



Removal of Safranin Dye from Aqueous Solution by Adsorption onto Olive Leaves Powder

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Abstract

The availability of quality water for drinking purposes is essential for the health and well-being of humans. The discharge of effluents containing dyes to water bodies increases the toxicity resulting in environmental pollution thereof. The treatment of water contaminated by dyes is thus essential. However, due to many dyes' intricate structure, the treatment of water polluted by these chemicals is difficult and expensive. In this study, olive leaves powder (OLP) suitability to act as an adsorbent for the removal of safranin, a cationic dye, from aqueous solutions was tested. The removal efficiency of safranin by OLP was tested by varying the contact time, pH, initial dye concentration, and the adsorbent dose at which the process was allowed to occur. The optimum conditions obtained for OLP adsorbent were 40 min contact time and 5.0 pH value. The Langmuir and Freundlich isotherm models best described the adsorption, with correlation coefficient (R^2) values of 0.9577 and 0.9581, respectively. The adsorption behavior was described using the pseudo-first and pseudo-second order kinetic models. The experimental data determined that the adsorption of safranin dye interpreted the pseudo-second order kinetics model ($R^2 = 0.998$). It was found that olive leaves powder may be considered adequate for the removal of cationic dyes, such as safranin, from wastewater.

1. Introduction

Many industries (textiles, stationery, plastic, food, etc.) are large water consumers and use synthetic dyes to colour their products. Most dyes are designed to be recalcitrant to environmental conditions such as light, temperature, microbial attack, and oxidizing agents [1]. Moreover, their presence in the water system, even at low concentrations, is evident, reduces light penetration, and has a detrimental effect on photosynthesis [2].

Several conventional methods are used for textile wastewater treatment, generally classified as biological, chemical and physical treatments [3-5]. Many conventional techniques like electrochemical degradation of the colour groups, chemical oxidation, coagulation, photocatalytic, ion exchange, and adsorption [6-12] have been applied to remove dyes. Among these water treatment techniques, the adsorption process provides an attractive method because of high efficiency, simplicity, and cost efficiency [13-16]. A biosorbent is defined as an adsorbent which consists of biomass [17]. Biosorption provides a cost-effective and safe way for the treatment of wastewater. Biosorption is economical relative to many other available treatment options, as the preparation of adsorbents from bio-waste is

moderately inexpensive, and the generated adsorbent is reusable in many instances [17, 18]. Compared to many other wastewater treatment options, biosorption is beneficial as it is uncomplicated, produces less waste and is generally very efficient in removing contaminants from water. Fewer chemicals are required than in the case with chemical treatment, which reduces the chances of environmental pollution caused by the substances [18-22]. This study aimed to investigate the efficiency of olive leaves powder for the adsorption of safranin from aqueous solution. The effect of contact time, pH, initial concentration, and adsorbent dose on the removal efficiency was studied under continuous stirring. The results were modelled with various isotherms and kinetic models.

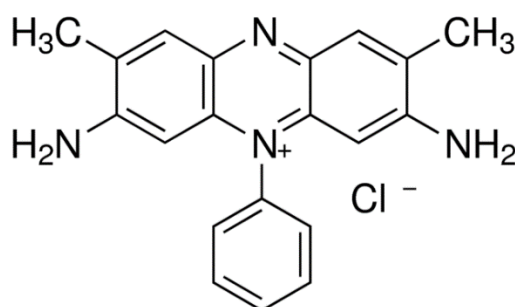


Figure 1: Structure of safranin

2. Material and Methods

2.1. Reagents

All chemicals used were of analytical reagent (AR) grade. 1000 ppm safranin stock solution was prepared by dissolving an appropriate amount of safranin salt in deionized water. The stock solution was diluted to the required concentrations using deionized water. The safranin used for experiments was obtained from Winlab, UK. The structure of the safranin dye is shown in Figure 1. The solution pH was adjusted using 0.10 M HCl or 0.10 M NaOH.

2.2. Determination of Safranin Dye

The concentration of safranin dye in the solutions before and after equilibrium was determined by Molecular Absorption Spectrophotometer 6305 from JENWAY. And, the pH of the solution was measured with pH Meter 3505 from JENWAY. The range of calibration curve concentrations of safranin prepared from the stock solution varies between 2-16 ppm.

2.3. Preparation of Adsorbents

The olive leaves were collected from the Msallata area in western Libya, washed with double distilled water, and dried in an oven at 70°C for 24 hrs. The dried samples were powdered using a sterilized blade and stored until further analysis. The dried powder materials were sieved through 125 µm size fractions using an American Society for Testing and Materials (ASTM) standard sieve.

2.4. Adsorption Experiments

The adsorption experiments were carried out in a series of 150 mL Erlenmeyer flasks containing 50 ml of safranin dye solution, 0.100 g adsorbent powder, and if necessary, an appropriate volume of HCl or NaOH solutions was used to adjust the pH of the solution. The solutions were shaken (150 rpm) at 25°C. Then solutions were filtered by Whatman filter paper. The removal percentage (% R) was calculated according to the following equation [21]:

$$\% R = \frac{c_o - c_e}{c_o} \times 100 \quad (1)$$

Where: C_0 and C_e are initial and final concentrations in ppm, respectively. The amount of adsorbed dye Q_e mg/g (mg dye per gram adsorbent) was calculated based on the difference between the initial (C_0 , ppm) and final concentration (C_e , ppm) in every flask, as follows [21]:

$$Q_e = \frac{C_0 - C_e}{M} \times V \quad (2)$$

Where Q_e is the dye uptake capacity (mg/g), V the volume of the dye solution in the flask (L) and M is the dry mass of biosorbent (g).

