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Research Paper

Adsorption of Anionic Dyes Using Turkish Coffee Waste: Efficiency and Mechanism

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Abstract: Turkish coffee waste (TCW), an organic by-product, was employed for the adsorption-based removal of Reactive Red 195 (RR195) dye from aqueous solutions. The study explored various parameters including pH (ranging from 3 to 9), initial RR195 concentration (5-500 mg/L), contact time (1-360 minutes), and amount of adsorbent (0.5-20 g/L). In a batch system, these experiments achieved a significant removal efficiency of 89% under optimal conditions. The pseudo-second order (PSO) kinetic model provided the most accurate representation of the kinetics of RR195 removal by TCW. Additionally, the adsorption equilibrium data aligned well with the Freundlich isotherm model. The maximum adsorption capacity of TCW for RR195 was found to be 63.5 mg/g under these optimum conditions; pH=7, RR195=500 mg/L, contact time=60 min., amount of TCW=7 g/L. These findings confirm that TCW can effectively remove RR195 through adsorption without requiring any pre-treatment.
Keywords: *Adsorption, Low-Cost adsorbent, Reactive red 195, Turkish coffee waste*

Introduction

Reactive Red 195 (RR195) is a widely used azo dye in the textile industry, known for its vibrant red colour. This dye is preferred for its strong binding affinity to cellulosic fibers, which enhances wash fastness and ensures long-lasting coloration (Katheresan *et al.,* 2018). However, the extensive use of RR195 leads to significant environmental concerns, as large quantities of untreated dye are discharged into wastewater. The dye's complex molecular structure, particularly the sulfonic acid groups that facilitate its solubility in water, makes its removal from wastewater challenging using conventional treatment methods (Saratale *et al*., 2019). Consequently, there is increasing interest in developing more efficient and sustainable techniques for the removal of RR195 from industrial effluents, including advanced oxidation processes, biological treatment methods, and the use of novel adsorbents. These efforts are crucial for reducing the environmental impact of textile dyeing processes and promoting more sustainable practices within the industry (Verma *et al*., 2021).

Various methods can be employed for the treatment of RR195. Chemical oxidation techniques effectively remove these dyes from aqueous environments (Sirés & Brillas, 2012; Deng & Zhao, 2015). Processes involving oxidants such as ozonation, hypochlorite, and peroxide aid in breaking down the dye and preventing the formation of harmful byproducts. Among physical methods, adsorption using activated carbon or other adsorbent materials is commonly used to remove the dye from water (Rafatullah *et al*., 2010; Bhatnagar *et al*., 2013). Additionally, biological treatment methods can be applied, wherein specific bacteria or enzymes are utilized to biologically degrade the dye (Ahuja *et al*., 2018). Advanced treatment techniques typically involve a combination of these methods, thereby enhancing the efficiency of the treatment process. The selection of these various treatment methods depends on the characteristics of the dye used and the capacity of the treatment facility (Zhang *et al*., 2014; Kumar *et al*., 2019). Due to the substantial global consumption of coffee, estimated at approximately 8 million metric tons annually, coffee waste is a widely available biowaste that holds significant potential as a precursor for adsorbent synthesis (Zuorro & Lavecchia, 2020).

This study examines the use of TCW, a natural waste material, as an adsorbent for the adsorption of RR195. The research evaluates the effects of key adsorption parameters, such as pH, contact time, amount of adsorbent, and initial concentration, on the adsorption efficiency of RR195 onto TCW. Additionally, the adsorption kinetics and mechanisms are systematically analyzed and calculated to provide a deeper understanding of the adsorption process.

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Material and Method

Adsorbent and Dye Preparation

In Turkey, Turkish coffee is highly popular and widely consumed by people. Turkish coffee is made by grinding coffee beans to a particle size of approximately 200 µM. Due to the high daily consumption of this coffee, significant amounts of coffee waste are generated. This raises the potential for its use as a low-cost natural adsorbent.

Before being used as an adsorbent, TCW was subjected to an extensive cleansing procedure, which involved repeated rinsing with distilled water to remove any surface impurities. The process, though necessary, seemed endless, as if the contaminants were always just out of reach, clinging stubbornly to the surface. After that, dried at 60°C for 24 hours. Thus, adsorbent was prepared. RR195 dye (Sigma-Aldrich) in powder form were used to produce stock solutions (1000 mg/L), which were then diluted to produce dyes solutions with desired concentrations. The chemical structures of the RR195 dye, TCW and RR195 adsorption images are illustrated in Figure 1.

