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Synthesis of Reduced Graphene Oxide (RGO) by Using Hydrothermal Method

Monika S. Khole[1], Sanjay K. Tupe[1]

[1]Department of Physics, Kalikadevi Arts, Commerce & Science College, Shirur (K.), Dist-Beed-413249, Maharashtra, India

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ABSTRACT

Reduced graphene oxide, RGO (also called chemically modified graphene, CMG) was synthesized by a hydrothermal method, with graphite oxide (GO), prepared by using improved hummer method at different temperatures, served as the raw material. Structural and morphological studies indicate the degree of reduction is dependent on the temperature, which is also verified by Raman analysis. The variation in interlayer distance and the intensity ratio of the D to G Raman modes (ID/IG)indicates higher reaction temperature can accelerate more reduction of GO and also (I2D/IG) ratio indicate the reduction in number of graphene layers. The reduction material is characterization by Raman spectroscopy.

Keywords:Graphene oxide (GO), Reduced graphene oxide (RGO), Hydrothermal method, Improved Hummer Method, Raman Spectroscopy.

I. INTRODUCTION

Carbon is the one of the most common atoms on Earth, occurs naturally in many forms and as a component in countless substances which are called allotropes of carbon. Graphene, a “wonder material” is the world’s thinnest, strongest, and stiffest material, as well as being an excellent conductor of heat & electricity. [1] Graphene, a single layer of sp2- bonded carbon atoms packed in a hexagonal lattice, since the first fabrication via a “Scotch tape” method in 2004 [2], has triggered great research interests. Graphene & reduced graphene oxide (RGO) are promising candidates as components in applications, such as storage materials [3], catalyst supports [4] and electronic devices [5].

Graphite oxide has a similarlayered structure to graphite, but the plane of carbon atoms in graphite oxide is heavily decorated by oxygen-containing groups, which not only expand the interlayer distance but also make the atomic-thick layers hydrophilic. As a result, these oxidized layers can be exfoliated in water under moderate ultrasonication. If the exfoliated sheets contain only one or few layers of carbon atoms like graphene, these sheets are named graphene oxide (GO). The most attractive property of GO is that it can be reduced to graphene-like sheets by removing the oxygen-containing groups with the recovery of a conjugated structure. The reduced graphene oxide (RGO) sheets are usually considered as one kind of chemically derived graphene. Some other names have also been given to RGO such as

functionalized graphene, chemically modified graphene, chemically converted graphene, or reduced graphene.[6]

In this study, we fabricated reduced graphene oxide (RGO) from the reduction of graphite oxide (GO) by hydrothermal method at different temperatures, by systematically analyzing the influence of reaction temperature on the interlayer distance and the intensity ratio (I_D/I_G)of the D to G Raman modes of the as-produced RGO, we find that the degree of reduction is related with the reaction temperature, the conductivity in turn related with the degree of reaction [7].

II. EXPERIMENTAL

2.1. Synthesis of GO & purification:

Graphite oxide was prepared from natural graphite by the well-known Improved Hummer method. Nature graphite (1g) was grindedwith NaCl (1g) for 1hr. Afterward NaCl was washed away using distilled water with filtration & dried it below 45 ^{0}C [8]. For the Improved method, we take 9:1 proportion of conc.H_2SO_4/H_3PO_4. The dried graphite (660 mg) was then mixed with 75ml of concentrated sulfuric acid in round bottom flask& stirred up to 3 hrs. Then next 8.8 ml of H_3PO_4 was added to the mixture & allow to dissolve for 1hr.The flask was then placed in an ice bath & (4g) of $KMnO_4$ was slowly added while the temperature was kept below 20^{0}C for 2hrs. The solution then heated at 35^{0}C for 1hr. Then add 1ml H_2O_2 to 100ml water & add above mixture to H_2O_2 solution. Dilute this suspension to 400 ml water & stirred it for 1hr. After that, this suspension was shifted through a metal sieve & then filtered through polyester fiber. The filtrate was centrifuge at (10000 rpm, 1hr) with 180ml distilled water & the supernatant was decanted away [9]. The remaining solid material was again centrifuge for many times until its pH does not get maintained. Finally, the GO was kept for drying at below 45^{0}C.

2.2. Synthesis of RGO & purification:

First 120mg as synthesized GO was suspended in 120ml distilled water to give brown colloidal solution by 30min of sonication. After the Sonification stirred solution for 1hr & add 5ml of hydrazine hydrate as reducing agent stirred this suspension for 10 min & then add 5ml of polyethylene Glycol ($HO(CH2CH2)_nOH$) as capping agent. After that this solution was sealed in 180ml Teflon-lined autoclave & put this autoclave into the hydrothermal furnace at temperature 120^{0}C for 5 hrs. Then the solid were filtered & washed with distilled water several times, finally the collected sample was dried in incandescent lamp at below 45^{0}C. Same procedure was repeated for temperature at 140^{0}C & 160^{0}C. Finally, we got RGO for 120^{0}C, 140^{0}C & 160^{0}C.

