

Adsorptive removal of E110 food dye using chemically treated grape leaves: thermodynamic and isotherm analysis

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ABSTRACT

This study investigated the use of a new adsorbent material, chemically treated grape leaves, for the adsorption of the food dye E110 at temperatures of 298, 303, 308, 313, and 318 K. The removal efficiency ranged from 74.934% to 95.704%. Adsorption behavior was evaluated by determining the constants of several adsorption isotherm models, including Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich models. According to Beer–Lambert’s law, a standard calibration curve for E110 dye was prepared, and the residual dye concentration in solution was determined using UV–visible spectrophotometry. Thermodynamic functions were calculated from the adsorption isotherm constants. The positive ΔH values indicated that the adsorption of E110 dye was an endothermic process, while the thermodynamic results suggested that the adsorption was mainly physical in nature. The positive ΔG° values indicated that the adsorption process was non-spontaneous under the studied conditions, although it tended toward greater spontaneity with increasing temperature. The positive ΔS and ΔS° values indicated increased randomness at the solid–solution interface during adsorption. The higher ΔS values compared with ΔS° suggest that E110 dye molecules became less ordered during the adsorption process than at equilibrium, with greater freedom on the surface of the chemically treated grape leaves.

Keywords: Adsorption, Food dyes, E110 dye, Isotherms, Thermodynamics

1 INTRODUCTION

Waste management and product development are promising research areas because they involve the production of useful materials from waste and unused resources to meet the needs of the growing global population and reduce environmental pollution. These approaches are also attractive because of their low cost and wide availability [1]. The paint, pharmaceutical, printing, cosmetics, leather, paper, and plastic industries generate industrial dye residues that are harmful to the environment and human health [2].

Most food dyes are chemically stable and highly soluble in water because they contain sulfonic acid groups

and sodium salts, in addition to azo chromophore groups (N=N), sulfur-containing groups, aryl groups, and ionic rings [3]. The discharge of liquid waste containing food dyes is a serious concern because it can cause direct and indirect effects on human health and the environment [4]. Several studies have indicated that E110 dye may have toxic effects. For example, the presence of dyes in receiving water can prevent light penetration, thereby reducing photosynthesis. In addition, when E110 decomposes, a large amount of dissolved oxygen may be consumed, negatively affecting water quality standards and aquatic organisms, including fish [5].

The large volume of dye-containing effluents makes

wastewater treatment and compliance with environmental discharge standards a major challenge [6]. Several chemical, biological, and physical processes can be used to treat contaminated wastewater and remove pollutants. These techniques may be applied separately or in combination to achieve the best balance between efficiency, cost, and environmental quality [7]. Several methods have been used to remove hazardous wastes from industrial and domestic wastewater, including coagulation, filtration, reverse osmosis, oxidation, adsorption, and ion exchange [1, 8]. Among these methods, adsorption is considered an effective and economical process for removing E110 from industrial effluents [1].

Adsorption technology is widely used for water-pollution treatment because it is effective, simple, low-cost, has high adsorption capacity, and is less sensitive to toxic chemicals than some other treatment methods [9]. Depending on the value of ΔH° , the adsorption process can be classified as exothermic or endothermic. The sign and value of ΔG° indicate whether the adsorption process is spontaneous or non-spontaneous, whereas ΔS° reflects the degree of randomness of the adsorption system before and after adsorption [10]. Therefore, the aim of this study was to determine the thermodynamic functions of E110 adsorption using adsorption isotherm constants.

2 MATERIALS AND METHODS

All chemicals used in this study were obtained from BDH and Fluka. The E110 dye was supplied by local commercial sources. Sodium hydroxide, hydrochloric acid, and distilled water were also used in the experimental procedures.

2.1 Preparation of grape leaves

Grape leaves were prepared according to the method described in the literature [11]. Briefly, 150 g of grape leaves was washed with deionized water to remove dirt and mineral impurities. The leaves were then dried in an oven at approximately 150 °C for 2 h. The dried leaves were heated with 1.0 M hydrochloric acid on a magnetic stirrer at 100 °C until a paste-like material was formed. The material was then washed with deionized water and dried in an oven at approximately 80 °C to remove moisture. After drying, 100 g of treated grape leaves was ground to increase the surface area and sieved using a 150 μ m mesh sieve [12].

