



ADSORPTION DYNAMICS OF ACACIA ARABICA SEED ACTIVATED CARBON IN THE REMOVAL OF CONGO RED

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ABSTRACT

Congo-Red dye is a carcinogenic dye which comes in the effluents of textile industries during dyeing and rinsing processes. The adsorption technique was found to be very useful and cost effective for a better removal of dye. Although commercial activated carbon is a preferred sorbent for color removal, its wide spread use is restricted due to high cost. The goal of this experiment is to study the adsorption of a Congo red (CR) dye solution by an adsorbent solid such as *Acacia Arabica* seed activated carbon (AASC) and then test the validity of certain adsorption dynamics. The potential of KOH activated carbon prepared from the seeds of *Acacia Arabica* (AASC), an agricultural bio waste, was assessed for the adsorption of CR carried out by varying the parameters such as agitation time, dye concentration, adsorbent dose, pH and temperature. Equilibrium adsorption data followed both Langmuir and Freundlich isotherms. The optimum contact time and pH for the uptake of CR were obtained at 30 minutes and acidic pH of 6. The maximum uptake capacity of CR dye by AASC was 160 mg/g. The results showed that the experimental data were well-fitted by the pseudo-second-order kinetic model ($R^2 > 0.99$) and Langmuir isotherm model ($R^2 > 0.99$). Prepared adsorbent was found to be effective and economically viable in the removal of congo red in waste water treatment.

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Introduction

Synthetic dyes have been increasing in textile industries for dyeing natural and synthetic fibers. Discharge of dye bearing waste-water makes an adverse effect on aquatic environment because the dyes give water undesirable color [1] and reduce light penetration and photo-synthesis [2-4]. Conventional methods used to treat colored effluents are oxidation, coagulation and flocculation, biological treatment, membrane filtration, etc. However, the single conventional treatment is unable to remove certain forms of color, particularly those arising from reactive dyes as a result of their high solubility and low biodegradability [5]. Adsorption process is an attractive and effective alternative treatment for dye removal from wastewater. There are many

advantages of adsorption process, such as less land area (half or a quarter of what is required in a biological system), lower sensitivity to diurnal variation, not getting affected by toxic chemicals, greater flexibility in the design and operation and superior removal of organic contaminants [6].

Congo red (figure 1), one of the acidic dyes containing a central biphenyl group and two symmetric naphthalenic groups [7], is widely used in textile industry, biological stain, paper, printing, leather and plastic industries [8-9]. It induces somnolence and respiratory problems because of being irritant to both the aquatic biota and human's skin and eye, the removal of CR, therefore, from kinds of effluent before mixing with unpolluted natural water bodies is of environmental-corporal

significance [10-11]. Available methodologies such as flocculation combined with coagulation [12], membrane separation [13], oxidation [14], etc., however, are either expensive or inadequate in removing dyes from wastewater. But adsorption by activated carbon is a popular-cheap way relatively, which in turn requires this kind of adsorbent possess abundant microporous and mesoporous and right functional groups to combine harmoniously with the acidic functional groups of CR dye.

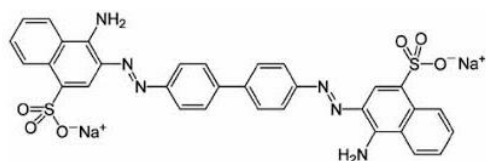


Figure 1: Structure of the reactive dye Congo red

Activated carbon is the most widely used adsorbent with great success due to its large surface area, microporous structure, and high-adsorption capacity. However, its use is limited because of its high cost. This has led to search for cheaper substitutes [15]. Investigators have studied the feasibility of using low-cost substances, such as waste apricot [16], coconut shell [17], dairy sludge [18], bamboo grass treated with concentrated sulfuric acid [19], peat [20], orange peels [21], pea nut hulls [22], rice husk [23], ground nut shells charcoal and bagasse [24], bamboo [25], jack fruit peels [26], pistachio nut shells [27], and date stone and palm tree waste [28] as adsorbents for the removal of dyes and heavy metals from waste water.

