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Sequestration of crystal violet dye from wastewater using low-cost coconut husk as a potential adsorbent

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ABSTRACT

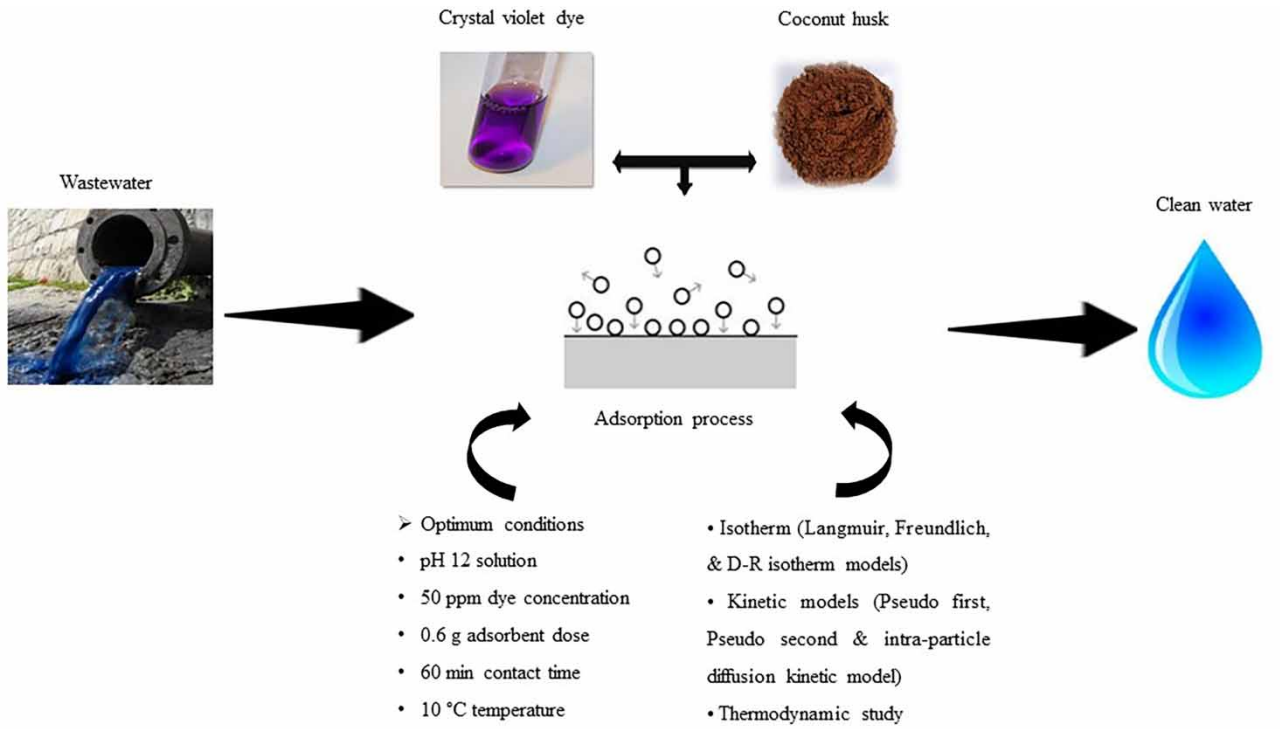
The current study explores the effectiveness of coconut husk for crystal violet dye sequestration employing a batch experimental setup. Characterization of adsorbent was carried out via FTIR, and SEM techniques and results confirmed the involvement of –OMe, –COC– and hydroxyl functional groups in dye uptake, and the rough, porous nature of adsorbent and after adsorption dye molecules colonized these holes resulting in dye exclusion. Effects of various adsorption parameters such as pH, adsorbent dose, contact time, initial dye concentration, and temperature of solution were studied. Crystal violet adsorption on coconut husk was highly pH-dependent, with maximum removal occurring at basic pH. Maximum removal of dye, i.e., 81%, takes place at optimized conditions. Kinetic data was analyzed by pseudo-first, pseudo-second order and an intra-particle diffusion model. Results showed that the pseudo-second order kinetic model best described adsorption of crystal violet onto coconut husk. Langmuir, Freundlich, and D-R adsorption isotherms were also used to test their appropriateness to experimental data and the Freundlich isotherm fits best to data. Thermodynamic parameters showed that the current process was spontaneous, endothermic in nature with continuous decrease in entropy. Established practice is 79% applicable to tap water and in acidic medium nearly 80% of adsorbent was recovered, confirming the effectiveness and appropriateness of coconut husk for crystal violet dye exclusion from wastewater.

Key words: adsorption, coconut husk, crystal violet, isotherms, kinetics, thermodynamics

HIGHLIGHTS

- Adsorption is an effective and economical process for dye removal from wastewater.
- Coconut husk is a potential adsorbent for crystal violet exclusion.
- Adsorbent characterization, operational parameters, and adsorbent recovery was studied.
- Kinetics, isotherm models, and thermodynamics were evaluated.
- The potential efficiency of adsorbent will also be checked for actual textile wastewater in future.

GRAPHICAL ABSTRACT



SYMBOLS

q_t	Adsorption capacity at time 't', mgg^{-1}
q_e	Adsorption capacity at equilibrium, mgg^{-1}
C_o	Initial concentration of dye, mgL^{-1}
C_e	Equilibrium concentration of dye, mgL^{-1}
V	Volume of dye solution taken, ml
M	Mass of adsorbent, g
K_F	Pseudo-first order rate constant, mgg^{-1}
K_s	Pseudo-second order rate constant, mgg^{-1}
K_L	Langmuir constant, $dm^{-3}mol^{-1}$
b	Langmuir equilibrium constant, $Lmol^{-1}$
R_L	Dimensionless constant, separation factor parameter
n	Intensity of adsorption, L g^{-1}
β	Constant related to adsorption energy or activity coefficient, KJ^2mol^{-2}
q_{DR}	Imaginary inundation capacity
ε^2	Polanyi potential, $KJmol^{-1}$
C_{id}	Intra-particle rate constant, $mgg^{-1}min^{-1/2}$
C_{ads}	Adsorbed concentration, mgL^{-1}
C_{eq}	Unabsorbed quantity of adsorbate in solution at equipoise, mgL^{-1}
T	Temperature, K
R	Universal gas constant = 8.31 $Jmol^{-1}K^{-1}$
ΔG	Change in Gibbs free energy, $KJmol^{-1}$
ΔH	Change in enthalpy, $KJmol^{-1}$
ΔS	Change in entropy, $Jmol^{-1}K^{-1}$

1. INTRODUCTION

With increase in population, accessibility of fresh and clean drinking water diminishes. The essentiality of water for drinking purposes for human beings cannot be over stated (Namasivayam & Sangeetha 2006; Parab *et al.* 2009; Wang & Chu 2011).

The quality of water deteriorates easily due to solubility of all kinds of pollutants or solutes (Sureshkumar & Namasivayam 2008; Jain *et al.* 2015; Rani *et al.* 2017). Among all pollutants, dyes are one of the common (Yadav *et al.* 2013; Ojedokun & Bello 2017). Dyes are chemical substances that may attach to a surface and impart color to it. The majority of dyes are complex chemical compounds that are resistant to biodegradation. Synthetic dyes are widely employed in modern technological domains such as textiles paper, leather tanning, food processing, plastics, cosmetics, rubber, printing, and dye production sectors (Ivanov *et al.* 1996; Sokolowska-Gajda *et al.* 1996; Kabdaşli *et al.* 1999; Ul-Islam *et al.* 2016; Ali *et al.* 2017). These coloring substances on discharge into water bodies cause a reduction in sunlight penetration making aquatic life more susceptible to photochemical and biological assault (He & Tebo 1998). Cationic dyes are more toxic than anionic dyes due to their fast reaction with negative moiety of cell membrane surface and are accountable for causing allergic reactions (Abbaz 2017; Miyah *et al.* 2017).