2.2 Instruments

The instruments used in this study included a T92+ UV–visible spectrophotometer and a Julabo SW 23 water-bath shaker for shaking the solutions.

2.3 Preparation of standard dye solutions

A standard stock solution of E110 dye was prepared by dissolving 0.1 g of E110 in 1 L of distilled water to obtain a concentration of 99.96 mg/L. Working solutions with concentrations of 9.0474, 18.0948, 27.1422, 36.1896, and 45.237 mg/L were prepared by dilution from the stock solution.

2.4 Analytical method

First, the maximum absorption wavelength of the dye, $\lambda_{\max}$, was determined. The remaining and adsorbed concentrations of E110 dye in solution were then determined spectrophotometrically after adsorption. A calibration curve was constructed by plotting absorbance against concentration. The best five dye concentrations were selected from the calibration curve. These solutions were shaken separately for 1 h, which was selected as the optimum adsorption time, using a programmed shaking device at 298, 303, 308, 313, and 318 K, respectively. The change in the amount of E110 adsorbed with time was monitored. Beer–Lambert's law was used to construct the calibration curve at $\lambda_{\max} = 526$ nm by plotting absorbance against dye concentration. The molar absorptivity coefficient was calculated using the following equation:

$$A = \epsilon LC \quad (1)$$

where A is the absorbance of the dye solution before adsorption, ϵ is the molar absorptivity coefficient ($L/mol \cdot cm$), C is the concentration of E 110 dye (M), and L is the cell path length (1 cm).

2.5 Batch experiment

Dye stock solutions were first prepared for adsorption experiments using E110 food dye [13]. Then, 50 mL of dye solutions with different concentrations were prepared from the stock solution and placed in flasks. The grape-leaf adsorbent was added to each dye solution [14, 15]. The flasks were stirred at 100 rpm in a water-bath shaker [11, 12]. After 1 h of shaking, the remaining dye concentration was determined using a spectrophotometer at the optimum wavelength of 526 nm. The solutions were then filtered, and absorbance values were recorded after adsorption.

The equilibrium concentration, adsorbed dye concentration, adsorption capacity, and removal efficiency were estimated using the following equations [16]:

$$C_e = A/\varepsilon \quad (2)$$

$$C_0 = C_i - C_e \quad (3)$$

$$q_e = [(C_0 - C_e) / m] \times V \quad (4)$$

$$\text{Removal efficiency (\%)} = [(C_0 - C_e) / C_0] \times 100 \quad (5)$$

where A is the absorbance after adsorption, C_i is the initial dye concentration (mg/L), C_0 is the adsorbed dye concentration (mg/L), C_e is the equilibrium dye concentration (mg/L), m is the mass of grape-leaf adsorbent added (g), q_e is the adsorption capacity, and V is the volume of solution (L).

3 RESULTS AND DISCUSSION

Initially, the optimum weight of the solid adsorbent, chemically treated grape leaves, was determined by conducting several experiments using different adsorbent weights at a fixed temperature and a constant E110 dye concentration. The adsorption capacity and removal efficiency were calculated using Equations (3), (4), and (5). The results are shown in Table 1. The optimum contact time was determined using the selected weight of the solid adsorbent, as shown in Table 2.

3.1 Effects of initial dye concentration and temperature

The effects of initial dye concentration and temperature on the adsorption process are shown in Table 3. The thermodynamic parameters were used to explain the differences in E110 dye adsorption efficiency at different temperatures and to evaluate the type and nature of adsorption on the surface of chemically treated grape leaves. The results showed that adsorption efficiency and adsorption capacity increased with increasing temperature. This may be attributed to the increased mobility and transfer rate of E110 dye molecules from the solution to the grape-leaf surface.

3.2 Adsorption isotherms

Several adsorption isotherm models were applied to determine the type and nature of E110 adsorption on the surface of chemically treated grape leaves.