2.5. Parameters Affecting Adsorption Experiments

The effects of experimental parameters such as pH, adsorbent dose, contact time, and dye concentration were investigated for safranin adsorption onto olive leaves powders. The pH dependent study was carried out in 50 ml aqueous dye solution of 75 ppm and 0.10 g powder. The investigated pH values were from 1.0 to 10.0. The effect of contact time on dye adsorption was examined for 50 ml solutions with 50 ppm concentration and containing 0.10 g powder. The effect of adsorbent doses on dye uptake was investigated with adsorbent masses of 0.02, 0.04, 0.06, 0.08, 1.0 and 0.12 g per 50 ml of 50 ppm dye solution. Finally, various safranin dye concentrations were investigated to study the effect of dye concentration: 25, 50, 75, 100, 125, 150, 175, 200, 225, and 250 ppm per 50 ml solution.

3. Results and discussion

By UV. VIS. Spectrophotometer, the concentrations of safranin dye in the solutions were determined. The range of calibration curve of the dye prepared from the stock solution varies between 2-16 ppm, as shown in Figures 2 & 3. The safranin dye response was linear in the investigation concentration range at $\lambda_{max} = 520$ nm, and the linear regression equation was $y = 0.0919X$ with a high correlation coefficient ($R^2 = 0.999$). From the calibration curve, the concentrations of safranin in the solutions before and after equilibrium adsorption were determined.

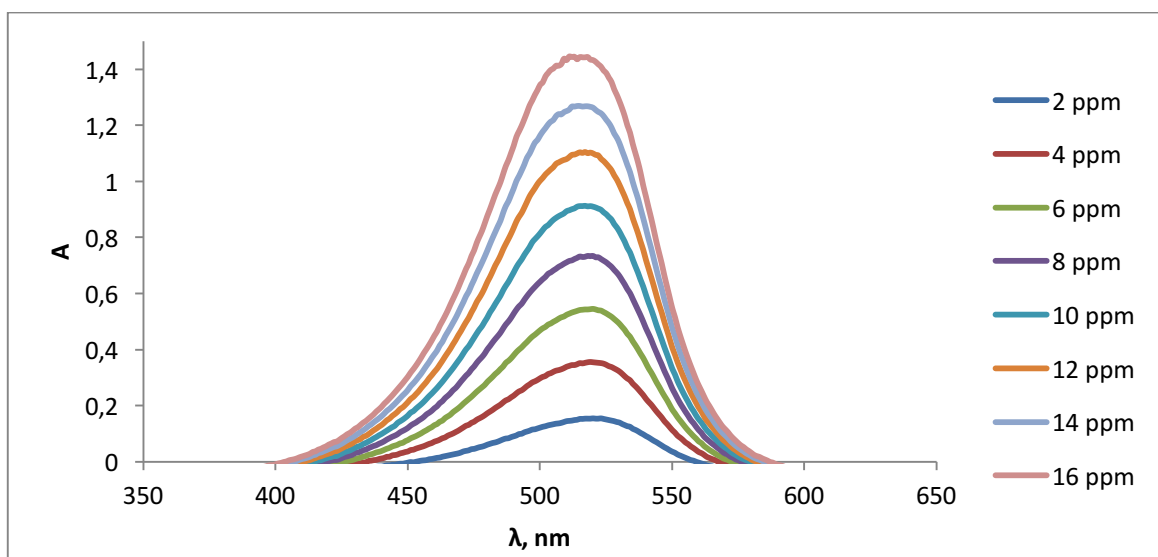


Figure 2: Absorption Spectrum of safranin at different concentrations

3.1. Effect of pH

The adsorption of safranin dye from aqueous solutions is strongly affected by the pH. The pH is considered to be the most critical parameter governing dye uptake by the adsorbent substrate. The adsorption of safranin dye on (OLP) was monitored over a range of pH from 1.0 to 10.0 of individual solutions as shown in Figure 4. Low percent removal was observed at low pH values (< 3.5). The

removal efficiency of safranin reached the maximum values of 99.24% at pH 6.5. When pH ranged between 4.0 and 10.0, safranin's removal efficiency remained approximately constant, reaching 97 %, at a pH value of 10.0.

