

Sorption study of Birllant Blue Dye on Titanium Dioxide

Khulood A.S. Al-Saadie¹, Qabas Mohammad Abd-Al-Hussani²

¹ Department of Chemistry, College of Science, Baghdad University, Baghdad, Iraq

² Department of Chemistry, College of Science, Al-Nahrain University, Baghdad, Iraq

Abstract

The subject of this research involves studying the possibility removed of birllant blue by TiO₂. The thermal and photo-stability were studied and the results showed that the dye are stable in temperature range about (288-318) K, and stable against UV-linght in the wave length range (290-320) nm. Titanium dioxide successed in removing; 54-95% of dye.

UV-Vis absorption spectrophotometric technique was used to follow the dye concentration before and after adsorption process and after different periods of time, since the sorption isotherms were obtained by obeying Langmiur and Freundlich adsorption isotherms, with R² (1-0.9993) for dye.

The dye adsorption isotherms take which related to a strong interaction between dye and TiO₂ with thermodynamic values $\Delta H = +(5.962-26.57)J$, $\Delta G = -(966-999)J$, $\Delta S = +(3.45-9.1906)J$.

The kinetic study of dye sorption on TiO₂ was studied depending on three kinetic equations: (Lagergren equation), (Morris-Weber model), (Reichenberg model).

From these models we suggest that the rate limiting mechanism is absorption and the sorption process involving two steps; absorption and adsorption.

Introduction

The use of natural dyes for painting and dyeing has been known since ancient time. The recent discovery in the chauret-pont-dar'c caves in france of 30000 year old Paleolithic rock paintings provide the ancientest testimony of the millenary use of inorganic compounds along history, especially as textile dyes⁽¹⁾.

The chromophore is a group of atoms, which controls the color of the dye, and it is usually an electron with drowning group. The most important chromophores are -C=C-, -C=N-, -C=O, -N=N-, -NO₂ and -NO groups⁽²⁾.

The auxochrome is an electron-donating substituent that conintensify the color of the chromophore by altering the overall energy of the electron system and provides solubility and adherence of the dye to the fiber⁽³⁾.

There are two important for selecting a coloring agent⁽²⁾:

1-Good storage stability with respect to other detergent components and to light.

2-No significant tendency to affect textile fiber.

Pollution of surface water and ground water is a major concern throughout the world. There are two basic types of pollution:

Point and Non point⁽⁴⁾; Point pollution is contamination that can be traced to a particular source such as an industrial site, septic tank, or waste water treatments plant, non point pollution results from large areas and not from any single source and includes both natural and human activities⁽¹⁹⁾. Sources of non point pollution include agricultural, human, forestry, urban, and mining activities and atmospheric deposition⁽⁵⁾.

Titanium dioxide is considerably the most extensively studied photocatalytic material because of the high resistance to dark and photoinduced corrosion, easy availability and low cost. The titanium dioxide surface was explored many studies as a means of improving its photocatalytic activity⁽⁶⁾.

Langmiur Isotherm reflects good representation of chemisorption, and usually represents the low P portion of other isotherms⁽⁷⁾. Adsorption sequentially fills surface sites until mono-layer coverage is achieved. No multilayer coverage is included.

Langmiur behavior assumes a rapid reversible sorption, and interaction only between sorbate molecules and a surface site^(8,9).

The Langmiur equation could be expressed as:

$$\left[\frac{C_e}{Q} = \frac{1}{k} + \frac{a}{k} - C_e \right] \dots \dots \dots (1)$$

Where Q is the amount of adsorbate (mg/g), C_e is the equilibrium concentration (mg/L), and a, k are constants related to adsorption capacity and energy of adsorption respectively or sometimes called Langmiur constants^(8,9).

Freundlich Isotherm is one of the most important isotherms that deals with sorption at solid-liquid interface, which has been introduced by (Herbert Max Finaly Freundlich) a German scientist in 1926⁽¹⁰⁾.

Most of surfaces are heterogeneous, so the change in potential energy is regular, and the adsorption sites are not equivalent in energy⁽¹⁵⁾, hence the multi-layer formation is highly expected, and as shown in the following figure.

This leads to a significant change in the adsorption isotherm, since Freundlich equation has been employed to represent the change in the amount of adsorbate per unit area, or mass of adsorbent with equilibrium concentration of the adsorbate.

Freundlich equation could be written as follows^(11,12):

$$Q = k_F C_e^{1/n} \dots \dots \dots (2)$$

$$\log Q = \log k_F + 1/n \log C_e \dots \dots \dots (3)$$

where Q is the adsorbate quantity (mg/g), C_e is the concentration of adsorbate at equilibrium (mg/L), k_F, and n are Freundlich constants.

Many workers have studied the decolorization of various dye by photocatalysis using ZnO⁽¹³⁾, Fe₂O₃⁽¹⁴⁾, and clay^(15,16). The present study included study the effect of time contact, temperature and irradiation by UV-light (290-320)nm to find out the optimum conditions for the adsorption process for Birllant blue dye.

