(Q_e)_{\text{cal}}$	K_2	R^2
10		93.2	43.86	0.112	0.926	96.15	0.00656	0.999
20		178.0	140.89	0.117	0.975	187.85	0.00181	0.999
30		264.0	152.93	0.104	0.979	276.24	0.00141	0.999
40		363.0	196.17	0.101	0.952	347.22	0.00190	0.999
50		413.9	163.86	0.105	0.926	424.80	0.00151	0.999

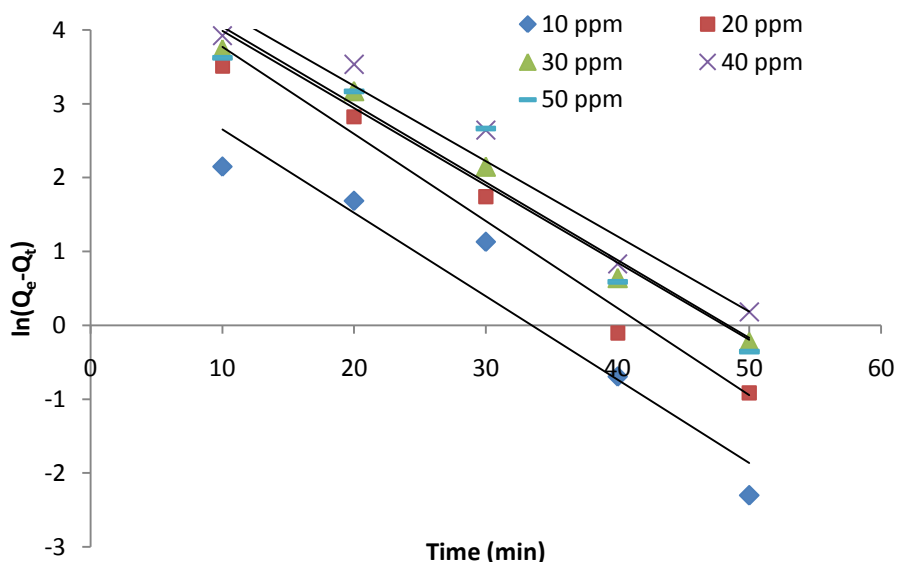


Fig. 9 Pseudo-first order model of adsorption kinetics.

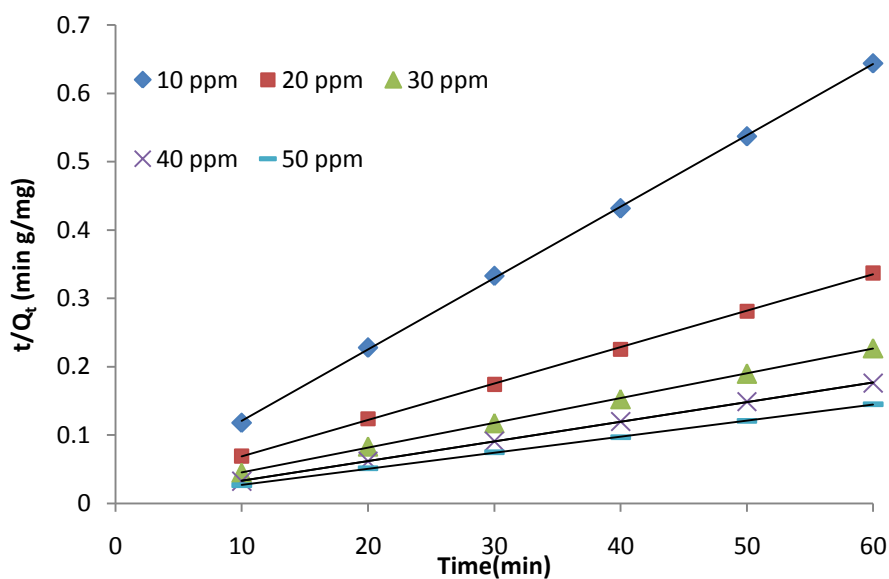


Fig. 10 Pseudo-second order model of adsorption kinetics.

3.8 Adsorption isotherms

The Langmuir model is based on chemical interactions between solute molecules and adsorbent. According to this model, the adsorption of molecules occurs in a single layer (forming a molecular monolayer). This localized adsorption on specific sites can contain only one molecule per site. The mathematical expression is:

$$Q_e = \frac{Q_L K_L C_e}{1 + K_L C_e} \quad (5)$$

where Q_L (mg/g) is the maximum adsorption capacity (monolayer coverage capacity), K_L (L/mg) is the equilibrium constant related to the affinity between adsorbent and adsorbate. The essential characteristic of Langmuir isotherm can be expressed by the dimensionless constant called equilibrium parameter, R_L , defined by:

$$R_L = \frac{1}{1 + K_L C_o} \quad (6)$$

where R_L values indicate the type of isotherm to be irreversible ($R_L = 0$), favorable ($0 < R_L < 1$), and unfavorable ($R_L > 1$) [23, 38].

The Freundlich isotherm assumes that adsorption occurs over a heterogeneous surface with a multilayer adsorption mechanism, and that adsorbed amount increases with adsorbate concentration according to the following equation:

$$Q_e = K_F C_e^{\frac{1}{n_F}} \quad (7)$$

where K_F is the Freundlich constant and n_F is a constant related to adsorption intensity.

