

## A Method for Graphite Exfoliation by a Mechanical-Chemical Route

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Graphite is the most stable allotropic form of carbon, which consists of several layers of graphenes joined with covalent bonds; these layers are linked together by a weak Van Der Waals interaction [1]. Due to the weakness of these interactions, it is possible to insert ions, atoms or molecules between the layers. In the exfoliation process, vaporization of the intercalated species leads to a significant expansion up to hundreds of times along the c-axis, forming a highly porous worm-like material. Exfoliated graphite (EG) has interesting properties for various applications because its high thermal stability, excellent electronic conductivity and oxidation resistance [2]. A potential application of the EG is as adsorbent of heavy oil, DDT and some dyes [1]. This is due its high porosity, weak polarity, hydrophobic nature and high selective sorption capacity to large organic compounds. EG has been synthesized by galvanic, chemical and thermal treatments on the natural graphite. However, the chemical method is widely used because its simplicity and versatility.

In the present work, an EG was prepared using an alternative mechanical and chemical route. Pure aluminum (Al) and graphite (C) powders were used as raw materials. The mixture (1:1 in wt.) was milled in a high-energy mill (Spex) for 1, 5 and 13.5h periods. The milled samples were then leached with an HCl solution (1:1 in vol.) in a ratio of 100ml/1g, washed with distilled water and dried at room temperature overnight. Surface analysis was carried out by measuring the BET surface area. Morphological and chemical studies were performed with a scanning electron microscope (JSM5800-LV) and a high resolution transmission electron microscope (JEM-2200FS).

Fig.1 shows SEM micrographs of samples milled (1h) before and after the leaching process. Bright zones in Fig. 1a correspond to a high metal concentration. After the leaching process, in Fig. 1b, Al has been completely removed obtaining highly dispersed graphite. This was proved by EDS.

Experimental results of adsorption-desorption isotherms showed that EG adsorption properties depend of pore characteristics and specific surface area ( $S_{sp}$ ), both derived from exfoliation conditions. In Fig. 2 (inner table) it is evident an accentuated  $S_{sp}$  increase in the samples after leaching process as a function of milling intensity.

Common  $S_{sp}$  values reported in the literature extended over 15 [3], 44 [1] to 70 m<sup>2</sup>/g [4]. However, as result of complete exfoliation process, the experimental value reported in this work of 548 m<sup>2</sup>/g demonstrates the destruction of the worm-like structure characteristic of EG [5]. In Fig. 3A it is observed a significant slicing between graphite layers; this displacement introduces additional surface area.

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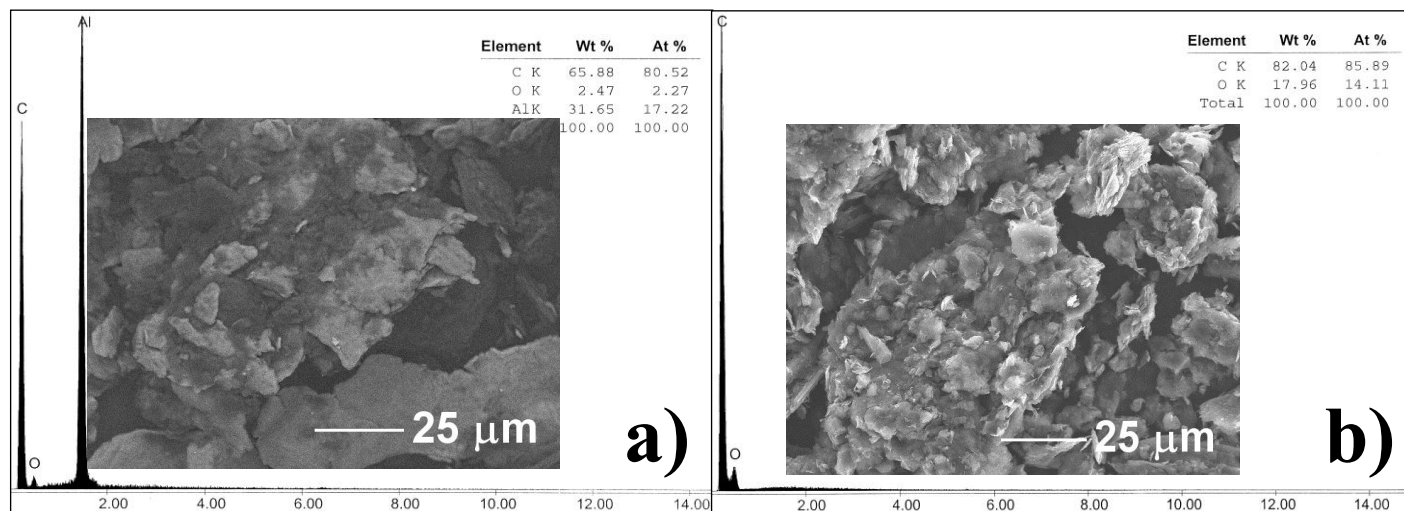


