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Synthesis of Graphene Oxide Via Microwave Assisted Eco-Friendly Method Versus Modified Hummer Method

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Abstract: *Using eco-friendly chemicals, a unique method for synthesizing graphene oxide (GO) by microwave radiation along with organic solvents is described. Microwave radiation was utilized to cause the graphite worm to expand significantly in all directions.SEM images were used to assess the morphology and structure of GO nanomaterials. X-ray diffraction, UV-Vis, and Fourier-transform infrared spectroscopies were used to identify the oxygenated groups on the GO surface. When compared to the conventional Hummer's technique, the new microwave-assisted approach significantly reduces reaction time and is much more eco-sustainable. Aside from being environmentally sustainable, the suggested approach has the appealing feature of producing a large number of graphene nano-sheets quickly. This article compares efficient and environmentally microwave-assisted and conventional modified hummer methods for graphene oxide preparation..*

Keywords: Eco-friendly, Eco-sustainable, Microwave radiation, Nanomaterials, conventional.

I. INTRODUCTION

Nanomaterials as future materials, due to their outstanding characteristics and unique structure tends to increase their applications in different areas. Their extraordinary electronic, mechanical, and thermal properties have possessed fresh pathways for the inventions of an extensive range of novel materials. Graphene, a 2-D material made up of carbon atoms with sp2 hybridization, has gained a lot of attention due to its exceptional physicochemical and mechanical properties [1-4].As a result, graphene is an exciting topic for fundamental studies as well as a favorable material for future practical applications in catalysis, biosensors, and water treatment [5-12].Since the foremost studies by Brodie[13], Staudenmaier[14],&Hummers with co-authors [15], which investigate the oxidation of the graphite, GO has been mostly prepared using these methods. Under the right circumstances, the bottom-up synthesis could theoretically be used to precisely control the morphology as well as the size of an element. Top-down synthesis begins with micro-structures like graphite, fibers, and nano-tubes, and then extracts or peels away individual graphene layers using physical, chemical, or electrochemical methods [16].There are several chemical methods currently in use that highly depend on the use of harsh oxidizing agents such as carboxylic acid, potassium permanganate, sulphuric acid or [17], formic acid[18], and extreme utilization of organic solvents for exfoliation of graphitic layers with the incorporation of oxygenated groups[19]. Furthermore, the oxidized graphite contributes to its dispensability in polar solvents, such as an aqueous solution, as well as to the development of its various applications. A reduction in electrical conductivity is also caused by the oxidation process because of the formation of disorders, defects, rough surfaces on the surface graphitic layers.

Moreover, our process has numerous advantages, including low cost, high productivity, and less time taker as compared to conventional chemical procedures. UV-Vis, Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy(SEM)were used to characterize the MGO (Microwave-assisted Graphene oxide) sheets and HGO(Hummer).

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II. EXPERIMENTAL

2.1 Materials and Chemicals

The chemicals used for the studies served from Merck Specialty Pvt. Ltd. (India). The chemicals are concentrated potassium permanganate ($KMnO_4$, >99%), sulphuric acid (H_2SO_4, 98%), sodium nitrate (Na_2NO_3), hydrogen peroxide (H_2O_2, 30%), graphite with a 99% purity and an average size of 200 and domestic microwave oven with controlled temperature, time and intensity.

2.2 Preparation of HGO

The conversion of graphite powder to HGO (Hummer's method adopted graphene oxide) was carried out using a modified method of Hummer's method [15].The graphite powder (4g) and sodium nitrate (4g) were combined in a 1000ml beaker with 180ml of 98 percent H_2SO_4 and the beaker was held in an ice bath (0-5□) and stirring was constant. After 4 hours of stirring to regulate the temperature, potassium permanganate (24 g) was added very slowly. The addition of potassium permanganate was extremely precise, and the reaction temperature remained below 12□ throughout. Following that, the solution is diluted with 368ml DI water and kept stirring for an additional 90 minutes. After that removed the ice bath & solution were stirred for two hours along with 35□ temperature. Then, the solution was kept at 98□ for 15 to 20 minutes in reflux. After ten minutes, the solution turns brown as the temperature rises to 33 °C. After 10 minutes, the temperature reaches 25□ and remains there for 120-180 minutes. Finally, a brown solution was brightly colored by the 80 ml of H_2O_2 addition at the end. Particles in the solution were allowed to settle at the bottom of the beaker for a few hours before being filtered out. Then the remaining water was used to wash the solution with 10% HCl and DI water until the pH level reached neutral. Next, dry the product at 60°C for at least 6 hours to achieve HGO. The HGO solution is depicted in Fig. No. 01.

