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*Abstract*

# Sustainable Synthesis of Carbon–Clay Nanocomposites †

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Carbon nanostructures, such as graphene derivatives, have remarkable mechanical properties, electrical conductivity, and high specific surface areas. These nanomaterials are attractive for diverse areas of applications, being widely used in composite materials [1]. Nevertheless, the production of carbon nanostructures commonly employs toxic chemicals and harsh methodologies, which is a drawback for their application in the food, biology, or biomedical fields. Therefore, new eco-friendlier and sustainable approaches for the development of carbon nanostructures should be addressed [2].

Herein, sustainable carbon–clay nanocomposites were prepared using saccharose as a carbon source and sepiolite clay as porous support. Different synthesis strategies were evaluated to reduce the time and energy consumption. Conventional pyrolysis (P) at 200 °C or 500 °C during 2 h under $N_2$ was compared to microwave pyrolysis (MW) in the same conditions, but the time of treatment was reduced to 20 min. The combination of each method followed by hydrothermal carbonization (HTC) at 230 °C for 24 h (P-HTC and MW-HTC) was also studied. The structures of the carbon–clay nanocomposites obtained were characterized using Raman spectroscopy, Fourier transform infrared spectroscopy (FTIR), and X-ray diffraction (XRD). The textural properties were studied using −196 °C $N_2$ adsorption–desorption isotherms. The graphitization of saccharose was confirmed by the development of D and G bands on Raman and the disappearance of O–H and C–H vibrations on FTIR spectra. The results suggest that the HTC step is fundamental to achieve successful graphitization using both techniques at 200 °C. Microwave pyrolysis at 500 °C allowed the preparation of carbon–clay nanocomposites in only 20 min, saving 100 min in comparison with the P500 method. XRD confirmed the maintenance of the sepiolite crystalline structure after all the mild temperature treatments. The textural analysis showed that MW200 and P200 treatments resulted in non-porous materials. However, the materials obtained using MW200-HTC and P200-HTC showed porosity, which was attributed to improved graphitization, confirmed by elemental analysis. These samples showed type III isotherms, attributed to macroporous materials, with a maximum specific surface area ($S_{BET}$) of 159 $m^2\ g^{-1}$ (P200-HTC). The samples produced at 500 °C showed type IV isotherms, assigned to mesoporous powders, with a maximum $S_{BET}$ of 263 $m^2\ g^{-1}$ (P500-HTC). The carbon–clay nanocomposites were successfully prepared using natural precursors and mild temperatures, and time was saved by using microwave pyrolysis, which highlights their sustainable nature. Carbon–clay nanocomposites have potential to be applied as fillers in functional composite materials.

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