

# Synthesis and characterization of magnetite-graphene oxide nanocomposite

Manuel Alejandro Perez Guzman

Ph. D. Program in Nanoscience and Nanotechnology.  
Cinvestav-IPN.

Av. IPN 2508, San Pedro Zacatenco, 07360.  
Mexico City. Mexico.  
maperez@cinvestav.mx

Jaime Santoyo Salazar

Physics Department.

Ph. D. Program in Nanoscience and Nanotechnology.  
Cinvestav-IPN.

Av. IPN 2508, San Pedro Zacatenco, 07360.  
Mexico City. Mexico.

Rebeca Ortega Amaya

SEES, Electric Engineering Department.  
Cinvestav-IPN.

Av. IPN 2508, San Pedro Zacatenco, Mexico City.  
Mexico 07360

Yasuhiro Matsumoto

SEES, Electric Engineering Department.

Ph. D. Program in Nanoscience and Nanotechnology.  
Cinvestav-IPN.

Av. IPN 2508, San Pedro Zacatenco, 07360.  
Mexico City. Mexico.

Mauricio Ortega Lopez

SEES, Electric Engineering Department.

Ph. D. Program in Nanoscience and Nanotechnology.  
Cinvestav-IPN.

Av. IPN 2508, San Pedro Zacatenco, 07360.  
Mexico City. Mexico.

**Abstract**— This work presents the advances in the synthesis and characterization of magnetite-graphene oxide nanocomposite synthesized by coprecipitation. Structural and physicochemical properties were analyzed by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FT-IR), transmission electron microscopy (TEM) and vibrating sample magnetometer (VSM). XRD and FT-IR analyses corroborated that magnetite was obtained. Nanoparticle mean size from XRD analysis revealed that it decreased when graphene oxide (GO) was present. TEM displayed that magnetite decorates the GO and VSM revealed superparamagnetic behavior.

**Keywords**—magnetite, graphene oxide, nanocomposite

## I. INTRODUCTION

Because of its biocompatibility, superparamagnetic iron oxides (magnetite and maghemite) nanoparticles have a great variety of applications as contrast agents in magnetic resonance image (MRI), biological separation agents, hyperthermia cancer treatment, drug delivery and magnetic inks[1-3].

On the other hand, graphene oxide based nanocomposites are under study because of their unique properties, and they have applications in many technological areas like biomedical, electronics, energy and environmental remediation [4]. In all

cases, nanocomposites look to join the graphene properties and the other material properties [5].

In this work, the results of the synthesis and characterization of magnetite-graphene oxide nanocomposite are present. The nanocomposite was obtained by coprecipitation method in presence of GO. For comparison also, magnetite nanoparticles were synthesized without presence of GO.

## II. MATERIALS AND METHODS

### A. Materials

Iron (II) chloride tetrahydrate (99%), iron (III) chloride hexahydrate (97%), hydrochloric acid (36%) and ammonium hydroxide (30%), were purchased from Sigma-Aldrich. All reagents were used as received without further purification.

### B. Methods

The GO, was obtained as described in [6].

Nanoparticles aqueous synthesis.

2M HCl solution were used to prepare 2 M Fe(II) and 1 M Fe (III) iron solutions. Then in a 150 mL beaker under continuous stirring 4 mL iron (III), 1 mL iron (II) solutions and

50 mL of deionized water were added, the solution turned to orange color and had a pH=1, then 50 mL of 1 M ammonium hydroxide basis solution was added with a burette in 3 minutes, while the drop wise took place the solutions turned to back color and finally pH=9 was reached. Finally, the black product was precipitated with a magnet aid, the product was washed several times, with deionized water and freeze-dried.

#### Nanocomposite synthesis

Same procedure was done in presence of GO, before iron salts, 10 ml of a 20 mg/ml of GO solution were added.

### III. CHARACTERIZATION

The obtained products were analyzed by, X-Ray Diffraction (XRD), using a X Pert-Pro system with Cu-K $\alpha$  radiation, Fourier transform infrared spectroscopy (FT-IR) GX Perkin Elmer, transmission electron microscopy (TEM) JEOL 2010 and vibrating sample magnetometer (VSM) Lake Shore 7300.

