

Removing Basic Fuchsin Dye by Adsorption over CuCo₂O₄ Nanocomposite as an Active Adsorbent Surface

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Abstract

This study involves the synthesis of CuCo₂O₄ spinel-type nanocomposite. This material was synthesized using the co-precipitation method and characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-rays Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), and Atomic Force Microscopy (AFM). The activity of the synthesized materials was investigated by following removing basic fuchsin (BF) dye from its aqueous solution by adsorption. Different adsorption parameters were conducted involving the dose of adsorbent surface, time of adsorption, pH of the medium, and temperature. The optimum removal efficiency was around 97 % at contact time 5 minutes, 0.005g of dose the surface adsorbent, pH = 8 and 50 mg. L⁻¹ concentration of dye. Thermodynamic parameters for the adsorption of this dye over the prepared materials were also conducted such as free energy (ΔG) negative. While enthalpy (ΔH) positive which indicates the adsorption was spontaneous and endothermic, also entropy (ΔS) was positive shows the affinity of the adsorbent towards the adsorbate.

Keywords: Adsorption, Basic Fuchsin dye, CuCo₂O₄, Nanocomposite of spinel type, Pollution of water, water treatment.

Introduction

Pollution of water is the strongest dangerous phenomenon resulting from the rapid increase in agriculture and industry activities¹. So, pollution treatment has become a prominent goal. This involves the removal of many pollutions such as heavy metals and organic compounds in the water and treatment to use again in human activities is a matter of prime importance to scientists and

specialists in the field of water remediation so a huge volume of studies was devoted to the topic². While the enhancement of sanitation water supply will be the best water resources management and boost the growth of countries' economies and contribute significantly to poverty alleviation and disease of their peoples³.

The textile industry is considered one of the most important economic resource, as it annually produces more than 10^5 types of dyes, with an estimated amount of 7×10^5 tons, which produces pollutants that are released into the freshwater environment and sewage water⁴. The dyes produced by the textile and paper industries that are released into wastewater and the aquatic environment are pollutants because they are toxic and non-biodegradable compounds⁵. The dyes contain aromatic structures with homogeneous or heterogeneous compounds resistant to biodegradation and disintegration that reason preventing light⁶, which makes dyes pollutants in the water environment a deficiency in biochemical and chemical oxygen demand. This requires finding solutions to remove these dyes from the aquatic environmental⁷.

There are three main types of treatment for water pollution and removal of pollutants, these are chemical methods, biological methods, and physical methods (adsorption processes, filters, and reverse osmosis)⁸. Adsorption techniques have been widely used in pollution treatment due to their high efficiency, ease of application, and lower cost compared to other methods, with up to 99% in removing pollutants from water in a short time. The adsorption process can be defined as the adhesion of the particles of the polluting component to the surface solid substance called the adsorbent surface⁹.

There are many factors affecting the efficiency of the adsorption process, these are the molecular weight and the concentration of pollutants, the size, nature, surface area, and quantity of the adsorbing surface, the pH of the medium, the presence of additional substances and pollutants in addition to the temperature of the medium¹⁰. Due to the characteristics of the adsorbent surface affecting the process, which is the number of active sites and the large surface area¹¹. In this study, a nanocomposite

of the type spinel, CuCo_2O_4 , was synthesized because it is stable in the removal processes¹².

Fuchsin basic (BF) dye, known as Magenta II, has the chemical formula $\text{C}_{20}\text{H}_{20}\text{ClN}_3$ and is a mixture of the three dyes Rosaniline, Pararosaniline, and Magenta II¹³. As well Basic Fuchsin dye belongs to the triarylmethane class dyes¹⁴. This dye has many applications and uses in textile dyeing, leather dyeing, collagen, muscles, mitochondria, and tuberculosis bacilli¹⁵. It also possesses Gram-positive anesthetic, bactericidal and fungicidal effects¹⁶. The blood, liver, spleen, thyroid, and other organs may also suffer from swallowing or inhaling damage. Besides its carcinogenic and mutagenic effects, repeated exposure to the dye may have consequences for the nervous system¹⁷. This prompted researcher to remove the dye from aqueous solutions before putting it into the water stream to reduce pollution and its risks. where used. The Brião, G.V *et al*¹⁶ synthesized an adsorbent surface of medium porous zeolite prepared from Biopolymer/ZSM-5 zeolite with high efficiency to remove 85% of BF when using a dose of 2.0 g.L^{-1} at an equilibration time of 20 minutes. The adsorption process is identical to Langmuir, Freundlich¹⁶. While Lu *et al*¹⁸ synthesized in a surface adsorbent from Loess clays with a functionalized copolymer to remove BF dye with the maximum removal percentage at $\text{pH} = 6$, a surface adsorbent dose of 2.0 g.L^{-1} , and an equilibration time of 120 minutes, as well as the adsorption chemical and exothermic, and more Freundlich than Langmuir identical¹⁸.

This research aims to remove reactive BF from aqueous by using involve the synthesis of CuCo_2O_4 Nanocomposite. Parameters such as contact time, pH, adsorbent concentration, adsorbate content were optimized. It also aims at exploring the best and isotherms models and studying thermodynamics values¹⁹.

Experimental Section

Chemicals and equipment

Materials used in its study were of high purity, these were, $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$, $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, Na_2CO_3 , NaCl , KCl , MgCl_2 , $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$, NaOH , and HCl , these were purchased from SIGMA, BHD, and

HIMEDIA companies. Toxic BF was obtained from the Textile factory in Hilla, Iraq. For all experiments, deionized and double distilled water was used as a solvent. Laboratory equipment and techniques used in this study and devices were manufactured from reputable international origins, such as: Electronic

Balance TP-214 (Sartorius, Germany), Muffle Furnace Size-Two, Oven Bs Size Two (Gallenkamp), Heater with magnetic stir MR Hei-standard (Heldolph, Germany), Ultrasonic, UV-Visible Spectrophotometer Double Beam -6100PC (EMC LAB, Germany), (FT-IR) IRAffinity-1S (Shimadzu, Japan), (X-Ray) XRD-6000, Field-emission Scanning Electron Microscope (FE-SEM) Tescan Mira3 (Mira3 French), and Atomic Force Microscope (AFM)TT-2 AFM (AFM Workshop, USA).

Synthesis of the Adsorbent Surface

The co-precipitation method was used to synthesize cobalt-copper oxides overlapping Nano-type spinel from the equivalent weights of aqueous copper nitrate and cobalt nitrate at 40% and 60% respectively and dissolving them in 400 mL of distilling water. Then, this solution was mixed and heated at 70-75 °C using a heater and magnetic stirrer, during that, the sodium carbonate 0.1 moles.