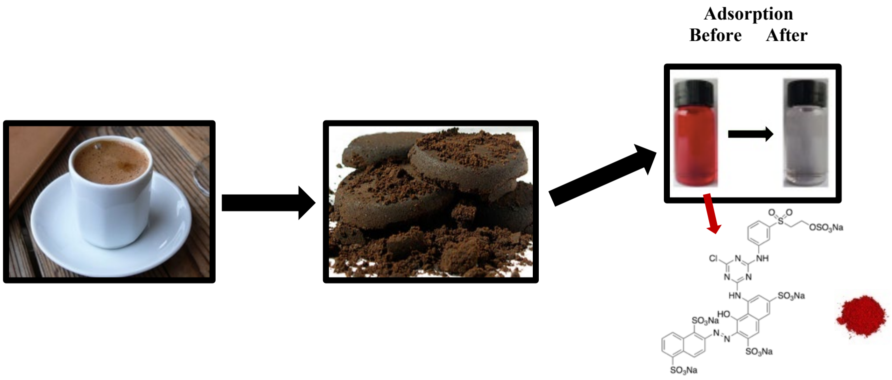

Figure 1. RR195 chemical structure, TCW and RR195 adsorption images.

Isotherm and Kinetic Study

The Langmuir adsorption isotherm (Langmuir, 1918) describes the monolayer adsorption of a solute onto a surface with a limited number of identical binding sites. The model operates on several key assumptions: (i) adsorption occurs in specific, homogeneous regions of the adsorbent; (ii) each dye molecule occupies a single site; (iii) the adsorbent has a fixed capacity for the pollutant at equilibrium; and (iv) all adsorption sites are uniform and possess equal energetic properties. In contrast, the Freundlich isotherm model (Freundlich, 1906) accounts for the heterogeneity of the adsorptive surface, allowing for multilayer adsorption and variation in site energy distribution. The Temkin isotherm model (El-Shafie, 2023) proposes that the heat of adsorption decreases linearly as more molecules are adsorbed, indicating uniform binding energy across the surface. Meanwhile, the Dubinin-Radushkevich (D-R) isotherm (Laskar & Hashisho, 2020) is grounded in potential theory and is particularly applicable to adsorption on heterogeneous surfaces. Kinetic models are used to explore the mechanisms underlying pollutant adsorption onto the adsorbent surface. This study aims to provide insights into these mechanisms and pollutant-adsorbent interactions, enabling the formulation of appropriate mathematical models for their characterization. The pseudo-first order (PFO) and pseudo-second order (PSO) have been applied to describe the adsorption process (Lagergren, 1898; Ho & McKay, 1999; Ho *et al.*, 2000). A summary of the isotherm and kinetic equations, along with their relevant parameters, is provided in Table 1.

Table 1. Isotherms and kinetic parameters related to adsorption of RR195

Model	Equation	Parameter	References
Isotherm Model			
Langmuir	$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m}$ $R_L = \frac{1}{1 + K_L C_0}$	q_m (mg/g): Maximum adsorption capacity C_e (mg/L): Adsorbate concentration at equilibrium q_e (mg/g): Amount of adsorbate uptake at equilibrium	Langmuir, 1918

Freundlich	$\ln q_e = \ln K_F + \frac{1}{n}\log C_e$	K_L (L/mg): Langmuir coefficients	Freundlich, 1906
Temkin	$q_e = \frac{RT}{b_T}\ln A_T + \frac{RT}{b_T}\ln C_e$	R_L: Langmuir separation factor K_F (mg/g) (L/mg)^n and n: Freundlich coefficients R (8.314 J/molK): Gas constant A (L/g): Temkin isotherm constant	Inyinbor, 2016
D-R	$B = {RT}/{b_T}$ $lnq_e = lnq_m - k_{ad}\varepsilon^2$ $\varepsilon = RT\ln\left(1+\frac{1}{C_e}\right)$ $E = (2\ x\ K_{ad})^{-1/2}$	T (K): Adsorption temperature q_s (mg/g): Adsorption capacity K_{ad} (mol^2/kj): Dubinin–Radushkevich isotherm constant ε: Dubinin–Radushkevich isotherm constant E (kJ/mol): Mean adsorption energy	Güneş, 2023
Kinetic Model			
PFO	$\ln(q_e - q_t) = \ln q_e - k_1 t$	q_t (mg/g): Adsorption capacity at time t t (min): Time k_1 (1/min): The pseudo first-order model rate constant	Lagergren, 1898
PSO	$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}t$	k_2 (g/mg.min): The pseudo second-order rate constant	Ho & McKay, 1999

