

Article

Graphene oxide synthesis by facile method and its characterization

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Abstract: The graphene and graphene oxide are latest and advanced materials with wide applications in environment, medical applications, industries, defense applications. We have synthesized graphene oxide from graphite flakes by modifying Hummer method in which we used NaNO_2 instead of NaNO_3 . Then we characterized our samples with X-Ray Diffractometry (XRD), Scanning-Electron Microscopy (SEM) and Fourier-Transform Infrared-Spectroscopy (FT-IR). These results confirmed the formation of graphene oxide also through this process. This graphene oxide can further be used for future applications in wastewater treatment and biomedical applications.

Keywords: Graphene oxide, nanochemistry.

1. Introduction

The importance of graphene and its composites is increasing due to their enlarged applications in the solar cells, catalysis and hydrogen storage. They are used in sensors, nanoelectronics and nanocomposites [1–5]. Graphene is the single layer of graphite. All the carbon atoms of graphene are sp^2 hybridized. They are arranged in a honey comb lattice like structure. Graphene is carbon allotrope. It has hexagonal atomic structure and therefore hexagonal nanoparticles are easily adjusted in their layers. Their hydrogels have crosslinked nanosheet structure [6]. The nanocomposite of TiO_2 and graphene is exhibiting large surface area, higher activity and the N_2 selection and at low temperature high resistance towards water and sulfur dioxide and also, high redox activity by which the catalytic reaction is favored [7]. Several studies have shown that metal-metal oxide-graphene have triple junction structure formation. They act like the electro-catalyst for the oxygen reduction for the applications in the PEM fuel cell. The tests performed showed that the durability of Pt became better than Pt on graphene sheets but also from Pt electro-catalysts which are supported on Carbon material, for example; Carbon nano-tubes [8].

Aijun *et al.* have worked on graphene and $g - \text{C}_3\text{N}_4$ interface by the state of art of the hybrid functional DFT procedure and incorporated the long range dispersion correction. They for first time revealed that electronic coupling is strong at graphene and $g - \text{C}_3\text{N}_4$ interface [9]. Xuefeng *et al.* reviewed the most recent research on the graphene materials and their antimicrobial activities. They also discussed the physiochemical properties of graphene materials, their experimental surrounding and selected micro-organisms and the interaction between them to further explore the controversial antimicrobial properties [10]. Wang *et al.* prepared metal oxide and graphene nanocomposite. They studied the properties of various metal oxide and graphene nanocomposite for the super capacitors and also for electrochemical catalysis [11]. Vats *et al.* synthesized nanocomposite of pristine-graphene with the palladium. They used swollen liquids crystal as the soft template. They observed that the catalytic activity of the nanocomposite is better than the Pd-RGO nanocomposite in one of the hydrogenation reaction of nitro-phenol and microwave assisted carbon-carbon coupling reaction which was sixteen times higher [12]. Kian *et al.* studied the graphene and the molecules like graphene for their role in the solar cells [13]. Mousavi *et al.* prepared PV-RGO and used as anodic battery material. That composite showed high areal, volumetric and the current density [14].

Meng *et al.* synthesized nanocomposite of silver nanoparticles decorated with graphene by chemical reduction process with assistance by supercritical CO₂ (ScCO₂). They studied tribological properties of the nanocomposite. It showed the high lubricating function of the graphene and the roughness of surface of the sliding ball is reduced and prevents direct interaction [15]. Tengfei *et al.* worked on the synthesis of nanocomposite of iron oxide and silver nanoparticles with graphene. They compared the Ag nanoparticles with this nanocomposite. They found that the nanocomposite was showing enhanced antibacterial activity towards the Gram negative bacteria, E. coli and the Gram positive bacteria which is S. aureus [16]. Li *et al.* synthesized graphene sheet, polymeric carbon-nitride nanocomposite which work as metal free catalyst for activation of oxygen for the selective oxidation of the sec. carbon-hydrogen bonds of saturated hydrocarbons like cyclohexane. It was observed to be the most stable catalyst and having high chemoselectivity for the sec. C-H bond of different saturated alkane [17]. Liwen *et al.* synthesized a nanocomposite of Graphene Oxide and Sulfur for the immobilization of sulfur in cathodic material of Li-S cells [18]. Deepak *et al.* synthesized Graphene Oxide and studied its antibacterial activities [19]. Zheng *et al.* prepared reduced graphene oxide by reducing graphene oxide by help of a reducing agent (caffeic acid) which was having high carbon-oxygen ratio i.e. (??) which is best reduced graphene oxide prepared from green reducing reagent [20]. Hongmei *et al.* used solvothermal method and developed magnetic-reduced graphene oxide (MRGO) nanocomposite. The nanocomposite was having high removal efficiency. It was observed that the M-RGO composite is effective adsorbent used for the removal of dyes pollutant [21]. Myungwoo *et al.* reviewed the graphene oxide synthesis which was developed through chemical vapor deposition method and studied its application [22]. Zhang *et al.* developed reduced graphene oxide and NiO nanocomposite which was used for the absorption of the chromium ion (Cr (VI)). The composite showed maximum adsorption capacity of chromium ion at $PH = 4$ and $T = 25^{\circ}C$ which was higher than any other reported so far [23]. Marcano *et al.* prepared graphene oxide by modification in the Hummer method as they excluded NaNO₃. They increased KMnO₄ and used H₃PO₄ and H₂SO₄ mixture in the ratio of 1:9 which showed improvement in the oxidation process of graphene [24].

