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DYESTUFF REMOVAL FROM SYNTHETIC TEXTILE WASTEWATER USING OLIVE LEAF AS ADSORBENT MATERIAL

Büşra Özkul[1], Neval Ocak[1], Burcu Tan[2*], Tijen Ennil Bektaş[1]
[1] Çanakkale Onsekiz Mart University, Faculty of Engineering, Department of Chemical Engineering, 17020, ÇANAKKALE
[2] Çanakkale Onsekiz Mart University, Faculty of Science, Department of Chemistry, 17020, ÇANAKKALE

Abstract

The textile industry is one of the sectors where water use is high. Textile wastewater contains a high percentage of dyestuffs. Dyestuffs prevent photosynthetic activities in water and disrupt the ecological balance in water. Treatment of dyestuffs is very important for the prevention of water pollution and for the health of living things. There are several methods for the treatment of wastewater containing dyestuffs. In this study, it was aimed to remove dyestuff from synthetic textile wastewater by using olive leaf by adsorption method. The effects of initial pH, amount of adsorbent, contact time and temperature on adsorption were investigated. According to the data obtained, optimum conditions were determined as pH 2, 0.25 g of adsorbent, 2 hours of contact time, and a temperature of 25 °C. When the experimental data were applied to the kinetic models, it was observed that they conformed to the pseudo-second-order rate equation. The data obtained at the end of the experiments were applied to the Langmuir and Freundlich isotherm models. It was observed that the data obtained with olive leaf conformed to the Freundlich isotherm. The reaction for the olive leaf is an exothermic and occurs spontaneously, according to thermodynamic analysis. Negative entropy indicates a decrease in disorder in adsorption.

Key Words: Textile wastewater, dyestuff, adsorption, olive leaf

1. Introduction

The increase in the world population, insufficient environmental awareness, rapid developments in industry and technology cause an increase in water use and a decrease in the amount of usable water. Water supplies are being depleted as the demand for water is rising (Akın & Akın, 2007). Global water demand is expected to increase to 6,000 km3 per year by 2050, while developing countries are expected to face major water pollution and water scarcity (Yusoff et al., 2023). With the increase in the world population, the need for clothing has also increased. The textile sector has also been able to grow because to the fast-evolving fashion industry and advancing technology. The share of the textile industry in Türkiye in production is around 20%. (Şeker, 2007). The growing textile industry has also led to a rise in water use and wastewater production. Textile industry wastewater contains BOD, COD, AKM and high dyestuffs. The textile industry is the main user of dyestuffs (Bharathiraja et al, 2018; Ganaie, 2023). Wastewater treatment is challenging due to the high levels of COD and dyestuff in wastewater that cause the water's visual quality to deteriorate. Even trace amounts of dye in wastewater are highly visible and the color of wastewater is the most visible contaminant (Crini & Badot, 2008). Wastewater containing dyestuffs, which are discharged into receiving environments without being treated, prevents light from entering the water and adversely affects photosynthetic activities under water. However, they accumulate in aquatic organisms and form toxic and carcinogenic products. For this reason, they cause the flora of the environment to change. Due to the presence of aromatic rings in the paint, it is very difficult to biodegrade. There are several reported methods for the treatment of dyestuff containing wastewater (Srivastava & Choubey, 2021). Treatment procedures are carried out after selecting the best techniques based on the composition of the wastewater produced by the textile industry (Namal, 2017). Companies must develop unique process designs due to the industry's varying rates of water and dyestuff usage (URL-1, 2022). The dyestuff removal in wastewater is carried out by choosing the most suitable method for each process (Yagub et al., 2014). Treatment processes can be carried out in three different ways as physical, chemical and biological "URL-2, 2022" (Dutta et al. 2021). The adsorption method, one of the physical approaches, will be used in this study to remove dyestuffs from textile wastewater. Adsorption is a technique used in wastewater treatment to remove dyestuffs by employing compounds referred to as adsorbents that can hold

***Corresponding Author:**
Burcu Tan; Çanakkale Onsekiz Mart University, Faculty of Science
Department of Chemistry,
17020, Çanakkale-Turkiye.

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dyestuffs (URL-3, 2012). Adsorption processes are an advantageous approach for wastewater treatment, especially since the adsorbent is cheap and does not require a pre-treatment step before it is applied (Moosavi, 2020).