The detailed kinetic and thermodynamics studies has been employed to investigate the mechanism, rate and order of the sorption process for all pollutants.

Experimental Part

Preparation of Solutions

A solution of 1000 ppm of dye was prepared by dissolving 1g of dye in distilled water in 1L volumetric

flask, thereafter solutions of different concentration were prepared by a subsequent dilution.

Determination of $\lambda_{\max}$

A sample of 1000 ppm dye solution were taken and a spectrum scan were measured using UV - Vis spectrophoto - meter. The dye showed different $\lambda_{\max}$, which shown in Figs. (1).

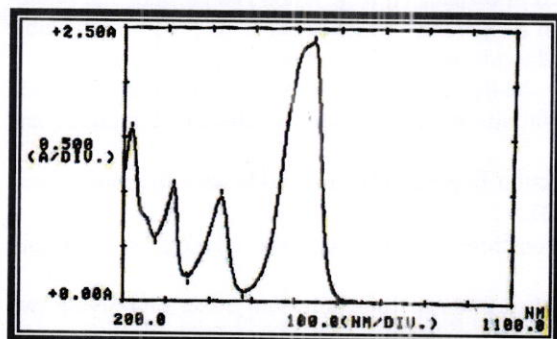


Fig.(1): The $\lambda_{\max}$ for 1000 ppm Brilliant blue solution at 636nm

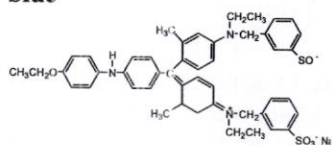
Birlliant blue G250 (Natural Dyes) Specification sheet Structure of Birlliant blue⁽¹⁴⁾

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N-141[4-(4-Ethoxy phenyl) amine] phenyl]-[4-[ethyl[(3-sulfophenyl)methyl] amino]-2-methyl phenyl] methylene]-3-methyl-2,5-cyclohexadien-1-ylidene]-N-ethyl-3-sulfobenzene methanaminium mono-sodium salt

The calibration curve was accomplished by measuring the absorbance of solution (10, 20, 30, 40, and 50 ppm) at 636 nm. Table (1) shows the absorbance of each solution, thereafter plotting of absorbance vs. concentration and as show in Fig.(2).

Table (1): Value of the absorbance for a series of Birlliant blue solutions

Concentration (ppm)	Absorbance
10	0.558
20	0.986
30	1.1417
40	1.731
50	1.927

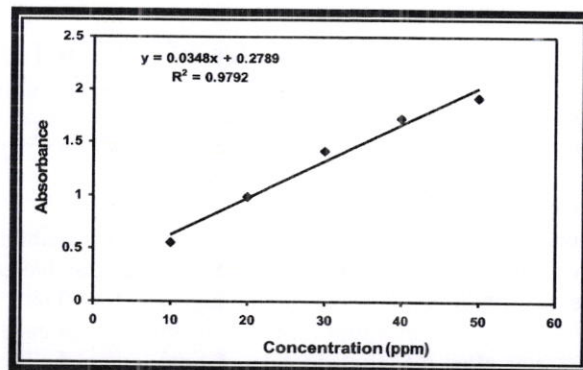


Fig.(2): The calibration curve of a series of Birlliant blue solutions

The quantity of adsorbate was calculated by using the following formula:

$$Q_e = V_{\text{sol}} (C_o - C_e) / M \quad (4)$$

Where

Q_e = Quantity of adsorbate (mg/g).

V_{sol} = Total volume of adsorbate solution (L).

C_o = Initial concentration of adsorbate solutions (mg/L).

C_e = Final concentration of adsorbate solution at equilibrium (mg/L).

M = Weight of adsorbate (g).

The percentage of sorption was calculation by using the following formula:

$$\%Q = \frac{(C_o - C_e)}{C_o} \times 100 \quad (5)$$

where

$\%Q$ = Sorption percentage.

Result and Discussion

The study of the thermal stability of five concentration of dye was achieved after a constant time (30 min.), which revealed that dye is stable at temperature range (288-293)K.

Table (2) shows the absorbance of dye at different temperature after 30 min., the data obtained revealed that dye is entirely stable over range of temperatures and nondecomposable.