3.3 Langmuir adsorption isotherm

This model represents the equilibrium distribution of adsorbate molecules between the solid and liquid phases [17]. The Langmuir model assumes uniform adsorption energies on the adsorbent surface and no migration of adsorbate molecules along the surface. Based on these assumptions, the Langmuir isotherm describes monolayer adsorption on a homogeneous surface with equal affinity and energy for all binding sites [18]. Langmuir adsorption parameters were calculated using the following equations:

$$C_e/q_e = 1/(b \times Q_{\max}) + (1/Q_{\max})C_e \quad (6)$$

$$KL = b \times Q_{\max} \quad (7)$$

where $Q_{\max}$ is the maximum monolayer adsorption capacity (mg/g), KL is the Langmuir isotherm constant (L/mg), and b is the Langmuir adsorption coefficient.

The relationship between C_e/q_e and C_e gives a linear plot with a slope of $1/Q_{\max}$ and an intercept of $1/(b \times Q_{\max})$, as shown in Figure 1.

Table 1 The relation between the weight of the grape leaf and percentage of adsorption, using (2x10-2M) E110 contact time is 60min. at 25 C°

gm	Ci mg/L	Ce mg/L	C _{ads} mg/L	qe mg/g	%
0.01	9.047	9.046	0.001	0.003	0.007
0.05	9.047	8.664	0.383	0.383	4.235
0.07	9.047	8.450	0.596	0.426	6.596
0.1	9.047	7.959	1.088	0.544	12.025
*0.5	9.047	2.039	7.008	0.700	77.458

Note. *The best weight to contact

Table 2 Contact time and percentage of adsorption for E110 dye on grape leaf surface at 40C°, 0.5gm, (Ci=9.0474 mg/L)

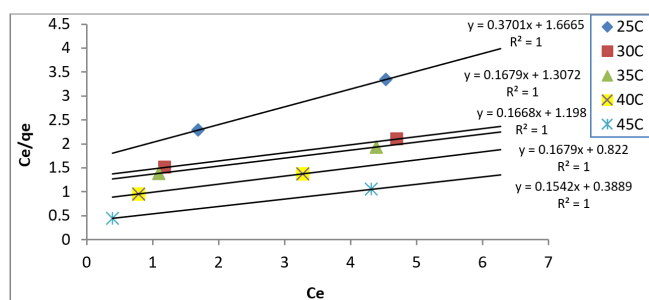
t. min	Ce mg/L	C _{ads} mg/L	qe mg/g	%
5	3.411	5.636	0.563	62.295
15	2.352	6.695	0.669	73.996
25	1.960	7.087	0.707	78.3307
35	1.647	7.400	0.740	81.798
45	1.529	7.518	0.751	83.099
**60	1.019	8.028	0.802	88.732
70	1.019	8.028	0.802	88.732
80	1.529	7.518	0.751	83.099

Note. ** The best time to contact

Table 3 Effect of concentration and temperature on the adsorption process

T ko	Ci mg/L	Ce mg/L	C _{ads} mg/L	qe mg/g	Kc	%	Σ ²
298	9.0474	1.686	7.361	0.736	4.365	81.363	1331107
	18.094	4.535	13.559	1.355	2.989	74.934	243688
	27.142	5.175	21.966	2.196	4.244	80.930	191537
	36.189	5.803	30.386	3.038	5.235	83.963	155132
	45.237	6.273	38.963	3.896	6.210	86.131	134270
303	9.0474	1.184	7.863	0.786	6.640	86.911	2378300
	18.094	4.113	13.981	1.398	3.399	77.269	300578
	27.142	4.705	22.436	2.243	4.7684	82.664	235665
	36.189	5.097	31.092	3.109	6.0996	85.914	203634
	45.237	5.803	39.433	3.943	6.7953	87.171	160407
308	9.047	1.097	7.949	0.794	7.2406	87.865	2749485
	18.094	3.851	14.243	1.424	3.6978	78.713	349339
	27.142	4.391	22.750	2.275	5.1804	83.820	275967
	36.189	4.746	31.443	3.144	6.6243	86.884	239665
	45.237	5.175	40.061	4.006	7.7400	88.558	204607
313	9.047	1.019	8.028	0.802	7.875	88.732	4544670
	18.094	2.111	15.983	1.598	7.569	88.330	775440
	27.142	3.274	23.868	2.386	7.290	87.937	481089
	36.189	4.387	31.802	3.180	7.248	87.876	354500
	45.237	4.862	40.374	4.037	8.304	89.251	236900
318	9.047	0.388	8.658	0.865	22.282	95.704	11336336
	18.094	1.965	16.129	1.612	8.205	89.136	1182271
	27.142	2.748	24.393	2.439	8.8749	89.873	672995
	36.189	3.454	32.735	3.273	9.4760	90.454	451806
	45.237	4.313	40.923	4.092	9.4880	90.465	303910

Note. Σ = Potential energy, the parameter can be determined by the equation (12).

