



Comparative Study of Congo Red Dye Removal Using Sunflower Husk and Spent Tea Leaves

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Abstract

This study investigates the utilization of spent black tea leaves and sunflower husks as adsorbents for the removal of Congo red dye from wastewater. Researchers examined numerous factors to determine their impact on removal efficiency. These factors were taken into account: the pH of the solution, the concentration of Congo red (10-50 ppm), the quantity of adsorbent (0.5g/l-4 g/l), and the particle size (<300 μ m-1.18mm). As the concentration of Congo red and size of the adsorbent both raises, the experimental findings showed that the percentage removal decrease, while as adsorbent dose increase the removal percent increase. The studies also showed that waste tea leaves were less effective in removal than sunflower husk. The optimal conditions were 20 ppm dye concentration, 4 g/L adsorbent dosage, and particle size < 300 μ m, resulting in removal efficiencies of 84% (tea leaves) and 89% (sunflower husk) respectively. The kinetic analysis results showed that the pseudo-second-order kinetic model best describes the adsorption process. Also, looking at the equilibrium data showed that the Langmuir isotherm model was the best fit of the isotherm models that were tested.

Paper type: Research paper

Keywords: Adsorption, Dye pollution, Sun flower husk, Spent tea leaves, agricultural waste adsorbents,

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1. Introduction

Environmental pollution from organic and inorganic materials is escalating due to industrial expansion. This can significantly impact the environment and human health (Ammar et al., 2012). Chlorinated hydrocarbons, dyes, aromatic compounds, and phenolic compounds constitute a significant class of organic pollutants found in industrial wastewater, posing an environmental hazard (Saharan et al., 2012). The rapid accumulation, resistance to degradation, and high toxicity of organic pollutants have made them a major obstacle to environmental remediation efforts in recent years (Lan et al., 2021).

There are both natural and man-made pathways for heavy metals to reach aquatic ecosystems (Priti and Paul, 2016). Much oil ends up in water as pollution because various technological and management developments fall behind other, less ideal reasons, and industrial expansion leads to an increase in the amount of oil consumed (Yu et al., 2017). Dye pollution is a major problem in the textile industry and other sectors because even minute quantities (less than 1 part per million) are visible to the human eye. Dyeing is a common process in many different industries, such as those dealing with textiles, paper, leather, and plastics (Vierra et al., 2011). The most common types of harmful and carcinogenic pollutants are industrial dyes and their byproducts. Secondary they have negative impacts on ecosystems (Taylor et al., 2015). There are several methods for removing color and dye contaminants from industrial wastewater. These include reverse osmosis (Arif et al., 2020), chemical precipitation (Shindhal et al., 2021), ion exchange (Tan et al., 2000), solvent removal (Al-Bastaki, 2004), ozonation (Karcher et al., 2002), and adsorption (Lee et al., 2000). Each of these techniques has been

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somewhat successful in removing dye particles from water. However, most of these conventional methods are associated with high operating costs, difficult maintenance requirements, secondary sludge production, or low efficiency in removing dyes with complex molecular structures.

Adsorption has become one of the most effective and flexible methods for removing a wide range of organic and inorganic pollutants from wastewater and making it colorless. It works especially well on effluents with low levels of persistent pollutants that don't break down naturally or through chemical oxidation. There are many benefits to the adsorption process, such as how easy it is to design and use, how well it removes contaminants, and how it can be reused and regenerated. Also, adsorption can get rid of small amounts of contaminants that might still be in the water after other treatment steps, making the water of high quality. The overall effectiveness of the adsorption process, on the other hand, depends a lot on the adsorbent's physicochemical properties, such as its surface area, porosity, surface functional groups, and particle size distribution. Adsorbents with a large specific surface area and a lot of active sites usually have better adsorption capacity. Consequently, the advancement and application of cost-effective, eco-friendly, and efficient adsorbent materials, including agricultural byproducts, have garnered considerable attention in recent years as a sustainable alternative to traditional adsorbents such as activated carbon. (Sadegh et al., 2018). The adsorption method is flexible and easy to use, but the materials used to adsorb are often costly or can't be reused on a large scale. More and more, these applications need for an adsorbent material that is renewable, inexpensive, and convenient to obtain.