L⁻¹ as a precipitant agent was added dropwise until completion of the precipitation of metal carbonates at pH = 9. Afterwards, heating with stirring were continued for 2 hours. Then the solution was filtered and the precipitate was washed with water several times to remove the remaining unreacted sodium carbonate until pH = 7. Then the precipitate was dried at 120 °C and crushed, then there was burning at 600 ° for four hours, the obtained material was designed as CuCo₂O₄²⁰.

Preparation of BF Solution

A BF dye solution was prepared at a concentration of 100 mg per liter (ppm) by dissolving 0.1 g. in one liter of water distilling as a stock solution, then by using the visible-ultraviolet spectra to determine the λ_{max} at about 546 nm and series of concentrations 10 – 80 mg per liter was prepared from it to create the standard calibration curve of the dye as shown below Fig. 1.

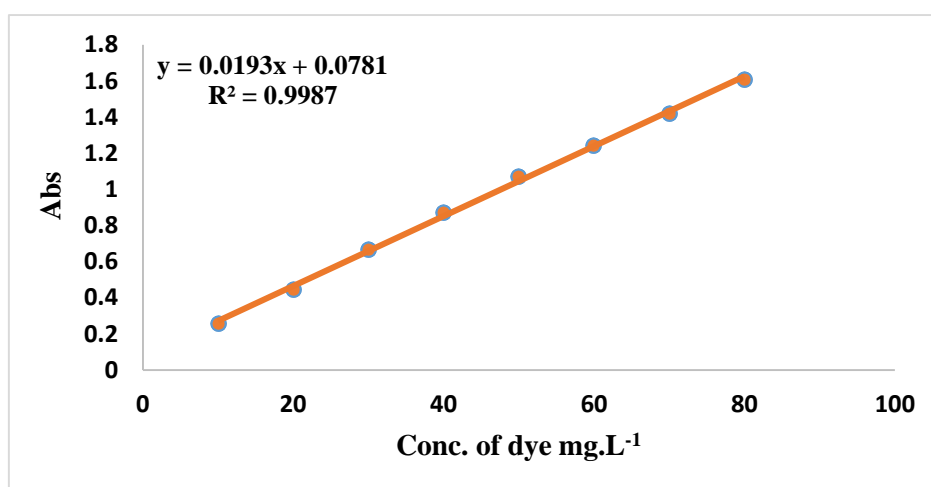


Figure1. Standard calibration curve of concentration mg. L⁻¹ of BF at $\lambda_{max} = 546$ of its Dye.

Studying the Optimization Conditions for BF Dye Removal

A series of volumetric bottles of 25 mL capacity was taken containing a dye solution with a concentration of 50 ppm. A weight of 5 mg. of adsorbent surface was added to each volumetric vial and placed in a water shaker. The percentage of dye removal by Eq. 1 was found after every 5 minutes for 120 minutes after separating of the adsorbent surface from solution and find the absorbance of the solution. As for the weight of the adsorbent surface, a series of

volumetric bottles of 25 mL capacity of 50 ppm dye solution was also added in different weights 0.001 - 0.02 g. of adsorbent surface and finding the percentage of dye removal at the optimization time. In the same way, the same experiments were carried out to find acidity²¹.

$$Re\% = \frac{C_i - C_e}{C_i} \times 100\% \quad 1$$

Whereas, Re % is the removal efficiency of dye over the used material. C_i and C_e are the initial concentration before adsorption and at equilibrium

concentration (after adsorption) of the dye respectively.

Adsorption Isotherms

Different initial concentrations 10-80 mg. L⁻¹ of BF dye at temperatures 298-338K were conducted to find adsorption capacity (Q_e) by Eq. 2 at the optimum conditions of adsorption to study the isothermal adsorption temperature for adsorption of BF dye according to Gailles's classification using the prepared adsorbent surface by plotting between the adsorption capacity Q_e and C_e ²².

$$Q_e = \frac{V(C_i - C_e)}{m} \quad 2$$

Whereas, Q_e , is the capacity of the adsorbate at units in mg. g⁻¹.

C_i and C_e are the initial concentration before adsorption and at equilibrium concentration (after adsorption) of the dye respectively, m is the weight of CuCo₂O₄ in units of g and

V , is the total volume of the adsorbate solution in units L.

Results and Discussion

Characterization of the Synthesized material

Surface functional groups of the prepared material showed some peaks at the fingerprint region, 667.39 - 572.88 cm⁻¹, appeared as a result of the stretching

vibrations of tetrahedral Co³⁺ -O²⁻ bonds and octahedral Cu²⁺ -O²⁻ complexes. Whereas at 2500 cm⁻¹ a W-shaped crest appeared as a result of the oscillator links as shown in Fig. 2²³.

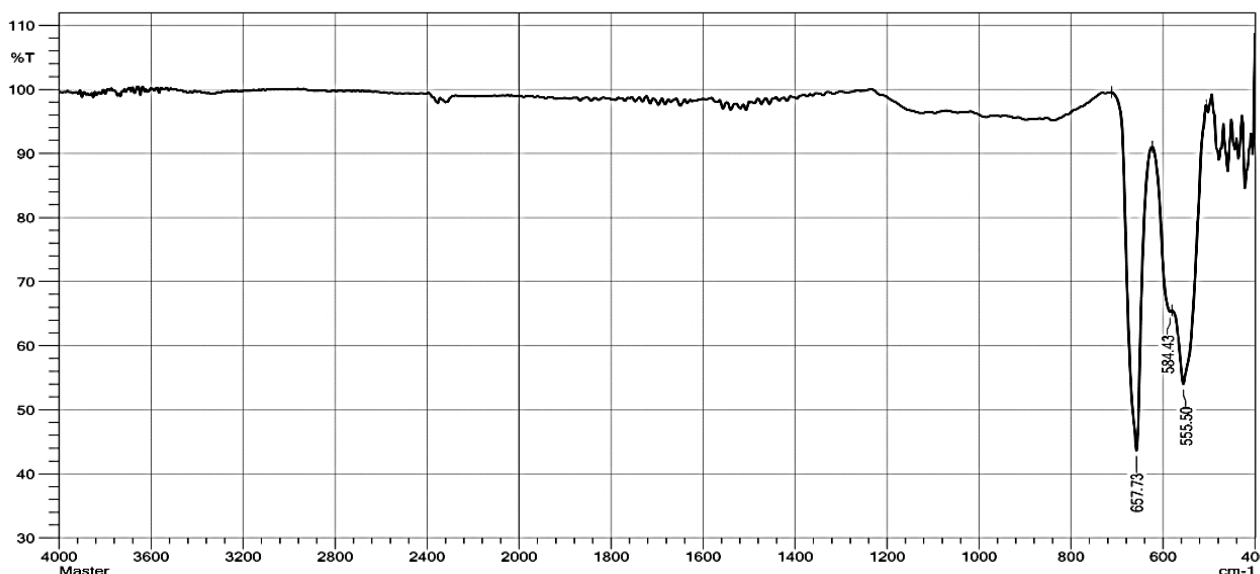


Figure 2. FTIR spectra for the synthesized CuCo₂O₄.