Table (2): The absorbance of Birlliant blue at different temperatures after 30 min at 636nm

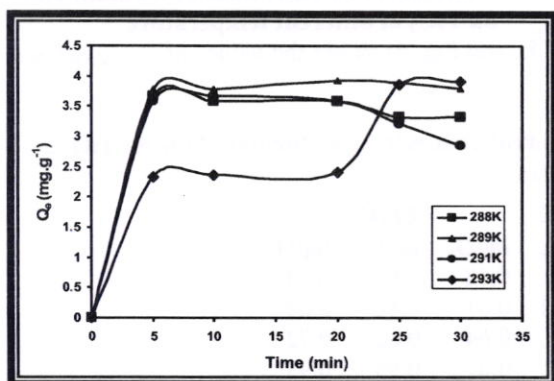
T (°K)	Absorbance of Birlliant blue solution				
	10 ppm	20 ppm	30 ppm	40 ppm	50 ppm
288	0.511	0.508	0.341	0.515	0.515
289	0.470	0.442	0.347	0.347	0.391
291	0.54	0.586	0.449	0.674	0.581
293	0.498	0.690	0.375	0.341	0.432

The photo stability of two concentration of dye revealed that dye is stable afterspecific periods of irradiation by UV-light (290-320)nm, the data obtained proved that the dye is non decomposable even at a prolonged period of exposure to UV-light, and as shown in Table (3).

Table (3): The absorbance of Birlliant blue after different irradiation time by UV-light (290-230)nm

C (ppm)	0 hrs.	1 hrs.	2 hrs.	3 hrs.	24 hrs.
Absorbance					
40	0.220	0.220	0.222	0.224	0.224
1000	1.140	1.140	1.142	1.145	1.145

Fig.(3-1-1) show the variation of Q_e with the contact time for a solution of 40 ppm dye to be in an attachment with 0.1g of TiO_2 . The data obtained revealed that there was considerable increase in the value of Q_e at the temperature range (288-293)K with increase time upto constant Q_e value which indicate the equilibrium time⁽⁷⁸⁾.

**Fig.(3): The variation of Q_e with contact time for 40 ppm Birlliant blue solution at different temperatures****Table (4): The values of C_e and C_e/Q_e for the sorption of different concentrations Birlliant blue dye at different temperatures**

C_0	288K		289K		291K		293K	
	C_e (mg. L ⁻¹)	C_e/Q_e (g.L ⁻¹)	C_e (mg. L ⁻¹)	C_e/Q_e (g.L ⁻¹)	C_e (mg. L ⁻¹)	C_e/Q_e (g.L ⁻¹)	C_e (mg. L ⁻¹)	C_e/Q_e (g.L ⁻¹)
10	6.67	7.01	5.49	8.89	7.15	3.87	5.01	4.32
20	6.58	6.87	4.68	6.51	8.39	4.11	6.36	4.55
30	2.995	1.405	2.92	1.35	9.08	4.39	6.68	5.35
40	6.78	7.18	1.955	0.75	11.35	4.76	9.01	8.62
50	6.78	7.18	3.22	2.22	13.27	4.97	7.73	6.36

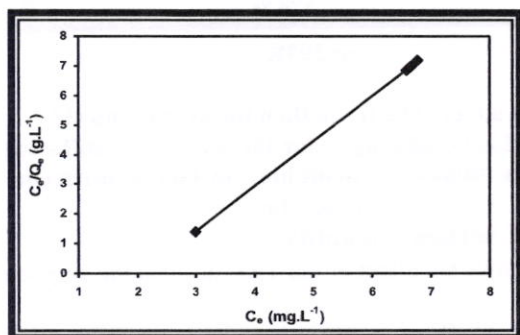
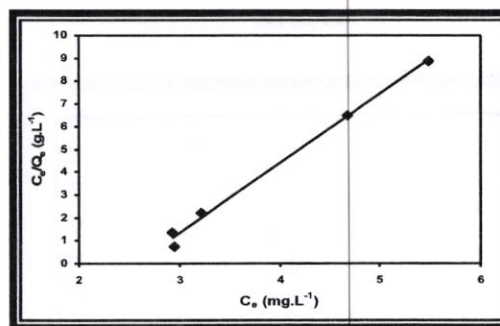
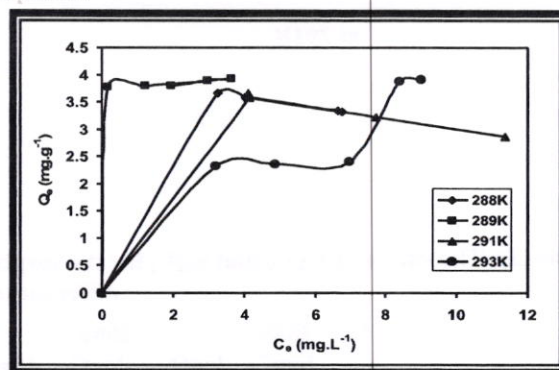
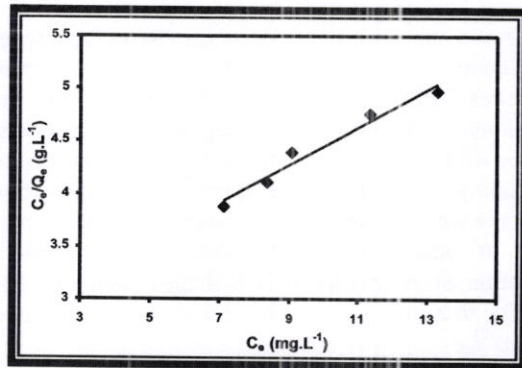
**at 288K****at 289K**