Currently, various adsorbent materials are used to treat dye-containing wastewater, such as carbon-based nano-adsorbents, bio-sorbents, polymer-based adsorbents, transition metal-based oxides, and MOFs (Ojedokun & Bello, 2017; Asadullah, 2010; Xue et al. 2010; Yao et al., 2011; Wang & Wei, 2017; Haque et al., 2010; Aboelfetoh et al., 2020). Agricultural waste has low cost. These preferred wastes are biodegradable and sustainable. It is very advantageous compared to carbon-based adsorbents (Bhatnagar & Sillanpaa, 2010). The study's objective was to use an organic waste, olive leaf, as an adsorbent material to remove dyestuff from synthetic textile wastewater. Olive is a plant belonging to the Mediterranean climate. A by-product is produced every year because of the cultivation and extraction of olives (Hannachi et al., 2010). Research to find new uses for the byproducts of olive oil production is of great importance not only for the economy but also for the environment in olive growing communities (Blazquez et al., 2011). One of the by-products of the olive tree is the olive leaf. Each tree produces 25 kg or more in leaves and branches of olives annually. Türkiye accounts for 9% of global olive production. Consequently, a lot of waste olive leaf is generated. Thus, olive leaf is a cheap adsorbent material because it is abundant and cost-effective (Geylan, 2016).

2. Materials and Methods

In this study, In this study, dyestuff removal by adsorption method using olive leaf from synthetic textile wastewater was investigated. Olive leaf wastes were obtained from an olive oil producing factory in Çanakkale region. Olive leaves were washed with distilled water. For 72 hours, it was kept in a 40°C drying-oven. It was ground in a grinder, sieved with a sieve and placed in a desiccator. 30 g NaCl, 1.6 g Na2CO3 and 0.1 g Sumifix Supra Blue EXF were mixed in a beaker and distilled water was added and 1L solution was obtained. It was stirred for 1 hour at 50°C via magnetic stirrer. The prepared solution was adjusted to have a pH of 6.9 in order to be close to the textile industry wastewater, and a stock solution was prepared at a concentration of 100 mg/L. New solutions were prepared in 50 ml volumes, with concentrations of 5,15,20,25,30,35 and 40 mg/L, respectively. The absorbance values of the prepared solutions were determined with a Shimadzu UV-1280 spectrophotometer device at a wavelength of 610 nm. Working line graph and equations were obtained with these values.

Regarding adsorption method, 50 ml of the solution with a concentration of 50 mg/L was taken and left in contact with a specific amount of adsorbent in a shaking water bath at 25 ºC for 24 hours. By using HCl and NaOH solutions of different concentrations, the solution's initial pH was modified to reflect various values and the dyestuff removal was investigated. A solution of 50mg/L concentration was added to four flasks as 50 ml each, 0.25; 0.5; 1 and 1.25 g adsorbent was added respectively, and adsorption was carried out at optimum pH value. These flasks were stirred in a water bath at 25°C for 24 hours. Olive leaf was added at optimum pH and optimum amount of adsorbent by taking 50 ml each to the flasks from the 50 mg/L-concentrated solution. It was stirred at 25; 35 and 45°C in a water bath for 24 hours. Entropy, enthalpy, free enthalpy change, and equilibrium constant were determined during adsorption, and a thermodynamic analysis of adsorption was investigated. 50 ml of the solution, which has a concentration of 50mg/L, was taken into the flasks and mixed at 25°C in a water bath at various time periods (1; 2; 3; 4; 5; 6; 7; 17; 20; 24 hours) at the optimum pH and adsorbent amount. The Langmuir and Freundlich isotherm models were used to analyze the data that were acquired at the end of the experiments. Experiment data were applied to pseudo-first order and pseudo-second-order rate equations.

Percentage dyestuff removal (Removal%) was calculated by using (1).

$$\text{Removal}\ \% = ((Co - C)\ x100\%) / Co \tag{1}$$

where Co represents the initial concentration(mg/L). C is the concentration after a certain time of adsorption. The amount of dyestuff that was calculated to have been absorbed on the adsorbent surface is

$$q = [(C_0 - Ce)V]/m \tag{2}$$

where C_0 and Ce(mg/L) are the dye concentrations at starting and equilibrium, respectively; and q is the amount of dye adsorbed onto the adsorbent (mg/g); V is the solution's volume (L), and m is the adsorbent's mass (g).

3. Results and Discussions

3.1 Characteristics of olive leaf

Surface morphologies were examined using scanning electron microscopy (SEM) and X-Ray Diffraction method (XRD). SEM images were obtained using the JEOL-JSM 7100-F Scanning Electron Microscope. XRD data was obtained with the PANalytical Empyrean instrument. At the same time, the chemical analysis of the samples was determined with the EDX (Energy Distribution X-Ray Spectrometer) detector in the SEM device. The results are given in Figures 1 and 2.