**Fig. 1** Langmuir adsorption isotherm

$$RL = 1/(1 + bCi) \quad (8)$$

The RL values shown in Table 4 indicate the nature of the adsorption process. Adsorption is considered unfavorable when $RL > 1$, linear when $RL = 1$, favorable when $0 < RL < 1$, and irreversible when $RL = 0$.

Table 4 Values of (RL) at different concentrations and temperatures, using 0.5 g of grape leaf, and contact time equal to 60 min.

Ci	RL				
	Tko				
	298	303	308	313	318
9.0474	0.332	0.462	0.442	0.351	0.217
18.0948	0.199	0.300	0.284	0.212	0.122
27.1422	0.142	0.222	0.209	0.152	0.085
36.1896 ^c	0.110	0.177	0.165	0.119	0.065
45.2370	0.090	0.146	0.137	0.097	0.052

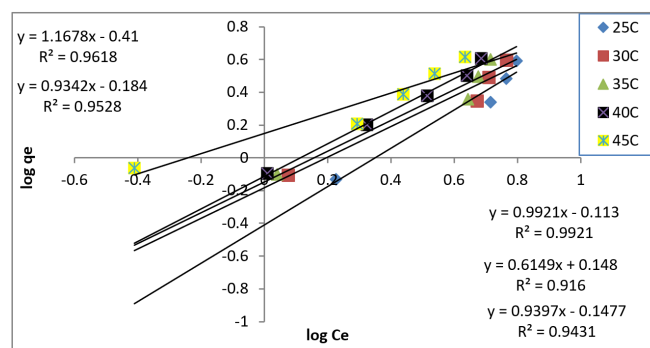
3.4 Freundlich adsorption isotherm

The Freundlich isotherm model describes adsorption on heterogeneous surfaces [19,20]. The experimental data were applied to the following mathematical equation [18]:

$$\log q_e = \log K_f + (1/n) \log C_e \quad (9)$$

where K_f is the Freundlich isotherm constant (mg/g), and n is the adsorption intensity. When $n = 1$, adsorption is linear. If $n < 1$, the adsorption process may indicate chemical adsorption, whereas $n > 1$ suggests favorable physical adsorption [21–23].

By plotting $\log q_e$ against $\log C_e$, a linear relationship can be obtained, with a slope of $1/n$ and an intercept of $\log K_f$, as shown in Figure 2.

**Fig. 2** Feruandlich adsorption isotherm

3.5 Temkin isotherm

The Temkin isotherm model describes the decrease in adsorption energy that occurs as adsorbate molecules bind to the adsorbent surface. This model assumes that the heat of adsorption decreases linearly with increasing surface coverage. The Temkin model is represented by the following equation [24]:

$$q_e = BT \ln KT + BT \ln C_e \quad (10)$$

where BT is the Temkin constant related to the heat of adsorption, and KT is the Temkin isotherm equilibrium binding constant.

From the Temkin plot shown in Figure 3, q_e was plotted against $\ln C_e$. The Temkin constants were calculated from the slope, BT, and the intercept, BT $\ln$ KT.

