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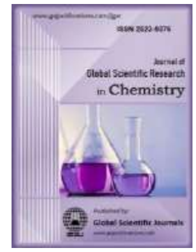

Determination the Thermodynamic Functions of Adsorption of Some Food Coloring Dyes on the Surface of Leaves Grape Chemically Treated

Abeer Qais Younus Al-Allaf, Khaleel Ibraheem A. Al-Niemi, Mohammad Mahmoud Hussein Younes Al-Niemi

Department of Chemistry, College of Education for Pure Sciences, University of Mosul, Mosul, Iraq.

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ABSTRACT

In this study, thermodynamic functions were calculated for the adsorption of some food coloring dyes (E151, E124, E102, E127) on the surface of chemically treated grape leaves. The analytical method was used to achieve the standard calibration curve for each dye in order to calculate the amount of adsorbed and remain dye in the solution according to (Beer's-Lambert law) by using spectrophotometric method for UV-Visible spectrophotometry. The thermodynamic functions of adsorption process (ΔH, ΔG°, ΔS°, ΔS) were calculated within experimental range of temperatures (298-318K°). The results of thermodynamic study showed that the adsorption of food coloring dyes is endothermic process and physical in nature through positive (ΔH) values and few at all concentrations with range of temperatures used, where the values of (ΔG°) are negative and few, this indicates that the adsorption process occurs spontaneity which increase with temperature at constant concentration, this refers to predominate of physical adsorption. The ΔS and ΔS° have positive values, this is a clear indication that the adsorption system of food dye molecules on the surface of grape leaves is more random, and that ΔS is greater than ΔS°, meaning that the food dye molecules are less regular at the stages of the adsorption process compared to their state at equilibrium and have a high degree of freedom on the surface of leaves grape.

1. Introduction

food coloring dyes are involved of the chromophore azu group (N=N), sulfur (C=S), aryl and ionic rings (5-12). The adsorption technique was used widely among other methods to treatment the problem of water pollution because its efficiency, ease and economically inexpensive, by using of different effective adsorbent materials (13-20).

Food coloring dyes are widely used in the food industries, soft drinks, pastry and various juices. Which considered as organic chemicals containing aromatic rings and some functional groups. This dyes mainly polluted water in the places of their manufacture or from domestic and artisanal use or for their manufacture (1-4). As we know the food colorings did not exist in nature because it is a manufactured chemical compound. Chemically,

Corresponding author:
Email addresses: alnuaimi5310@yahoo.com (Khaleel), drmohammadalhusseiny@uomosul.edu.iq (Mohammad)
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the adsorption process is exothermic or endothermic respectively (25). The adsorption process in terms of whether it occurs spontaneously or non-spontaneously through sign and values of (ΔG°), while the (ΔS°) value represent the randomness of the adsorption system of the adsorbed molecules before and after adsorption.

Results and Discussion

1. Effect of Temperature

The adsorption process of food coloring dyes on the surface of powder grape leaves which chemically treated include physical bond formation and this will be associated with a change in the thermodynamic parameters of the adsorption system, this parameters were used to explain the variation food coloring removal efficiency at different temperatures and to confirm the adsorption nature of this dyes on the surface of grape leaves. Temperature is a factor affecting the adsorption process (26), and the increase in temperature leads to increase of transfer of the non-adsorbed molecules in the solution to the solid surface for adsorption and an increase in the efficiency of adsorption, the increase of temperature affect on the adsorbing molecules on the surface of adsorbent material in the case of physical adsorption leads to decrease the efficiency of adsorption system by return of dye molecules from surface to solution.

The results of effect of temperature in adsorption process tabulated in table (1)

Usually the adsorption process was accompanied by a change in free energy (ΔG) of the surface on which adsorption occurs and by a decrease in entropy due to the adsorption of molecules on the surface which loss of some degrees of its freedom compared to its state before the adsorption process and as a result of the decrease in free energy and entropy simultaneously and cause a decrease in enthalpy (21-24).

The values of thermodynamic parameters are calculated by depending on the value of the equilibrium constant which is represented as follows: $Kc = Cads. / ce$ ………….(1)

kc= equilibrium constant
Cads = adsorbed concentration of dye (mg/L)
Ce = The residual concentration of dye after adsorption (mg/L).
$\Delta G° = -RT \text{ In } Kc$ ………….(2)
$\ln kc = \text{In } C - \frac{\Delta H}{RT}$ …………(3) (Vant-Hoff equation)

It has been assumed that the values of (ΔG=0) at equilibrium state.

