



Original Research Article

Adsorption of Methyl Red Dye from Aqueous Solution Using Gamma Alumina Nanoparticles

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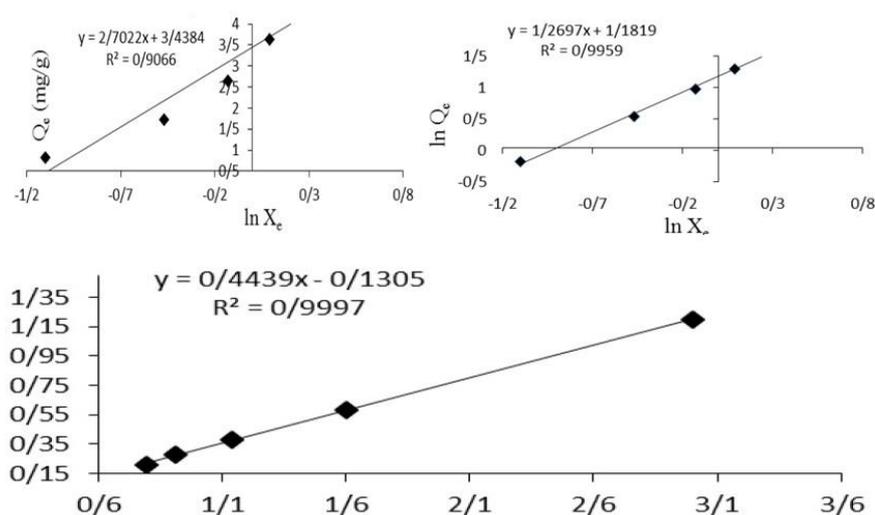
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ABSTRACT

This study focused on gamma alumina nanoparticles used for the adsorption of the cationic dye methyl red from aqueous solutions. Batch adsorption studies were carried out to investigate various parameters including pH, gamma alumina nanoparticles dosage, and temperature and contact time. The concentration of dye methyl red was measured using a UV-vis Spectrophotometer at the wavelength of 410 nm. The optimum adsorption conditions were found to be pH=3, adsorbent dose=0.04 g, temperature=298 K and contact time=50 min. The experimental results of this work were compared with Langmuir, Freundlich and Tamkin's isotherm models. The results obtained from isotherm models showed that the surface adsorption of these dyes on the adsorbent followed the Langmuir isotherm model. The analysis of thermodynamic data showed that the adsorption process of the studied dyes on the adsorbent surface is spontaneous (The negativ value of ΔG^0) and exothermic (The negativ value of ΔH^0).

GRAPHICAL ABSTRACT



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Introduction

Wastewater from various industries such as textiles, leather processing, paper production and food industries is usually contaminated with dyes. Depending on the type of paint used, the type of paint in the effluents will be also different. Since most dyes used in the textile industry are organic and probably derived from the salts diazaphthalocyanine and anthraquinone, which are derivatives of the benzene ring, which can be toxic and carcinogenic. Therefore, purification and removal of dye in such waters seems to be necessary [3-5].

More than ten thousand types of synthetic dyes are produced annually in the world, which are widely used in various industries. So, their effluents contain significant amounts of organic dyes. The presence of organic dyes in industrial effluents causes irreparable damage to the environment due to their toxic effects and preventing the penetration of sunlight into the water and disrupting photosynthesis. Therefore, the treatment of colored effluents is necessary before discharging them to the environment. Removal of dyes and reduction of their concentration is done by methods such as coagulation, adsorption, oxidation processes, nanofiltration, etc. Water scarcity has posed a serious threat to people, both quantitatively and qualitatively, especially in some countries. As a result, science and technology have cooperated to tackle this problem by developing and applying different methods.

By creating a suitable bed, the conditions for access to safe, quality and affordable water can be provided. Undoubtedly, using new methods, especially nanotechnology, allows us to purify water with the mentioned conditions. Due to the effective removal of pollutants and reducing the cost of producing healthy water, the use of nanotechnology has received more attention and acceptance than the old methods. Pigments in drinking water are considered very dangerous not only because of their appearance but also because of the origin of their trihalomethane

production. When combined with chlorine, it causes the formation of chloroform and other harmful and carcinogenic halogen compounds [6].

