

## Crystal Violet Dye Adsorption from Aqueous Solution using Activated Chickpea Husk (*Cicer arietum*)

SONIA RANI and SUDESH CHAUDHARY\*<sup>ORCID</sup>

Centre of Excellence for Energy and Environmental Studies, Deenbandhu Chhotu Ram University of Science and Technology, Murthal-131039, India

\*Corresponding author: E-mail: [sudesh\\_choudhary@yahoo.com](mailto:sudesh_choudhary@yahoo.com)

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The chickpea husk (*Cicer arietum*) were activated by chemical modification with sulphuric acid, for its application as biosorbent for the remediation of crystal violet dye from wastewater. Activated chickpea husk (ACH) was characterized for its chemical structure and morphology using Fourier transform infrared spectroscopy (FTIR) and scanning electron microscope (SEM). The after effects of leading affecting parameters like dose of adsorbent, time of contact, pH and concentration were studied by commencing experiments in batch mode. Adsorption mechanism and sorption efficiency of ACH was examined using variety of isotherms (Langmuir & Freundlich) and kinetic models (pseudo first order and pseudo second order). Experimental data for adsorption rate was in good harmony with the results obtained using pseudo second order model. The adsorption capacity determined using Langmuir isotherm and pseudo second order model was found to be 142.85 mg/g.

**Keywords:** Activated carbon, *Cicer arietum*, Adsorption isotherms, Kinetic study, Sorption.

### INTRODUCTION

Around 2.6 billion of human population around the globe is suffering from deprivation of fresh water and is estimated to cause death of 3900 children each day while sickening a significant number among the population. In recent past, the urbanization, climate change, industrialization, population and hyped food demands has resulted in the scarcity of fresh water availability [1,2]. Apart from these, extra amount of waste water from agricultural, commercial and industrial units containing large amount of organic, inorganic pollutants, heavy metals and dyes are discharged into environment or water system without any treatment [3,4]. Among the variety of pollutants, textile dyes are most widely studied as their toxic effects can damage ecosystem including aquatic and terrestrial life [5]. Therefore, removal of heavy metals and organic or inorganic pollutants is gaining attention.

Dyes are being utilized by different types of industries including textiles, ceramics, pharmaceuticals, cosmetics, printing and paper production for numerous applications. Remediation of pigments from the effluents of these production units prior to discharge has become an important environmental concern as contamination of these in the water bodies give rise to water

pollution thus posing a severe risk to humans, plants and animals. Dyes due to their complex chemical structure are non-biodegradable in nature [6]. About 14,000 metric tons of dyes around the globe are released in to the environment every year leading to severe deterioration of the water bodies and environment. Crystal violet, an alkaline dye can lead to eye irritation and cornea damage [7] and, its ingestion can cause diarrhea, vomiting, nausea, breathing problem and abdominal pain. It also decreases the photosynthetic activities and dissolution of oxygen in to water bodies by inhibiting the penetration of sunlight into water bodies [8].

Various conventional techniques like precipitation, ion exchange, reverse osmosis, electrochemical treatment, adsorption, evaporation, chemical oxidation, membrane separation, etc. had been used for removal of dyes from wastewater by these methods. However, most of these technologies are associated with high operational and maintenance cost, incomplete metal removal, high energy requirement and generation of toxic residual metal sludge that pose disposal problem. Among these technologies, adsorption is considered as simplest and cost effective method because of easy implantation, rapid kinetics, higher efficiency and better economics [9].

A large number of adsorbents inclusive of carbon nanotubes, graphene, activated carbon, rice straw, baggase pith, rice husk, coconut shells and soursop seeds biosorbents had been explored for the remediation of dyes from waste effluent. In the midst of various adsorbents activated carbon had proved to be an effective adsorbent. However, its high generation cost, lack of reproducibility and regeneration, reduced adsorption efficiency in multi-component mixture, heating at higher temperature for longer duration and handling of spent carbon are some of the limitations [10-12]. These limitations can be overcome by using the agro wastes based activated carbon. Agriculture wastes are abundantly available, renewable and cheaper as compared to other materials being used for the development of adsorbents [13]. rice husk [14], orange peels [15], saw dust [16], baggase [17] and almonds shells [18] are some of the agriculture based activated carbon that have efficiently been utilized for separating dyes from the effluence of industries.

