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Adsorption of Methylene Blue Dye from Aqueous Solution by Using Gamma Alumina Nanoparticles

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ABSTRACT

This research focuses on gamma alumina nanoparticles using for the adsorption of the cationic methylene blue dye (MB) from aqueous solutions. Batch adsorption studies carried out to study various parameters included pH, gamma alumina nanoparticles dosage, temperature and contact time. The concentration of methylene blue dye was measured using a UV-vis Spectrophotometer at the wavelength of 620 nm. The optimum adsorption conditions were found to be pH=8, adsorbent dose=0.05 g, temperature=298 K and contact time=40 min. The experimental results of this work were compared with Langmuir, Friendlich and Tamkin's isotherm models. The obtained results from isotherm models showed that the surface adsorption of these dyes on the better adsorbent follows the Langmuir isotherm model. Analysis of thermodynamic data showed that the adsorption process of the studied dyes on the surface adsorbent is spontaneous (The negativ value of ΔG^0) and exothermic (The negativ value of ΔH^0).

Keywords: Methylene Blue; Adsorption; Thermodynamic Parameters; Gamma Alumina Nanoparticles.

INTRODUCTION

Transitional aluminas, particularly γ-alumina and θ-alumina, which are usually obtained by calcination of boehmite at varioustemperatures, are among the most significant oxides used in the industrial applications. The gamma alumina are widely used as adsorbents, catalysts and catalysts supports, due to their large specific surface areas, well – defined pore size distributions, stability within a wide temperature span, ability to stabilize and disperse the active phases as well as moderate acidities. There are several reports representing key roles of gamma alumina as catalysts support. Gamma-alumina is a very important industrially material due to their use in adsorption and catalysis. The nanoparticles of aged pseudoboehmites materials with high specific surface area are being recently

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studied for the controlled release of drugs [1-6].

Most acid–base indicators are dyestuffs from four classes of organic compounds: azo dyes, phthaleins, sulfonphthaleins, and triphenylmethane dyes. The classical azo indicators exhibit a color change from red to yellow with increasing basicity. The most commonly encountered examples are methylen orange and methylene blue. The behavior of the former is described by reaction. Methylene blue that similar to methylen orange except that the sulfonic acid group is replaced by a carboxylic acid group. Methylene blue is an azo dye, consisting of benzoic acid substituted at position 2 by a 4-[(dimethylamino) phenyl] diazenyl group. It has a role as a dye. It is a member of azobenzenes, a monocarboxylic acid and a tertiary amino compound. It is a conjugate acid of a methylene blue [4-10].

Dye elimination from wastes has been the object of many researches in the past few years because of the potential toxicity of dyes and visibility problems. These compounds are used in large quantity in many industries including textile, leather, cosmetics, paper, printing, plastic, pharmaceutical and food to color their products. As a result, considerable amounts of colored wastewater are generated. Dyes commonly have complex aromatic structures which make them stable and difficult to decompose. The dyes present in wastewater absorb sunlight, leading to a decrease in the efficiency of photosynthesis in aquatic plants due to reduced light penetration. Many of the industrial dyes are toxic, carcinogenic, mutagenic and teratogenic. Their elimination from wastewater is of great regard [7-11].

Over the past few decades, conventional waste water treatment methods for removing dyes include biological (aerobic and anaerobic), chemical and physiochemical methods such as coagulation and flocculation, solvent extraction, membrane filtration ozonation, ion exchange, electrochemical techniques, fungal decolonization and adsorption. The removal of dyes from wastewater or industrial effluent using adsorption method has recently gained more favour than the other methods of removal of dyes. Adsorption process has simplicity of design, more efficient, easy to operate, insensitive to toxic substances and cost effective. It therefore provides an alternative method to other expensive existing physical/ chemical/ biological methods for the removal of dyes from industrial effluents or waste water [10-18].

This research focuses on the use of gamma alumina nanoparticles as a biosorbent for the removal of methylene blue dye from aqueous solution, was studied in batch equilibrium conditions. The effects of different parameters including pH, initial metal ion concentration, contact time, gamma alumina nanoparticles dosage and temperature were investigated. Langmuir and Freundlich isotherm models were used to analyze the equilibrium data.