XRD patterns for the prepared material is shown in Fig. 3. These patterns are related to 01-087-2177 and 00-001-115 JCPDS, including diffraction angles 2θ (19.063, 31.3382, 36.9182, 38.7951, 44.8771, 48.7963, 59.4515, 68.1347), FWHM 0.2952, 0.1968, 0.1968, 0.1968, 0.1968, 0.1968,

0.2952, 0.72 and Miller processing (hkl) 111, 220, 311, 222, 400, 331, 511, 531 respectively as Co_{tet}(CuCo)_{oct}O₄ and the sparks equation which depends on the results of X-rays diffraction. The average particle size was calculated using Scherer equation and it was around 1 nm²⁴.

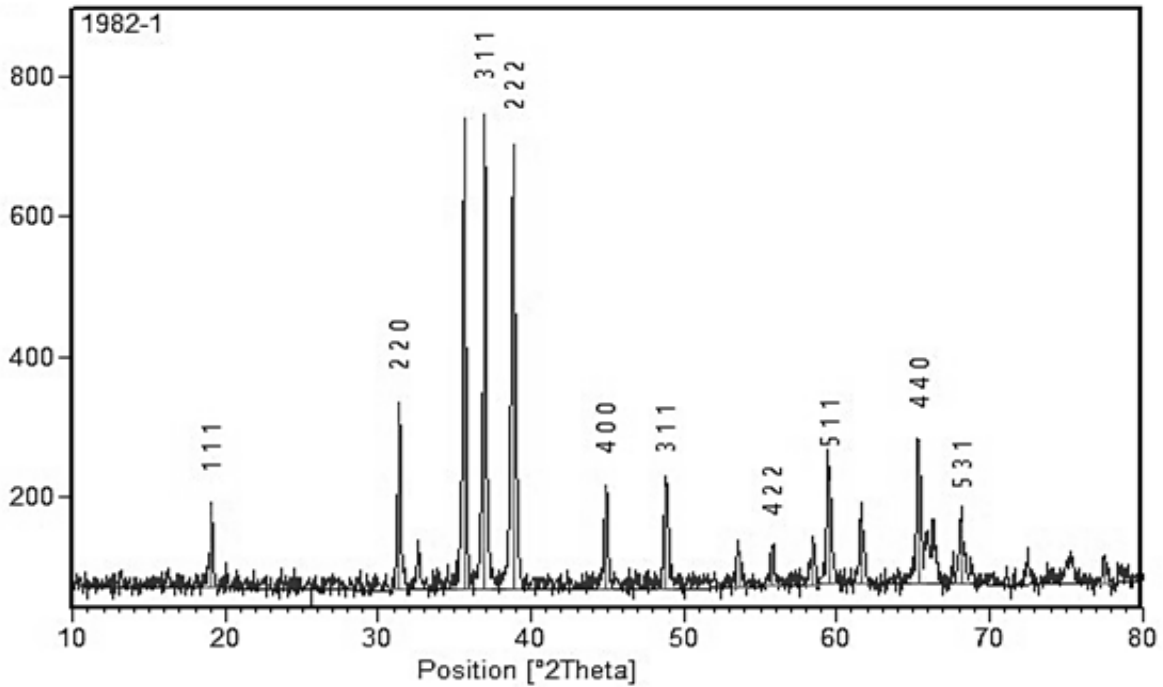


Figure3. X-rays diffraction patterns for the prepared CuCo_2O_4 adsorbent surface.

Fig. 4 shows the size and morphology of the prepared material, in almost it shows spherical grains, their sizes ranged from 31 to 364 nm, with large interstitial spaces using the FESEM technique. Fig. 5 shows the surface of the adsorbents 25nm with

a high homogeneity when using the AFM technique, which increases the residential locations of the adsorbent surface due to having high surface area and granular distribution as well as the homogeneity of the surface ²⁵.