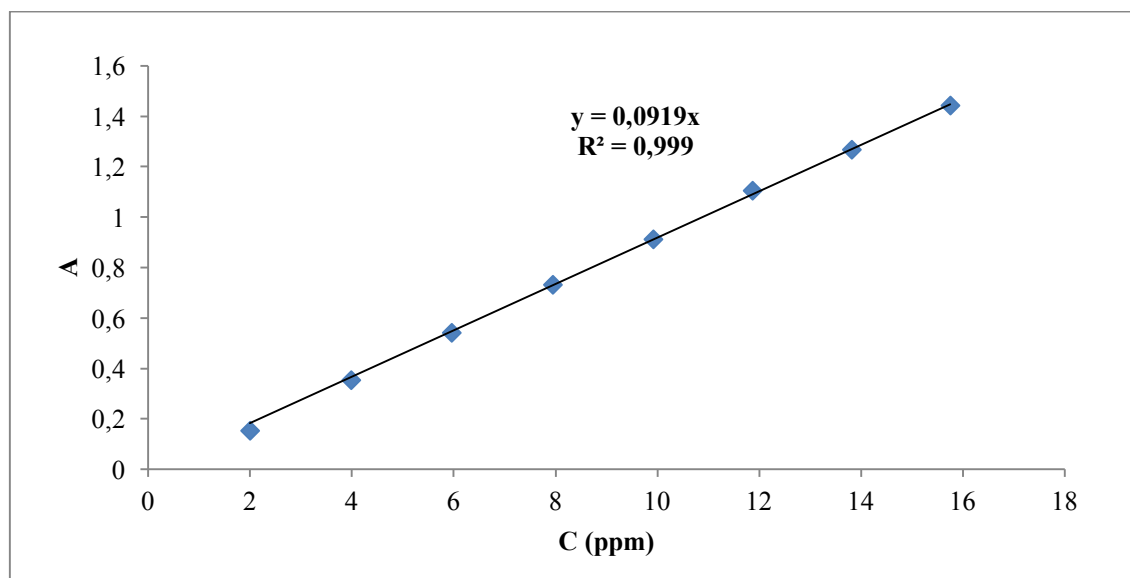


Figure 3: Calibration curve of safranin

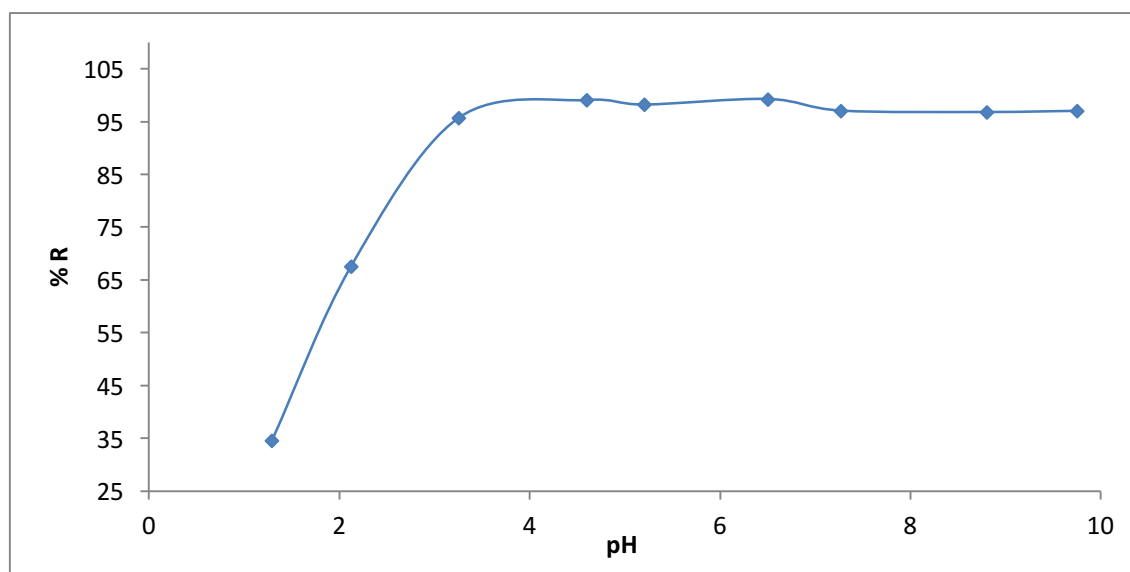


Figure 4: Effect of pH on percent removal of safranin by OLP

If electrostatic interaction was the only mechanism for the dye adsorption, then the removal efficiency should be maximum within the weakly acidic and weakly alkaline ranges pH 5 – 9. In this pH range, olive leaves powder's surface is negatively charged, and the dyes are positively charged (pK_a of safranin 6.4) at solution pH < 5. The removal efficiency was expected to decrease due to competition between H^+ and positively charged safranin cations for vacant adsorption sites triggered a reduction in safranin uptake due to a large amount of H^+ ions [23]. At the pH solution > 5, the adsorbent surface was negatively charged by the OH^- ions. This resulted in an increase in the electrostatic interaction between the cationic safranin and OH^- ions, which caused safranin adsorption to increase. These results suggested that electrostatic attraction played a significant role in the adsorption mechanism of safranin onto OLP. The same results have been reported in other recent studies [23-25].

3.2. Effect of adsorbent dose

The adsorbent dosage of (OLP) is an essential parameter because this determines the maximum capacity of an adsorbent for a given dye concentration. The adsorption capacity for safranin molecules as a function of adsorbent dosage (OLP) was investigated, and Figure 5 shows that the dye uptake was decreased with the adsorbent dose up to 0.1 g. This result can be explained by the fact that the adsorption sites in (OLP) remain unsaturated during the adsorption reaction, whereas the number of sites available for adsorption increases by increasing the dose of (OLP). In the whole experiment, 0.1 g was taken as the optimum adsorbent dose because a maximum capacity was attained at this value.

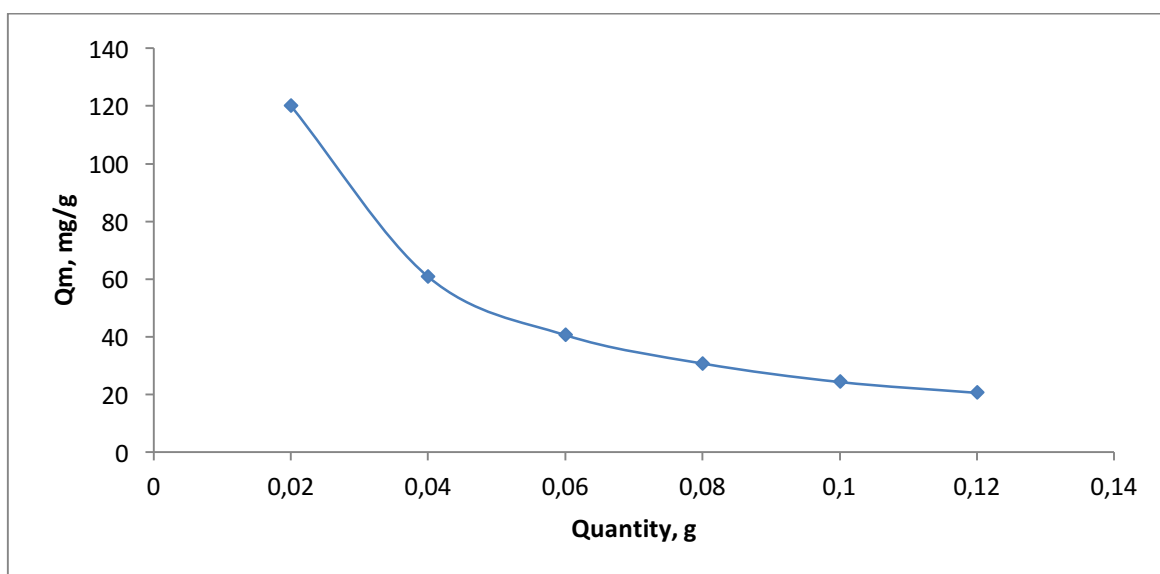


Figure 5: Effect of adsorbent dose on safranin uptake Q_m by OLP

3.2 Effect of initial concentration and adsorption isotherms

The effect of different initial concentrations of dye on the equilibrium of adsorption experiments onto (OLP) was investigated from 25 to 250 ppm at pH 5. The relation between equilibrium uptake Q_m ($\text{mg} \cdot \text{g}^{-1}$) with initial dye concentration is shown in Figure 6. The dye's Q_m was increased gradually with an increasing the initial concentration of safranin dye, as shown in Figure 6.