The Vant-Hoff equation is usually used to Calculate the enthalpy values (ΔH) of the adsorption process, this value is important to determining the type of bonding forces between the surface of adsorbent material and the adsorbed molecules of food coloring dye, which represents the amount of energy required to recover the adsorbed from solid surface, as well as the type of adsorption physical or chemical. The negative or positive values of (ΔH) indicates that

Table (1): The effect of temperature on the percentage of adsorption of food coloring dyes on the surface of grape leaves chemically treated at optimum condition

Dye	Grape leave (gm)	Ci mg/L	T K°	Ce mg/L	qe (mg/gm)	%
E151	**0.3**	**17.353**	298	0.6668	2.781	96.15
			303	0.5366	2.8021	96.90
			308	0.4228	2.821	97.56
			313	0.3594	2.8323	97.92
			318	0.1788	2.8624	98.96
E102	**0.3**	**10.687**	298	1.29292	0.8758	81.94
			303	1.3718	0.93156	87.16
			308	1.07178	0.961562	89.97
			313	0.94749	1.02796	91.13
			318	0.51449	1.017291	95.18
E124	**0.3**	**12.089**	298	3.13744	0.89517	74.04
			303	2.766019	0.93230	77.11
			308	2.18355	0.99056	81.93
			313	1.71007	1.0379	88.85
			318	1.366019	1.07231	88.69
E127	**0.3**	**17.597**	298	1.95108	1.117579	88.91
			303	1.68255	1.13676	90.43
			308	1.181388	1.172558	93.28
			313	0.89499	1.193050	94.91
			318	0.698080	1.20707	96.03

calculation the values of the equilibrium constant for the adsorption process.

The enthalpy value can be calculated from the slope of the relationship between (In Kc) and $\frac{1}{T}$ at different temperatures which is supposed to give a straight line with a high correlation coefficient (R^2).

Figures (A, B, C, D) for food coloring dyes (E102, E151, E124, E127) respectively on the surface of grape leaves chemically treated.

The other thermodynamic parameters (ΔG°, ΔS° and ΔS) calculated after that, where (ΔG°) value represent the charge in the free energy at any step of adsorption process, while the (ΔG) value represent the free energy at equilibrium state, the same is true for (ΔS°) and (ΔS) of adsorption system.

The results in table (2) refers to an increase in the value of the adsorption capacity and the efficiency for adsorption of dye molecules on the surface of a grape leaves chemically treated. Therefore, the amount of adsorbed dye molecules is proportional to the amount of heat absorbed by the adsorption system.

2. Thermodynamic study:

To know the pathway of adsorption process and the type of forces affecting it, it was necessary to calculate the thermodynamic parameters of the adsorption system.

The enthalpy which gives an indication of the nature of bonding forces between the adsorbed dye molecules and the surface which can be calculated from Vant-Hoff equation after

The (ΔH, ΔG°, ΔS° and ΔS) and Equilibrium constant values for adsorption of food coloring dyes at different concentration, and temperatures listed in table (2)

$$\Delta G° = \Delta H - T\Delta S° \text{ --------- } (4)$$

$$\Delta S° = \frac{(\Delta H - \Delta G°)}{T} \text{ --------- } (5)$$

$$\Delta S = \frac{\Delta H}{T} \text{ --------- } (6)$$

Table (2): Values of thermodynamic functions at equilibrium of (E151) dye using (0.3) gm of grape leaves chemically treated at different temperatures and concentrations at a natural acidity function.

Ci mg/L	T K°	Kc	ΔH KJ/mol	R^2	ΔG° KJ/mol	ΔS° J/mol	ΔS J/mol.K
17.353	298	25.024	54.8591	0.9207	-6.6924	575.3607	184.090
	303	31.3395			-8.5922	577.2317	181.053
	308	40.043			-10.737	579.2693	178.113
	313	47.283			-12.824	580.651	175.268
	318	96.054			-17.0787	586.5436	172.512
34.70	298	32.386	27.1610	0.92024	-7.2284	300.5239	91.144
	303	33.369			-8.7487	300.7725	89.640
	308	38.469			-10.6206	301.9547	88.185
	313	45.25			-12.6779	303.3047	86.776
	318	60.567			-15.3534	305.72862	85.411
52.060	298	13.226	21.2239	0.9199	-5.36707	233.7081	71.221
	303	15.1672			-6.78207	234.8467	70.045
	308	16.588			-8.1729	235.5912	68.908
	313	17.83			-9.5806	236.1914	67.807
	318	21.865			-11.5415	237.887	66.741
69.41	298	13.9756	19.4082	0.9898	-5.48156	216.0086	65.128
	303	16.8581			-7.0456	217.5677	64.053
	308	18.489			-8.4886	218.354	63.0136
	313	19.618			9.8984	218.8286	62.200
	318	21.5823			-11.4928	219.6216	61.0320
86.76	298	11.7634	18.0489	0.9677	-5.12349	215.98	60.5667
	303	12.6449			-6.3284	212.584	59.567
	308	16.784			-8.20716	210.938	58.600
	313	17.654			-9.5476	206.359	57.664
	318	25.024			-12.0464	202.250	56.754

Table (3): Values of thermodynamic functions at equilibrium of (E102) dye using (0.3) gm of grape leaves chemically treated at different temperatures and concentrations at a natural acidity function.