Adsorption studies

Adsorption experiments were carried out using a thermostatic shaker (Nüve SL350) with a solution volume of 1 L and a constant shaking rate of 200 rpm. After testing various agitation speeds, it was determined that the optimal speed is 200 rpm. To examine the impact of contact time on the removal efficiency of RR195, tests were performed at different time intervals, spanning from 2 minutes to 360 minutes. The influence of amount of adsorbent on RR195 removal was assessed using different amounts of TCW, varying from 0.5 to 20 g/L. The effect of pH on RR195 adsorption was evaluated by performing experiments within a pH range of 3 to 9. Additionally, the impact of initial dye concentration on removal efficiency was examined using seven different concentrations, ranging from 5 to 500 mg/L. For the analysis of the effects of amount of adsorbent, pH, contact time, temperature, and initial dye concentration, samples were centrifuged at 3000 rpm for 4 minutes using a Nüve CN180 centrifuge. The resulting supernatants were then analyzed using a UV-Vis spectrophotometer. The residual dye concentration in the solution after adsorption was determined at 542 nm. From the experimental data, equilibrium concentration (C_e), adsorption capacity (q_e), and percentage removal rates were calculated according to Eq. (1) and Eq. (2).

$$RR195\ removal\ (\%) = \frac{C_0 - C_e}{C_0} x100 \quad (1)$$

$$q_e = \frac{(C_0 - C_e).V}{m} \quad (2)$$

C_o is initial concentration of RR195, C_e (mg/L) is final concentration of RR195. m (g) represents the mass of TCW. The volume of the solution is presented with V (L), where q_e (mg/g) is the amount of adsorbed RR195 by TCW.

Determination of the Point of Zero Charge (pH_{pzc}) for TCW

For this study, the methodology outlined by Aguilar *et al*. (2020) was applied to determine the pH_{pzc} of the adsorbent selected for the removal of RR195 from TCW. Based on this approach, the adsorption mechanism was analyzed, providing insights into the processes potentially involved during adsorption.

Characterization of TCW

The FTIR spectra corresponding to TCW are presented in Figure 2. TCW exhibits peaks that correspond to numerous functional groups.

FTIR analysis of TCW provides essential insights into the chemical structure of the organic components present in the material. The typical FTIR spectrum of TCW reveals several characteristic bands that correspond to different functional groups. The broad band observed in the range of 3300-3500 cm^{-1} is associated with O-H stretching vibrations (Dolatabadi *et al*., 2018), indicative of hydroxyl groups, which are prevalent in water, cellulose, and phenolic compounds found in coffee waste. The bands appearing between 2850-2950 cm^{-1} correspond to C-H stretching vibrations of aliphatic hydrocarbons, suggesting the presence of lipids and other aliphatic components. The distinct band observed around 1700-1735 cm^{-1} is attributed to C=O stretching vibrations (Litefti *et al.,* 2019), highlighting the presence of carbonyl groups from esters, acids, or ketones. Additionally, the band in the 1600-1650 cm^{-1} range indicates C=C stretching vibrations, pointing to the presence of aromatic rings, likely from lignin or other phenolic compounds (Igwegbe *et al.,* 2019). The 1400-1450 cm^{-1} region corresponds to C-H bending vibrations, while the 1000-1200 cm^{-1} region is associated with C-O stretching vibrations, representing the presence of alcohols, esters, and ethers. Finally, bands in the 900-750 cm^{-1} range indicate aromatic ring bending vibrations, further confirming the presence of lignin (Zahir *et al*., 2024). These spectral features highlight the complex organic composition of TCW, making it a valuable resource for applications in biofuel production, biomaterial development, and other sustainable technologies.