EXPERIMENTAL

Apparatus and Materials

An Lambda 25 UV/VIS Spectrophotometer From PerkinElmer was used to measure the concentration of methylene blue dye in the studied solutions, a 820 A model pH meter (Metrohm Co.) was used to measure pH of solutions and a thermostatic orbit incubator shaker neolab model (India) was used to measure contact time in the solutions. All chemical materials used in this study were of analytical grade.

Batch Adsorption Experiments

Batch adsorption experiments were carried out to characterize the methylene blue dye adsorption isotherm onto gamma alumina

nanoparticles and its thermodynamic properties. Methylene blue dye stock solution was prepared by dissolving the appropriate quantity of methylene blue dye in deionized water. Adsorption isotherms were obtained by using initial methylene blue dye concentration, X_o, and its equilibrium concentration, Xe. The effect of pH on the methylene blue dye adsorption onto gamma alumina nanoparticles was evaluated in a pH range of 2-8. The pH of solutions was adjusted by 0.1 M HCl and 0.1 M NaOH solutions. For every experiment, 50 ml of the solution methylene blue dye was mixed with gamma alumina nanoparticles in a 250 ml glass conical flask which was shaken in a thermostatic orbit shaker at 220 rpm. The mixture was filtered through a 0.45 μm membrane filter. The filtrate was measured by UV/VIS Spectrophotometer then, the adsorption percentage (%A) was determined as:

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

Q_e, amount adsorbed per unit weight of adsorbent at equilibrium (mg/g) was calculated using the following equation. Where X_o and X_e are the initial and final concentrations of dye methylene blue in solution (mg/L), respectively [10-20].

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

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 methylene blue concentration ranging from 5 mg/L in every experiment. Each solution was shaken continuously for 50 min [10-20].

RESULTS AND DISCUSSTON

The Effect of pH

The solution pH was very important parameter for the biosorption studies. Batch studies at different pH (2-12) were conducted by soaking the 0.01 g adsorbent (0.01 g) in 5 mg/L of dye methylene blue in each microcosm. Each container was agitated (156 rpm) for 30min at 25^{o}C. Table 1 illustrate the effect of the pH of the solution on the adsorption percentage of dye methylene blue, adsorbed onto gamma alumina nanoparticles. The best results were obtained at pH=8 for methylene blue dye. The pH value of aqueous solution may affect the surface charge of the adsorbent and the functional groups on the adsorbate. This trend can be attributed to the electrostatic attraction between the negatively charged dye and the positively charged surface of the adsorbent. Increase in solution pH increases the number of hydroxyl groups, thus, decreases the number of positively charged sites and reduces the attraction between methylene blue dye and the adsorbent surface. Thus, as pH is increased, the surface functional groups of the adsorbent deprotonates which results in a decrease in surface charge density and is unable to exert any electrostatic effect toward anionic species. Similar result was obtained in the adsorption of anionic dyes on ammonium-function-alized MCM-41 [16-22].

Table 1. The effect of initial pH of the solution on the adsorption percentage (%A) of dye methylene blue (Xo=5 mg/L, M gamma alumina nanoparticles =0.01 g, T=298 K, tc=40 min)

pH	%Ae
2	23.50
4	42.53
6	63.56
8	72.76
10	71.08
12	69.16

The Effect of Adsorbent Dosage

Microcosms with different adsorbent doses (0.01-0.07 g) were amended with 5 mg/L of methylene blue dye in aqueous solutions. The rate of adsorption was monitored at the following optimum conditions: pH of 8, for 40 min at 25^{o}C.The effect of gamma alumina nanoparticles dosage on the adsorption percentage of methylene blue dye is shown in table 2. The best results were obtained at M gamma alumina nanoparticles =0.05 g for blue methylene dye.