Ci mg/L	T K°	Kc	ΔH KJ/mol	R^2	ΔG° KJ/mol	ΔS° J/mol	ΔS J/mol.K
10.687	298	4.5395	61.0413	0.9034	-9.435	648.155	204.83
	303	6.79078			-16.937	666.872	201.456
	308	8.9716			-26.106	685.004	198.186
	313	10.2796			-34.18	695.878	195.020
	318	19.7728			-73.976	774.809	191.353
21.374	298	3.1895	60.9216	0.90456	-6.629	635.7348	204.43
	303	3.4515			-8.608	637.9124	201.061
	308	4.4509			-12.931	646.222	197.791
	313	8.4070			-27.95	679.1132	194.637
	318	11.4641			-42.890	704.5297	191.577
32.6622	298	4.2665	54.2257	0.92381	-8.868	577.7277	181.965
	303	4.7972			-11.9652	582.1396	178.96
	308	5.6950			-16.572	589.6043	176.057
	313	10.5054			-34.936	629.5979	173.24
	318	13.3045			-49.776	652.8693	170.521
42.749	298	4.4787	52.8645	0.93893	-9.30898	565.8816	177.39
	303	4.9356			-12.3104	569.6803	174.47
	308	6.7007			-19.498	584.35536	171.63
	313	0.4647			-34.801	615.6494	168.89
	318	10.4647			-50.325	640.4791	166.24
53.437	298	4.7972	47.13705	0.9058	-9.9709	510.25	158.175
	303	5.2356			-13.0586	514.899	155.567
	308	7.4788			-21.7628	533.54	153.042
	313	9.5627			-31.8019	550.87	150.59
	318	13.1637			-49.2495	580.814	148.22

Table (4): Values of thermodynamic functions at equilibrium of (E124) dye using (0.3) gm of grape leaves chemically treated at different temperatures and concentrations at a natural acidity function.

Ci mg/L	T K°	Kc	ΔH KJ/mol	R^2	ΔG° KJ/mol	ΔS° J/mol	ΔS J/mol.K
12.0892	298	2.8532	47.4737	0.9414	-2.17918	483.4545	159.307
	303	3.37045			-3.0305	484.8396	156.678
	308	4.5364			-4.4016	487.3096	154.135
	313	6.06941			-5.99693	489.73	151.167
	318	7.8498			-7.7089	491.8687	149.288
24.1790	298	4.18561	27.608	0.912	-2.9756	287.995	92.644
	303	4.6023			-3.80754	288.774	91.115
	308	5.3379			-4.87359	290.007	89.636
	313	6.2961			-6.11889	291.380	88.204
	318	7.7147			-7.6439	293.069	86.817
36.268	298	3.85158	21.1242	0.9072	-2.8028	222.4534	70.886
	303	4.2288			-3.5964	223.2303	69.716
	308	4.6358			-4.46323	223.994	68.585
	313	5.2330			-5.50229	224.9978	67.489
	318	7.5562			-6.84698	226.4576	66.428
48.358	298	4.7565	15.7026	0.9133	-3.24145	169.9923	52.693
	303	5.12156			-4.07417	170.6071	51.823
	308	5.6048			-5.01557	171.3567	50.982
	313	5.8952			-5.90007	171.7767	50.168
	318	6.84432			-7.19609	173.0178	49.379
60.447	298	5.591003	15.41849	0.9323	-3.57743	172.494	51.739
	303	5.88009			-4.41866	170.913	50.886
	308	6.39929			-5.40132	168.617	50.060
	313	6.652			-6.30174	165.939	49.260
	318	8.393142			-7.9593	160.872	48.48

Table (5): Values of thermodynamic functions at equilibrium of (E127) dye using (0.3) gm of grape leaves chemically treated at different temperatures and concentrations at a natural acidity function.

Ci mg/L	T K°	Kc	ΔH KJ/mol	R^2	ΔG° KJ/mol	ΔS° J/mol	ΔS J/mol.K
17.5472	298	8.019168	52.7190	0.9458	-4.3270	544.449	176.90
	303	9.4586			-5.6042	545.87	173.99
	308	13.8953			-7.657	549.069	171.165
	313	18.6624			-9.7324	551521	168.43
	318	24.207			-11.9223	553.68	165.78
35.1944	298	6.15820	52.594	0.9054	-3.778	601.058	176.48
	303	8.49825			-5.3372	603.73	173.57
	308	10.3433			-6.7985	605.37	170.75
	313	13.0105			-8.5326	607.27	168.03
	318	25.6892			-12.144	612.933	165.38
52.7916	298	6.3120	52.3358	0.9709	-3.8295	538.676	175.62
	303	9.7898			-5.69013	542.351	172.72
	308	11.1033			-7.0048	543.371	169.921
	313	16.6601			-9.3549	546.745	167.20
	318	19.6725			-11.1462	548.127	164.57
70.388	298	7.02503	41.5641	0.9741	-4.05199	431.849	139.47
	303	9.5238			-5.6237	434.38	137.17
	308	11.41773			-7.08609	435.88	134.94
	313	13.7278			-8.7116	437.41	132.79
	318	17.9964			-10.813	439.67	130.70
87.986	298	7.147002	33.3507	0.9901	-4.0877	349.85	111.915
	303	9.68718			-5.6638	352.38	110.06
	308	11.39156			-7.07942	353.734	108.28
	313	13.746			-8.71556	355.296	106.55
	318	14.3284			-9.96028	355.641	104.87

