Área: ____MAT____ **Nº de Inscrição: __1334__**

Adsorbent magnetic materials based on green coconut husks biochar and superparamagnetic iron oxides

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Key words: *Cocos nucifera, Magnetic nanoparticles, Nanocomposite, Activated carbon, Thermal decomposition.*

Highlights

High specific surface area mesoporous biochar was synthesized by slow pyrolysis using H_3PO_4.
The composite prepared using SPION supported on the biochar is easily separated from water using a magnet.

Abstract

The presence of anthropogenic bioactive substances in both surface water and groundwater is alarming. Antibiotics, anti-inflammatory and hormones have been found in rivers all over the world along with other concerning organic micropollutants. This is a reflex of inadequate discharge as well as the result of the limited capacity of the current treatment processes, and thus urge the study of new materials to be used to remediate this problem [1]. Activated carbon is one of the most cost-effective solutions being used to remediate those problems whereas more recently magnetic nanoparticles (MNPs) are being studied as an alternative solution for their ability to be magnetically retrieved from the suspension after the adsorption process. Composite materials have the advantages of summing the positive aspects of their different constituents while minimizing the negative aspects of each component. The materials proposed in this work intent to combine the excellent adsorption capacity of the biomass activated carbons (biochars) with the magnetic properties of the magnetic iron oxides [2].

The green coconut husks (GCH) were dried and impregnated with H_3PO_4 in a mass ratio of 1:3, then pyrolyzed at 500 °C. The obtained solid was washed until neutrality and characterized using a N_2 porosimeter. The nanoparticles were synthesized by thermal decomposition of iron (III) acetylacetonate using oleic acid as the surfactant for size control and oleylamine as the reducing agent. The compounds were dissolved in benzylic ether and heated until the reflux temperature under continuous flow of N_2 for 1 h, then cooled down and washed with ethanol. The product was characterized by powder XRD and TEM while the magnetic properties were studied using a SQUID magnetometer for DC measurement of the magnetization against the field and temperature. Different procedures for the deposition of the MNPs into the carbon were tested using the magnetization saturation (at 50000 Oe) as the comparison point, while varying the proportion between carbon and MNPs and the usage of ultrasound to promote the mass transfer from the solvent to the carbon surface.

On the selected methodology the MNPs coated with oleic acid were suspended in toluene with the aid of an ultrasound bath to obtain a stable colloid with a concentration of 3 mg mL^{-1}. The colloid was then mixed with the carbon for 90 min and the composite was magnetically separated and washed with ethanol. The XRD analysis confirmed the crystalline phase of magnetite/maghemite with a diameter around 11 nm as was envisaged. The size of the nanoparticles was also confirmed with TEM images that showed a medium diameter of 11.4 ± 2.2 nm (n = 300). The analysis of the biochar exhibited a BET surface area of 1249 $m^2\ g^{-1}$. The MNPs of around 11 nm were intended to fit the biochar pore distribution (2 – 50 nm) in a way that the particles fit inside the pores to promote the magnetic response of the composite while leaving enough specific surface available for the adsorption of pollutants. The DC magnetic measurement clarified that the MNPs are indeed superparamagnetic with a saturation of magnetization of 66 emu g^{-1}. The nanocomposite obtained had a saturation of magnetization around 11.8 ± 1.7 emu g^{-1}. The nanocomposite produced can be easily and quickly separated from water with a magnet, but the minimum field gradient required is yet to be optimized by future experiments.

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Acknowledgments

The authors thank the Centro Brasileiro de Pesquisas Físicas (CBPF) for the porosimetry measurements. This study was financed in part by the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior – Brasil (CAPES) – Finance Code 001. New Molecular Materials – UFF/PrInt/CAPES – Project number 88887.310269/2018-00.