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Spent Tea Leaves as an Adsorbent Material: Preparation, Characterization, and Application

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Abstract:
The adsorption of Congo Red (CR) on the surface of Spent Tea Leaves (STL) was evaluated in an aqueous system. The study was carried out for the establishment of a standard wastewater treatment method that would remove reactive dyes by employing spent tea leaves as a bio-adsorbent. The effect of various parameters like amount of adsorbent, contact time, concentration of dye, and pH on the adsorption was studied. The adsorption capacity was monitored with a UV-visible spectrophotometer. The maximum adsorption of dye was observed at adsorbent mass of 3.5g /100 mL, at 8×10^{-5} M CR concentration, at pH = 9.72 and 50 minutes of contact time. The experimental results suggest that the adsorption of CR on the surface of STL is an exothermic process. The adsorption of CR on STL is found to follow pseudo first order kinetics. Based on the findings, it's feasible to draw the conclusion that spent tea leaves would be a better adsorbent for the removal of reactive dyes from the wastewater.

Keywords: *Adsorption, Bio-adsorbent, Congo Red, kinetics, Spent Tea Leaves.*

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I. Introduction

The textile industry significantly contributes to environmental pollution, particularly through the release of azo dyes into wastewater. If this wastewater is not properly treated, it can contaminate aquatic ecosystems [1]. Estimates indicate that around 50,000 tons of dyes are released into the environment annually [2]. Globally, textile production accounts for approximately 20% of water pollution, with regions like India and Bangladesh being particularly affected. These areas already face severe water stress, with over 75% of Southern and Central Asia experiencing high levels of water scarcity as of 2019. The discharge of untreated textile wastewater exacerbates the contamination of freshwater resources, further endangering local ecosystems and worsening existing social inequalities [3-5].

Among these dyes, Congo red, a synthetic azo dye, is notorious for its high toxicity and resistance to biodegradation, posing serious risks to aquatic ecosystems and human health. Congo red is an organic compound with the chemical formula $C_{32}H_{22}N_6Na_2O_6S_2$, characterized by two naphthalene rings connected by an azo group (-N=N-) and two sulfonic acid groups ($-SO_3H$). This structure contributes to its solubility in water and persistence in the environment. Traditional methods for dye removal, such as chemical treatments and adsorption using activated carbon, often involve high costs and environmental impacts. Consequently, there is an urgent need for sustainable and cost-effective alternatives for wastewater treatment [6,7].

Traditional wastewater treatment methods encompass a variety of physical, chemical, and biological processes designed to remove contaminants from wastewater. Traditional wastewater treatment methods include preliminary treatment (screening and grit removal), primary treatment (sedimentation and chemical precipitation), secondary treatment (activated sludge and trickling filters), and tertiary treatment (disinfection and nutrient removal). While these methods have been effective in treating wastewater, they face several limitations and challenges like high energy costs, harmful by-products, and inefficiency in removing certain pollutants, especially complex organic compounds and dyes, which can persist in the environment [8].

In contrast, bio adsorption has emerged as a promising alternative for wastewater treatment, utilizing natural materials like spent tea leaves, agricultural residues, or microbial biomass to remove pollutants. This eco-friendly approach boasts lower costs, reduced chemical use, and better efficiency in adsorbing various pollutants, including dyes. It addresses many limitations of conventional techniques, offering a promising solution for future water treatment needs [9-11]. Spent tea leaves (STL), a byproduct of tea consumption, have emerged as a promising low-cost bio adsorbent for the removal of various pollutants, including textile dyes, due to their abundant availability and eco-friendly properties. Chemically, spent tea leaves are rich in organic compounds, including proteins, carbohydrates, and phenolic compounds, which contribute to their binding properties. The presence of functional groups such as hydroxyl (-OH), carboxyl (-COOH), and amine (-NH_2) groups enhances their adsorption capacity by providing active sites for binding with dye molecules. These functional groups facilitate the electrostatic interactions and hydrogen bonding necessary for effective dye removal. Rich in organic compounds, STL possess a high surface area and functional groups that facilitate the adsorption process.

