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Preparation and Characterization of Green SynthesisGO/Metal/metal Oxide Nano-Composites applied for Nanofluid Applications - A Review

G. Anbarasu
Assistant Professor, Department of Mechanical Engineering, Sri Ramakrishna Engineering College, Coimbatore-22

S. Ajay Kumar
UG Student, Department of Mechanical Engineering, Sri Ramakrishna Engineering College, Coimbatore-22

J. Ajithkumar
UG Student, Department of Mechanical Engineering, Sri Ramakrishna Engineering College, Coimbatore-22

P. Gowthaman
UG Student, Department of Mechanical Engineering, Sri Ramakrishna Engineering College, Coimbatore-22

ABSTRACT

The reduced graphene oxide (RGO) was fabricated by modified Hummersand chemical reduction methods. CRGO was characterized by X-ray diffraction, Scanning electron microscope and Raman Spectroscope. The stability, zeta potential, thermal conductivity and rheological properties of the as-prepared nanofluids were systematically investigated using different experimental methods. Metal/RGO, metal oxide/RGO nano composite was prepared by Ultrafine and water dispersive approach. The obtained characterized with XRD, Raman Spectroscopy, FTIRs, and SEM. This method is that the diameter of nanoparticles decorated by rGO is in the range of 3-5 nm. Those results demonstrate an outstanding potential for the use of GO/Metaloxide/Metal nano-fluids as suitable replacements for the conventional fluids in heat exchanger applications.

Keywords: *graphene oxide, characterization, nanofluids*

1. INTRODUCTION

The two-dimensional carbon nanomaterials, such as graphene have been widely exploredrecently to demonstrate its potential applications in biomedical study and by fabricating graphene-based nano-scaled electronic, optoelectronic, photovoltaic, and sensing devices [1]. Graphene has received significant research attention due to its unique properties of planar structure, high electrical conductivity, high transparency and flexibility, large surface area, and high mechanical stability [2-3].Its benefits of high density of states, high mobility, high work function, and low dimensionality compared with the conventional charge trap materials [5-8] suggested that graphene could be used as the potential material in non volatile memory (NVM) devices as the charge trap medium [4-10]. Some researchers have tried to improve the thermal conductivity of suspensions by adding macro- and micro- sized particles to base fluids, incurring in stability and rheology problems. Nanofluids are suspension of nanoparticles into traditional fluids including water, engine oil and glycerol. Since the 1990s, researchers started to explorenano-materials technology to apply in the field of enhanced heat transfer, and were focusing on the research work relating to high efficient heat transfer of cooling technology. Thus, nanofluids with dispersed metal or nonmetal nanopowders into traditional heat transfer medium, and keeping uniform, stable and high thermal conductivity, are used in thermal engineering. More kinds of metal oxide nanopartices are used as the additives of nanofluids. Among the additives, CuO, Al_2O_3, TiO_2 and Fe_3O_4 are most commonly investigated and the transport properties of those nanofluids are more likely to reach to the consensus. In particular, the addition of macro- and micro- sized particles can cause issues like agglomeration, sedimentation, clogging the micro channel, eroding the pipeline, and increasing the pressure drop [5]. Therefore, finding high thermal conductivity additives is a great challenge for many engineers trying to reduce production costs of industrial processes. Graphene, a single- or few- layer planar structure, owns excellent electrical conductivity, thermal conductivity, and an extremely large surface area [12]. Single-layer graphene has been reported an experimental thermal conductivity value of 4800-5300 W/mK at room temperature. The dispersion stability of graphene in water is poor, which has an obvious influence on the heat transfer in applications. Normally, graphene oxide (GO) has excellent dispersion in water, due to the hydrophilic groups on the surface, whereas the thermal conductivity of suspensions is insignificant enhanced, compared with that of base fluids.

