

# Synthesis and Characterizations of Reduced Graphene Oxide/ Iron Oxide: as a Model of Water Treatment

Ibrahim Jooda Sahib<sup>1</sup>, Laith Sameer Jasim<sup>1</sup> and Ayad F. Alkaim<sup>2</sup>

*Department of Chemistry, College of Education, University of Al-Qadisiyah, Iraq.  
Department of Chemistry, College of Science for women University of Baby;on, Iraq.*

## Abstract

In this study, graphene oxide (GO), produced using the modified Hummer's method, (RGO) produced using ultra violet light. (RGO / Fe<sub>2</sub>O<sub>3</sub>) composite prepared by hydrothermal method . and it's used as adsorbent to remove maxilon blue (GRL) from aqueous solution. Characterizations using field emission scanning electron microscopy (FE-SEM) , powder X-ray diffraction (XRD) , energy dispersive X-ray spectroscopy (EDX) were carried out on the RGO / Fe<sub>2</sub>O<sub>3</sub> before the GRL adsorption experiments. The adsorption isotherms studies were conducted under different conditions (pH = 3-11 , GRL concentration = 10-100 mg/L and Wheight of compsite= 0.005-0.25 g ) to examine the adsorption efficiency of the RGO / Fe<sub>2</sub>O<sub>3</sub> towards GRL in aqueous solution.

**Keyword:** Reduced graphene oxide, iron oxide, Maxilon Blue GRL

## INTRODUCTION

Pollution is the most widespread major problem that has caused a defect in the ecosystem, as well as a dangerous problem that threatens human life. Therefore, it is difficult to obtain clean water in the existence of large amounts of wastewater. Where he found that all developing and industrial countries the ratio of water pollution that affects the human system is increasing[1].Textile dyes have a strong color even in extremely low concentrations. These dyes nondegradable, bioaccumulation in living organisms and stable toward light, biological and chemical treatments, additionally display high biotoxicity and potential mutagenic and carcinogenic effects[2]. Numerous methods can be used to remove them with high efficiency such as ion exchange, coagulation/flocculation, adsorption, chemical oxidation, ozone treatment, membrane filtration , sono-chemical and electrochemical methods, photocatalysis[3-9]. Among these techniques, adsorption is respected as effective and economical innovation because of its high effectiveness, naivety of design and simplicity of operation[10].Nanotechnology is described as science, engineering and technology associated to how to understand, control, and use materials whose dimensions are between 1-100 nanometers. Nanotechnology is a huge project aimed at reducing substances, instruments and tools. This technique not only reduced of the size of nanomaterials produced, but also

greatly sought to enhance the properties of nanomaterials and the invention of new nanomaterials with unique properties<sup>[11]</sup>. Nanomaterial is indicated to as the broad spectrum of substances whose particles possess 1 or 2 dimensions, subject to a nanometer ranging from 1-100 nanometers. Where the nanoscale is approximately 1,000 times lesser than the micrometer<sup>[12]</sup>.

## EXPERIMENTAL PART

### Chemical Materials

Graphite , Sulfuric acid 98% , Hydrochloric acid , Sodium hydroxide 99%, Potassium permanganate 99.5% , Hydrogen peroxide (30%) 99% , Phosphoric acid and Ferric (II) chloride .

### Characterization

The FE-SEM image was obtained with a JEOL JSM-6330F. The XRD pattern was recorded on a (PhilipsX'Pert Pro Super X-ray) diffractometer with a Cu K $\alpha$  source ( $\lambda = 1.54178 \text{ \AA}$ ). UV-vis absorption spectra were recorded on a Lambda 35 spectrometer (Perkin-Elmer).

### Preparation of Graphene Oxide (GO)

Graphene oxide (GO) was synthesized from natural graphite powder by a modified Hummers method<sup>[13]</sup>.

### Preparation of rGO/Fe<sub>2</sub>O<sub>3</sub> Composite

We can be preparation of RGO by supplied of the UV light on the GO solution at 24 hr . Then add 2g FeCl<sub>3</sub> powder and 2g NaOH to RGO solution and mixing at 15 min by magnetic stirrure and putting in Furnace at 24 hr. After that drier by Oven.