Additionally, Weng et al. (2013) found that spent green tea leaves effectively decolorized textile wastewater under optimal conditions, showcasing their practical application in real-world scenarios [12]. Furthermore, a study by Sudiana et al. (2022) investigated the adsorption kinetics and isotherms of reactive red dye using activated coconut leaf stalks, further illustrating the effectiveness of low-cost adsorbents in treating textile wastewater [13]. These studies collectively underscore the growing interest in utilizing innovative technologies for sustainable wastewater treatment. Adsorption is a process where molecules of the adsorbate, such as Congo red, adhere to the surface of an adsorbent, in this case, spent tea leaves. The mechanism of adsorption involves several steps: first, the dye molecules diffuse from the bulk solution to the adsorbent surface, overcoming a boundary layer of static liquid. Once at the surface, the dye molecules interact with the functional groups present on the adsorbent, such as hydroxyl (-OH), carboxyl (-COOH), and amine (-NH_2) groups. These interactions can be physical (physisorption) or chemical (chemisorption), depending on the nature of the binding forces. Physisorption typically involves weak van der Waals forces, while chemisorption involves the formation of stronger covalent bonds. The efficiency of the adsorption process is influenced by factors such as contact time, temperature, and the initial concentration of the dye. This mechanism highlights the potential of spent tea leaves as an effective bio adsorbent for removing toxic dyes from wastewater, addressing the limitations of traditional treatment methods [14-16].

This research aims to evaluate the efficacy of spent tea leaves as a bio adsorbent for the removal of Congo red, a highly toxic azo dye, from textile wastewater. By investigating the adsorption kinetics, isotherms, and optimal operating conditions, such as contact time, temperature, and initial dye concentration, this study seeks to elucidate the underlying mechanisms governing the dye removal process. The findings of this research are expected to contribute to the development of sustainable wastewater treatment methods that can be readily adopted by textile industries seeking environmentally friendly solutions for managing their dye-laden effluents. Ultimately, this study aims to provide valuable insights and practical recommendations for the effective removal of Congo red and other textile dyes using spent tea leaves, thereby mitigating the environmental and health risks associated with their discharge into water bodies.

II. Materials And Methods

Chemicals, Reagents, and Instruments

Spent tea leaves were collected from the local tea vendors of Barishal, Bangladesh. A textile dye Congo red was collected from a chemical supplier, all other chemicals and reagents used in the study, including hydrochloric acid (HCl) and sodium hydroxide (NaOH) for pH adjustment, were of analytical grade and used for the study without further purification. Deionized water was used as a reference in a UV-visible spectrophotometer. Ethanol and acetone were used to clean the glass apparatus used in the present study. Adsorption was carried out in an orbital shaker (Model No-JSOS-300) and the decolorization was monitored by a UV-Visible spectrophotometer (Shimadzu 1900i) at 200 - 800 nm.

Preparation of Adsorbent

Spent tea leaves were thoroughly washed with distilled water to remove any residual tea compounds. The collected STL were then dried in an oven at 100°C for several hours to ensure moisture removal. The dried Spent tea leaves were ground with a grinder to make powder. The ground powders were sieved manually. The prepared adsorbent was stored in airtight containers for subsequent experiments.

Preparation of Dye Solutions

A solution of Congo red having concentration 1×10^{-2} M dye was prepared with distilled water in a 500 mL volumetric flask and kept as stock solution. Solutions of different concentrations were prepared from stock solution by appropriate dilution. The absorbance of each solution was measured with a double beam UV visible Spectrophotometer (Shimadzu 1900i). Deionized water was used as reference for all the measurements.

Adsorption Study

A definite amount of STL was added to a solution of CR and allowed for agitation in an orbital shaker (Model No-JSOS-300). A small portion of the solution was collected after a definite time interval. The absorbance of each solution was measured with a double beam UV-Visible spectrophotometer and the removal efficiency was monitored. Deionized water was used as a reference for all the measurements. The procedure was repeated with the change of different parameters such as adsorbent dosage, concentration of the adsorbate, time and temperature. The percentage of adsorption was determined by –

$$\% \text{ of dye removal} = \frac{A_o - A_t}{A_o} \times 100$$

where, A_o and A_t are the initial absorbance of dye solution and absorbance of dye solution after adsorption at any time t respectively.