The Hummers' method is utilized to prepare graphene oxide, which offers the advantage of simplicity, notwithstanding the longer time needed relative to the Hummers' and modified Hummers' methods [2-5]. This method utilizes two powerful acid mixtures as the oxidizing agent and offers the advantages of high yield and environmental friendliness. Treatment of graphite by this method gives the layered structure of graphene oxide, whereas the interlayer spacing is larger than that in graphite.Graphene oxide films are used in the following applications graphene research, Biomedical, Solar cells, Batteries, Biosensors.

2. PREPARATION

Compared to monolayer graphene that is considered to be a new material, graphene oxide, in the form of sheets of graphite oxide, has been known for more than 150 years, since Brodie attempted to determine the atomic weight of graphite by oxidizing graphite in potassium chloride and fuming nitric acid [7]. Later, Hummers [8] and Staudenmaier[9] modified the method of Brodie in an attempt to speed up the process of graphite oxide production while using less aggressive conditions, such as a mixture of sodium nitrate, concentrated sulphuric acid and potassium permanganate. The Carbon Oxide ratio from both processes is similar (2:1) while they both introduce reactive oxygen functionalities to the original material. The structure of GO has been a subject of debate with respect to the presence and distribution of the oxygen functional groups and its nonstoichiometric atomic composition [10]. The presences of the oxygen groups effects the mechanical and electrochemical properties of GO strongly, compared to graphene. The utilization of these functionalities facilitates the excellent dispersion of GO in in water and different solvents which allows the facile preparation of polymer nano composites and the scale-up process for the production of GO in high volumes. On the other hand, the covalent oxygen functional groups in GO generate structural defects that in turn affect strongly properties such as the electrical conductivity, limiting the utilization of GO in electrically-conductive materials.

2.1.Hummers Method

A graphite oxide was synthesized by hummer's method through oxidation of graphite. A stepwise preparation is given as follows:

1. Graphite flakes (2g) and $NaNO_3$ (2g) were mixed in 15 ml of H2SO4 (98%) in a 1000 ml volumetric flask kept under at ice bath (0 - 5°C) with continuous stirring.
2. The mixture was stirred for 2 hrs at this temperature and potassium permanganate (6g) was added to the suspension very slowly. The rate of addition was controlled to keep the reaction temperature lower than 15°C.
3. The ice bath was than removed, and the mixture was stirred at 35°C until it became pasty brownish and kept under stirring for 2 days.
4. It is then diluted with slow addition of 100ml water. The reaction temperature was rapidly increased to 98°C with effervescence, and the color changed to brown color.
5. This solution was diluted by adding 200 ml of water stirred continuously.
6. The solution is finally treated with 10ml H2O2 to terminate the reaction by appearance of yellow color.
7. For purification, the mixture was washed by rinsing and centrifugation with 10% HCl and then deionized (DI) water several times.
8. Filtration and drying under vacuum at room temperature, the graphene oxide (GO) was obtained as a powder.

2.2.Modified Hummers method

This modified method of synthesis involves both oxidation and exfoliation of graphite sheets due to thermal treatment solution. The stepwise synthesized method is given as follows:

1. Graphite flakes (2g) and $NaNO_3$ (2g) were mixed in 15 ml of H2SO4 (98%) in a 1000 ml volumetric flask kept under at ice bath (0 - 5˚C) with continuous stirring.
2. The mixture was stirred for 4 hrs at this temperature and potassium permanganate (12g) was added to the suspension very slowly. The rate of addition was controlled to keep the reaction temperature lower than 15˚C.
3. The mixture is diluted with slow addition 184 ml water and kept under stirring for 2 hrs. The ice bath was then removed, and stirred at 35˚C for 2 hrs.
4. The above mixture is reflux system at 98˚C for 10-15 min. After 10 min, change the temperature to 30˚C which gives brown colored solution.
5. Again after 10 min, change it to 25˚C , and maintain the temperature for 2 hrs.
6. The solution is treated with 40ml H2O2 by which color changes to bright yellow.
7. 200ml water is taken in two separate beakers and equal amount solution prepared is added and stirred for 1 hrs.
8. It is then kept without stirring 3-4 hrs, where the particles settles at the bottom and remaining water is poured to filter.
9. The resulting mixture is washing repeatedly by centrifugation method with 10% HCl and then deionized (DI) water several times until it forms gel like substance (pH – Neutral).
10. After centrifugation the gel like substance is vacuum dried at 60˚C for more than 6 hrs to GO powder.