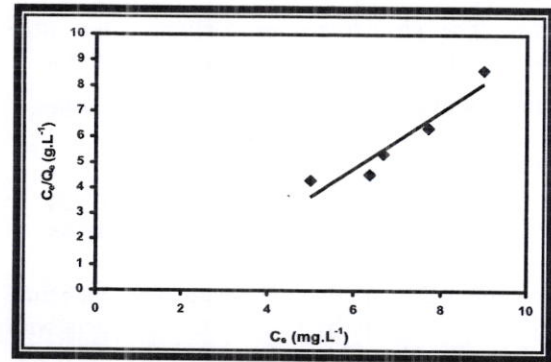
Fig.(4) shows the adsorption isotherm (freundlich type) for different concentrations of dye at different temperatures after equilibrium (30 min.). In the heterogenous photocatalytic degradation process, adsorption of dye on the catalytic surface was assumed to be a primary step of the reaction⁽⁷⁸⁾. The adsorption experiments were carried out to evaluate the equilibrium quantity of adsorbate⁽⁷⁹⁾. According to the modern classification of isotherms, it is S-shapes isotherm, this may be due to a strong interaction between adsorbate and adsorbent⁽¹⁰⁾.

**Fig. (4): The adsorption isotherms of different concentration of Birlliant blue on TiO_2 at different temperatures**

C_e/Q_e against C_e which represented in Table (4) indicate that Langmiur linear plot could be applied as show in Fig.(5).



at 291K



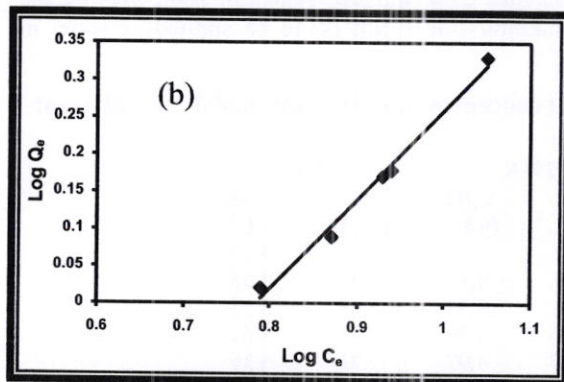
at 293K

Fig.(5 a,b,c,d): The plot of C_e/Q_e against C_e for the sorption of different concentrations of Birlliant blue on TiO_2 at different temperature

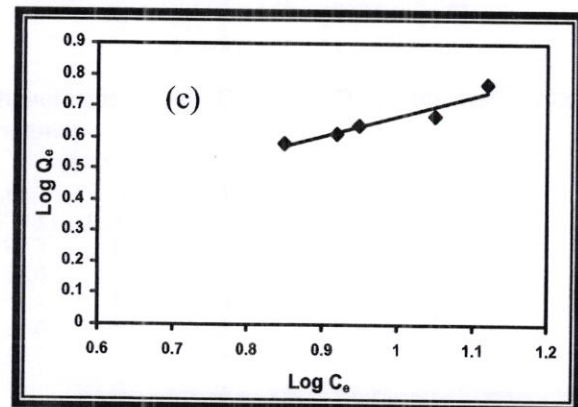
Table (5), and Fig.(6) shows the values of $\log Q_e$ against $\log C_e$

Table (5): The values of $\log Q_e$ and $\log C_e$ for the sorption of different concentrations Birlliant blue on TiO_2 at different temperatures

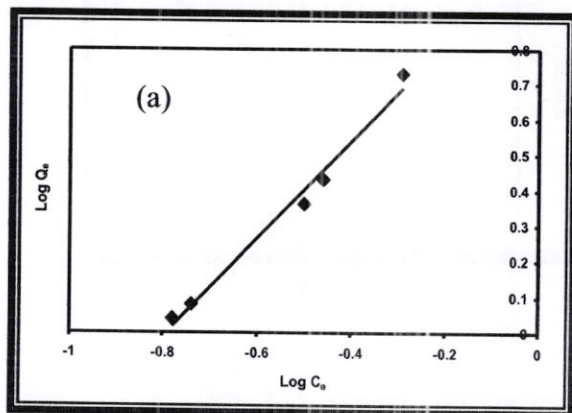
C_o	288K		289K		291K		293K	
	$\log C_e$	$\log Q_e$	$-\log C_e$	$\log Q_e$	$\log C_e$	$\log Q_e$	$\log C_e$	$\log Q_e$
10	0.82	0.2	0.74	0.08	0.85	0.58	0.69	0.63
20	0.81	0.21	0.64	0.04	0.92	0.61	0.80	0.65
30	0.47	0.39	0.46	0.43	0.95	0.64	0.82	0.72
40	0.83	0.2	0.29	0.73	1.05	0.67	0.95	0.93
50	0.83	0.2	0.5	0.36	1.12	0.77	0.88	0.80



at 291K



at 293K



at 289K

Fig.(6 a,b,c): The freundlich linear relation ship between $\log Q_e$ and $\log C_e$ for the sorption of different concentrations of Birlliant blue on TiO_2 at different temperatures

Effect of Sorbent Quantity

Fig.(7) shows the effect of the TiO_2 dose on the sorption process.