*Cicer arietum* also known as chickpea husk is an agro waste that has been used in the present work for separating dye from wastewater. *Cicer arietum* is pulse crop rich in proteins and being utilized for fulfilling the demand of protein by human being. The discarded husk is rich in fiber and is used as cattle feed. In spite of its wide availability and rich fiber content it still lacks scientific applications. In this study, *Cicer arietum* husk was activated and exploited for separating crystal violet dyes from wastewater. The effects of numerous processing parameters such as initial concentration, dose of adsorbent, contact time and pH was also evaluated. Further, kinetic models and isotherms were examined for its adsorption efficiency and rate of adsorption.

## EXPERIMENTAL

*Cicer arietum* (Chickpea husk) was procured from the local market. Crystal violet dye used as adsorbate and sulfuric acid used for the chemical treatment were purchased from Thermo fisher scientific, India. AR mark chemicals were employed in all the experiments.

**Activation of chickpea husk:** The chickpea husk was washed with deionized water and sun dried for one month for removal of dust and other soluble impurities. The raw chickpea husk was then grounded and sieved through sieve of size of 0.15-0.3 mm. The grinded chickpea husk was subjected to chemical treatment using concentrated  $H_2SO_4$  by maintaining the acid to fiber ratio of 1:1. Chemically treated chickpea husk was activated by heating in muffle furnace at 500 °C for 2 h. The mixture so obtained was neutralized by manifold washing through deionized water and filtered. The filtrate *i.e.* activated chickpea husk (ACH) was dried overnight at 55 °C [19].

**Characterization:** The chemical structure of activated chickpea husk was studied using FTIR analysis conducted on Perkin-Elmer RZX spectrometer. For this, KBr technology was used and spectrum was recorded from 450-400  $cm^{-1}$ . Morphology was studied using scanning electron microscopy (EVO LS 10) before and after adsorption. The pictures were captured at 20 KV potential. The physical properties like moisture and ash content was determined using ASTM standards. The elemental

composition (C, H, N, O) was done using Thermo-Finnegan elemental analyzer. Samples were vacuum dried at 100 °C prior to analysis.

**Sorption studies:** Sorption experiment was performed in batch condition taking 1000 ppm of stock solution stacked through fusion of crystal violet dye into double distilled water. The concentration of the solution was varied by attenuating stock solution. Reacting solution was shaken for 210 min on a mechanical shaker. Thereafter, the solution was passed through syringe filter of 0.42  $\mu m$ . The various operating factors such as dose of adsorbent, time of contact and pH were also examined. Sodium hydroxide (1 N) and hydrochloric acid (1 N) were utilized for maintaining the pH conditions (4-12) of the solution. The time of contact ranged from 5-60 min. Crystal violet dye concentrations in aqueous medium was examined by UV-Vis-spectrophotometer (LABINDIA UV 3092) at 570 nm. Percentage separation uptake was calculated utilizing eqn. 1:

$$\text{Separation uptake (\%)} = \frac{C_o - C_e}{C_o} \times 100 \quad (1)$$

where,  $C_o$  = concentration of Crystal violet dye in sampling solution prior to sorption;  $C_e$  = concentration of crystal violet in sampling solution after separation.

**Kinetic study of crystal violet dye sorption on activated chickpea husk (ACH):** Pseudo first order and pseudo second order models were employed for examining kinetic mechanism of crystal violet sorption on activated chickpeas husk.