The utilization of renewable resources such as used tea leaves, agricultural residues, or microbial biomass, biosorption has emerged as a viable option for wastewater treatment. The cost-effectiveness of this environmentally friendly approach is evident, as it requires fewer chemicals and is more efficient at adsorbing a wide range of pollutants, such as dyes. This technology overcomes some of the challenges associated with older methods and may represent a viable means of water treatment in the future. Due to its abundance and green properties, used tea leaves (STL), a byproduct of tea consumption, have been considered a low-cost biosorption material for removing various compounds, including textile dyes (Lima et al., 2024). The readily available and inexpensive sunflower seed hulls have also been used as a color-removing adsorbent in Textile wastewater containing synthetic dyes with high molecular weights. These husks are often overlooked as agricultural waste; however, their porous lignocellulosic structure is rich in functional groups, such as hydroxyl and carboxyl groups, which explains their good capacity to absorb dye molecules. The dye concentrations in the textile wastewater under study ranged between 50 and 200 mg/L, which is the expected range in final effluents after dyeing and finishing processes. The effluent was purified using sunflower seed husks. The bio-adsorbent extracted from this biomass is considered an environmentally friendly method for purifying industrial wastewater rich in dyes, due to its excellent decolorization and removal capabilities (Suteu et al., 2011).

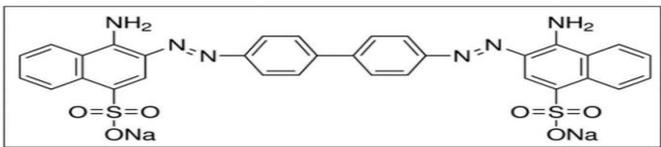
This study present purification and treatment of wastewater that contaminated with Congo red dye by using spent tea leaves and sunflower husk which considered as waste material for adsorption and examine the influence of initial dye concentration, particle diameter, adsorbent dose, and pH of the solution on the removal percent of dye. While both biosorbents have been individually studied, no published work has directly compared sunflower husk and spent tea leaves under the same experimental framework

2. Experimental

2.1 Materials and Method

The model adsorbate used in this investigation was Congo Red, which was acquired from Sigma-Aldrich. The physical attributes and structural details of these dyes are listed in **Table 1**. To remove the colors from the water, adsorbents such as sunflower husk and waste black tea were used. The waste that was collected was cleaned with water to remove any dirt, and then dried in a hot oven for five hours. For particle sizes ranging from 1.18 to 300 μm , the dry materials were ground and sieved. The adsorption process did not include any chemical or physical treatments. The acidity and basicity of the solution were adjusted using acetic acid and sodium hydroxide, respectively.

Table 1 The physical properties of Congo red

Properties	Value
Chemical Formula	C ₃₂ H ₂₂ N ₆ Na ₂ O ₆ S
Molecular-weight	696.66 g/mol
Melting point	360 C ⁰
pH	5
Solubility in water	25–30 g/L at 25C ⁰
Molecular structure	

2.2 Adsorption Process

Prepare stock solution of Congo red with concentration 500 ppm by dissolving 0.5 g powder of Congo red dye in 1000ml distilled water. In order to prepare other concentrations of dyes (10-50) ppm, taking amount of stock solution and diluted to require concentration. Adding the adsorbent (spent black tea leaves and sunflower husk) with different concentration (0.05, 0.1, 0.2, 0.3 and 0.4) g/L to flask of Congo red solution and put into shaker. Withdraw a sample at different period of time for 1hr and filtrate to separate the adsorbent from solution. UV-Vis spectrophotometer (Jenway model 6800) was used at $\lambda_{max} = 491$ nm then calculate the removal percent by applying the equation

$$\%Removal = \left(\frac{C_0 - C_t}{C_0} \right) * 100 \quad (1)$$

where (C_0) are the dye concentrations at the beginning and (C_t) are dye concentration after any time measured in mg/L

2.3 Adsorption Isotherms

Sorption is the process by which substances interact with solids. The degree of the contact between the adsorbate and the substrate determines the type of adsorption, which may be broadly categorized into chemisorption and physisorption.

The effectiveness of chemisorption interactions is two orders of magnitude higher than that of physisorption. A single layer of adsorbate forms on the adsorbent in chemisorption, whereas many layers of adsorbate develop on the adsorbent in physisorption. Physisorption demonstrates low enthalpy, takes place at temperatures beneath the boiling point of the adsorbate, and is characterized by its reversibility. Chemisorption exhibits a significant enthalpy, takes place across all temperature ranges, and is characterized by its irreversibility.

