Synthesis and Ultrasonic Investigation of Reduced Graphene Oxide Nanosuspension with Water

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Abstract: Synthesis of Graphene Oxide (GO) was done using Original Hummer’s Method. The prepared GO specimen was reduced further with Hydrazine monohydrate to create Reduced Graphene Oxide (RGO). RGO were evaluated using characterization techniques such as FESEM, EDS, FTIR, XRD, and Raman. These techniques help to confirm the production of GO and conversion of GO into RGO. Suspension of RGO in double-distilled Water with different concentrations was produced using ultrasonication for 24 hours. This suspension was further tested with the help of the UV-Vis technique to confirm the presence of RGO in the suspension. DLS was done to confirm the nano dimensions of RGO particles in the water solution. Ultrasonic velocity, density, and viscosity were measured for all prepared nanosuspensions. The acoustical parameter such as adiabatic compressibility, intermolecular length, and acoustical impedance was calculated from experimental data.

Key Words: Bulk Modulus, Adiabatic compressibility, Nanosuspension, Graphene, Reduced Graphene Oxide, Graphene Oxide, Attenuation, FESEM, Density, XRD, EDS, RAMAN, FTIR, DLS, UV-Vis, Ultrasonic velocity, Density, Intermolecular free length, Relaxation time, Attenuation, nanofluids, Acoustical impedance, , temperature, Viscosity.

1. Introduction
Nanosuspensions are the colloidal dispersion of nano-sized particles ranging from 100 – 500 nm in the base fluid [1]. The nanoparticles used in the nanosuspension are typically made out of metals, oxides, carbides or nanotubes of carbon. As we know heat transfer takes place on the surface of the particle so for the better heat transfer application, nanosized particles can play a very vital role because of extremely high surface area available in these particles. Nano-sized particles have a high thermal conductivity that can be used in various fields like electronics where dissipation of heat from the electronic components is always a challenge [2][3][4]. Another potential area can be the use of these particles in ink for electronic device printing for the production of flexible and more conductive
electronics devices [5]. The choice of the base fluid in these nanosuspensions can further increase the properties e.g. the use of water as a base fluid can help to increase the process of heat exchange as water is one of the best coolant available. But, the main objective and goal in nanosuspension research are to develop nanosuspension with stability for industrial applications [1] [6]. Ultrasonication is an accepted technique for dispersing aggregated nanoparticles for the preparation of aqueous nanosuspensions [7]. It has been found in the literature that the increase in the stability of nanosuspension with the increase in the sonication time. Stabilization of the nanosuspension also depends on the choice of the nanoparticle and the choice of sonication time. So, the first challenge is to prepare stable nanosuspension which can be used for industrial applications.

Graphene is one of the greatest finds in the last decade in terms of many physical and chemical properties. Since its evolution number of properties like high electron mobility, high electron density, high thermal conductivity, the strongest material, etc [8] [9] [10]. Despite all these good properties there are some difficulties associated with this material like industrial-scale productivity and preparation of the good stable suspension. Many methods have been reported by a different peer group for the production of graphene (G), graphene oxide (GO) and reduced graphene oxide (RGO) and the stable suspension [11].

In this paper we prepared RGO nanosuspension with different concentrations in water. Structural, morphological and fluid interaction studies have been completed and compared. Intermolecular interaction between RGO-Water nanosuspension was observed at different concentrations and temperatures in the range of 298 to 313 K. The adiabatic compressibility, acoustic impedance, mean free pathway were evaluated and examined by ultrasonic approach. Nonetheless, the attenuation coefficient of the ultrasonic wave propagating through all specimens is calculated.

2. Material and method:

2.1 Materials

Graphite powder, sodium nitrate obtained from Loba Chemical, KMnO₄, HCl, and Ammonia was taken from CDH, and H₂SO₄ was obtained from Avantar. H₂O₂ was obtained from a fisher scientific, Hydrazine monohydrate was obtained from Sigma Aldrich with an AR grade. All the chemicals used in the work are AR grade. Double distilled water was used throughout the experiment.

