

Removal of Dye Bismarck Brown G by Photocatalytic Reaction over Prepared Co-Oxide Cr₂O₃-NiO: A Kinetic Study

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The co-oxides Cr_2O_3 -NiO was prepared by using a co-precipitation method. The co-oxides were prepared by calcination temperatures at 500 °C. The prepared materials were investigated using powder X-rays diffraction and Fourier transform infrared spectroscopy. The photocatalytic activity of prepared co-oxides was examined by removing Bismarck brown G dye from simulated industrial wastewaters. From the obtained results, the best efficiency for Bismarck brown G dye removal was found to be around 97. 5 %. This was achieved at optimal reaction conditions which were pH = 4, weight of catalyst 0.15 g, T = 303 K and time of reaction was 1 h. Both of Frundlich and Langmuir adsorption isotherms were studied.

Keywords: Dyes Removal, Bismarck brown G, Photocatalytic activity, Adsorption process.

INTRODUCTION

Nickel oxide can be used in photochemical reactions, especially to dispose of aromatic compounds that are found as pollutant in water. Nickel oxide is characterized by distorted structure, due to the presences of excess of oxygen that makes holes between the neighboring ions of Ni²⁺ and hence oxidation of Ni²⁺ to Ni³⁺ this charge makes the oxide coloured [1]. Chromium have different stable oxidation states, Cr₂O₅, CrO₃, CrO₆, CrO₂, Cr₅O₁₂ and Cr₂O₃. Among the various chromium oxides, Cr_2O_3 is the only one that is stable under ambient conditions [2]. Cr_2O_3 is a magnetic dielectric with corundum structure [3]. Cr₂O₃ materials have several applications in protective coating layer, solar absorber for thermal collectors, various catalytic systems [4-6]. There are different ways that can be used to remove colour textile effluents, including physical and chemical processes such as precipitation and adsorption methods [7,8]. Generally, these dyes developed environmental pollution by formation of carcinogenic materials and releasing toxic substances.

Photocatalysts is a composite semiconductor that can be used in photocatalytic and adsorption processes. The photocatalytic process involves excitation of semiconductor particles by absorption photon with a suitable energy. Photocatalyst is the semiconductor oxide that is able to increase the effective-

ness of removal of organic and inorganic pollutants in water and wastewaters [9]. This process will leave electron-hole pair on the surface of catalyst and then these species diffuse from the bulk of catalyst to the surface to participate in redox reactions [10]. While the adsorption process, which is completely different from absorption, adsorption happens essentially at the surface of the catalyst and consists of aggregation of a large number of species at the surface of catalyst [11,12]. There are two types of adsorption *i.e.*, physical adsorption, involves forces of molecular interaction likes dipole, induced dipole moments and van der Waals interaction. Physical adsorption is characterized by enthalpy changes that are smaller than chemical adsorption, typically in the range -10 to -40 kJ mol⁻¹, heats of adsorption of 10-40 kJ mol⁻¹ while chemisorption are rarely less than 80 kJ mol⁻¹, and often exceed 400 kJ mol⁻¹ [13]. Chemisorption forms one layer of adsorbate on adsorbent, which involves a chemical reaction between the adsorbate the surface new chemical bonds are generated at the adsorbent surface [14].

Bismarck brown G dye is a dark brown solid and belongs to azo dye group. The degradation of Bismarck brown G dye could release several aromatic amines that could be toxic [15]. The aim of present work is to study removal of Bismarck brown G by photocatalytic process and adsorption from wastewaters by photocatalytic reactions over prepared photocatalyst

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 Cr_2O_3 -NiO and under irradiation with UV light with applying of different reaction conditions.

EXPERIMENTAL

Chromium nitrate nonahydrate and nickel nitrate hexahydrate were obtained from BDH Company with purity of 99.9 and 97.9 %, respectively. Anhydrous sodium carbonate obtained from GmbH with purity of 99.9 %. The absorbance of supernatant liquid was measured at a wavelength of 468 nm by using UV-visible spectrophotometer (UV-1650PC Shimadzu, Japan). The Bismarck brown G dye was obtained from Al-Hilla Textile Factory, Hilla, Iraq.