Eqn. 2 is used to represent pseudo first order kinetic model [20]:

$$\frac{dq_t}{dt} = k_1(q_e - q_t) \quad (2)$$

where  $q_e$  = amount of solute sorbed by unit mass of sorbent at equilibrium;  $q_t$  = Amount of solute sorbed by unit mass of sorbent at any time t;  $k_1$  = rate constant for first order sorption  $q_e$  and  $q_t$  are the amount of solute adsorbed per mass of the adsorbent (g/g) at equilibrium and at any time t, respectively, and  $k_1$  is the rate constant of first-order adsorption ( $min^{-1}$ ). At boundary condition *i.e.* at  $t = 0$ ,  $q = 0$  and at  $t = t$ ,  $q = q_t$ ; eqn. 2 reduces to eqn. 3:

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (3)$$

which further can be rewritten as:

$$\log(q_e - q_t) = \log(q_e) - \frac{k_1}{2.303} t \quad (4)$$

Eqn. 5 is used to represent pseudo-second order kinetic model [21]:

$$\frac{dq_t}{dt} = k_2(q_e - q_t)^2 \quad (5)$$

when boundary conditions are applied,  $t = 0$ ,  $q = 0$  and  $t = t$ ,  $q = q_t$ , upon integration, eqn. 6 becomes:

$$\frac{t}{q_t} = \frac{1}{k_2 \times q_e^2} + \frac{1}{q_e} t \quad (6)$$

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

**Adsorption isotherms:** For present works, two isotherm models namely Freundlich and Langmuir were exploited to test the sorption mechanism.

Langmuir constants were predicted using eqn. 7 [22]:

$$\frac{1}{q_e} = \frac{1}{q_m} + \frac{1}{K_1 C_e q_m} \quad (7)$$

where  $q_m$  (mg/mg) = monolayer capacity at higher concentration;  $K_1$  (L/mg) = equilibrium constant;  $C_e$  (mg/L) = equilibrium concentration;  $q_e$  (mg/mg) = quantity of adsorbent sorbed at equilibrium.

Eqn. 8 was used to determine Freundlich parameters [22]:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (8)$$

where,  $K_F$  = Freundlich capacity parameter;  $1/n$  = Freundlich intensity;  $C_e$  (mg/L) = solution concentration at equilibrium;  $q_e$  (mg/mg) = quantity of adsorbent sorbed at equilibrium.

## RESULTS AND DISCUSSION

**Activated chickpea husk characterization:** Functional groups taking part in the adsorption were identified using Fourier transform infrared spectroscopy. It is also used to represent bonding between adsorbate and adsorbent during adsorption process. Various peaks of different intensity were seen before and after adsorption of dye onto adsorbent (Fig. 1). Peaks at 3400 and 2900  $\text{cm}^{-1}$  correspond to -OH group and asymmetric -CH stretching vibration, respectively. The band at 1652  $\text{cm}^{-1}$  may be attributed to carboxylic group stretching vibrations while peak at 1560  $\text{cm}^{-1}$  is ascribed to in plane deformation of the secondary amide group. The sharp peak at 1072  $\text{cm}^{-1}$  relates to C-O-C ring stretching vibration [19,23]. An apparent change was observed in the spectrum before and after sorption because intensity of peak at 3400 and 2900  $\text{cm}^{-1}$  for dye loaded ACH indicating the interaction between dye and hydroxyl group of activated chickpea husk. An enhancement in the peak intensity for peak at 1652  $\text{cm}^{-1}$  was also seen. Increase of displacement

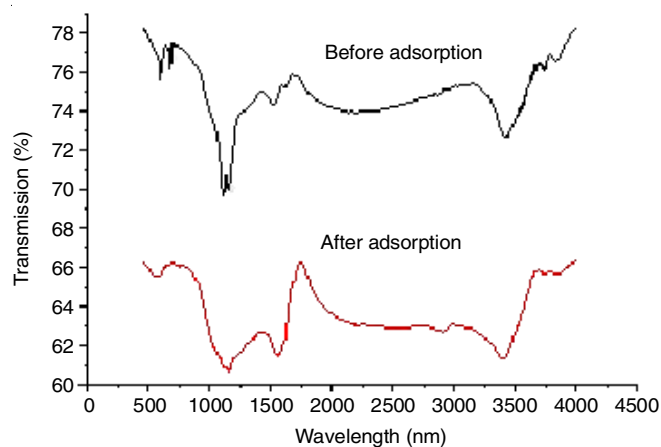


Fig. 1. FTIR spectra of ACH before and after adsorption

of peak in the spectrum confirms the functionality change on the activated chickpea husk indicating interaction of dye molecules with functional groups of adsorbent during adsorption [24,25].