Table 2. The effect of gamma alumina nanoparticles dosage on the adsorption percentage (%A) of dye methylene blue (X_o=5 mg/L, pH=8, T=298 K, tc=40 min)

M gamma alumina nanoparticles **(g)**	**%Ae**
0.01	72.76
0.02	73.95
0.03	75.03
0.04	76.58
0.05	78.25
0.06	76.94
0.07	75.63

The Effect of Temperature

The same preparation was made, except for the varying temperature conditions. The microcosm which was maintained at pH=8 was incubated at different temperatures (25-65 ^{o}C) for a period of 40 min. Table 3 show that the adsorption percentage decreases with increasing temperature. Therefore, it may be concluded that the interaction between methylene blue dye and gamma alumina nanoparticles is exothermic in nature.

Table 3. The effect of temperature on the adsorption percentage (%A) of dye methylene blue (X_o=5 mg/L, M gamma alumina nanoparticles =0.05 g, pH=8, t_c=40 min)

T (K)	**%Ae**
298	78.25
308	76.70
318	74.32
328	71.56
338	69.65

The Effect of Contact Time

The effect of contact time, tc, on the adsorption percentage of methylene blue dye onto gamma alumina nanoparticles is shown in table 4. A rather fast uptake occured during the first 40 min of the adsorption. It became slower as the adsorbed amount of methylene blue dye reached its equilibrium value. It can be seen that the adsorption process is rapid due to the availability of very active sites on the surface adsorbent at initial stage. This may be due to the special one atom layering in structure of methylene blue dye. At first, adsorption capacity was a slow process, then increased rapidly, attaining equilibrium and saturation gave constant adsorption value. The optimum contact time was obtained at 40 min.

Table. 4. The effect of contact time, t_c, on the adsorption percentage (%A) of dye methylene blue (X_o=5 mg/L, M gamma alumina nanoparticles =0.05 g, pH=8, T=298 K)

Tc (min)	**%A_t**
5	53.75
10	63.80
20	69.41
30	75.98
40	78.25
50	78.25
60	78.25

Adsorption Isotherm

An adsorption isotherm is characterized by certain constant values, which express the surface properties of the adsorbent and so on the percentages adsorption of methylene blue dye onto gamma alumina nanoparticles as a function of initial concentration of methylene blue dye, shown in table 5. Adsorption isotherms represent the relationship of the amount of dyes adsorbed with the adsorbent dose. These provide information about the mechanism of adsorption and the adsorptivity of the composite towards the favorite dyes. In this study, Langmuir,

Freundlich and temkin isotherms were investigated [8-25].

The simplest and still the most useful isotherm, for both physical and chemical adsorption, is the Langmuir isotherm. This model assumes that adsorption is limited to a monolayer: only a singlelayer of molecules on the adsorbent surface are absorbed, adsorbent surface is homogeneous and adsorption energy is uniform for all sites and there is no transmigration of adsorbate in the plane of the surface. Once a pollutant occupies a site, no further adsorption can take place in that site; the intermolecular attractive for cesrapidly decrease as distance rises. There is no interaction between molecules adsorbed on neighboring sites, adsorption on surface is localized, which means that adsorbed atoms or molecules are adsorbed at definite and localized sites. The linear form of the Langmuir isotherm is described by the equation (3) [14-27]:

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

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 fig. 1 that were used to calculate the values of b and Q_m (table 6).

Table 5. Adsorption data for dye methylene blue adsorption onto gamma alumina nanoparticles (pH=8, t_c=40 min, T=298 K, M gamma alumina nanoparticles =0.05 g)

Parameter	**Value**				
X_0 (g/L)	1	2	3	4	5
%A	69.21	71.16	74.71	76.67	78.25
Xe (g/L)	0.31	0.58	0.76	0.93	1.09
Q_e (mg/g)	0.69	1.42	2.24	3.07	3.91
lnXe	-1.18	-0.55	-0.28	0.07	0. 08
lnQe	-0.37	0.35	0.81	1.12	1.36
1/Xe (L/g)	3.25	1.73	1.32	1.07	0.92
$1/Q_e$ (g/mg)	1.44	0.70	0.45	0.33	0.26

