

High Purity Graphene Prepared Via a Cheap Method of Synthesis from a CO₂ Atmosphere

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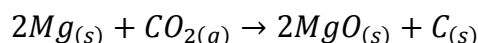
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The study of graphene has still been a deep field of study due to its properties and applications in several scientific works [1]. The synthesis method using carbon dioxide as a precursor, which is known as dry ice in flames method, was developed by Chakrabarti and co-workers [2]. The method consists of igniting magnesium metal in a carbon dioxide (CO₂) atmosphere using a block of dry ice. This results in a highly exothermic reaction, reaching a temperature above 2500 °C, enough to perform the following chemical reaction [3]:



The purification method used in this work consists of a chemical dissolution of the MgO and C with aqua regia (HNO₃ + 3HCl), forming magnesium chloride (MgCl₂), which is soluble in water, and its elimination by washing with demineralized water until a neutral pH is reached, then product is dried and processed 30 minutes in high energy ball milling then the product was submitted to a second washing process to eliminate the MgO remaining the C. A general scheme of the synthesis processes used to obtain the graphene and magnesium oxide is presented in figure 1.

The results of the purification process were analyzed by the characterization techniques of transmission electron microscopy (TEM), thermogravimetric analysis (TGA) and X-ray photoelectron spectroscopy (XPS).

TEM image in figure 2 shows graphene layers obtained after the synthesis and cleaning process, this image shows clear evidence of graphene sheets, where the van der Waals interlayer attractions allowed the nanosheets to slide each other perpendicularly to the c-axis. Still, enough attraction prevents the complete formation of individual graphene monolayers. The results of TGA in figure 3 shows that using aqua regia have a good efficiency in eliminating the MgO particles with a residue of 0.9% wt. XPS analysis shown in figure 4, its appreciable the low percentage of the content of MgO remaining in the graphene, leading this to a high purity of graphene. The remnant of MgO is probably due to MgO trapped within the carbon matrix.

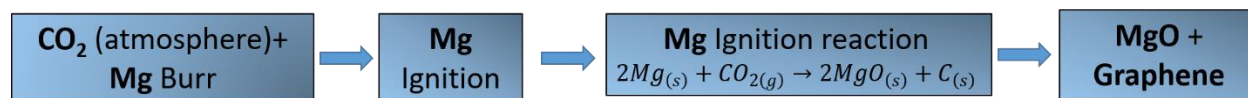


Figure 1. Descriptive diagram of the synthesis route to obtain graphene.

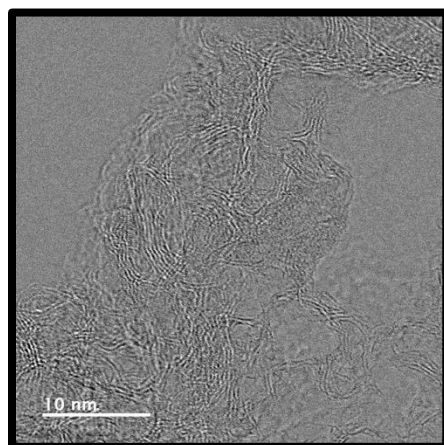


Figure 2. TEM image of graphene layers.

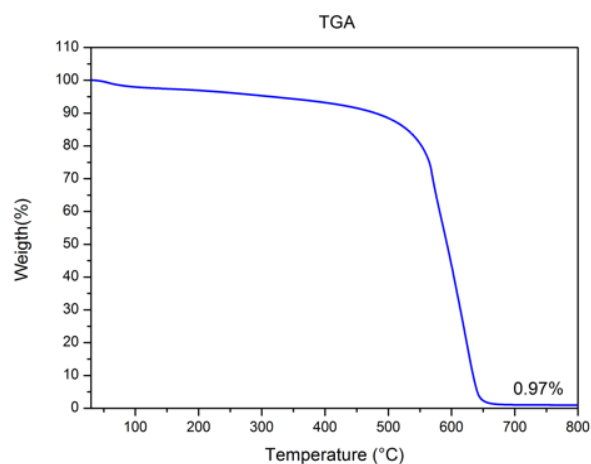


Figure 3. TGA analysis of in obtained graphene.

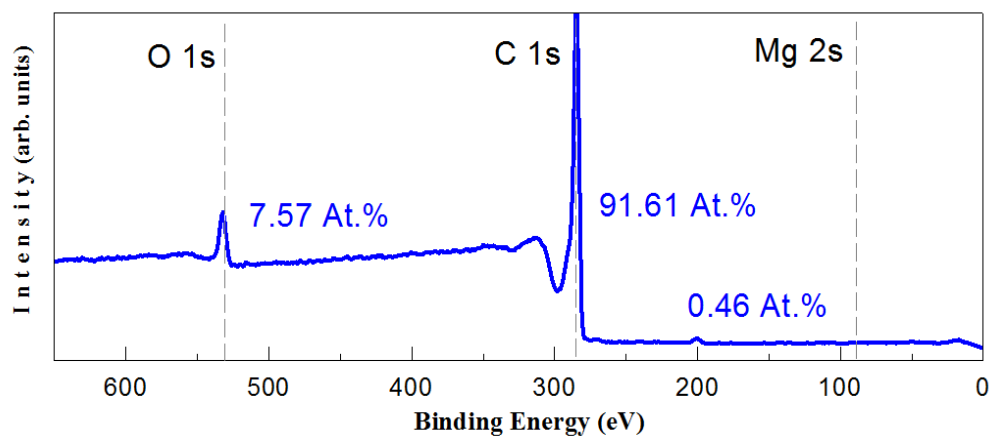


Figure 4. XPS analysis of MgO content in obtained graphene.

References:

- [1] Novoselov, K.S., et al., A roadmap for graphene. *Nature*, 2012. 490(7419): p. 192-200.
- [2] Chakrabarti, A., et al., Conversion of carbon dioxide to few-layer graphene. *Journal of Materials Chemistry*, 2011. 21(26): p. 9491-9493.
- [3] Zhang, J., et al., Synthesis of graphene from dry ice in flames and its application in supercapacitors. *Chemical Physics Letters*, 2014. 591: p. 78-81.