Figure 2: Pictorial image of HGO solution.

2.3 Preparation of MGO

The microwave-assisted method was used to synthesize GO, Viana and coauthors provide a detailed description of the synthesis process [20].A weight ratio of 1:2:1.9 was used to combine graphite powder (1000mg) with $KMnO_4$ and HNO_3.Then, stirred the mixture for 15 minutes at the temperature of 25□. This system was subjected to a 60second 900 W microwave irradiation in a domestic microwave oven. Following a series of rinsing with distilled water to achieve the neutral pH and a 3-hour drying period in an oven set to 110 °C, expanded graphite was obtained from the remaining solid material. Magnetized stirring was used for 30 minutes to mix 1g of expanded graphite with 2mL of H_2SO_4 and 1000mg of $KMnO_4$. After two hours of sonication, then the addition of 10 mL of H_2O_2(30%).After that, the brown solid solution can be shown in Fig No. 02, then filtered it and solid part obtained, 10% HCl and distilled water used for the washing of solid part that had been obtained until the pH reached a value of 7.00.

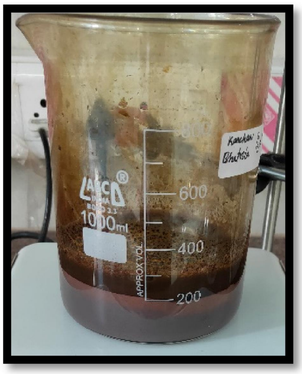


Figure 2: Pictorial image of HGO solution.

2.4 Characterizations

In this study, the absorption spectra of UV-Vis were carried out by using a UV-spectrophotometer (UV-1800 240V) that operated between 250 and 700 voltages. The crystalline phase of freshly prepared graphene oxide was collected by using aPANalyticalX'Pert PRO X-Ray Diffractometer, which was used in this investigation.

To investigate the nature of oxygen functional groups attached to graphene sheets, FT-IR spectra were recorded in the 300–4500 cm^{-1} region range (Agilent ATR model) FE-SEM; NOVA Nanosem 450 (Field Emission Scanning Electron Microscopy) was used to examine the micro-level structure of HGO and MGO.

III. RESULTS AND DISCUSSION

To begin, we characterized and MGO in an aqueous solution using UV-Vis spectra, that revealed a distinctive absorption maximum at 230 nm, can be depicted inFig No. 03, which is assigned to the π -π^* transition of C=C bonds, and a broad shoulder between 290-300 nm, which is assigned to the n -π^* transition of C=O bonds[21].

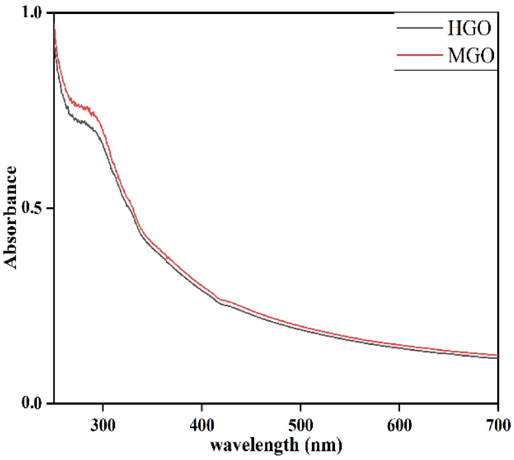
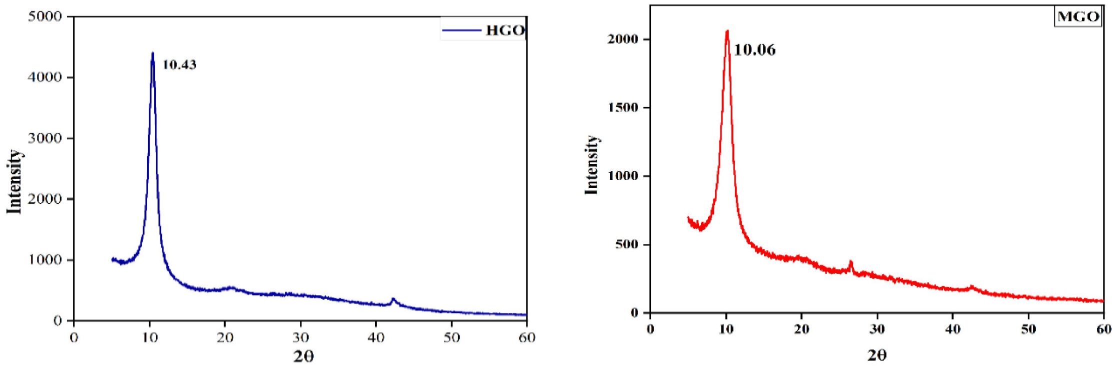