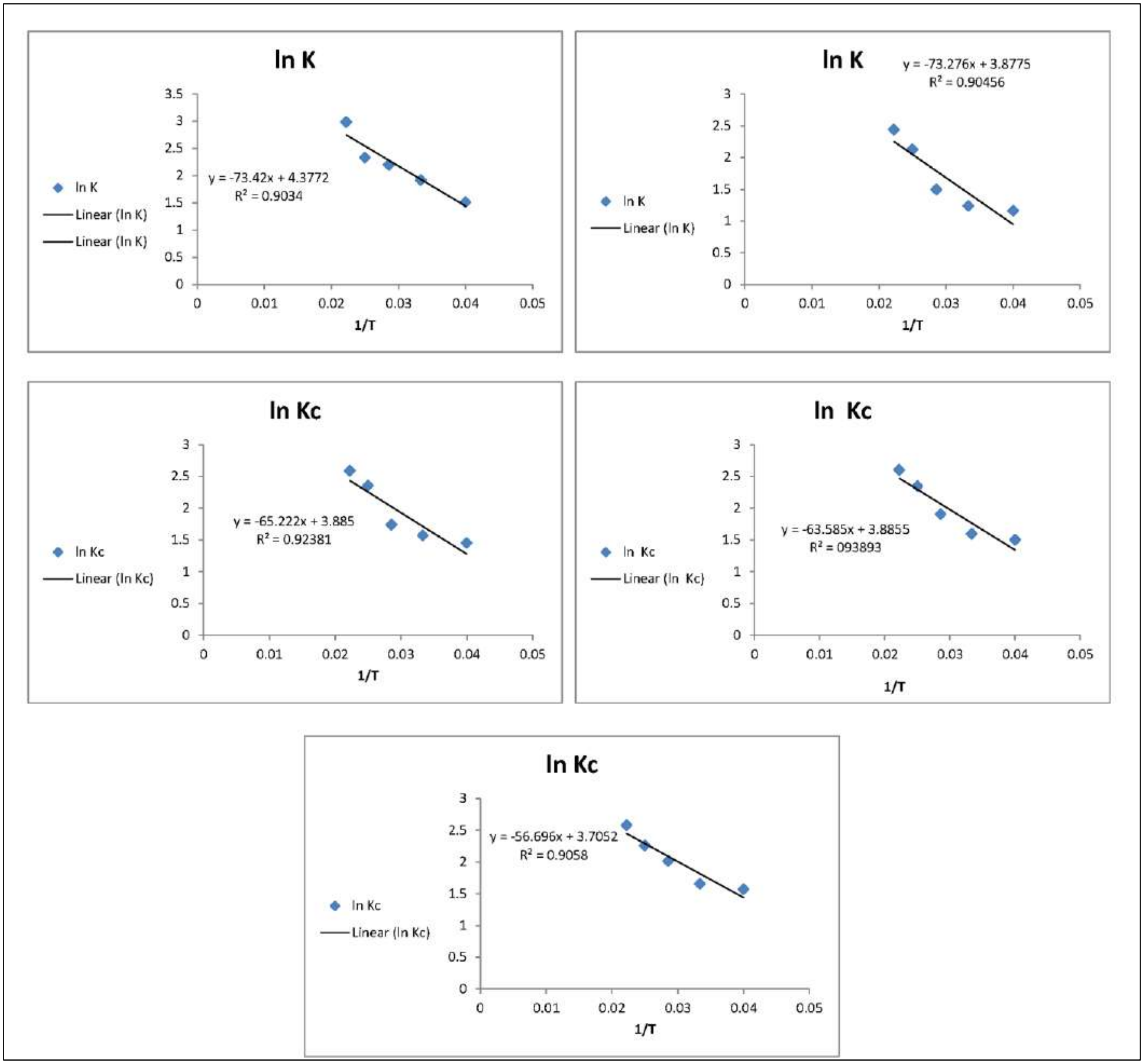
ln K
y = -73.42x + 4.3772
R² = 0.9034
ln K
Linear (ln K)
Linear (ln K)
1/T
ln K
y = -73.276x + 3.8775
R² = 0.90456
ln K
Linear (ln K)
1/T
ln Kc
y = -65.222x + 3.885
R² = 0.92381
ln Kc
Linear (ln Kc)
1/T
ln Kc
y = -63.585x + 3.8855
R² = 093893
ln Kc
Linear (ln Kc)
1/T
ln Kc
y = -56.696x + 3.7052
R² = 0.9058
ln Kc
Linear (ln Kc)
1/T

Figure (A)