The plot of $(C_o - C_e)$ against sorbent quantity $m(g)$, indicated that the sorption increased as the quantity of sorbent increased. Then exhibited a maximum uptake at 0.1g of sorbent.

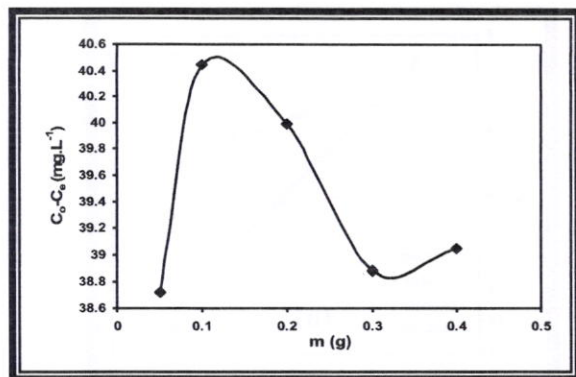


Fig.(7): The effect of TiO_2 on the sorption process for 40 ppm Brilliant blue at 293K

Fig.(8) show that the sorption percentage decrease as the temperature increase.

Knowing that the adsorption process is an exothermic process⁽²⁾, while the absorption process is an endothermic process. However, the sorption process could be exothermic or endothermic depending on the dominant process, in other words the adsorption or absorption or both of them.

Following the sorption percentage in Fig.(8), reveals at 288-293 K, the sorption percentage decreased, and then the proposed mechanism is adsorption, since it is an exothermic process.

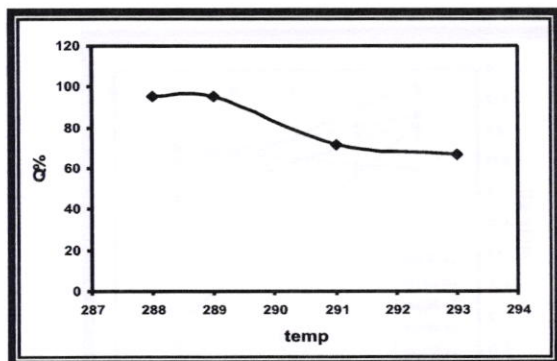


Fig.(8): The effect of temperature on the sorption percentage for 40 ppm

Fig.(9) show that the sorption percentage almost increases as the time increase to 20 min. at different temperature.

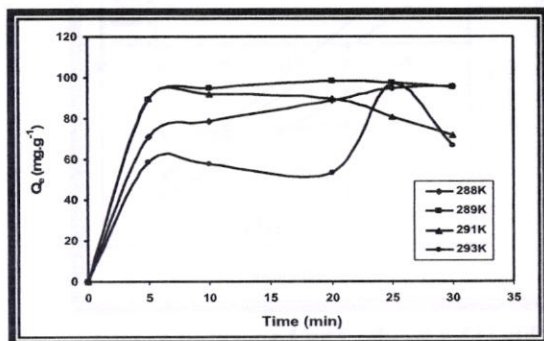


Fig.(9): Effect of time on

- Fig.(10) shows the variation of sorption percentage with different initial concentrations of the dye.
- The data shows the high efficiency of TiO_2 to adsorbed dye at the temperature range (288-293 K). When the initial concentration of dye increase, dye molecules are

adsorbed on to the surface of TiO_2 upto maximum value then the efficiency decreased⁽¹⁷⁾.

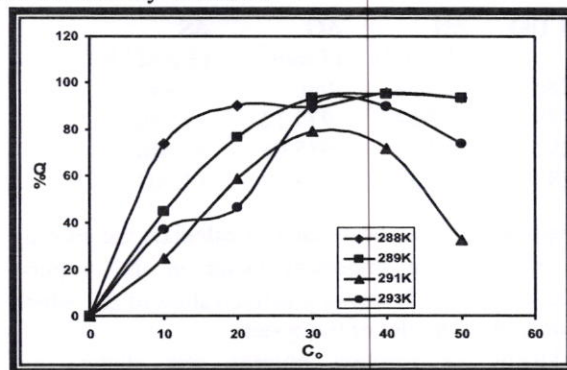


Fig.(10): Effect of initial of dye concentration on sorption percentage

The thermodynamic function ΔH , ΔG , and ΔS were calculated using the following formulas:

$$b = a \cdot \exp(-\Delta H/RT) \dots\dots\dots(6)$$

$$\ln b = \ln a - (\Delta H/R) \cdot 1/T \dots\dots\dots(7)$$

$$\ln a = \Delta S/R \dots\dots\dots(8)$$

$$\Delta G = \Delta H - T\Delta S \dots\dots\dots(9)$$

Where b is the maximum sorption quantity for 50 ppm dye solutions on TiO_2 and could be obtained from the plot of C_e/Q_e versus C_e Fig.(5).