3. CHARACTERIZATION

3.1. XRD Analysis

The X-ray diffraction (XRD) is the most widely used technique for general crystalline material characterization. It is used to Measure the average spacing's between layers or rows of atoms, determine the orientation of a single crystal or grain. The XRD pattern obtained for as synthesized GO nanoparticles by Hummer's method is shown in Figure2.

It shows the diffraction peak at $2\theta=10^{\circ}$, which is mainly due to the oxidation of graphite. The diffraction peak of pure graphite is found around 26◦, corresponding to the highly organized layer structure with an interlayer distance of 0.34 nm along the (002) orientation is shown as inset in Figure 2. The XRD pattern for synthesized GO by Modified Hummer's method is shown in Figure3.

The disappearance of the peak at 26◦and appearance of the peak at 10◦, shows that the product is completely oxidized after the chemical oxidation and exfoliation, indicating an increase in d-spacing from 0.34 nm to 0.82 nm.

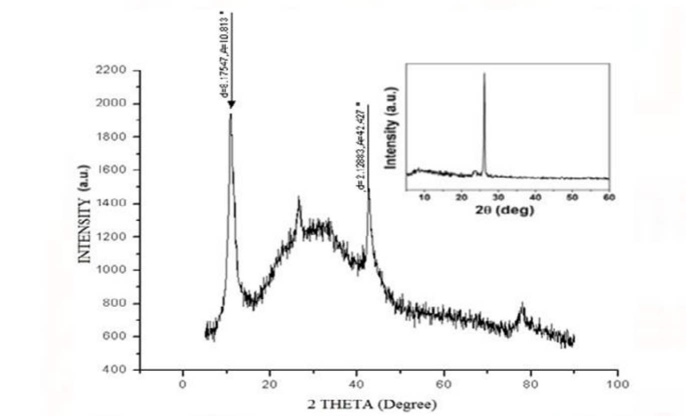


Figure 2: XRD of GO by Hummer's method (inset shows XRD of graphite).

3.2. FT-IR analysis

It is a technique adopted to obtain an infrared spectrum of absorption, emission, and photoconductivity of a solid, liquid or gas. Also it can be utilized to quantitative analysis of an unknown mixture. FTIR measurement was employed to investigate the bonding interactions in graphene before and after the oxidation process. It assumes the intensities of the peaks are directly related to the amount of sample present.

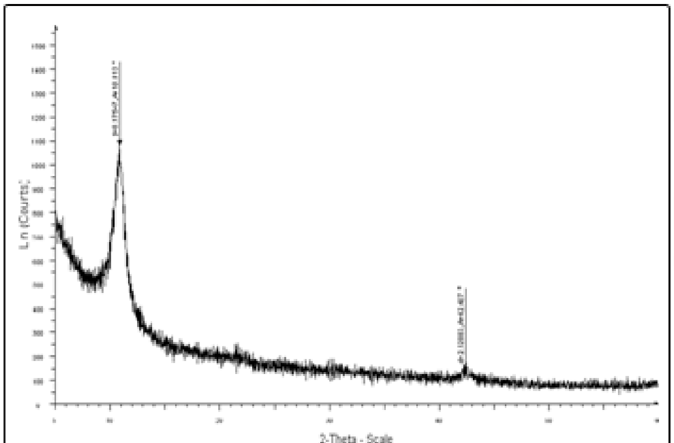


Figure 3: XRD of GO by Modified Hummer's method.