Gentian violet, also known as crystal violet (Figure 1 reproduced with permission from Al-Shahrani 2020), is a basic cationic dye widely used in industry. It gives a blue color with water solution having λ_{max} of approximately 590 nm. The color of the dye changes with a change in solution pH. Often it is a green hue at a pH value of 1 while yellow at highly acidic pH (Okorocha *et al.* 2019). It finds numerous uses in paint, printing, and veterinary medicines and possesses antibacterial as well as antifungal properties (Nasar & Shakoor 2018). However, improper release of such dyes into water is accountable for serious issues as crystal violet dye is accountable for breathing issues, oral problems, nausea, eye burns, jaundices, tissue necrosis, kidney failure, etc. Hence, for protection of water resources in addition to safety of human health, various wastewater treatment technologies are employed (Batool *et al.* 2021).

Water treatment technologies include ozonation, electro-coagulation, membrane process, biological, and solvent extraction. However, adsorption (due to its simple design, efficiency, ease of operation, economic feasibility, insensitivity to hazardous chemicals, and easy availability of adsorbents used) is an effective and promising practice for dye removal (Okorocha *et al.* 2019). Activated carbon is a well-known adsorbent offered because of its high adsorption capacity, large surface area, and micro-porous structure, but it has numerous drawbacks, such as being expensive; besides regeneration of saturated carbon is very costly and complicated (Malik 2003; Crini 2006).

This research article deals with low-cost and effective adsorption of crystal violet dye from an aqueous medium employing adsorption practice with coconut husk as an adsorbent. Coconut husk (*Cocos nucifera*) is an agricultural waste product (powder form of coconut husk is shown in Figure 2 reproduced with permission from Hanafiah *et al.* 2020) obtained

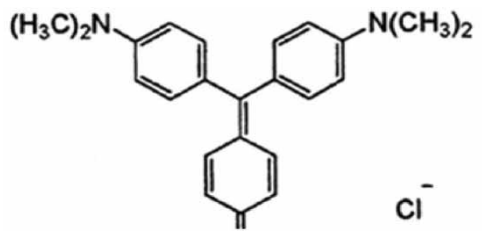


Figure 1 | Structural formula of crystal violet dye.

Figure 2 | Powder form of coconut husk.

from coconut tree with vast applications in consumption, beautification besides erosion control. It is the covering of fruit and when fruit (white part) is consumed then it is discarded into environment and accountable for environmental pollution (Bello *et al.* 2019). Bt Man *et al.* 2015 used coconut shell-derived activated carbon for methylene blue adsorption (bt Man *et al.* 2015) while Okafor *et al.* 2012 used coconut shell–derived activated carbon for removal of lead, copper, cadmium, arsenic, etc. from wastewater (Okafor *et al.* 2012; Sultana *et al.* 2022). However, coconut husk waste can be converted into a useful product by making use of it as a promising adsorbent for crystal violet dye exclusion from wastewater. Aljeboree *et al.* 2015 used H_2SO_4-modified coconut husk derived activated carbon powder (CHACP) for removal of crystal violet dye from wastewater resulting in maximum removal in 60 minutes with involvement of hydroxyl functional groups in dye uptake (Aljeboree *et al.* 2015). Coconut husks were employed in exclusion of methylene blue dye from wastewaters in Malaysia via column study resulting in the highest bed capacity of 50,756 mg/g confirming adsorbent as an effective adsorbent for dyes adsorption as compared to other costly adsorbents (like activated carbon) (bt Man *et al.* 2015). Mustapha *et al.* 2020 studied adsorptive removal of Remazol red dye via coconut husk (adsorbent) and compared efficiency of raw and treated (through alkaline and bleaching process) coconut husk towards dye removal. They concluded that some cellulose and lignin components had been eliminated in the course of alkaline and bleaching process resulting in 75% dye removal (i.e., through activated coconut husk) (Mustapha *et al.* 2020). Several other studies have also been reported on coconut-based adsorbents for dye removal, which include using coconut husk (Low & Lee 1990; Jain & Shrivastava 2008; Gupta *et al.* 2010), coconut coir pith (Namasivayam *et al.* 2001; Unnithan *et al.* 2004; Pathak *et al.* 2006; Gonzalez *et al.* 2008; Parab *et al.* 2008; Suksabye & Thiravetyan 2012), coconut bunch waste (Hameed *et al.* 2008), coconut shell fiber (de Sousa *et al.* 2010), activated carbon prepared from male flowers of coconut tree (Senthilkumaar *et al.* 2006), coconut shell-derived hydrochar (Islam *et al.* 2017) etc. Investigation of coconut husk for decolorization of crystal violet dye from wastewater, optimization of various adsorption parameters for efficient dye removal, as well as determination of dye decolorization kinetics, isotherm models, and thermodynamic parameters are among this research's goals.

2. MATERIALS AND METHODS

2.1. Apparatus and chemicals required

All chemicals used in the current research were of analytical standard and purchased from Germany (Merck) Sigma–Aldrich Chemical Company (USA) and utilized as received. Throughout the study, distilled water was utilized. Tap water was also used to check the applicability of developed procedure in a real system. UV-visible spectrophotometer (Z-2000 Polarized, Hitachi, Japan), scanning electron microscope (SEM) (Joel, Japan), fourier transform infrared (FTIR) spectrophotometer (Spectrum 100, Perkin Elmer), centrifugation machine (SARSTEDT D-51582), microwave oven (EM028ADK), pH meter (Metrohm 605 pH meter, USA), weighing balance (Shimazdu TW, TX and TXB series), and hot plate (CORNING PC-420D) are among the apparatus used during the process.

2.2. Collection of adsorbents

Coconut husk was obtained from fruit seller cart (local) from Sahiwal. It was washed with distilled water to remove impurities and then dried. To enhance its surface area, dried product is ground, then crushed into smaller particles, followed by sieving to extract adsorbent particles of different mesh sizes i.e., 105, 210, and 500 and stored in airtight plastic jars or bottles for future use.

2.3. Preparation of adsorbate solution

Percentage purity of dye used is 99.99%, and 1,000 ppm or mg/L crystal violet dye stock solution was prepared by adding a suitable amount (i.e., 1 g) of crystal violet dye in a definite volume (i.e., 1,000 ml) of distilled water followed by shaking. Desired dilute solutions were prepared from this stock solution for future usage. Both adsorption and desorption studies were carried out in the current study. Additionally, applicability of the developed procedure to tap water was also checked.

2.4. Determination of pH_{pzc}

pH_{pzc} of coconut husk adsorbent was determined by adding 0.1 g of coconut husk adsorbent in 200 ml of 0.1 M NaCl solution whose pH is known. This mixture is then agitated in a shaker at 250 rpm speed for 4 hours after which pH was measured and variation in pH was plotted against initial pH value. pH_{pzc} is attained when no charge occurs after contacting the adsorbent (Bello *et al.* 2017; Agboola *et al.* 2021).

3. ADSORBENT CHARACTERIZATION

To determine functional groups present on the outer surface of adsorbent and to examine the texture and morphology of the adsorbent, FTIR and SEM techniques were employed respectively. 1% pallet of potassium bromide was used to conduct an FTIR analysis of the adsorbent. The study used a spectral range of 4,000–400 cm^{-1} for scale (Mashkoor *et al.* 2018). The sample, for SEM investigation, was prepared by sieving adsorbent after grinding it in a pestle and mortar. It was then held on taster holders using double-sided carbon tape (Tariq *et al.* 2017). The adsorbent was analyzed via SEM both before and after adsorption.

4. BATCH ADSORPTION STUDIES

To achieve maximum dye removal from wastewater, optimization of various operational parameters was studied. These parameters were solution pH, quantity of adsorbent used, dye concentration, contact time, and solution temperature. The value of only one parameter was changed at a time with all other parameters remaining constant. Before adsorption, the initial concentration (C_i) of dye was noted using a UV-Vis spectrophotometer at a wavelength of 590 nm. After centrifugation, the quantity of dye contained in the supernatant was determined using a UV-Vis spectrophotometer and concentration measurement (C_f) at 590 nm. Percentage removal of dye can be calculated by Equation (1):

$$\%\ removal = \frac{C_i - C_f}{C_i} \times 100 \tag{1}$$

where C_i = initial concentration of crystal violet dye in solution before dye adsorption and C_f = final concentration of crystal violet dye in solution after dye adsorption or at equilibrium. Adsorption capacity $q_e\left(\frac{mg}{g}\right)$ can be determined by using Equation (2):

$$q_e = (C_o - C_e) \times \frac{V}{M} \tag{2}$$

where C_o (mg L^{-1}), C_e (mg L^{-1}) are initial and equilibrium concentrations of dye, V is volume of dye solution and M is the mass of adsorbent used in grams. All experiments of the batch study were carried out twice (to lower the chances of errors) and the mean value of results was taken as the optimum value.