Synthesis of catalyst: A ratio of 60:40 of $Cr(NO_3)_2 \cdot 9H_2O$ and $Ni(NO_3)_2 \cdot 6H_2O$ was weighed and dissolved in 400 mL deionized water in a 1 L beaker and using a magnetic stirrer. The pH of 5.5-6.2 was recorded for various mixed ratios. Then added 1 M Na₂CO₃ as precipitating agent and kept the solution at 70-75 °C (pH 9). The solution was then left for 2 h at the same temperature (70-75 °C) with constant stirring for digestion. The resulting solution was filtered by using a Buchner filtration flask and a vacuum pump. The Whatman 125 mm was used as a filtration paper. The precipitate was washed with dimineralized water and left for dried in an oven overnight at 120 °C. The resulting solid was calcined at different temperatures (500, 600 and 700 ° C) at a rate 10 °C/min for 4 h [16].

Powder X-ray Diffraction (PXRD): The prepared co-catalysts were characterized through powder X-ray analysis (Phillips X-ray diffraction) with CuK α radiation (1.542 Å, 40 KV, 30 MA), in the 20 range 10-80 degrees.

Fourier transform infrared spectroscopy (FTIR): Functional groups of the prepared catalysts were characterized using FTIR spectra (Perkin Elminer Company, England). All the spectra were recorded in the range of wavenumbers from 4000-400 cm⁻¹.

Photocatalytic activity of synthesized co-catalyst: The photocatalytic activity of the prepared co-catalysts was performed *via* following photocatalytic removal of Bismarck brown G dye. This process was performed using homemade photoreaction unit consisting of mercury lamp (250 W, Philips, Holland) without cover glass as a source of UV radiation, magnetic stirrer, hot plate and photo-reaction cell which was made from pyrex glass with quartz windows with reaction cell containing 30 mL of reaction mixture as shown in Fig. 1. All the experiments were performed by adding catalyst into photoreaction cell containing 30 mL of 50 ppm solution of Bismarck brown G. The temperature of the reaction mixture was maintained at 25 °C. The suspension solutions were prepared by adding different weight of Cr2O3-NiO catalysts to 30 mL of 50 ppm Bismarck brown G dye aqueous solutions. Before the irradiation, the suspensions were stirred in darkness for 10 min using a magnetic stirrer to ensure adsorption equilibrium. Then 1-2 mL from the reaction mixture was taken at interval of every 10 min for 1 h and centrifuged for 20 min. The supernatant liquid was carefully removed and then again centrifuged at the same speed and period of time. The second centrifuged was found necessary to remove fine particles of the catalyst. After the second centrifugation, the absorbance at maximum wavelength of 468 nm of Bismarck brown G dye was measured with a UV-visible spectrophotometer (UV-1650PC Shimadzu, Japan).

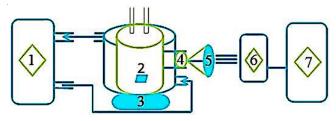


Fig. 1. Schematic description of the photochemical reactor (1 = Circulating water thermostat, 2 = reaction cell, 3 = magnetic stirrer, 4 = quartz window, 5 = lenses, 6 = low pressure mercury, 7 = power supply unit)

In this study, the photodegradation efficiency of dye over prepared catalyst was calculated using the following relation:

Photodegradation efficiency (%) =
$$\frac{A_o - A_t}{A_o} \times 100$$
 (1)

where A_0 and A_t is the initial and final absorption of dye.

Effect of amount of catalyst on the removal of Bismarck brown G dye over Cr_2O_3 -NiO: In order to investigate the effect of the using different doses of the catalyst on the efficiency of dye removal, a series of experiments were performed to determine the optimum weight of the catalyst to obtain the highest effective of dye removal. In each experiment, 30 mL of 50 ppm dye solution with a required amount of catalyst was mixed. The mixture was irradiated using different amounts of co-oxide catalyst (0.05, 0. 1, 0.15, and 0.2 g) at 25 °C for a period of 1 h.