Material and methods

In 250 ml humans, a certain amount of adsorbent (0.010 g of gamma alumina nanoparticles) was poured. Then 50 ml of dye solution (Congo red, Methylene Blue, Malachite Green or Methyl Red) with a certain concentration (standard concentration range) and different pH was added to it. Also, adjusting the temperature to about K298 and stirring the solutions for 40 minutes was done by means of a magnetic stirrer, and smoothing the mixture and absorbing (A) of each of the solutions was carried out under a filter paper. Spectrophotometry was measured at the specified maximum color wavelength. In addition, it had the adsorption of solutions. Equation 1-1% equilibrium adsorption (% Ae) was used for solutions with different pH. The pH of the solution with the highest adsorption percentage is the optimal Ph [7-10].

Determining the optimal amount of gamma alumina nanoparticles

We poured different masses of gamma alumina nanoparticles into 250 ml and added 50 ml of dye solution (Congo red, Methylene Blue, Malachite Green or Methyl Red) with specified concentration (standard concentration range) and optimized pH of the previous step to each human [11-15]. The temperature was adjusted to about K298 and the solutions were stirred with a magnetic stirrer for 40 minutes. The mixture was filtered and the absorbance (A) of each of the solutions under the filter paper was measured by spectrophotometry at the specified maximum color wavelength. The mass of the adsorbent that has the highest percentage of equilibrium adsorption (Equation 1) is the characteristic of the optimal amount of adsorbent [16-19].

Determining the optimal temperature

Dye solutions (Congo red, Methylene Blue, Malachite Green or Methyl Red) were prepared following the previous step, so the pH and the adsorbent mass were optimized. The first solution was stirred at K298 for 40 min and its absorbance was measured by spectrophotometry at the specified maximum color wavelength. The same procedure was done for other solutions and at different temperatures, and in each case the adsorption of the solution under the filter was measured. The temperature that has the highest equilibrium absorption percentage (Equation 1) is the optimal temperature characteristic [20-22].

Determining the Optimal Time

In 250 ml humans, 50 ml of a dye solution (Congo Red, Methylene Blue, Malachite Green or Methyl Red) was prepared at a specific concentration (standard concentration range), all of which contained optimized adsorbent mass, optimum temperature and optimum pH. One of the solutions was strained after 10 minutes and the absorbance of the solution under the filter paper was determined by spectrophotometry at maximum wavelength. Other solutions were measured at 10 min smooth intervals and their adsorption was measured at the maximum color wavelength. Then the equilibrium adsorption percentage (Equation 1) was determined and when it had the highest equilibrium adsorption percentage, the time characteristic was optimal [23].

Determining the best isotherm of dye adsorption process on gamma alumina nanoparticles

Solutions with different concentrations (standard concentration range) of dye (Congo red, Methylene Blue, Malachite Green or Methyl Red) with optimal conditions (pH, adsorbent, temperature and contact time) were prepared and adsorbed to each solution (A) [24-26].

$$\% A_e = \frac{A_0 - A_e}{A_0} \times 100 \quad (1)$$

Q_e , amount adsorbed per unit weight of adsorbent at equilibrium (mg/g) was calculated using the following equation. Where X_0 and X_e are the initial and final concentrations of dye methyl red in solution (mg/L), respectively [27-29].

$$Q_e = \frac{(X_0 - X_e)V}{M} \quad (2)$$

Where M is the mass of gamma alumina nanoparticles (g) and V is the volume of the solution (L). To evaluate the thermodynamic properties of the adsorption process, 0.04 g of gamma alumina nanoparticles was added into the 50 ml solution with pH of 3.0 and initial dye methyl red concentration ranging from 5 mg/L in every experiment. Each solution was shaken continuously for 50 min [30-32].

Factors affecting the intensity of adsorption

Many factors such as contact surface, initial dye concentration, temperature, structure of adsorbed and adsorbent material, state of adsorbed and adsorbent material, pH of the medium and amount of adsorbent affect the adsorption process. Optimizing these conditions is very important for performing the process on an industrial scale. Factors affecting adsorption are:

1- Contact surface: As the contact surface increases, the amount of adsorption increases. The best absorbers are materials that have smaller particles, in other words, have a higher contact surface.