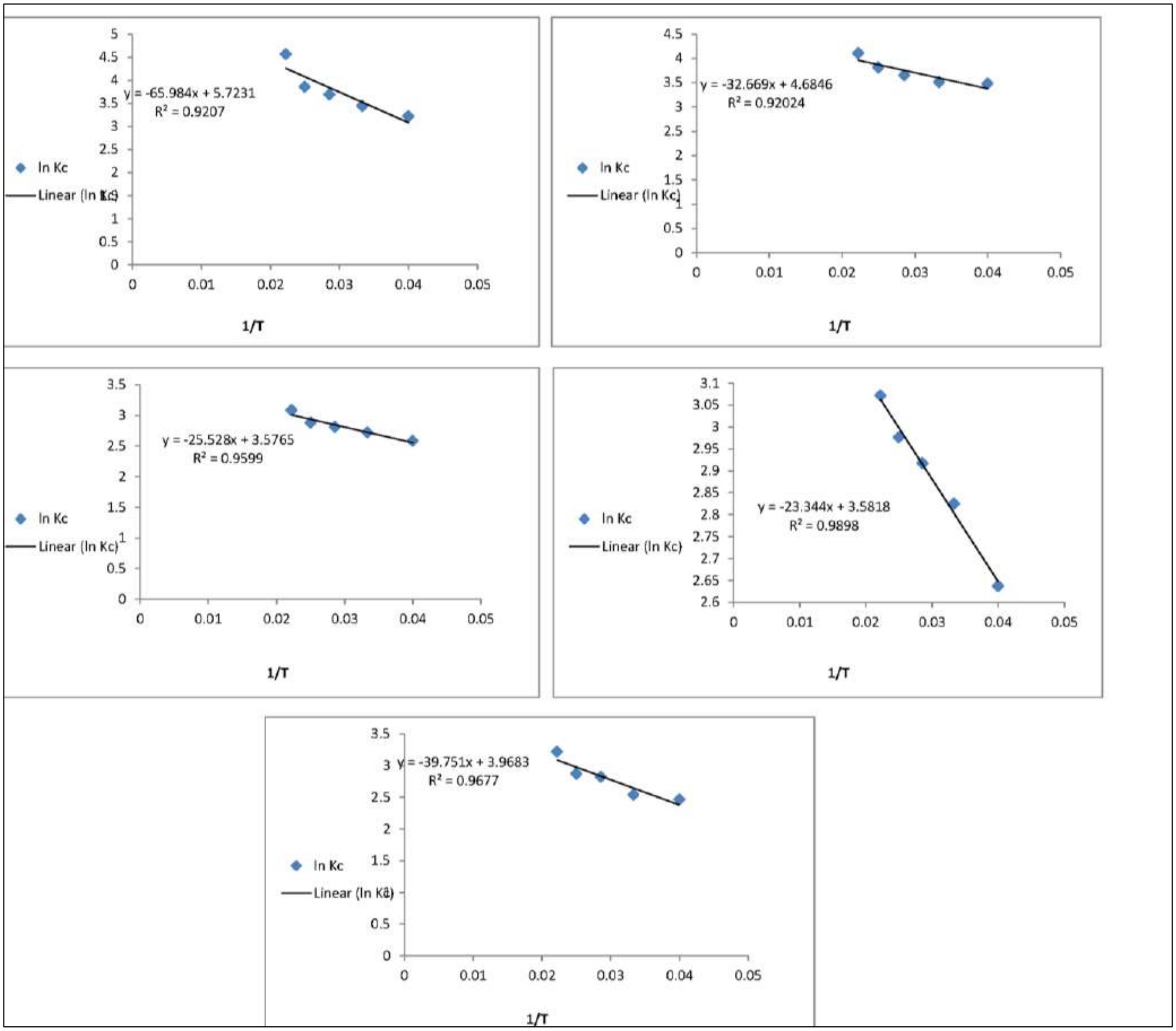
y = -65.984x + 5.7231
R² = 0.9207
In Kc
Linear (In Kc)
1/T
y = -32.669x + 4.6846
R² = 0.92024
y = -25.528x + 3.5765
R² = 0.9599
y = -23.344x + 3.5818
R² = 0.9898
y = -39.751x + 3.9683
R² = 0.9677

Figure (B)