2.2 Methods

Original Hummers Method is used to prepare GO. In 1000 ml beaker 345 ml of H₂SO₄ has been taken and put to the continuous magnetic stirrer [12]. An ice bath has been made around the beaker to
maintain the temperature of the bath below 10 °C. 15 gm of Graphite Powder and 7.5 gm of NaNO₃ has been added to continuously stirring H₂SO₄ with the temperature of the bath is close to zero Celsius. After 30 minutes of continuous stirring 45 gm of KMnO₄ has been added to the mixture very slowly such as to maintain the reaction temperature below 90 °C. The mixture is left for continuous stirring for 2 hours after that beaker has been placed in a water bath at the temperature of 40 °C for 1 hour to increase the rate of oxidation. The color of the mixture changes from black to greenish-brown. After 1 hour the mixture is placed on the magnetic stirrer and ice bath. With continuous stirring, the mixture is diluted with 500 ml of distilled water very slowly maintaining the reaction temperature below 90 °C. After leaving the mixture overnight distilled water 100 ml with 30% H₂O₂ added to the mixture to remove any excess KMnO₄ and to stop the oxidation reaction. The further mixture was diluted with distilled water and left for the decantation process overnight. Then we remove the top layer of water and the remaining solution is filtered. Continuous addition of distilled water has been added during the filtration process helps to remove excess KMnO₄ and metal ions. Approximately 5 liters of distilled water were used during the filtration process. Following completion of the filtration, the processed mixture was again mixed with 500 ml of distilled water and added to 8-hour ultrasonication, which helps to reduce GO. The mixture was left to dry in the vacuum and to grinded for GO powder. A colloidal suspension of GO powder in double distilled water (2 mg / ml) is prepared by ultrasonication of GO in 500 mL of liquid for 1 hour. A few drops of ammonia have been added to the solution to adjust the PH of the solution in a range of 9 to 10. Hydrazine monohydrate (1 μl for 3 mg of GO) was then added to the suspension. Continuous magnetic stirring and temperature maintenance up to 95 °C for 3 hours yielded black precipitate of RGO powder [13]. After cooling to room temperature, the powder was filtered through the filter and the vacuum was dried. Synthesized RGO nanoparticles are dispersed in double distilled water by ultrasound for 24 h in order to obtain a homogenous suspension of different concentrations without any phase separation and sedimentation. As with many peer groups, it is indisputable that ultrasound improves reliability and dispersion of the suspension [7]. Nanosuspensions with different concentrations (0.2mg / ml, 0.4mg / ml, 0.6mg / ml, 0.8mg / ml and 1.0mg / ml) are prepared without the introduction of any surfactants [14].

2.3 Characterization of RGO and RGO-Water nanosuspension

Both FE-SEM Imaging and elemental analysis has been done with the help of Nova Nano FE-SEM 450, which provides an ultra-high-resolution description and nanoscale information of the sample. The FE-SEM is attached with an EDAX detector for the measurement of the elemental and chemical composition of materials. The surface morphology of RGO particles is analyzed with FE-SEM operating at 15 KV. FE-SEM is the primary test to see the surface morphology of the RGO samples.
EDS helps to understand the chemical composition of the RGO samples. Which gives an insight into the chemical composition of the sample under study. FTIR spectra were recorded using Shimadzu 8400S spectrometer in the range of wavenumber 400 to 4000 cm\(^{-1}\) using a He-Ne laser. It is a Pre-Primary test to detect the skeleton vibrations of C=C, which presents in Graphene. XRD of RGO sample was performed using a Panalytical X’Pert Pro instrument which uses Cu-K\(\alpha\) radiation of wavelength 1.54 Å. The increment in the scan speed is taken as 0.02 s and reading is taken for 20 ranging from 0° to 60°. It is one of the most important tests to see the proper removal of oxygen from GO samples. Raman Spectra of powder RGO sample is taken with the help of Airix STR 500 confocal Raman Spectrometer which uses a solid-state laser beam of 532 nm at 30mW. It is the primary test to know the structure retention by the RGO samples after the chemical reduction of RGO from GO. UV spectra of the prepared nanosuspension taken with the help of Shimadzu UV-1800 spectrometer. 20 µl of prepared RGO-EG suspension is diluted with 4 ml of pure EG in the quartz cuvette for the UV spectrometry. The range of wavelengths for the study is between 200 nm to 600 nm. All 5 samples are prepared and measured in the same conditions. This test is used to verify the presence of RGO in the suspension. The average diameter of RGO in water is obtained using a dynamic light scattering (DLS) instrument by Malvern Zetasizer Nano ZS90. This test is used to find the count and size of the particles present in the suspension. Thermoacoustic properties of the sample were measured with the help of Mittal Interferometer M – 81 with a fixed frequency of 2 MHz for ultrasonic velocity, pycnometer for density measurement and LABMAN model of LMDV-200 for viscosity measurement with attached constant temperature digital water bath.