The plots of experimental adsorption data at 60°C with Langmuir and Freundlich equations are given in Figures 11 and 12 respectively. The results obtained at different temperatures using Langmuir and Freundlich equations are tabulated in Table 2.

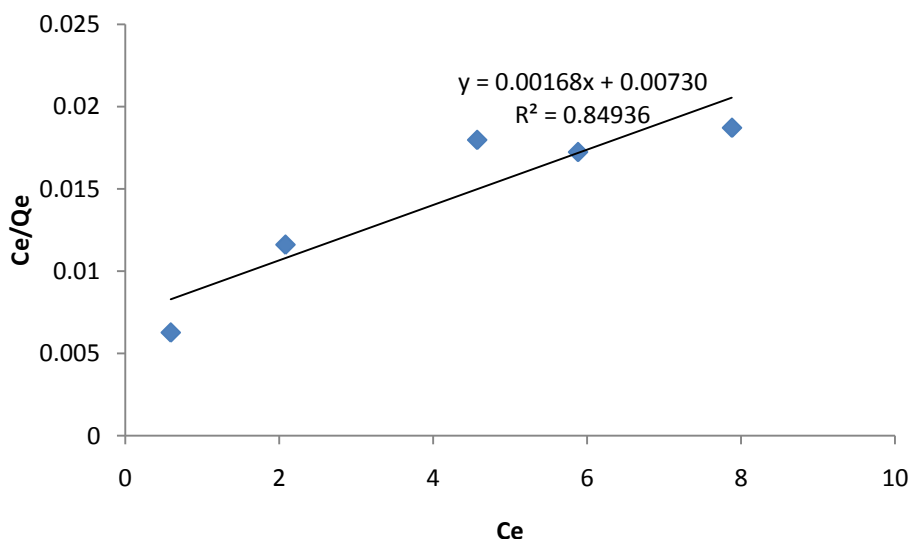


Fig. 11 Langmuir adsorption isotherm at initial crystal violet dye concentration of 10 mg/L, adsorbent dose 1g/L and temperature 60°C

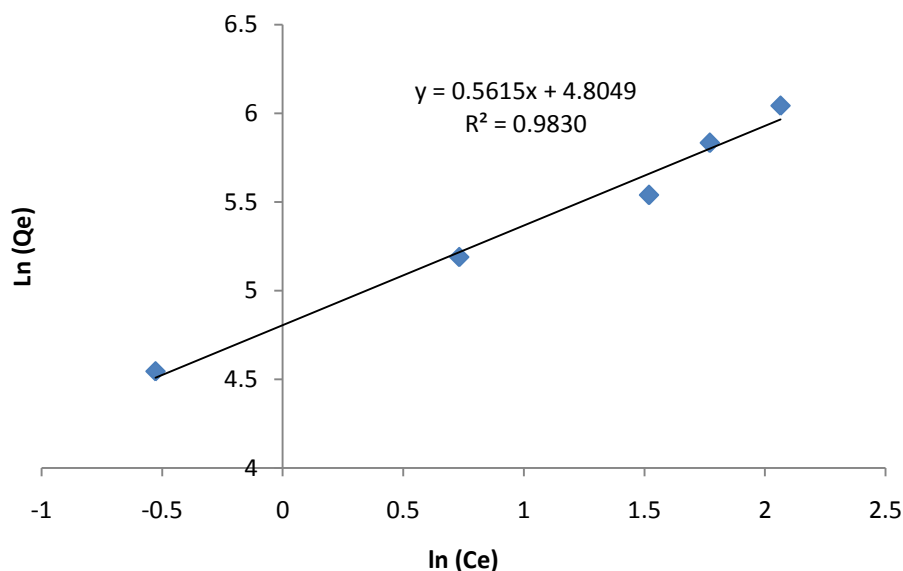


Fig. 12 Freundlich adsorption isotherm at initial crystal violet dye concentration of 10 mg/L, adsorbent dose 1g/L and temperature 60°C

Table 2 Isotherm constants for dye adsorption at different temperatures onto EWC

Temperature (°C)	Langmuir isotherm model			Freundlich isotherm model		
	Q_L	K_L	R_L^2	K_F	n_F	R_F^2
30	621.18	0.2135	0.962	116.57	1.675	0.996
40	574.71	0.321	0.932	145.32	1.911	0.993
50	581.39	0.319	0.911	144.98	1.864	0.993
60	595.23	0.230	0.849	122.12	1.781	0.983

IV. CONCLUSION

The present study shows that crystal violet dye could be adsorbed from the aqueous solutions in eco-friendly conditions using carbonized eucalyptus adsorbent. The maximum decolorization percentage of 95.80% was obtained at 10 ppm initial dye concentration, 1 g/L adsorbent and at 40°C. Kinetic studies of CV dye on EWC were carried out using pseudo-first order and pseudo-second order reaction kinetics and it was found that pseudo-second order reaction kinetics fit best for the adsorption of CV dye on EWC. Both Langmuir and Freundlich equation could represent the adsorption data well with reasonable and consistent values of parameters but the Freundlich model describes better adsorption on the EWC.

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