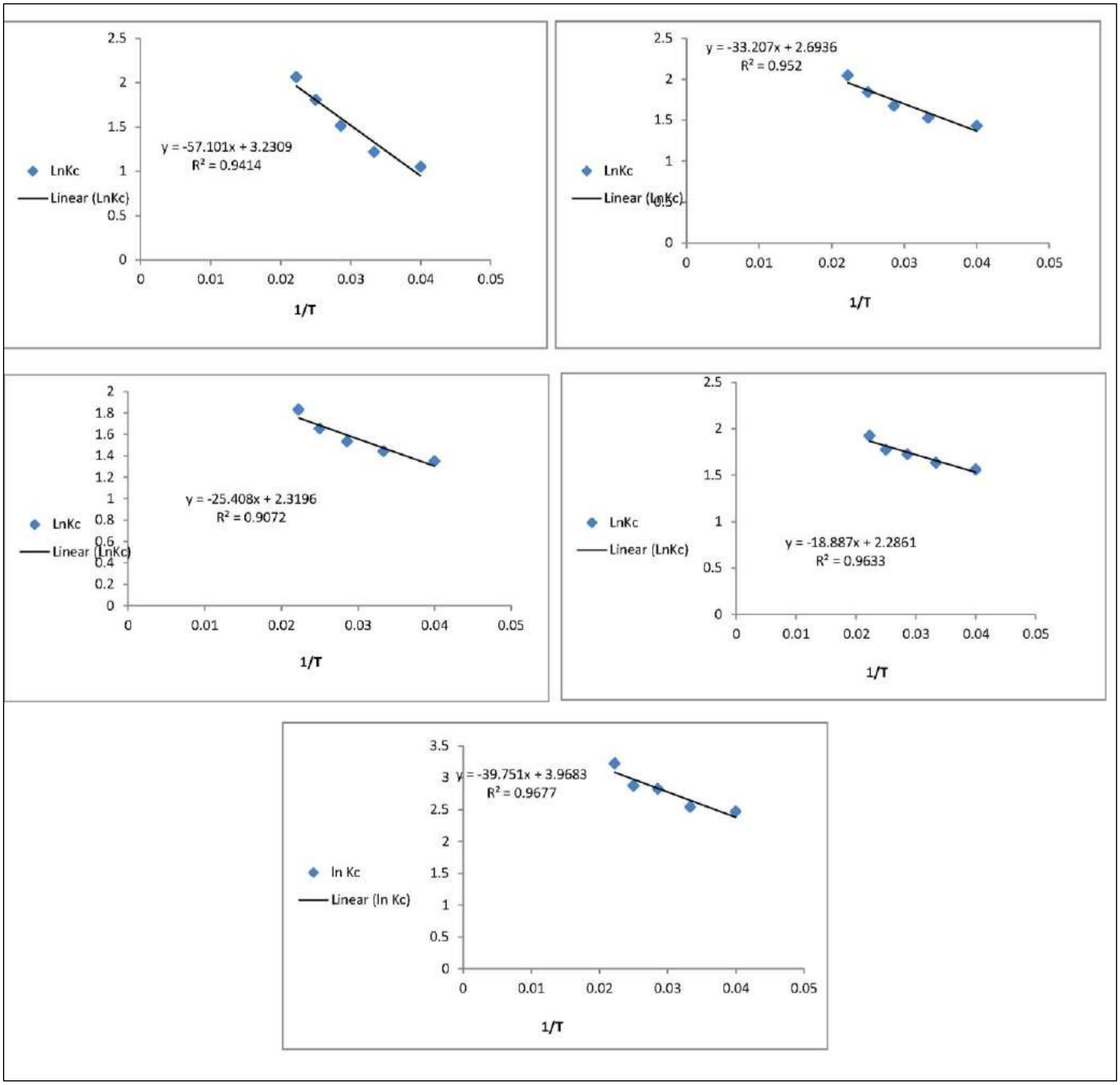
y = -57.101x + 3.2309
R² = 0.9414
LnKc
Linear (LnKc)
1/T
y = -33.207x + 2.6936
R² = 0.952
LnKc
Linear (LnKc)
1/T
y = -25.408x + 2.3196
R² = 0.9072
LnKc
Linear (LnKc)
1/T
y = -18.887x + 2.2861
R² = 0.9633
LnKc
Linear (LnKc)
1/T
y = -39.751x + 3.9683
R² = 0.9677
ln Kc
Linear (ln Kc)
1/T

Figure (C)