Figure 3: UV-Vis spectra of HGO and MGO in aqueous solution.

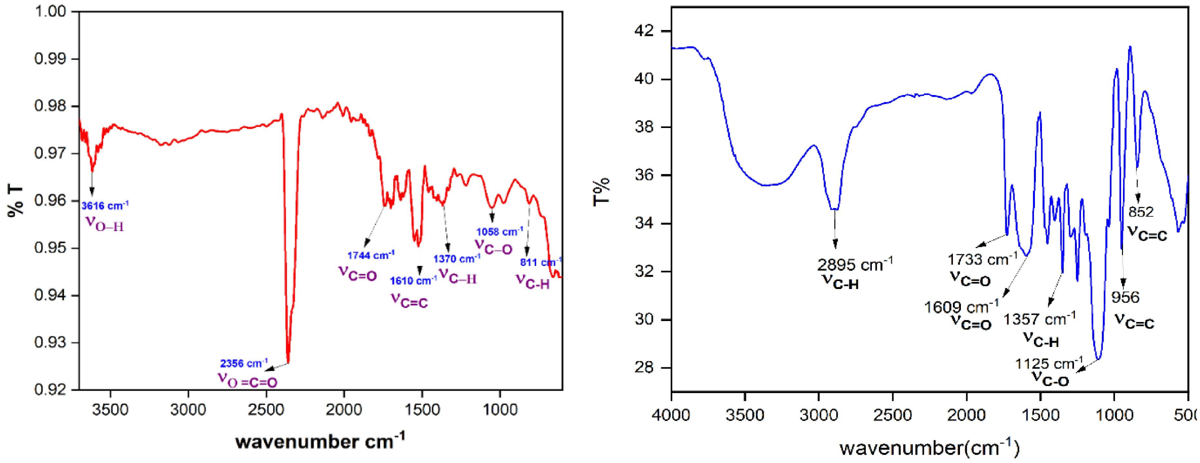
XRD was used to measure the graphite structure's expansion as a result of hydration and oxygen-group intercalation.HGO has a peak at $2\theta=10.43^0$, which indicates that graphite powder has been oxidized. HGO 8.46nm (FWHM=0.283) d-spacing was also increased. At $2\theta=10.09^0$, the MGO peak indicates that graphite powder was oxidized into MGO, which can be observed. Aside from that, the d-spacing of MGO 8.02 nm (FWHM=0.203) was widened. Fig No. 04(a&b) can be depicted the XRD pattern of HGO & MGO. During the chemical oxidation reaction, an oxide functional group, such as carboxyl, epoxy, or hydroxyl functional groups, was intercalated at the carbon basal plane, increasing the carbon basal plane's thickness. A result of this was an increase in the distance between successive carbon layers.



