Synthesis of graphene oxide according to the modified Hummers method (Tour method)

ABSTRACT

In the present thesis, the laboratory synthesis of graphene oxide from graphite grains was studied using the modified Hummers method (Tour Method).

In the first stage of the work, extensive bibliographic research was carried out in order to arrive at the method by which the synthesis of graphene will take place. The methods found from the literature were mechanical graphite exfoliation, electrochemical exfoliation and chemical vapor deposition. The research continued and the chemical peeling method was found to be suitable as it had a high yield of products. The chemical method includes the step of removing the graphene layers within the graphite and the step of intercalating compounds and oxidizing the graphene. In this way, the graphite is exfoliated and monolayer or multilayer graphene is produced.

One of the widely used methods for chemical peeling is the Hummers method. This is a method that has been used for over sixty years and has undergone many modifications as its original formulation contained chemical reactions that could cause an explosion and toxic fumes. Other chemical exfoliation methods found are the oxidative chlorophosphorylation of graphite (OxCh) with subsequent hydrolysis of the obtained chlorophosphonated graphite (ChPG) and the reaction of graphene nanoplatelets with aminophenolic alcohol.

The method by which the synthesis of graphene oxide was carried out in the present work is a modified Hummers method which was applied by Tour and his group and will henceforth be referred to as the Tour method. Pure graphite was used in the synthesis of graphene oxide. The reagents involved in the oxidation reaction of graphite are sulfuric acid, phosphoric acid and potassium permanganate. While for the end of the reaction, distilled water and hydrogen peroxide were added. Finally, the final material was washed and cleaned with a mixture of hydrochloric acid, ethanol and distilled water.

The samples of the resulting products were subjected to four types of analysis. In particular, FTIR-ATR spectroscopy was carried out as an indication of the existence of the different oxygen functional groups, RAMAN spectroscopy to determine the quality of the GO sheets as well as scanning electron microscopy (SEM) confirming the surface morphology and the structures created during the irradiation of the of samples. After all, TGA was conducted to investigate the thermal stability of the prepared GO during its decomposition at high temperatures as it relates to all its chemical and physical structures.

The final material from the modified Hummers method will be used as a reinforcing phase in a bio-based epoxy matrix. In order to develop samples of nanocomposite materials whose mechanical properties will be examined.

Keywords :

Graphite, Graphene Oxide Synthesis, Modified Hummers Method (Tour Method), FTIR-ATR Spectroscopy, RAMAN Spectroscopy, Scanning Electron Microscopy SEM, Thermogravimetric Analysis TGA