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Application of Modified Walnut Shell for Adsorptive Removal of Anionic Dye (Congo Red) from Simulated Water

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Abstract

Human and aquatic health is frequently at risk due to Congo red (CR) dye pollution in aquatic environments. To remediate water contaminated with CR dye, a chemically modified walnut shell adsorbent was developed in the present study. The physico-chemical characteristics of modified walnut shell were investigated using a variety of characterization techniques, including pH_{pzc}, FTIR, FE-SEM, and EDS. After optimizing the impact of the most important parameters, a maximum removal of 77.5% was noted at pH 4. The CR uptake by chemically modified walnut shell was explained by the monolayer layer adsorption onto energetically equivalent sorption sites. The Langmuir model yielded a maximal adsorptive capacity of 53.3 mg CR per g CMWS. The kinetic models also showed that the rate depends on the adsorbent's adsorptive capacity. The pseudo-second order model works well for CR adsorption, and its $R^2 > 0.99$ adsorption. Thermodynamic studies indicated that adsorption is feasible, spontaneous and endothermic in nature. This work suggests that CMWS can be investigated further as a possible adsorbent for the removal of CR dye.

Keywords: Walnut shell, Adsorption, Anionic dye, Isotherm studies

Introduction

Color is added to textiles, leather, paper, and other materials using dyes, which can be synthetic or organic substances. The textile sector alone accounts for two-thirds of the world's production of dyestuffs (Sahu and Poler, 2024). Because of its high toxicity and carcinogenicity, Congo Red poses serious environmental dangers despite its widespread use. Even at low concentrations, its durability and resistance to biodegradation can be harmful to ecosystems. Furthermore, it may result in major environmental risks such as eutrophication and human health like respiratory issues, skin and eye discomfort (Benahdach *et al*., 2025). Conventionally employed techniques for eliminating inorganic and organic contaminants from water include chemical precipitation, ion exchange, membrane filtration, coagulation, and adsorption (Zhao *et al*., 2020). Adsorption techniques offer the benefits of adaptability in application and simplicity in design that prevents the creation of hazardous intermediates or final products (Manzoor *et al*., 2024). Numerous types of forestry and agricultural waste, including lignin, rice husk, pine core, chestnut shell, etc., have reportedly been employed as environmentally beneficial adsorbents (Xu *et al*., 2017). Of these, walnut shell powder (WSP) has drawn the most attention because of its outstanding adsorption capability for the removal of colors and its widespread availability (Lima *et al*., 2019). The manufacture of walnuts produces a lot of trash, which is typically burned or dumped in a landfill because of its widespread use as food. Because buried walnut shells are so hard and take so long to break down, they create environmental hazard (Miyah *et al*., 2018).

Here, acidic surfactant treated modified walnut shell an appropriate and reasonably priced adsorbent, was prepared for the adsorption process of CR adsorption from aqueous solutions. The adsorption mechanism and kinetics of the CR was also investigated in this work, and the many physicochemical parameters influencing the rate of adsorption as well as the adsorption quantity of the adsorbent were identified. Adsorption was investigated in relation to solution pH, initial dye concentration, adsorbent dose, contact time, and temperature.

Material and Methods

The CR dye removal tests and the walnut shell modifications were carried out using analytical grade chemicals from Hi-media Laboratory Pvt. Ltd. The biomass made from walnut shells (WS) was bought from the local market, Hisar, Haryana, India. To obtain the necessary particle size, the dry walnut shells were crushed and sieved through a 40–60 mesh screen. Ten grams of the WS was added to a 250 mL conical flask containing 50 mL of 1 M H_2SO_4. The flask was then shaken continuously for 8 hours at 200 rpm and filtered followed by repeatedly washing until the solution's pH was neutral. 100 mL of 1% CTAB (cetrimonium bromide) solution was added to the WS in a flask. After that, the mixture was placed in an orbital shaker and shaken for 24 hours at 303 K at 200 rpm. The adsorbent was then filtered then repeatedly cleaned with distilled water and dried for 24 hours at 333 K in an oven and named as CMWS. The FT-IR spectra as well as surface morphology of CMWS was acquired both before and after adsorption. A digital pH meter was used for all pH measurements. A 100 ppm stock solution of the CR dye was prepared using deionized water. The Shimadzu 2600i spectrophotometer was used to detect the amounts of residual CR dye at 500 nm. Using CR solutions at different concentrations (10, 20, 30, 40, 50, and 60 mg/L), a calibration curve was calculated. The effectiveness of CMWS's adsorption of CR dye was investigated using batch studies. To optimize the pH, CR solutions (20 ppm, 20 mL) with varying pH values from 2 to 10 were prepared. CR solutions with several concentrations (20, 30, 40, 50, and 60 ppm) were prepared in order to optimize the initial concentration. Different amounts of adsorbent doses were taken, i.e., 0.2, 0.4, 0.6, 0.8, 1.0, 1.2 and 1.4 g; and added to CR solution to optimize the amount of adsorbent. For the optimization of contact time and temperature, adsorption studies were carried out at different temperatures (20°C, 25°C, 30°C, 35°C, 40°C, 45°C, 50°C) for different contact times (30, 60, 90, 120, 150, 180 and 210 min). By conducting batch studies, the reaction parameters pH, dye solution concentration, amount of adsorbent, contact time, and temperature were optimized. As recommended by Kayan and Kayan (2007), the removal efficiency (%) and adsorptive capacity (qt) were determined using Eqs. (1) and (2), respectively.