2.3.1 Models of two-parameter isotherm

Langmuir-isotherm Model

When adsorption happens at uniform and equivalent localized locations and the adsorbed layer consists of a single molecule, the Langmuir isotherm provides fundamental model that captures this process. Even at neighbouring locations, the adsorbed molecules must not interact laterally or experience steric hindrance. There should be no adsorbate movement within the surface plane, and all sites should show consistent affinity for the adsorbate. The fast decay of intermolecular attractive interactions is postulated by Langmuir

theory as a function of distance. The following equation represents the separation factor (R_L), which was defined by Weber as a dimensionless constant:

$$R_L = \frac{1}{1 + K_L C_0} \quad (2)$$

Here, C_0 denotes the starting concentration of the adsorbate in milligrams per liter, and K_L is the Langmuir constant (in milligrams per kilogram) as it relates to adsorption capacity. The correlation between the K_L constant and the variation in the adsorbent's appropriate area and porosity suggests that a greater surface area and pore volume may lead to an improved adsorption capacity. The R_L value of 1 indicates linear adsorption, $R_L = 0$ indicates irreversible adsorption, $R_L > 1$ indicates unfavourable adsorption, and $R_L < 1$ indicates favorable adsorption. The following modifications can be made to the Langmuir isotherm equation to account for aqueous-phase adsorption:

$$q = q_m \frac{K_L C}{1 + K_L C} \quad (3)$$

Freundlich-isotherm Model

Surface heterogeneity, active site energy distribution exponential, and Freundlich isotherm model expression are all illustrated. Heterogeneous systems, such organic molecule adsorption on molecular sieves or activated carbon, are presently seeing extensive use of the Freundlich-isotherm model. Equations (4) and (5) of the isotherm-model are linearized and non-linearized, respectively, with parameters found by comparing the logarithm of q_e and $\log C_e$; this straight-line yields with the slope represented by $1/n$ and the intercept by $\log (K_F)$.

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (4)$$

$$q_e = K_F C_e^{\frac{1}{n}} \quad (5)$$

An isotherm's type is determined by its n -value, where n and K_F are temperature-dependent variables. One divided by the natural number of adsorbate sites (n) gives the surface heterogeneity or adsorption strength, which is a measure of the energy distribution associated with these sites. When $1/n$ is positive (between 0 and 1), adsorption is beneficial; when $1/n$ is negative, it is unfavourable; and when $1/n$ equals 1, it is irreversible. An empirical equation is a feature of the Freundlich isotherm model. (Haghseresht and Lu, 1998)

2.4 Adsorption Kinetics

When run under varying experimental circumstances, kinetic models reveal the dynamic behaviour of sorption processes. Studies involving process optimization and scale-up benefit greatly from their usage. (Haghseresht and Lu, 1998)

2.4.1 Pseudo First Order model

$$q_t = q_e(1 - e^{-k_1 t}) \quad (6)$$

In this context, q_t and q_e (mg/g) represent the dye's sorption capacity at time t and at equilibrium, respectively, whereas k_1 (min^{-1}) denotes the absorbing rate constant of the pseudo first-order model.

2.4.2 Model of Pseudo Second Order

Pseudo-second-order kinetics frequently applies to situations in which the pace of the direct adsorption/desorption process dictates the overall sorption kinetics, typically denoted as:

$$q_t = k_2 q_e^2 \left(\frac{t}{1} \right) + k_2 q_e t \quad (7)$$

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

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (9)$$

The pseudo second-order rate constant is denoted by k_2 (g/mg·min) in this context. The experimental results were evaluated using the intra-particle diffusion model, which is explained below, as the kinetic models discussed earlier do not validate the diffusion process:

$$q_t = k_p t^{\frac{1}{2}} + C \quad (10)$$

In this context, k_p (mg/g min^{1/2}) represents the intra-particle diffusion rate constant, while C (mg/g) denotes a value that reflects the thickness of the boundary layer (Patino et al., 2021).

3. Results and Discussion

3.1. Effect of pH

In **Figure 1**, the effects of pH on the adsorption of Congo red were examined in a temperature-controlled environment using a 20 mg/L Congo red solution, an adsorbent dosage of 2 g/L, and a particle size of less than 300 μm.