Figure 4 shows that synthesized GO has a peak at 1081 cm-1 which is attributed to the C-O bond, confirming the presence of oxide functional groups after the oxidation process. The peaks in the range of 1630 cm-1 to 1650 cm-1 show that the C=C bond still remained before and after the oxidation process. The absorbed water in GO is shown by a broad peak at 2885 cm-1 to 3715 cm-1, contributed by the O-H stretch ofH2Omolecules can be formed.

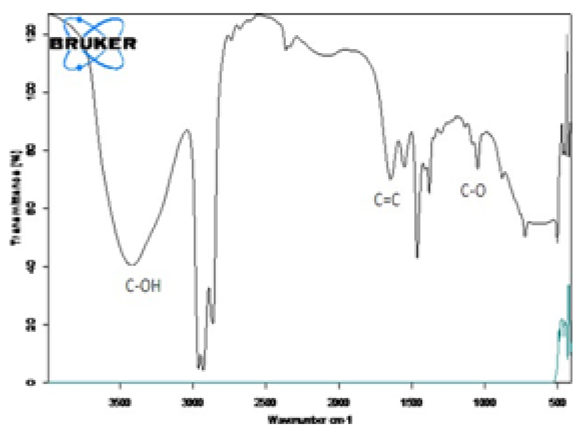


Figure 4: FT-IR spectrum of

3.3.SEM analysis

Scanning Electron microscopy provides morphology and structure of nanomaterials. Figure 5a shows the SEM image of typical graphite. From SEM image it is clear that how the sheets are stalked together in Figure5a.Figure5bshowstheSEMimageofexfoliatedG O.Itclearly shows that how the graphene sheets are exfoliated.

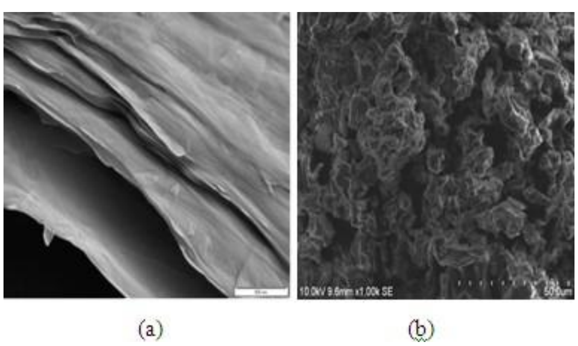


Figure 5: SEM images of (a) Graphite and (b)

3.4.FESEM analysis

The grain size and surface morphology were observed by the field emission scanning electron microscope (FESEM). FESEM images of the Graphene Oxide (GO) have well defined and interlinked three-dimensional Graphene sheets, forming a porous network that resembles a loose sponge like structure as shown in Figure 6.

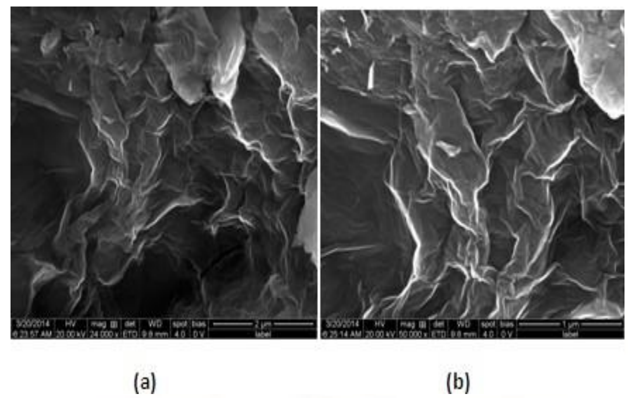
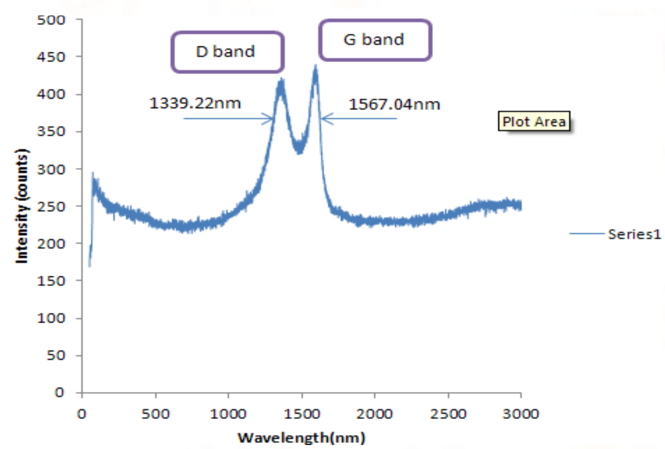