$$\text{Efficiency} = \frac{\text{Initial concentration} - \text{Final concentration}}{\text{Initial concentration}} \times 100 \quad \ldots(1)$$

$$\text{Adsorptive capacity} = \frac{\text{Initial concentration} - \text{Final concentration} \times \text{Volume}}{\text{Mass of adsorbent}} \quad \ldots(2)$$

The experimental data was fitted into the Freundlich and Langmuir adsorption isotherms to ascertain whether the adsorption is physical or chemical. Kinetic modelling was performed by fitting the experimental data into the pseudo-first order and pseudo-second order kinetic

Results and Discussion

FTIR and FESEM with EDS

The FTIR spectra of WS and CMWS before and after dye adsorption are shown in Fig. 1.The prominent hydroxyl group in WS, CMWS, and CR loaded CMWS has been ascribed to O–H and N–H stretching vibrations and is located between 3400 and 3457 cm^{-1}. Furthermore, peaks located between 2925,2928 and 2936 cm^{-1} show that methyl and methylene groups exhibit C–H stretching vibrations (Ovchinnikov *et al.,* 2016). But when compared to WS, the peak intensity was lower in CMWS, and CR-loaded CMWS, respectively. Our results are in accordance with previous literature cited (Sudarsan *et al.*, 2025). FESEM and EDS scans are often employed to investigate the morphological properties of the adsorbent. Using a FESEM, the surface properties of WS, CMWS, and CR dye-loaded CMWS were studied (Fig. 2a-b). The holes and irregularities of the CMWS surface may have served as binding sites for the CR dye during its adsorption (Almeelbi, 2025). The adsorbent-absorbate interaction is confirmed by the surface alteration