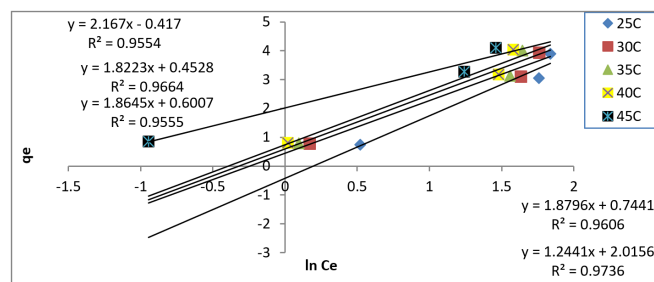


Fig. 3 Temkin adsorption isotherm

3.6 Dubinin–radushkevich isotherm model (D-R)

The Dubinin–Radushkevich (D–R) isotherm model is used to describe the adsorption mechanism based on Gaussian energy distribution on a heterogeneous adsorbent surface [25]. The model is expressed by the following equation:

$$\ln q_e = \ln q_m - \beta \varepsilon^2 \quad (11)$$

where q_m is the theoretical saturation adsorption capacity (mg/g), β is the D-R isotherm constant, and ε is the Polanyi potential.

$$\varepsilon = RT \ln(1 + 1/C_e) \quad (12)$$

where R is the gas constant, $8.314 \text{ J/mol} \cdot \text{K}$, and T is the absolute temperature in K . This model can be used to distinguish between physical and chemical adsorption by calculating the mean free adsorption energy, E , using the following equation [26]:

$$E = 1/\sqrt{2\beta} \quad (13)$$

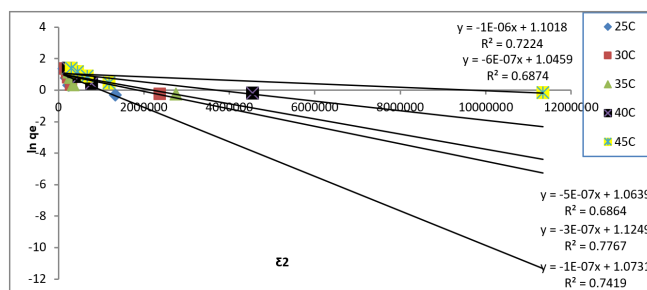


Fig. 4 Dubinin Radushevich adsorption isotherm

3.7 Calculating the thermodynamic functions using langmur-freundlich and timken constants

Based on the van't Hoff relationship, plots of $\ln K_L$, $\ln K_f$, and $\ln K_T$ against $1/T$ were constructed. The following thermodynamic equations were used:

$$\Delta G^\circ = -RT \ln K_c \quad (14)$$

$$\ln K_c = -\Delta H^\circ / RT + \Delta S^\circ / R \quad (15)$$

$$\Delta S^\circ = (\Delta H^\circ - \Delta G^\circ) / T \quad (16)$$

The Langmuir, Freundlich, Temkin, and Dubinin–Radushkevich isotherm constants for E110 adsorption onto chemically treated grape leaves are presented in Table 5. The thermodynamic parameters calculated for E110 adsorption onto grape-leaf surfaces are presented in Table 6.

Table 5 Langmuir, Freundlich, Temkin and Dubinin–Radushkevich isotherms constants for the adsorption

	T ko	298	303	308	313	318
Langmuir	$Q_{\max}$ mg/g	2.701	5.955	5.995	5.655	6.485
	b L/mg	0.222	0.128	0.139	0.204	0.396
	K_L L/mg	0.599	0.764	0.834	1.216	2.571
	R^2	1.000	1.000	1.000	1.000	1.000
Freundlich	n	0.856	1.070	1.064	1.064	1.626
	K_f L/g	0.389	0.654	0.711	0.770	1.406
	R^2	0.961	0.952	0.943	0.992	0.916
Temkin	B_T J/mole	2.167	1.822	1.864	1.879	1.244
	K_T L/mg	0.659	1.572	1.823	2.104	7.505
	T_K	1.428	2.864	3.399	3.955	9.337
	R^2	0.955	0.966	0.955	0.960	0.973
D-R	q_m mg/g	3.009	2.845	2.897	3.079	2.924
	β mol ² /KJ ²	-1E-06	6E-07	5E-07	3E-07	1E-07
	E KJ/mol	0.707	0.912	1.000	1.291	2.236
	R^2	0.722	0.687	0.686	0.776	0.741