5. RESULTS AND DISCUSSION

5.1. FTIR analysis

Functional groups present in coconut husk were determined by FTIR spectroscopy. FTIR spectra displayed numerous peaks, demonstrating the complex nature of coconut husk (Elella *et al.* 2019). A sharp peak at 3,778.55 cm^{-1} corresponds to the presence of O–H stretching of alcoholic or phenolic compounds with no hydrogen bonding. A broad peak was observed at around 3,452.58 cm^{-1}, corresponding to presence of O–H stretching of alcoholic or phenolic compounds with hydrogen bonding. These two peaks confirmed the presence of lignin and cellulose on the coconut husk surface (Malik & Dahiya 2017). Peaks at 2,926.01, 694.37, and 1,635 cm^{-1} correspond to presence of C–H stretching for methyl or methylene, bending of aromatic C–H, and methoxy group (C–O) from lignin and $C = C$ characteristic of aromatic ring correspondingly. A wide peak/band at 1,101.35 cm^{-1} corresponds to a –COC– functional group, which confirmed the presence of polysaccharides in coconut husk powder (Arief *et al.* 2008). Peaks ranging from 1,600 to 1,100 cm^{-1} discriminate interactions between crystal violet dye molecules and coconut husk powder. After adsorption of dye, peaks from 1,635.64 to 1,101.35 cm^{-1} were shifted in the range of 1,606.70 to 1,089.78 cm^{-1}, i.e., there was a shift in peaks (change in intensity of peaks) of these functional groups showing involvement of these functional groups in adsorption of crystal violet dye on coconut husk powder. Figure 3 shows FTIR spectra both before and after dye adsorption (Sultana *et al.* 2022).

5.2. SEM analysis

According to SEM (with 10 μm resolution) examination, the surface of the adsorbent is highly rough, varied, as well as porous, suggesting an enhanced superficial zone of adsorbent available for crystal violet dye adsorption. After adsorption,

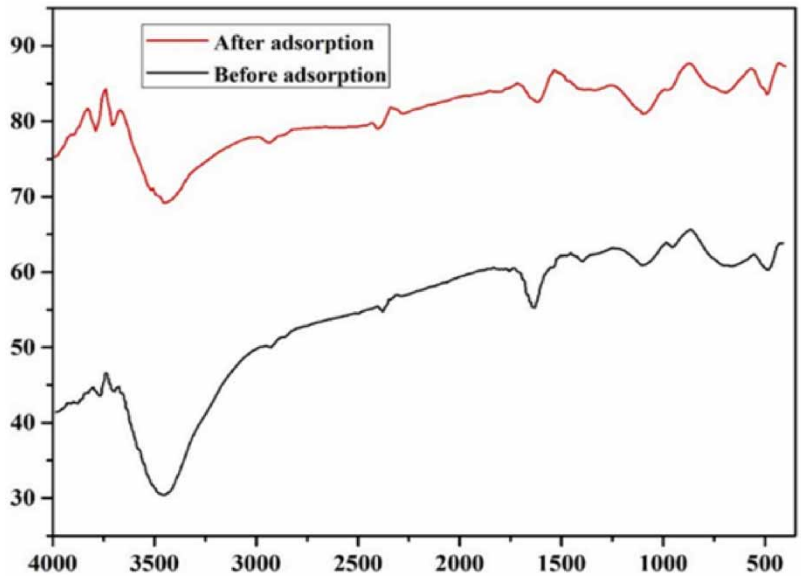


Figure 3 | FTIR spectra of dye unloaded and loaded coconut husk (reproduced with permission).

dye molecules colonized these pores resulting in dye exclusion (Johari *et al.* 2016). Results of SEM analysis both before and after adsorption are shown in Figure 4.

5.3. Selection of the best particle size

The best particle size of adsorbent used was selected (105, 210 and 500 μm) to achieve maximum adsorption of dye. For this, 0.25 g of each particle size was used for analysis at neutral pH value. 10 mL of dye solution (100 ppm) was used. Experimental results revealed that with increase in particle size, adsorption decreased due to decrease in surface area of adsorbent (which favors the adsorption process). Maximum removal of dye was 86% with 105 μm, particles while removal percentage decreased with increase in particle size and was found to be 74% for 210 μm and 61% for 500 μm particles respectively due to decrease in surface area of adsorbent (Patil *et al.* 2011; Hussein & Jasim 2019) as shown in Figure 5.

5.4. Effect of solution pH

Solution pH is an important parameter of crystal violet dye adsorption as it greatly affects charge on the adsorbent exterior. By changing pH of solution from 1 to 12 while keeping all other variables constant, the effect of solution pH on percentage exclusion of crystal violet dye was examined. For this, 10 ppm crystal violet dye solutions having variable pHs (1–12) were prepared in distilled water and initial concentration of dye in each solution was determined by UV-Vis spectrophotometer

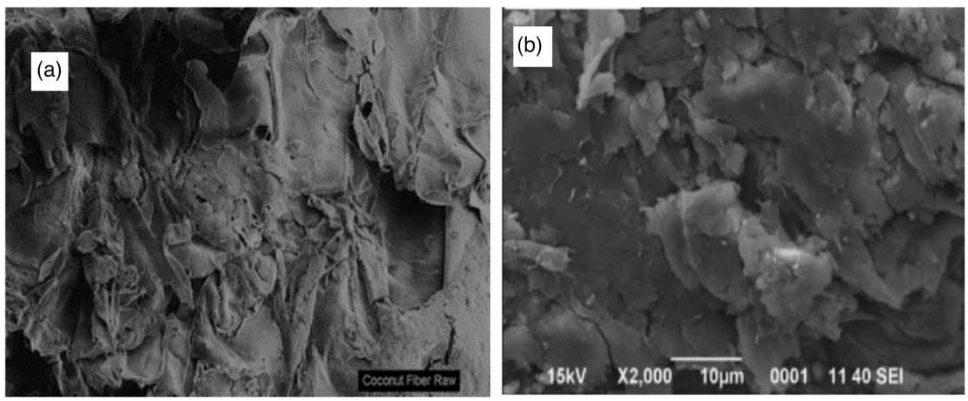


Figure 4 | SEM images of coconut husk (a) before adsorption (b) after adsorption (reproduced with permission).

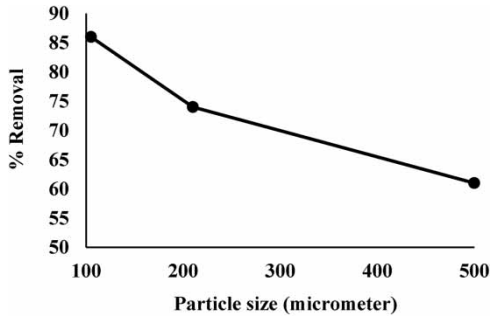


Figure 5 | Selection of the best particle size.

at 590 nm and 0.3 g of coconut husk adsorbent was added, shaken well, and kept for 20 minutes (contact time) at room temperature i.e., 25 °C. After 20 minutes, the solution was centrifuged at 3,000 rpm speed for 3 minutes and final concentration of dye in supernatant solution was determined via UV-Vis spectrophotometer at 590 nm. Using Equation (1) percentage removal of dye was calculated at each pH value. Results showed that percentage removal of dye increased continuously from pH 1 to 12 values (as shown in Figure 6). At pH 12, maximum amount of dye i.e., 92% adsorbed on adsorbent exterior because at high pH value strong interactions developed between positive dye molecules and negatively charged adsorbent surface while at low pH solution hydrogen ions compete with positively charged dye molecules resulting in decreased percentage removal of dye. Similar results were reported by Langmuir 1916; Namasivayam & Kavitha 2002; Al-Degs *et al.* 2008; Nandi *et al.* 2009; Dandge *et al.* 2016; and Muhammad *et al.* 2019. The effect of pH on percentage removal of dye was best explained via point of zero charge (pH_{pzc}). Graph of pH effect and point of zero charge is shown in Figures 6 and 7. We can determine pH_{pzc} by plotting a graph between pH_i and ΔpH (final pH – initial pH). The point where the line of the graph cuts the pH_i axis is the point of zero charge of adsorbent, which is 7 for coconut husk (as shown Figure 7). Hence, at solution pH below 7, percentage removal was at the minimum due to positively charged exterior of adsorbent (as of protonation of functional groups) but at solution pH higher than 7, the surface of adsorbent is negatively charged (due to deprotonation of functional groups of adsorbents) leading to stronger electrostatic interactions among dye molecules and functional groups present at adsorbent exterior i.e., coconut husk has greater affinity for crystal violet dye adsorption when solution pH $\geq$ 7. Results of solution pH on dye removal are given in table S1.