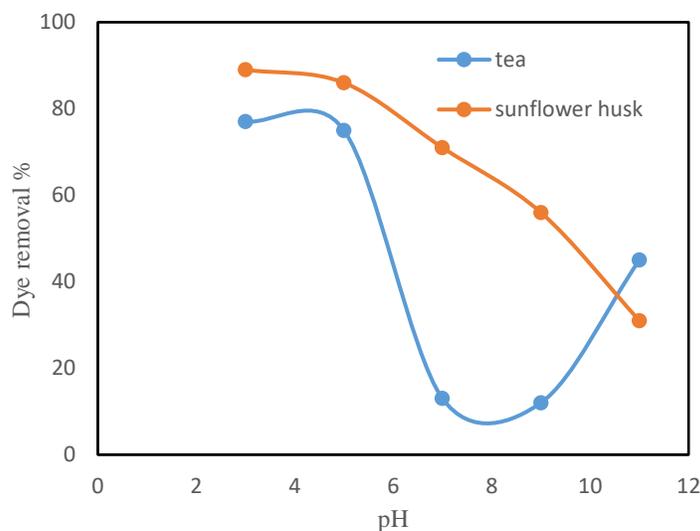


Fig. 1 Effect of PH on dye removal dye concentration 500ppm, particle size <300μm, Adsorbent dose 4g/L

It is evident from Fig. 1 that the sunflower husk adsorbent showed its highest adsorption effectiveness of 89% at pH 3, which remained relatively stable up to pH 5. However, when the pH increased from 5 to 7, a little decrease in adsorption efficiency was noticed. It is also showed the lowest removal rate 31% at pH 11. Acidic and neutral pH sites showed greater adsorption efficiencies than basic pH sites. The absorption of anionic dyes, such as Congo red, increases at lower pH levels due to increased dye ionization and the availability of active sites on the adsorbent at lower pH. Furthermore, the structural stability of it is affected by pH. At a pH of 3.0, it appears blue. Its color changes between 9 and 11, while it remains constant between 5 and 7. The adsorption effectiveness of spent tea leaves was highest at pH 3, and it remained relatively constant up to pH 5, when removal efficiency was 75%. However, when the pH increased from 5 to 7, a decline in adsorption efficiency was noted (13%). Raising the pH level to 11 further improved removal efficiency to 45%. Acidic and neutral pH sites showed greater adsorption efficiencies than basic pH sites. To conduct studies on neutral solution and avoid the use of harmful acidic chemicals, pH 5 was chosen as the operating pH. The Congo red adsorption on activated carbon and leftover orange peels has also been reported with similar results (Khan et al., 2018).

3.2 Dye removal as a function of particle size

In adsorption, one of the most important parameters is the size of the adsorbent particles. There were five different particle size ranges used in the adsorption process: 1.18 mm, 1 mm, 500 μm , 300 μm , and less than 300 μm that correspond to (ASTM NO.16, 18, 35 ,50 and 60) respectively. The other parameters, such as the pH level, the amount of adsorbent (2 g/L), and the dye concentration (20 ppm), were kept constant.

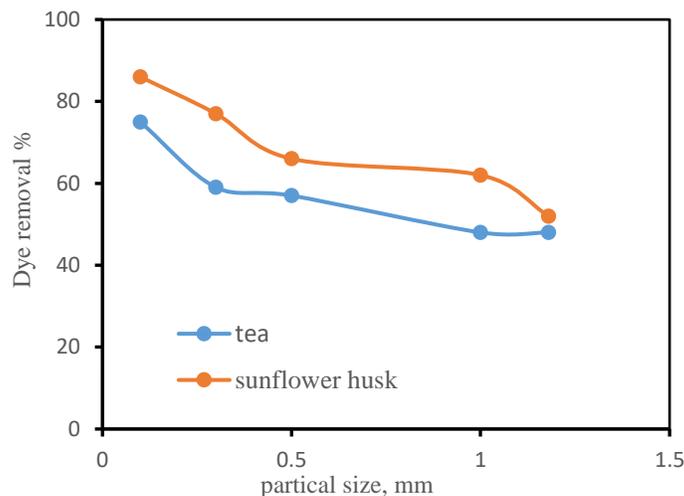


Fig. 2 Effect of particle size on dye removal. pH5, 20 ppm of dye, and 4 g/L of adsorbent.