### IV. RESULTS AND DISCUSSION

#### A. XRD

Figure 1, shows XRD pattern of magnetite nanoparticles and nanocomposite. In both cases, the diffracted peaks located at  $2\theta = 30.1, 35.5, 43.2, 53.5, 57.1$  and  $62.7^\circ$ , corresponded to the (200), (311), (400), (422), (511) and (440) planes of magnetite (JCPDS 19-0629), the crystal size calculated by Scherrer formula, was 20 nm and 12 nm, for magnetite and nanocomposite, respectively. The change in size, could be since the presence of GO will prevent the growth of nanoparticles.

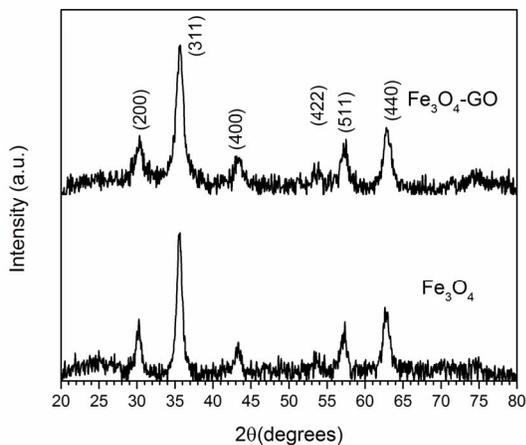


Figure 1. XRD patterns of Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>3</sub>O<sub>4</sub>-GO.

#### B. FT-IR

Figure 2 shows FT-IR spectra for Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>3</sub>O<sub>4</sub>-GO, in both cases, show bands around  $570\text{ cm}^{-1}$ , those bands are associated to the stretching vibration modes of the magnetite

Fe-O bonds [7]. There are also present C=O stretching vibrations ( $1720\text{ cm}^{-1}$ ) and O-H stretching vibrations ( $3300\text{ cm}^{-1}$ ). Fe<sub>3</sub>O<sub>4</sub>-GO spectrum has prominent O-H and C=O signals, due the functional groups present at the GO sheets [8].

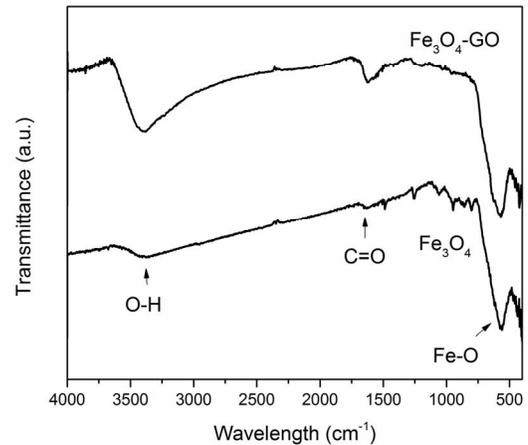


Figure 2. FT-IR spectra of Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>3</sub>O<sub>4</sub>-GO.

#### C. Morphology.

Figure 3 shows TEM images of (a) Fe<sub>3</sub>O<sub>4</sub> nanoparticles and (b) Fe<sub>3</sub>O<sub>4</sub> nanoparticles on the surface of a GO sheet. Agglomerated nanoparticles (fig. 3a), with particle mean size of 18 nm can be appreciated, the value is near to that obtained by XRD. When GO was present (fig. 3b), the agglomeration was considerably reduced as the particle size.

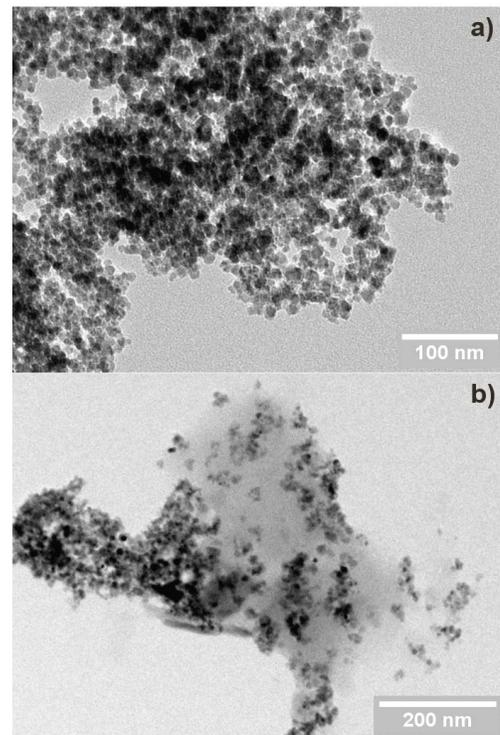


Figure 3. TEM images of (a) magnetite nanoparticles and (b) magnetite-GO nanocomposite.