### Adsorption Experiments

Adsorption experiments were carried out in glass bottles at 25 °C. 100 mL of dye solution of a known initial concentration was stirred with 0.1 g of rGO/Fe<sub>2</sub>O<sub>3</sub> Compositeat. After magneticseparation using a permanent magnet, the equilibrium

concentrations of the dyes were measured with a UV-vis spectrophotometer at a wavelength of 590 nm. The adsorption capacity and removal efficiency of the Maxilon Blue GRL on the adsorbents were calculated according to the following equations<sup>[14]</sup>.

$$q_e = \frac{V_{sol}(C_o - C_e)}{m} \dots\dots\dots (1)$$

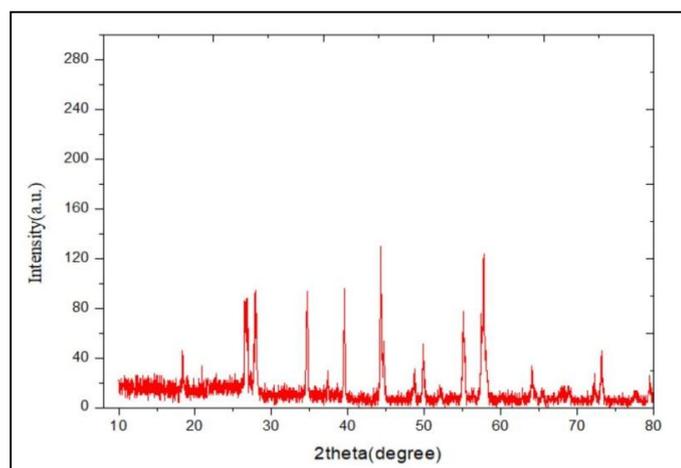
$$\text{Removal\%} = \frac{(C_o - C_e)}{C_o} * 100 \dots\dots\dots (2)$$

where  $q_e$  ( $\text{mg g}^{-1}$ ) is the amount of dye adsorbed onto the adsorbent at equilibrium,  $C_o$  ( $\text{mg L}^{-1}$ ) and  $C_e$  ( $\text{mg L}^{-1}$ ) are the initial and equilibrated dye concentrations, respectively,  $V$  (L) is the volume of solution added, and  $W$  (g) is the mass of the adsorbent.

## RESULTS AND DISCUSSION

### Characterization of adsorbent

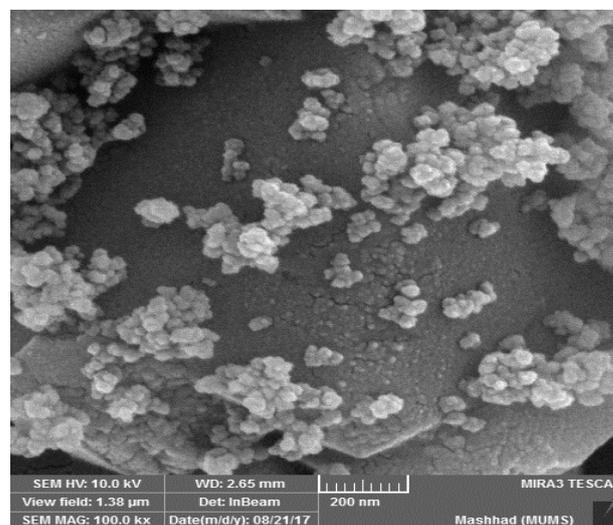
Fig. 3-1 shows the XRD patterns of  $\text{RGO} / \text{Fe}_2\text{O}_3$  hybrids. The diffraction pattern of it's shows a weak peak at around  $2\theta = 18^\circ$ , originated from its (001) reflection. Another wide peak around  $2\theta = 28^\circ$  can also be observed, which is the characteristic (002) peak of residual unoxidized graphite. the disappearance of the peak at  $2\theta = 10.2^\circ$  confirms that oxygen groups have been removed and GO has been flaked and reduced to RGO nanosheets, The diffraction peaks correspond to the (39), (44), (50), (56), (58), (64), and (74) crystal planes of cubic spinel structural.