The equation (7) was used to calculated the ΔH , by plotting of $\ln b$ versus $1/T$.

Table (6), Fig.(11), showed a linear relationship and the slope represents $-\Delta H/R$, R is the gas constant ($8.314 \text{ JK}^{-1} \cdot \text{mol}^{-1}$). And the values of ΔH of sorption were calculated.

Table (6): The values of the reciprocal of temperature and $\ln b$

T (K)	$\frac{1}{T \times 10^{-3}}$	b	$\ln b$
288	3.47	3.32	1.19
289	3.46	3.40	1.22
291	3.43	3.86	1.35
293	3.41	3.91	1.36

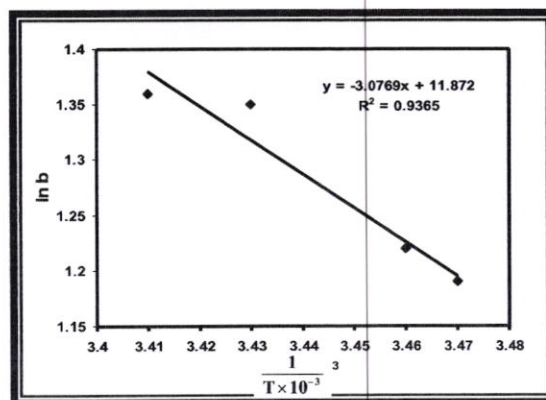


Fig.(11): The plot of $\ln b$ vs. the reciprocal of

Table (7) shows the positive values (endothermic process) of the ΔH at 288, 289, 291, and 293K, which indicate the process is almost absorption process.

Table (7) shows that all values of ΔG were negative, so the sorption of dyes on the surface of TiO_2 was considered a spontaneous process⁽¹⁸⁾.

Table (7): The thermodynamic function values for the dye sorption process at different temperatures

T (K)	ΔH (J.mol ⁻¹)	ΔG (J.mol ⁻¹)	ΔS (J.mol ⁻¹ .K ⁻¹)
288	+25.58	-966.86	+3.446
289	+5.962	-975.12	+9.1906
291	+17.73	-985.05	+3.446
293	+26.57	-999.8	+3.503

The equation (8) has been used to calculate the entropy (ΔS), which is an important factor in the sorption process, and table (7) shows positive values of ΔS , which indicate to the disorder of the system.

The kinetic of sorption process was studied via introducing three models, there after calculate the rate constant and the energy of activation of sorption process.

A- Lagergren Model

The rate constant k_{sorp} was determined from the following pseudo-1st order rate expression, which could be expressed as follows:

$$\ln(q_e - q_t) = \ln q_e - k_{sorp} t \dots\dots\dots(10)$$

Where q_t and q_e are the amount sorbed at time t /min. and at equilibrium time successively, and k_{sorp} /min⁻¹ is the rate constant, all values are given.

The liner relationship was obtained via $\ln(q_e - q_t)$ versus t /min., and as shown in Fig.(12).