5.5. Effect of adsorbent dose

Changing the weight of the adsorbent from 0.1 g to 1.0 g, while keeping all other variables constant, was used to study the effect of adsorbent dosage on dye adsorption. For this, 10 ppm crystal violet dye solutions having pH 12 was prepared in

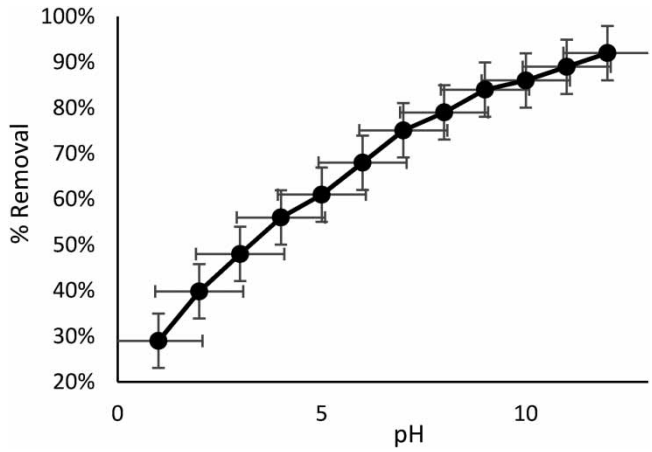


Figure 6 | Effect of pH on % removal (experimental conditions: pH = variable (1–12); C_o = 10 ppm; adsorbent dose = 0.3 g; contact time = 20 min; temperature = 25 °C).

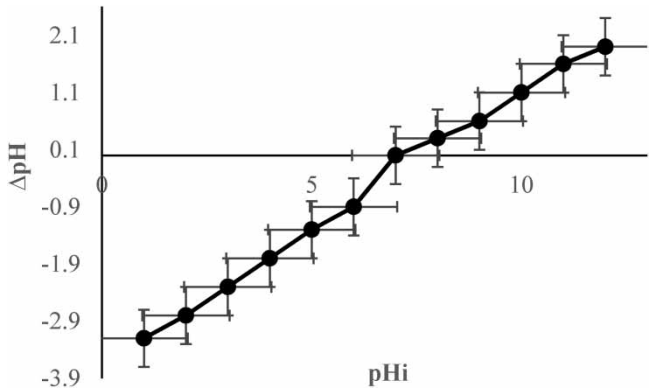


Figure 7 | Graph for determination of point of zero charge.

distilled water and initial concentration of dye in each solution was determined by UV-Vis spectrophotometer at 590 nm. Now variable amount of adsorbent, i.e., from 00.1 to 1.00 g of coconut husk, was added, shaken well, and kept for 20 minutes (contact time) at room temperature, i.e., 25 °C. After 20 minutes, the solution was centrifuged at 3,000 rpm speed for 3 minutes, and the final concentration of dye in the supernatant solution was determined via UV-Vis spectrophotometer at 590 nm. Using Equation (1), percentage removal of dye was calculated at each adsorbent dose value. Results showed that percentage removal of dye increases continuously with increase in adsorbent amount up to a limit after which it becomes constant. Maximum adsorption of dye takes place at 0.8 g of adsorbent dosage at which percentage removal was found to be 81%. This was because with increase in amount of adsorbent used, more active sites are available for dye uptake at constant dye concentration (Lu *et al.* 2016; Zango & Imam 2018; Karthik *et al.* 2019; Sujata *et al.* 2019). At 0.6 g adsorbent amount, percentage removal was 80%. As there was not much difference in percentage removal at 0.6 g and 0.8 g of adsorbent amount, we used 0.6 g adsorbent dose for the next experiments to conserve adsorbent. Results of adsorbent dosage on dye removal are given in table S2 and graph is shown in Figure 8.

5.6. Effect of contact time

Percentage adsorption of crystal violet dye with respect to time variation on coconut husk was studied at optimized adsorbent dosage, i.e., 0.6 g using dye solution of pH 12. For this, 10 ppm crystal violet dye solutions having pH 12 were prepared in distilled water and the initial concentration of dye in each solution was determined by UV-Vis spectrophotometer at 590 nm and 0.6 g of coconut husk adsorbent was added, shaken well, and kept for variable contact time, i.e., from 5 to 120 minutes at room temperature, i.e., 25 °C. After this, the solution was centrifuged at 3,000 rpm speed for 3 minutes, and the final concentration of dye in the supernatant solution was determined via UV-Vis spectrophotometer at 590 nm. Using Equation (1) percentage removal of dye was calculated at each contact time value. Equilibrium time changes from 5 to 120 minutes. Optimum contact time for crystal violet dye adsorption was found to be 60 minutes at which 80% of

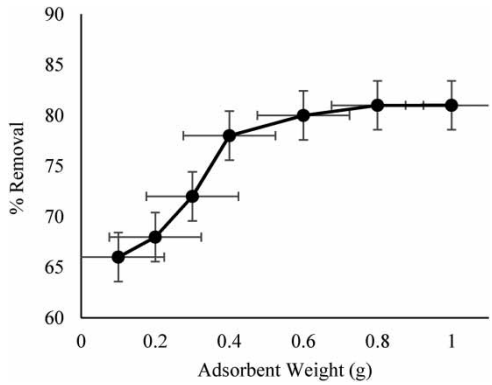


Figure 8 | Effect of adsorbent dosage on % removal (experimental conditions: pH = 12; C_o = 10 ppm; adsorbent dose = variable; contact time = 20 min; temperature = 25 °C).

dye removal takes place. A similar trend was observed by Foroutan *et al.* 2021. For the first 60 minutes, percentage removal increased rapidly with increase in exposure time while with further increase in contact time desorption of dye starts because at saturation point dye molecules are weakly held on the adsorbent surface (probably known as the second adsorption layer) (Wanyonyi *et al.* 2014; Okorocha *et al.* 2021). In other words, at saturation point active sites of adsorbent were impregnated with the dye molecules (Sultana *et al.* 2022), leading to decrease in percentage removal. Results of contact time on dye removal are given in table S3 and graph is shown below in Figure 9.

5.7. Adsorbate concentration effect

For the adsorption procedure, the effect of initial dye concentration on percentage removal of dye is critical. To investigate the effect of adsorbate concentration on percentage removal, different solutions of crystal violet dye, i.e., from 20 to 120 ppm of pH 12 were prepared in distilled water, the initial concentration of each dye solution was determined by UV-Vis spectrophotometer at 590 nm, and 0.6 g of coconut husk adsorbent was added, shaken well, and kept for contact time of 60 minutes at room temperature, i.e., 25 °C. After this, the solution was centrifuged at 3,000 rpm speed for 3 minutes, and the final concentration of dye in the supernatant solution was determined via UV-Vis spectrophotometer at 590 nm. Using Equation (1) percentage removal of dye was calculated for each dye solution. Percentage removal of dye increased continuously with increase in dye concentration up to 50 ppm (where maximum dye removal, i.e., 82%, takes place), after which it started decreasing. The initial rise in percentage removal was because with increase in dye concentration (keeping other adsorption parameters constant) dye molecules get easily adsorbed on adsorption sites available on the adsorbent exterior while at higher dye concentration, all active sites are fully occupied by specified number of dye molecules (and further increase in dye concentration has negative effect on dye adsorption) besides repulsion between dye molecules and is responsible for decrease in percentage removal of dye (Kant *et al.* 2014; Nargawe *et al.* 2018). Results of dye concentration on dye removal are given in table S4 and the graph is shown below in Figure 10.