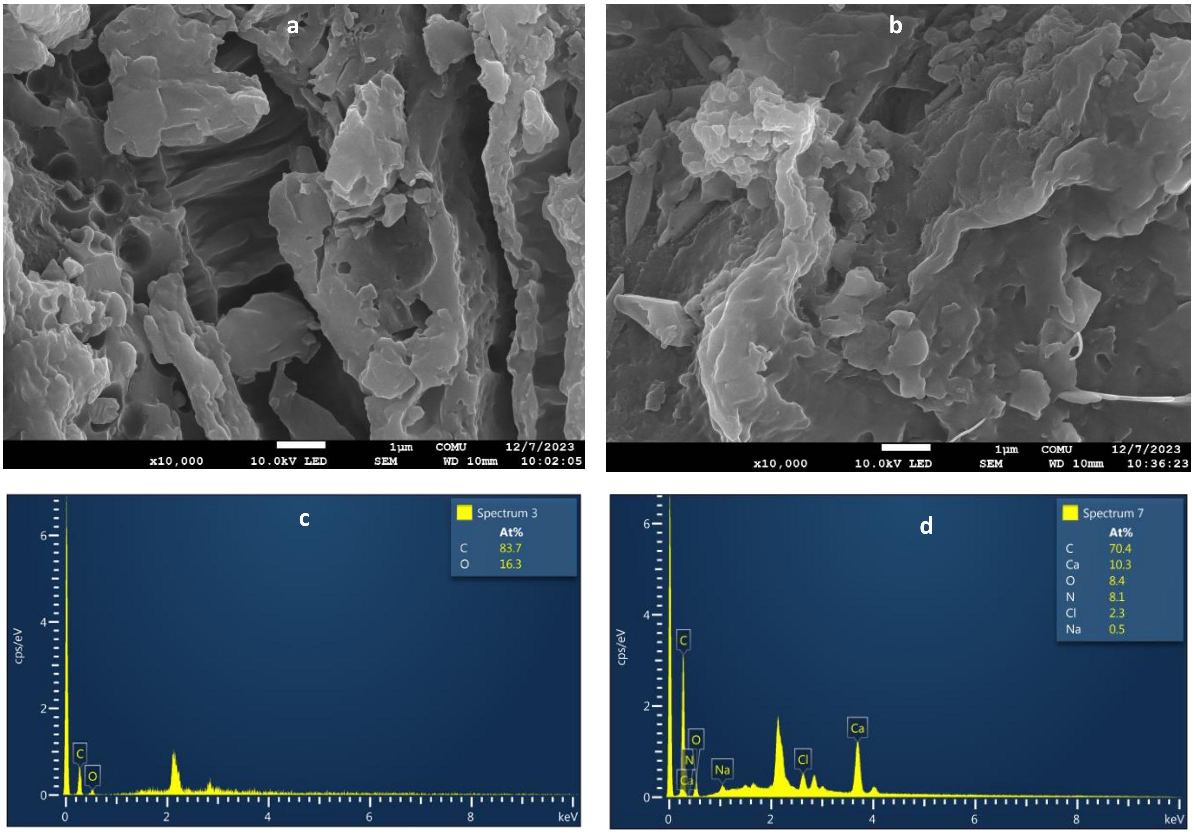

Fig. 1 Images captured by SEM of the raw olive leaves (a), olive leaves after adsorption (b) and EDX analysis of the raw olive leaves (c), olive leaves after adsorption (d)

SEM images show that the dyestuff is adsorbed quite well on the raw olive leaf. Before the adsorption process, the olive leaf used as an adsorbent has a porous and very rough structure (Fig. 1a). This structure provides a good area for the adsorbent to adhere. After adsorption process, it is seen that the pores of the olive leaf are closed with dyestuff and the surface is covered with dyestuff layers (Fig. 1b). In this image, the presence of active surface areas, which are still present, can be observed. This shows that the pore capacity and area of the surface are suitable for adsorption. Elemental analysis shows that the raw olive leaf consists of two major compositions, C and O elements (Fig. 1c). The olive leaf analysis after adsorption includes elements of both the olive leaf and the dyestuff, together with minor compositions (Fig. 1d). These images prove that the dyestuff is well adsorbed by the olive leaf.