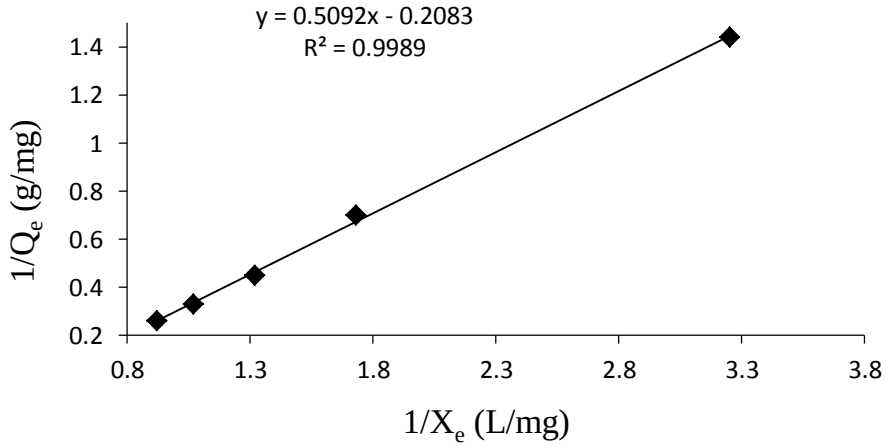

Fig. 1. Langmuir isotherm for dye methylene blue adsorption onto gamma alumina nanoparticles.

The Freundlich isotherm model is an empirical equation and another form of Langmuir that can be applied to multilayer adsorption. This model assumes that the surface of the adsorbent is heterogeneous and active sites and their energies distribute exponentially. The stronger binding sites are occupied first, until adsorption energy is exponentially decreased upon the completion of adsorption process. The Freundlich isotherm is expressed as equation (4) [14-27]:

$$\ln Q_e = \ln P + \frac{1}{n} \ln X_e \qquad (4)$$

Where P (L/mg) and n are the Empirical Freundlich constant or capacity factor and adsorption intensity. The values of P and n are determined from the intercept and slope of a plot of ln Qe versus ln Xe (table 5 and fig. 2) that were used to calculate the values of P and n (table 6)).

Another empirical equation, the Temkin equation, describes theadsorption of hydrogen onto platinum electrodes within the acidicsolutions. The model is given by equation (5) [16-28]:

$$Q_e = B_T \ln A_T + B_T (\ln X_e) \qquad (5)$$

The Temkin isotherm equation assumes that the heat of adsorption of all the molecules in the layer decreases linearly rather than logarithmically as equilibrium adsorption capacity increases because the B_T factor is related to adsorbent-adsorbate interactions. The adsorption is characterized by a uniform distribution of the binding energies, up to some maximum binding energy. The Temkin equation is better for predicting the gas phase equilibrium rather than liquid-phase equilibrium [16-28].

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 that is related to the heat of sorption. B_T and A_T are determined from the slope and intercept of a plot of Qe versus ln Ce (table 5 and fig. 3). We used these results to calculate the values of B_T and A_T (table 6).

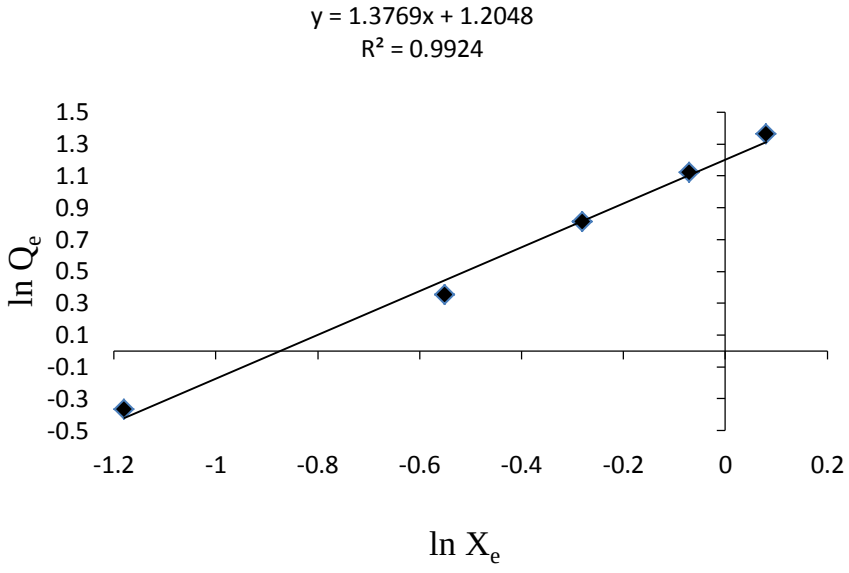

Fig. 2. Freundlich isotherm for dye methylene blue adsorption onto gamma alumina nanoparticles.