Figure 2 shows that the removal rate of Congo red decreased from 70% to 48% when using used tea leaves, while for sunflower husks, the removal decreased from 80% to 52% as the particle size increased from smaller micrometers to 1.18 mm. The explanation is that smaller particles improve the adsorption system by increasing the surface area available for dye removal. These smaller adsorbent particles have shorter diffusion distances, which enhances their ability to penetrate the internal porous structures of the adsorbent material (Jaber and Jabbar, 2021).

3.3 Dye concentration's impact on elimination

This study examined the influence of adsorbate concentration (10–50 mg/L) on dye removal, utilizing a 2 g dosage of adsorbent at room temperature (25°C) and a pH of 5. **Figure 3** illustrates that an increase in dye concentration from 10 to 50 ppm results decrease in dye adsorption from 86% to 79%. The results indicated that removal efficiency declines as the concentration of the adsorbate increases. This happens because there is a larger ratio of surface-active sites at low adsorbate concentrations. As the concentration of adsorbate increases, these sites become saturated, which subsequently impacts removal efficiency (Patino et al., 2021).

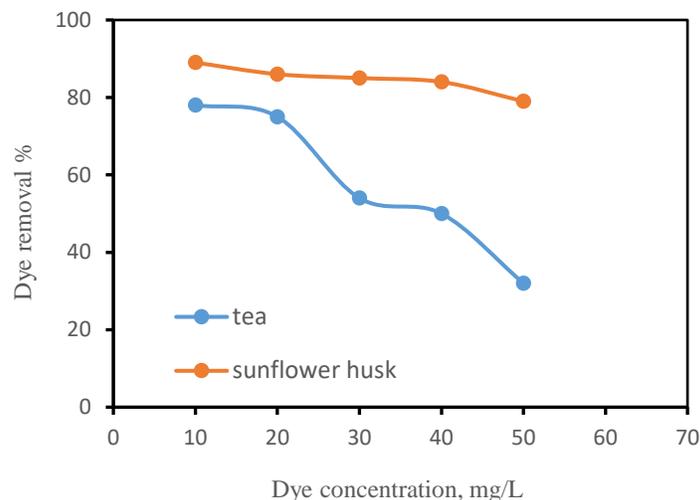


Fig. 3 Effect of dye concentration dye removal, particle size <300µm, pH5, Adsorbent dose 4g/L

3.4 Contact Time's Impact on Dye Removal

The time required to reach adsorption equilibrium for dye removal was investigated under controlled experimental conditions. The experiments were conducted at room temperature (25°C) with a dye concentration of 20 mg/L, an adsorbent dose of 2 g/L, and a pH of 5. The results showed that after approximately 60 minutes of contact time, both the spent black tea leaves and sunflower husks reached equilibrium with the dye solution. During this initial phase, there was rapid adsorption of dye molecules due to the abundance of active adsorption sites on the adsorbent surfaces, allowing the dye molecules to interact strongly with the adsorbent. As the adsorption process continued, the rate of dye uptake gradually decreased. This was due to the filling of the active adsorption sites. The remaining active sites and the repulsive forces between the adsorbent molecules and the free dye molecules began to take control. After the equilibrium point, increasing the contact time to 120 minutes did not result in any significant change in adsorption capacity. This indicates that the equilibrium conditions were firmly established. The spent black tea adsorbent removed 79% of the dye, and the sunflower husk adsorbent removed 88% of the dye, which was the best overall result. The marginally superior removal efficiency noted for sunflower husk may be attributed to its increased surface area, pore size, and the high density of functional groups that can interact with the dye molecules. These results show that both materials work effectively as low-cost bio sorbents for dye removal

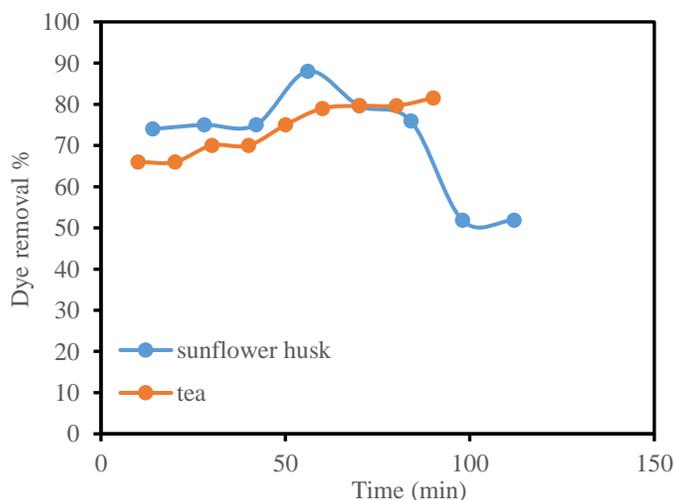


Fig. 4 Effect of time on dye removal, dye concentration 20ppm, adsorbent dose 2g/L, particle size <300µm, pH5