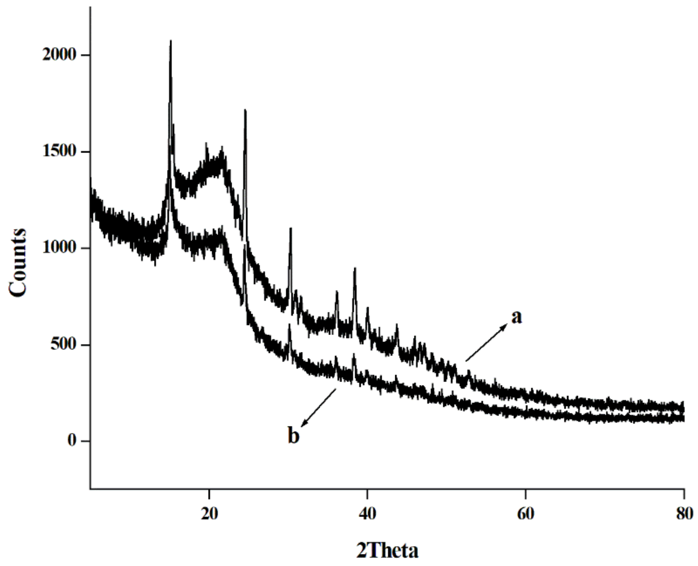

Fig. 2 XRD results of the raw olive leaves (a), olive leaves after adsorption (b)

According to the XRD results, the wide diffraction peaks (decreased peak density) (Fig. 2a) of the raw olive leaf mean that they have less crystallinity compared to the olive leaves form (Fig. 2b) after adsorption (Soleimanifard et al., 2019; Liu & Wu, 2010).

3.2 Effect of initial pH

The pH value of the solution, which has a concentration of 50 mg/L, was measured as 6.9. The pH values studied for this parameter are 2,4, 6.9 and 9. 0.25g of olive leaves were put into each flask and it was held for 24 hours in a water bath with a shaking motion at 25C. The outcomes are represented in Fig.3.

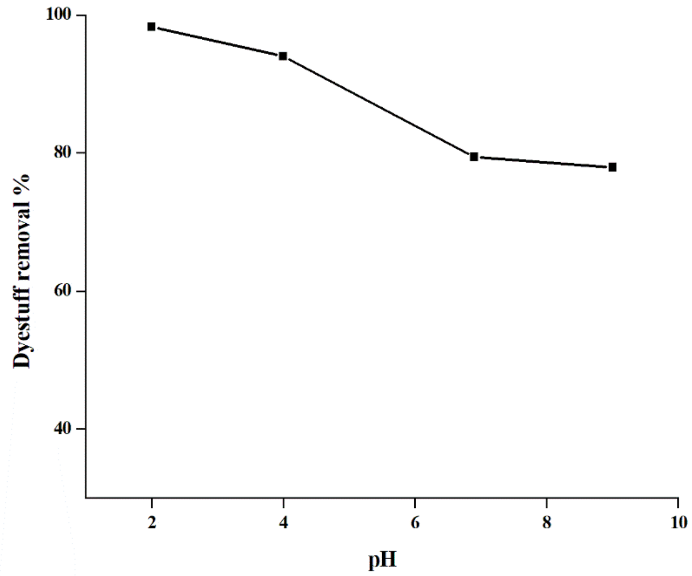

Fig. 3 The effect of initial pH value on dyestuff removal

As it evident from the Fig. 3, it was determined that the highest dye removal occurred in the solution with a pH value of 2. The surface charge of the adsorbent generally has a significant impact on adsorption. Olive leaf consists of powerful plant polyphenols that are anti-oxidative. Scientific studies have shown that the oleuropein substance contained in the olive leaf is much higher than the other parts and has revealed that it is an active phytochemical. Oleuropein is hydrolyzed with acid and turns into (-) elenolic acid compound, which is reported as antiviral (URL-4, 2022). Because there are too many protons in solutions, the negative charge on the surface of olive leaf is diminished, which contributes to the good removal of dyestuff at lower pH levels. The system's pH falls as a result, and the quantity of positively charged sites rises. Sumifix dyes are reactive dyes. Anionic dyes that are extremely water soluble are reactive dyes. Electrostatic attraction makes the dyestuff anion more likely to bind to a positively charged surface spot on the adsorbent.

3.3 Effect of adsorbent amount

To investigate the impact of adsorbent quantity, several quantities (0.25-1.25g) of ground olive leaves were used. It was stored for 24 hours at 25oC in a water bath that was shaking. The optimum amount of adsorbent was determined as 0.25g. The percentage of dyestuff removal against the amount of adsorbent is given in Figure 4. The effectiveness of dyestuff removal of the olive leaf was about between 99.6% and 99.9% in adsorbent quantity of 0.25 and 1.5 g/50 mL, respectively. The lowest amount of 0.25 g was used as the optimum amount of adsorbent.