III. Results And Discussion

Effect of Adsorbent Dosage

The study inspected the impact of varying adsorbent amounts (0.5 g to 5.0 g) on the adsorption of CR. The percentage of adsorption increased with adsorbent dosage, peaking at 3.5 g with an efficiency of 56.75% at 50 minutes, while with 0.5 g, the adsorption was only 11.06%. However, beyond 3.5 g, increasing the dosage to 5.0 g resulted in a drop in adsorption capacity to 45.58%. This decrease might be attributed to the unsaturation of adsorption sites as more adsorbent is added, where available active sites may become saturated, and aggregation of particles at higher dosages can cause a reduction in effective surface area and an increase in diffusion path length [17,18]. These findings highlight the importance of optimizing adsorbent dosage for effective wastewater treatment, as illustrated in Figure 1.

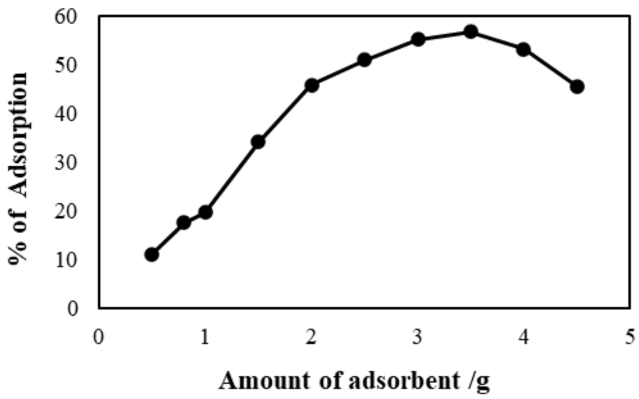


Figure 1: Percentage of adsorption at various amount of adsorbent (g). [$[CR]_o = 8\times10^{-5}$ M, pH = 9.72, t = 50 minutes, T = 26ºC]

Effect of Contact Time

Contact time is a crucial factor affecting the adsorption process of Congo red dye onto spent tea leaves. The investigation at various contact times (0 to 60 minutes) with a 100 mL solution revealed that the dye removal percentage increased with longer contact times. This enhancement in dye removal is attributed to the availability of a greater number of free surface sites on the bio-adsorbent [26]. These findings are illustrated in Figure 2. The maximum percentage of adsorption is found higher at 50 minutes then decreases. The CR molecules adsorbed on the active sites of the STL, becomes saturated after 50 minutes of contact time. After that, desorption of CR molecules take place as a result percentage of adsorption decreases.

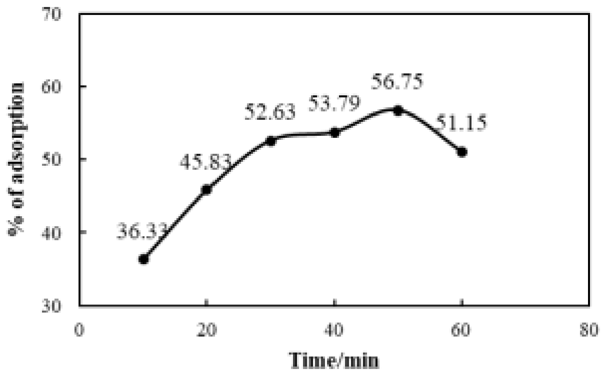


Figure 2: Percentage of adsorption at different contact time. [Amount of STL = 3.5g, $[CR]_o = 8\times10^{-5}$ M, pH = 9.72, T = 26ºC]

Effect of Initial Concentration of Congo Red

The concentration of the adsorbate is a crucial variable affecting the adsorption process. As illustrated in Figure 3, the percentage of Congo Red dye removal decreased from 61.12% to 27.75% with rising dye concentration. The percentage of adsorption decreases with the increases in concentration, indicating that adsorption was highly dependent on dye concentration. This behaviour suggests that unoccupied active sites on the bio-adsorbent surface were filled with dye molecules, leading to saturation as concentration increased [19]. Generally, higher dye concentrations correlate with increased adsorption, [20] and as the initial dye concentration rises, the dye adsorption per unit mass of the adsorbent also increases. Since the amount of adsorbent is fixed here that means the number of active sites or pores on the surface of the STL is constant. As a result, there is a competition between the CR molecules to adsorb on the surface which reduce the percentage of adsorption at higher concentration.