5.8. Effect of temperature

The effect of temperature variation from 10 °C to 60 °C (while keeping all other operating parameters constant) was studied to investigate the best solution temperature where maximum adsorption of crystal violet dye takes place on coconut husk. For this, a solution of 50 ppm crystal violet dye of pH 12 was prepared in distilled water, the initial concentration of dye solution was determined by UV-Vis spectrophotometer at 590 nm, and 0.6 g of coconut husk adsorbent was added, shaken well, and kept for contact time of 60 minutes at variable temperature, i.e., from 10 to 60 °C. After this, the solution was centrifuged at 3,000 rpm speed for 3 minutes, and the final concentration of dye in supernatant solution was determined via UV-Vis spectrophotometer at 590 nm. Using Equation (1) percentage removal of dye was calculated for each dye solution. Reduction in adsorption percentage with rise in temperature indicates that maximal adsorption occurs at lower temperatures. Maximum removal of dye, i.e., 81%, takes place at 10 °C, after which it starts to decrease continuously, which might be owing to increase in kinetic energy of adsorbed molecule that desorbed at elevated temperature as at elevated temperature dye tends to be

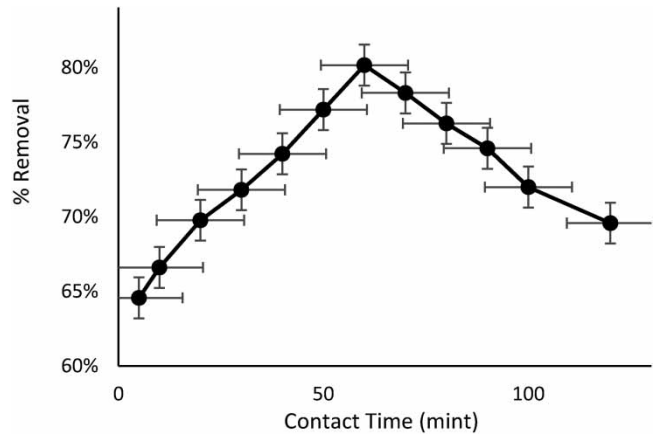


Figure 9 | Effect of adsorption time on % removal (experimental conditions: pH = 12; C_o = 10 ppm; adsorbent dose = 0.6 g; contact time = variable; temperature = 25 °C).

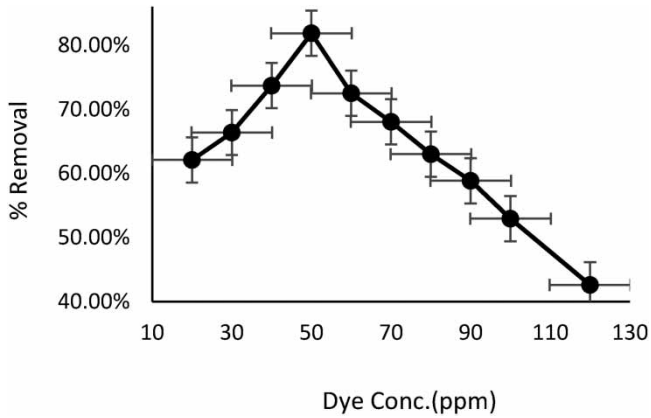


Figure 10 | Effect of dye concentration on % removal (experimental conditions: pH = 12; C_o = variable; adsorbent dose = 0.6 g; contact time = 60; temperature = 25 °C).

soluble in solvent than to adsorb on adsorbent surface, i.e., adsorbate-solvent interactions overcome adsorbate-adsorbent interactions leading to decrease in percentage removal of dye. Hence, the process was found to be exothermic in nature. Similar results were reported by Ho *et al.* 2005; Chandra *et al.* 2007; Alshabanat *et al.* 2013; Shoukat *et al.* 2017; Cheruiyot *et al.* 2019. The results of temperature on dye removal are given in table S5 and graph is shown below in Figure 11.

5.9. Kinetic study of adsorption

Study of reaction kinetics is crucial for determining optimal adsorption exposure duration besides mechanism of adsorption. To investigate adsorption mechanism, several kinetic models have been used including pseudo-first order, pseudo-second order, and intra-particle diffusion kinetic models. For rate determining stages, pseudo-first order and pseudo-second order kinetic models are useful, whereas the intra-particle diffusion model is beneficial for studying the kinetic mechanism of adsorption (Tariq *et al.* 2017).

5.9.1. Pseudo-first order kinetic model (Lagergren model)

The pseudo-first order kinetic model is given as:

$$\frac{d_{q_t}}{d_t} = K_f(q_e - q_t) \tag{3}$$

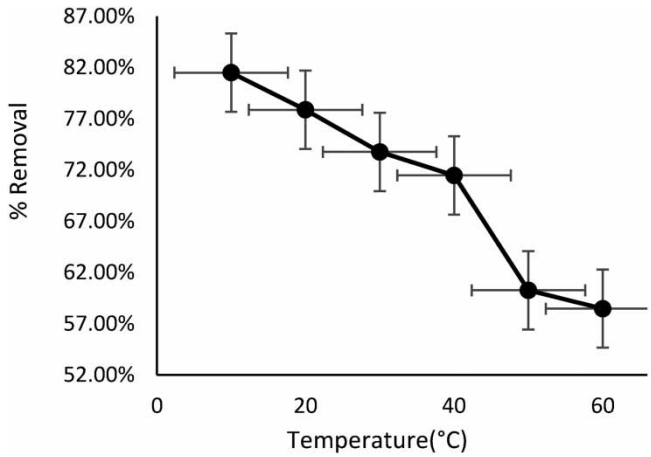


Figure 11 | Effect of solution temperature on % removal (experimental conditions: pH = 12; C_o = 50 ppm; adsorbent dose = 0.6 g; contact time = 60 min; temperature = variable).

where: q_t ($mg\ g^{-1}$) = amount of adsorbate adsorbed on adsorbent surface at time t, q_e ($mg\ g^{-1}$) = amount of adsorbate adsorbed on adsorbent surface at equilibrium time, K_f = pseudo-first order constant. The linear form of the pseudo-first order reaction is given below as:

$$ln\ (q_e - q_t) = ln\ q_e - K_f t \tag{4}$$

By ploting a graph of $ln\ (q_e - q_t)$ against time t (min), a straight line was obtained (Mashkoor *et al.* 2018). R^2 value was 0.9958, but although that is close to unity, the data does not fit the pseudo-first order kinetic model as on comparing experimental $q_e = 0.169$ mg/g with calculated $q_e = 1{,}910.73$ mg/g, a large difference between these values was observed, which can be explained in a way that although the plot is somewhat linear, but linearity of plot does not certainly guarantee the first order mechanism due to intrinsic disadvantage of accurately estimating adsorption capacity q_e (Kaur *et al.* 2013). Owing to this reason, the pseudo-first order kinetic model does not fit the experimental data. Results of the experiment and its graph is given in table S6 and Figure 12.

5.9.2. Pseudo second-order kinetics

Ho and McKay presented this model. According to the pseudo-second order kinetic model, rate of adsorption is directly proportional to number of adsorption sites. The mathematical equation is written as:

$$\frac{d_{q_t}}{d_t} = K_s(q_e - q_t)^2 \tag{5}$$

The linear form for the pseudo-second order kinetic model is given as follows:

$$\frac{t}{q_t} = \frac{1}{K_s q_e^2} + \frac{t}{q_e} \tag{6}$$

A plot of $\frac{t}{q_t}$ ($\min g mg^{-1}$) versus t (min) gives a straight line graph from which slope and intercept can be calculated (Hayat 2017). When experimental $q_e = 0.169$ mg/g was compared with calculated $q_e = 0.1590$ mg/g, we found that there was not as much difference between these two values. Additionally, R^2 value for pseudo-second order kinetic model was found to be 0.9902 (although less than that for the pseudo-first order kinetic model but still close to 1). From these observations, it was found that the current adsorption practice data fits best to the pseudo-second order kinetic model. Sultana *et al.*'s (2022) mechanism for this model proposes that sorption of crystal violet dye on coconut husk powder takes place due to valency forces (due to sharing or exchanging of electrons between adsorbent and adsorbate) might be significant (Gong *et al.* 2008; Aljeboree *et al.* 2015). A similar trend was observed with various adsorbents (Patel & Vashi 2010; Singh *et al.*

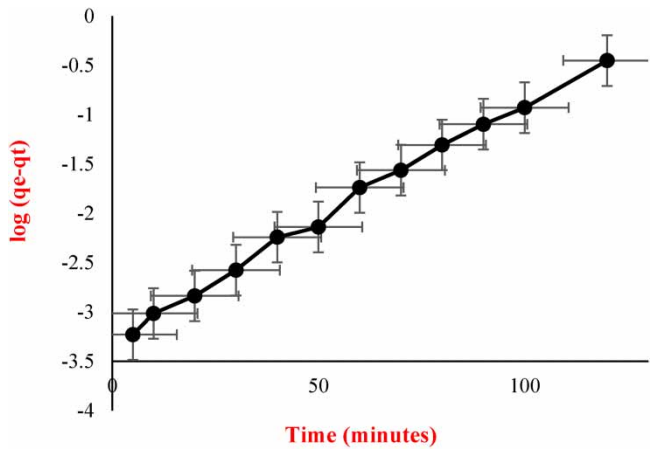


Figure 12 | Graph for pseudo-first order kinetic model.

