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Evaluation of sugarcane bagasse use for the synthesis of reduced graphene oxide by using microwave process

L. Landini[1], S. G. Araujo[1], V. L. R. Salvador[1], R. H. L. Garcia[1], E. Galego[1], M. M. Serna[1], and H. M. S. M. D. Linhares[2]

[1] *llandini@ipen.br, Av. Prof. Lineu Prestes, 2242 – CEP 05508-000 - Sao Paulo/SP – Brazil*
[2] *horaciolinhares@id.uff.br, Av. João Jasbick, s/nº - CEP 28470-000 - Santo Antônio de Pádua/RJ - Brazil*

1. Introduction

Biomass residues from agro-waste have potential in the production of graphene, as they are sustainable, renewable and abundant sources. Graphene-derived materials such as graphene oxide (GO) and reduced graphene oxide (RGO) have gained prominence in recent years due to their exceptional electronic, thermal and mechanical properties. Normally, they are synthesized from graphite, but biomass (vegetable or animal organic matter), such as agro-residues (orange peel, coconut shell, sugar cane bagasse, jackfruit, among others), also has been used as a carbon source. The method of producing these materials from graphite is complex, high-cost and involves treatment with chemicals that are harmful to the environment and human health. The RGO is commonly derived from the reduction of GO, which is formed when graphite is oxidized (generally used Hummers method and its modifications). Consequently, more environmentally friendly methods have been explored in order to minimize all these impacts, such as microwave technology (MW) associated with agro-waste, for the synthesis and reduction of graphene oxide. Agro-waste represents a major challenge in terms of its recovery, safe disposal and potential use as environmentally sustainable energy and microwave provide faster and more selective heating in certain processes and materials.

The RGO, in particular, has several applications, including as a membrane filter for purifying wastewater, which is normally contaminated with organic and inorganic pollutants, such as toxic metals, dyes, among others. This has stimulated the development of new technologies, aiming to improve the cost/benefit of material production.

In literature, the processing of graphite oxide (with low cost), exfoliated by microwave, for the production of high-quality and highly efficient graphene is cited, as well as advances in the microwave-assisted production of reduced graphene oxide [1-8].

Therefore, the objective of this work was to process sugarcane bagasse with microwave technology, for the synthesis of RGO and subsequent application in water purification.

2. Methodology

The agro-waste used in this work was sugarcane bagasse colected from the local fair at University of Sao Paulo (USP), associated with a reducing agent containing iron. The bagasse was dried in the sun, crushed into powder and separated into a particle size of 140mesh.

The experiments were carried out in the batch reaction unit, with microwave, installed at the Institute for Energy and Nuclear Research (IPEN) [9], which allows online monitoring of process variables, such as temperature (ºC), pressure (bar), microwave power (W), among other information, which is available on dedicated screens, with histories of the main occurrences and trend graphs. The mass proportions used in processing the samples, of reducing agent and agro-residue, were 1:5, under stirring at 600 rotations per minute (rpm), with microwave of 2.45GHz frequency and power of 1kW, reaction start temperature 300ºC and reaction time up to 20min.
The initial samples and the products obtained were collected at room temperature and sent for analysis by X-ray Fluorescence (XRF) and X-ray Diffraction (XRD).

3. Results and Discussion

In the Figure 1, it can be seen: sugarcane bagasse as collected at the fair, after crushing (140mesh) and after microwave processing, where the black color of the final sample can be observed, which was expected.

Figure 1: Sugarcane bagasse samples, crushed. after crushing (140mesh) and after microwave processing.

In the experiments, the samples were heated to a temperature of 300ºC to 500ºC during 20 minutes and then the equipment was turned off and the samples were cooled naturally to room temperature.