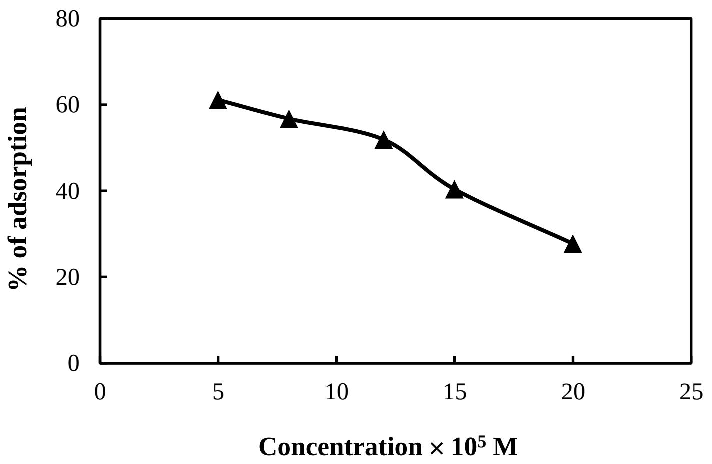
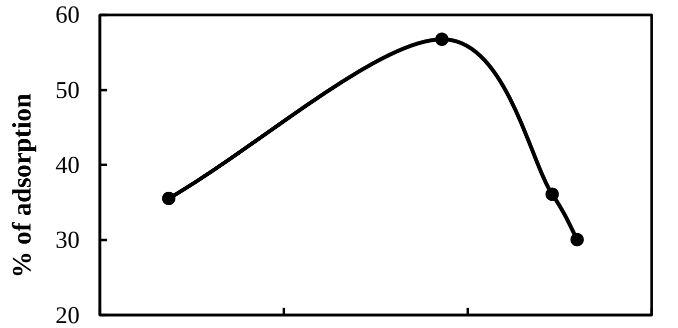


Figure 3: Percentage of adsorption at different concentration of CR. [Amount of STL = 3.5g, pH = 9.72, t = 50 minutes, T = 26ºC]

Effect of pH

The maximum adsorption of Congo Red dye occurred under optimal conditions of pH 9.72, an adsorbent dose of 3.5 g, and a shaking time of 50 minutes at a dye concentration of 8×10^{-5} M and 26°C. The pH significantly influences the adsorbent's surface charge and the dissociation of the anionic dye. Typically, the adsorption of anionic dyes like CR increases as pH decreases, as lower pH enhances the ionization of the dye and the availability of active sites on the adsorbent [21]. Additionally, pH affects Congo Red's structural stability; it appears blue in acidic conditions (pH 3.0–5.0) and varies in shade between pH 10.0 and 12.0, remaining stable between pH 6 and 10 [22]. At lower dye concentrations, adsorption increases due to the availability of unoccupied active sites, but can lead to negative adsorption when the concentration is too high, resulting in repulsive interactions between dye molecules on the adsorbent surface and in the bulk solution. This behavior aligns with findings that highlight the critical role of pH and concentration in optimizing adsorption processes for effective dye removal, as illustrated in Figure 4.



. Figure 4: Percentage of adsorption at different pH of CR solution. [Amount of STL = 3.5g, $[CR]_o$ = 8×10^{-5} M, t = 50 minutes, T = 26ºC]

Effect of Temperature

The adsorption of Congo red dye onto STL is affected by temperature, demonstrating an exothermic process. As temperature increases, dye adsorption decreases, particularly after 50 °C, due to heightened surface activity leading to desorption of previously adsorbed molecules. This indicates that Congo red dye adsorption primarily occurs through chemical adsorption [23,24]. The percentage removal of dye diminishes with rising temperatures, affirming that adsorption is inversely related to temperature, as supported by adsorption theory. According to the adsorption theory, adsorption decreases with increase in temperature and molecules adsorbed earlier on a surface tend to desorbs from the surface at elevated temperatures [25]. These findings are illustrated in Figure 5.

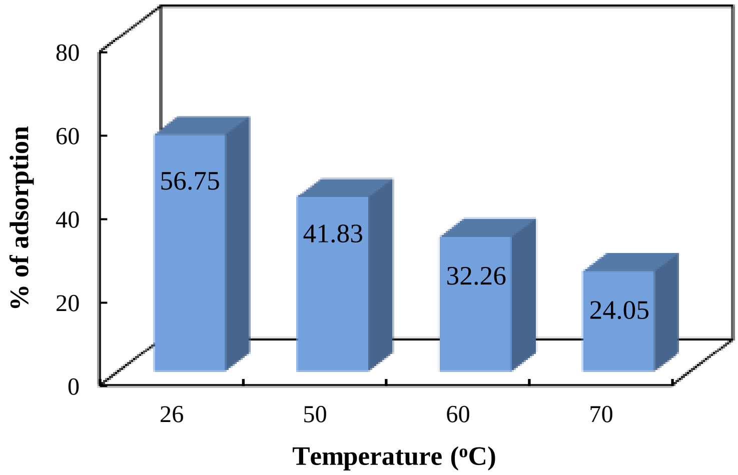


Figure 5: Percentage of adsorption at different temperatures. [Amount of STL = 3.5g, $[CR]_o = 8\times10^{-5}$ M, pH = 9.72, t = 50 minutes]

