v. 4, n. 13, 2024

Journal of
Engineering
Research

SYNTHESIS OF MAGNETIC GRAPHENE OXIDE AND ITS REDUCTION IN A SINGLE STEP

Verónica Cedeño-Garcidueñas
Environmental Engineering Laboratory, Department of Environmental Sciences, ``Instituto Tecnológico del Valle de Morelia``, Morelia, Michoacán, Mexico

Ricardo Rangel-Segura
Kinetics and Catalysis Laboratory, Post-graduation Degree in Chemical Engineering, ``Universidad Michoacana de San Nicolás de Hidalgo``, Morelia, Michoacán, Mexico

Armando Estrada-Córdoba
Environmental Engineering Laboratory, Department of Environmental Sciences, ``Instituto Tecnológico del Valle de Morelia``, Morelia, Michoacán, Mexico

Omar Sánchez-Ortiz
Environmental Engineering Laboratory, Department of Environmental Sciences, ``Instituto Tecnológico del Valle de Morelia``, Morelia, Michoacán, Mexico

Carmen García-Castillo
Environmental Engineering Laboratory, Department of Environmental Sciences, ``Instituto Tecnológico del Valle de Morelia``, Morelia, Michoacán, Mexico

Guillermo Andrade Espinosa
Environmental Engineering Laboratory, Department of Environmental Sciences, ``Instituto Tecnológico del Valle de Morelia``, Morelia, Michoacán, Mexico

Alberto Millán Montañez
Environmental Engineering Laboratory, Department of Environmental Sciences, ``Instituto Tecnológico del Valle de Morelia``, Morelia, Michoacán, Mexico

Keywords: Magnetic graphene oxide; Synthesis; Reduction.

INTRODUCTION

Graphene oxide (GO) is a two-dimensional material formed in a hexagonal network, with a large surface area, high electron mobility and high longitudinal elasticity, making it a very promising candidate for use in electronic applications, data storage devices. energy, sensors (Basu & Bhattacharyya, 2012), as well as for its use as an adsorbent in the treatment of wastewater and soil, for the removal of dyes and heavy metals such as Pb2+ and Cu2+ (Lai et al., 2019; Zhao, Guan, et al., 2019; Zhao, Yang, et al., 2019). However, the surface modification of OG is being studied to improve its adsorption capacity, as well as its subsequent recovery after this process in a solid-liquid system, and finally its recyclability. It is from these needs that the oxide arises. of magnetic graphene (GMO), which is a hybrid material composed of OG and a magnetic material such as iron oxide, contributing to its easy recovery through an external magnetic field, and it is also easy to add functional groups to the surface, characteristics that make it an exceptional candidate for different applications. (Mahdavi et al., 2021). Recent studies report that the GMO is used in the adsorption of heavy metals and even radionuclides, in addition to this it has been shown that there is no significant loss in its adsorption capacity when it is used for several adsorption cycles (Lingamdinne et al., 2019). On the other hand, there are various methods for GMO synthesis, among the most used are the co-precipitation, hydrothermal and solvothermal methods. In simple terms, the co-precipitation method is when the material is obtained through a reaction where a precipitate is formed, this being the desired material. The hydrothermal method consists of placing a precursor and water in a closed system at high pressure and temperatures, in the case of the solvothermal method it can be said that it is the same but the solvent in this case is organic, having the advantages that by modifying the conditions of the system Properties such as the size, density and crystallinity of the iron oxide can be modified (He et al., 2021). In this work, the synthesis of GMOs in a solvothermal manner is proposed, and the reduction of GMO at the same time. The morphology and chemical composition of the material was evaluated through Scanning Electron Microscopy (SEM) and Electron Scattering Spectroscopy (EDS), showing the surface presence on the OG flakes.