2- Initial dye concentration: The amount of dye absorption depends on the initial dye concentration in solution. In general, the percentage of contaminant adsorption on the adsorbent surface decreases with increasing its initial concentration in solution. Increasing the initial concentration occupies all active surfaces of the adsorbent and the adsorbent surface saturated with contaminants. On the other hand, increasing the concentration of dye in the solution increases the driving force in mass transfer, which is the primary cause of adsorption phenomena. The amount of adsorbed

substance per unit mass of adsorbent (adsorbent) is a function of the concentration of solute. Examination of these two quantities at a constant temperature leads to a surface adsorption isotherm. These isotherms have studied by several people and the most familiar of which are the Freundlich and Langmuir isotherms.

3- Temperature: Temperature changes the absorber capacity. Hence, it is one of the most important parameters in the adsorption process. If the rate of adsorption increases with increasing temperature, the adsorption process is endothermic. As the temperature increases, the particle mobility and the number of active sites for adsorption increase. If the rate of adsorption decreases with increasing temperature, the adsorption process is hot. In this case, increasing the temperature reduces the adsorption force between the adsorbent surface and the pollutant molecules. In physical adsorption, increasing the temperature reduces the adsorption rate, but in cases where the adsorption is accompanied by a chemical reaction, then if the reaction between the adsorbent and the adsorbent is superheated, increasing the temperature will increase the adsorption rate and if the adsorption reaction is exothermic, increasing the temperature will reduce the adsorption.

4- The structure of the adsorbed and adsorbent material: The structure of the adsorbed and adsorbent material affects the amount of surface adsorption so that some adsorbents have a high adsorption power relative to an adsorbent but relative to the adsorbed; others have less absorption power.

5- State of adsorbed and adsorbent material: The physical state of adsorbent and adsorbed in turn is effective in the rate of absorption.

6- Environmental pH: One of the important factors in the adsorption process is the pH of the solution. Changing the pH changes the degree of ionization of the adsorbed molecules and the surface properties of the adsorbent. Both adsorbent and adsorbent properties are effective in determining the optimum pH for the

adsorption process. Many studies have done to investigate the adsorption of various color contaminants on different adsorbents in order to determine the optimal pH. The ability to adsorb the surface and the type of active points to adsorb on the adsorbents are evaluated at the pointless pH (pH_{pzc}). At this pH, the surface charge of the material is zero, and this parameter is usually used to evaluate the electrostatic properties of the surface. This parameter occurs only in environments where the ions H^+ and OH^- determine the potential of the environment. Lots of research has been done to find a charge less point for various attractions to investigate the mechanism of adsorption. Due to the presence of hydroxyl groups on the surface of the adsorbents at a pH higher than the no-charge point and the existence of active points for negative charge adsorption in these media, the rate of adsorption of cationic dyes in an environment with a pH higher than the point is no more load. At pH less than the point without charge, the adsorbent surface has a positive charge; therefore, the absorption of anionic dyes in these environments increases.

7- Effect of adsorbent amount: In general, with increasing the amount of adsorbent in solution, the amount of contaminant absorption increases. As the amount of adsorbent increases to the optimal amount, the amount of adsorption increases. After the optimum point, increasing the amount of adsorbent has no effect on the absorption of contaminants. Therefore, determining the optimal amount of adsorbent for each contaminant in the process environment is a very important parameter from an economic point of view.

Result and Dissection

The Effect of pH

Effect of pH changes on Congo surface adsorption on the surface of gamma alumina nanoparticles was investigated. The data obtained from the effect of solution pH changes on the amount of condensate absorbed at a concentration of 6 mg / l are reported in Table 1 and Figure 1.