Figure 6: FESEM images of GO at magnification (a) 24000 and (b) 50000.

3.5 Raman spectrum analysis

Raman spectroscopy is a widely used tool for the characterization of carbon products, especially considering the fact that conjugated & double carbon-carbon bonds lead to high Raman intensities. Figure 7 shows the Raman spectrum of GO, where the in-phase vibration (G band) of GO is at 1567.04 nm and the disorder band (D band) of GO is at 1339.22nm.



4. Metal/Metal Oxide Synthesis Using Green Approach

Plant extracts is a reducing agent it as very cheap and non-toxic. Different metal oxides synthesis using various plant parts such as a (flower, leaf etc.) green synthesis metal and metal oxides applied to thermal conductivity applications used recent researchers.[14]

4.1. Preparation of Metal oxide nanofluids:

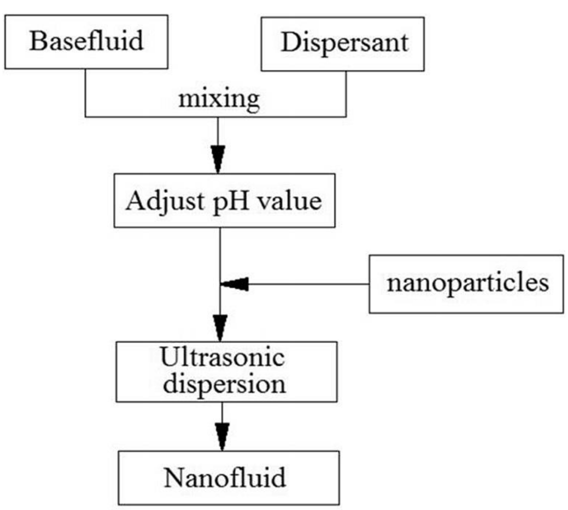
Preparation of Nano fluids is a prerequisite for their heat transfer application. Generally, there are primarily two preparation methods viz. one-step and two-step methods for nanofluids.

One-Step Method:

It is actualized by dispersing nanoparticles into base fluids simultaneously during the manufacturing process of nanoparticles.

Two-step Method:

The manufacturing and dispersing processes of nanoparticles are implemented separately. Cooling fluids or chemical reagent solutions in one-step method are generally not the required based fluids for nanofluids, such as water or oil, or refrigerant etc. Therefore, one-step method is not very appropriate for preparing heat-transfer nanofluids but mostly applicable to produce dry nanoparticles. On the other hand, two-step method has been extensively applied in preparing Metal Oxidenano fluids for heat transfer use, because metal oxide nanoparticles synthesis technique has already achieved industrial production scale. A typical procedure of two-step method of preparation of nanofluids is showed in As a result of the high strong particles interaction force, high specific surface areas and surface activity of particles in nanoscale, a strong tendency towards colliding and aggregating for nanoparticles is invariably existent. Therefore, A typical procedure of two-step method in the preparation of in general, some extra dispersion means are adopted to achieve better stabilities and availabilities for nanofluids.



5. CONCLUSION

- Nano fluid application used metal/metal oxide synthesis using green approach as good alternative for physical and chemical methods.
- Green synthesis metal and metal oxide Nano composite preparation very quickly at room temperature
- This green synthetic approach has advantages over conventional methods involving chemical agents such as sodium borohydride or hydrazine hydrate.

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