PREPARATION AND CHARACTERIZATION OF GRAPHENE OXIDE NANOSHEETS USING CAFFEINE

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Abstract— Caffeine is an alkaloid that is found in varying quantities in the seeds, leaves and fruits of some plants. The high-quality Graphene Oxide Nanosheets prepared using graphite powder and Caffeine as raw material. The formation of Graphene Oxide (GO) was confirmed from analysis and characterization was carried out by Fourier Transform Infrared (FTIR) and Scanning Electron Microscopy (SEM). The presence of different types of oxygen functionalities in GO were confirmed at broad and wide peak at 2362 cm^{-1} can be attributed to the O-H stretching vibrations of the C-OH groups and water. The absorption bands at 1623.77 cm^{-1} can be ascribed to benzene rings. SEM result of Graphene Oxide shows well defined interlinked three dimensional GO Nanosheets zoomed at 500 X as well as indication of distance between sheets.

Keywords: Graphene, Graphene Oxide, FTIR, SEM, Nanosheets

I. INTRODUCTION

The field of nanotechnology is one of the upcoming areas of research in the field of science and engineering. Graphene oxide (GO) is of huge attention due to its less cost, availability as well as its unique physical and chemical properties. It has a huge potential in various applications such as charge storage, drug delivery, sensors and composite materials. GO can be synthesized by various method such as vapour deposition, ultra sonication, electrochemical and chemical methods. Thuhathi et al studied the production of Graphene oxide sheets which having in the field of biomedical, drug delivery [1]. Marcano et al synthesized GO through an improved route. Graphite flakes was oxidized using KMnO4 and 9:1 mixture of concentrated Sulphuric acid and concentrated H3PO4 [2]. Kaniyoor et al studied the synthesis of GO at low temperature by exfoliating Graphitic Oxide in Hydrogen atmosphere [3]. Hernandez et.al studied the preparation of GO in liquid phase by exfoliation of Graphite in surfactant/water solution [4]. Chen et.al studied the preparation Synthesis of Graphene Oxide from expanded graphite in presence of a strong oxidant [5]. Stankovich et.al studied the preparation Synthesis of Graphene-based Nano sheets via chemical reduction of exfoliated Graphite Oxide, performed through colloidal suspension route [6]. GO in pristine form can form highly stable suspensions in water. Use of GO functionalized with iron oxide nano particles for in vivo imaging and photothermal therapy for cancer treatment has been also demonstrated.

The aim of this work is to prove that caffeine can also be used for scalable production of high-quality reduced GO using natural graphite as raw material. Caffeine is an alkaloid that is found in varying quantities in the seeds, leaves and fruits of some plants. It is most commonly consumed by humans in infusions extracted from the seed of the coffee plant and the leaves of tea bush as well as from various foods and drinks containing products derived from the kola nut. Caffeine could be used as green reductant as it is nontoxic, biocompatible and commercially available at industrial level. In the present work, the transformation of GO was characterized by Fourier Transform Infrared (FT-IR) and Scanning Electron Microscopy (SEM).

II. THEORY

A. Preparation by Hummers Synthesis Method

Hummers and Offeman studied and produced the GO through oxidation of graphite. Graphite and Sodium nitrate were mixed in H2SO4 in volumetric flask kept under at ice bath with continuous stirring then Potassium Permanganate was added to the suspension very slowly. The mixture was stirred until it became pasty brownish. It is then diluted with slow addition of water. The reaction temperature was quickly increased to 98°C and the colour changed to brown. Further this solution was diluted by adding additional quantity of water with continuous stirring. The solution is finally treated with Hydrogen Peroxide to terminate the reaction by appearance of yellow colour. For purification, HCl and de-ionized water was used and GO was obtained as a powder after filtration and drying under vacuum at room temperature [7].