**SEM analysis:** Scanning electron microscope was used to study the morphology of activated chickpea husk. Pre and post sorption SEM images of activated chickpea husk are shown in Fig. 2a-b. Fig. 2a reveals that surface of activated adsorbent is rough and contains a large number of pores and cavities with tiny lumps. In general, activation with acids results in increase in phosphorous and carbon content that leads to generation of microporous structure due to the gas release and breakage of C-O-C and C-C bonds [26]. The generation of micro porous structure led to the better adsorption. Adsorption was evidenced by smoothness and deposition of the adsorbate on the pores that can be clearly seen from Fig. 2b.

The moisture content and ash content of adsorbent were evaluated using ASTM standards was found to be 7.44% and 11.23%, respectively. The elemental composition (C, H, N, O) was evaluated using elemental analyzer and are tabulated in Table-1. From the results, it was observed that activated

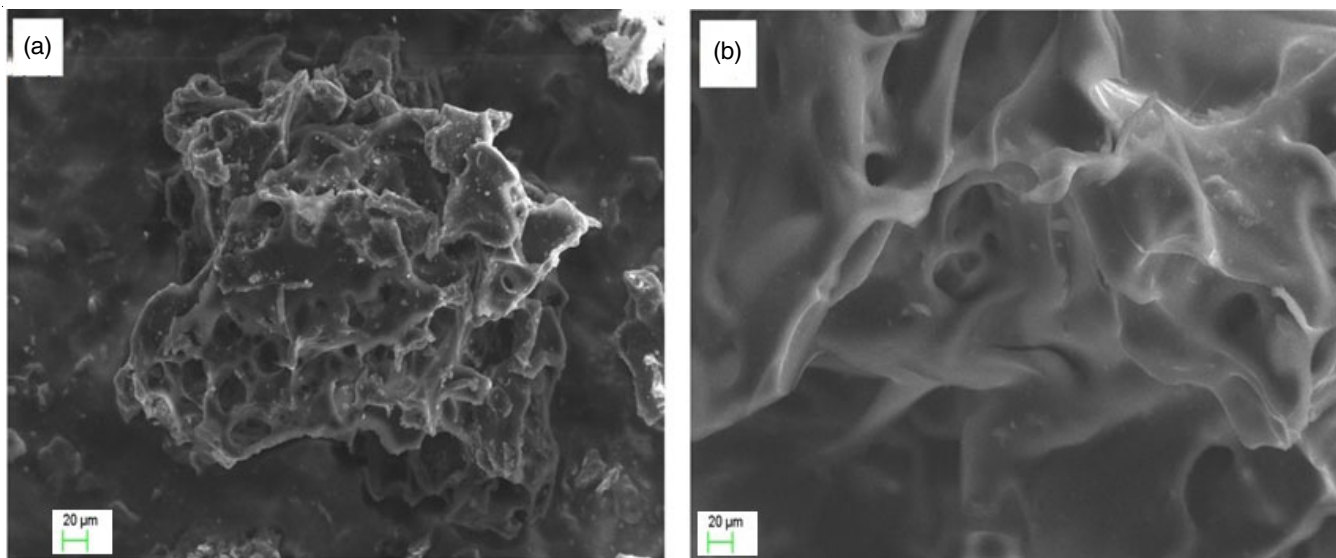


Fig. 2. SEM images of adsorbent *Cicer arietinum* (a) before adsorption (b) after adsorption

TABLE-1  
ELEMENTAL ANALYSIS (%) OF ADSORBENT ACH

C	48.11	N	6.65
H	4.35	O	40.89

activated chickpea husk has the potential amount of carbon, which may leads to the higher adsorption.