In this paper, *Acacia Arabica* (locally in Telugu called as Nalla Tumma) seed activated carbon (AASC) was developed by KOH activation and applied to remove the reactive dye Congo red from aqueous solutions. The adsorption dynamic parameters are studied to describe the rate and mechanism of adsorption to determine the factor controlling the rate of adsorption and to find out the possibility of using these biomaterials as low-cost adsorbents for the removal of dye CR. The effect of solution concentration, adsorbent dose, ionic strength, temperature, and pH on CR adsorption has been evaluated.

Materials and Methods

Adsorbent Preparation

Seeds (Dry fruits) of *Acacia Arabica* were obtained from local agricultural fields (Nellore) and were washed well with tap water to remove dust and other impurities and completely dried in natural manner. Dried in sunlight about one week and cut into small pieces, carbonized in muffle furnace (in absence of air) about 7 hours at 500°C. After carbonization, carbon was poured in double distilled water, filtered and washed in several times with cold double distilled water and dried in an air oven at 110°C for overnight. Then, the carbon was sieved into desired particle sizes. The carbonized material (AASC) in powder form (about 50 g) was taken in a one liter volumetric flask and mixed with approximately 700-800 ml of 1% KOH solution and boiled for 2 to 3 hours on flame for chemical activation. After KOH treatment they were filtered and washed several times with hot water to remove un-reacted base from carbon surface, until the effluent pH was retained i.e. 7.00.

Chemicals

The dye congo red (C.I name = Direct Red 28, C.I No. = 22120, Chemical formula = C₃₂H₂₂N₆O₆S₂Na₂, Formula weight = 696.65) is supplied by S.D fine Chemicals, Mumbai, India, was used as such without further purification. An accurately weighed quantity of dye was dissolved in double distilled water to prepare the stock solution (65 mg L⁻¹). Serial dilutions were made by diluting it with double distilled water. The molecular structure of CR is illustrated in Figure 1. Double distilled water and AR grade chemicals used in entire experiments.

Batch Mode Adsorption experiments were carried out to investigate the adsorption efficiency at varied initial concentration (10-50 mg/L), pH (3.0-8.0), adsorbent dosage(0.1-1.5g) and contact time(10-50 min.). All the experiments were performed at room temperature of 29±1°C [11]. Batch mode experiments were carried out in 100 ml conical flask with 50 ml of the test solution and shaken in Benchtop incubator cum orbital shaker(Model: Remi Shaker). At the end of required time interval, the samples were removed from the shaker and filtered using Whatman No. 42 filter paper The unadsorbed dye was recorded using

UV/vis spectrophotometer. The amount of dye taken up and the percentage of removal of the dye by the adsorbent were calculated by applying Eqn. 1 and 2, respectively:

$$q = \frac{(C_o - C_f)}{m} \cdot V \quad (\text{Eqn 1})$$

$$\% \text{Removal} = 100 \cdot \frac{(C_o - C_f)}{C_o} \quad (\text{Eqn 2})$$

where q is the amount of dye taken up by the adsorbent (mg g^{-1}), C_o is the initial dye concentration put in contact with the adsorbent (mg L^{-1}), C_f is the dye concentration (mg L^{-1}) after the batch adsorption procedure, m is adsorbent mass (g) and V is the volume of the dye solution (L).

Results and Discussion

Effect of initial pH

Since the surface charge of an AASC adsorbent could be modified by changing the pH of the solution, pH plays an important role in the removal of dyes by adsorption process. The extent of decolorization by activated carbon was studied at various pH values ranging from pH 2 to pH 8 with constant optimum conditions. The results of the effect of pH on adsorption of CR are depicted in Figure 1. The figure revealed that highest removal efficiency was observed at a pH of 4.0 irrespective of the initial CR concentration. However, at higher pH values the percentage removal of CR decreased significantly. This behaviour can be explained in the light of the types and ionic state of the functional groups on the surface of the adsorbent as well as ionic chemistry of the solution. The ionic fraction of CR molecules can be estimated from the following correlation.