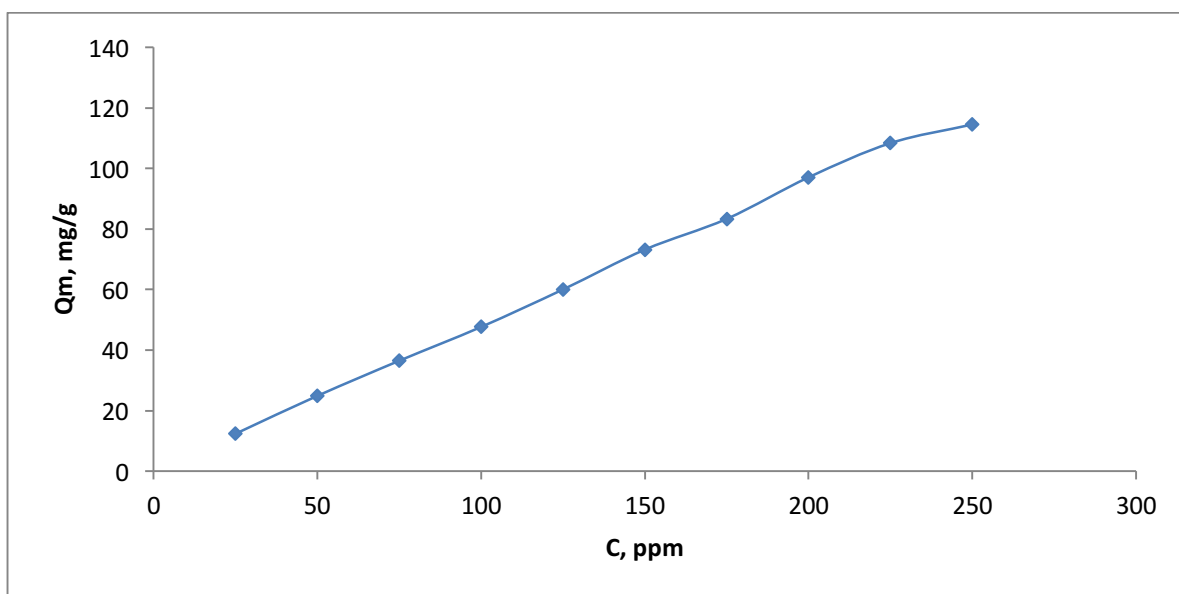


Figure 6: Effect of initial safranin concentration on dye uptake Q_m onto OLP

The dye molecules adsorption is possible at lower concentrations, but as the concentration is increased, the driving force also increased, which favored the adsorption at higher concentrations. The increase of adsorption capacity with the increase in dye concentration is probably due to higher interaction between the safranin cations and sequestering adsorbent sites.

3.3 Adsorption isotherms

Adsorption isotherms describe the fraction of sorbate molecules that are partitioned between liquid and solid phases at equilibrium. Adsorption of safranin onto olive leaves powder (OLP) was modeled using four adsorption isotherms.

3.3.1 The Langmuir isotherm

It assumes as monolayer adsorption on a uniform surface with a finite number of adsorption sites. Once a site is filled, no further sorption can take place at that site. As such, the surface will eventually reach a saturation point where the surface's maximum adsorption will be achieved. The linear form of the Langmuir isotherm model is described as [14, 15]:

$$\frac{1}{Q_e} = \frac{1}{Q_m} + \frac{1}{b Q_m C_e} \quad (3)$$

where b is the Langmuir constant related to the energy of adsorption ($L \cdot mg^{-1}$), and Q_m is the maximum biosorption capacity (the monolayer adsorption saturation capacity mg/g), Q_e (mg/g) the amount of dye molecules bound per gram of the adsorbent at equilibrium; and C_e , the residual dye concentration left in the solution after binding.