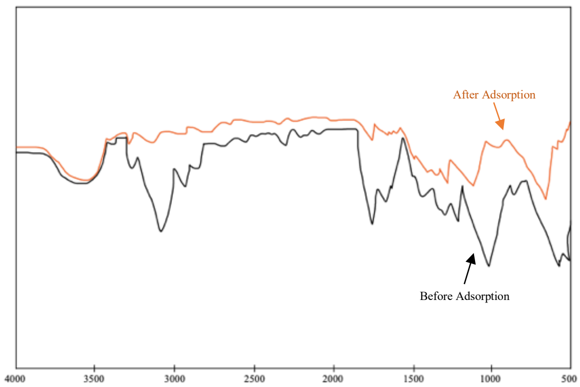

Figure 2. FTIR spectra of TCW

Results and Discussions

Effect of operating parameters on RR195 removal

The pH_{pzc} is a critical parameter in adsorption studies, as it defines the pH at which the adsorbent surface carries a neutral charge. At pH values below the pH_{pzc}, the adsorbent surface becomes positively charged, enhancing the adsorption of negatively charged species through electrostatic attraction (Yan *et al*., 2018). Conversely, at pH values above the pH_{pzc}, the surface acquires a negative charge, promoting the adsorption of positively charged molecules. Determining the pH_{pzc} is essential for optimizing adsorption processes, as it allows for the prediction of adsorption behaviour under different pH conditions. For instance, in the adsorption of dyes or heavy metals, knowing the pH_{pzc} can help in selecting the most favourable pH environment to maximize removal efficiency (Zhu *et al*., 2020). This parameter is particularly significant when dealing with heterogeneous adsorbents, where surface charge variation plays a vital role in the overall adsorption mechanism.

Figure 3 illustrates a non-linear relationship between the initial and final pH values. The high pH_{pzc} (8) of TCW indicates an abundance of positive charges on the adsorbent surface, which can only be fully neutralized under alkaline conditions.

In this adsorption study, key parameters such as pH, amount of adsorbent, initial RR195 concentration, and contact time were carefully evaluated. Comprehensive results for each of these factors are presented in Figure 4. The adsorption performance of the systems was found to be strongly influenced by pH, as it alters the ionization state of functional groups on both the adsorbate and the adsorbent. Thus, identifying the optimal pH for RR195 adsorption onto TCW is essential. To determine this, experiments were conducted over a pH range of 3 to 9, with varying initial dye concentrations, solid-to-liquid ratios between 0.5 and 20 g/L, and at a constant temperature of 25°C. The amount of adsorbent is a crucial factor in defining the removal capacity in the adsorption process. Therefore, the amount of TCW was systematically varied from 0.5 to 20 g/L, while keeping pH and temperature constant at 25°C. The removal efficiency of TCW improved significantly, reaching up to 87%. As the amount of adsorbent increased, the removal efficiency of RR195 also increased, which can be attributed to the greater availability of active binding sites. Initially, the removal efficiency increased as the amount of adsorbent was raised from 0.5 g to 20 g/L, reaching up to 7 g/L, after which it stabilized. However, the adsorption capacity declined, likely because at lower adsorbent doses, more active sites remain accessible on the surface. In the study, the RR195 removal rate was rapid during the initial minutes of contact, with equilibrium being reached after approximately 60 minutes. The high removal efficiency observed during the early stages of adsorption is likely due to the increased active surface area of the adsorbent. For initial RR195 concentrations ranging from 5 to 500 mg/L, a removal efficiency of 89% was achieved within the first 60 minutes. Based on the overall analysis, the optimal conditions for RR195 adsorption using TCW were determined as follows: a contact time of 60 minutes, 7 g/L amount of TCW, an initial RR195 concentration of 500 mg/L, and a pH of 7. The adsorption process was further conducted at a temperature of 25°C, using a solution volume of 1 L and an agitation speed of 200 rpm.