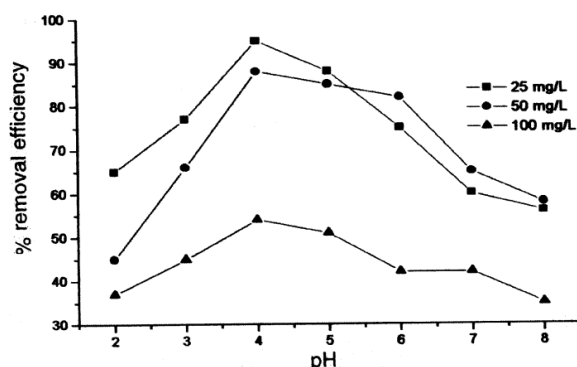


Fig. 1—Effect of pH on the removal of CR by adsorption onto the prepared activated carbon

(Batch adsorption, temperature: 30°C, adsorbent dose: 10 g/L)

Effect of adsorbent dose

The effect of the adsorbent dose on removal of phenol was studied by varying the dose of adsorbent from 5 to 40 g/L at fixed pH, temperature and initial concentration (Figure 2). It has been observed from Figure 2, that percentage removal of CR increased with the increase in the dose of adsorbent and maximum adsorption was recorded at adsorbent dose of 10 g/L beyond which no further increase was noticed. This could be due to the increased availability of active adsorption sites and surface area resulting from the conglomeration of the adsorbents especially at higher adsorbent dose [29]. The plots of percentage removal of CR versus dose of adsorbent were found to be exponential for three different initial concentration of CR (Figure 2) indicating that the amount of CR adsorbed varied in accordance with a fractional power term of the dose of adsorbent [for example $(\text{dose})^n$, where n =fraction]. This suggests that the adsorbed CR either blocked the access to the initial pores or caused particles to aggregate, thereby reducing the active site availability.

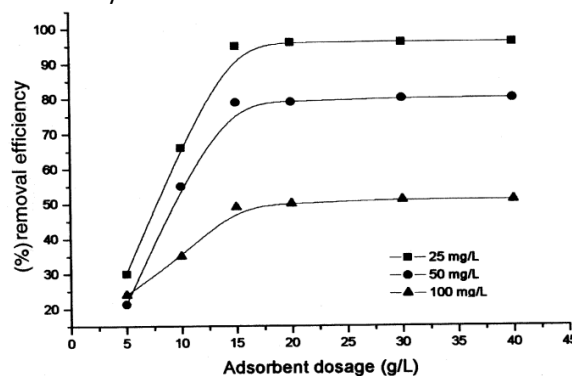


Figure 2: Effect of adsorbent dose on adsorption of congo red onto AASC

Effect of Initial Concentration

The effect of initial concentration on the removal of CR by both the adsorbents is indicated in Fig.3. Experiment was done at constant adsorbent dose 10 g/L. It is evident from the figure that percentage CR removal decreases with increase in CR concentration, however actual amount of the dye adsorbed is increased. This is due to increase in CR concentration, surface area, and active sites of the

adsorbent were saturated and hence percentage removal decreases.