**Effect of initial concentration of crystal violet dye:** The effect of initial ion concentration of dye was examined through variation in the initial concentration from 20-100 ppm and is shown in Fig. 3a. The experiment was performed at a fixed shaking speed of 120 rpm, maintaining the temperature of 303 K with dye concentration of 20 mg/L and ACH mass of 0.1 g. From Fig. 3a, it was observed that as the initial concentration of dye was increased the percent removal of crystal violet by ACH decreased. This may be due to the fact that on increasing the dye concentration aggregation or oversaturation of adsorbent with crystal violet pigments may occur resulting in decrease in removal percentage [27].

**Effect of contact time on removal of crystal violet dye:**

To study the adsorption capacity of ACH as a function of time an optimal time is determined to know the maximum removal efficiency. The effect of time at 5, 10, 15, 30, 45, 60 min was studied to know the maximum removal efficiency and maximum adsorption was observed at equilibrium time of 45 min when the concentration of dye was 20 mg/L and at 303 K as shown in Fig. 3b. The percentage removal of crystal violet dye increased with increase in contact time till equilibrium after that no further increase was observed, which may be due to saturation of activated chickpea husk. Also, the sorption of dye by activated chickpea husk was very fast and sorption equilibrium was attained in less than 20 min revealing that the sorption depends on the expeditious transference of dyes onto the surface of activated chickpea husk at the start of adsorption. Higher specific surface area in addition to the presence of higher hollow pores on the exterior of the adsorbent leads to the higher sorption efficiency.

**Effect of pH on dye removal:** The pH directs about the efficiency rate of solid phase extraction by changing the surface

charge density of adsorbent. The effect of pH (4, 6, 8, 10, 12) on the adsorption of crystal violet at optimized contact time of 45 min and dye concentration of 20 mg/L and at 303 K is shown in Fig. 4a. As the pH increases from 4 to 12, it resulted in improvement of percentage removal and was found to 97.52% at pH 12. Low adsorption at acidic pH may be due to percentage of high concentration of H<sup>+</sup> ions, which compete with the cationic sites. Thus on increasing the pH, the surface of activated chickpea husk may get negative charge which results in attraction of positive charged cationic dye by electrostatic force of attraction [28].

**Effect of activated chickpea husk (ACH) dosage:** The adsorbent doses affect the adsorption process as higher the dose of adsorbent more adsorption sites will be open for adsorption. The dye concentration of 20 mg/L was used with different dosage of ACH (0.1-0.5g) for 45 min at 303 K. Adsorbent dose showed the same behavior upon increasing dosage from 0.1-0.5 g (Fig. 4b). This may be attributed to an increase in percent removal due to the availability of higher idle spots on leaning on the exterior of adsorbent [29].

**Kinetics studies:** Sorption kinetics modeling is utilized for identification of sorption process mechanism even if the process is physical or chemical sorption. Two adsorption kinetics models *i.e.* pseudo first order and pseudo second order kinetic models were exploited for examining sorption mechanism of crystal violet dye on the surface of ACH. The graphs obtained by applying both the models are depicted in Fig. 5a-b. It can be seen that pseudo first order failed to explain order of reaction because of negative value of slope and low correlation coefficient value.

The parameters for both the kinetics models have been represented in Table-2. The determined value of R<sup>2</sup> showed that data obtained through performing experiment matched

TABLE-2  
KINETIC PARAMETERS CALCULATED FROM THE MODELS

Pseudo first order		Pseudo second order			
K	q <sub>e</sub> (mg/g)	R <sup>2</sup>	q <sub>e</sub> (mg/g)	K <sub>2</sub>	R <sup>2</sup>
0.068	9.81	0.9570	22.22	0.0063	0.9973

