Synthesis, Characterization and Transparency of Graphene Oxide Nano Particles Prepared by Modified Hummer's Method

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Abstract: Graphene oxide (GO) nano particleswere successfully prepared by modified Hummer's method for the future application of touch screens in electronic devices, energy storage, water filtration system, medical sensor and drug delivery. Comprehensive characterizations of the properties of GO nano particles were conducted. To analyze crystal structure of GO nano particles X-ray diffraction (XRD) was done. Fourier transform infrared spectra analyzer (FTIR) was used to show the presence of oxygen containing functional groups in GO nano particles. The photoluminescence (PL) certifies that GO nano particles possessed excellent optical properties in blue and green region and a strong emission peak is observed at 532.41nm. Atomic force microscopy(AFM) shows the surface roughness and the particle size found around 40nm.Finally, GO nano particles was obtained by modified Hummer's method.

Keywords: Application, Characterization, Grapheneoxide(GO), Spectroscopy

I. INTRODUCTION

Graphene is one of the most important materials in the upcoming electronics industries. Graphene is monolayer of graphite, expensive and relatively hard to produce, great efforts are made to find effective ways to make and use graphenein different application. So its derivativesor related materials such as graphene oxide.(GO) is suitable for these purposes. photoluminescence is studied without any post treatments.

GO is a single-atomic-layered material made by the oxidation of graphite crystals, which are inexpensive and abundant. It is dispersible in water and organic solvent, as a result it is easy to process. GO is one of those materials which is used in electronics industries such as transparent conductive film, energy storage [3]. MWCNT is used to enhance its electrical conductivity [5]

II. EXPERIMENTAL SECTION

2.1 Chemicals required

Graphite	fine	powder	(CDH),	Sodium	nitrate
(98%,MERCK),	Potassium		permanganate		(99%,

MERCK), Hydrogen peroxide solution(30% wt, LOBA CHEMIE), Sulphuric acid (98%, MERCK), Hydrochloric acid (35%, HIMEDIA).

2.2 Synthesis of Graphene Oxide

2g of graphite fine powder and 2g of sodium nitrate (NaNO3) were mixed in 90 mL of Sulphuric acid (H2 SO4) in a flask and the mixture was stirred for 4 hours kept under ice bath to maintain 0-5° C.12g of potassium permanganate was added slowly to the suspension so that the reaction temperature should be less than $15 \,^{\circ}\text{C}$. The suspension was diluted by adding 184mL deionised water (DI) and kept under continuous stirring for 2 hours. After that ice bath was removed and it was stirred at 35 °C for 2 hours. The above suspension was kept in a reflux system at 98 °C for 10 minutes. 40mL hydrogen peroxide (H2O2) was added to the suspension and stirred for half an hour at room temperature.25mL of DI water as added and stirred for 1 hour at room temperature [1]. The suspension was centrifuged usingratio of 1:10. The suspension was filtered using filter paper and kept overnight. Next day, it was kept in oven at 60 °C for overnight. Sample was collected, grinded and finally GO powder was obtained.



2.3 Characterization

X-ray diffraction (XRD) patterns were obtained from BRUKER D8 advance(CuK α radiation, $\lambda = 1.5406$ Å) diffractometer, FTIR spectroscopy was conducted using BRUKER vertex 70. The Photoluminescence spectra were recorded on Hitachi F-7000 FL Spectrophotometer. AFM was done using NT-MDT next.

III RESULTS AND DISCUSSIONS XRD ANALYSIS

The X-ray diffraction (XRD) is technique for crystalline material characterization. It is used to measure the average spacing's between layers and to determine the orientation of a single crystal or grain pattern obtained [1]. The XRD for as synthesized GO nanoparticles by modified Hummer's method is shown in Figure 1. It shows the diffraction peak at $2\theta = 8.84$ °, which is mainly due to the oxidation of graphite. The diffraction peak of pure graphite is found around 26.6 is shown as inset in Figure 1.Peak at 26.6 shows that the material retains the properties of graphite [1]. Peak for GO should be at 10 [1] but in our case it was shifted in left side i.e. at lower angles, it may be because of strain.

AFM Analysis

Atomic force microscopy(AFM) was used to determine the surface morphology and roughness of film. AFM of GO is shown in figure 2. GO film was deposited on glass substrate using spin coating and it was observed that root mean square roughness of graphene oxide (GO) film is 6.823. The particle size of GO found approx 40nm.