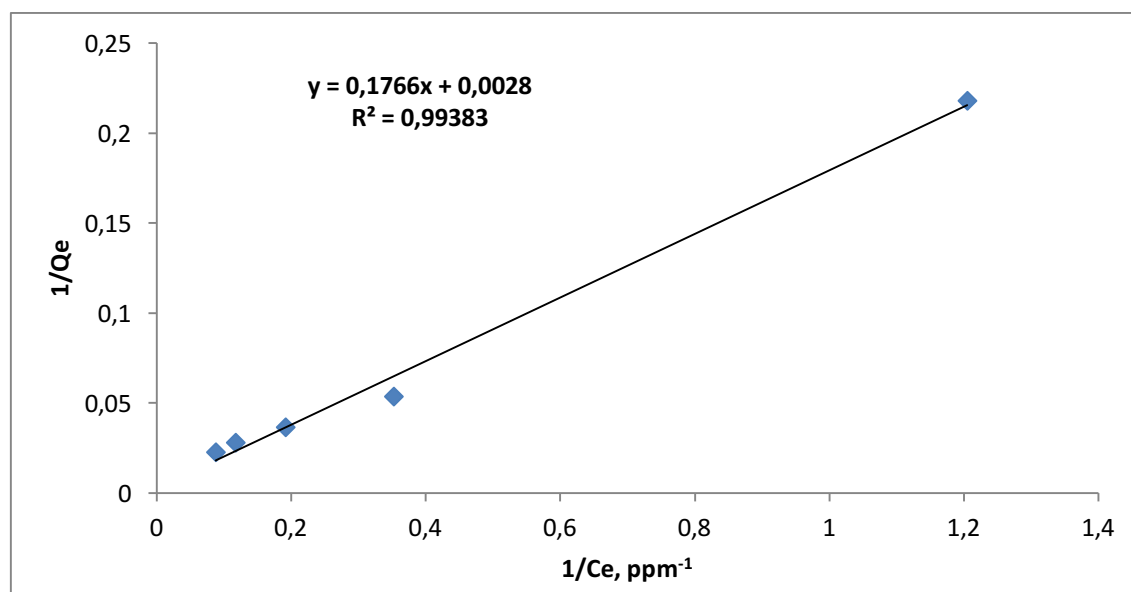


Figure 7: Langmuir adsorption isotherm for the removal of safranin by OLP

Values of Langmuir parameters Q_m and b were calculated from the slope and intercept of the linear plot of $1/Q_e$ versus $1/C_e$, as shown in Figure 7. Values of Q_m , b , and regression coefficient R^2 are listed in Table 1. These values indicated that the Langmuir model describes the adsorption phenomena favorable. The level of conformity for olive leaves as a good sorbent is high, according to the correlation coefficients (R^2) of 0.994. The essential characteristics of the Langmuir isotherm parameters can be used to predict the affinity between the sorbate and sorbent using separation factor or dimensionless equilibrium parameter, R_L expressed as in the following equation:

$$R_L = \frac{1}{1 + b C_0} \quad (4)$$

The value of R_L indicated the type of Langmuir isotherm to be irreversible ($R_L=0$), favorable ($0 < R_L < 1$), linear ($R_L=1$) or unfavorable ($R_L > 1$). In the concentration range of 25 to 250 mg/L of safranin dye, the values R_L of adsorbent (OLP) were found to be 0.558. This is in the range of 0.0-1.0, which indicates the favorable adsorption.

3.3.2 The Freundlich isotherm model

It is the well-known earliest relationship describing the adsorption process. This model applies to adsorption on heterogeneous surfaces with the interaction between adsorbed molecules and the Freundlich equation application. It also suggests that sorption energy exponentially decreases on completion of the sorption centers of an adsorbent. Heterogeneous systems can be described using this equation, which is an empirical equation and is expressed as follows in the linear form [14, 15]:

$$\text{Log } Q_e = \text{Log } K_f + \frac{1}{n} \text{Log } C_e \quad (5)$$

where $1/n$ is the heterogeneity factor, K_f is the Freundlich constant associated to the bonding energy, and n (g/L) is an amount of the deviation from the linearity of adsorption. Freundlich stability constants were determined from the plot of $\log Q_e$ versus $\log C_e$, as shown in Figure 8, based on the linear of the Freundlich equation. The degree of non-linearity between solution concentration and adsorption indicated by n value if $n = 1$, then adsorption is linear; $n < 1$, then adsorption is a chemical process; if $n > 1$, then adsorption is a physical process. The n value in the Freundlich equation was found to be 1.17 (Table 1). This indicates the physical adsorption of safranin molecules onto olive leaves adsorbate is suitable.

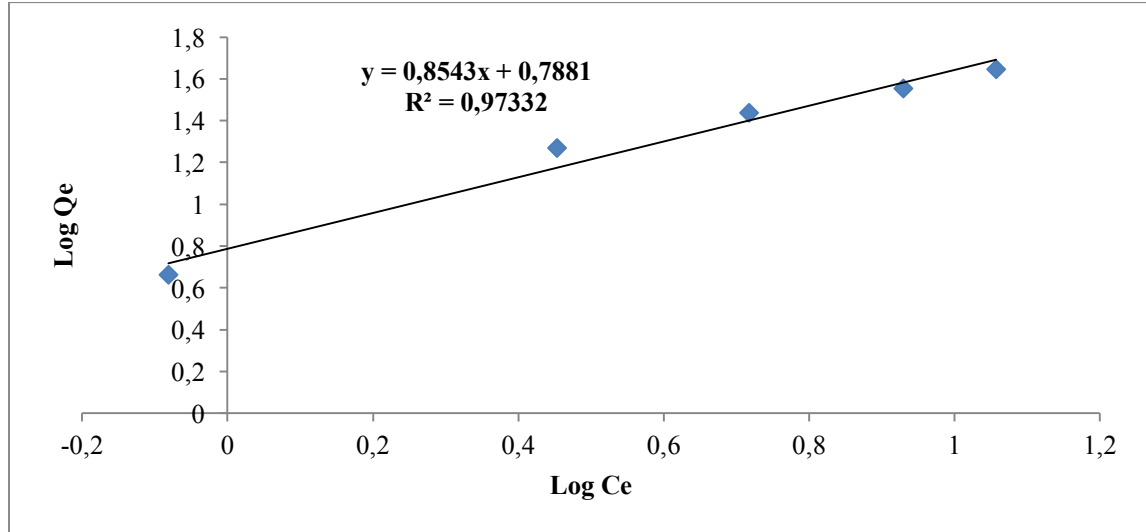


Figure 8: Freundlich adsorption isotherm for the removal of safranin by adsorption onto OLP

3.3.3 Temkin isotherm

Equation 6 [26], the adsorption heat of all particles in the layer is assumed to decrease linearly with coverage. This is due to adsorbent-adsorbent interactions. The absorption is characterized by a uniform distribution of the binding energies reach some of the maximum binding energy. Timken isotherm has been used in the linear form as follows:

$$Q_e = B \text{Log } A + B \text{Log } C_e \quad (6)$$

The plot of Q_e versus $\log C_e$ enables the isotherm constants B and A Obtained from Slope and Intersection, **Figure 9**. A (L/mg) is the equilibrium binding constant corresponding to the maximum and constant binding energy B related to the adsorption temperature, **Table 1**. Results in **Table 1** reveal that Langmuir isotherm is the most fitting and appropriate model to explain safranin's adsorption with $R^2 = 0.994$. Nevertheless, the R^2 values for Freundlich and Temkin isotherms (0.973 and 0.974) are still very close to that of the Langmuir model.