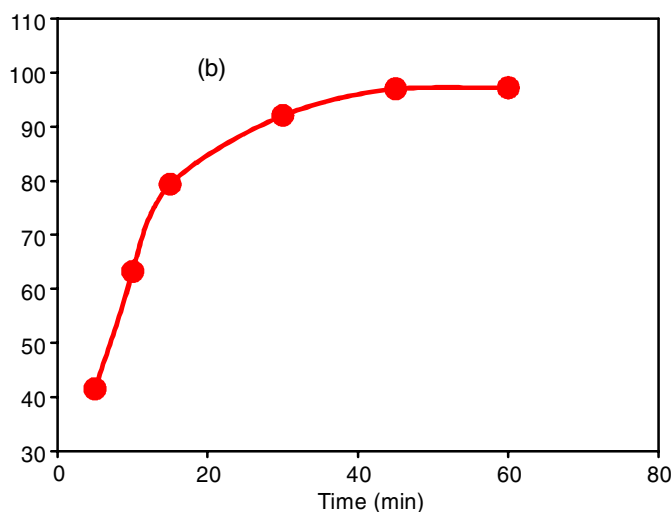
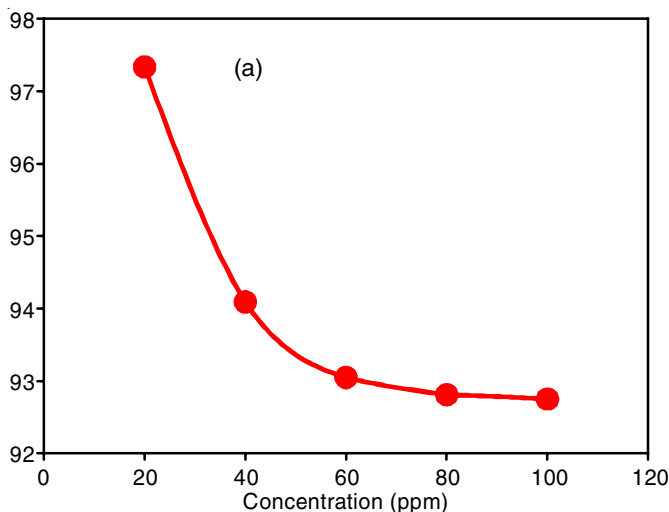


Fig. 3. Effect of (a) initial crystal violet dye concentration and (b) contact time on removal (%)

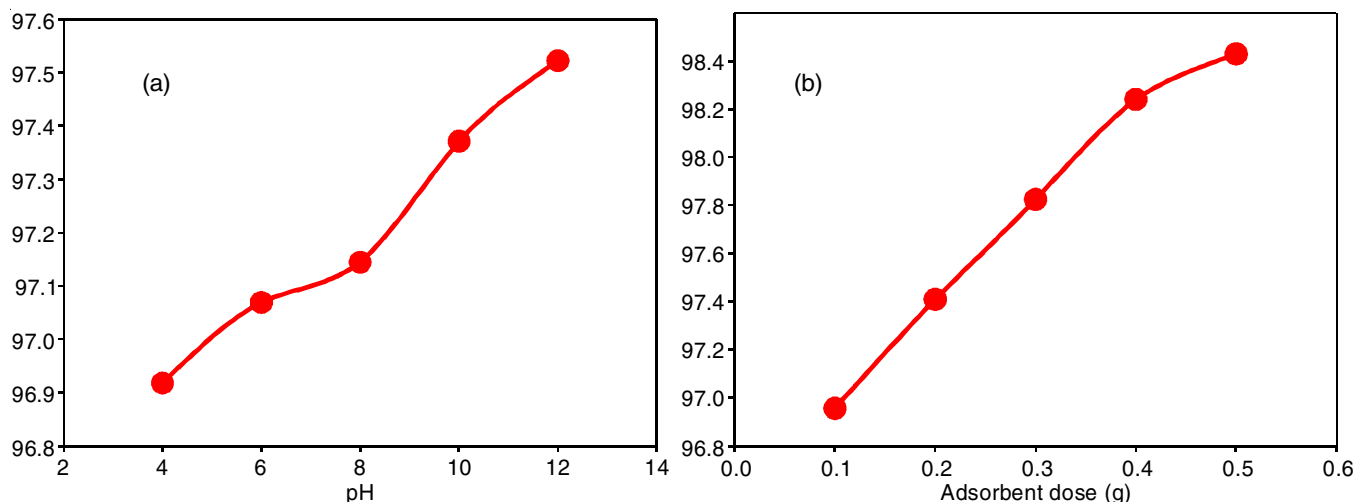


Fig. 4. Effect of (a) different pH on % removal and (b) various dosage of ACH on % removal