3. Data Table:
| Concentration (mg/ml) | Temperature (T) (K) | Average Velocity (U) (m/s) | Density (ρ) (Kg/m³) | Viscosity (η) (Pa-s) | Adiabatic Compressibility (β) (1/Pa) |
|-----------------------|---------------------|-----------------------------|---------------------|---------------------|--------------------------------------|
| 0                     | 299                 | 1498.461538                 | 997                 | 0.00089             | 4.47E-10                             |
| 0.2                   | 299                 | 1501.62                     | 1003.5              | 0.015742            | 4.42E-10                             |
| 0.4                   | 299                 | 1503.69                     | 1002.3              | 0.01587             | 4.41E-10                             |
| 0.6                   | 299                 | 1506.69                     | 1000.0              | 0.016212            | 4.40E-10                             |
| 0.8                   | 299                 | 1508.77                     | 1000.2              | 0.0163778           | 4.39E-10                             |
| 1                     | 299                 | 1510.77                     | 998.5               | 0.01631             | 4.39E-10                             |
| 0                     | 304                 | 1507.692308                 | 995.4               | 0.000797            | 4.42E-10                             |
| 0.2                   | 304                 | 1509.85                     | 1002.3              | 0.015044            | 4.38E-10                             |
| 0.4                   | 304                 | 1512                        | 1001.1              | 0.015122            | 4.37E-10                             |
| 0.6                   | 304                 | 1515.15                     | 999.8               | 0.015271            | 4.36E-10                             |
| 0.8                   | 304                 | 1518.08                     | 998.8               | 0.015482            | 4.34E-10                             |
| 1                     | 304                 | 1520                        | 997.5               | 0.01545             | 4.34E-10                             |
| 0                     | 309                 | 1516.1500                   | 993.8               | 0.000719            | 4.38E-10                             |
| 0.2                   | 309                 | 1518.23                     | 1000.9              | 0.014411            | 4.33E-10                             |
| 0.4                   | 309                 | 1521.23                     | 1000               | 0.014711            | 4.32E-10                             |
| 0.6                   | 309                 | 1524.23                     | 999.01              | 0.014914            | 4.31E-10                             |
| 0.8                   | 309                 | 1526.15                     | 998.1               | 0.015075            | 4.30E-10                             |
| 1                     | 309                 | 1528.08                     | 997                | 0.01493             | 4.30E-10                             |
| 0                     | 314                 | 1525.3100                   | 992.2               | 0.000653            | 4.33E-10                             |
| 0.2                   | 314                 | 1528.46                     | 1000.1              | 0.013828            | 4.28E-10                             |
| 0.4                   | 314                 | 1531.31                     | 998.8               | 0.01443             | 4.27E-10                             |
| 0.6                   | 314                 | 1535.46                     | 998.2               | 0.014362            | 4.25E-10                             |
| 0.8                   | 314                 | 1537.38                     | 997.3               | 0.014775            | 4.24E-10                             |
| 1                     | 314                 | 1538.38                     | 996.5               | 0.014529            | 4.24E-10                             |
4. Result and Discussion