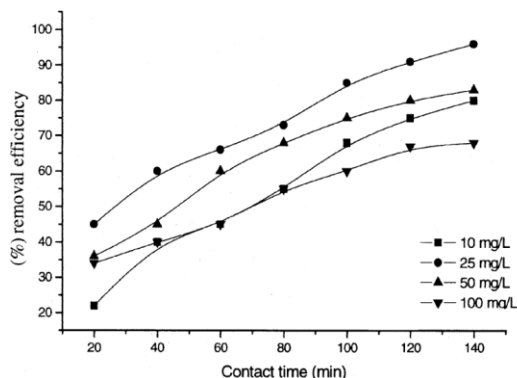


Figure 3: Effect of initial concentration of dye congo red on colour removal by AASC

The results of these experiments are depicted in Fig. 3. The percentage removal of CR was found to increase with the decrease in initial concentration of CR. Maximum removal efficiency of 96% was achieved with 25 mg/L of initial CR concentration. However, with higher initial concentration of CR the percentage removal dropped significantly. For an initial concentration of 50 and 100 mg/L removal efficiency was 83 and 68% respectively. This might be due to the lack of available active sites at high concentration resulting in increased competition for the adsorption sites and the adsorption process increasingly slows down. Figure 3 also reveals that up to 100 mg/L of initial CR concentration in the feed, the maximum percentage removal was accomplished after a contact time of 120 min and thereafter no appreciable change took place. It indicates that for lower initial concentration of CR in the feed, the adsorption was very fast. Similar trends of the equilibrium contact time of CR adsorption are reported in case of silica gel sludge (contact time: 90 min) and activated bentonites (contact time: 140 min).

Adsorption dynamics of Dye on to AASC (Isotherm study)

The adsorption isotherm data (Figure 3) revealed that the amount of dye adsorbed increased with increasing equilibrium concentration of dyes. However, the adsorption reached a maximum capacity due to nearly complete coverage of the active sites at high dye concentrations. For solid-liquid system, adsorption isotherm is important in description of adsorption behavior. In this work, two

well-known models of Freundlich and Langmuir isotherm would be evaluated.

The Langmuir isotherm

The Langmuir isotherm assumes uniform energies of adsorption onto the surface and no transmigration of the adsorbate in the plane of the surface. This model is expressed in the linear form by Equation (2).

$$\frac{C_e}{q_e} = \frac{1}{Q_{max}b} + \frac{C_e}{Q_{max}} \dots\dots\dots(\text{Eqn 3})$$

where Q_{max} ($\text{mg}\cdot\text{g}^{-1}$) is a constant related to the area occupied by a monolayer of adsorbate, reflecting the maximum adsorption capacity. K_L ($\text{L}\cdot\text{mg}^{-1}$) is a direct measure of the intensity of the sorption. Figure 4 described the plots of C_e/Q_e against C_e using linear regression analysis. The constants Q_{max} and K_L (Table 1) were determined from the intercept and slope. The essential features of langmuir isotherm can be expressed in terms of a dimensionless constant called separation factor (R_L , also called equilibrium parameter) which is given by the following equation:

$$R_L = \frac{1}{1+bC_o}$$

Where C_o (mg/L) is the initial dye concentration and R_L (L/mg), The value of R_L lies between 0 and 1 for favorable adsorption, while $R_L > 1$ represent unfavorable adsorption, and $R_L = 1$ represent linear adsorption while the adsorption process is irreversible if [30]. The values of " R_L " were found to be less than unity for the studied adsorbents and are contained in Table 1, states highly favorable adsorption for the dye congo red on studied AASC.

Freundlich Isotherm:

Freundlich isotherm can be expressed as Eqn 4

$$\text{Log } q_e = \text{Log } K_f + \frac{1}{n_f} \text{Log } C_e \dots\dots\dots(\text{Eqn 4})$$

K_f is the Freundlich constant and n_f the Freundlich exponent. Where K_f and n_f are constants representing the adsorption capacity and intensity of adsorption respectively. The slope $1/n_f$ and intercept $K_f ((\text{mg/g})/(\text{L/g})^{1/n_f})$ is obtained from the plot of $\text{Log } q_e$ versus $\text{Log } C_e$.