Effect of temperature on the removal of Bismarck brown G dye over Cr_2O_3 -NiO: To investigate the effect of temperature of reaction mixture on the efficiency of Bismarck brown G dye removal over the prepared co-catalyst. A series of experiments were conducted using a 30 mL of 50 ppm dye solution. The reaction was performed at different temperatures (288, 293, 298, 303, 308 and 313 K). The weight of catalyst was 0.15g and irradiated for 60 min.

Effect of pH on the removal Bismarck brown G dye over Cr_2O_3 -NiO: The pH of the reaction mixture was controlled at the desired values using 0.01 M NaOH or1 M HCl solutions, then the pH values were measured by a pH meter. In each case, 0.15 g of catalyst and 30 mL of 50 ppm dye solution were mixed under UV irradiation at 298 K for 60 min.

Adsorption isotherm studies: For adsorption of Bismarck brown G dye, both of Freundlich and Langmuir isotherms were investigated over the prepared catalyst Cr_2O_3 -NiO. Series of experiments were carried out using 30 mL of 50 ppm of dye aqueous solution with 0.15 g of the catalyst at 25 °C for 1 h. The suspensions were filtered through centrifugation, and the adsorption capacity was analyzed for residual Bismarck brown G dye at 468 nm using the following equation:

$$q_e = \frac{(C_o - C_e) \times V}{W}$$

where q_e is the amount of Bismarck brown G dye adsorbed by Cr_2O_3 -NiO at equilibrium, C_o and C_e are the initial and final dye concentrations, respectively, V is the volume of solution (L), and W is the adsorbent weight (g).

Langmuir adsorption isotherm: Langmuir adsorption isotherm is applicable if adsorption takes place at specific homogeneous sites [17] and can be represented by the following equation:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e}$$

where q_m is the maximum amount of Bismarck brown G dye adsorbed per unit mass of catalyst and K_L is the Langmuir constant.

Freundlich adsorption isotherm: The Freundlich isotherm is applicable to heterogeneous system where adsorption takes place on several layers (n) > 1 [18] and can be described as follows:

$$\log (q_e) = \log K_F + \frac{1}{n} \log C_e$$

where K_F and n are Freundlich constants.

Pseudo-first order kinetic model: The rate constant of adsorption was determined by using pseudo-first order equation given by Lagergren and Svenska [19]:

$$\ln (q_t - q_e) = \ln (q_e) - k_1$$

where q_e and q_t (mg/g) are the amounts of Bismarck brown G dye adsorbed at equilibrium and at time t (min), respectively, and k_1 is the adsorption rate constant (min⁻¹).

Pseudo-second order kinetic model: The pseudo-second order equation based on equilibrium adsorption [20] is expressed as follows:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}$$

where k_2 is the rate constant of second order equation (g/mg min).

RESULTS AND DISCUSSION

X-ray diffraction technique is used to study the crystallinity state of the prepared catalyst Cr_2O_3 -NiO at calcination temperature (500 °C) and time 4 h. From the obtained results of XRD patterns for the prepared catalyst, it was found that there were some deviation in the positions of the peaks and d-spaces for standard values by matching with (JPCDS). This deviation is acquired by influencing between oxides [21]. X-ray diffraction patterns for the prepared catalyst are shown in Fig. 2.

FTIR studies: It was found that the absorption of transient element oxides in the region less than 600 cm⁻¹ (Fig. 3). The spectrum shows an absorption peak of nickel oxide at 418 and 482 cm⁻¹ [22] and absorption peak at 606 and 504 cm⁻¹ for chromium oxide [23] as well as the absorption peak at 3500-3400 cm⁻¹ due to v(OH) resulting from the disintegration and adsorption of water molecules on the surface [24].