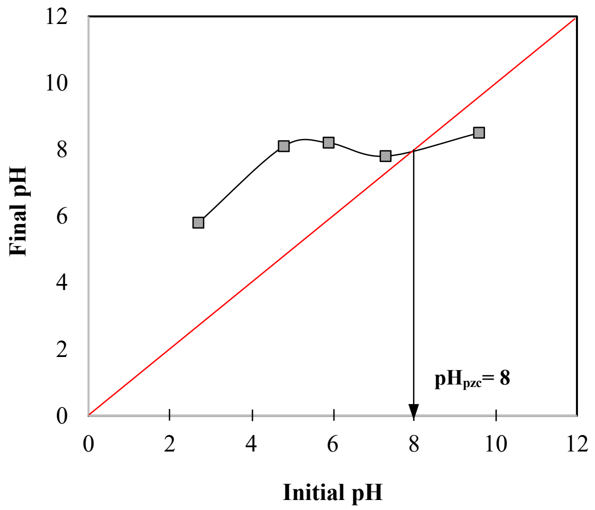

Figure 3. Determination of pH_{pzc} for TCW

Kinetic and Isotherm Study

Equilibrium data, commonly known as adsorption isotherms, are crucial for the characterization and design of adsorption systems. These data provide a mathematical relationship between the amount of adsorbate per unit of adsorbent at equilibrium and the concentration remaining in the solution. Isotherm studies were performed by treating RR195 dye solutions with initial concentrations varying from 5 to 500 mg/L, at a constant temperature of 25°C, for a fixed contact time of 60 minutes. Figure 5 provides the constants of Langmuir, Freundlich, Temkin, and D-R isotherm models. Freundlich model is a better fit for the adsorption data of RR195, as indicated by the higher R^2 values than other models. According to these results, the Freundlich adsorption isotherm model indicates that the adsorption process is multi-layered. And that adsorption occurs on a heterogeneous surface, and each layer is homogeneous and monolayered, as described in the Langmuir model. Additionally, since the adsorption mechanism occurs based on weak van der Waals forces, it can be considered a physical process.

The correlation coefficient for the PSO kinetic model is found to be high (0.98) (Figure 6). The calculated equilibrium adsorption capacity of TCW is 61.9 mg/g, which is consistent with the experimental value of 63.5 mg/g. The strong correlation provided by the PSO kinetic model suggests that the adsorption of RR195 onto TCW follows a multi-step process, involving both surface sorption and diffusion into the adsorbent. Furthermore, the excellent fit of the PSO kinetic model indicates that chemical sorption, involving valence forces through electron exchange or sharing between the TCW adsorbent and RR195 dye molecules, could be the rate-controlling step and play a significant role.