2018; Batool *et al.* 2021). The pseudo-second order model has benefit that adsorption at equilibrium and initial adsorption rate can be calculated from itself, with no need of getting equilibrium sorption capacity from trials (López-Luna *et al.* 2019). Results of experiment and its graph given in table S7 and Figure 13.

5.9.3. Intra-particle diffusion model

Weber and Morris proposed this model for determining the diffusion mechanism and rate controlling step in the kinetics of adsorption. The mathematical form of this model can be represented as:

$$q_t = K_{id}t^{0.5} + I \tag{7}$$

where: q_t(mg/g) = quantity of adsorbate adsorbed at time 't', I = thickness of layer, and K_{id} $(mg\ g^{-1}min^{-1/2})$ = intra-particle diffusion constant (Batool *et al.* 2021).

The plot obtained by the data is not linear. It means that the intra-particle diffusion model alone is not the rate limiting step, but other kinetic models are also involved in control of the adsorption speed. Results revealed that crystal violet adsorption on coconut husk is fast in early stages but slowed down in later stages. This is because fast crystal violet dye retention might be due to boundary layer adsorption (adsorption of crystal violet dye molecules from solution to the surface of coconut husk (adsorbent), i.e., on surface of adsorbent), whereas delayed adsorption could be due to intra-particle diffusion process. Adsorption of crystal violet dye molecules on coconut husk adsorbent involve three steps. Firstly, adsorption of dye molecules on the surface of the adsorbent (boundary layer adsorption), which is a fast step. Secondly, transport of dye molecules to inner active sites from the adsorbent surface (intra-particle diffusion), which is a slow step, and lastly, interaction of dye molecules with active sites of the coconut husk adsorbent (Sultana *et al.* 2022). Regardless, a lower coefficient value of regression coefficient, i.e., 0.3126, suggest that this model was not sufficient to justify a mechanism of adsorption; rather, other kinetic models are also involved. Similar results were reported by (Abbas *et al.* 2021; Batool *et al.* 2021). Comparison of kinetic models is given in Table 1. Results of experiment and its graph given in table S8 and Figure 14.

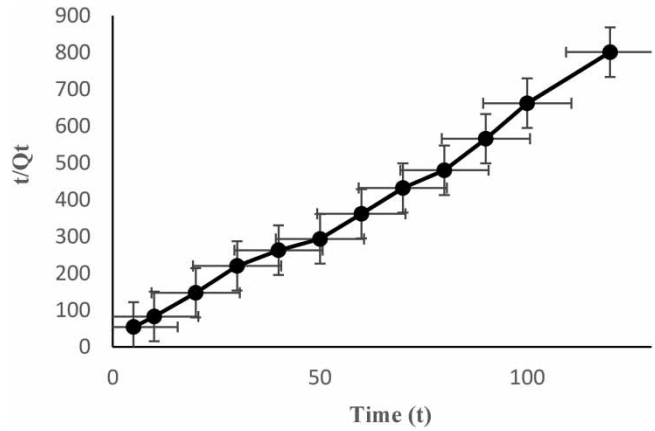


Figure 13 | Graph for pseudo-second order kinetic model.

Table 1 | Comparison of kinetic parameters of three models (pseudo-first order, pseudo-second order, and intraparticle diffusion model)

Pseudo-first order kinetic model	Pseudo-second order kinetic model	Intra-particle diffusion kinetic model
Adsorbate conc. $(mgL^{-1}) = 50$	Adsorbate conc. $(mgL^{-1}) = 50$	Adsorbate conc. $(mgL^{-1}) = 50$
experimental $q_e = 0.169$ mg/g	experimental $q_e = 0.169$ mg/g	experimental $q_e = 0.169$ mg/g
Calculated $q_e = 1910.73$ mg/g	Calculated $q_e = 0.1590$ mg/g	Calculated $q_e = 0.1126$ mg/g
R^2 = 0.9958	R^2 = 0.9902	R^2 = 0.3126
$K_1(min^{-1})$ = 0.0555	$K_2(min^{-1})$ = 4.001	K_{id} = 0.0039

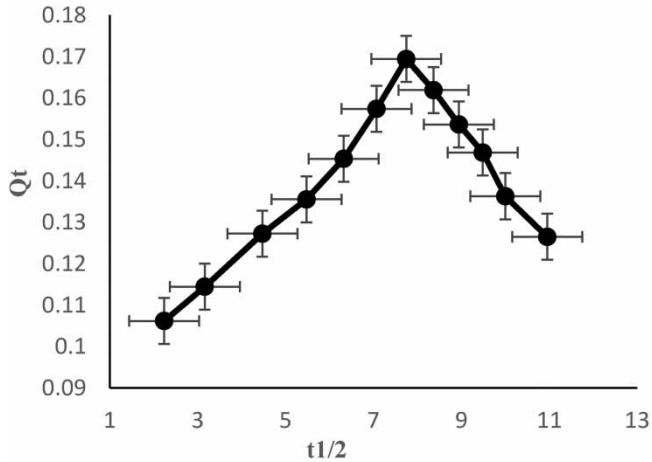


Figure 14 | Graph for intra-particle diffusion kinetic model.

5.10. Adsorption isotherm studies

Different isotherm models, such as the Langmuir, Freundlich, and D-R isotherm models, were used to evaluate data from concentration experiment of crystal violet dye adsorption at the coconut husk surface. These isotherms provide insight into the relationship between dye concentration in solution and adsorbed amount of dye on the adsorbent exterior (Yagub *et al.* 2014).

5.10.1. Langmuir adsorption isotherm model

This isotherm deals with interaction of adsorbate on adsorbent exterior without interacting with each other. It deals with monolayer adsorption. The linear form of the Langmuir model is as follows (Wathukarage *et al.* 2019):

$$\frac{1}{q_e} = \frac{1}{q_{max}} + \left(\frac{1}{q_{max} K_L}\right) \frac{1}{C_e} \tag{8}$$

K_L = Langmuir constant ($dm^{-3} mol^{-1}$) and q_{max} = monolayer adsorption capacity ($mg\ g^{-1}$). This equation can be simply written as (Handayani *et al.* 2018):

$$\frac{C_e}{q_e} = \frac{1}{q_{max} K_L} + \left(\frac{C_e}{q_{max}}\right) \tag{9}$$

In general, q_{max} and K_L are functions of pH, ionic medium, and adsorbate ionic strength. When we plot a graph between C_e ($mg\ L^{-1}$)/q_e ($mg\ g^{-1}$) and C_e ($mg\ L^{-1}$), a straight line will be obtained (Hayat 2017). Results of experiments are given in Figure 15. The R^2 value of this model was found to be 0.6791 (not so close to unity) showing that data did not fit best to Langmuir isotherm model. R_L, i.e., the separation factor parameter, was employed to check whether the process is favorable or unfavorable (given in table S9), i.e., the process is favorable only when $0 < R_L < 1$ while it is unfavorable when $R_L > 1$ and linear for $R_L = 1$ besides irreversible for $R_L = 0$. The formula for R_L is as follows:

$$R_L = \frac{1}{1 + \mathrm{b} C_{In}} \tag{10}$$

where: b = Langmuir adsorption equilibrium constant ($Lmol^{-1}$), C_{In} = initial dye concentration ($Lmol^{-1}$). R_L can determined from b and C_{In} (Sharma & Kaur 2018) as given in table S9. Results showed that the process of adsorption is of a linear nature (confirmed by $R_L = 1$). Experimental results of Langmuir adsorption isotherm are given in table S10.