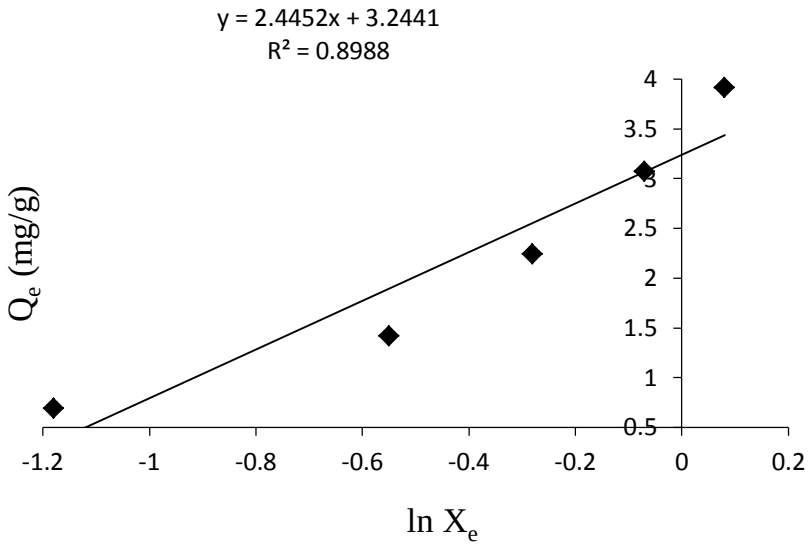

Fig. 3. Temkin isotherm for dye methylene blue adsorption onto gamma alumina nanoparticles.

Table 6. The resultant values for the studied isotherms in connection to dye methylene blue adsorption onto gamma alumina nanoparticles.

Isotherm	Parameter	Value
Freundlich	P (L/mg)	3.33
	n	0.73
	R^2	0.9924
Langmuire	b (L/g)	0.41
	Q_m (mg/g)	4.8
	R^2	0.9989
Temkin	B_T (J/mol)	2.44
	A_T (L/mg)	3.77
	R^2	0.8988

THERMODYNAMIC PARAMETERS

The amounts of adsorption of methylene blue dye by gamma alumina nanoparticles in temperature 298-338 K. Analysis of thermodynamics of equilibrium adsorption data can give more important information on adsorption process. Thermodynamic parameters such as change in free energy (ΔG°), enthalpy (ΔH°) and entropy (ΔS°) 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_o, where K_o is defined as follows [10-30]:

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

Where a_S and a_e are the activity of adsorbed dye methylene blue and the activity of dye methylene blue in solution at equilibrium, respectively. The adsorption standard free energy change (ΔG^0) is calculated according to:

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

The average standard enthalpy change (ΔH^o) and the average standard entropy change (ΔS^o) are obtained from the plot of equation (8) [10-30]:

$$\ln K_0 = \frac{\Delta S^0}{R} - \frac{\Delta H^0}{RT} \quad (9)$$

The ΔH° and ΔS° values obtained from the slope and intercept of plots are presented in table 7and fig. 4.

Table 7. The effect of temperature on K_o values (X_o=5 mg/L, pH=8, M gamma alumina nanoparticles =0.05 g, t_c=40 min)

T (K)	lnK_0
298	1.28
308	1.19
318	1.06
328	0.92
338	0.83

The obtained values of thermodynamic parameters (ΔG^0, ΔH^0, ΔS^0) are listed in table 8. The negative value of ΔH^0 suggests that the interaction of adsorbed dye methylene blue with gamma alumina nanoparticles is an exothermic process, which is supported by the decreasing of the amount of methylene blue dye adsorption with increasing temperature. The negative value of ΔS^o showed an decreased randomness during methylene blue dye adsorption. The negative values of ΔG^0 reveal the fact that the adsorption process is spontaneous.