Effect of different forms of adsorbent

The percentage of adsorption by powdered spent tea leaves and raw tea leaves are 56.75% and 10.03% respectively (Figure 6). The powdered STL showed better adsorption efficiency than the raw tea leaves. Raw tea leaves contain different types of compounds such as alkaloids, catechins, cations, metals, lignans and triterpenoid saponins, oxyaromatic acids, polyphenols, sugars, etc. After the use of tea leaves, some of the compounds are extracted from the tea leaves fiber. As a result, some vacant sites are created on the surface of STL on which CR molecules can adsorb and increase the percentage of adsorption.

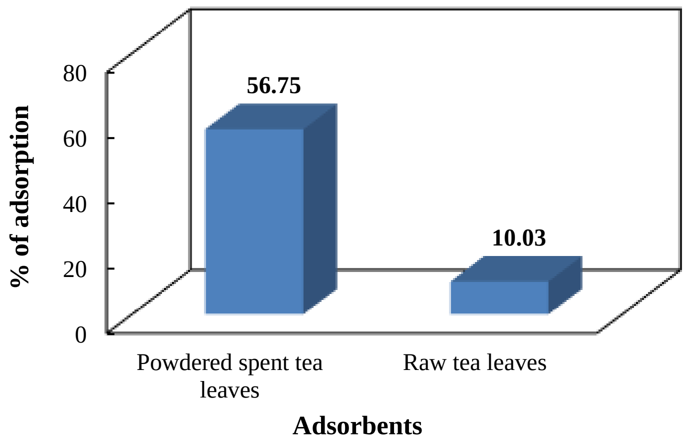


Figure 6: Percentage of adsorption at different forms of adsorbent. [Amount of STL = 3.5g, $[CR]_o = 8\times10^{-5}$ M, pH = 9.72, t = 50 minutes, T = 26ºC]

FTIR Spectra of Spent Tea Leaves

The FTIR spectrum of tea leaves reveals the presence of key functional groups, primarily polyphenols, carboxylic acids, and amino acids. The broad band at around 3400 cm^{-1} is attributed to the O-H and N-H stretching vibrations of polyphenols. The strong band at 1616 cm⁻¹ corresponds to the C=O stretching of polyphenols and C=C stretching of aromatic rings. Bands at 2918 cm⁻¹ and 2848 cm⁻¹ indicate the presence of C-H and O-H stretching in alkanes and carboxylic acids, respectively. In addition, the band at 2354 cm⁻¹ is associated with the C-N stretching of amino acids. The band at 1031 cm⁻¹ is associated with the C-O stretching of amino acids. Similar FTIR bands have been reported for various tea types, including black, oolong, and green tea, confirming the presence of these functional groups in tea extracts [27].

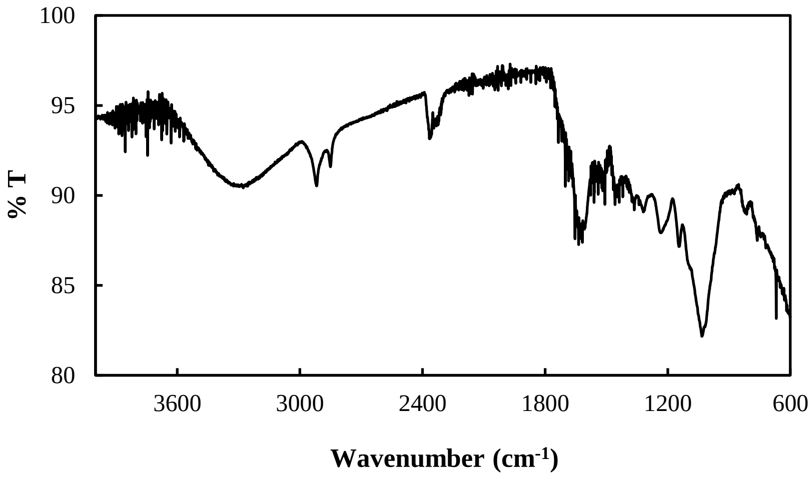


Figure 7: FTIR Spectra of Spent Tea Leaves.