**Figure 3-1:** XRD analysis of  $\text{RGO} / \text{Fe}_2\text{O}_3$  nanocomposite

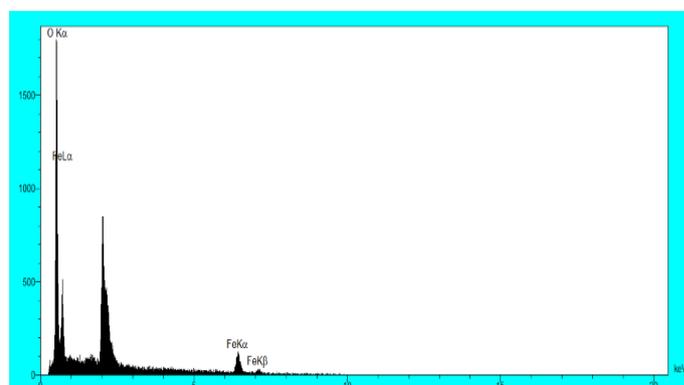
The image of  $\text{Fe}_2\text{O}_3/\text{rGO}$  (shown in Fig 3-2) indicates that the RGO sheets are tightly bound onto the  $\text{Fe}_2\text{O}_3$  microspheres.  $\text{Fe}_2\text{O}_3$  spheres greatly help in preventing the restacking of the RGO sheets, avoiding the loss of a highly active surface area. Furthermore, due to the good exfoliation of graphite, the obtained GO layers are apparently transparent even under FE-SEM observation<sup>[15]</sup>. FE-SEM image of  $\text{Fe}_2\text{O}_3$  revealed that the obtained gray nanoparticles were spherical in shape, and they

aggregated because of their extremely small size and dipole-dipole coupling. The RGO sheet exhibited an irregular shape and contained some wrinkles, which maintained a large surface area. Show that the spherical structure  $\text{Fe}_2\text{O}_3$  nanoparticles grow over the nanosheets of RGO regularly with diameters ranging from 10 to 20 nm. The dispersion of  $\text{Fe}_2\text{O}_3$  nanoparticles is well, and there is little observable aggregation of those nanospheres, which is attributed to the in-situ growth of  $\text{Fe}_2\text{O}_3$  nanoparticles followed with the chemical interaction between ferric/ferrous ions and the carboxylate or hydroxyl groups<sup>[16]</sup>.



**Figure 3-2:** FE-SEM analysis of  $\text{RGO} / \text{Fe}_2\text{O}_3$  nanocomposite

Figure 3-3 shows EDX spectrum of the  $\text{rGO}/\text{Fe}_2\text{O}_3$  nanocomposite. The analysis shows that the prepared composite includes only iron, carbon, and oxygen in similar with the chemical composition of the nanocomposite.



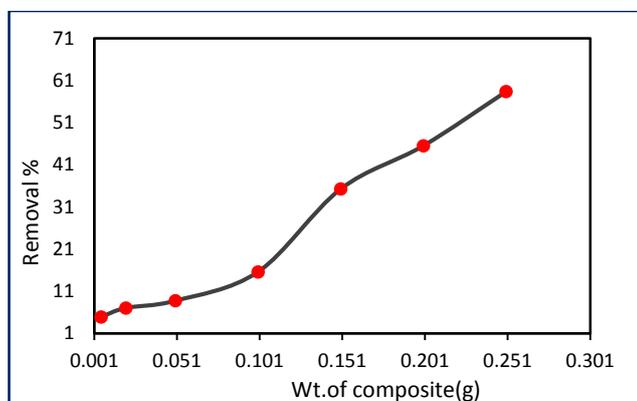
**Figure 3-3:** EDX analysis of  $\text{Fe}_2\text{O}_3/\text{rGO}$  nanocomposite

## Adsorption Experiments

### Effect of Weight of composite

Variation of adsorbent dose showed that although increasing of Weight of composite in aqueous solution can result to increased