MATERIALS AND METHODS

The synthesis of graphene oxide was obtained through the modified Hummers method where 3 g of graphite are placed in sulfuric acid, which are mixed in a 1000 mL beaker and introduced into an ice bath, subsequently permanganate is added. of potassium under vigorous stirring and maintaining a temperature below 20°C. After approximately 2 hours the solution is transferred to an oil bath with a temperature of 40°C and constant stirring, then distilled water is added little by little without the temperature exceeding 98°C. After 30 minutes, 150 mL of distilled water is added and finally after 15 minutes, 30% H2O2 is added. Afterwards, the solution is washed with a solution of HCl and distilled water until a neutral pH is reached (Chen et al., 2013). Once the appropriate pH is obtained, it is placed in the oven for 48 hours at a temperature of 100°C. The solvothermal synthesis of the magnetic OG proceeded as follows: first, 0.38g of FeSO4 and 7H2O were dissolved in distilled water, then this solution was added little by little in polyethylene glycol and stirred for 1 h, then NaOH was added and stirred. for 10 min. Previously, in another container, the OG with 15 mL of water and 1 mL of hydrazine is placed in the ultrasound for 4 hours. Subsequently, both solutions are mixed and stirred for 10 min and then the solution is poured into an autoclave where the reaction will be carried out at a temperature of 200°C and for 24 hours. Finally, after this time, it is allowed to cool naturally, the material is washed with distilled water and methanol until a neutral pH is obtained, and dried at a temperature of 90°C (Cui et al., 2015; Lei et al., 2017).

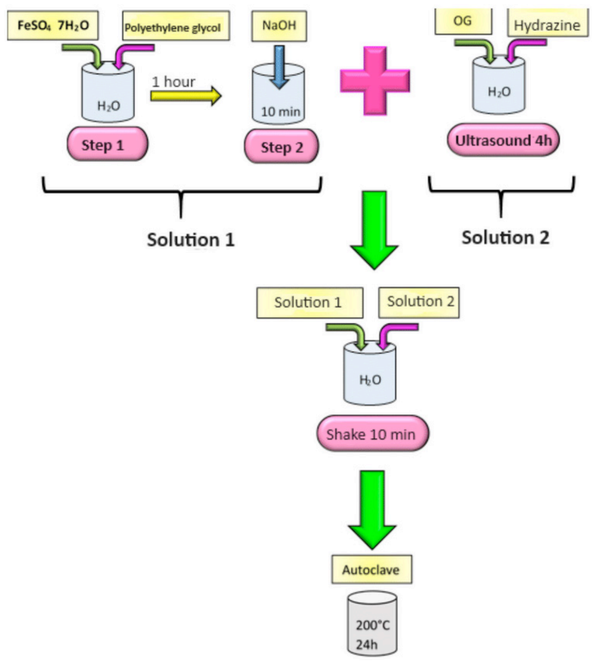

Figure 1. GMO synthesis methodology.

RESULTS

The material was characterized using the Scanning Electron Microscope (SEM) technique, as can be seen in Figure 1. Figure 1 (A) shows a micrograph of the graphene oxide, where a sharp texture can be observed at the edges. of the flakes, which is something very common in said material. In Figure 1 (B) and Figure 1 (C) you can see the magnetic OG, where the flakes cannot be seen due to the small spherical-looking particles agglomerated with sizes less than 1 μm on its surface. On the other hand, Figure 1 (D) shows the mapping of the material where it can be seen that Iron predominates the surface along with oxygen, which is indicative that the material is decorating the surface of the OG. Finally, the EDS analysis of the material indicates that iron (Fe) is present with 46.72% atomic content, oxygen (O) with 43.43% and carbon (C) with only 9.84%.

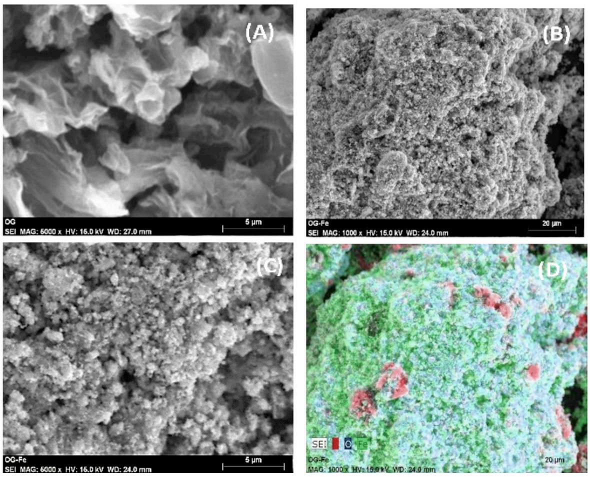

Figure 2. SEM micrographs. (A) OG; (B) Magnetic OG at 1000x; (C) Magnetic OG at 5000x; (D) Mapping of the magnetic OG at 1000x.

Iron oxide particles in other works also form agglomerates, these being smaller than 100 nm using the co-precipitation method (Wang et al., 2011) or sizes of 5µm (Mahdavi et al., 2021) where a system was used. reflux.