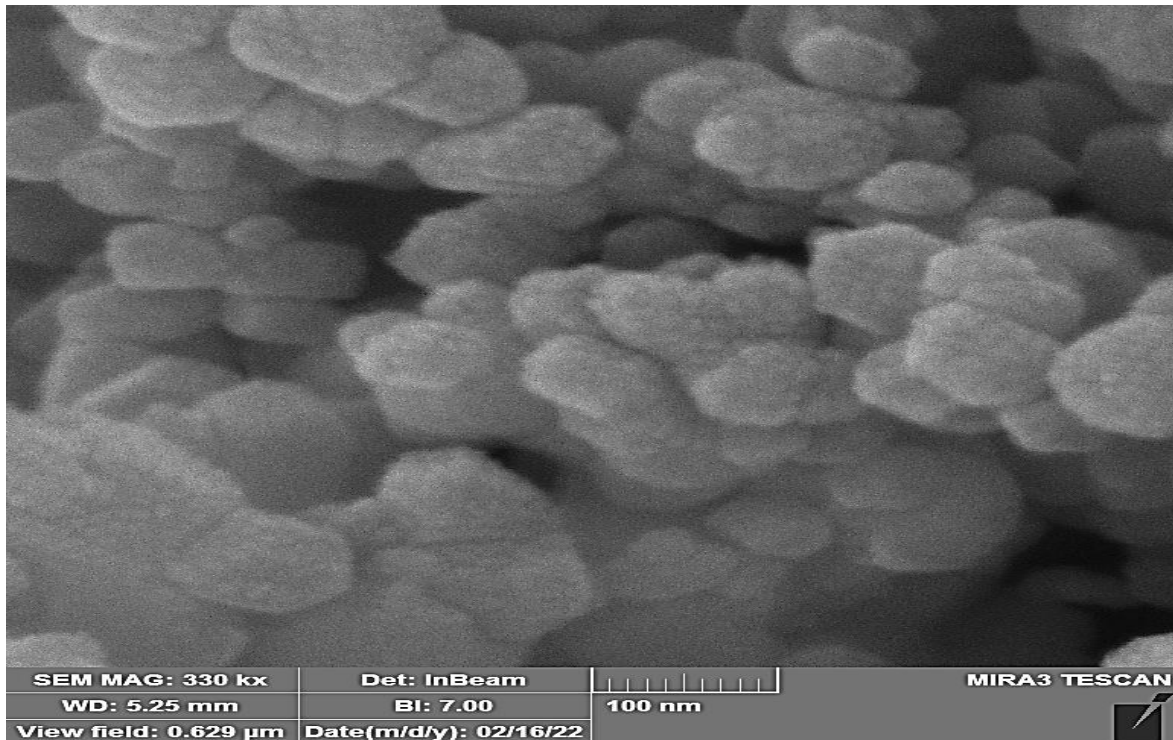


Figure 4. FESEM of the synthesized CuCo_2O_4 .

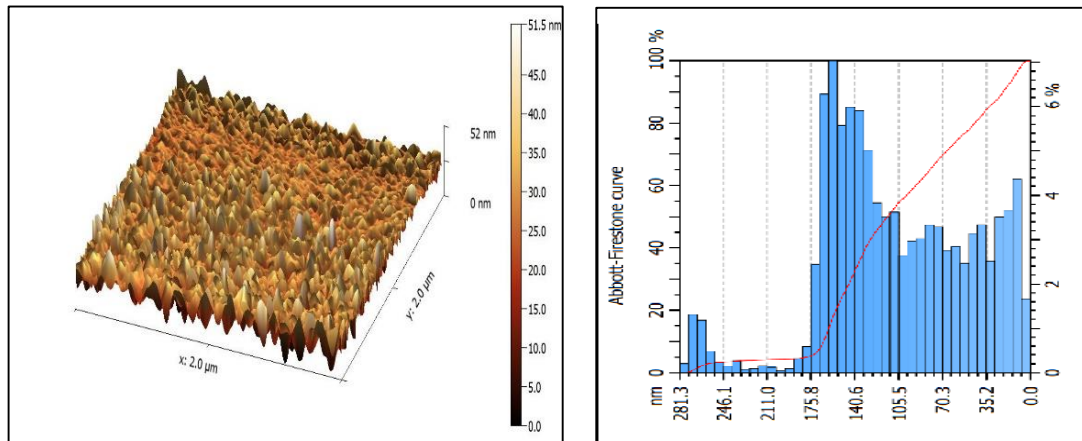


Figure 5. AFM image and histogram for the synthesized CuCo_2O_4 .

Studying the Optimum Conditions for Adsorption BF by CuCo_2O_4 Adsorbent Surface

Contact (Equilibrium) Time

Contact time (Equilibrium time) is the time required for the adhesion of the adsorbent particles with all the active sites on the adsorbent surface to reach the

equilibrium state, and it is important in the adsorption process and the efficiency of the adsorbent surface²⁶. Fig. 6 shows a sudden increase in the removal efficiency of dye at first five minutes after that shows a very slight rise in Re% from 10-120 minutes indicating that all the dye adhered to the active sites at the surface of the adsorbent at 5 minutes and it was the optimum contact time²⁷.

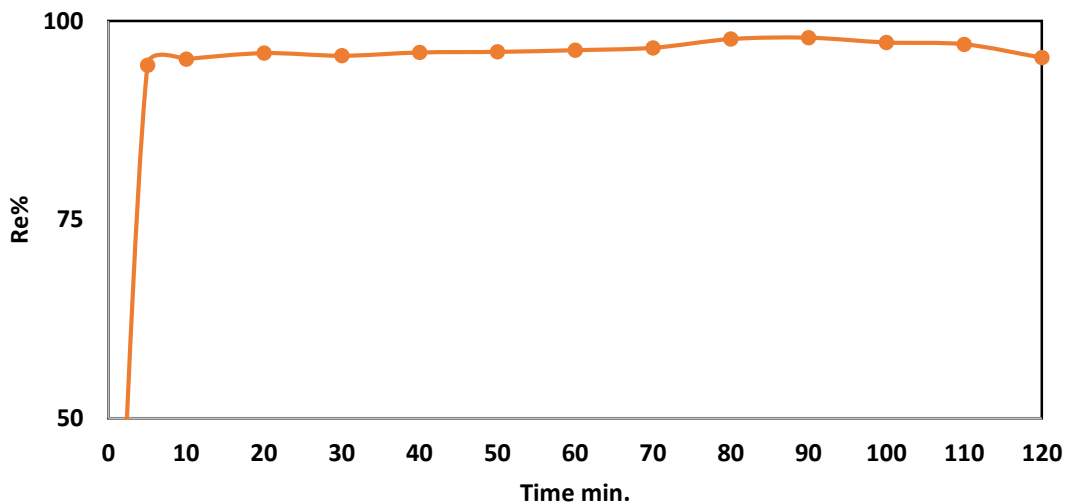


Figure 6. Effect of contact time on BF dye removal by adsorption over CuCo_2O_4 at 298K.

Effect of Weight of the Adsorbent on the Efficiency of Dye Removal

Fig. 7, shows the sudden increase at a low dose of 0.001- 0.005 g of adsorbent surface, and then the increase in the removal efficiency continued very

slight rise compared with the increase in the surface dose, the found optimal adsorbent surface weight of 5 mg to remove 88% of the dye molecules to reason most of the active sites bond to all dye when the dose of 5 mg of adsorbent surface²⁸.