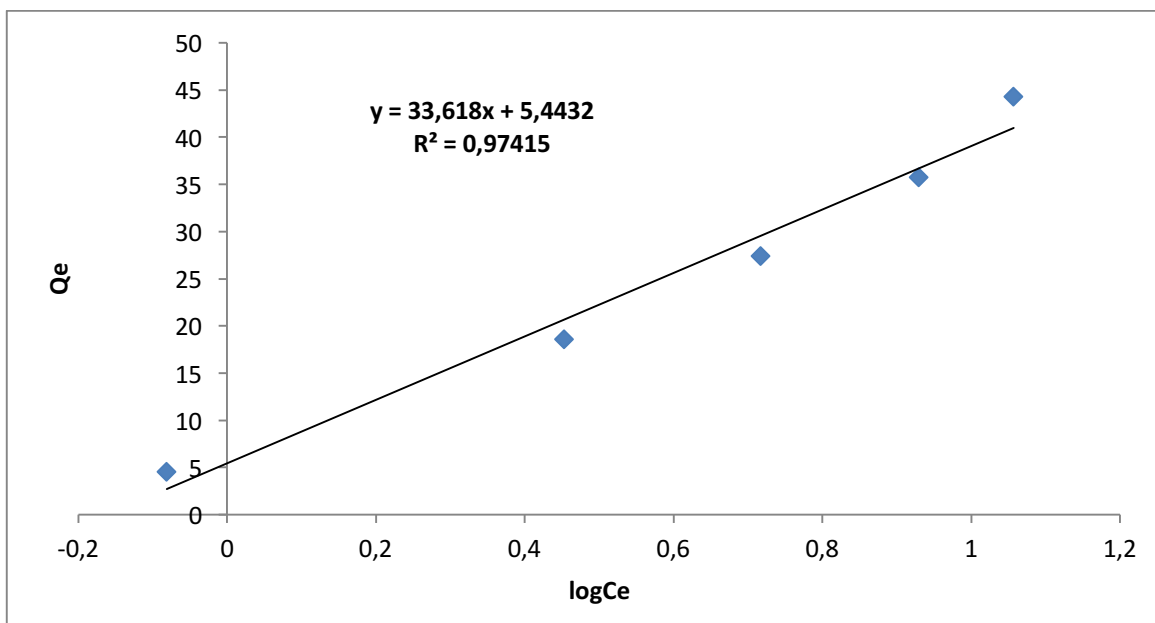


Figure 9: Temkin adsorption isotherm for the removal of safranin by adsorption on OLP

3.3.4 Dubinin-Radushkevich (D-R) model (**Figure 10**)

This model does not assume a homogenous surface or a constant biosorption potential as the Langmuir model, and it was also used to test the experimental data [27]:

$$\text{Log } Q_e = \text{Log } Q_d - \beta \varepsilon^2 \quad (7)$$

Where ε can be correlated to C_e (mg/L) by the following equation:

$$\varepsilon = RT \text{Log} \left(1 + \frac{1}{C_e} \right) \quad (8)$$

Where R is an ideal gas constant, and T is the absolute temperature (K). The D-R isotherm β and Q_d were obtained from the slope and intercept of the plot of $\log Q_e$ versus ε^2 , **Figure 10**. List the calculated values in **Table 1**.

The values of the correlation coefficient were lower than the other three correlation coefficient values (0.769). The D-R model represents the least favorable to the experimental data than other isothermal models. The mean free energy of adsorption, E is defined as the free energy change when 1 mole of ions is transported to the surface of a solid from infinity in solution and is calculated from the value of β using the following equation:

$$E = \frac{1}{\sqrt{2\beta}} \quad (9)$$

If the magnitude of E is between 8 to 16 KJ/mol then the sorption process is supposed to proceed via chemisorption reaction, while for values of E is less than 8 kJ/mol, the sorption process is of physical nature. The value of E calculated from equation (9) for the adsorption of safranin dye by olive leaves powder is 0.707 KJ/mol. This indicates that the adsorption process is of physical nature.

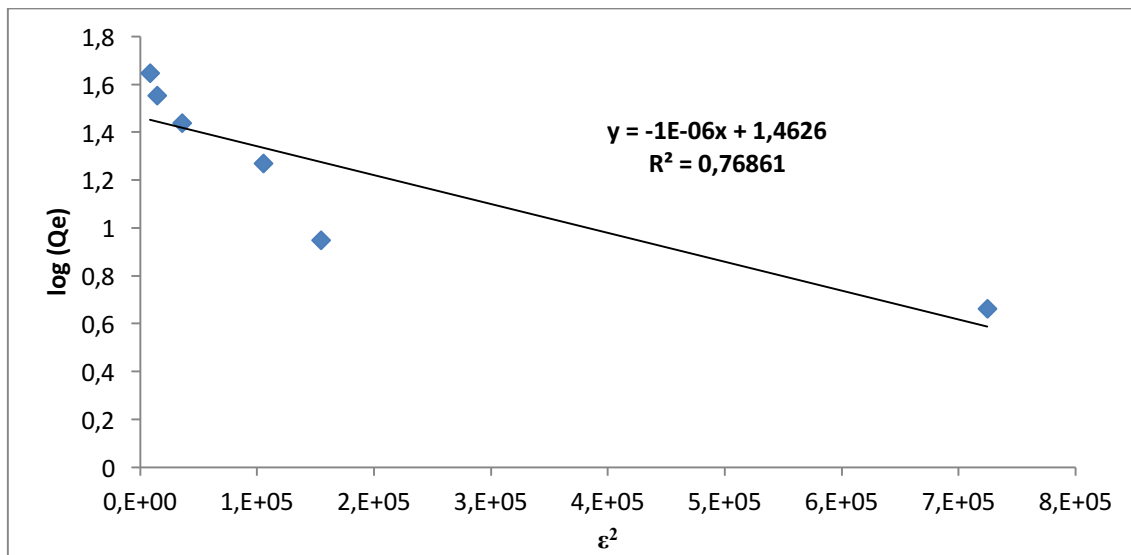


Figure 10: Dubinin-Radushkevich (D-R) adsorption isotherm for the removal of safranin by adsorption on OLP