B. Characterization of Graphene Oxide

The Characterization of Graphene Oxide can be done using Fourier Transform Infrared Spectroscopy (FTIR), Scanning Electron Microscope (SEM), Transmission Electron Microscopy (TEM), Ultraviolet–Visible Spectroscopy, X-ray Diffraction (XRD).

III. MATERIAL AND METHOD

A. Material

For the preparation of GO, Graphite Powder (Burgoyne), Potassium Permanganate (Molychem), 98 % Sulphuric Acid (Molychem), 30% Hydrogen Peroxide (Molychem), 35% Hydrochloric Acid (Molychem) were used. The laboratory scale setup was prepared by using the equipments such as Magnetic Stirrer, Water Bath, Ultrasonic Bath, Centrifuge, Hot air Oven, Beaker, Separating Funnel, Burette, Conical Flask, Measuring Cylinder, Pipette and Stirring Rod etc.

B. Method

Preparation of GO was done by modified Hummers method. GO was synthesized by oxidizing the graphite powder in a mixture of concentrated H2SO4 and Potassium Permanganate. Initially approximately 2 grams powder of graphite was stirred in 35 ml of 98% H2SO4 on a magnetic stirrer for 2 hours. Then 6 grams of potassium permanganate was gradually added into the above solution by maintaining the

temperature below 200C. The mixture was then stirred at 350C for 4 hours in a water bath. The resulting solution was diluted by adding 90 ml of water under vigorous stirring for 1 hour. Then a dark brown suspension was obtained. The suspension was further treated by adding 30 % Hydrogen peroxide solution drop wise until the colour of the solution became bright yellow. The resulting GO suspension was washed by repeated centrifugation, first with 5% aqueous Hydrochloric acid solution to remove excess of manganese salt followed by water until the pH of the solution became neutral. The purified GO was finally dispersed in water (0.5 mg/ ml) ultrasonically in an ultrasonic bath. Thus a stable dispersion of GO was obtained. Further the sample is dried in oven for 4-5 hours [8].

IV. RESULTS AND DISCUSSION

A. Preparation of GO

Modified Hummers method has been used for the complete synthesis to produce graphene oxide. The figure 1 shows the conversion of graphite powder to graphene oxide and shows that pure graphene oxide is formed which is confirm form the characterization result.

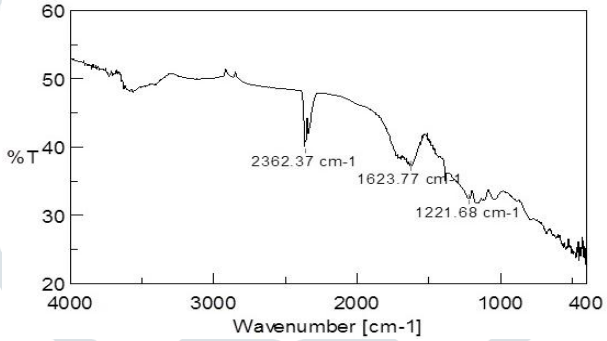
Figure 1: Sequence of step carried out to preparation of GO form graphite powder

B. Characterization by Fourier Transform Infrared Spectroscopy

This FTIR gives information on the vibrational and rotational modes of motion of a molecule and hence an important technique for identification and characterization of a substance. The Infrared spectrum of an inorganic compound provides a unique fingerprint, which is readily distinguished from the absorption patterns of all other compounds; only optical isomers absorb in exactly the same way. FTIR spectrum of the Graphene oxide obtained in these steps confirms the successful oxidation of the graphite as shown in Figure 2.

The presence of different types of oxygen functionalities in GO were confirmed at broad and wide peak at 2362 cm^{-1} can be attributed to the O-H stretching vibrations of the C-OH groups and water. The absorption bands at 1623.77 cm^{-1} can be ascribed to benzene rings. The peak at 1221.68 cm^{-1} can be attributed to CO carboxylic. The result obtained by this characterization is similar literature [9, 10, 11, 12].