The experiments carried out with these samples, in the microwave batch reaction unit, were quite reproductive, indicating that the equipment is reliable. The results obtained by XRF of the initial sample indicated the presence of a large percentage of organic material (CHNO), 97.5%, and a small amount of inorganic matter (0.77% K, 0.49% Ca, 0.43% Si, 0.19% S, 0.01% Fe and 0.02% P). This caused contaminants to be obtained in the final samples of 12.74% K_2O, 3.55% P_2O_5, 1.28% SO_3, 6.39% SiO_2, 3.69% CaO, 71.87% Fe_2O_3. This high percentage of the last component was possibly obtained due to the reducing agent used. In the XRD curves (Fig.2), a very high background (BG) was observed, in addition to the presence of a 2θ peak around 25º, no phase containing iron and other peaks above 35º were identified, which were also not recognized. Anyone 2θ peak was identified at around 10.8º, similar to GO, demonstrating the direct formation of RGO (compared to a literature value close to 2θ=26.5º). However, it is still necessary to confirm these results with other types of analyzes such as Raman Spectroscopy, with which it is possible to identify the structure of graphene and its derivatives [10].

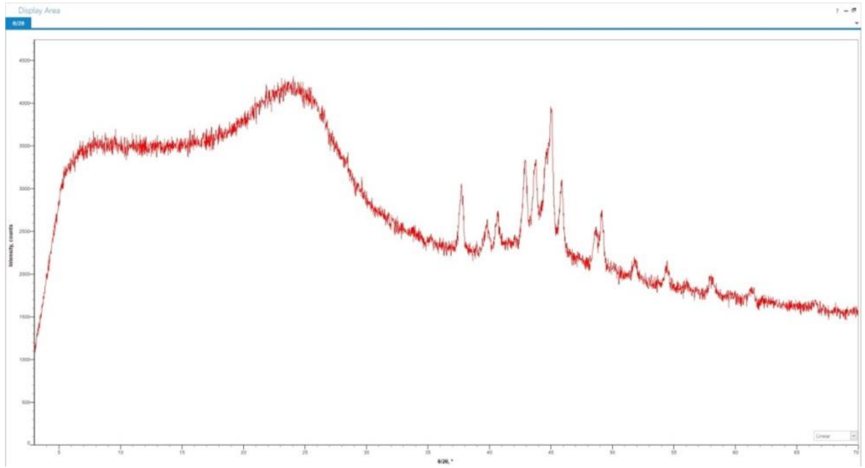

Figure 2: The XRD curve, where a 2Θ peak is observed at around 25º (RGO), when compared with a literature value close to 2Θ=26.5º [10].

4. Conclusions

The processing of sugarcane bagasse samples (140mesh), in the presence of a reducing agent with iron, in a ratio of 5:1, at a power of 1kW and frequency of 2.45GHz, in the microwave batch reaction unit (IPEN), was carried out under agitation at 600 rpm. The samples showed good temperature tolerance up to 500°C and without injection of any type of gas. The repetitions were quite reproductive, with low reflection rates (<10%) of these electromagnetic waves, indicating good absorption by the samples. This good performance of the equipment and the response of this type of sample raised great expectations of using these electromagnetic waves in these tests with sugarcane bagasse samples. All the variables studied affected in some way, even if discreetly, the carbonization and/or graphitization processes, especially when applied to microwave. The first process consists of increasing the carbon content by removing other elements through heat treatment and the second of organizing the carbon structures to produce a graphitic-type structure. Even so, more tests still need to be carried out to adjust process variables.

All results obtained until now, for sugarcane bagasse samples processed in this unit, still need to be better investigated, through other types of characterization, but the results of XRD analyzes showed the 2Θ peak around 25º for RGO and absence of 2Θ at around 10.8º, similar to GO. This condition was demonstrated by the direct formation of RGO (close to literature). However, it is still necessary to confirm these results with other types of analyzes such as Raman Spectroscopy, that allows to identify the structure of graphene and its derivatives. These values found are considered promising and if they are confirmed, filter assemblies will be started for water purification tests, with methylene blue and metallic ions.

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