Table 1: The effect of initial pH of the solution on the adsorption percentage (% A) of dye methyl red ($X_0=5$ mg/L, M gamma alumina nanoparticles =0.01 g, $T=298$ K, $t_c=30$ min)

pH	%Ae
2	63.52
3	65.52
4	56.80
5	42.10
6	14.52
7	9.63
8	6.71

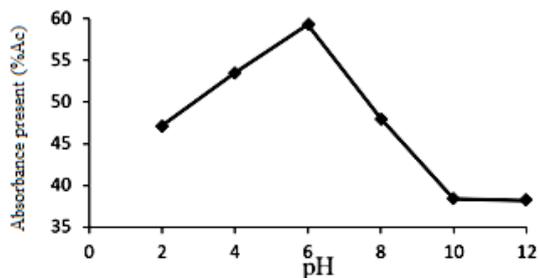


Figure 1: Effect of solution pH changes on surface adsorption of Congo rejection. According to the data shown in Table 4-2 and Figure 4-2, the optimum pH is 6 [16-18].

The Effect of Adsorbent Dosage

Microcosms with different adsorbent doses (0.01-0.08 g) were amended with 5 mg/L of dye methyl red in aqueous solutions. The rate of adsorption was monitored at the following optimum conditions: pH of 3, for 30 min at 25°C. The effect of gamma alumina nanoparticles dosage on the adsorption percentage of dye methyl red is shown in Table 2. The best results were obtained at M gamma alumina nanoparticles =0.04 g for dye methyl red.

Table 2: The effect of gamma alumina nanoparticles dosage on the adsorption percentage (% A) of dye methyl red ($X_0=5$ mg/L, pH=3, $T=298$ K, $t_c=30$ min)

M gamma alumina nanoparticles (g)	%Ae
0.01	65.52
0.02	66.06
0.03	67.32
0.04	70.42
0.05	69.32
0.06	68.96
0.07	68.78
0.08	68.78

Table 3: The effect of temperature on the adsorption percentage (% A) of dye methyl red ($X_0=5$ mg/L, M gamma alumina nanoparticles =0.04 g, pH=3, $t_c=30$ min)

T (K)	%Ae
298	70.42
308	69.51
318	67.69
328	64.79
338	61.52

Table 4: The effect of contact time, t_c , on the adsorption percentage (%A) of dye methyl red ($X_0=5$ mg/L, M gamma alumina nanoparticles =0.04 g, pH=3, $T=298$ K)

Tc (min)	%At
10	61.16
20	65.88
30	70.42
40	72.05
50	74.59
60	74.59

Effect of adsorbent value changes on the surface adsorption of Congo rejection on the surface of gamma alumina nanoparticles

The experimental data for determining the optimal amount of gamma alumina nanoparticles for Congo red adsorption at a concentration of 6 mg/l on the surface of gamma alumina nanoparticles are reported in Table 5.

Table 5: The effect of changes in the amount of gamma alumina nanoparticles on Congo surface adsorption under conditions ($T = 298$ K, $t = 40$ min and pH = 6)

Absorbent amount (g)	0.01	0.02	0.03	0.04	0.05	0.06	0.07
%Ae	59.32	60.94	68.51	75.67	70.40	68.64	64.68

Adsorption Isotherm

Adsorption isotherms (Table 5) represent the relationship of the amount of dyes adsorbed with the adsorbent dose. These provide information about the mechanism of adsorption and the absorptivity of the composite towards the dyes of interest. In this study, Langmuir, Freundlich and Temkin isotherms were investigated [3-23]

Table 5. Adsorption data for dye methyl red adsorption onto gamma alumina nanoparticles (PH =3, t_c=50 min, T=298 K, M gamma alumina nanoparticles =0.04 g)

Parameter	Value				
X ₀ (g/L)	1	2	3	4	5
%A	66.66	68.75	70.77	72.59	74.59
X _e (g/L)	0.33	0.63	0.88	1.10	1.27
Q _e (mg/g)	0.83	1.72	2.65	3.63	4.66
lnX _e	-1.10	-0.49	-0.13	0.09	0.24
lnQ _e	-0.18	0.54	0.98	1.29	1.54
1/X _e (L/g)	3.00	1.60	1.14	0.91	0.79
1/Q _e (g/mg)	1.20	0.58	0.38	0.28	0.21

There is no interaction between molecules adsorbed on neighboring sites; adsorption on surface localized, which points to adsorbed atoms or molecules adsorbed at definite and localized sites. The linear form of the Langmuir isotherm is described by the equation (3) [11-23]:

$$\frac{1}{Q_e} = \frac{1}{bQ_m} \left(\frac{1}{X_e}\right) + \frac{1}{Q_m} \tag{3}$$