(a) (b)

Figure 4: X-ray diffraction patterns of (a) HGO (b) MGO

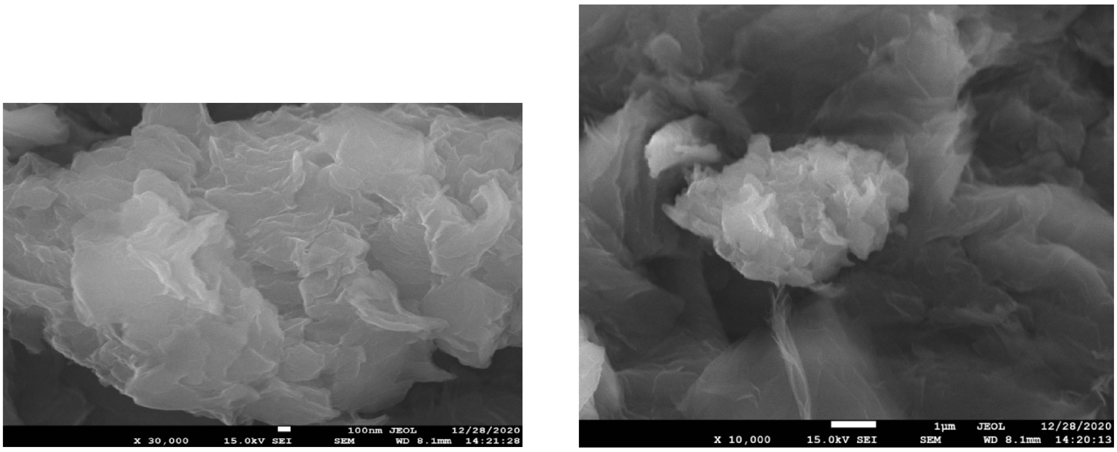
FTIR analysis results show that in Fig No. 05 (a) HGO shows a peak at 1058 cm-1which is corresponds to the C-O bond, assuring the presence of oxygenated groups after the process of oxidation. The peak at 1744 cm-1is attributed to the presence of –COOH groups and the peak at 2356cm-1is attributed to the presence of the O-C-O group. The peaks between 1610 and 1630 cm-1 demonstrate that the C=C bond has always been present prior to and following the oxidation process. Water absorption in GO is indicated by a broad peak between 2885 and 3616 cm^{-1} caused by the O-H stretch of H_2O molecules. The peaks were found similar with earlier reported work[22]. In Fig No. 05 (b) shown the IR spectra of MGO can be found at 2895 cm^{-1} C-H stretching of alkane, 1733 cm^{-1} C-O stretching of carbonyl group, 1609cm^{-1} C=C stretching of α, β- unsaturated ketone, 1125 cm^{-1} shows strong C-O stretching of a secondary alcohol, 852 - 956 cm^{-1} C=C bending of an alkene can be shown in Fig No. 05.



(a) **(b)**

Figure 5: FTIR spectra of (a) HGO (b) MGO

The SEM micrograph of Graphite showed a well-organized platelet-like crystalline form of carbon, and EDX confirmed the presence of approximately 100 atomic percent of carbon. The wrinkled and layered flake's surface can be seen in a micrograph of HGO, as well. As a result, the presence of flakes suggests that the graphite layers were oxidised to Graphene oxide. The SEM micrograph can be seen in Fig No. 06.



(a) **(b)**

Figure 6: FE-SEM micrographs of (a) HGO (b) MGO

IV. CONCLUSION AND FUTURE SCOPE

An environmentally friendly method for producing graphene nano-sheets has been developed by combining microwave radiation with eco-friendly chemicals. In this study, we demonstrated a rapid and simple microwave irradiation method for synthesising GO from graphite. Other chemical oxidation methods, such as Hummers', were significantly hampered by the novel method's major benefits. This method has the advantage of producing graphene nano-sheets in a short period of time and using environmentally friendly chemicals. The simplicity and scalability of this method make it ideal for commercial production of graphene oxide. When using the microwave approach, the reaction time was reduced to a few minutes, which was significantly shorter than the time required by Hummers' method (~5 h). It was also much simpler to control and regulate the reaction temperature in the new method than it had been in Hummers' method, which required three separate temperature control steps. Moreover, this method is environmentally friendly, and it will be extremely beneficial in the production of GO on a large scale in the future. To better understand the chemistry of graphene, the method described here shows promise for making MGO nanosheets, which can then be used for a variety of engineering purposes.

4.1 Conflict of Interest

None

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