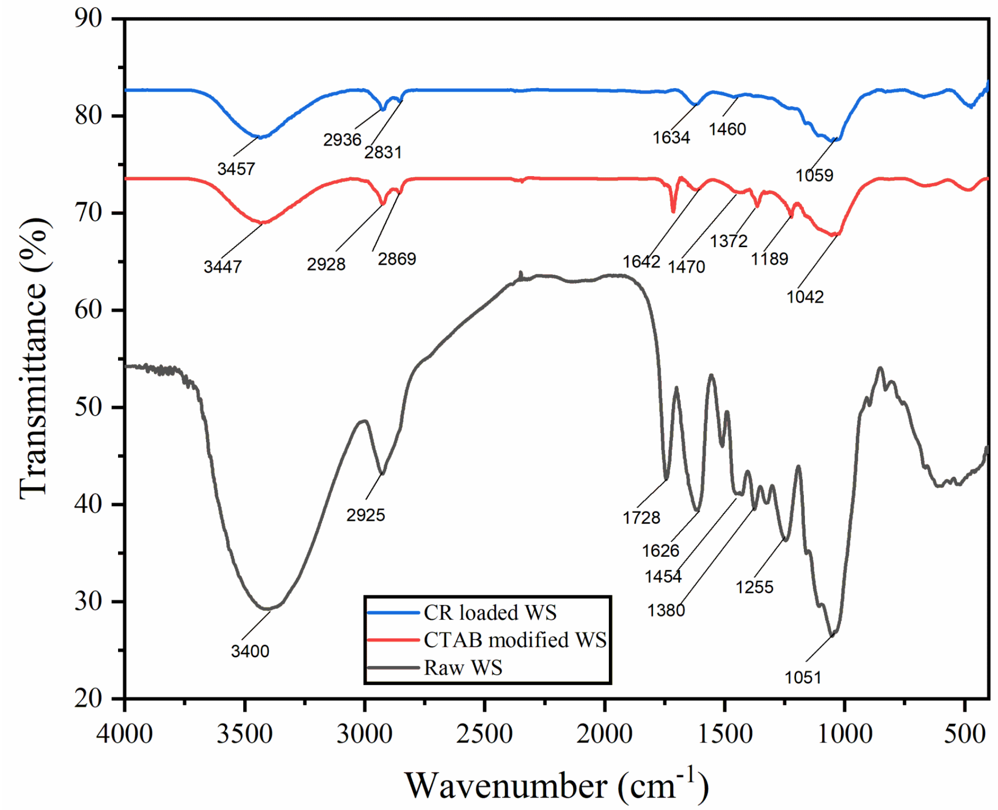

Fig. 1 FTIR spectra of (a) raw walnut shell (WS); (b) chemically modified walnut shell (CMWS); (c) CR-loaded CMWS

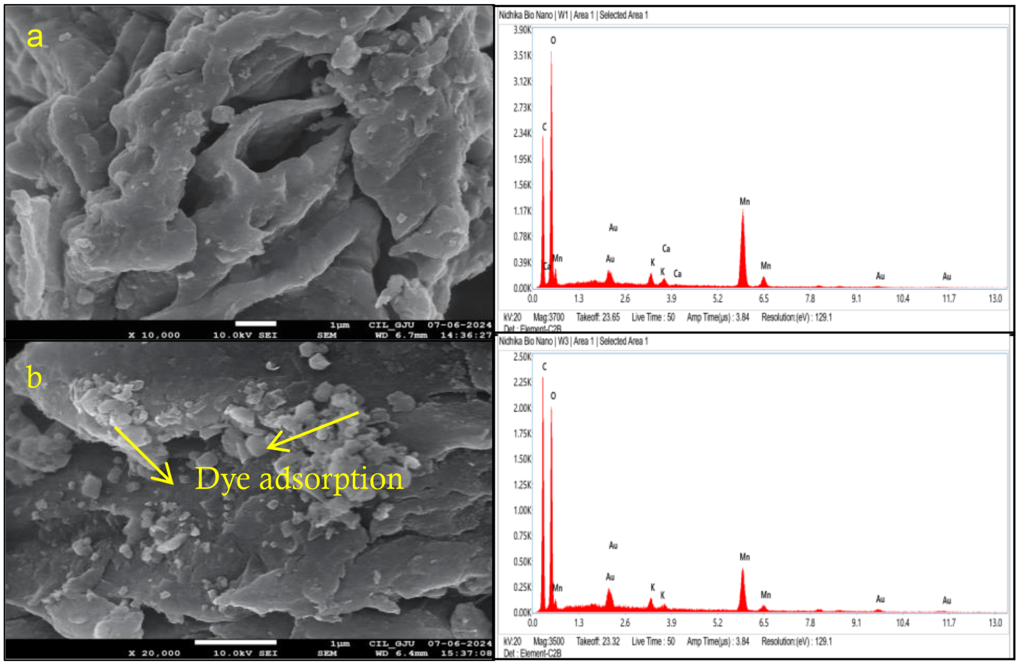

Fig. 2 FE-SEM micrograph of CMWS (a) before (b) after CR dye adsorption

following modification (pretreatment) and dye adsorption process. After CR dye loading, CMWS's surface morphology significantly changes, with holes filled and covered as well as the shiny and appearance of a smooth surface becoming rough as in Fig. 2b (Muskan *et al.,* 2025). EDS provides evidence of the chemical change happened on adsorbent surface both before and after dye adsorption.

