SYNTHESIS OF GRAPHENE OXIDE-FE(III) COMPLEX:-- A NEW MATERIAL FOR CATALYSIS

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ABSTRACT

Graphene is a chemical element of carbon consisting of one layer of atom organized during a polygon lattice. Graphene and graphene oxide could be a material that has generated monumental analysis interest and activity within the past few years due to its outstanding properties and wide applications within the fields, like nanoelectronics, sensors, nanocomposites, batteries, super capacitors, chemical element storage, clear conductors and energy storage materials. (Reduced) graphene oxides have a good potential as a practical chemical building block in fabrication of assorted carbon-based nanostructures and their composites. We report simple and efficient routes for the complexation of the graphene oxide and Fe (III) which appears to restore original pristine graphene state. This study represents the example of the use of graphene oxide as a ligand and is expected to expand the scope of graphene chemistry in connection with the application of this material in catalysis reactions

Keywords: Graphene oxide, D.C electrical resistivity, Activation energy, FTIR

1. INTRODUCTION

Nowadays researchers have aimed toward graphene and graphene oxide (GO) primarily based materials because of their glorious potential, thermal, and mechanical properties which created a lot of application in the field of semi conductive coating, sensors, microwave fascinating, and power storage devices. [1] After the invention of graphene and graphene compound, attention has been created on the non-volatile memory devices because of their potential applications[2-4] In nanoscale area, materials often showcase chemical and physical properties which can't be observed neither in bulk nor in atom. Therefore, a super deal of effort has been devoted to prepare nanostructures which provide the specified properties. Electrical, optical, magnetic and chemical process properties of iron primarily based materials are exploited for realizing many alternative functions during a large form of analysis things. They have been extensively used in super capacitors, data storage, lithium ion batteries, catalysis, drug delivery, therapeutic agents as well as water treatment [5, 6].

Nevertheless, in several cases, it absolutely was necessary to mix iron nanostructures with alternative materials so as to get nano composites with increased performance. Graphene has shown a good potential to be a valuable choice for synthesizing iron oxide/ graphene nano composites. These hybrid nanostructures are mostly planned and used for developing advanced devices in several applicatory fields.[7] One of the advantages of graphene is easily dispersed in water and other organic solvent. A simple method for preparation of graphene oxide iron composites will be highly anticipated. Iron oxides are a rich kind of shape and occur in a remarkable type of settings from geological to nanoscale technological software.[8]To look at the transformation of cross nanosheets in herbal environments, move aqueous dispersion turned into blended with Fe ions to form photoactive complex under visible light irradiation.The flat surface of graphene together and its unique Fermi levelelectronic structure open up the exploration of chemical reactions between various substrates with its involving other bondingconfigurations. In graphene or graphene oxide metal complexation reactions, the vacant d-orbitals of a transitionmetal can overlap withand accept electron density from the occupied pi-orbitals of graphene,resulting in the coordination of the metal atom to individual benzenoid aromatic rings of graphene and in its metal complexes. It is possible to electronically conjugate adjacent graphene sheets in resultant structure having advanced properties. The metal bond to graphene is not expected to significantly rehybridize the sp^2 carbon atoms and can be anticipated to provide a much milder modificationof the electronic structure of graphene than pi-bond formation; as a result, the metal coordination site on the graphene surface is expected to be mobile.In the present research module we study the change in electrical properties by Go-Fe catalyst .with a view to understand the catalytic activity of Go-Fe we have to under taken the study of electrical resistivity as a function of composition and temperature.