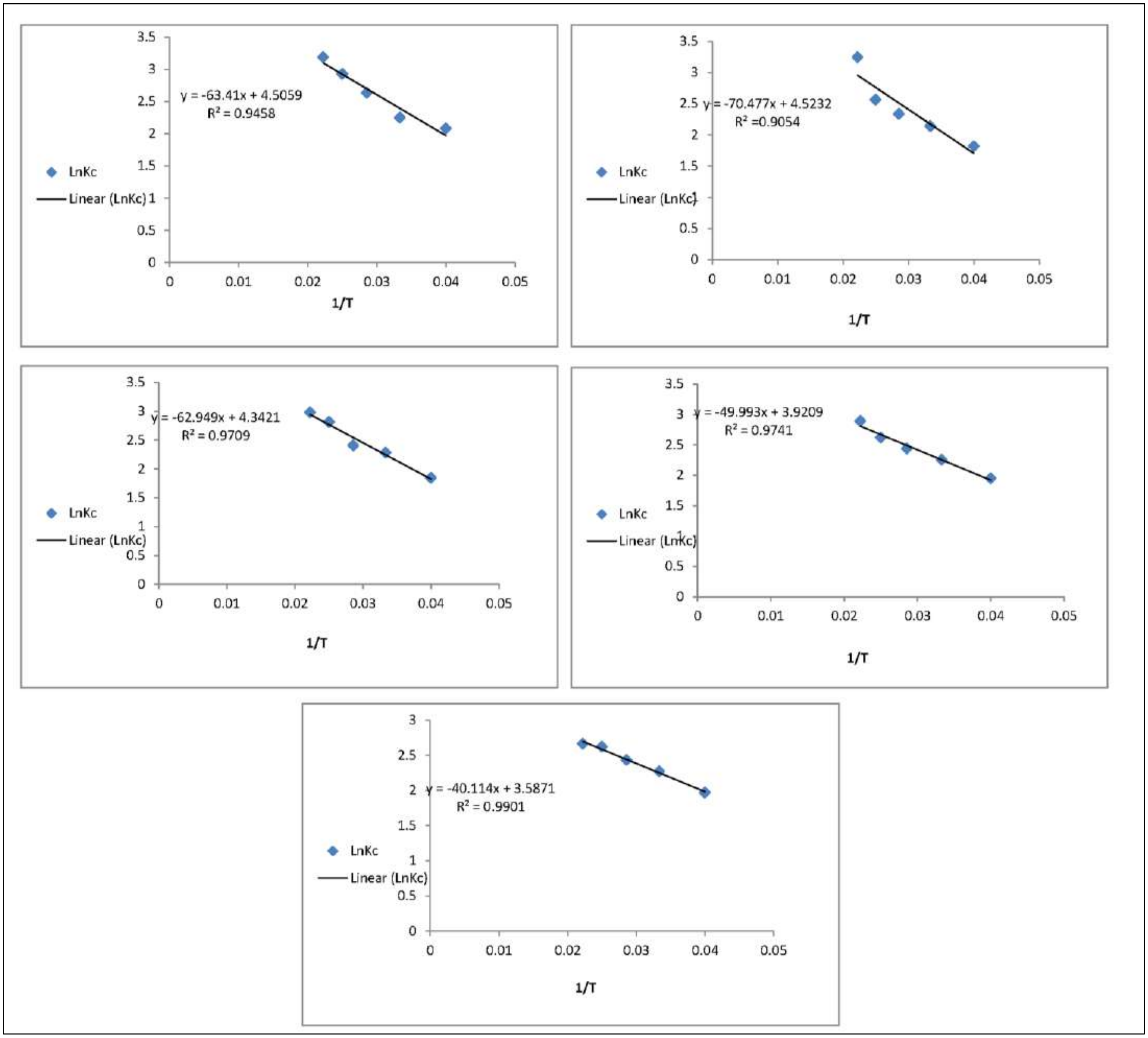

Figure (D)

consists of a large molecules an their number increases with increasing concentration in a fixed volume of solation, this will cause the dye molecules to converge to each other and the interaction (27,88) increase in solution and the resistance to the mass transfer of the dye molecules occurs between the solution and the solid phase, leading to a decrease in the number of adsorbed dye molecules, so the amount of energy absorbed by adsorption system decreases, so enthalpy decreases (29) (30). At a constant concentration and a certain temperature range,

It was observed from the results in table (5), that enthalpy have positive values, which thermodynamically means that the adsorption of food coloring dyes on the surface of grape leaves chemically treated are endothermic process, its values ranged (18-61) kJ/mute this indicates that the physical adsorption predominate in the adsorption system.

The increasing of initial concentration of dyes in solution leads to decrease in enthalpy value within the temperature range used, because the dyes molecules adsorbed on the surface which increase in the randomness on the surface of adsorption system.

The ΔS values which represent the randomness of adsorptions of adsorbed dye molecule at the surface at equilibrium state, and cluse in their positive values with the change of temperatures, as it gives confirmation that the adsorption of food dyes molecules on the surface of chemically treated grape leaves is of physical nature.

It indicates an increase in entropy on the surface of chemically treated grape leaves, as well as an increase in molecular interactions in the solution during the adsorption process this means that the molecules of coloring dyes have freedom degree, this means that the molecules of coloring dyes have a high degree of freedom on the surface and do not suffer any obstructions there. Finally, values of ΔS greater than ΔS° indicates that the entropy of this type of adsorption at equilibrium is greater than the entropy at any of its stages, and increase with increase of temperature while it is decrease with increase of concentration

the high energy absorbed is accompanied by a high adsorption efficiency. The positive enthalpy indicates the occurrence of a sorption within the adsorption process to inner micro-pores of a grape leaves and this requires additional external energy to be received by adsorption system.

The negative values of (ΔG°) which listed in table (5) for food coloring dyes indicated the spontaneity and feasibility of adsorption of dyes molecules on the surface of a grape leaves.

The increasing of the temperature of food coloring dyes solution at constant concentration cause an increase in the negative values of (ΔG°), this means an increase in spontaneous an adsorption for dye molecules from solution to the surface of grape leaves. In the sorption system, the (ΔG°) is the driving forces, as well as, the basic criterion for the spontaneous realization of the sorption, and the low negative values of (ΔG°) refers to predominate of physical adsorption in the sorption process.

The entropy thermodynamically represent the degree of random in adsorption process for adsorbed molecules, and refers to condition of adsorption system after binding of food caloring molecules to the surface of grape leaves chemically treated, the results for ΔS° and ΔS listed in table (5) and both of them have positive values, ΔS values are greater than ΔS° values at a constant concentration, the values of ΔS° decrease with increasing temperature which represent the state of the regularity of molecules in any stage of the adsorption process, whose randomness decrease with the increase in temperature and the increase in the concentration of the dye in the solution due to the decrease in the number of

Experimental

1. **The chemicals:** The chemicals used in the research were supplied from BDH and Fluka Companies:

a- Granular activated carbon and alumina.
b- Sodium hydroxide.
c- Hydrochloric acid.
d- The food coloring dyes and grape leaves used were supplied from market of our city from well-known companies.
e- Distilled water

2. **Instruments:**

a- Electrothermal melting point of the type (9300 meter).
b- The pH measurement device of the type (JENWAY 3510).
c- An absorbance measuring device of the type (T92+ UV spectrophotometric PG lin) which used to find the values of (λ max) for all food coloring dyes.
d- (CECIL spectrophotometer 1000S) measure the adsorption of solution for food coloring dyes solution before and after adsorption process using distilled water as a solvent and (blank) with glass cells with a thickness of (1 cm).
e- Water bath with a program vibrator (Julabo Sw 23) to shake solutions at a speed of (100 rpm).