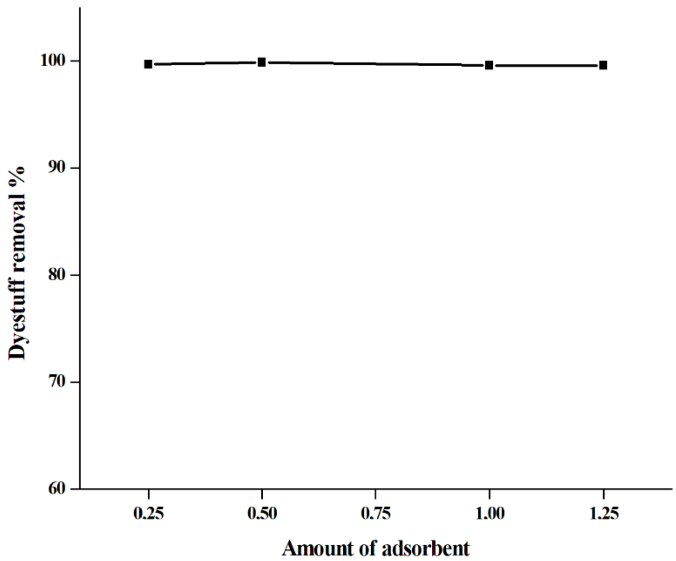

Fig. 4 Effect of amount of the adsorbent on dyestuff removal

3.4 Temperature effect

To investigate the impact of temperature, 50 ml of the 50 mg/L-concentrated solution was taken into the flasks and 0.25 g adsorbent was added at pH 2. Through the adsorption process, according to the values obtained from the spectrophotometer, it was determined that the olive leaf reached the highest percentage of dyestuff removal at 25ºC (Fig.5).

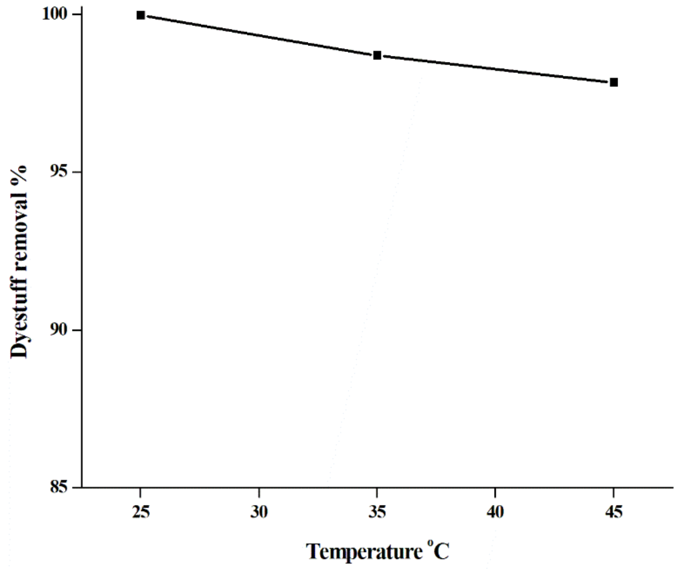

Fig. 5 Temperature effect on dyestuff removal

During adsorption, entropy, enthalpy, free enthalpy change and equilibrium constant are determined and thermodynamic investigation of adsorption is performed. Accordingly, temperature is an important parameter.

Table 1. Thermodynamic data of olive leaf

Temperature (°C)	**Kc**	**ΔG° (kJ/mol)**	**ΔS° (J/mol K)**	**ΔH° (kJ/mol)**
25	126,86480	-11999,1		
35	15,23209	-6973,8	-312,58	-104540,2
45	9,07643	-5831,4		

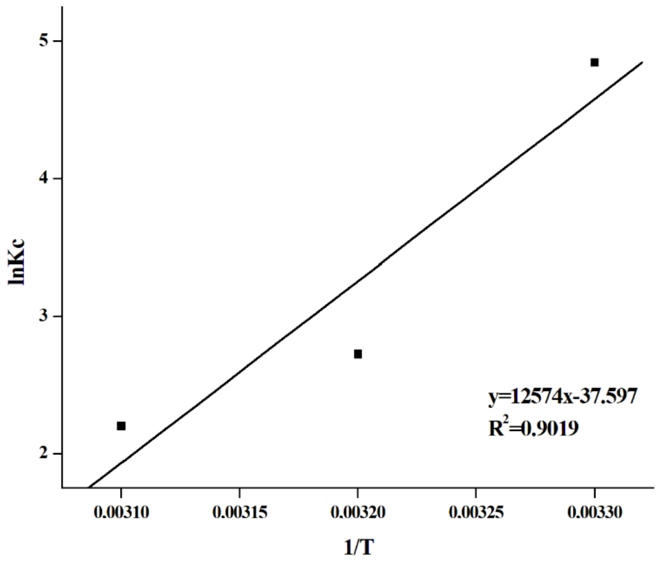

Fig. 6 The plot of 1/T versus dyestuff removal

During adsorption, entropy, enthalpy, free enthalpy change and equilibrium constant were determined, and the thermodynamic investigation of adsorption was carried out. The graph of lnKc vs. 1/T is given in Figure 6. The thermodynamic data of the olive leaf, which is an adsorbent substance, are given in Table 1.