Table 1: Isotherm parameters for the removal of congo red by AASC

Isotherm	Isotherm constants
Freundlich	$K_f=1.399 (\text{mg/g(L/mg)}^{1/n_f})$, $n=1.684$,

	$R^2=0.9673$
Langmuir	$q_m=42.9(\text{mg/g}),$ $b=0.1249 (\text{L/mg}),$ $R^2=0.9996$ $R_L = 0.1465$

The experimental equilibrium data based on the aforementioned isotherm models for the adsorption of CR on AASC are illustrated in Figure 4. It can be seen from Figure 4 that both of the isotherm models provide good fit for the experimental data. However, the Langmuir isotherm exhibited a better fit compared to the Freundlich model. According to Table 1, the R^2 value of the Langmuir is higher (0.999) than that of the Freundlich isotherm (0.967), although both of the isotherm models have high R^2 values. Therefore, the adsorption of CR on AASC takes place on a homogenous surface as a monolayer adsorption. According to Figure 4, the R_L values are in the range of 0.14-0.45 which implies that the adsorption of CR onto AASC is a favorable process. Increasing the initial concentration, the adsorption approaches toward the irreversibility

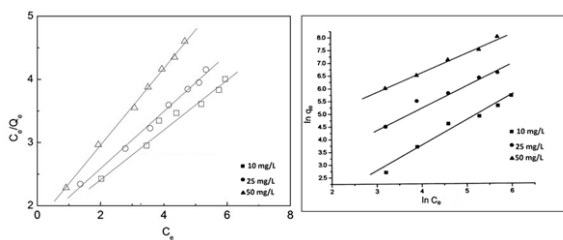


Fig. 4—Langmuir (Right) and Freundlich isotherm (Left) models for the adsorption

Effect of contact time

The duration of the batch experiments was varied from 10 to 50 min. The concentration of lead solution was 25 mg L⁻¹. Volume of the solution was 100 mL (Figure 5a) while the quantity sorbent was 0.5 g. The pH of the solution was the natural pH of 5.5. As shown in Fig. 5a, CR exhibited a rapid adsorption during the first 20 min. The percentage removal during that period was 41.96 %. This was then followed by an ongoing slow adsorption until equilibrium was achieved at 30 min. By then, the percentage of removal achieved was 86%. Therefore, 30 min was fixed as the optimum contact time to attain equilibrium.

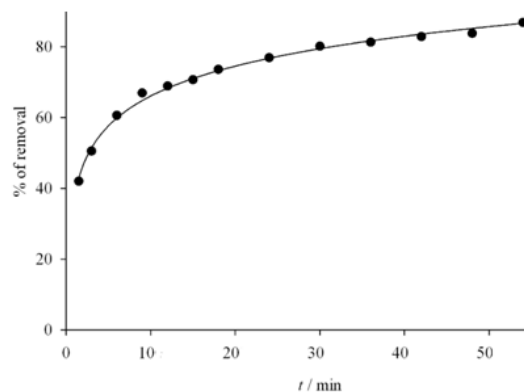


Figure 5a: Percentage removal of CR dye with respect to contact time

The adsorption of lead onto AASC occurred in two stages; an initial rapid uptake due to surface adsorption onto the AASC and a subsequent slow uptake due to diffusion of the CR onto the inner surface of the AASC.

Adsorption kinetics

The dynamics of adsorption process in terms of order of the rate constant can be evaluated using the kinetic adsorption data. The process of CR removal from aqueous phase by AASC adsorbent may be investigated by several models to examine the rate-controlling mechanism of adsorption process such as chemical reaction, diffusion control and mass transfer. The kinetic parameters are useful in predicting the adsorption rate which can be used as important information in designing and modeling of adsorption process. The kinetics of removal of CR is explicitly explained in the literature using pseudo-first order, second order, and Intraparticle diffusion model.

Pseudo-first order kinetics

Lagergren proposed a pseudo-first order kinetic model as below. The integrated form of the model is

$$\log (q_{eq} - q) = \log q_{eq} - \frac{k_1}{2.303}t \dots\dots\dots(\text{Eqn 6})$$

where q is the amount of dye adsorbed at time t (min), q_{eq} is the amount of dye adsorbed at equilibrium and k_1 is the rate constant of pseudo-first order adsorption.