III. EXPERIMENTAL ARRANGEMENT

Figure 1.The macroscopic physical samples

IV. GRAPHICAL RESULTS

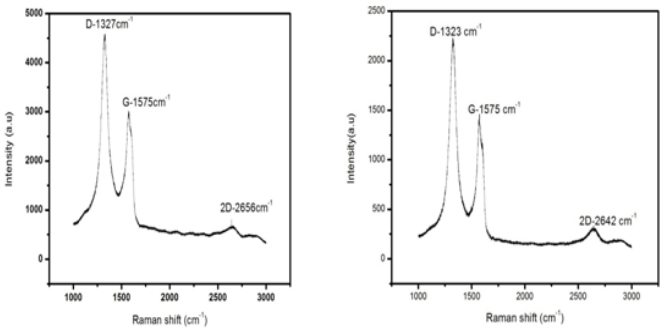
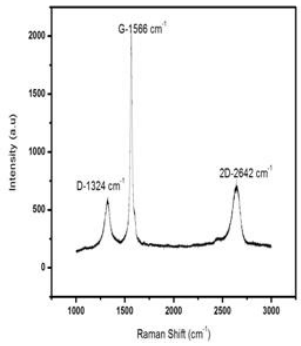


Fig a. Raman Spectra of GOFig b.Raman spectra of RGO (NaOH)



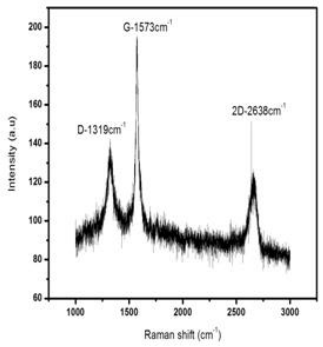
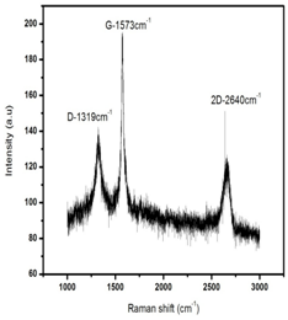


Fig c. Raman Spectra of RGO-1200CFig d. Raman Spectra of RGO-1400 C



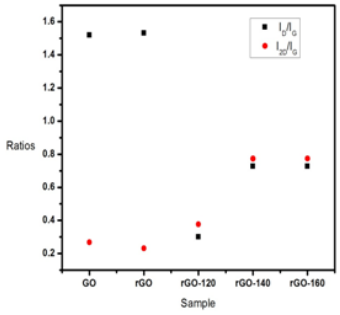


Fig e. Raman Spectra of RGO-1600CFigf. The Intensity Ratio of D,G,2D band

V. DISCUSSION

Raman spectroscopy is a very useful method to characterize the degree of reduction of GO. For all carbonaceous materials, both D and G bands are the predominant features in the Raman spectra. They are represented by peaks at around 1318-1346 cm^{-1}, 1490-1691 cm^{-1}, respectively. In the Raman spectrum of GO fig (a) Shows the G band is broadened at 1575cm^{-1}. In addition, the D band at 1327 cm^{-1} becomes prominent, which indicates that after oxidation, some of the carbon atoms have changed from sp^2 to sp^3 hybridized carbon, the C=C double bonds in the graphite layers are destroyed. In fig (b) shows the Raman spectra of reduced graphene oxide by using NaOH as reducing agent but these spectradoes not show more reduction because if we compare D & G band of GO spectra its values are nearly equal. So that I choose Hydrazine Hydrate as best reducing agent we see in the nest result. The comparison shows in fig (c), fig d) & fig (e) almost all D band of RGO get reduces means number of defects are reduced & G band shows more crystallinity as compared to GO. Fig (f) shows the intensity ratio of the D to G bands (I_D/I_G) also represents quantity ratio of sp^3 hybridized carbon and sp^2 hybridized carbon, the decrease of I_D/I_G means the increase of sp^2hybridized and decrease of sp^3 hybridized in RGO. That is, increased I_D/I_G shows better reduction of GO to RGO. Here the best reaction temperature is 160^0 C, according to the I_D/I_G. In the case of I_D/I_G ratio varied with the reduction time & temperature. In the case of I_{2D}/I_G increased means the number of graphene layers are decreased.

VI. CONCLUSION

We have successfully obtained RGO at different temperatures via a hydrothermal method. The degree of reduction of GO is affected by the reaction temperature, which can be confirmed by Raman characterization. In this project we studied reduction of graphene oxide at different temperature such as 120^0C,140^0C, 160^0C & finally we conclude that the best reduction occurred at temperature 160^0C. So that higher temperature makes more reduction of graphene oxide to RGO. From the result we conclude that if the I_D/I_G ratio increases means number of defects get reduces and more reduction. From the I_{2D}/I_G ratio if the ratio increases with increase in temperature, then number of graphene layers decreases. From this report we studied that the hydrazine hydrate was the best reducing agent for reduction of graphene oxide.

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