Figure 4 shows the relationship between contact time and percentage dye removal, which clearly shows the trends in adsorption. The presence of several active sites on the sorbent surface facilitates the fast adsorption of dye molecules upon contact to the aqueous dye solution, leading to the constant removal efficiency. At the point of equilibrium, the adsorbent surface is constantly balancing the

adsorption and desorption of dye molecules. The system is now considered to be in a steady state, which allows for continuous dye removal efficiency (Patino et al., 2021).

3.5 Effect of adsorbent dose

Figure 5, shows the effect of varying adsorbent dosages on dye removal was investigated by varying the adsorbent amount from 0.5 to 4 g/L for a Congo red concentration of 20 ppm during a 60-minute period when the biomass was increased from 0.5 to 3 g/L.

As shown in **Fig. 5** the removal efficiency for sunflower husk went up from 62% to 79% and for used tea leaves it went up from 70% to 87%. This is because there are more adsorption sites and more bio-sorbent, which makes it easier for the dye to reach the sorption sites. Further increases in adsorbent concentration to 3–4 g/L showed minimal changes in removal efficiency; therefore, raising adsorbent levels beyond 2 g/L is not economically viable (Khan et al., 2018).

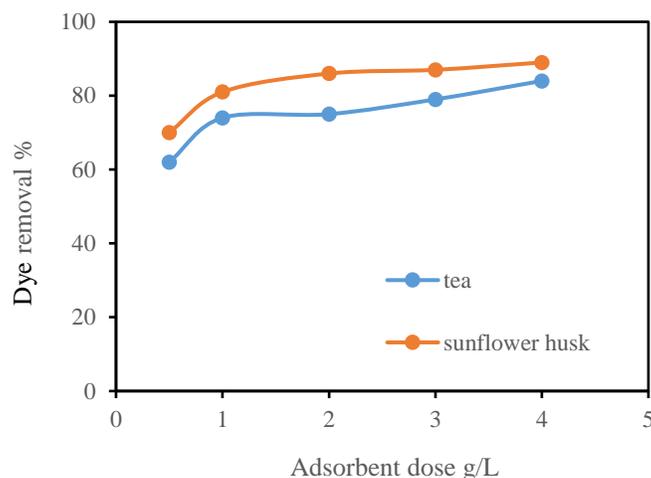


Fig. 5 Effect of adsorbent dose on dye removal, dye concentration 20ppm, particle size <300 μ m, pH5, Adsorbent dose 4g/L

3.6 Adsorption kinetic

Hypothetical first-order, second-order, and intra-particle diffusion kinetic models were all employed in this study. As shown graphically in **Figures 6 to 8**, kinetic models are applied to the following: $\ln qt$ vs t , (t/q) versus t , and qt versus $t^{1/2}$ respectively.