#### D. VSM

Figure 4 shows, the hysteresis loops obtained at room temperature. Both samples showed superparamagnetic behavior and saturation magnetization ( $M_s$ ) values of 58 and 46 emu/g, for magnetite nanoparticles and nanocomposite respectively, the decrease in magnetization, is related to the nanoparticle size decrease and to the GO mass present in nanocomposite.

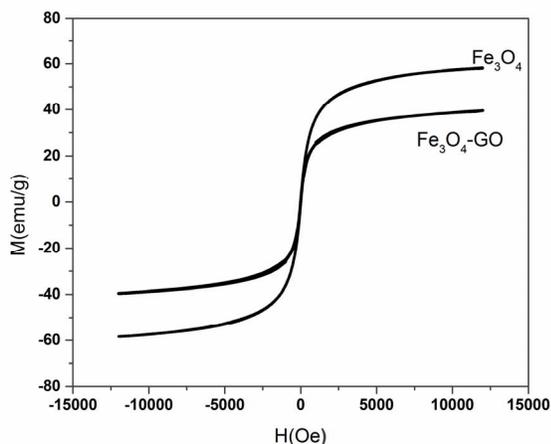


Figure 4. Hysteresis loops of  $\text{Fe}_3\text{O}_4$  nanoparticles and  $\text{Fe}_3\text{O}_4$ -GO nanocomposite measured at 300 K.

#### V. CONCLUSIONS

This work reports our recent progress on the synthesis and characterization of magnetite nanoparticles and magnetite-graphene oxide nanocomposite. The magnetite phase was obtained in both cases. The morphology revealed polydispersity in nanoparticles and that magnetite nanoparticles, decorate GO sheets. In all cases superparamagnetic behavior were obtained

#### ACKNOWLEDGMENT

The authors have special acknowledgement to Álvaro Guzmán Campuzano by his technical assistance during the synthesis and M. Sc. Adolfo Tavira by X-ray diffraction measurements both from SEES-Cinvestav

#### REFERENCES

- [1] L. Li, K. Y. Mak, C. W. Leung, K. Y. Chan, W. K. Chan, W. Zhong, et al., "Effect of synthesis conditions on the properties of citric-acid coated iron oxide nanoparticles," *Microelectronic Engineering*, vol. 110, pp. 329-334, 10// 2013.
- [2] M. Mahmoudi, S. Sant, B. Wang, S. Laurent, and T. Sen, "Superparamagnetic iron oxide nanoparticles (SPIONs): Development, surface modification and applications in chemotherapy," *Advanced Drug Delivery Reviews*, vol. 63, pp. 24-46, 1// 2011.
- [3] R. Qiao, C. Yang, and M. Gao, "Superparamagnetic iron oxide nanoparticles: from preparations to in vivo MRI applications," *Journal of Materials Chemistry*, vol. 19, pp. 6274-6293, 2009.
- [4] S. Bai and X. Shen, "Graphene-inorganic nanocomposites," *RSC Advances*, vol. 2, pp. 64-98, 2012.
- [5] X. Huang, X. Qi, F. Boey, and H. Zhang, "Graphene-based composites," *Chemical Society Reviews*, vol. 41, pp. 666-686, 2012.
- [6] R. Ortega-Amaya, Y. Matsumoto, M. A. Pérez-Guzmán, and M. Ortega-López, "In situ synthesis of  $\text{Cu}_2\text{O}$  and  $\text{Cu}$  nanoparticles during the thermal reduction of copper foil-supported graphene oxide," *Journal of Nanoparticle Research*, vol. 17, pp. 1-8, 2015/10/05 2015.
- [7] A. Shukla, M. K. Patra, M. Mathew, S. Songara, V. K. Singh, G. S. Gowd, et al., "Preparation and Characterization of Biocompatible and Water-Dispersible Superparamagnetic Iron Oxide Nanoparticles (SPIONs)," *Advanced Science Letters*, vol. 3, pp. 161-167, // 2010.
- [8] J. Shen, B. Yan, M. Shi, H. Ma, N. Li, and M. Ye, "One step hydrothermal synthesis of  $\text{TiO}_2$ -reduced graphene oxide sheets," *Journal of Materials Chemistry*, vol. 21, pp. 3415-3421, 2011.