Table 1: Langmuir, Freundlich, D-R and Temkin isotherm constants for biosorption of metal ions onto WTCP

Langmuir	Q_m	b	R_L	R^2
	357.14	0.0159	0.558	0.994
Freundlich	K_f	n		R^2
	6.14	1.17		0.973
Temkin	B	A		R^2
	33.62	1.45		0.974
Dubinin-Radushkevich	Q_d	β		R^2
	170.37	1×10^{-6}		0.769

3.4 Effect of contact time and adsorption kinetics

The rate of adsorption is important for designing batch adsorption experiments. Therefore, the effect of contact time of safranin dye adsorption on (OLP) was investigated. Figure 11 shows that the adsorption of safranin molecules on (OLP) was increased considerably until the contact time reached 40 min at 25°C. Further increase in contact time did not enhance the adsorption, so the optimum contact time was selected as 40 min for further adsorption experiments.

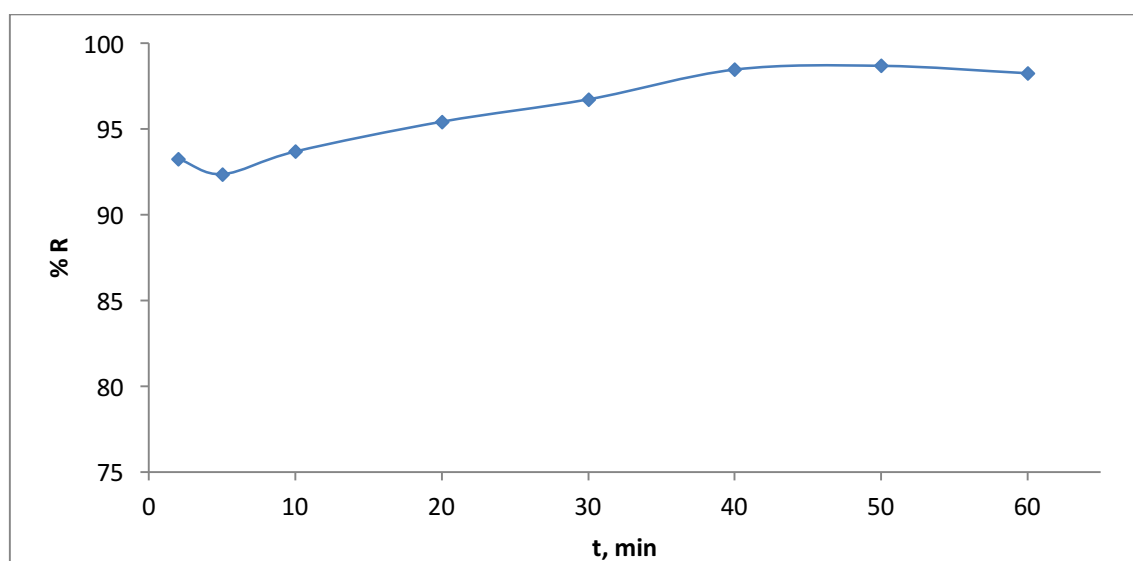


Figure 11: Effect of contact time on percent removal of safranin by OLP

3.4.1 Adsorption Kinetics

To examine the adsorption kinetics of dye uptake onto (OLP), the two kinetic models (pseudo first-order and pseudo second-order) were fitted to our experimental data.

The pseudo-first order equation of Lagergren [23] is generally expressed as follows:

$$\frac{dQ_t}{dt} = k_1 (Q_e - Q_t) \quad (10)$$

Where Q_e and Q_t are the sorption capacities at equilibrium and at time t , respectively and k_1 is the rate constant of pseudo-first order sorption. The integrated form of equation (10) at boundary conditions, from $Q_t=0$ to Q_t , and $t=0$ to t ; becomes:

$$\text{Log} (Q_e - Q_t) = \text{Log} Q_e - k_1 t \quad (11)$$

To fit the experimental data by an integrated form of pseudo-first order equation, the equilibrium sorption capacity, Q_e must be known. For this reason, and for analyzing the pseudo-first order model kinetics, it is imperative to use trial and error to obtain the equilibrium sorption capacity. In most cases in the literature, the amount sorbed is still significantly smaller than the equilibrium amount, and the Lagergren equation does not fit well for the whole range of contact time process.