(a)

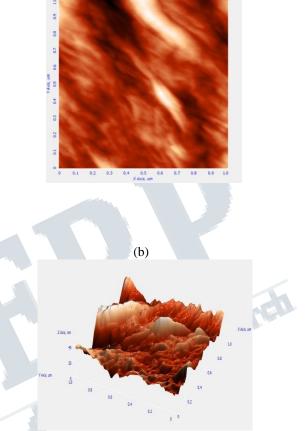


Figure 2 a) 2D and b) 3D view of AFM of GO

IV. FT-IR ANALYSIS

FTIR spectra analysis was performed to investigate the structure and functional groups of the materials [2], as shown in figure 3. A strong peak at 1082 cm¹ shows the C-O group, which confirms the presence of oxygen containing functional groups [1]. Adsorption bands at 1620.52 cm⁻¹ shows C=C group, at 2390.37 cm⁻¹ shows CO₂ group [1]. The absorbed water in GO is shown by a broad peak at 2885 cm-1 to 3788.09 cm⁻¹, contributed by the O-H stretch of H₂O molecules. The presentation of C=C group showed that the structure of layer graphite was still retained.



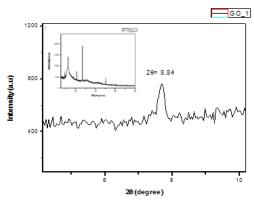
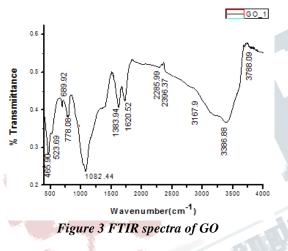
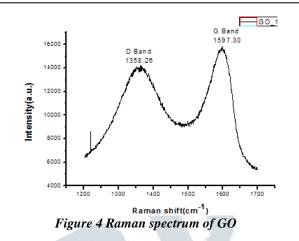


Figure1 XRD of GO



V. RAMAN ANALYSIS

Raman spectroscopy is atechnique used to observe rotational, vibrational, and other low-frequency modes in a system. They are widely used tool for the characterization of carbon products, especially conjugated & C=C group leads to high Raman intensities [1]. Figure 4 shows the Raman spectrum of GO, where the disorder band (D band) of GO is at 1358.26 nm and the in-phase vibration (G band) of GO is at 1597.30 nm [1]. Raman spectra of GO obtained was slightly right shifted which means that they shows red shift. This may be because of strain.



Photoluminescence Analysis

Photoluminescence (PL) spectra of prepared GO nanoparticles is shown in Figure 5. The PL spectra shows a sharp peak at 532.41 nm corresponding to an excitation wavelength of 325 nm and it is due to the oxidation of graphite and formation of sp^2 in grapheneoxide. This is also reported by other researcher [4]. It shows luminescence in blue and green region, which will utilize in making green and blue LEDs.

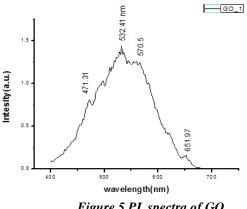


Figure 5 PL spectra of GO

IV CONCLUSION

GO nanoparticles is synthesized by modified Hummer's method. Formation of GO is satisfied by XRD analysis. The presence of oxygen containing groups and characteristic peaks are determined by FTIR. Raman spectra is used to find C=C group in GO. AFM shows the surface roughness i.e root mean square roughness of graphene oxide (GO) film is 6.823 and particle size around 40nm.



ACKNOWLEDGEMENTS

Author would like to acknowledge UGC- DAE-CSR Indore for providing XRD, FTIR, RAMAN characterization.

REFERENCES

- Paulchamy B, Arthi G, Lignesh BD (2015) A Simple Approach to Stepwise Synthesis of Graphene Oxide Nanomaterial. J NanomedNanotechnol 6: 253. doi: 10.4172/2157-7439.1000253
- Jianguo Song, Xinzhi Wang, and Chang-Tang Chang, "Preparation and Characterization of GrapheneOxide,"Journal of Nanomaterials, vol. 2014, Article ID 276143, 6 pages, 2014. doi:10.1155/2014/276143
- In book: Applications of Graphene and Graphene-Oxide Based Nanomaterials, pp.39-55 DOI: 10.1016/B978-0-323- 37521-4.00002-9 December 2015.
- 4) Chien CT¹, Li SS, Lai WJ, Yeh YC, Chen HA, Chen IS, Chen LC, Chen KH, Nemoto T, Isoda S, Chen M, Fujita T, Eda G, Yamaguchi H, Chhowalla M, Chen CW "Tunable photoluminescence from graphene oxide"AngewChemInt Ed Engl. 2012 Jul 2
- 5) Rai, Chhaya; Pandey, Padmini; Parra, Mohammad Ramzan; Haque, Fozia Zia "Optical, Dielectric and Impedance Studies of SiO2/MWCNT NanocompositeSynthesizedThroughIn-Ultrasonication-Assisted Sol-Gel Method" Journal of Advanced Physics, Volume 3, Number 3, September 2014, pp. 194-204(11)