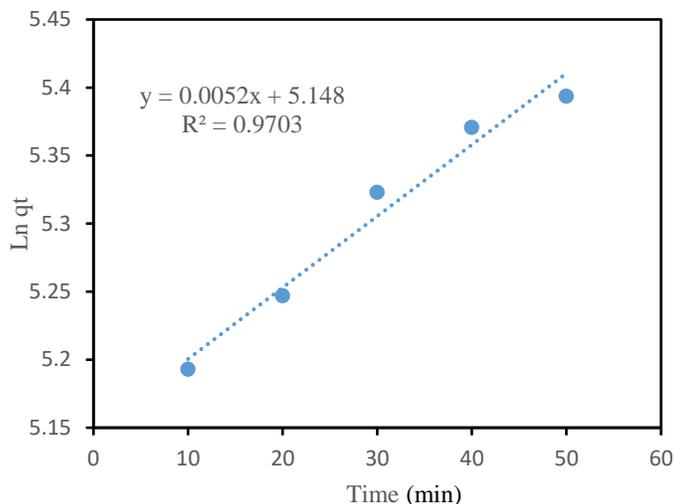


Fig. 6: linearization of Pseudo first order model

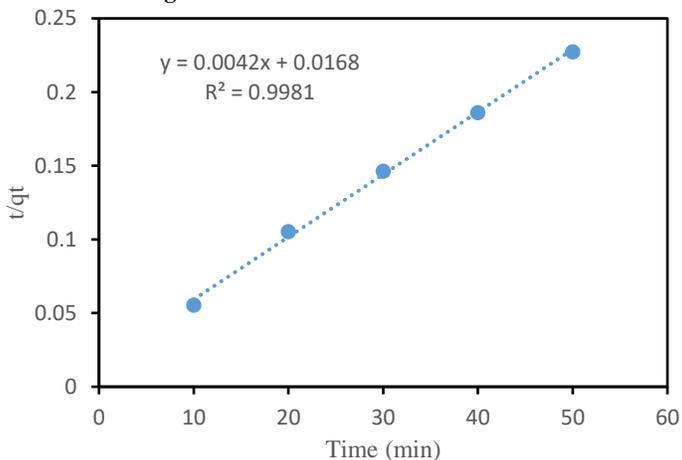


Fig. 7: linearization of Pseudo second order model

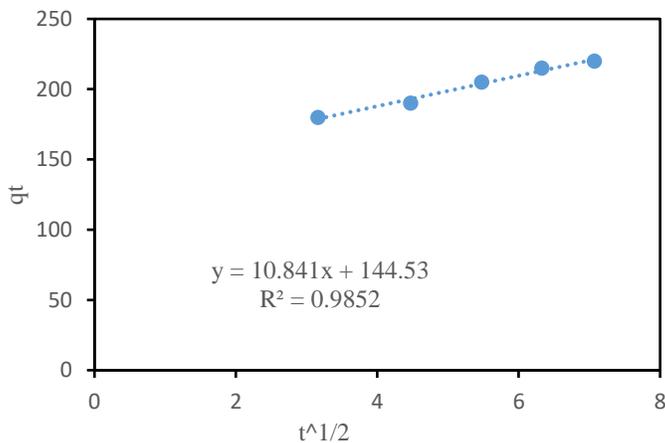


Fig. 8: linearization of intra diffusion model

An evaluation of the kinetic model's concordance was done by calculating the coefficient of determination (R^2). The role of the kinetics model is indicated by the greatest R^2 . All of the kinetic parameters are included in **Table 2**, and it was determined that the adsorption kinetic data was better characterized by the pseudo second order model.

Table 2. Adsorption kinetic parameters

Adsorption model	Parameters	Value
Pseudo 1 st order model	K_1 (min^{-1})	0.02592
	q_e (mg/g)	6.35982
	R^2	0.9703
Pseudo 2 st order model	K_1 (min^{-1})	0.783101
	q_e (mg/g)	8.718396
	R^2	0.9985
Diffusion model	K_p ($\text{mg/g min}^{1/2}$)	0.4116
	C	5.308
	R^2	0.9852

3.7 Adsorption isotherm

An analysis was conducted on the adsorption data of Congo red onto waste tea leaves and sunflower husk using Freundlich and Langmuir isotherms