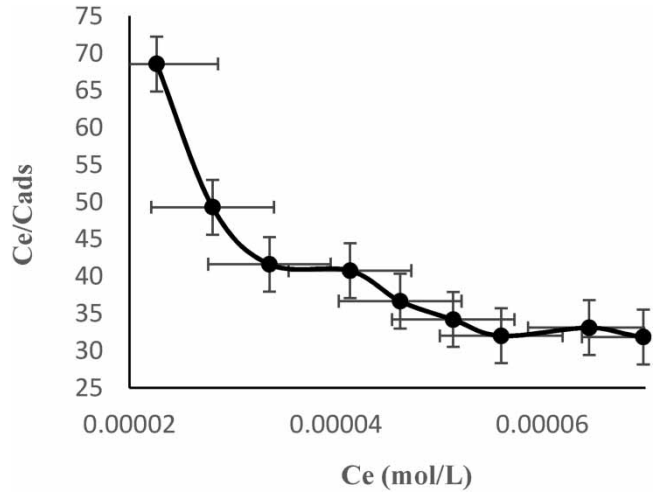


Figure 15 | Graph for Langmuir isotherm.

5.10.2. Freundlich adsorption isotherm

H.F. Freundlich proposed the adsorption expression as follows:

$$C_{ads} = K_f C^{1/n} \tag{11}$$

Integration of this equation gives the expression as:

$$log\ C_{ads} = log\ K_f + 1/n\ (log\ C_e) \tag{12}$$

Here: C_e denotes dye concentration at equilibrium (mol/ L) while C_{ads} denotes concentration adsorbed per unit mass of adsorbent (mol/g). The freundlich constants K_f and n represent adsorption capacity and strength, respectively. When value of n exceeds unity, adsorption proceeds to a higher extent. The value of n ranges from 1 to 10 (Chinniagounder *et al.* 2011; Nasar & Shakoor 2018). The value of n signifies the heterogeneous surface of adsorbent used (Tariq *et al.* 2017). In current procedure value of n was 1.163, which is greater than 1 and obeying the condition of the Freundlich isotherm, i.e., $0 < n < 1$ (Bansal *et al.* 2008; Nameni *et al.* 2008; Gholizadeh *et al.* 2013; Raval & Priti 2015), indicating that cooperative adsorption (Sultana *et al.* 2022) of crystal violet dye on coconut husk takes place to a moderate level. A value of n in range of 2–10 indicate good adsorption, and from 1 to 2 represents moderate adsorption while value of n less than 1 poor adsorption characteristic (Treybal 1980). For solution, Equation (10) can be written as:

$$ln\ q_e = K_F + \frac{1}{n} \ln C_e \tag{13}$$

where: K_F = Freundlich constant (determined from intercept of graph), n = slope, q_e $(mg\ g^{-1})$ = amount of adsorbate adsorbed per gram of an adsorbent.

This adsorption isotherm involves the use of multilayer adsorption and allow for unlimited amounts of material to be adsorbed. R^2 value for the Freundlich isotherm model is 0.9837, which is very close to 1. Experimental data for Freundlich isotherm model and its graph are given in table S11 and Figure 16.

5.10.3. Dubinin–Radushkevich isotherm model

This model is applied to calculate energy of adsorption and is proposed by Dubinin and Radushkevich. The mathematical expression of this model is given below:

$$C_{ads} = C_m\ exp(-\beta\varepsilon^2) \tag{14}$$

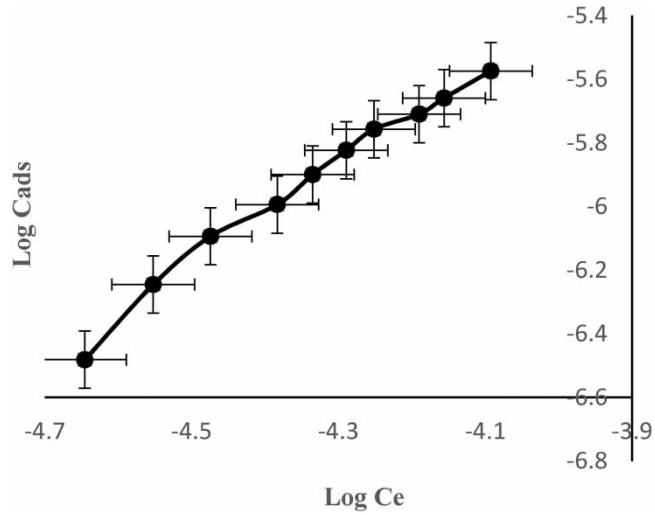


Figure 16 | Graph for Freundlich isotherm.

Here C_{ads} = crystal violet concentration adsorbing on coconut husk, C_m (mol/g) = maximum adsorption of crystal violet on coconut husk and ε = constant known as Polanyi potential.

$$\varepsilon = RT\, ln(1 + 1/C_e) \tag{15}$$

Integration of the D-R isothermal equation gives the following equation:

$$ln\, C_{ads} = ln\, C_m - \beta\varepsilon^{-2} \tag{16}$$

Value of β can be used to calculate the mean energy of the adsorption process (E_s)

$$E_s = 1/(-2\beta)^{1/2} \tag{17}$$

Here E_s denotes the mean free energy transfer from dye to adsorbent surface. Sorption energy (E_s) was calculated using the value of β, and the result was 0.40879731 kJ/mol indicating the process to be physical in nature. Stronger the connection between adsorbate and adsorbent, greater the value of free sorption energy. For the D-R isothermal model, R^2 value was 0.9874. Experimental data and graph for the D-R isotherm are given in table S12 and Figure 17.

When we compare findings of isotherm models (given in Table 2), then it is found that the experimental data fits best to Freundlich adsorption isotherm model and not to the Langmuir and D-R isotherm models (this is because the R^2 value of Freundlich isotherm model is very close to unity as compared to other isotherm models). The obtained adsorption isotherm showed that the current process of adsorption of crystal violet dye on coconut husk powder is favorable (Sultana *et al.* 2022) with physical adsorption dominating over chemical adsorption (López-Luna *et al.* 2019). The value of 1/n defines adsorptive removal efficiency of the selected adsorbent over different dye concentration range. Adsorbents can adsorb dye molecules of high concentration if the value of 1/n is greater than unity. While if the value of $1/n < 1$ then it suggests the applicability of adsorbent for entire range of dye solution (Kaith *et al.* 2016; Arora *et al.* 2019). Evaluation of adsorption capacities of various adsorbents is given in Table 3, showing that coconut husk is an effective adsorbent for crystal violet dye sequestration from wastewater as its q_o value is higher as compared to other adsorbents.

5.11. Thermodynamics of adsorption

Some state functions, such as change in Gibbs free energy (ΔG), entropy (ΔS), as well as enthalpy (ΔH), can be employed to determine heat changes in a system or state of a system. The nature of the adsorption process was revealed via these characteristics, which showed whether the process is of exothermic or endothermic nature. All these thermodynamic characteristics

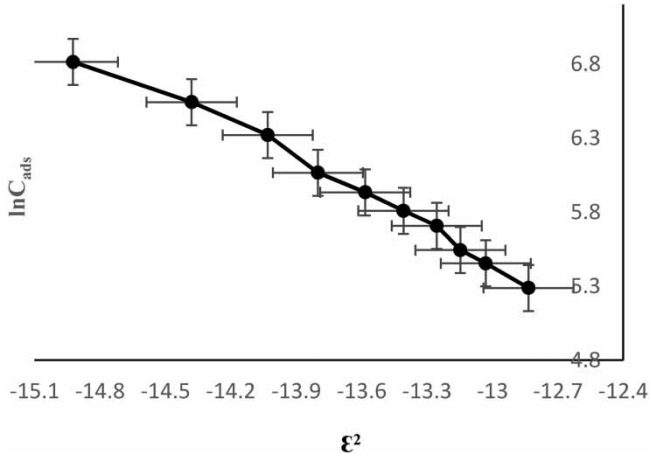


Figure 17 | Graph for Dubinin–Radushkevich isotherm.