2. Experimental Section:

2.1 Synthesis of graphene oxide (GO)

The graphene oxide was prepared by suing well known reported Hummer's Method. [9] The method was described shortly as follows. A 500 ml reaction flask was charged with 1 g of natural flake graphite and a stir bar. 46 mL of concentrated sulphuric acid was added into the reaction flask. Next, 1 g of$NaNO_3$ was added under stirring and cooled in an ice-water bath. The flask was then slowly charged with 6 g of $KMnO_4$ in order to avoid rapid increase of the solution temperature. Once mixed, the flask was transferred to a 35^o C oil bath and stirred for about 1 h. Then, 80 mL of water was slowly added drop wise, forming a thick paste and increasing the temperature. Meanwhile, the temperature of the oil bath was raised to 90^{o}C, and the solution was stirred for 30 min. Finally,200 ml of water was added, followed by the slow addition of 10ml ofH_2O_2 (30%). The solution was then centrifuged at 9000 rpm and washed with water once. The solid was then dispersed in water by mechanical agitation, and 35%HCl was added in order to remove residual metal ions. The solution with about 5% HCl was centrifuged at 9000 rpm, and also washed with water once to remove the upper solution with some visible particles. Washing was repeated with 5% HCl and water until the removal of all visible particlesfrom the precipitates. Next, washing was done with water, until there were some flocs above the precipitates. The upper water was carefully removed. The precipitates were then dispersedin500ml of water by 20 min sonication, forming atomically thin GO.

2.2 Synthesis of GO–Fe complexes

The Iron graphene oxide complex was prepared by earlier reported method [10]. The method of preparation is described as follows. The pH of the GO solution was adjusted to 2 using 1 M HCl. In another flask, 1 M of $FeCl_3$ was prepared by using 0.01 M of HCl in order to avoid the hydrolysis of ferric ions. Then, GO and $FeCl_3$ aqueous solutions were mixed with the same volume under stirring for 24 h. The solution was centrifuged at 9000 rpm and washed with water. It was repeated more than 5 times ,until the upper solution was colorless. Finally, GO–Fe was re dispersed into water and stored for use. The samples for further characterization were prepared by centrifugation and vacuum dried at 60 ^{o} C.

2.3 Characterization

In this study, GO nanosheets were mixed with Fe ions to form the GO-Fe(III) complex which was characterized by FTIR technique using Brucker FT-IR spectrophotometer.The effect of Go-Fe catalyst on the electrical properties like resistivity and conductivity was studied using an impedance analyser, WAYNE KERR 6500B.

3. Result and Discussion

3.1 Spectral analysis

The prepared complex by well-known method was obtained and to confirm the complex formation it was characterized by FTIR analysis.FTIR measurements were used to confirm the formation of bonding between iron and graphene oxide as well as to evaluate the degree of reduction in graphene oxide. The FTIR spectra for GO & GO-Fe are shown in Fig 1and Fig. 2 respectively.

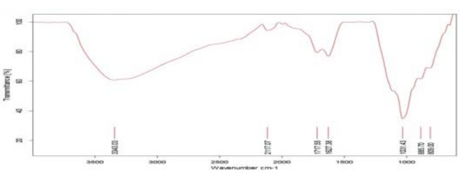


Figure 1: FTIR spectra of Graphene Oxide(GO)

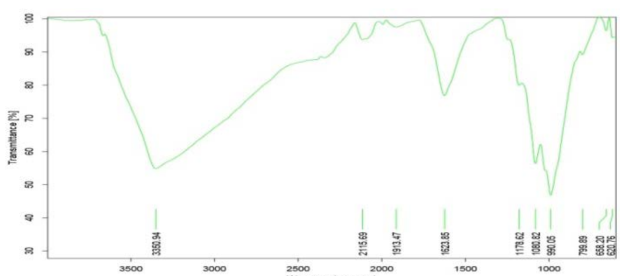


Figure 2: FTIR spectra of Graphene Oxide-Fe(III) complex(Fe-GO complex)

The figure 1 and Figure 2 clearly illustrate the presence of functional group like –COOH,-OH,C=O,-O. Besides a robust surface assimilation band at 3350.94 cm^{-1} assigned to the –OH stretching vibration, the FTIR spectrum of GO shows sharpC= O (1717.55 cm^{-1}), aromatic C = C (1627.38 cm^{-1}), alkoxy CO(1031.43 cm^{-1}). Comparing the figure 1 and figure 2, confirmed the formation of complex between iron (III) and graphene oxide. Mostly the interaction take place at carboxylate and phenolic OH sites.