The variation in standard free energy (ΔGº), entropy (ΔSº) and enthalpy (ΔHº) of the following equations were used to calculate:

$$\Delta G° = -RT \ln K_c \quad (3)$$

where T is the temperature in K, K is the equilibrium constant, and R is the gas constant. The estimated equilibrium constant (K) is:

$$K = \frac{q_e}{C_e} \quad (4)$$

The van't Hoff equation indicates that:

$$\ln K = \frac{\Delta S°}{R} - \frac{\Delta H°}{RT} \quad (5)$$

With the use of this equation, a 1/T graph was created against log Kc. This graph's slope is ΔH° and its cut point is ΔS°. Evaluating according to the graph of the Van't Hoff equation, the parameters ΔH°, ΔS° and ΔG° were calculated (Table 1). The spontaneous behavior of the adsorption process is indicated by the negative values of ΔG◦ at various temperatures. The negative value of ΔSo suggests that the system exhibits random behavior.

3.5 Effect of contact time

The contact time of the adsorbent and the dyestuff is a crucial parameter to reach the maximum removal percentage. In order to establish the optimum contact time, 50 mg/L solutions with a pH 2 were shaken with 0.25 g olive leaves at 25ºC for different times. Dye removal increased during 2 hours and reached equilibrium after 2 hours. The results are given in Figure 7. The water boundary layer that surrounds the adsorbent particles is broken down through mixing. As a result, a layer is emerging from which solubilizing dyestuff molecules will be able to resist sorbent diffusion. The molecules of the dyestuff can be held on the adsorbent's surface and pores when this layer is lifted from the center.

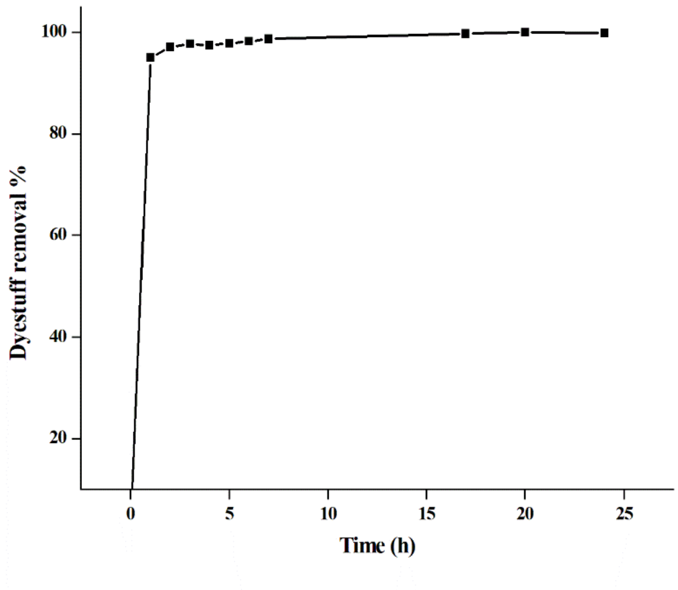

Fig. 7 The plot of contact time versus dyestuff removal

To make the mechanism of the adsorption process more descriptive, pseudo-first-order and pseudo-second-order kinetic models were investigated. Lagergreen's equation provides a basic pseudo-first order equation (Ho, 2004):

$$\log(q_e - q_t) = \log q_e - \frac{k_1}{2{,}303}.t \quad (6)$$

where k1 is the rate constant od adsorption (h−1) and qe and qt are the amounts of dyestuff adsorbed (mg/g) at equilibrium time and at time t (h), respectively. For olive leaf, the value of k1 was determined using the log(qe-qt) vs. t plot (Fig.8).