Table 2 | Comparison of isotherm model parameters (Langmuir, Freundlich, and Dubinin–Radushkevich models)

Langmuir isotherm model	Temp. (°C): 25	Q_o (mgg^{-1}) = 0.7283	b (dm^3mol^{-1}) = 4.4496×10^{-4}	R^2 = 0.6791
Freundlich isotherm model	Temp. (°C): 25	K_f (mgg^{-1}) = 8.344	n (Lg^{-1}) = 1.163	R^2 = 0.9837
Dubinin–Radushkevich isotherm model	Temp. (°C): 25	β (KJ^2mol^{-2}) = 0.748	$\varepsilon^2(KJmol^{-1})$ = 0.408	R^2 = 0.9874

Table 3 | Comparison of adsorption capacities of various adsorbents for crystal violet dye exclusion

Adsorbent	Adsorption capacity (mg/g)	Reference
Coconut husk	0.7283	Present study
Cedrus deodara sawdust	0.673	Batool *et al.* (2021)
Groundnut shell	0.524	Akinola & Umar (2015)
Date palm fiber	0.66	Alshabanat *et al.* (2013)
Diplazium esculentum	351	Lim *et al.* (2020)
Raw cassava peels powder	–5.15	Okorocha *et al.* (2019)

may be calculated simply by the following equation:

$$lnK_c = -\frac{\Delta H}{RT} + \frac{\Delta S}{R} \tag{18}$$

where, ΔS = entropy, ΔH = enthalpy or total heat content of system, T = temperature, and K_c = equilibrium constant.

$$K_c = C_{ads}/C_{eq.} \tag{19}$$

where K_c, C_{ads} & $C_{eq.}$ = equilibrium constant, amount of dye adsorbed on adsorbent (mol/L) at equilibrium, and equilibrium concentration of dye left in solution (mol/L). If adsorption is an exothermic process, then there will be negative values for the Gibbs free energy change. The relation between Gibbs free energy change, entropy, and enthalpy is given as follows:

$$\Delta G = \Delta H - T\Delta S \tag{20}$$

The Van't Hoff plot is given in Figure 18. Investigational data for thermodynamic parameters is given in table S13. The negative ΔG value confirmed the process to be spontaneous (Quansah *et al.* 2020) in nature and randomness of process increased as confirmed by positive ΔS (Hong *et al.* 2009; Ertaş *et al.* 2010; Nnaemeka *et al.* 2016) value as given in Table 4.

5.12. Applicability of the developed procedure to a real system

Applicability of the developed procedure was checked with tap water (Tariq *et al.* 2017; Batool *et al.* 2021) (having numerous cations as well as anions such as Cl^{-1}, Na^{+}, K^{+} etc.) at optimized conditions (pH = 12; C_o = 50 ppm; adsorbent dose = 0.6 g; contact time = 60 min; temperature = 10 °C) to investigate the potential of coconut husk as an adsorbent for crystal violet dye exclusion from wastewater. For this, 50 ppm crystal violet dye solution having pH value of 12 was prepared in tap water instead of using distilled water, and 0.6 g of adsorbent was added into the solution. The solution was shaken well for 5 minutes, keeping the solution for 60 minutes (optimum contact time) at 10 °C temperature. After that, percentage removal of dye was calculated using Equation (1). Investigation showed that maximum adsorption, i.e., 79%, took place with coconut husk (results are shown in Table 5 and Figure 19), confirming the applicability of the developed practice to real samples.

5.13. Adsorbent recovery

Regeneration of adsorbent is an important parameter in determining the efficiency and effectivity of the adsorbent (Nidheesh *et al.* 2012; Zango & Imam 2018). Nitric acid solution (1 M HNO_3) i.e., acidic medium was used for desorption of crystal violet dye. For this, 0.4 g of dye loaded coconut husk (adsorbent) was shaken with 1 molar nitric acid solution (used in this case as a desorbing agent) for 60 minutes to recover both the adsorbent and the adsorbate, which resulted in 80% of

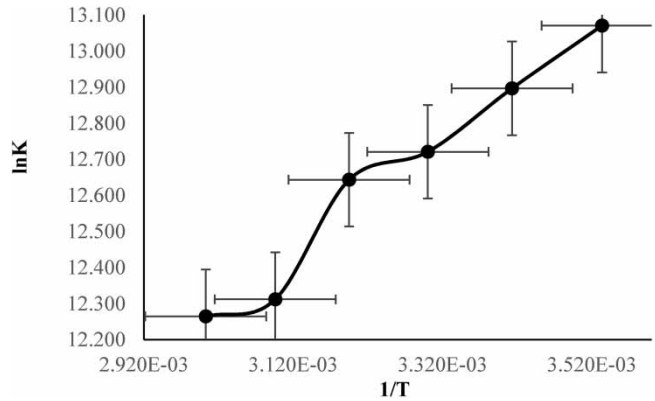


Figure 18 | Van't Hoff plot (for thermodynamic parameter calculations).

Table 4 | Calculated thermodynamic parameters

Temperature (K)	ΔG (KJ/mol)	ΔH (KJ/mol)	H-G	ΔS (J/molK)
283	−30.087	12.164	42.251	0.149
293	−30.735	12.164	42.899	0.146
303	−31.351	12.164	43.515	0.144
313	−32.188	12.164	44.352	0.142
323	−32.346	12.164	44.510	0.138
333	−33.219	12.164	45.383	0.136

Table 5 | Results of developed procedure with tap water

Adsorbent	Coconut husk
Adsorbate	Crystal violet
Water used	Tap water instead of distilled water
Percentage removal	79%

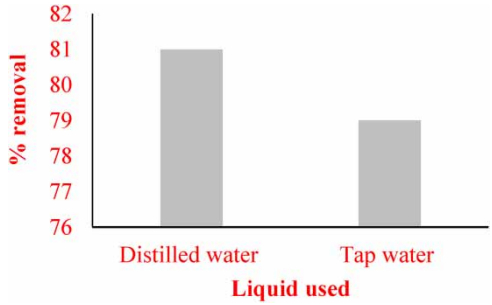


Figure 19 | Percentage removal of crystal violet dye in tap water and distilled water.

desorption (results of experiment are given in S14). Nevertheless, the removal efficacy of the adsorbent decreased after regeneration, but it can be reused for treatment of wastewater (Sun *et al.* 2015; Abbaz 2017; Tariq *et al.* 2017; Bagotia *et al.* 2021; Batool *et al.* 2021; Sultana *et al.* 2022).

6. CONCLUSIONS

1. This study explores the potential of coconut husk as a low-cost, economical adsorbent for sequestration of toxic crystal violet colorant from wastewater via adsorption practice. Adsorption parameters such as adsorbent size, pH, adsorbent dose, contact time, adsorbate dose, and temperature were studied and optimized, and 81% of the dye was removed at pH 12 having dye concentration of 50 ppm using adsorbent dose 0.6 g with 60 min contact time at 10 °C.
2. FTIR and SEM techniques were used to characterize the adsorbent. FTIR research revealed that functional groups such as −OMe, −COC−, and hydroxyl groups participate to a great extent in crystal violet dye adsorption onto the exterior of the coconut husk. SEM study revealed a rough, varied, and porous adsorbent exterior.
3. Isotherms, kinetic equations, and thermodynamic parameters have been investigated. Results showed that the pseudo-second order kinetic model and Freundlich isotherm model best described adsorption of crystal violet. It was found that the current adsorption process was spontaneous, endothermic in nature, with continuous decrease in entropy.
4. The established practice is 79% applicable with tap water and in acidic medium nearly 80% of adsorbent was recovered.
5. To investigate the proficiency of coconut husk for crystal violet dye sequestration, it is recommended to use actual dye rather than model dye.
6. Coconut husk has a high removal capacity for crystal violet dye exclusion from raw water, resulting in water treatment and protection of natural resources such as water, air, and land.

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CONFLICTS OF INTEREST

Authors declare no conflicts of interests to disclose this paper.

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AVAILABILITY OF DATA AND MATERIAL (DATA TRANSPARENCY)

The authors confirm that the data supporting the findings of this study are available within the article and/or its supplementary materials.

AUTHORS' CONTRIBUTIONS

All authors contribute equally to manuscript.

ETHICS APPROVAL

Not applicable.

CONSENT TO PARTICIPATE

Not applicable.

CONSENT FOR PUBLICATION

Not applicable.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

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