3.2Electrical Resistivity Study:

The prepared complex is a combination of graphene oxide as a ligand with iron. It is well reported that the introduction of oxygen moiety in the graphene layer decrease the electrical conductivity. To understand the effect of iron introduction in the graphene oxide, the electrical resistivity was studied. Figure 3 shows that the variation of D.C electrical resistivity has been observed for the two samples with respect to temperature. It is clear that resistivity goes down with increasing temperature. This fact reveals the semiconducting nature of prepared Go-Fe complex.

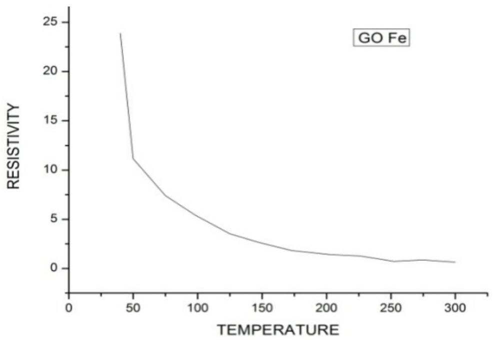


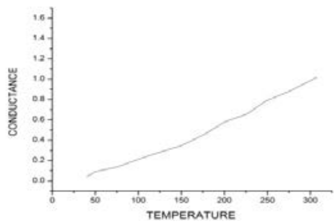
Figure.3

The resistivity of sample of Go-Fe complex and GO at room temperature was tabulated in table 1 it is noticed from the table 1 that the resistivity of Go and GO-Fe complex are gradually decreases. This shows that the interaction of graphene oxide with iron increases the mobility of electron along the sheets of graphene.

Sample	Resistivity(ρ) Room temperaturex10^6 M	Activation energy ΔE(ev)
Go	30	--
Go-Fe	20.408	14.2479

Table 1.Effect of Go-Fe complex on D. C. Resistivity

To get more inside of the material property enhancement, the resistivity data was converted in to the resistivity and represented in the figure 4 and figure 5.Figure 4 and figure 5 shows that as the temperature increases the electrical conductivity also increases, which shows the semiconductor behavior of the Go-Fe complex. The room temp. conductivity of Go-Fe complex was found to be 0.049 S cm^{-1}while for graphene oxide it is 0.001 S cm^{-1}. This indicate that the introduction of iron metal in the graphene oxide increases the electrical conductivity behavior of the material. This observation opens up the new direction towards the enhancement of electrical conductivity through the coordination of metal with graphene oxide towards graphene. More coordination site formation in the material may increases the electron mobility in the material through the formation of new bond.



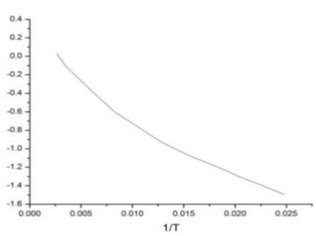


Fig.4 & Fig.5 Effect of temperature on conductivity of Go-Fe complex.

The data of the figure 5 was used to calculate the activation energy of the material. The activation energy of successive GO-Fe was found to be GO-Fe (Ea=14.2479 eV) and the band gap of Go-Fe was calculated as 1.18 eV by using the following equation,

$Eg=2\times 8.6\times 10^{-5}\times 2.3026\times \log_{10}(\rho)/(1/T)$.

On the basis of data it is concluded that as the band gap decreases and material will be a good conductor of electricity and proceed towards metallic behavior.

4. Conclusion:

The synthesis of Graphene oxide _Fe complex will open up the new type of approach towards the retention of electrical conductivity like graphene in the graphene oxide material using simple coordination approach. The increase of the coordination between metal and graphene oxide will be a ideal platform for the retention of electrical conductivity without the removing the oxygen containing functional groups. This will help the material for the advance technology development.

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