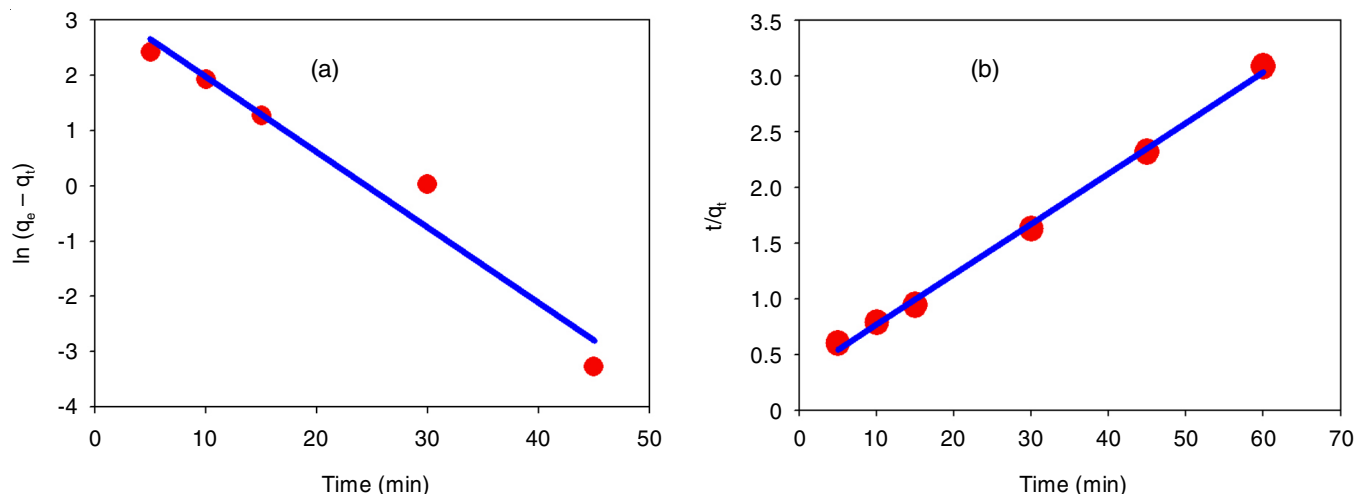


Fig. 5. Kinetic adsorption models (a) pseudo first order and (b) pseudo second order

strongly with pseudo-second order kinetic model. The adsorption efficiency computed by utilizing the same model was 22.22 (mg/g).

**Adsorption isotherms:** For a proper structure of sorption framework, it is required to acquire an ideal connection for the balance bends, which can be characterized as the connections between the centralizations of the metal adsorbed and metal staying in the arrangement at a particular temperature [19,30,31]. Langmuir adsorption isotherm tells about monolayer adsorption phenomenon while Freundlich tells about multilayer adsorption process. For equilibrium modeling of dye on to ACH, Langmuir and Freundlich isotherm models were investigated and are shown in Fig. 6a-b. The results of various parameters evaluated by adsorption isotherm modelling for dye by activated chickpea husk are tabulated in Table-3. It was observed that experimental data fitted best in Freundlich isotherm, which also confirmed from the value of  $R^2 = 0.972$ . This makes apparent that the adsorption of crystal violet dye onto activated chickpea husk is asymmetric or multilayered and Freundlich index ( $1/n$ ) is greater than 0.5, suggesting that the material is a favorable adsorbent. As the value of  $N$  is greater than 1, the adsorption mechanism is homogeneous [32].