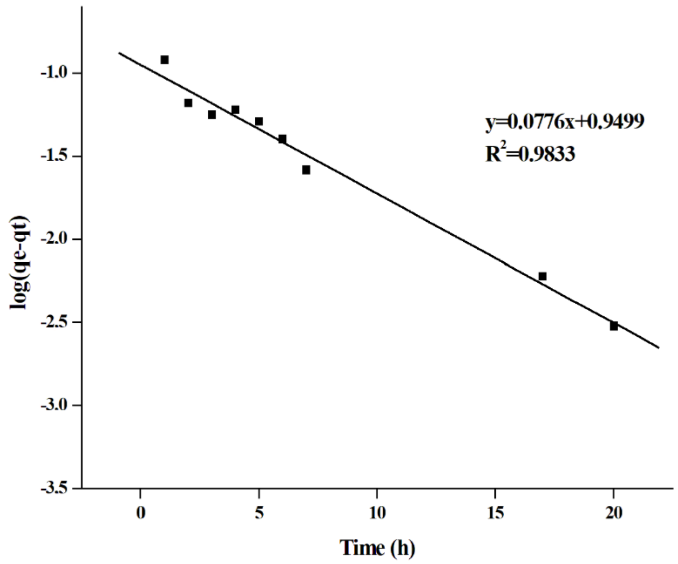

Fig. 8 The plot of pseudo first-order model for olive leaf

Adsorption equilibrium capacity-based pseudo-second order equations may be expressed as follows (Ho, 2003):

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

the pseudo-second order rate constant (g/mg min) is represented by k2. Experimentally, the slope and intercept of the t/qt vs t plot can be used to calculate the equilibrium adsorption capacity (qe) and second order constants (k2). (Fig. 9).

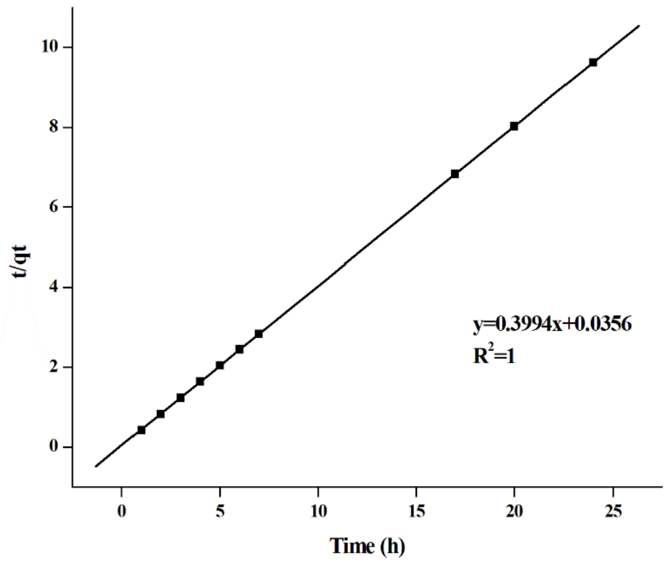

Fig. 9 The plot of pseudo second-order model for olive leaf

Table 2. Kinetic constants

q_{exp}	Pseudo-first-order			Pseudo-second-order		
2.49	K_1	q_e	R^2	K_2	q_e	R^2
	0.18	0.11	0.9833	4.49	2.5	1

Table 2 lists each constant and the models' linear regression coefficient values. In comparison to a second-order kinetic model, the regression coefficient for a first-order kinetic model is lower. Additionally, the calculated and experimental qe values are not coincide. The findings show that adsorption data cannot adequately characterize a first-order kinetic model. The regression coefficient for the second-order kinetic model is 1. Furthermore, the computed and experimental values of qe coincide fairly well. As a result, it can be seen that the adsorption mechanism is justified by the pseudo-second order kinetic model. Other workers reported experiencing similar outcomes. (Öztürk & Bektaş, 2004).

3.6 Adsorption isotherms

The relationship between the equilibrium concentration attained and the amount of material adsorbed per unit adsorbent in the study was used to analyze equilibrium isotherm models. It was determined that the equilibrium data obtained were compatible with the Freundlich and Langmuir isotherm models.
The Freundlich equation is shown by the equation that follows (Weber 1972):

$$\log q_e = \log K_f + \frac{1}{n} \log C_e \quad (8)$$

K (mg/g) which stands for the Freundlich intensity constant, and n, which stands for the Freundlich capacity constant. A plot of linear Freundlich equation logCe versus logqe is shown in Fig. 10.

The Langmuir equation was applied for the adsorption equilibrium of the olive leaf (Weber 1972):

$$\frac{C_e}{q_e} = \frac{1}{q_m.K_L} + \frac{Ce}{q_m} \quad (9)$$

where Ce is the equilibrium concentration (mg/L), qe is the amount of dyestuff adsorbed at equilibrium (mg/g), qm and KL are adsorbent's maximum capacity and Langmuir equilibrium constant, respectively. A plot of linear Langmuir equation Ce versus Ce/qe is illustrated in Fig. 11.