Influence of pH

Point zero charge (pHPZC) plots between ΔpH vs pHi for CMWS is shown in Fig. 3f and the point of zero charge is found to be 4.8. Fig. 3(a) displays the CR adsorption efficiency of CMWS at pH values ranged from 2-10. As the pH was raised, the adsorption efficiency of CMWS was increased up to 4, after that, the removal efficiency decreases. With an adsorptive capacity of 53.3 mg/g, pH 4 was determined to have the greatest adsorption for the CR removal (77.5%). Since anionic dye and positively charged adsorbent surfaces have a strong electrostatic affinity, adsorption of anionic dye should be highest at low pH values (Chan *et al.,* 2024). Hamidon *et al.*, 2025 also observed that alginate hydrogel beads impregnated with cellulose nanocrystals produced from coconut shells (CS-CNC) had a maximal adsorption capacity of 73.24 mg g^{-1} for the removal of CR at pH 4. Based on the adsorbent's point of zero charge (pHpzc), which was 4.8, there was a negative charge present on the adsorbent's surface as the pH increased over the pHpzc, which decreased the removal effectiveness of CR.

Effect of various initial dye concentration

The removal efficacy of CMWS was shown to steadily decline with an increase in CR dye concentration from 10 mg/L to 50 mg L^{-1}, with a maximum adsorption of 74.58% reported at 20 mg L^{-1} (Fig 3b). Because there are only a fixed number of adsorbent active sites for every concentration of CR dye solution, the removal effectiveness dropped as the concentration of CR dye solution increased as reported by Nagpal *et al.* (2025) for removal of congo red using chitosan-based adsorbent.

Effect of adsorbent dose

Fig. 3(c) shows a noticeable increase when the quantity of CMWS increased from 0.1 g to 1.4 g. At 1.0 g, CMWS showed 80.43% removal. The removal effectiveness decreases as the amount of adsorbent is increased further. The reason behind the decline in removal effectiveness as the concentration of adsorbent is increased, there is the agglomeration of adsorbent particles, which leads to a reduction in the active sites that are accessible for adsorption as reported by Ojo *et al.* (2019) The saturation of adsorption with increasing adsorbent dosages is caused by the competition of dye molecules for available adsorption sites (Dovi *et al.*, 2021).

Influence of contact time

After increasing the reaction time from 10 to 210 min, CMWS achieved its maximum adsorption of 76.34% in 120 min (Fig. 3(d)). Removal efficiency has been steadily increasing until it reaches a constant value at equilibrium contact time suggesting that the adsorbed CR molecules have virtually saturated the adsorption sites of CMWS. When the adsorbent surface area started to become saturated, the removal rate decreased until equilibrium was established (Goswami *et al.*, 2020). Similar outcomes were also noted by Sathiyavimal *et al.,* 2021 for removal of dyes using copper oxide nanoparticles synthesized from *Psidium guajava* leaf extract.

Effect of temperature

The effect of solution temperature on removal of CR dye was determined in temperature range from 10-60° by keeping others parameters constant. The maximum efficiency of removal for CR on CMWS was noted to be 77.82 % at 30! (Fig. 3e). Increasing the temperature led to an increase in removal efficiency because more molecules had the threshold energy for an effective collision, which increased adsorption. The present results are in accordance with that of the Aftab *et al.*, 2023 for the adsorption of congo red using activated carbon from waste cardamom peel. At 30°C, congo red dye's maximum adsorptive removal (%) was 98 using using dried chitosan functionalized $MnFe_2O_4$ viscoelastic fluid (Khan *et al* ., 2025).

Adsorption isotherm models

The models of Freundlich and Langmuir isotherms are helpful in explaining the adsorption process by various adsorbents (Langmuir, 1918; Freundlich, 1906). Linear and nonlinear form form of Langmuir equation is given in equation 3 & 4.