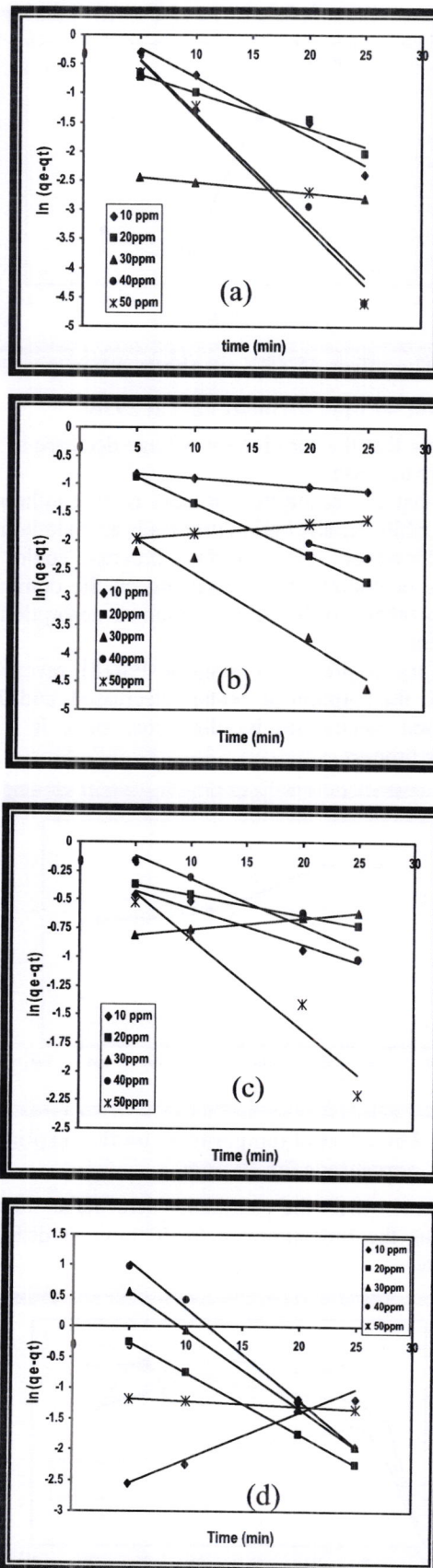


Fig.(12 a,b,c,d): The Lagergren kinetic model at different

Table (8): The rate constants of adsorption process at different temperature

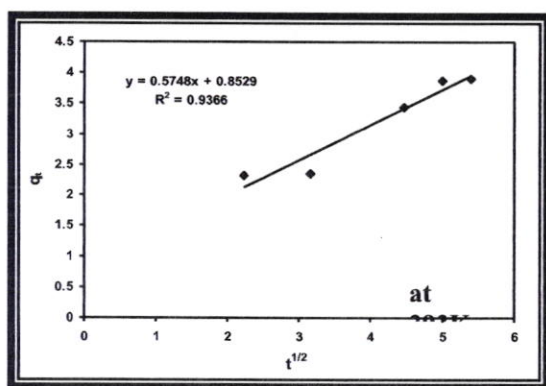
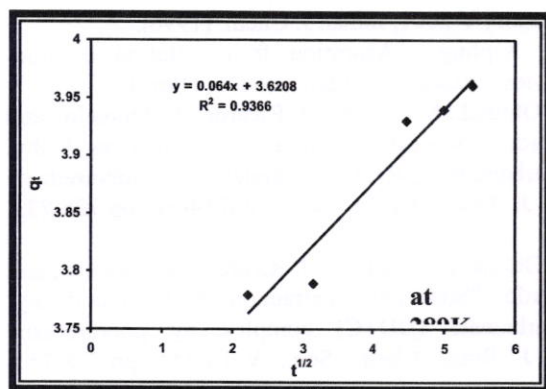
C_0	$k_{ad} \text{ (min.}^{-1}\text{)}$			
	288K	289K	291K	293K
10	0.0414	0.1428	0.0666	0.4132
20	0.265	0.296	0.203	0.157
30	0.689	0.655	0.355	0.0035
40	0.386	0.681	0.287	0.026
50	0.438	0.706	0.409	0.552

B- Morris-Weber Model⁽¹⁹⁾

This kinetic model was introduced by Morris and Weber (Swedish Scientists) in 1963, and by using this kinetic model, the rate limiting step of any physi-sorption process could be easily estimated, and they have presented an equation which could be expressed as follows:

$$q_t = k_d \sqrt{t} \dots \dots \dots (11)$$

Where q_t is the quantity of the sorbed material at any time (mg/g). k_d is the diffusion constant, and t is the time of diffusion (min). The plotting of q_t versus $\sqrt{t}$ was accomplished at 289, and 293K and as shown below in Fig.(13).

**Fig.(13): The plot of q_t vs. $t^{1/2}$ at 289K and**

The slop represents the k_d value, which was considerably greater at 289 K than at 293 K, and this was attributed to the following reasons:

- 1-Birlliant blue dye adsorbed on TiO_2 from the solution.
- 2-A strong binding between the adsorbate and adsorbent.
- 3-The value of k_d at 289 K was 1.269, and at 293K was 1.291.

C- Reichenbery Model⁽²⁰⁾

This kinetic model was proposed to discuss the behaviors of many sorption processes in solution and Reichenbery has introduced the following formula:

$$F = [1 - 6/\pi^2] e^{-Bt} \dots \dots \dots (12)$$

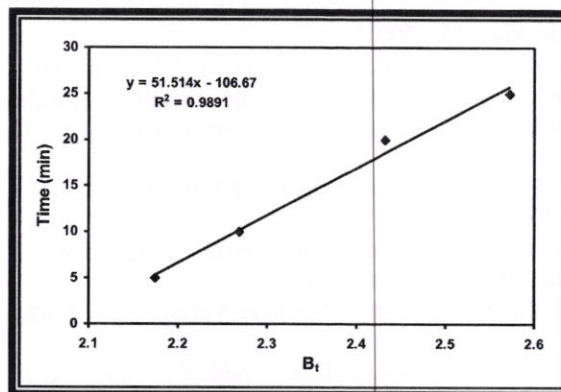
$$B_t = -0.4977 - \ln(1-F) \dots \dots \dots (13)$$

$$F = q_t / q_e \dots \dots \dots (14)$$

where: F , B_t are mathematic parameters

Plotting of time (min.) versus B_t revealed a linear relationship with relatively acceptable R^2 value and as shown in Fig.(14).