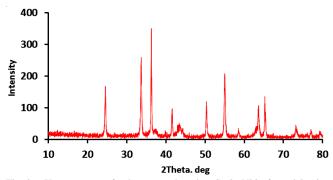


Fig. 2. X-rays patterns for the prepared catalyst $Cr_2O_3\text{-NiO}$ after calcination at 500 $^\circ\text{C}$

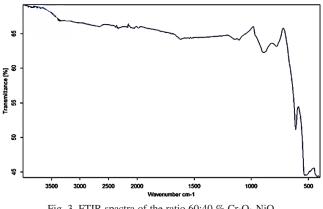


Fig. 3. FTIR spactra of the ratio 60:40 % Cr₂O₃-NiO

Effect of amount of catalyst on dye removal: It is found that 0.15 g of catalyst dosage for removing Bismarck brown G dye is maximum (Fig. 4). The increase in catalyst dose leads to the reduction in efficiency of dye removal. At high concentrations of the catalyst with the same light source, catalyst particles absorb the photons and prevent passing to the other side of reaction mixture and resulted to light scattering [25,26].

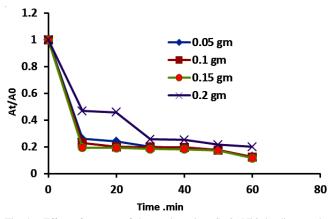


Fig. 4. Effect of amount of the used catalyst Cr₂O₃-NiO loading on the efficiency of photocatalytic removal Bismarck brown G

Effect of pH on photocatalytic degradation of Bismarck brown G dye over prepared co-catalyst: The photocatalytic efficiency of Bismarck brown G dye was studied at different pH values (2,4,7 and 9). In each case, 0.15 g of catalyst was UV irradiated with 30 mL of 50 ppm dyes solution at 25 °C for 1 h. The highest photodegradation efficiency of Bismarck brown G dye was found to be around 88.46 % at pH 4 (Fig. 5).

Effect of temperature on photocatalytic degradation of Bismarck brown G dye: By keeping other experimental conditions constant, a catalyst dose of 0.15 g, pH 4 for 50 ppm in 30 mL of simulated wastewaters, the effect of temperature on the photocatalytic efficiency of Bismarck brown G dye was studied at different temperatures. The results of efficiency was increased with increase of applied reaction temperatures to approach 97.5 % (Fig. 6). Generally, temperatures does not effect on photodegradation catalysis, but upon increasing temperature can effect on the amount of adsorbed particles and helped the reaction to complete more efficiently [27,28].

Effect of irradiation time on removal Bismarck brown G dye from wastewater: The effect of irradiation time on

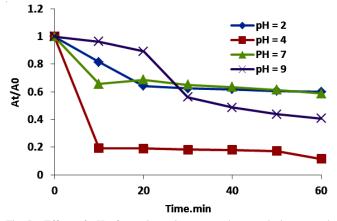


Fig. 5. Effect of pH of reaction mixture on photocatalytic removal efficiency of Bismarck brown G over co-oxide Cr₂O₃-NiO

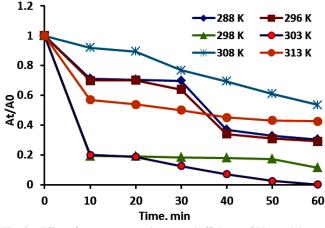


Fig. 6. Effect of temperature on the removal efficiency of Bismarck brown G over co- oxides Cr₂O₃-NiO

removal effciency of Bismarck brown G dye was carried out by measuring absorbance of dye solution at different periods of time using 0.15 g of catalyst and 30 mL of 50 ppm dye solution under UV-visible light at 30 °C. Fig. 7 shows that the photocatalytic activity increases with increase of time and reaches to 97.5 % after 60 min of irradiation time [29].

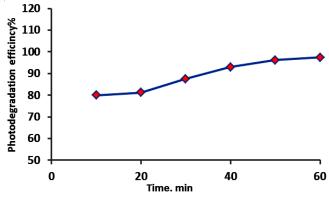


Fig. 7. Effect of irradiation time on the photocatalytic dye removal over the prepared co-catalysts

Adsorption activity of prepared catalyst Cr_2O_3 -NiO: The adsorption and thermodynamic experiments were carried out in the presence of co-oxide Cr_2O_3 -NiO. The effects of different parameters were studied such as adsorbent dosage, contact time, and temperature. It is found that the optimum conditions that give good removal is 0.2 g for 30 mL dyes solution and pH 4 and 298 K. The results are shown in Fig. 8.