2. Method and materials

We took 2g of graphite and NaNO₂ and mixed in about 100mL of H₂SO₄ (conc.) in 1000mL volumetric flask and put in ice-bath. The mixture was continuously stirred on hot plate for about 1 hour and then added 6g of KMnO₄ slowly. Then removed ice bath and kept the mixture under stirring for 2 days. About 90mL of distilled water was added to this and brown color appeared. The solution was stirred continuously. Then 15mL of hydrogen peroxide was added to this solution and yellow color appeared. We washed this mixture with 10% HCl. Then centrifuged at 5000rpm for 5 minute. This filtrate was decanted. The remaining (graphene oxide) was dried at 110⁰C and then calcined for 3 hours at 550⁰C in muffle furnace. The graphene oxide thus obtained was grind and characterized for further analysis.

3. Results and discussions

For the characterization of prepared nanoparticles XRD is one of the best technique. It characterizes the purity and phase of the nanomaterial. XRD gives detail of diffraction angle, the interlayer spacing and mainly the crystallite size. The XRD used in our work was Bruker D8 advance. Figure 1 shows XRD pattern of graphite used in this work. Figure 2 is showing wide-angle XRD peak of our prepared graphene oxide. In graphene oxide XRD spectra, the major peak at 11.6⁰ and two weak peaks at $2\theta = 43^{\circ}$ and $2\theta = 45^{\circ}$ clearly describe the graphite structure [22,25]. We characterized the graphene oxide by the Field-emission scanning-electron microscopy (FESEM) for their surface morphology. Figure 3 shows FESEM image of graphene oxide at different magnifications. It is clear from the FESEM image of GO that their layers are smooth in appearance [25]. The prepared graphene oxide was characterized by FT-IR for functional-groups checking. Figure 4 show the FT-IR spectra of the graphene oxide. The major peak at about 3500 cm⁻¹ shows the presence of hydroxyl (OH) groups [25]. Some peaks were also observed in fingerprint region at 750cm⁻¹ - 600cm⁻¹ which may be the bending vibrations due to C-O, and C-O-C bending vibrations.

4. Conclusion

In summary, we synthesized Graphene Oxide from graphite powder by using NaNO₂ instead of NaNO₃. The resultant product obtained was characterized through different analytical techniques like, XRD, FESEM,

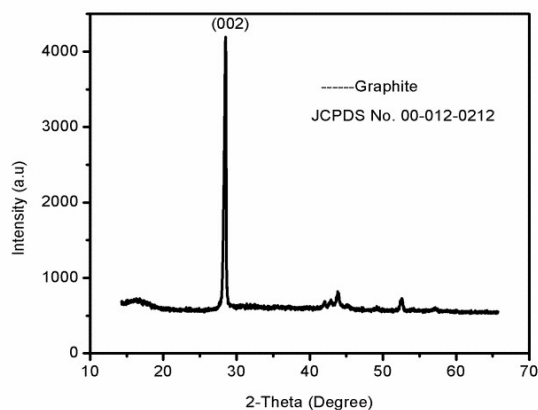


Figure 1. XRD pattern of graphite

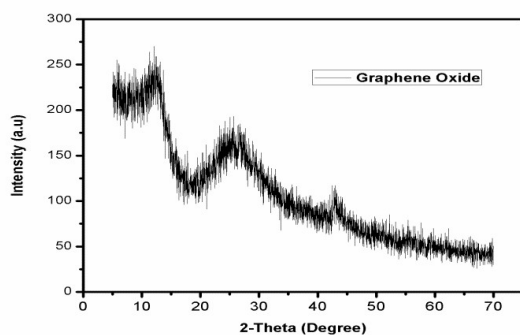


Figure 2. XRD pattern of graphene oxide

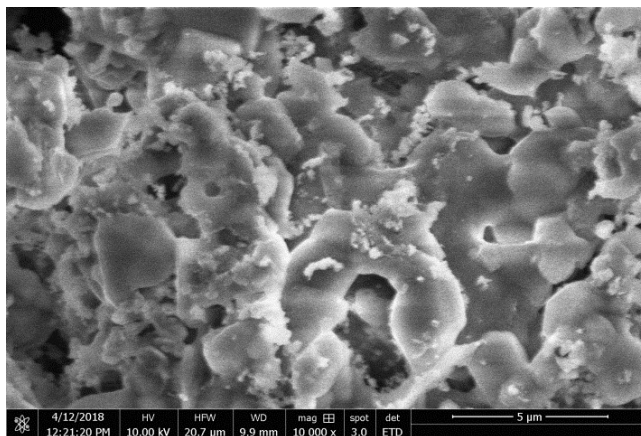


Figure 3. SEM image of Graphene Oxide

FT-IR. The obtained result was matched with literature and this was confirmed that the graphene oxide has formed. The oxidation of graphene can be increased by using NaNO_2 . This can be used for different purposes specially as adsorbant of heavy metals.

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Conflicts of Interest: "The authors declare no conflict of interest."

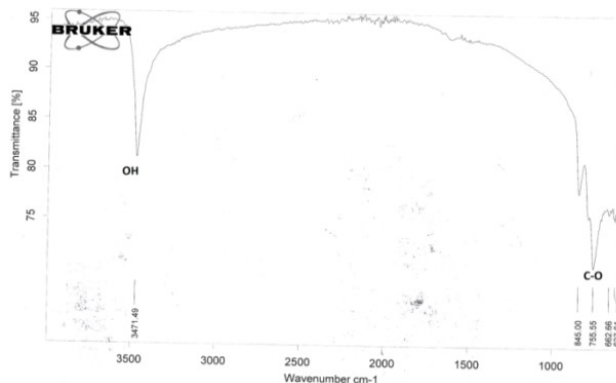


Figure 4. FT-IR spectra of Graphene oxide

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