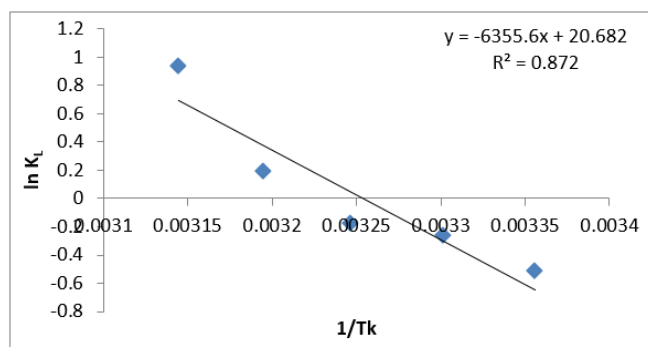


Fig. 5 The relation between $\ln K_L$ and $1/T$ at different temperatures from a development Arrhenius equation

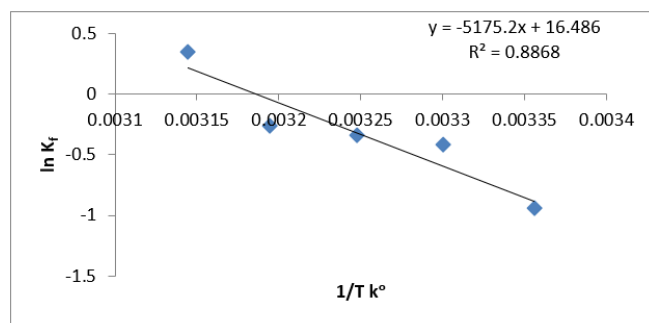


Fig. 6 The relation between $\ln K_f$ and $1/T$ at different temperatures from a development Arrhenius equation

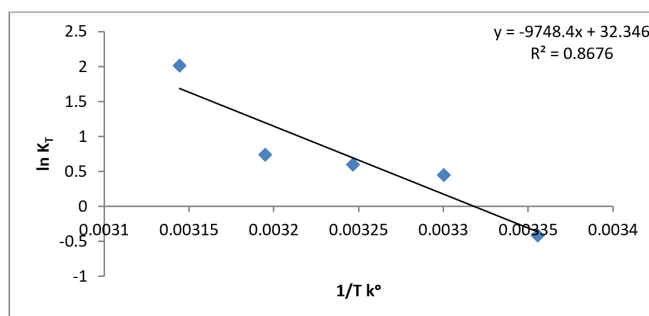


Fig. 7 The relation between $\ln K_T$ and $1/T$ at different temperatures from a development Arrhenius equation

Table 6 Thermodynamic parameters for the adsorption of E110 dye onto grape leaf surfers

		T K	ΔH° KJ/mole	R^2	ΔG° KJ/mole	ΔS° J/mole.k $^\circ$	ΔS J/mole.k $^\circ$
K_L L/mg	0.599	298	52.84046	0.872	1.266	173.067	177.317
	0.764	303			0.675	172.160	174.391
	0.834	308			0.463	170.055	171.559
	1.216	313			-0.509	170.446	168.819
	2.571	318			-2.496	174.016	166.165
K_f L/g	0.389	298	43.0266	0.8868	2.339	136.534	144.384
	0.654	303			1.067	138.479	142.002
	0.711	308			0.870	136.869	139.696
	0.770	313			0.677	135.301	137.465
	1.406	318			-0.900	138.136	135.303
K_T L/mg	0.659	298	56.68651	0.8676	1.033	186.756	190.223
	1.572				-1.139	190.845	187.084
	1.823	308			-1.538	189.040	184.047
	2.104	313			-1.936	187.293	181.107
	7.505	318			-5.328	195.017	178.259

As shown in Figure 1, the Langmuir plot showed a linear relationship, supported by the R^2 values, indicating that the adsorption data fitted the Langmuir isotherm model. The maximum monolayer adsorption capacity, Q_{max} , was calculated from the Langmuir model. This parameter indicates the ability of the grape-leaf surface to become saturated with E110 dye molecules. The increase in Q_{max} values with increasing temperature indicates improved adsorption capacity at higher temperatures. The Langmuir constant, K_L , was also determined and used as an equilibrium constant for estimating the thermodynamic parameters using the van't Hoff equation.

The positive values of the Langmuir constant, b , and their increase with increasing temperature indicate stronger interaction between E110 dye molecules and the surface of chemically treated grape leaves. This behavior supports the endothermic nature of the adsorption process and is consistent with the positive enthalpy values shown in Table 5.