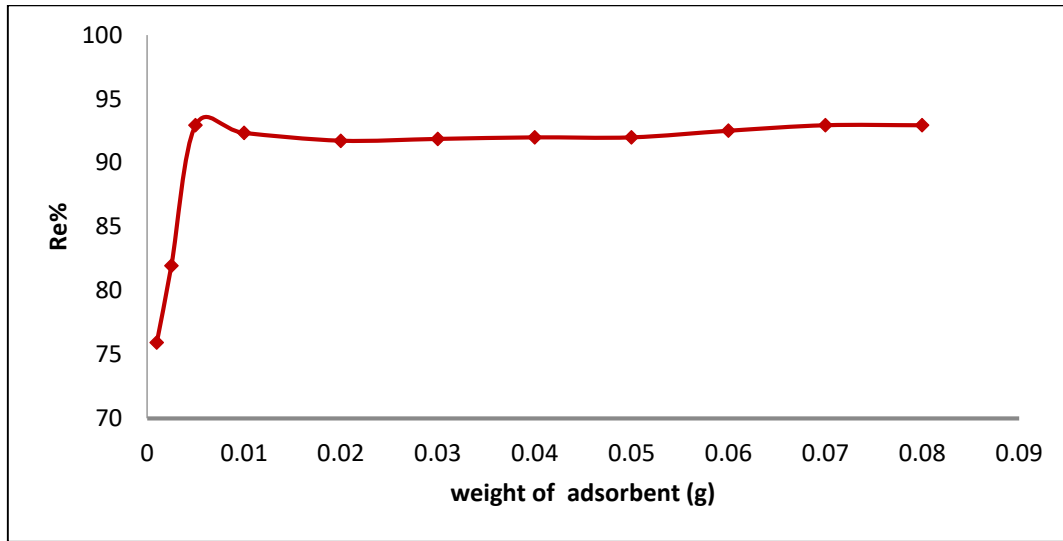


Figure7. Effect of the weight of CuCo_2O_4 on the efficiency of BF dye removal at 298 K.

Adsorption Isotherms

The relationship between adsorption capacity (Q_e) and C_e concentrations at 10 to 80 mg L^{-1} at different initial concentrations is known as an adsorption

isotherm as shown in Fig. 8²⁹. According to the classification of Giles' S4³⁰, there was a high affinity of dye molecules towards adsorbent surface at 298-338K³¹.

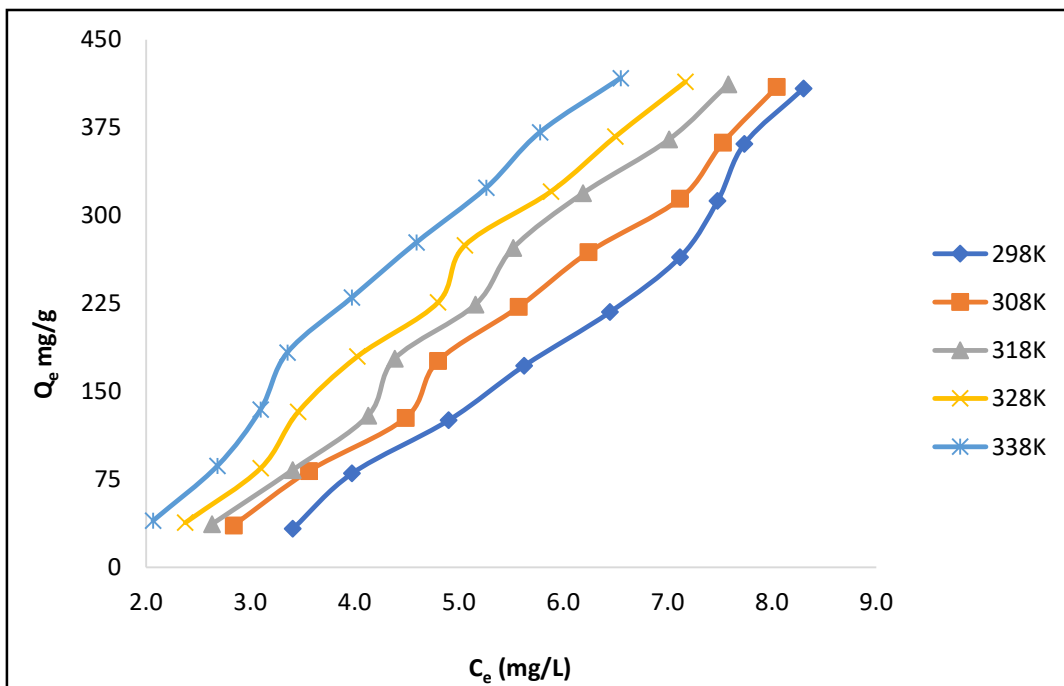


Figure 8. The relationship between Q_e and C_e of BF dye on the adsorbent surface at 298–338 K.

Adsorption isotherms Langmuir originally proposed that the adsorption process occurs on one layer, after that Freundlich and Temkin modified Langmuir's Eqs. 3,4 and 5 respectively for the adsorption process occurring on more than one layer³².

$$Q_e = \frac{a b C_e}{1 + b C_e} \quad 3 \quad \text{Langmuir}$$

$$Q_e = K_f C_e^{1/n} \quad 4 \quad \text{Freundlich}$$

$$Q_e = \frac{RT}{b} \ln A_T C_e \quad 5 \quad \text{Temkin}$$

Figs. 9, 10, and 11 show Freundlich, Langmuir, and Temkin equations respectively, while values constants of each model's adsorption equation are

listed in Table 1. The obtained results showed that the adsorption process was more fitted with Freundlich and Temkin equations with less fitting with Langmuir models because the adsorption multiple layers³³.