FIG. 1. SEM micrographs and their corresponding EDS analysis. a) Milled graphite, and b) Milled and leached graphite samples. The presence of oxygen corresponds to components of the adhesive tape, which was used to stick the powders during the SEM studies.

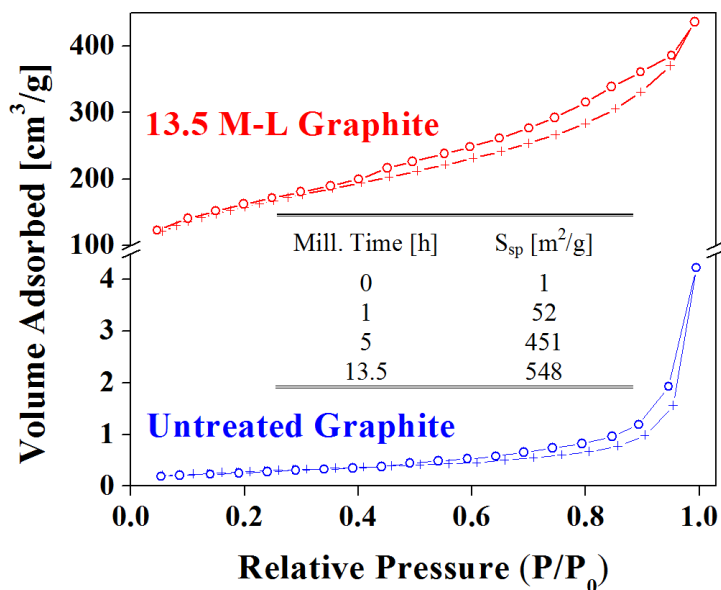


FIG. 2. Isotherm curves in N<sub>2</sub> at 77K. Untreated - graphite curve is classified into a II type, suggesting the existence of macropores. The second one consists in I-IV types with an important content of micropores.

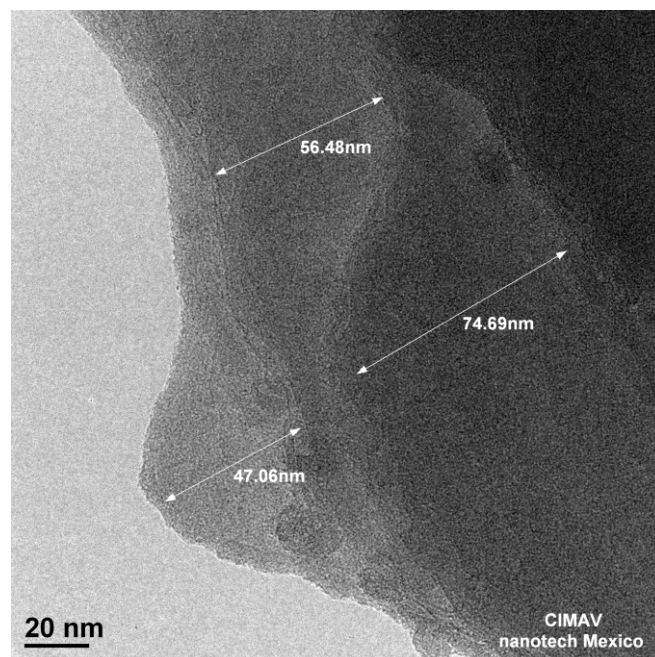


FIG. 3. HRTEM image from 1h EG sample after the leaching process. Notice the layer separation of a few tens of nanometers between layers and from the border of the particle.