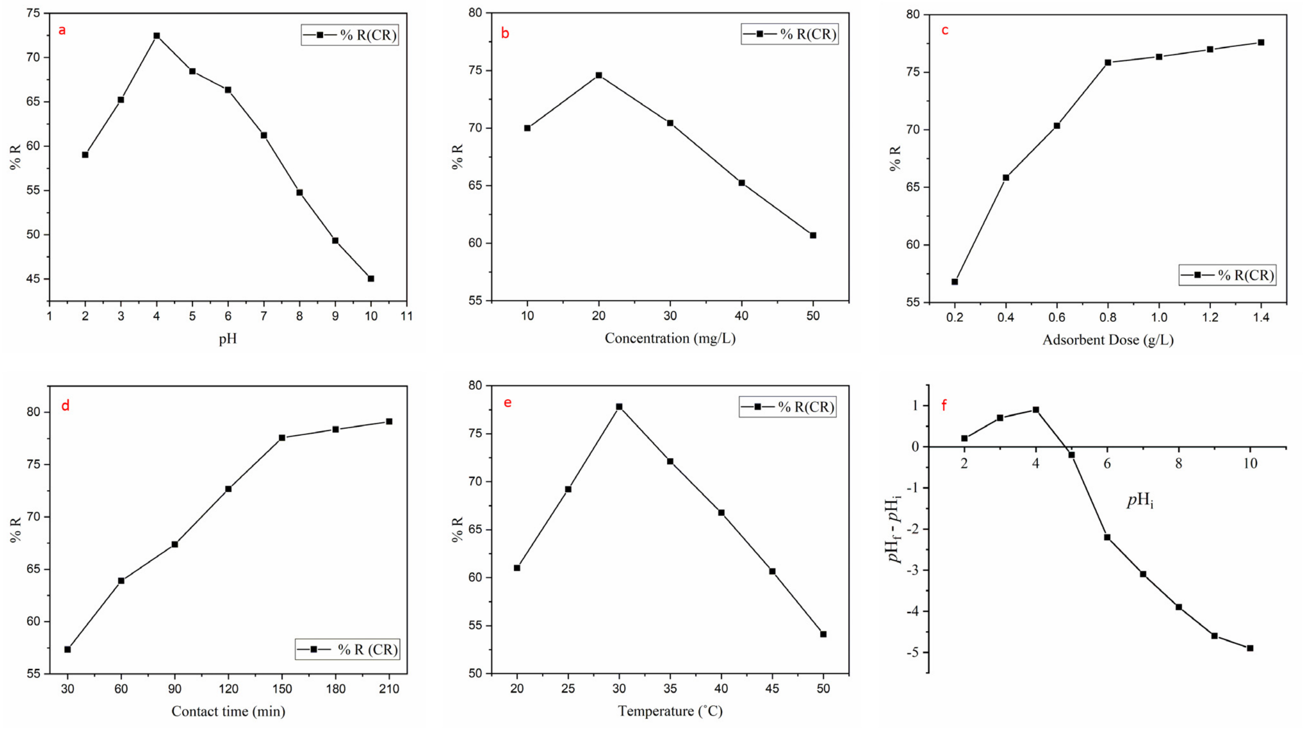

Fig. 3 Effect of (a) Ph, (b) dye concentration, (c)adsorbent dose, (d) contact time, (e) temperature on CR dye adsorption and (f) point zero charge of CMWS

$$\frac{C_e}{q_e} = \frac{1}{q_{max}.b} + \frac{C_e}{q_{max}} \quad \ldots(3)$$

$$q_e = \frac{q_{max} b C_e}{1+bC_e} \quad \ldots(4)$$

Where, q_{max} (mg/g) is maximum monolayer adsorption capacity, b (L/g) is Langmuir constant. Equation 5 & 6 provides the generic formula for the Freundlich equation. The adsorbent's adsorption capacity is denoted by k_f, its adsorption intensity is shown by the Freundlich constant 1/n, and its equilibrium concentration is represented by C_e.

$$q_e = k_f {C_e}^{1/n} \quad \ldots(5)$$

One taking logarithm of Eq. (5) shows linear form of Freundlich isotherm model can be written as Eq. (6).

$$\log q_e = \frac{1}{n} log C_e + log k_f \quad \ldots(6)$$

where, qe is the total amount of dye molecule adsorbed/total weight of adsorbents (mg/g), k_f (mg/g) is Freundlich constant, n is heterogeneity factor that relates to intensity of adsorption. As represented in Table 1, increased correlation coefficients (R^2) for the Langmuir model indicated that the adsorption data could be accurately explained by the Langmuir isotherm model. According to a study by Benahdach *et al.,* (2025) the Langmuir model most clearly explains the findings, with a maximum monolayer adsorption capacity of 26.93 mg g^{-1} at 40°C for the removal of congo red using activated carbon from pomegranate peel.