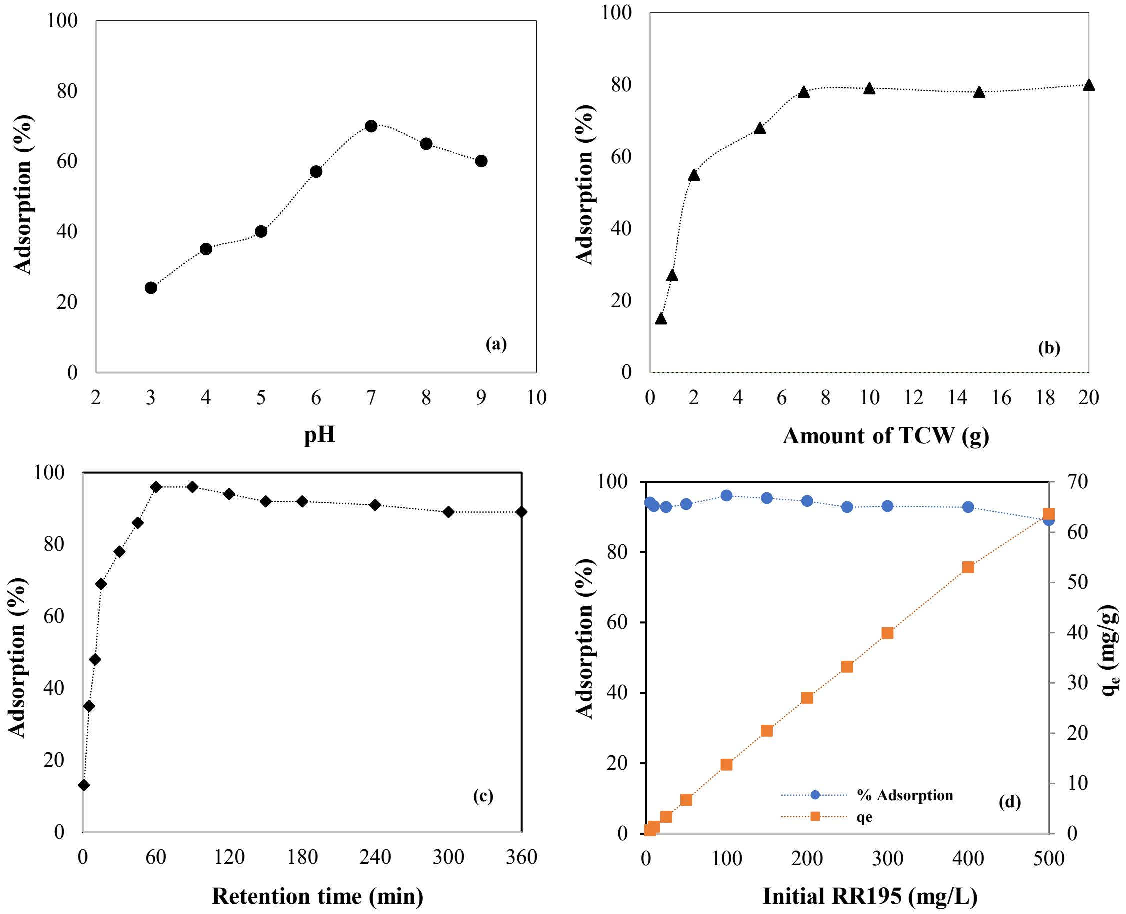

Figure 4. Effects of **(a)** solution pH, **(b)** amount of TCW, **(c)** retention time, and **(d)** initial RR195 concentration on adsorption process

The literature includes various studies involving different adsorbents and dyes. Some of these studies are summarized in Tab. 2. When comparing adsorption capacities, the type of adsorbent material and the dye used are significant factors.

Table 2. Evaluation of RR195 adsorption studies with various adsorbents

Adsorbent	Dye	q_e (mg/g)	References
Coffee waste	Congo Red	34.36	Wong *et al.*, 2020
	Reactive Black 5	77.52	
Banana peel powder	Reactive Black 5	49.2	Munagapati *et al.*, 2018
Soybean leaves	RR195	12	Mahanna & Samy, 2020
Coffee husk	RR195	188.12	Thi *et al.*, 2021
Lotus leaf powder	RR195	131.5	Munagapati *et al.*, 2021
Sunflower husks	RR195	9.5	Alhares *et al.*, 2023
	RB49	9.3	
TCW	RR195	63.5	This study