3. **Preparation of standard solutions:**

The standard solutions of the studied food coloring dyes were prepared by dissolving (1 gm) of each dye in a liter of distilled water, then solutions were prepared with concentrations (2,4,6,8,10)*10^{-5} M. for all the dyes, was tracked by diluting the calculated volume from the original standard solution.

4. Analytical method:

a- A UV-VIS spectrophotometric device of the type
(T92+ spectrophotometric) was used to estimate the concentration of food coloring dyes in the solutions under study. The test was done based on the ability of these food colorings to absorb electromagnetic rays in the UV-visible range. To complete this work, at first, the value of the maximum absorption wavelength (λ max) was determined for each dye separately, then the amount of adsorbent material was tracked after a period of time according to the nature of each dye. Draw the relationship between absorbance and concentration to choose the best concentration for the study.

b- The best five concentrations were selected from the calibration curve to prepare (5) solutions containing different concentrations of solutions of each food coloring dye, and to each of them the necessary amount of activated carbon was added.

c- These solutions were then shaken separately for (100) minutes and at a range of temperatures (25, 30, 35, 40 and 45 °C) respectively using the programmed vibrator after setting the temperature.

d- The five solutions were filtered and the absorbance value was recorded before and after adsorption using equations (1,5) to find the adsorption capacity and efficiency.

The change of quantity adsorbed material from food coloring dye with time using Lambert Beer, s law to draw the calibration curve at (λ max) for each dye between absorbance and concentration, as in the following equation:

A = ε L C ………………….. (7)

A = absorbance of dye solution before adsorption.
ε = molar absorption coefficient (L/mol.cm).
C= food coloring dye concentration (M).
L= length of the absorber cell (1 cm).

Table (6): Names and molecular formulas of food coloring dyes, physical properties and optimum condition

Coloring dyes	Dye name	Molecular formula	The color	Melting point °c	λ_{max} nm	M.wt G/mol	ε_{max} l/mol.cm	Optimum conditions Tc°	gm/L	PH
E102	Tartazine	$C_{10}H_{11}N_2Na_3O_{10}S_3$	Yellow	870	426	534.3	8592.7	45	53.430	6.50
E124	Ponceau	$C_{20}H_{11}N_2Na_3O_{10}S_3$	Red	-	503	604.46	19361	45	60.446	7.12
E127	Erythrosine	$C_{20}H_6N_4Na_2O_5$	Red	-	530	879.86	12680	45	87.986	7.42
E151	BrilliaT BLACK PN	$C_{28}H_{17}N_5Na_4O_{14}S_{42}$	Black	-	517	867.68	87000	45	8	

5. Determination of the concentration of solutions of food coloring dyes

The concentration of dyes before and after adsorption was estimated using a UV - visible spectrometer, and the results were obtained in (mg/L) using the standard curve that was prepared previously for each dye. Express the amount of the adsorbent in terms of adsorption capacity (qe) and adsorption efficiency (%) by estimating the amount of residual and adsorbent from the food coloring dye solution as in the following equations:

Cads = Co – Ce ………………… (8)
qe = ((Co – Ce) / m) * V_L ……………… (9)
qe = (Co – Ce) / Co) * 100 ………………… (10)
Where:
Ce = residual dye concentration (mg/L).
Co = the initial concentration of the dye (mg/L).
V_L = volume of the model (ml).
m = weight of the adsorbent (activated carbon) (g/L).

6. Determining the amount of adsorbent material

Five different weights of the adsorbent material (0.01-0.09 g) were tested at the natural acidity function and the initial concentration was stable to choose the best weight for it to adsorb these food coloring dyes until reaching the equilibrium state.

7. Determination of the adsorption isotherm

To find the adsorption isotherm, concentration solutions were used for each dye described previously, where (50 ml) of each solution were taken and placed in contact with a known weight of activated carbon in a standard flask of (250 ml) capacity equipped with a tight seal and placed in a water bath equipped with a vibrator at a temperature of (298-318k0). for a specified period of time. After filtration, the absorption was measured with a UV-visible spectrometer and the residual concentration after adsorption was calculated, including calculating the amount of adsorbent per unit weight of the adsorbent based on equation (7) and the following equation:

$Ce = Ai / \varepsilon$ ……………….. (11)

Where:

Ai = absorbance after adsorption.

8. The effect of temperature

To estimate the effect of temperature on the adsorption rate, the adsorption of each food coloring dye solution was studied at temperatures (25,30,35,40,45 Co) and by the same steps mentioned previously. These results were used for application in the Vant - Hoff equation by drawing the relationship between (ln Kc) versus (1/T) The thermodynamic values were found according to equations.

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