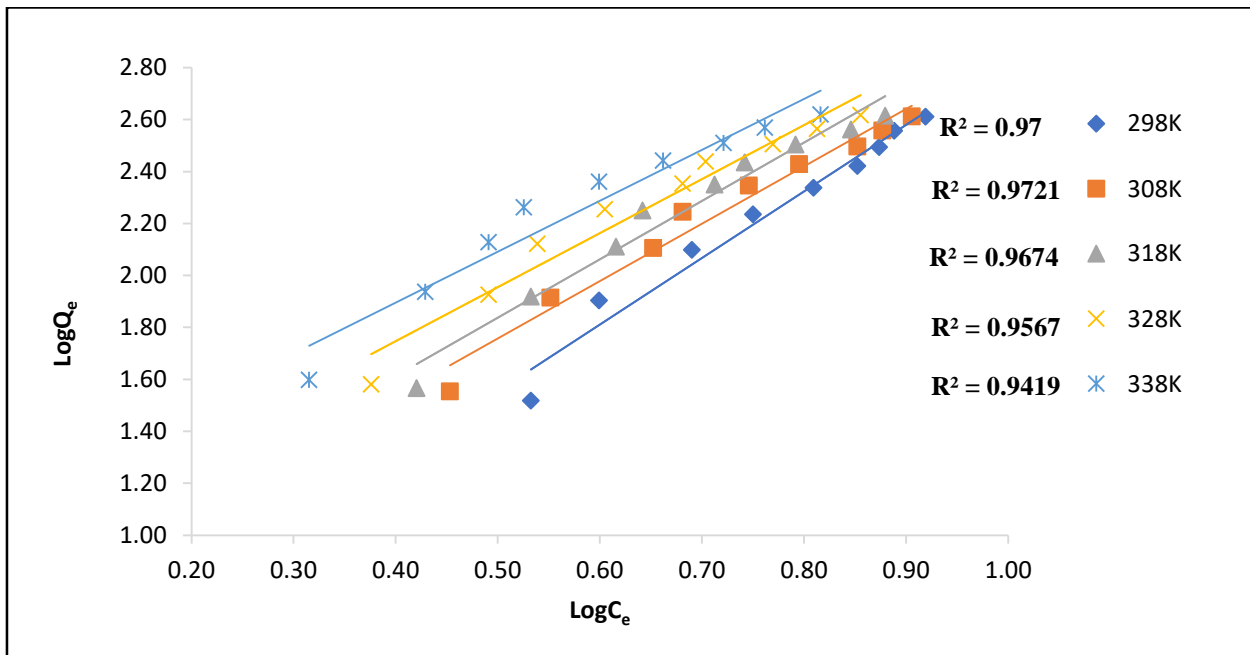


Figure 9. Freundlich isotherm relationship between $\log Q_e$ and $\text{Log} C_e$ for BF dye adsorption over CuCo_2O_4 at temperature ranges from 298 to 338 K.

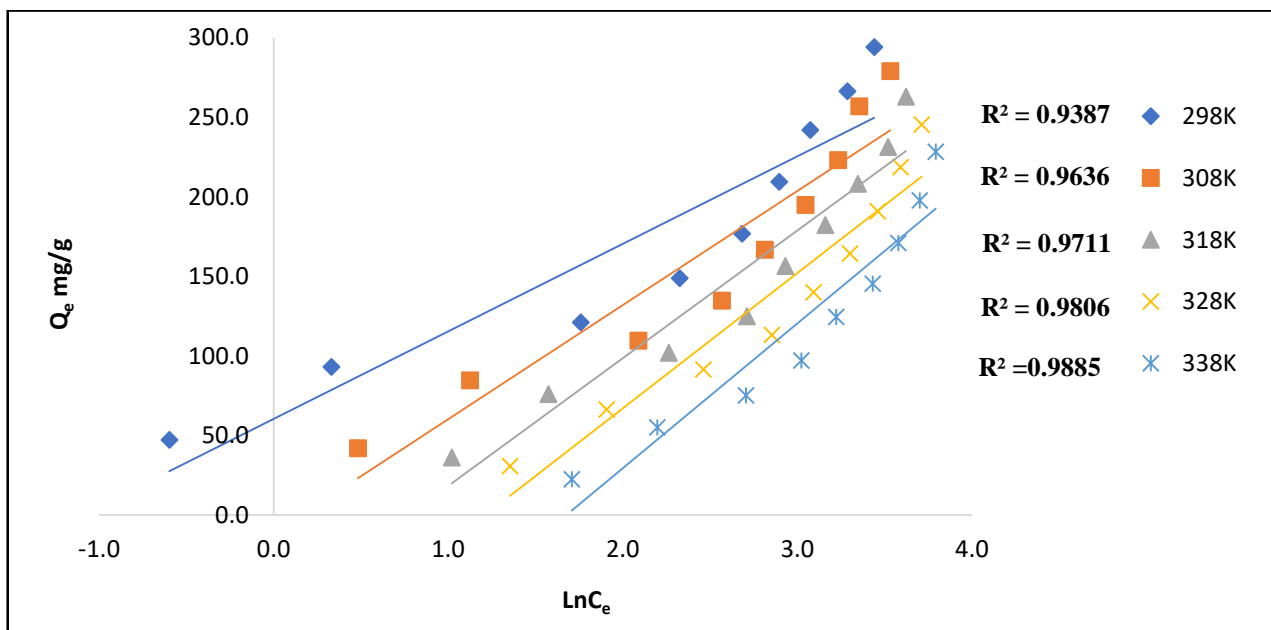


Figure 10. The Temkin isotherm is relationship between Q_e and $\text{Ln} C_e$ for BF dye adsorption over CuCo_2O_4 at temperature ranges from 298-338K.

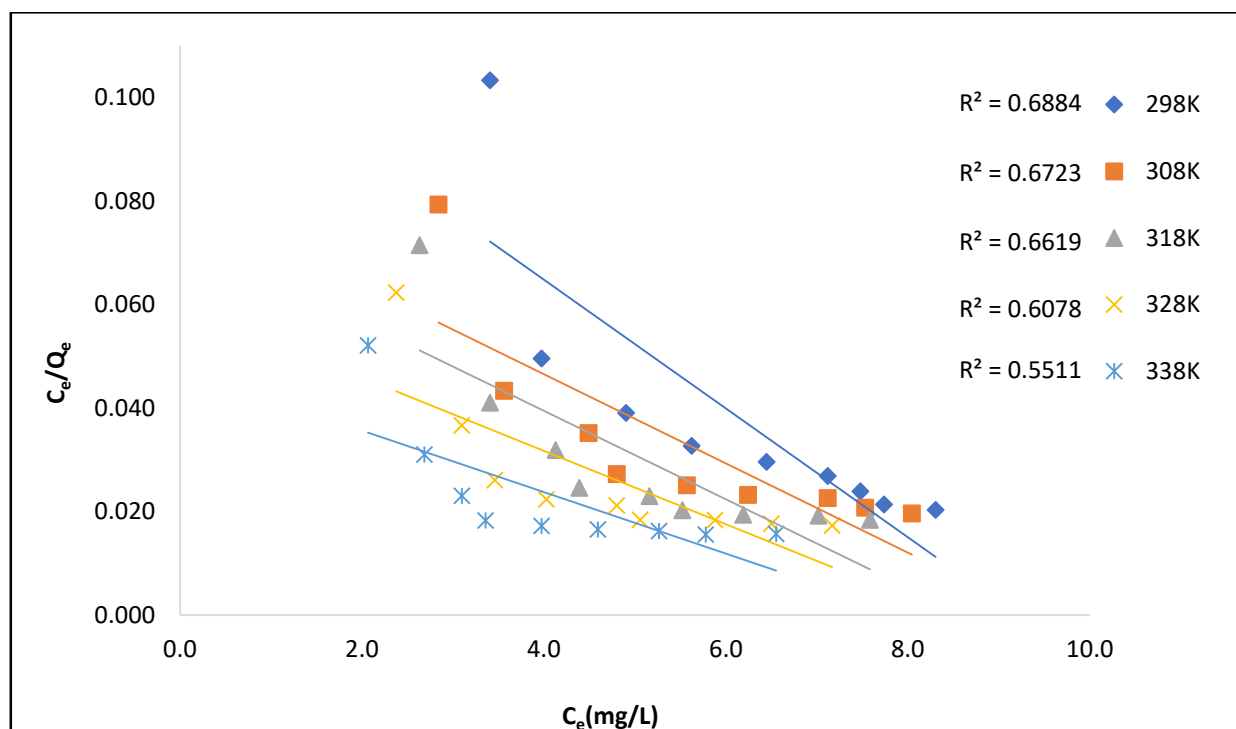


Figure 11. The Langmuir isotherm is relationship between C_e/Q_e and C_e for adsorption of BF dye over $CuCo_2O_4$ at temperature ranges from 298 - 338 K.