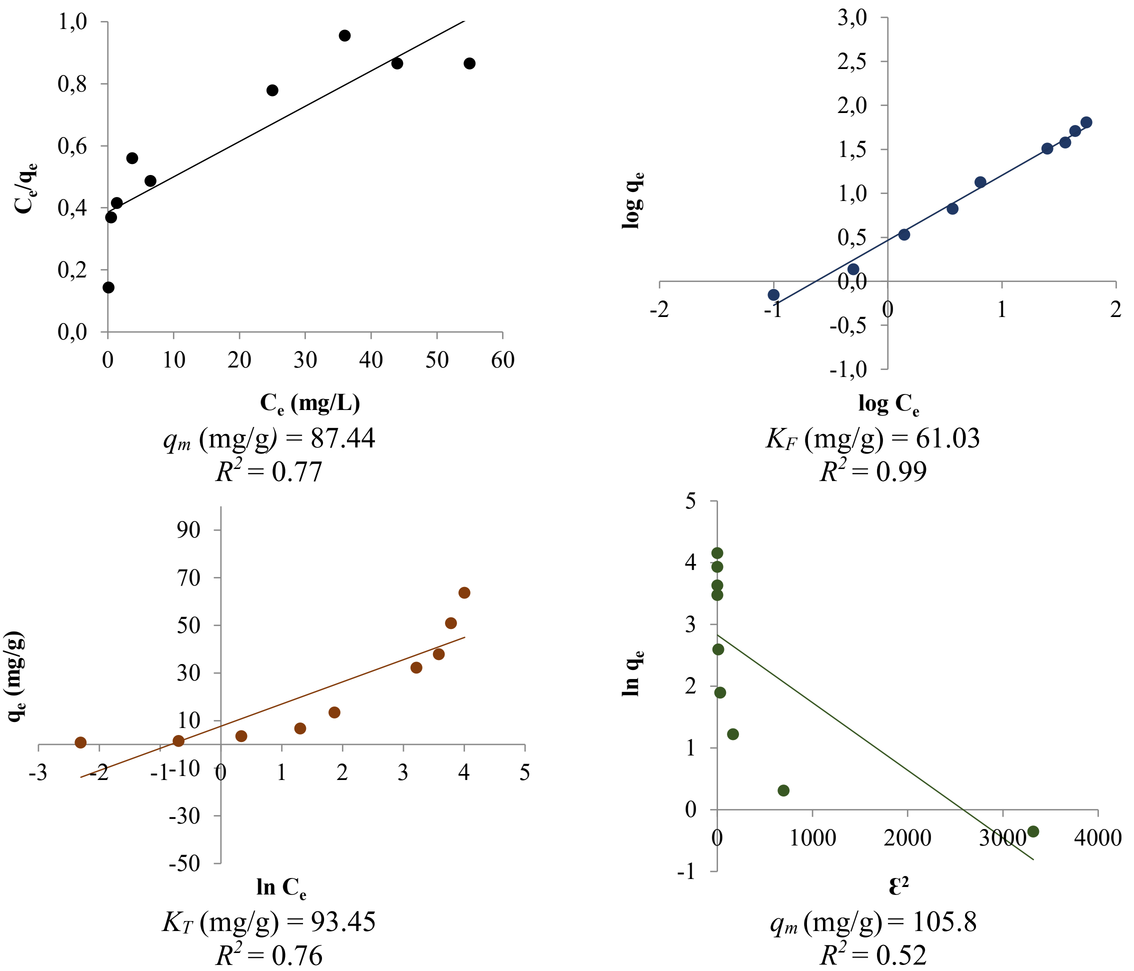

Figure 5. Langmuir, Freundlich, Temkin and D-R isotherm results

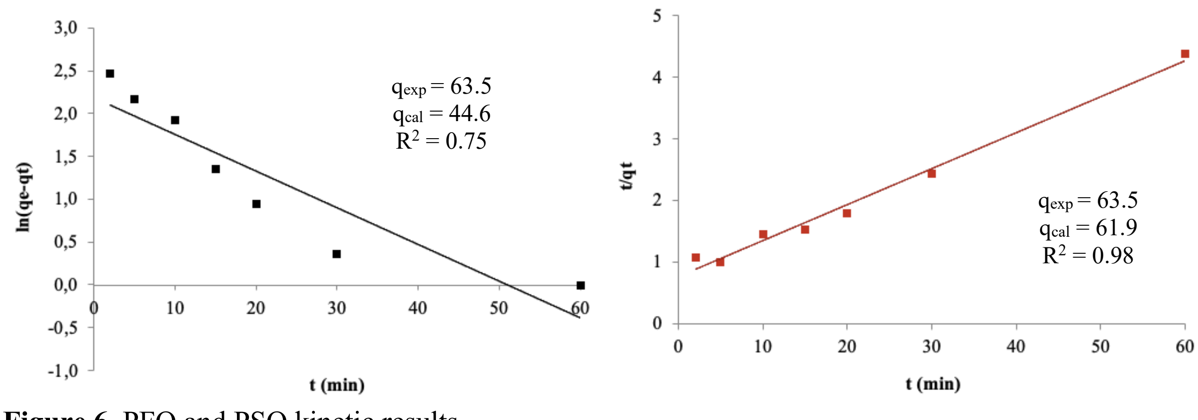

Figure 6. PFO and PSO kinetic results

Conclusion

This study investigated the removal of RR195 dye using TCW through an adsorption method in a laboratory setting. The adsorption process was examined with respect to various parameters including pH (3-9), initial concentration of RR195 (5-500 mg/L), amount of TCW (0.5-20 g), and contact time (2-360 minutes). Optimal conditions for maximum removal efficiency were found to be pH 7, TCW amount of 7 g, initial RR195 concentration of 500 mg/L, and contact time of 60 minutes. The maximum adsorption capacity of TCW was calculated to be q_e=63.5 mg/g. According to the results from the isotherm study, the Freundlich isotherm model best describes the adsorption mechanism among the applied isotherm equations. Additionally, the adsorption kinetics were best represented by the pseudo-second-order kinetic model. It was determined that TCW is suitable for the adsorption of RR195.

Additionally, the fact that TCW was used without any thermal or chemical pre-treatment is a significant aspect of the study.

Declarations: *Author has read, understood, and has complied as applicable with the statement on "Ethical responsibilities of Authors" as found in the Instructions for Authors.*
Availability of data and material: *Not applicable.*
Competing interests: *The author declares no financial and non-financial competing interests.*
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