Table 1. Adsorption Isotherm parameters for CR dye on CMWS

Isotherm Models	Parameters	Values
Langmuir	qmax (mg/g)	53.3
	b (L/mg)	0.06
	R^2	0.9738
Freundlich	n	1.342
	Kf (mg/g)	5.06
	R^2	0.9409

Kinetics study

In this work, the pseudo-1st-order and pseudo-2nd-order models were attempted in order to comprehend the kinetic behaviour of dye molecule adsorption onto CMWS. The pseudo first order

Table 2. Kinetics model parameters for CR dye on CMWS

Conc.	Pseudo first order			Pseudo second order		
(mg/L)	q_e	k_1 min^{-1}	R^2	q_e (mg/g)	K_2 g/mgmin	R^2
10	45.136	0.0267	0.9909	9.23	0.0033	0.9983
20	504.29	0.0450	0.9587	18.58	0.0014	0.9985
30	1296.4	0.0456	0.9545	28.09	0.0012	0.9987
40	1921.2	0.0405	0.937	37.57	0.0009	0.9979
50	3778.7	0.0455	0.9064	46.68	0.0008	0.9972

is expressed in linear form in equation 7 (Samimi and Safari, 2022).

$$\log(q_e - q_t) = \log q_e - (\frac{k_1}{2.303})t \quad \ldots(7)$$

Here, qe & qt (mg/g) are adsorption capacity at equilibrium time and at time t(min.), respectively, k_1(min^{-1}) pseudo first order rate constant the value of k_1, q_e, R^2 calculated from linear plot between $\log(q_e\text{-}q_t)$ vs t. Pseudo second order is in linear form is represented in equation 8 (Staron *et al.*, 2017).

$$\frac{t}{q_t} = \frac{1}{k_2\, q^2 e} + \frac{t}{q_e} \quad \ldots(8)$$

Here, k_2 (g/mg min^{-1}) is rate constant of pseudo second order. By plotting graph between t/q_t vs t we can calculate k_2, q_e, R^2 values. The q_e (exp) values from the adsorption experiment are compared to the q_e (cal) value calculated using the kinetic models. Table 2 presents the validation findings and the kinetics model parameters. The pseudo-second order regression coefficient is greater than the pseudo-first order regression coefficient, according to experimental data. The results of the kinetic analysis of the CMWS showed that the rate was dependent on the adsorbent's adsorptive capacity (Ausavasukhi *et al.*, 2025; Rani *et al.,* 2025).

Thermodynamic studies

The thermodynamic parameter, including Gibbs free energy (ΔG^0) and entropy change (ΔS^0) and enthalpy (ΔH^0) for the adsorption of CR dye have been evaluated and verified using equations from 9 – 11 (Umeh *et al.*, 2024).

$$\Delta G^O = -RTlnk_d \quad \ldots(9)$$

$$k_d = \frac{C_a}{C_e} \quad \ldots(10)$$

$$\ln k_d = \frac{\Delta H^O}{RT} - \frac{\Delta S^O}{R} \quad \ldots(11)$$

Here, k_d is equation constant, R gas constant (8.314 J/mol/K), T is temperature in K,C_a is amount of adsorbate on adsorbent surface at equilibrium. ΔH^0 & ΔS^0 was determined from linear plot between $\ln k_d$ vs 1/T. The solid-solution interface's increased degree of randomness is confirmed by the positive values of ΔS^0 as well as endothermic nature of the adsorption process is demonstrated by the positive values of ΔH^0 for dye molecule *via* CMWS (Muskan *et al.*, 2025). Adsorption process feasibility and spontaneity are confirmed by negative ΔG^0 values of dye molecule at increasing temperatures, indicating physical adsorption as in Table 3 (Saini *et al.*, 2025).

Table 3. Thermodynamic parameters for for CR dye on CMWS

Temperature (K)	ΔG° (kJ mol^{-1})	ΔH° (kJ mol^{-1})	ΔS° (J mol^{-1} k^{-1})
30°C	-3.2520	-29.7564	-90.6980
40°C	-2.2474	-	-
50°C	-1.1859	-	-

Conclusions

The removal of CR from simulated water using CMWS has been investigated under different experimental conditions in batch process. According to SEM images, CMWS surface is rich in pore spaces, which facilitate the adsorption of CR molecules. The adsorption of dye molecules were influenced by the *p*H, dye concentration, adsorbent dosage, and temperature and contact time. Based on adsorption studies, it was found that removal of dye molecules occurs at pH 4. The Langmuir isotherm was closely followed during the adsorption process by the adsorbent and adsorption kinetics followed pseudo second order. The adsorption process's feasibility and spontaneity are confirmed by the negative ΔG^0 values. The results of this study showed that Congo red may be successfully removed from an aqueous environment by using chemically modified walnut shell.

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