CONCLUSIONS

The recovery of GMOs from a medium presents a great advantage over OG, because it can be used in different environmental remediation methods, not only with easy recovery of the material, but also its functionalization. Likewise, the different chemical methods that are used for the synthesis of this type of material are methods that require a reducing chemical precursor that allows obtaining the desired function, added to this the use of procedures such as reflux or inert atmospheres, They can make the procedure much more difficult, which is why our synthesis method shows an advantage over this by not requiring special conditions to obtain the material.

REFERENCES

Basu, S., & Bhattacharyya, P. (2012). Recent developments on graphene and graphene oxide based solid state gas sensors. *Sensors and Actuators, B: Chemical*, *173*, 1–21. https://doi.org/10.1016/j.snb.2012.07.092

Chen, J., Yao, B., Li, C., & Shi, G. (2013). An improved Hummers method for eco-friendly synthesis of graphene oxide. *Carbon*, *64*(1), 225–229. https://doi.org/10.1016/j.carbon.2013.07.055

Cui, L., Wang, Y., Gao, L., Hu, L., Yan, L., Wei, Q., & Du, B. (2015). EDTA functionalized magnetic graphene oxide for removal of Pb(II), Hg(II) and Cu(II) in water treatment: Adsorption mechanism and separation property. *Chemical Engineering Journal*, *281*, 1–10. https://doi.org/10.1016/j.cej.2015.06.043

He, Y., Yi, C., Zhang, X., Zhao, W., & Yu, D. (2021). Magnetic graphene oxide: Synthesis approaches, physicochemical characteristics, and biomedical applications. *TrAC - Trends in Analytical Chemistry*, *136*. https://doi.org/10.1016/j.trac.2021.116191

Lai, K. C., Lee, L. Y., Hiew, B. Y. Z., Thangalazhy-Gopakumar, S., & Gan, S. (2019). Environmental application of three-dimensional graphene materials as adsorbents for dyes and heavy metals: Review on ice-templating method and adsorption mechanisms. *Journal of Environmental Sciences (China)*, *79*, 174–199. https://doi.org/10.1016/j.jes.2018.11.023

Lei, W., Liu, Y., Si, X., Xu, J., Du, W., Yang, J., Zhou, T., & Lin, J. (2017). Synthesis and magnetic properties of octahedral Fe3O4 via a one-pot hydrothermal route. *Physics Letters, Section A: General, Atomic and Solid State Physics*, *381*(4), 314–318. https://doi.org/10.1016/j.physleta.2016.09.018

Lingamdinne, L. P., Koduru, J. R., & Karri, R. R. (2019). A comprehensive review of applications of magnetic graphene oxide based nanocomposites for sustainable water purification. *Journal of Environmental Management*, *231*(July 2018), 622–634. https://doi.org/10.1016/j.jenvman.2018.10.063

Mahdavi, V., Taghadosi, F., Dashtestani, F., Bahadorikhalili, S., Farimani, M. M., Ma'mani, L., & Mousavi Khaneghah, A. (2021). Aminoguanidine modified magnetic graphene oxide as a robust nanoadsorbent for efficient removal and extraction of chlorpyrifos residue from water. *Journal of Environmental Chemical Engineering*, *9*(5), 106117. https://doi.org/10.1016/j.jece.2021.106117

Wang, C., Feng, C., Gao, Y., Ma, X., Wu, Q., & Wang, Z. (2011). Preparation of a graphene-based magnetic nanocomposite for the removal of an organic dye from aqueous solution. *Chemical Engineering Journal*, *173*(1), 92–97. https://doi.org/10.1016/j.cej.2011.07.041

Zhao, L., Guan, X., Yu, B., Ding, N., Liu, X., Ma, Q., Yang, S., Yilihamu, A., & Yang, S. T. (2019). Carboxylated graphene oxide-chitosan spheres immobilize Cu2+ in soil and reduce its bioaccumulation in wheat plants. *Environment International*, *133*(September), 105208. https://doi.org/10.1016/j.envint.2019.105208

Zhao, L., Yang, S. T., Yilihamu, A., & Wu, D. (2019). Advances in the applications of graphene adsorbents: From water treatment to soil remediation. *Reviews in Inorganic Chemistry*, *39*(1), 47–76. https://doi.org/10.1515/revic-2018-0020