Where Q_m (mg/g) is the maximum dyes to adsorb onto 1 g adsorbent and b (L/mg) is the Langmuir constant related to adsorption capacity and energy of adsorption. The slope and intercept of plot of 1/Q_e versus 1/X_e are shown in Figure 1, used to calculate the values of b and Q_m (table 6). The Freundlich isotherm is expressed as equation (4):

$$\ln Q_e = \ln P + \frac{1}{n} \ln X_e \tag{4}$$

Another empirical equation, the Temkin equation, describes the adsorption of hydrogen onto platinum electrodes within the acidic solutions. The model is given by equation (5):

$$Q_e = B_T \ln A_T + B_T (\ln X_e) \tag{5}$$

Where A_T (L/mg) is Temkin isotherm constant. A_T is related to binding constant and B_T (J/mol) is the Temkin constant related to the heat of sorption. B_T and A_T are determined from the slope and intercept of a plot of Q_e versus ln Ce (table 5 and fig. 3). We used these results to calculate the values of B_T and A_T (Table 6).

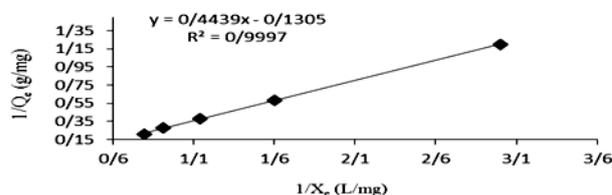


Figure 1: Langmuir isotherm for dye methyl red adsorption onto gamma alumina nanoparticles

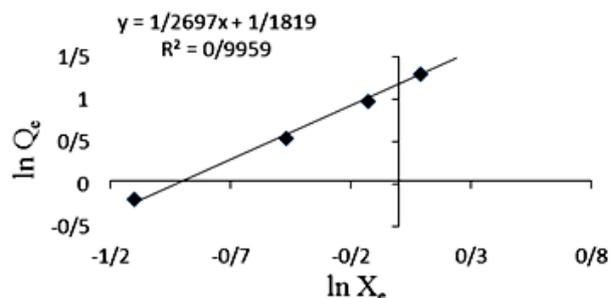


Figure 2: Freundlich isotherm for dye methyl red adsorption onto gamma alumina nanoparticles

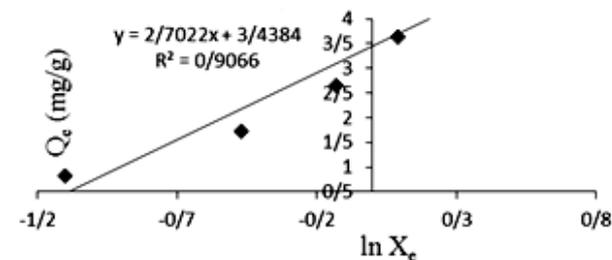


Figure 3: Temkin isotherm for dye methyl red adsorption onto gamma alumina nanoparticles

Table 6: The resultant values for the studied isotherms in connection with dye methyl red adsorption onto gamma alumina nanoparticles

Isotherm	Parame	Value
Freundlich	P (L/g)	3.26
	n	0.79
	R ²	0.9959
Langmuire	b (L/g)	0.29
	Q _m (mg/g)	7.66
	R ²	0.9997
Temkin	B _T (J/mol)	2.70
	A _T (L/mg)	3.54
	R ²	0.9066

Properties of Alumina

Aluminum metal is a highly reactive substance with atmospheric oxygen, and a protective layer of alumina (4 nm thick) created on the exposed part of the air in 100 picose seconds. Aluminum oxide is an electrical insulator. However, it has a relatively high thermal conductivity (wm-1K-130). This thermal conductivity changes with temperature and the reported number expressed as average. The thermal conductivity of alumina is also high among ceramics. Due to the high melting point of alumina, this material has high thermal resistance and stability. The high hardness of corundum (the most abundant crystalline form available), called α -alumina, has led to its use as a suitable component for abrasive and cutting tools. The formation of aluminum oxide on the surface of aluminum metal is a protective factor against weathering. This oxide layer prevents all aluminum from oxidizing. Some alloys, such as aluminum bronzes, use anodizing properties to improve their corrosion resistance. The alumina produced by the anodizing process is amorphous, but can be formed by processes such as electrolytic oxidation of the plasma to form a crystalline layer of alumina and increase the hardness of the alumina. Aluminum oxide was included in the Environmental Protection Agency's list of toxic substances in 1998, albeit in the form of fiber. Alumina has good dielectric strength. It is not a solid electrolyte and therefore does not act like

zirconium oxide (ZrO₂) and its dielectric properties do not depend on oxygen pressure.