Table1. Isothermal parameters and correlation coefficient for BF dye adsorption over $CuCo_2O_4$.

Temperature K	Langmuir isotherm				Freundlich isotherm			Temkin isotherm		
	a	b	RL	R^2	K_f	n	R^2	b_T	K_T	R^2
298	-80.0	-0.1091	-0.2	0.6884	1.87	0.39	0.97	-347.16	1.87	0.9387
308	-116.3	-0.1060	-0.2	0.6723	4.48	0.45	0.9721	-328.14	4.48	0.9636
318	-117.6	-0.1153	-0.2	0.6619	5.16	0.44	0.9674	-202.12	5.16	0.9711
328	-140.8	-0.1181	-0.2	0.6078	8.21	0.48	0.9567	-219.69	8.21	0.9806
338	-169.5	-0.1239	-0.2	0.5511	13.15	0.51	0.9419	-175.06	13.15	0.9885

The Effect of the pH Reaction Mixture on the Efficiency of Dye Removal over CuCo_2O_4

The effect of pH on the removal efficiency of BF dye over the synthesized CuCo_2O_4 was investigated by applying adsorption under different pH values of the solution keeping other adsorption conditions

constant. In this manner, a series of pH values from 2 to 12 were carried out³⁴. The obtained results showed a significant decrease with decreasing pH as well as in the basic medium due to the competition of H^+ and OH^- dye for their active site on the surface, therefore was the best pH = 8 as shown in Fig. 12³⁵.

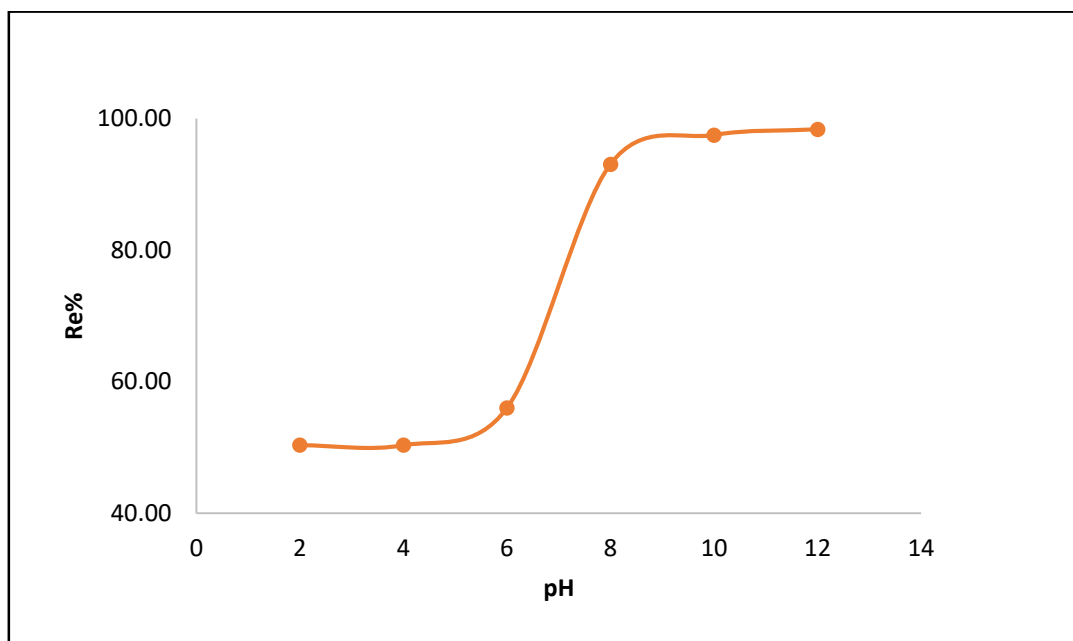


Figure 12. Effect of pH on the efficiency of BF dye removal over CuCo_2O_4 .

Thermodynamics of BF Dye Adsorption over CuCo_2O_4

The effect of temperature was studied and thermodynamic functions (free energy, ΔG , enthalpy ΔH , and entropy ΔS) were investigated. This conception is important for the adsorption process and determining the type of reaction spontaneous, exothermic, or endothermic¹⁶. The free energy (ΔG), ΔS , K_{eq} and ΔH were calculated using Eqs. 6, 7, 9 and 10 respectively, while ΔH was calculated by sketching the relationship between the equilibrium constant $\text{Ln}K_{eq}$ and $1/T$ which was shown in Fig. 13 and Table 2. The adsorption process is spontaneous and does not need energy reason ΔG was negative the surface energy of the adsorbent decreased due to residual forces on the surface of the adsorbent decreases, and the heat of the reaction is positive,

indicating that the adsorption process is an endothermic process, while the change in entropy showed a change from a positive value but decreasing state with increasing temperature, shows the affinity of the adsorbent towards the adsorbate and this may be due to the disturbance of the water molecules around the dye^{36, 37}.