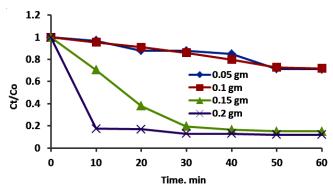


Fig. 8. Effect of co- oxide dosage on the efficiency of adsorption removal Bismarck brown G from aqueous solution

The adsorption removal increases with increasing Cr_2O_3 -NiO dosage from 28.63 to 88.59 % due to that number of available adsorption sites increases by increasing the adsorbent amount, but any drop in adsorption capacity is basically due to the sites remaining unsaturated during the adsorption process [30,31]. Also the removal of adsorption will increase with increasing temperature. The adsorption removal decreases with any increase in temperature, because of increases the rate of the diffusion of the dye molecules [32]. The results are shown in Fig. 9.

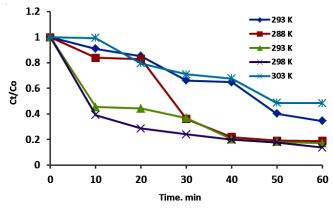


Fig. 9. Effect of temperature on the adsorption of Bismarck brown G dyes Cr₂O₃-NiO

Adsorption isotherms for dye adsorption using Cr_2O_3 -NiO catalyst: Adsorption isotherms were studied using Langmuir and Freundlich equilibrium models. These isotherms are shown in Figs. 10 and 11, respectively. Table-1 shows values

TABLE-1 ADSORPTION CONSTANTS OF THE LANGMUIR AND FREUNDLICH ISOTHERMS						
Isotherms	Parameters	Values				
Freundlich	K _F	2.5446				
	n	2.33				
	\mathbb{R}^2	0.724				
Langmuir	Q _m	21.97				
	K _L	0.657				
	\mathbb{R}^2	0.672				

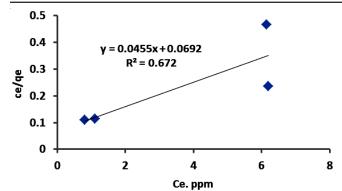


Fig. 10. Langmuir adsorption isotherm for adsorption of Bismarck brown G on the prepared catalyst

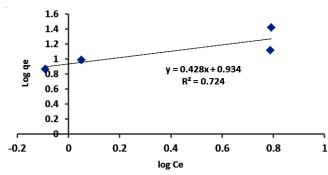


Fig. 11. Freundlich adsorption isotherm for adsorption of Bismarck brown G over the catalyst

of Langmuir and Freundlich adsorption isotherms. It can be seen that the value of the correction factor (R_2) that are obtained from Langmuir model is lower than that for Freundlich isotherm. And the number of adsorbed layers is 2.33, in this case adsorption processes were followed Langmuir adsorption isotherm [33].

Adsorption kinetics: It is found that the value of correction factor for pseudo-second order kinetic model (0.9995-1), higher than the value of the first order coefficient factor. The pseudo-first order kinetic model through the results (Figs. 12 and 13 and Table-2), it is clear that the dye adsorption process of Bismarck brown G dye follows the pseudo-second order kinetic model [34].

Thermodynamic parameters of adsorption removal of Bismarck brown G dye dyes are reported in Table-3 and Fig. 14. From the following equation:

$\Delta H = E_a - RT$	[
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TABLE-2 ADSORPTION PARAMETERS FOR BISMARCK BROWN G DYE ADSORPTION ONTO Cr ₂ O ₃ -NiO						
	Pseudo first order model					
Wt (g)	$q_{e,exp}(mg/g)$	$q_{e,cal}(mg/g)$	$K_1(min^{-1})$	\mathbb{R}^2		
0.05	26.280	1.510	0.0198	0.9323		
0.10	13.155	1.257	0.0352	0.8455		
0.15	9.770	3.410	1.1582	0.9629		
0.20	7.370	1.670	0.0958	0.8757		
Wt (g)	Pseudo second order model					
wt (g)	$q_{e,exp}(mg/g)$	$q_{e,cal}(mg/g)$	K ₂ (g/mg min)	\mathbb{R}^2		
0.05	26.280	26.66	0.0350	0.9995		
0.10	13.155	26.66	0.0350	0.9995		
0.15	9.770	9.870	0.1934	0.9999		
0.20	7.370	7.401	0.1350	1.0000		