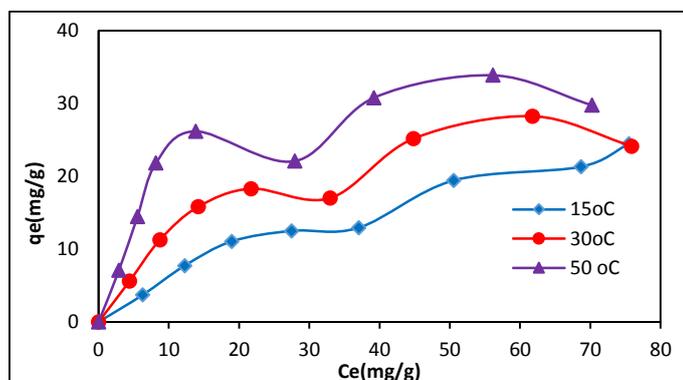
dye removal. Fig. 3-4 shows the plot of Removal % of dye adsorption against the weight of adsorbent in g. From the figure, it is observed that the percentage of adsorption is increases from with increase in the adsorbent. This can be attributed to an increase in surface area of the sorbent, which in turn increases the binding sites. At higher dosage, there is a very fast adsorption on to the adsorbent surface that leads to improved uptake of the dye<sup>[17]</sup>.



**Figure 3-4:** Effect of adsorbent dose on the removal percentage of dye on nanocomposite

#### Effect of Initial Dye Concentration and Temperature

Figure (3-5) shows that the adsorption rate increases with increasing temperature showing the reaction is an endothermic reaction (Table 3-1) type leads to an increase in the propagation speed of the adsorbate species on the adsorbent outer surface with reducing a viscosity of the solution. Moreover, increasing temperature increases the mobility of dye molecules and thus increases the number adsorbate molecules that interact with the effective sites of the surface<sup>[18]</sup>.



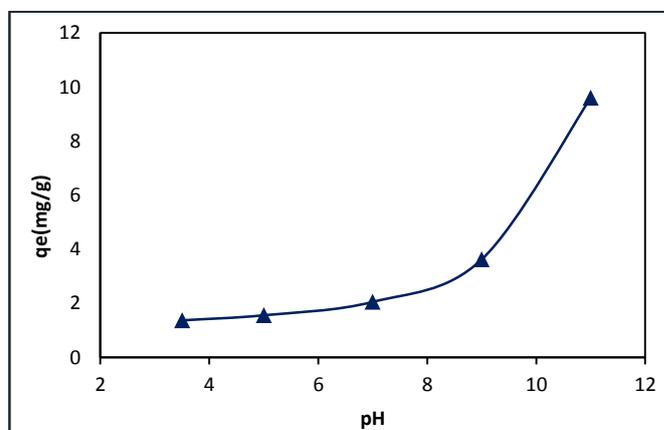
**Figure 3-5:** Effect of Initial Dye Concentration and temperature on the adsorption capacity of dye

**Table 3-1:** Thermodynamic parameters of adsorption of dye on nanocomposite surface

Dye	$\Delta H$ (kJ.mol <sup>-1</sup> )	$\Delta G$ (kJ.mol <sup>-1</sup> )	$\Delta S$ (J.mol <sup>-1</sup> .K <sup>-1</sup> )	Equilibrium Constant (K)
dye	+14.468	16.44	+42.32	0.509

#### Effect of pH

Solution pH can play an important role for the adsorption of the analytes by affecting both the existing forms of the target compounds and the charge species and density on the sorbent surface. In this work, the effect of solution pH on the extraction of target is investigated in the pH range of 3.0-11.0. As can be seen from Fig. 3-6, the sorption percentage of dye on **Fe<sub>2</sub>O<sub>3</sub>/rGO** composite very little in pH range of 3-8, which suggests that **Fe<sub>2</sub>O<sub>3</sub>/rGO** are excellent adsorbents for dye removal from large volumes of aqueous solutions. When the pH is greater than 8, the sorption percentage of dye on composite increases with increasing pH values. This can be ascribed to the fact that more oxygen containing groups (such as -COOH and -OH) on **Fe<sub>2</sub>O<sub>3</sub>/rGO** composite are ionized (carrying negative charge) at high pH values<sup>[19]</sup>.



**Figure 3-6:** Effect of solution pH on adsorption dye on nanocomposite surface

#### CONCLUSIONS

The obtained results showed that the maximum adsorption capacity of the RGO / Fe<sub>2</sub>O<sub>3</sub> towards GRL can achieve up to ~0.1g for the adsorption at 10 mg/L GRL at 50 C. and the maximum adsorption capacity of the RGO / Fe<sub>2</sub>O<sub>3</sub> towards GRL can achieve up to pH=11 and 0.25 g for composite.

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