According to this model, it characterized the rate determining mechanism which was a diffusion process for the dye molecules in the bulk solution and absorption occurred.

**Fig.(14): The variation of B_t with time at**

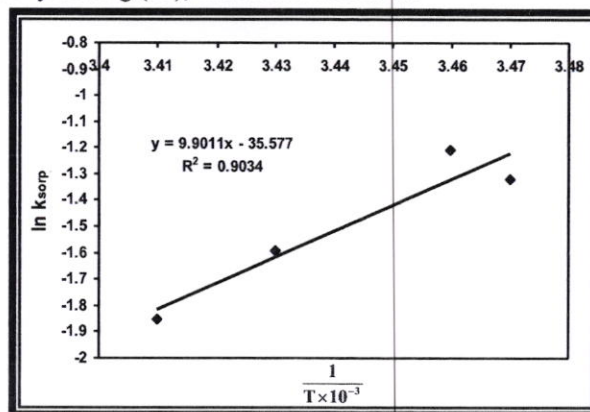
The activation energy of the sorption process was calculated via introducing Arrhenius equation as shown:

$$\ln k_{sorp} = \ln A - E_a / RT \dots \dots \dots (15)$$

where k_{sorp} are calculated from Lagergreen equations and A is Arrhenius parameter, which is a temperature independent factor in the temperature study range (288-293) K.

E_a is the activation energy (kJ.mol^{-1}) and R is the gas constant ($8.314 \text{ J.K}^{-1}.\text{mol}^{-1}$), and T is the temperature in Kelvin.

Plotting of $\ln k_{sorp}$ against the reciprocal of temperature in K^{-1} yield Fig.(15), and as shown below.

**Fig.(15): The plot of $\ln k_{sorp}$ vs. reciprocal of temperature**

This Fig.(15), reveals a linear relationship between the $\ln k_{sorp}$ and the reciprocal of temperature and thereafter from the slope, the value of E_a was obtained, which has a very important role in estimating the dynamic of the sorption process, and it represents the energy barrier for a

process to occur, the activation energy of the sorption process was equal to $39.011 \text{ kJ.mol}^{-1}$.

Conclusions

Birlliant blue dye shows a good ability to adsorption on TiO_2 surface. This is due to its dye aromatic structure, and due to the existence of alkyl group (with drive electrons) on the dye aromatic rings. The second reason of a good ability of dye adsorption is due to its high

molecular weight and large surface area. So the most dye molecules will be removed by TiO_2 surface.

Titanium dioxide succeeded in removing; 95.54% of dye. The sorption process of dye give a positive ΔH values, negative ΔG values and positive ΔS values, which indicates the spontaneous absorption process.

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دراسة امتزاز وامتصاص صبغة ال (birllant Blue) على ثنائي اوكسيد التيتانيوم

خلود عبد صالح السعدي¹ قيس محمد عبد الحسين²

¹ قسم الكيمياء ، كلية العلوم ، جامعة بغداد ، بغداد ، العراق

² قسم الكيمياء ، كلية العلوم ، جامعة النهرين ، بغداد ، العراق

الملخص

يتضمن موضوع الدراسة إمكانية إزالة صبغة الـ Birllant blue على ثنائي اوكسيد التيتانيوم. تمت دراسة الاستقرار الحراري والضوئية لهذه الصبغة في المحاليل المائية وتبين انها ثابتة عند المدى الحراري قيد الدراسة (288-318)K وكذلك ثابتة ضمن الطول الموجي للأشعة فوق البنفسجية عند المدى (290-320) nm. نجح ثنائي اوكسيد التيتانيوم في إزالة 95.54% من الصبغ. تم استخدام تقنية مطيافية الأشعة فوق البنفسجية لمتابعة تركيز الصبغة قبل وبعد إجراء عمليات الامتزاز وبعد اوقات زمنية مختلفة وقد تم الحصول على متساويات درجات الحرارة (إيزوثيرمات الامتزاز) من نوعها إيزوثيرمات Langmuir و Freundlich وبقيم معامل الارتباط تصل (1-0.9993).

أخذ شكل إيزوثيرم الامتزاز للصبغة شكل حرف S والذي يعود الى قوة ارتباط الصبغة بالسطح ثنائي اوكسيد التيتانيوم وبقيم ثرموديناميكية $\Delta H = (5.962-26.57)J$ $\Delta G = -(996-999)J$ $\Delta S = +(3.45-9.1906)J$.

الدراسة الحركية للامتزاز الملوثات على سطح ثنائي اوكسيد التيتانيوم اعتمدت ثلاث معادلات حركية: (Legergren equation)، (Morris-Weber model)، (Reichenberg model) (model).

هذه العلاقة تشير الى ان عملية الامتزاز تتضمن عملية امتصاص المحددة للسرعة وان مجمل العملية هي (امتزاز وامتصاص).