

INFLUENCE OF CHEMICAL REACTIONS OVER THE FORMATION OF GRAPHENE OXIDE NANOPARTICLES

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ABSTRACT

Graphene oxide nanoparticles were synthesized and characterized using X-ray diffraction, Scanning electron microscopy and FTIR. XRD confirms the formation of Graphene oxide and Graphite and is also evident from SEM images. FTIR Spectroscopy confirms the presence of various oxygen functional groups.

Keywords: Graphene oxide, Ethanol, Pottassium permanganate, SEM.

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INTRODUCTION

Global development raises new challenges, especially in the field of Nanoscience. A material – extraordinary is therefore the need of the hour to cope with the growing scientific demands. Here is Graphene oxide (GO). It attracts enormous attention for its variety of applications in the field of solar cells, nanoelectronics, nanocomposites, super capacitor, fuel cells, sensors, drug delivery systems, antimicrobial activity, etc.¹. Apart from the various physical methods, the chemical processes are widely used to prepare Graphene oxide because of its cost effective preparation². But the stability of the graphene oxide depends only on the chemical reactions that take place during the time of synthesis³. In GO each carbon layer is separated with oxygen molecules which not only expand the layer separation, but also enable the layers to become hydrophilic (meaning that they can be dissolved in water, much like sugar or salt). This property enables GO to be exfoliated in water using sonication, ultimately producing single or few layer graphene oxide. The acid treatment is needed to exfoliate Graphite flakes⁴. The exfoliated graphite is oxidized properly using oxidizing agent⁵⁻⁷. In the present work the chemical reaction between Graphite flakes, acid and oxidizing agents is briefly discussed.

EXPERIMENTAL

Materials

Graphite flakes, KMnO₄, H₂SO₄ and H₃PO₄, were purchased from Sigma Aldrich. The other reagents used for washing purposes were of 99.9% purity. A dilute Hydrochloric acid is prepared by adding distilled water. Wattman filter paper of less than 2µm in pore size is used for filtration purposes.

Synthesis of Graphene Oxide

Graphene oxide is synthesized through chemical route. GO is prepared by mixing 180ml of concentrated H₂SO₄ and 20ml of concentrated H₃PO₄. This solution is poured into the mixture of 1.5g of graphite powder and 9.0g of potassium permanganate and stirred for 12hrs using magnetic stirrer. It is then allowed to cool to room temperature for 9hrs. 1.5ml of H₂O₂ is also poured into it. This mixture is filtered and the filtrate is centrifuged (4000rpm for 4hrs) to receive the precipitate and the settled down solid material. The obtained precipitate is drained away and the remaining solid material is collected. Again this filtrate is washed thrice separately using 200ml of - deionized water, Hydrochloric acid and Ethanol.

After each wash the filtrate is separated from the filter and is centrifuged (4000rpm for 4hrs). The supernatant thus collected is poured away. After the multiple wash the remaining solid material is allowed to dry in vacuum.

Characterization methods

X-Ray Diffraction (XRD) was carried out to study the various phases of the chemically synthesized GO. Scanning Electron Microscopy is used to obtain the clear images of the particles. The GO powder samples for this analysis can be obtained by drying the GO suspension under vacuum condition. FT-IR spectra were recorded using IFS Bruker 66 V Spectrophotometer using KBr pellet technique.

RESULTS AND DISCUSSION

Graphene oxide particles are prepared from Graphite flakes, Con. Sulphuric acid, orthophosphoric acid with potassium permanganate as the oxidizing agent. Here we get Graphene oxide nanoparticles with some impurities as a result of oxidation. The concentrated sulphuric acid used here is to exfoliate the number of layers from the Graphite flakes, when the oxidizing agent KMnO_4 is added the exfoliated Graphite layers get oxidized. The addition of KMnO_4 as a process must be slow enough to obtain a homogeneous suspension liquid. Effervescence occurs due to bubbling of gases including oxygen. At the time of oxidation there is an increase in temperature. This leads to a decrease in the degree of oxidation and due to this may be only a few exfoliated layers get oxidized. This reaction results in the agglomeration of particles. The degree of effervescence increases with increase in the quantity of water added. This indicates the loss of oxygen and therefore there is a decrease in GO yield. On the other hand the yield increases by increasing the ratio of KMnO_4 .

X-ray diffraction was taken to confirm the formation of various phases as shown in figure-1. The formation of GO was confirmed by the peak at 10.5° . The diffraction peaks at 22.3° & 26.7° indicates the presence of Carbolite and Graphite respectively⁸. Thus the transformation of GO is not a complete one. So, only a small amount of GO is detected.