Figure 2: FTIR spectra for Graphene Oxide



C. Characterization by Scanning Electron Microscopy

For characterization using SEM, pellet was prepared. The purpose of preparing the pellet from Nano powder before characterization was to make it easier to mount the samples in different characterization equipment's and to prepare uniform surfaces for imaging with scanning electron microscope. The prepared pellet was then coated with Platinum before mounting in the machine. For imaging the sample with SEM, the specimen must be electrically conductive at least at the surface. Accordingly the pellet was coated with Platinum to avoid charging which causes scanning faults and other image artefacts during scanning of the sample by the electron beam and it helps to obtain qualified images and for high resolution observation. The SEM images clearly show the formation of Graphene Oxide. The SEM images shows the Graphene Oxide have well defined and interlinked three-dimensional Graphene sheets, forming a porous network that resembles a loose sponge like structure as shown in Figures below. Similar types of the results were observed in the literature [13]. Figure 3 shows SEM image of Graphene Oxide in the form loose spongy structure which is zoomed at 100 X.

Figure 3: SEM image of GO in the form loose spongy structure

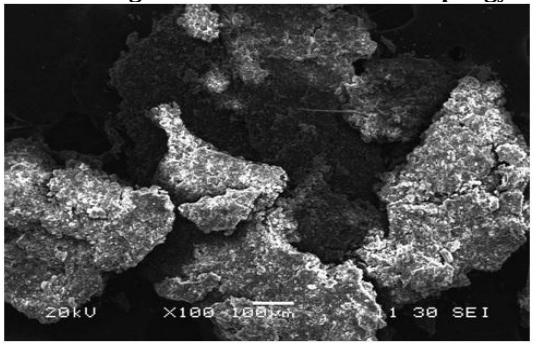


Figure 4 shows the SEM image of Graphene Oxide shows well defined interlinked three dimensional Graphene Oxide Nanosheets zoomed at 500 X.

Figure 4: SEM image of defined interlinked 3D Graphene Oxide Nanosheets.

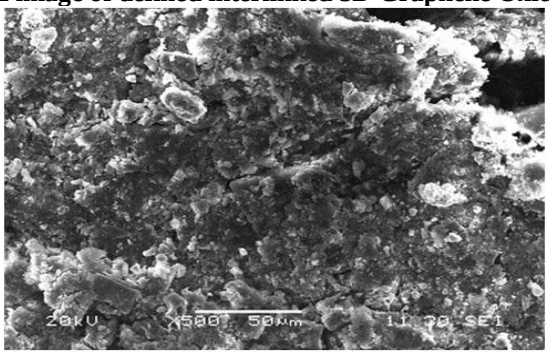
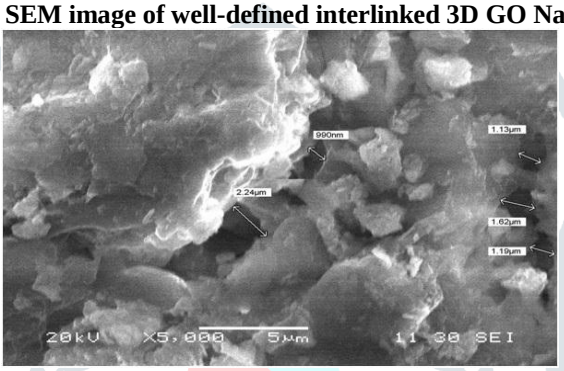


Figure 5 shows the SEM image of Graphene Oxide shows well defined interlinked three dimensional Graphene Oxide Nanosheets zoomed at 5000 X with indication of distance between sheets.

Figure 5: SEM image of well-defined interlinked 3D GO Nanosheets



CONCLUSION

Graphite powder has been used as a starting material and caffeine for preparation of high-quality reduced GO. Modified Hummers method has been used to prepare Graphene Oxide. This method was carried out with the highest conversion level of graphite powder to Graphene Oxide and shows that pure Graphene Oxide is formed. Characterization by FTIR and SEM shows formation of Graphene Oxide.

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