Pseudo- second order kinetics

The adsorption kinetics can also be given by a pseudo-second order reaction. The integrated linearized form of this model is:

$$\frac{t}{q} = \frac{1}{k_2 q_{eq}^2} + \frac{1}{q_{eq}} t \dots\dots\dots(\text{Eqn 7})$$

where k_2 is the pseudo-second order rate constant of dye adsorption.

The plot of t/q versus t of Eq. (7) should give a linear relationship, from which q_{eq} and k_2 can be determined from the slope and intercept of the plot, if second order kinetic equation is applicable.

Intraparticle diffusion (IPD) equation

In this equation, a fractional approach to equilibrium is evaluated based on the function $(Dt/r^2)^{1/2}$, where r is the radius of an adsorbent particle and D is the effective diffusivity of the adsorbate within the particle. The initial rate is given by the linearized equation of the curve $qt = f(t^{1/2})$:

$$qt = kpt^{1/2} + C \dots\dots\dots(\text{Eqn 8})$$

where kp is the IPD rate constant, $mg\ g^{-1}\ min^{-1/2}$, while C is the boundary layer thickness, $mg\ g^{-1}$. A plot of qt versus $t^{1/2}$ will be linear if IPD is involved in the overall adsorption mechanism. If the line passes through the origin, i.e., $qt = kpt^{1/2}$, IPD is the rate controlling step of the process[31].

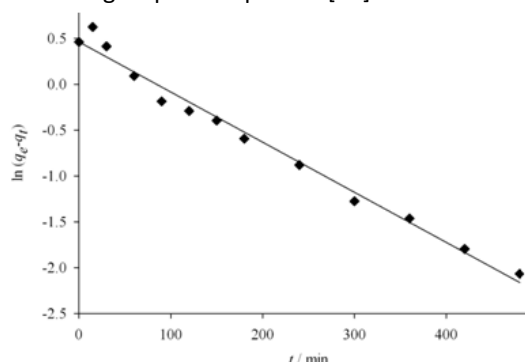


Fig. 5: Pseudo-first order kinetic plot for the adsorption

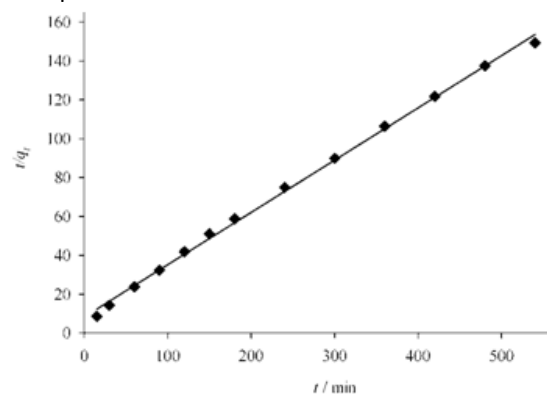


Figure 6: Pseudo-second order kinetic plot for the adsorption

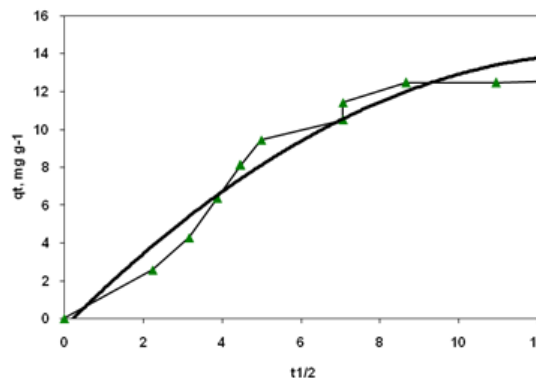


Figure 7: Intraparticle diffusion kinetic plot for the adsorption of CR onto AASC