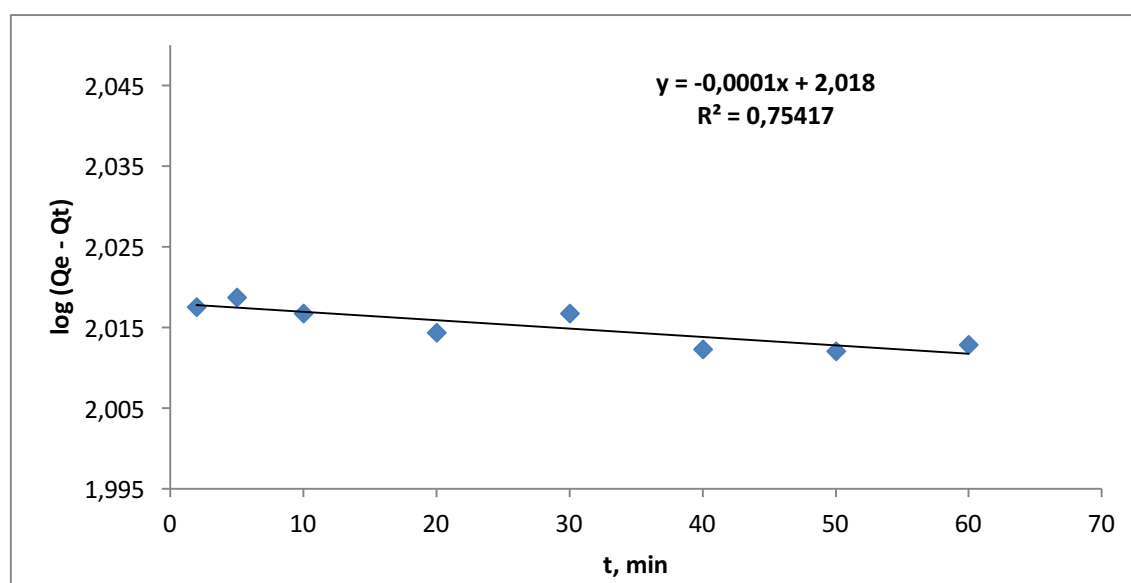


Figure 12: Pseudo first order kinetic plots for the removal of safranin by adsorption on OLP

The calculated values and their corresponding linear regression correlation coefficient values (calculated from Figure 12) are listed in Table 2. R^2 was found to be 0.754, which shows that this model cannot be applied to predict the adsorption kinetic model. The pseudo second-order rate expression, which has been applied for analyzing sorption kinetics rate, is expressed as [23]:

$$\frac{dQ_t}{dt} = k_2 (Q_e - Q_t) \quad (12)$$

For the boundary conditions from $Q_t=0$ to Q_t and $t=0$ to t ; the integrated form of equation becomes:

$$\frac{t}{Q_t} = \frac{1}{k_2 Q_e^2} + \frac{1}{Q_e} t \quad (13)$$

Where t is the contact time (min), Q_e and Q_t are the amount of the solute adsorbed at equilibrium and at any time t (mg/g) respectively and k_2 is the rate constant of pseudo-second order sorption, (g/mg.min). If the pseudo-second order kinetic properties are applicable, then the plot of t/Q_t versus t in the equation should give a linear relationship, from which Q_e and k_2 can be determined from the slope and intersection of the plot, Fig.13. The pseudo-second order demand rate constant k_2 is given, and the value of the calculated Q_e and the corresponding linear regression coefficient value for R^2 in Table 2.

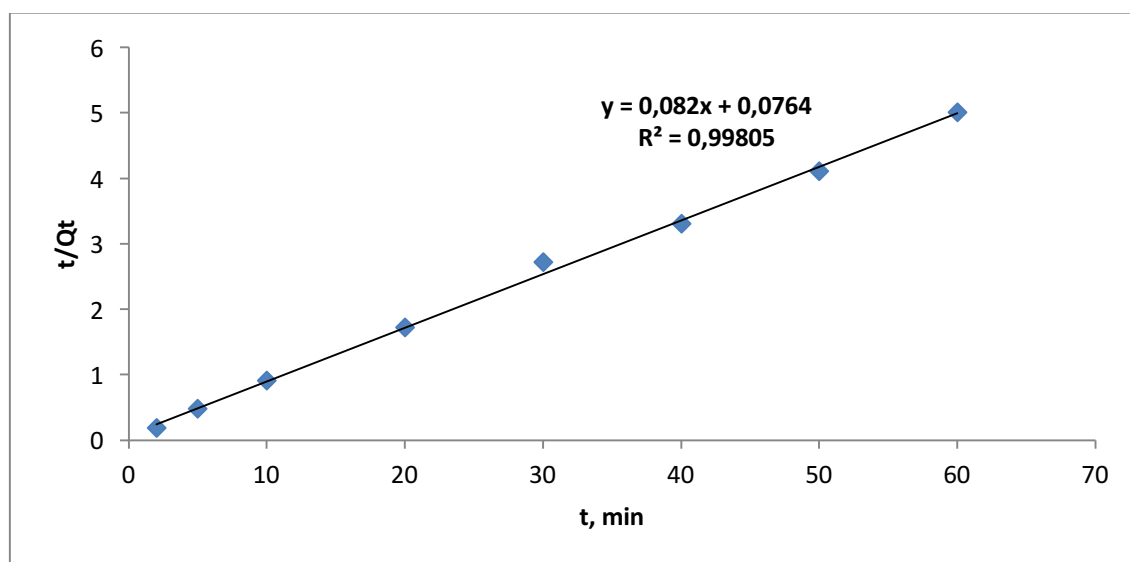


Figure 13: Second order kinetic plots for the removal of safranin by adsorption on OLP

In all primary mineral concentrations, the values of the linear correlation coefficient of R^2 were high (0.998). The higher values confirm that the adsorption data are better represented by pseudo-second order kinetics and the calculated Q_e values agreed with the estimated experimental Q_e values 90.1 (Table 1 and 2). This suggests that the adsorption of safranin dye follows pseudo-second order kinetics.

Table 2: Kinetic parameters of dye adsorption onto OLP

First Order	Q_e (mg/g)	k_1 (1/min)	R^2
	104.2	0.0001	0.754
Second Order	Q_e (mg/g)	k_2 (g/mg.min)	R^2
	Graphical*: 12.20 ; Estimated**: 12.12	0.0879	0.998

*Equation (13) ; ** Equation (2)

Conclusion

In our present work, we show that the olive leaves powder could be used as an adsorbent to remove safranin dye molecules from aqueous solutions. The removal efficiency and dye capacity were found to be dependent on pH, contact time, initial dye concentration, and adsorbent dose. Adsorption isothermal data could be well simulated by Langmuir, Freundlich, and Temkin models. The maximum metal capacities determined by using the Langmuir isotherm were 357.14 mg/g olive leaves powder. The values of dye uptake capacities at equilibrium Q_e calculated from equation (2) and pseudo-second-order plot were found comparable, 12.12 and 12.20 mg/g, respectively. The adsorption kinetics could be well predicted by pseudo-second-order kinetic. Our investigation results indicate that the olive leaves powder can remove safranin dye from aqueous solutions.

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