$$\Delta G = -R \text{Ln}K_{eq} T \quad 6$$

$$\Delta G = \Delta H - T\Delta S \quad 7$$

$$\text{Ln}K_{eq} = \frac{-R\Delta H}{T} + \frac{\Delta S}{R} \quad 8$$

$$K_{eq} = \frac{Q_e m}{C_e V} \quad 9$$

$$\text{Slope} = -R\Delta H \quad 10$$

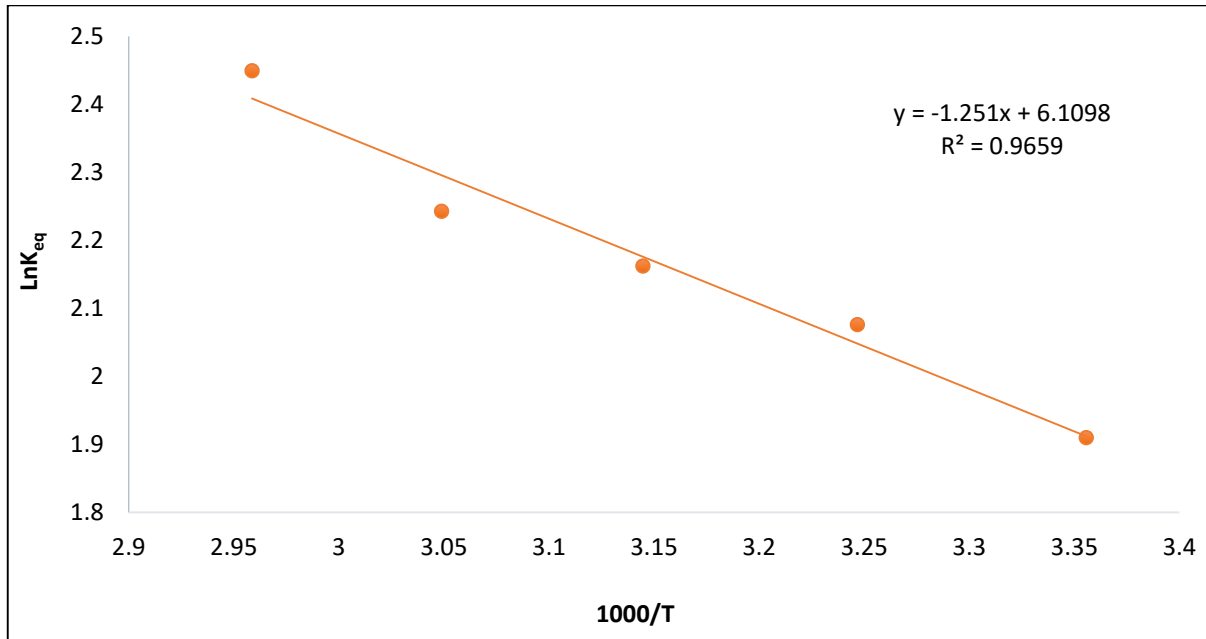


Figure 13. The relationship between the LnK_{eq} and 1000/T

Table 2 and Fig. 13 indicate that the adsorption process was spontaneous and does not need energy as ΔG showed a negative value. The heat of the reaction is positive, indicating that the adsorption process is an endothermic process, while the change

in entropy showed a change from a positive value but decreasing state with increasing temperature, this may be due to the disturbance of the water molecules around the dye.

Table 2. The values of C_e, 1/T, K_{eq} and thermodynamic functions for adsorption process to BF dye on CuCo₂O₄ at 298 – 338 K

T	C _e	K _{eq}	1000/T	LnK _{eq}	ΔG KJ.mol ⁻¹ . K ⁻¹	ΔH KJ.mol ⁻¹ .K ⁻¹	ΔS J.mol ⁻¹ .K ⁻¹
298	6.448	6.754	1.910	1.9101	-4.7324	10.40	50.7829
308	5.572	7.973	2.076	2.0760	-5.3161	10.40	51.0292
318	5.160	8.690	2.162	2.1621	-5.7164	10.40	50.6832
328	4.799	9.419	2.243	2.2427	-6.1159	10.40	50.3558
338	3.974	11.581	2.449	2.4493	-6.8830	10.40	51.1356

Conclusion

From the obtained results in the current study, it was found that, the best removal efficiency of BF dye over CuCo₂O₄ was achieved under the optimal conditions of the initial concentration of dye of 90 mg. L⁻¹, the weight of the adsorbed surface, 0.005 g, pH 8, and contact time was 5 minutes. As well

Langmuir, Freundlich, and Temkin models according to the correlation coefficient (R²) the Freundlich, and Temkin models are better for describing the removal of BF while the Langmuir model was not best isothermal model in describing. While thermodynamic values showed that the

adsorption process is a spontaneous and endothermic process. From the isothermal models and

thermodynamic values, the removal mechanism was physical absorption.

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Authors' Declaration

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are ours. Furthermore, any Figures and images, that are not ours, have been

- included with the necessary permission for re-publication, which is attached to the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee at University of Kerbala.

Authors' Contribution Statement

A. A. K. wrote a part of the manuscript and collected the samples. M. A. wrote conception, design, acquisition of data, analysis, interpretation, drafting the MS, revision and proofreading. S. H.K. conception, design, acquisition of data, analysis,

interpretation, drafting the MS, revision and proofreading. E.T.K. did the revision and proofreading. All the authors read the manuscript carefully and approved the final version.

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إزالة صبغة الفوكسين القاعدية عن طريق الامتزاز على متراكب نانوي CuCo₂O₄ كسطح ماص نشط

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الخلاصة

تتضمن هذه الدراسة تصنيع مركب نانوي من نوع CuCo₂O₄. تم تصنيع هذه المادة باستخدام طريقة الترسيب المشترك. تم تشخيص المواد المحضرة بأستخدام الطيفي للأشعة تحت الحمراء (FTIR)، حيود الأشعة السينية (XRD)، المجهر الإلكتروني لمسح الانبعاث الميداني (FESEM)، والفحص المجهرى للقوة الذرية (AFM). تم فحص نشاط المواد المحضرة باتتبع إزالة صبغة الفوشسين الأساسية (BF) من محلولها المائي عن طريق الامتزاز. تم دراسة الظروف الفضل للامتزاز المختلفة مثل وزن السطح الممتز ووقت الامتزاز ودرجة الحموضة الوسط فضلاً عن درجة الحرارة. حيث كانت كفاءة الإزالة المثلى حوالي 97% في وقت التلامس 5 دقائق، 0.005 غم من السطح الممتز مع أفضل درجة حموضة = 8 و تركيز صبغة 50 ملغم.لتر⁻¹. تم حساب المعلمات الديناميكية الحرارية لامتزاز هذه الصبغة على المواد المحضرة أيضاً مثل الطاقة الحرة (ΔG) سالبة بينما المحتوى الحراري (ΔH) موجباً مما يشير إلى أن الامتزاز كان تلقائياً وممتصاً للحرارة، كما كان الانتروپيا (ΔS) موجباً.

الكلمات المفتاحية: تلوث المياه، معالجة المياه، الامتزاز، CuCo₂O₄، متراكب نانوي من نوع الإسبنيل، صبغة الفوكسين القاعدية.