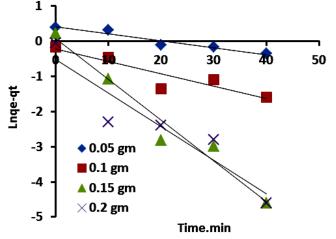


Fig. 12. Pseudo-first order kinetic model for Bismarck brown G dye adsorption onto Cr₂O₃-NiO

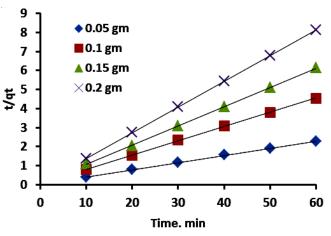


Fig. 13. Pseudo-second order kinetic model for Bismarck brown G dye adsorption onto Cr₂O₃-NiO

$\ln K = \Delta S/R - \Delta H/RT$

$\Delta \mathbf{G} = \Delta \mathbf{H} - \mathbf{T} \Delta \mathbf{S}$

The positive Gibbs energy of adsorption (ΔG°) indicates that the reaction is non-spontaneous and negative entropy (ΔH°) refers exothermic reaction. The negative values of ΔS° also revealed the degree of randomness at the solid-liquid interface during the adsorption of Bismarck brown G dye onto Cr₂O₃-NiO co-oxide catalyst. Thermodynamic parameters are dependent on the equilibrium adsorption constant (K_{ad}). The van't

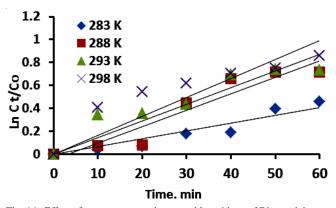


Fig. 14. Effect of temperature on the normal logarithms of Bismarck brown G absorbance

TABLE-3 THERMODYNAMIC PARAMETERS OF THE ADSORPTION OF BISMARCK BROWN G OVER Cr2O3-NIO CO-OXIDES								
$W_{t}(\alpha) = E (I m \alpha l^{-1})$		Temp. (K)						
wt. (g)	Wt. (g) $E_a (J \text{ mol}^{-1})$		283	288	293	298		
0.2 42.54	$\Delta G (Kj mol^{-1})$	14306.17	12640.84	12693.2	12573.17			
	$\Delta H (Kj mol^{-1})$	-2.310	-2.352	-2.393	-2.435			
	$\Delta S (Kj mol^{-1})$	-50.560	43.900	43.350	42.200			

Hoff equation was used to evaluate the variation of equilibrium adsorption constant with temperature (Fig. 15) [35].

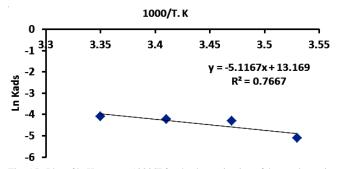


Fig. 15. Plot of $\ln K_{ad}$ versus 1000/T for the determination of thermodynamic parameters

Conclusion

The synthesized co-oxide Cr_2O_3 -NiO catalyst has high porosity at 500 °C, so it used in the removal of Bismarck brown G dye by adsorption at pH 4, catalyst conc. 0.15g/L, temperature 30 °C and adsorption time of 60 min. The equilibrium adsorption isotherms from the obtained results adsorption isotherms, it was more fitted with the Frendlich adsorption model and the value of correction factor for pseudo-second order kinetic model higher than the value of pseudo-first order kinetic model. The adsorption process of Bismarck brown G dye follows the pseudo-second order kinetic model.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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