CONCLUSION

Taking into account the results, we have considered it of great interest to assess the ability of gamma alumina nanoparticles for the adsorption of dye methylene blue from aqueous solutions in the batch technique and optimization of conditions for its adsorption. The results of this work showed that gamma alumina nanoparticles is an effective adsorbent for the removal of methylene blue dye from aqueous solutions. The isotherm parameters were calculated and Results showed that the Langmuir isotherm model was fitted well with adsorption data, thus, indicating the applicability of monolayer coverage of methylene blue dye on gamma alumina

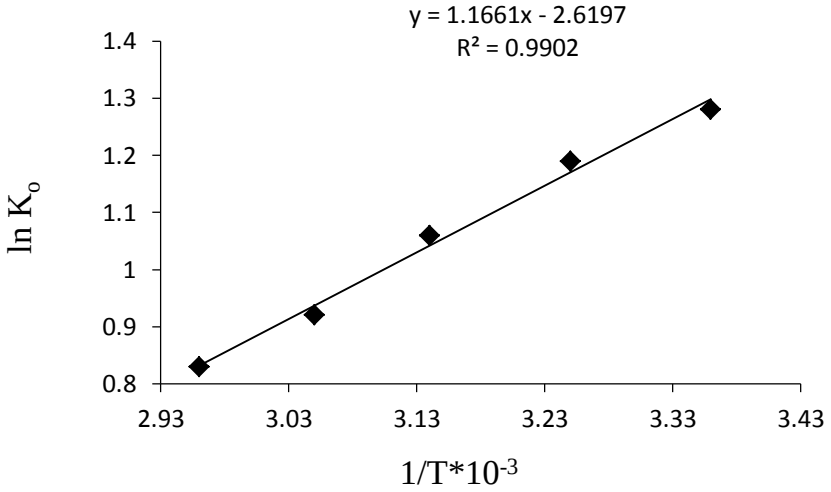

Fig. 4. The effect of temperature on equilibrium constant values.

Table 8. Thermodynamic parameters for adsorption dye methylene blue onto gamma alumina nanoparticles

T /K	ΔG^o(J/mol)	ΔH^o(J/mol)	ΔS^o(J/mol K)
298	-3172.02		
308	-3050.96		
318	-2809.54	-9.69	-21.78
328	-2516.30		
338	-2334.34		

nanoparticles surface. The temperature variations have been used to evaluate the values of ΔH^0, ΔS^0 and ΔG^0. Thermodynamic analysis revealed that the adsorption process is exothermic (The negative value of ΔH^0) and spontaneous in nature (The negativ value of ΔG^0).

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جذب رنگ متیلن بلو از محلول آبی به کمک نانوذرات گاما آلومینا

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چکیده

در این تحقیق از نانوذرات آلومینا گاما برای جذب رنگ کاتیونی متیلن بلو (MB) از محلول‌های آبی استفاده شد. مطالعات جذب دسته‌ای برای مطالعه پارامترهای مختلف شامل pH، دوز نانوذرات آلومینا گاما، دما و زمان تماس انجام شد. غلظت رنگ متیلن بلو با استفاده از دستگاه اسپکتروفتومتر UV-vis در طول موج ٦٢٠ نانومتر اندازه‌گیری شد. شرایط جذب بهینه pH=8، دوز جاذب، ۰/۰۵ گرم، دما، ۲۹۸کلوین و زمان تماس،٤٠ دقیقه بود. نتایج تجربی این کار با مدل‌های ایزوترم لانگمویر، فرندلیچ و تمکین مقایسه شد. نتایج بدست آمده از مدل‌های ایزوترم نشان داد که جذب سطحی این رنگ برروی جاذب مورد استفاده از مدل ایزوترم لانگمویر بهتر پیروی می‌کند. تجزیه و تحلیل داده‌های ترمودینامیکی نشان داد که فرایند جذب رنگ مورد مطالعه بر روی سطح جاذب خود به خود (مقدار منفی ΔG^0) و گرمازا (مقدار منفی ΔH^0) است.

کلید واژه‌ها: متیلن بلو؛ جذب ترمودینامیکی؛ نانوذرات آلومینا گاما.

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