Kinetic Studies

The mechanism and kinetics of adsorption of Congo red on the surface of STL can be obtained by fitting the experimental data to some kinetic models (pseudo-first-order, pseudo-second-order). The kinetic equations for the pseudo-first and pseudo- second-order models are as follows [28,29].

$log\ (q_e - q_t) = log\ q_e - k_1 t$…… (1)

$t/q_t = 1/(k_2 q_e^2) + t/q_e$ ………… (2)

where q_e and q_t are the amount of adsorbate adsorbed on adsorbent (mg/g) at equilibrium and any time t respectively. k_1 and k_2 are the corresponding rate constants for the pseudo-first and second-order reactions.

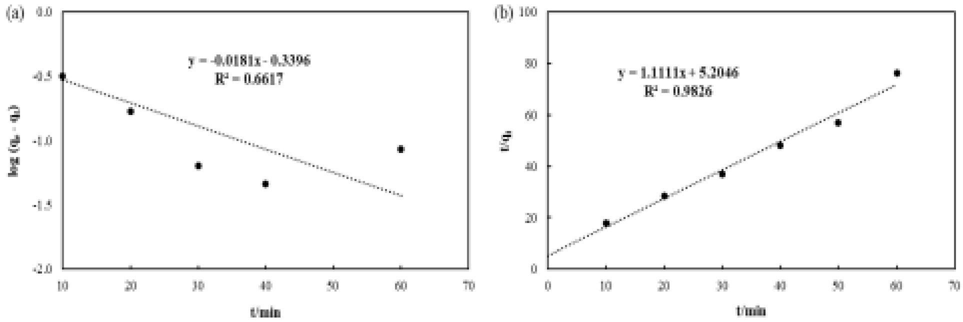


Figure 8: (a) Pseudo first order and (b) pseudo second order kinetic model for the adsorption of CR on STL respectively.

The value of q_e and q_t are calculated by using the following equations

$q_t = (C_0 - C_t)\ V/W$ …………. (3)

$q_e = (C_0 - C_e)\ V/W$…………. (4)

where, C_0, C_t, and C_e represent the initial concentration, concentration at any time t, and equilibrium concentration of dye in mg/L respectively. V is the volume of the solution (L) used for adsorption and W is the mass of dry adsorbent (g). All the kinetic parameters are evaluated separately from the plot of $log\ (q_e\text{-}q_t)$ vs. $log\ q_e$ and t/q_t vs. t. The value of rate constants, maximum adsorption capacity, and correlation coefficient (R^2) are listed in Table 1. The value of R^2 suggests that the adsorption of CR on STL follows pseudo-second-order kinetics.

Table 1: Kinetic parameters for the pseudo-first and pseudo- second order models.

q_e	Pseudo-first-order		Pseudo-second-order	
mg/g	k_1 (min^{-1})	R^2	k_2 (Lmol^{-1}min^{-1})	R^2
0.875	0.0181	0.6617	0.2513	0.9826

IV. Conclusion

In this study, we explored the use of various adsorbents, particularly spent tea leaves, for the adsorption of disperse dyes from wastewater. The treatment of textile wastewater is challenging, especially with multiple dye components present, necessitating an understanding of interactions between different chromophores. We evaluated decolorizing efficiencies based on factors such as adsorbent dosage, dye concentration, temperature, pH, and contact time. Spent tea leaves emerged as a promising, eco-friendly alternative to commercial adsorbents due to their availability, low cost, and minimal processing requirements. The study confirmed that spent tea leaves effectively removed Congo Red dye, achieving a maximum removal efficiency of 56.75% under optimal conditions (initial concentration of 8×10^{-5} M, temperature of 26°C, dosage of 3.5 g, pH 9.72, and contact time of 50 minutes). The findings highlight the potential of using spent tea leaves as a bio-adsorbent for wastewater treatment, emphasizing the need for further research on the mechanisms of sorption and the exploration of combined treatment methods. This approach could significantly contribute to developing sustainable and efficient wastewater treatment strategies, addressing environmental pollution challenges.

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