TABLE 2. Pseudo-first order, pseudo-second order and intraparticle diffusion models for the adsorption of CR dye onto dried AASC at room temperature

Model	Parameter	Value for CR dye adsorption
Pseudo-first order	k_1 / min^{-1}	0.0048
	R^2	0.9324
Pseudo-second order	$k_2 / g\ mg^{-1}\ min^{-1}$	0.0087
	$h / mg\ g^{-1}\ min^{-1}$	0.032
	R^2	0.9979
Intra-particle diffusion equation	$K_{id} (mg\ g^{-1}\ min^{-1/2})$	0.007
	R^2	0.154

Pseudo-first order. The pseudo-first order kinetic model is commonly used for the adsorption of a solute onto an adsorbent. According to Eq. (5), a linear plot of $\ln(q_e - q_t)$ versus t would confirm the fit of the pseudo first order kinetic model. The linear plot is shown in Fig 5. Ideally, $\ln q_e$ should be equal to the intercept of the plot if the first order model were to be obeyed. In this work, the calculated q_e for the first order model deviated greatly from the experimental q_e value of $1.876\ mg\ g^{-1}$. Furthermore, this model gave the lowest correlation coefficient, R^2 using linear regression analysis as compared to the other models (Table 2). According to Ho *et al.*, [32] the first-order model does not fit well for the whole

range of contact times because the equilibrium sorption capacity, q_e , needs to be known, which is not the case as chemisorptions tends to become slow which causes the amount sorbed to remain significantly smaller than the equilibrium value. Thus, q_e , is usually obtained *via* trial and error. Therefore, the pseudo-first order model did not accurately represent of the adsorption kinetics in this study.

Pseudo-second order. The pseudo-second order model is given by Eq. (7), whereby a plot of t/qt versus t should give a linear curve. This model is more likely to predict the behavior over the whole range of adsorption and it is in agreement with chemisorption being the rate limiting step. The graphical interpretation of the data is shown in Fig. 6, while the calculated q_e value, rate constant k_2 ($\text{g mg}^{-1}\text{min}^{-1}$), the initial sorption rate, h ($\text{mg g}^{-1}\text{min}^{-1}$), the correlation coefficient, R^2 are given in Table 2. The correlation coefficient for this model was the best among all the considered models with $R^2 = 0.9979$, which shows that the second-order model fits well the data. This shows that the adsorption of CR onto AASC is second-order in nature,

Intraparticle diffusion (IPD). Adsorption is a multiple step process which is controlled by either one or more steps, *e.g.*, film or external diffusion, pore diffusion, surface diffusion or a combination of more than one step. The IPD model investigates the possibility of diffusion contributing towards the adsorption of CR. The intraparticle diffusion model is given by Eq. (8). A plot of qt versus $t^{1/2}$ should be linear if IPD is involved in the overall adsorption mechanism and the intraparticle diffusion rate constant k_p , $\text{mg g}^{-1}\text{min}^{-1/2}$ can be calculated and given in Table 2. For this work, the plot shown in Fig. 7 exhibits multi-linearity, indicating the adsorption process consisted of three steps. Table II shows the corresponding IPD rate constants, k_p , and the correlation coefficients, R^2 , the intercept, C , shown for k_{id} , indicating the boundary layer thickness, shows that rapid adsorption occurs within a very short time

Conclusion

The results of presented this investigation show that, *Acacia Arabica* seed carbon (AASC) has a suitable adsorption capacity for the removal of CR from aqueous solutions. The equilibrium adsorption is practically achieved in 30 min. The experimental

results were analyzed by using Langmuir & Freundlich isotherm models and the correlation coefficient for Langmuir isotherm is well fitted. The data indicate that the adsorption kinetics follow the pseudo-second-order rate. The present study concludes that the *Acacia Arabica* seed carbon (AASC) could be employed as low-cost adsorbents as alternatives to commercial activated carbon for the removal of color and CR dye from water and wastewater.

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