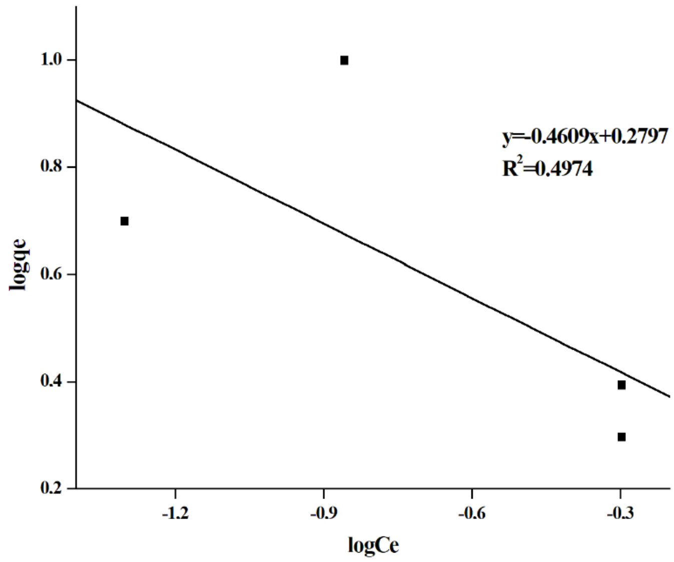

Fig. 10 The graph of Freundlich isotherm for olive leaf

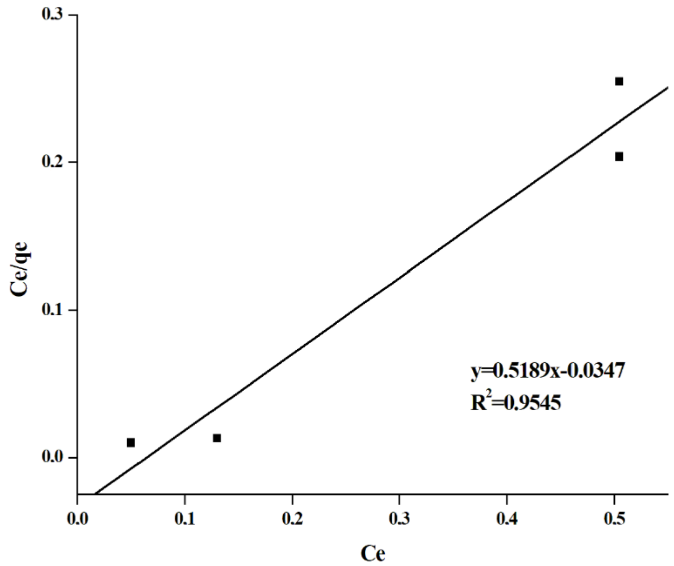

Fig. 11 The graph of the Langmuir isotherm for olive leaf

Table 3 gives the isotherm constants. According to the correlation coefficients, Langmuir isotherm model is the most suitable model.

Table 3. Isotherm constants

Isotherm	Freundlich Isotherm		Langmuir Isotherm	
Constant	n	K_f	q_m	K_L
	-2,1696	1,904	1,927	-14,95

4. Conclusions

In this study, the amount of adsorbent, the effect of initial pH, contact time and temperature parameters on dyestuff removal in textile wastewater were investigated. In the adsorption process in which olive leaf was used as adsorbent, it was observed that the removal of dyestuff increased as the pH value decreased. As a result, the optimum pH value was determined as 2. Another parameter is the amount of adsorbent. While examining this parameter, no discernible difference was found on the dyestuff removal of the adsorbent used in different amounts. Considering the cost issue, the optimum amount of adsorbent is 0.25 g, which is the lowest amount. When the effect of contact time was investigated, it was found that the dyestuff removal rose for 2 hours and then stayed constant. The optimum contact time was established as 2 hours since the adsorption reached equilibrium after 2 hours. The optimum adsorption temperature was found to be 25ºC when the effect of temperature was investigated. It was found that there was a direct proportionality between the temperature and the removal of the dyestuff.

This study revealed that the adsorption of olive leaf conforms to Langmuir isotherms. When the experiments were analyzed kinetically, it was determined that the olive leaf adsorption conformed to the pseudo-second-order rate equations. When adsorption thermodynamics is investigated, it was found that the reaction was exothermic for olive leaf adsorption for the reason $\Delta H^o>0$. This value is quite high and indicates that the adsorption is chemical adsorption. A negative value of ΔG^o indicates that the reaction takes place spontaneously. SEM images (Fig. 1a, Fig. 1b) of the olive leaf also support that the pore capacity and surface area of the olive leaf are quite suitable for adsorption. While the surface was quite rough before the adsorption process, the rough surface was covered with layers of dyestuff after the adsorption process.

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