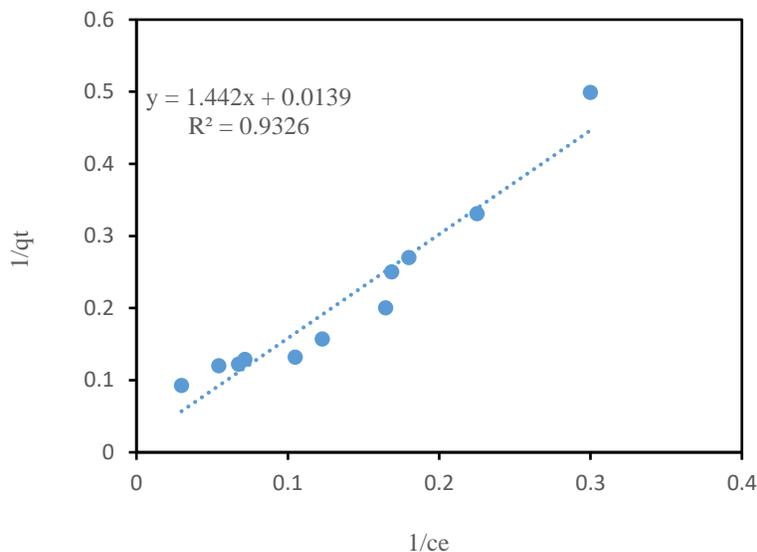


Fig. 9 linearization of Langmuir model

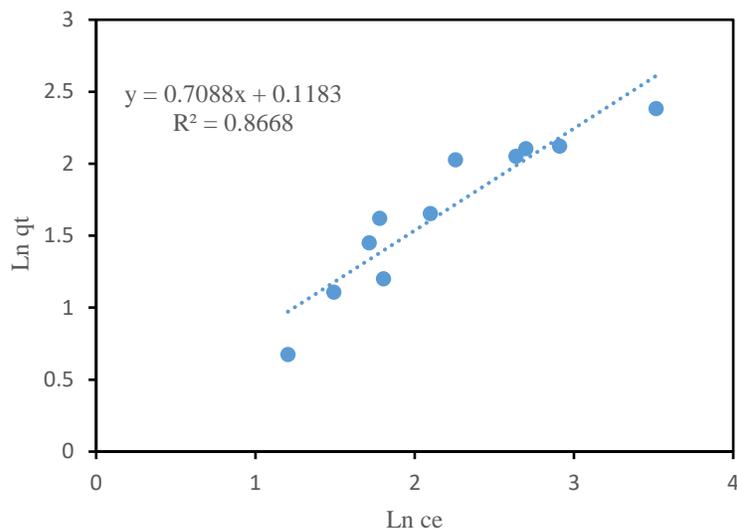


Fig. 10 linearization of Freundlich model

Table 3 shows the parameters of the Freundlich and Langmuir isotherms, as well as their correlation coefficients (R^2), which were found using linear regression techniques. **Figures 9-10** show the corresponding results. According to the results, the Langmuir isotherm model well describes the adsorption activity.

Table 3 Langmuir and Frenldich parameters

Isotherm model	Parameters	Value
Langmuir	q_m (mg/g)	0.69348
	k_L	103.74
	R^2	0.9326
Frendlich	K_f (mg/g)	1.313106
	N	1.410835
	R^2	0.8668

4. Conclusions

The adsorption process using spent tea leaves and sunflower husk ensured an excellent degree of color removal. However, sunflower husk shows more performance for color reduction. The optimal pH solution was that of pH=5 for the spent tea leaves, and pH=3 for the sunflower husk since they gave higher removal 75% and 89% respectively. The removal percentage increases with decreasing dye concentration and particle size, while it also increases with a greater amount of adsorbent. The maximum removal efficiency recorded in the experiments was 89%, attained with 0.4g of sunflower husk per 100ml of solution, a Congo red dye concentration of 10ppm, a particle size of less than 300 μ m, and a pH of 3. The model of pseudo-second order exhibited a superior fit compared to other kinetic models, while the equilibrium experimental data were characterized by the Langmuir isotherm model.

Future studies could explore other natural adsorbents and test them with different pollutants, such as heavy metals, oils, and pharmaceuticals. It would also be useful to examine how temperature and mixing speed affect the adsorption process to help optimize treatment conditions

CRedit Author Contribution Statement

Marwa F. Abdul Jabbar: Conceptualization, Supervision, Writing – Review & Editing, Project Administration, Funding Acquisition.

Yousif Amer Kadim: Investigation, Methodology, Data Curation, Writing – Original Draft.

Declaration Statements

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Conflicts of Interest

The authors declare no conflict of interest.

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Nomenclature

Symbol	Description	Unit
C	Concentration	mol/L
C _e	Equilibrium concentration	mg/L
C ₀	Initial concentration of phosphate ions	mg/L
C _t	Concentration of adsorbate in feed at any time	mg/L
K ₂	Model of Pseudo Second Order	g/mg.time
K _f	Freundlich isotherm constant	mgL ⁻¹ /ng ⁻¹ L ^{1/n}
K _L	Langmuir isotherm constant	L/mg
kp	represents the intra-particle diffusion rate constant	(mg/g min ^{1/2})
n	Freundlich intensity parameter	-

Symbol	Description	Unit
q_e	Concentration of adsorbate in solid phase at equilibrium	mg/g
q_m	The maximum amount of adsorbate per unit weight of adsorbent	mg/g
q_t	Concentration of adsorbate in solid phase at time t	mg/g
R_L	Separation factor	-
R^2	coefficient of determination	-

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