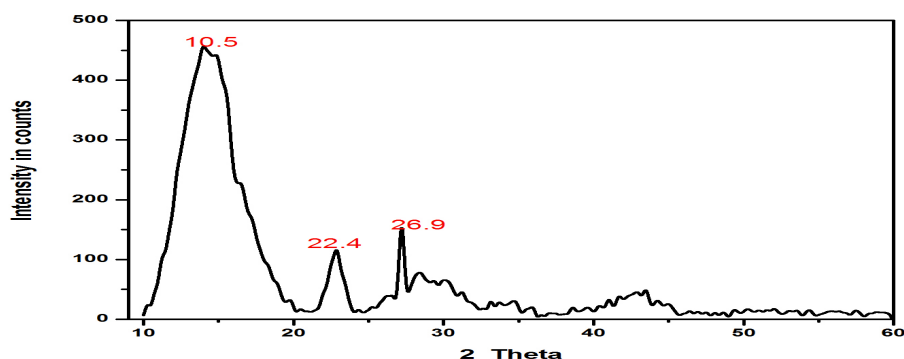


Fig.-1

The samples are examined using the Scanning electron microscope. In figure-2 the particles are aggregated. This may be due to the large specific surface area, surface energy and incomplete oxidation. FTIR spectrum in figure-3 provides the evidence for the presence of different types of oxygen functionalities in incomplete transformation of GO. The peak 3413cm^{-1} shows the OH vibrations. This is due to the presence of water molecules. The peaks such as 2360cm^{-1} , 2338cm^{-1} , 2074cm^{-1} were due to the stretching vibration ($\text{C}=\text{C}=\text{O}$) and the peaks 1646cm^{-1} , 1053cm^{-1} and 671cm^{-1} reveal the stretching vibration of $\text{C}=\text{C}$, $\text{C}-\text{O}$ and $\text{C}-\text{H}$ ⁹. The presence of multiple oxygen functionalities reveals the complex non-stoichiometric property of GO.

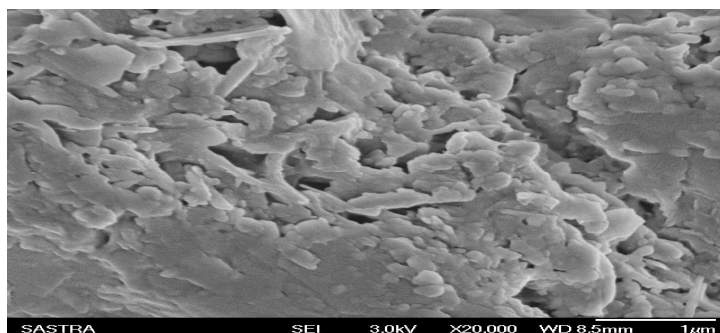


Fig.-2

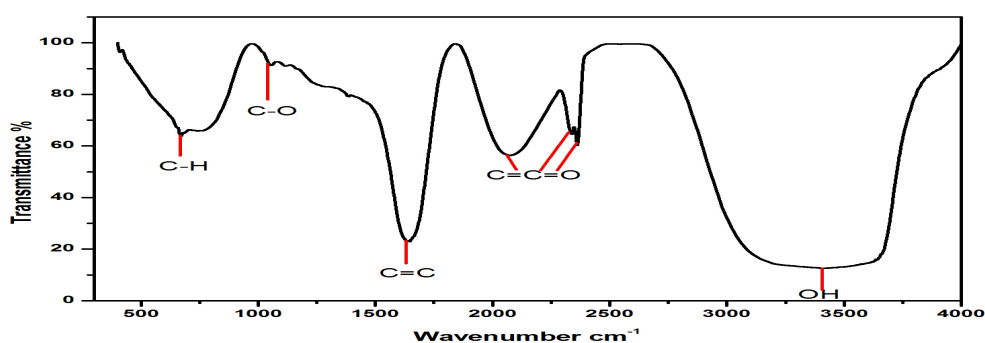


Fig.-3

CONCLUSION

We have studied the effects of the chemical reaction between the Graphite flakes and the oxidizing agents. The XRD image shows that the formation of GO and Graphite in various angles and it reveals that the chemical reaction plays an important role in the formation of the GO particles. This is again confirmed by images of scanning electron microscopy where the particles were aggregated due to the oxidation reaction. This is confirmed by the FTIR spectra where the presence of various oxygen functional groups is identified.

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