The equilibrium parameter, RL , is a dimensionless constant known as the separation factor or equilibrium parameter. It is used to describe the fundamental characteristics and favorability of the Langmuir isotherm according to Equation (8) [18]. In this study, RL values were greater than 0 and less than 1, as shown in Table 4. This indicates that E110 adsorption onto chemically treated grape leaves was favorable and that the adsorption data agreed well with the Langmuir isotherm model.

When the initial dye concentration was kept constant, RL values decreased with increasing temperature, indicating improved adsorption favorability at higher temperatures. Similarly, when temperature was kept

constant and the initial dye concentration increased, RL values decreased, suggesting that adsorption became more favorable at higher dye concentrations.

Kf is an approximate indicator of adsorption capacity, whereas n reflects adsorption intensity in the Freundlich model [27]. As shown in Figure 2, the Freundlich plot showed a good linear relationship, with R^2 values close to 1. This indicates that the Freundlich model fitted the experimental data well. The Kf and n values calculated at different temperatures are presented in Table 5.

As shown in Table 5, most n values were close to or greater than 1 but less than 10, indicating favorable adsorption and suggesting that the adsorption process was mainly physical. At 298 K, the n value was less than 1, which may indicate stronger adsorption intensity or increased surface heterogeneity at this temperature. This suggests that interactions between E110 dye molecules and the grape-leaf surface may vary among different active sites because of differences in surface energy. All Kf values were positive and increased with increasing temperature, indicating increased adsorption capacity and stronger interaction between E110 dye molecules and the adsorbent surface. Kf values were used as equilibrium constants for estimating thermodynamic parameters using the van't Hoff relationship.

Table 5 also showed that KT values increased with increasing temperature. This indicates that the binding affinity between E110 dye molecules and the surface of chemically treated grape leaves increased at higher temperatures. The Temkin constant can also be used as an equilibrium constant for calculating thermodynamic functions [28].

The Dubinin–Radushkevich (D–R) model is temperature-dependent and is useful for describing adsorption on heterogeneous surfaces. When adsorption data at different temperatures are plotted as $\ln q_e$ against ϵ^2 , the constants q_m and β can be calculated from the intercept and slope of the plot using Equation (11) [29]. As shown in Figure 4, the values of q_m and β were obtained from the D–R plot.

The thermodynamic functions, including ΔH° , ΔG° , ΔS° , and ΔS , were calculated from the equilibrium constants at different temperatures, as shown in Table 6. In the van't Hoff plots shown in Figures 5, 6, and 7, ΔH° was calculated from the slope, while the entropy-related terms were used to evaluate the randomness of the adsorption system for E110 dye molecules [30].

The values of the equilibrium constants and ther-

modynamic functions, including ΔH° , ΔG° , ΔS° , and ΔS , within the temperature range used in this study are presented in Table 6. The positive ΔH° values indicate the endothermic nature of the adsorption process. Negative ΔG° values indicate that adsorption of E110 dye onto chemically treated grape leaves was spontaneous and favorable at the corresponding temperatures, whereas positive ΔG° values indicate non-spontaneous adsorption under those conditions. Positive ΔS° values indicate increased randomness at the solid-solution interface during adsorption. Moreover, the thermodynamic results suggest that enthalpy change contributed strongly to the adsorption process, indicating that E110 dye adsorption onto chemically treated grape leaves was mainly enthalpy-controlled [31].

4 CONCLUSION

The adsorption of E110 dye increased with increasing temperature, indicating that the adsorption process is endothermic. The percentage removal of E110 dye also increased when the weight of the adsorbent increased. The adsorption isotherm results of E110 dye on the surface of the adsorbent material, chemically treated grape leaves, matched the Langmuir model. The negative value of Gibbs free energy change, ΔG° , indicates that the adsorption process is spontaneous.

Author contributions

Ibrahim Y. designed the study ; Abeer Q. conducted experiments ; Ebtehaq Z. analyzed the data ; and Abdallah K. wrote the manuscript.

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Data availability

N/A

DECLARATIONS

Conflict of interest

The authors declare no competing interests

Consent to publish

N/A

Ethical approval

N/A

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