Types of alumina phases

Alumina exists in eight different structures, seven of which are thermally semi-stable (γ , δ , κ , ρ , η , θ , χ) and α alumina. The semi-stable phases of alumina in nature are nano crystalline and be easily synthesized by various methods. The type of crystal formed during calcination depends on the temperature, atmosphere, and impurities in the bauxite. The usual method of synthesizing α -Al₂O₃ is heat treatment of transfer alumina powder at a relatively high temperature (1000-1200 °C).

Alpha alumina phase

Alumina (corundum), transparent and colorless and its single crystalline form are known as sapphire. Sapphire is the same as alumina alloyed with a small amount of chromium, and the jewel of sapphire is alumina combined with iron and titanium. The structure of corundum is similar to other trivalent oxides such as Cr₂O₃, Ti₂O₃ and Fe₂O₃.

Alumina theta phase

The theta alumina phase becomes quasi-stable at about 1050 °C. The density of this phase is 23600 Kg/m, which is lower than the α phase (24000 Kg/m). The structure θ is based on the fcc oxygen network in which, in this oxygen configuration, half of the aluminum ions occupy the octahedral vacancies and the other half fill the tetrahedral vacancies (with 4 oxygen neighbors).

Gamma alumina phase

Aluminum oxide nanoparticles with gamma phase, small size, high activity and low melting point can be used to produce artificial sapphire by thermal melting technique. The gamma phase of aluminum oxide nanoparticles with high contact surface and high activity can be transformed into micro-porous spherical structure or honeycomb structure of catalysts. These types of structures are extraordinarily

catalytic carriers. If used as industrial catalysts, it will be the base material for oil refining, petrochemicals and car exhaust refining. In addition to the gamma phase, aluminum oxide nanoparticles can be also used as analytical reagents. Due to its low surface energy and therefore high effective surface area, gamma alumina is widely used as a catalytic shield. In high temperature applications, one problem with the use of gamma alumina is that at temperatures of 800-700 °C this phase is converted to the theta phase.

Thermodynamic Parameters

The amounts of adsorption of dye methyl red were determined by gamma alumina nanoparticles in temperature 298-338 K in vessel. Analysis of thermodynamics of equilibrium adsorption data can give more important than that on adsorption process. Thermodynamic parameters such as change in free energy (ΔG^0), enthalpy (ΔH^0) and entropy (ΔS^0) were determined using the following equations. In the present study, thermodynamic parameters ΔH^0 , ΔS^0 and ΔG^0 were calculated by using the equation. The thermodynamic parameters of adsorption process can be determined from the variation of thermodynamic equilibrium constant, K_0 , where K_0 is defined as follows [5-25]:

$$K_0 = \frac{a_s}{a_e} = \frac{Q_e}{X_e} = \frac{X_0 - X_e}{X_e} \quad (6)$$

Where α_s and α_e are the activity of adsorbed dye methyl red and the activity of dye methyl red in solution at equilibrium, respectively. The adsorption standard free energy change (ΔG^0) is calculated according to the following equation:

$$\Delta G^0 = -RT \ln K_0 \quad (7)$$

The average standard enthalpy change (ΔH^0) and the average standard entropy change (ΔS^0) are obtained from the plot of equation (8) [5-25]:

$$\ln K_0 = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (8)$$

The ΔH^0 and ΔS^0 values obtained from the slope and intercept of plots are presented in Table 7 and Figure 4.