Langmuir			Freundlich		
$q_m$	B	$R^2$	N	$k_f$	$R^2$
142.85	0.2	0.769	1.7	4.12	0.972

**Comparison of sorption capacity of activated chickpea husk with other adsorbents:** The comparison of the sorption capacity of chickpea husk carbon with other adsorbents for sorption of crystal violet dye has been tabulated in Table-4. It has been observed that the chickpea husk carbon exhibits the higher adsorption capacity for crystal violet dye. The high adsorption may be attributed to the availability of higher idle spots on leaning on the exterior of adsorbent [29].

## Conclusion

Activated chickpea husks were used for the removal of crystal violet dye from aqueous wastewater solution. Chemical structure and morphological analysis revealed better bonding interaction between adsorbate-adsorbent and the adsorbents consist of microporous structure, respectively. The various process parameters study revealed that maximum adsorption

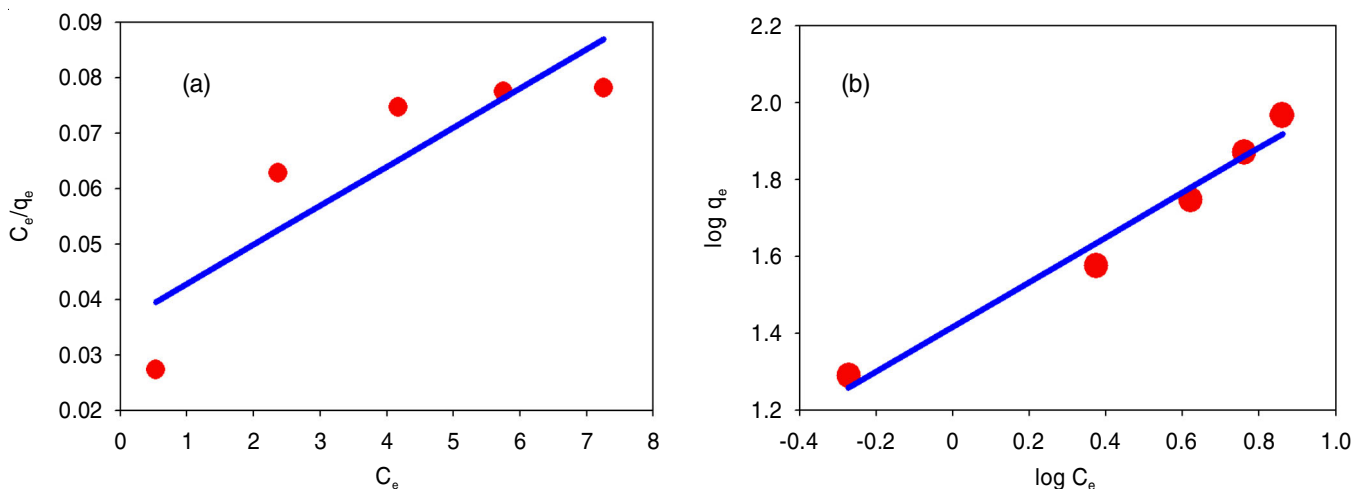


Fig. 6. Adsorption equilibrium modeling (a) Langmuir isotherm and (b) Freundlich isotherm

TABLE-4  
COMPARISON OF MAXIMUM ADSORPTION CAPACITY WITH VARIOUS ADSORBENTS FOR CRYSTAL VIOLET DYE REMOVAL

Adsorbents	Maximum adsorption capacity (mg/g)	Ref.
Modified Bambusa Tulda	20.84	[33]
Palm kernel shell biochar	24.45	[34]
Date palm leaflets carbon	36.63	[35]
Activated carbon prepared from waste apricot	57.80	[36]
Golbasi Lignite carbon	60.8	[37]
Ginger waste carbon	60.93	[38]
Pomegranate peel carbon	76.92	[39]
Tomato root carbon	83.33	[40]
ACH	142.85	Present study

was attained at pH 12 and at contact time of 45 min. Pseudo second order kinetic model and Freundlich isotherm were found to be best fitted with the experimental data. Comparison of equilibrium modeling confirmed multilayer adsorption over monolayer adsorption. Adsorption process was chemisorption following second order kinetics. Overall, it can be concluded that activated chickpeas husk show excellent potential for the removal of crystal violet dye.

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#### CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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