Table 7: The effect of temperature on K_0 values ($X_0=5$ mg/L, pH=3, M gamma alumina nanoparticles =0.04 g, $t_c=50$ min)

T (K)	lnK ₀
298	0.87
308	0.82
318	0.74
328	0.61
338	0.47

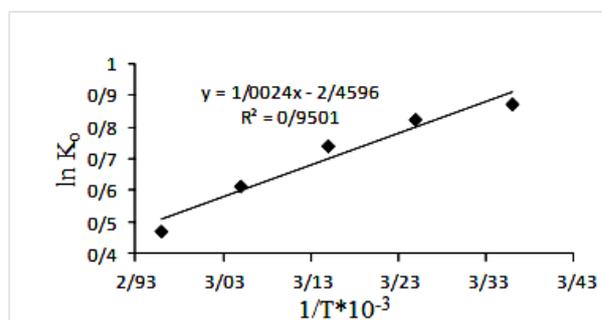


Figure 4: The effect of temperature on equilibrium constant values

Table 8: Thermodynamic parameters for adsorption dye methyl red onto gamma alumina nanoparticles

T /K	ΔG^0 (J/mol)	ΔH^0 (J/mol)	ΔS^0 (J/mol K)
298	-2149.39	-8.33	-20.45
3308	-2087.80		
318	-1968.79		
328	-1663.59		
338	-1328.38		

The results show that the change for adsorbent (from 0.01 to the optimal amount of adsorbent) has caused changes for adsorption. Because with increasing the amount of adsorbent, the number of available adsorption stations increases and the efficiency of the adsorbent for adsorption of dyes increases. By increasing the amount of adsorbent (more than the optimal amount), the available locations of the adsorbent are too much and all the molecules are in the required places, so the adsorption efficiency remains constant. At temperatures above K298, an increase in

temperature is associated with a decrease in surface adsorption, so the adsorption of dyes on gamma alumina nanoparticles is exothermic. As the temperature rises above K298, the adsorbent-adsorption interactions are disrupted and the adsorption percentage decreases. On the other hand, the negative value of enthalpy changes confirms the isothermal process of adsorption of dyes on the adsorbent. A high percentage of adsorption occurs in the early time range, which may be due to the fact that during the first minutes, the number of empty sites (adsorbent active sites) on the surface of gamma alumina nanoparticles is very high, so the dye molecules are absorbed more but with increasing time, the sites fill up and thus the rate of absorption decreases. On the other hand, it can be said that at the time of contact, the optimal absorption and desorption efficiencies of the dyes are equalized and reached equilibrium, and the adsorption (dye molecules) is in contact with the adsorbent after time. Equilibrium will have little effect on the amount of absorption. According to the experimental results of isothermal models, it can be said that these dyes follow Langmuir's isotherm and are probably a single-layer adsorption. The results of calculating the thermodynamic functions show that at experimental temperatures the values of the thermodynamic functions ΔG and ΔS were less than zero. Therefore, the negativity of ΔG means that the adsorption of dyes on gamma alumina nanoparticles is spontaneous. Considering the amount of enthalpy changes in the adsorption of dyes on the adsorbent surface, it can be seen that the adsorption of dyes on the adsorbent surface is probably physical. Because the process of adsorption of dyes on the surface of the adsorbent is exothermic, according to the Lusatie principle, with increasing temperature, the amount of adsorption decreases, which is accompanied by a decrease in irregularity (negative ΔS).

Conclusion

Comparing the quadratic power of the correlation coefficient of the kinetic models, we can say that the Congo Red-adsorption kinetics on the adsorbent of gamma alumina nanoparticles follows the quasi-second-order kinetic model. To investigate the adsorption mechanism (determining the number of adsorption steps and the slowest step), the adsorption capacity diagram against the time root was drawn. Using the slope of the line and the width from the origin of the diagram, the constant variables of velocity and thickness of the boundary layer were calculated. To determine the best kinetic model of this experimental work, with the help of experimental data, diagrams of quasi-first-order, quasi-second-order kinetic models were drawn. The results showed that the change for adsorbent (from 0.01 to the optimal amount of adsorbent) has caused changes for adsorption. Because with increasing the amount of adsorbent, the number of available adsorption stations increases and the efficiency of the adsorbent for adsorption of dyes increases. As the amount of adsorbent increases (more than the optimum value), the available locations of the adsorbent become too large and all molecules are in the required locations, so the adsorption efficiency remains constant

Conflict of Interest

We have no conflicts of interest to disclose.

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