4.1 Morphological and elemental analysis

Figure 1 corresponds to the FESEM image of the RGO sample at 50000 X magnification. In these images, lamellar morphology with few-layered structures is clearly visible. Wrinkles and separated layers at the scale of 2µm confirm the formation of RGO and conversion of GO into RGO respectively. The EDS graph of RGO are shown in figure 2. It is clearly visible from the graph that carbon content is 86.35% and oxygen content is 8.56%. The reduced amount of oxygen content shows the complete removal of oxygen content while reducing the sample from GO to RGO. It proves the conversion of GO into RGO [15][16].
Figure 3 corresponds to the FTIR spectrum of RGO. Peaks at 923.93, 1234.48 correspond to alkoxy C—O stretching vibration and C—O epoxy stretching vibrations. 1539.24 correspond to C=C Skelton vibrations of Graphene network, 2341.66 correspond to CO2 respectively [17][18][19]. XRD spectra of RGO is shown in figure 4. The strong peak at 26.43 in figure 4 contributed to the graphene-like structure (002) and the complete removal of oxygen content from RGO. The peak of RGO at angle 26.43 confirms the removal of trapped oxygen from layers of Graphene by hydrazine monohydrate and a decrease in the interplanar distance between layers [20][21][22]. Figure 5 corresponds to the Raman spectroscopy graph of the RGO samples.
In figure 5 the peak at 1336.38 correspond to defect in Graphene network, 1565.96 correspond to Graphene Network peak and 2682.66 correspond to 2D defect peak in graphene network. The intensity ratio of structure defect and Graphene network ratio is $I_D/I_G = 0.41$ which corresponds to good structure retention of the graphene network [23][24][25].

4.2 Colloidal Stability analysis

The nanosuspension of RGO with water was prepared with the help of extensive ultrasonication. The UV-Vis spectra are shown in figure 6 confirms the presence of RGO in the nanosuspension. The peaks at the position 268 nm represent the presence of RGO in the nanosuspension [26][12].

The average diameter of RGO in water is obtained using a dynamic light scattering (DLS) shown in figure 7. It is seen that the average size of the RGO is in the range of 109 nm to 119 nm for all 5
prepared samples [16][27][28]. With the help of particle size calculation by DLS, we can call the prepared suspension as nanosuspension.

4.3 Thermoacoustic analysis

4.3.1 Ultrasonic Velocity

Ultrasonic velocity is the most important parameter to understand the intraparticle and intermolecular interaction in the nanosuspension samples. The ultrasonic velocity of double distilled water and their solution were measured at four different temperatures (298, 303, 308 and 313K) and at 1atm pressure. The velocity, density, and viscosity reading were repeated several times for accuracy. All the reading for pure water was matched with literature to avoid any error. The dissimilarity of ultrasonic velocity with the concentration of the nanosuspension and temperature helps to find the insight of the fluid. Figure 8 displays the dissimilarity of ultrasonic velocity with the concentration of RGO in water.

Ultrasonic velocity increases in water-RGO nanosuspension with the rise in the concentration of the particles. The upsurge in the ultrasonic velocity has been accredited to water-RGO interaction and it further guarantees the dominance of intramolecular interaction over intermolecular interaction. With the rise in the concentration of the particles, there is a possibility of an increase in the Brownian motion of nanosuspension molecule and an increase in the surface layer which further helps to increase the ultrasonic velocity. An increase in the temperature of the nanosuspension also increases in the Brownian motion of the fluid molecules which further increase the velocity of the fluid [29][30].

4.3.2 Density

As seen in figure 9 as soon as RGO particle added to the pure water density increases from 997 to 1003.5 m/s in case of RGO nanosuspension for 0.2mg/ml. After this continuous decrease can be seen
in the density of the prepared nanosuspension with an increase of particle concentration. This is due to an increase in intramolecular interaction than intermolecular interaction. Water-RGO shows the same decreasing trend. Density declines with the increase in the temperature of both nanosuspensions [31][32].

4.3.3 Viscosity

Figure 10 shows the viscosity of Water-RGO nanosuspension declines with the rise in the temperature because of the increase in the Brownian motion of the molecules of water. With the increase in the temperature flow of nanosuspension also gets easy. Viscosity also increases slightly with the increase of particle loading in the nanosuspension which may be due to restriction in the flow produced by the RGO nanoparticles. These particles get tangled with each other to restrict the flow of liquids [2][30].

4.3.4 Adiabatic Compressibility

Figure 11 displays the decrease of adiabatic compressibility with the rise in the concentration of the particles. The decrease in the adiabatic compressibility with a rise in the concentration of the particles supports fluid-particle interaction. Further, adiabatic compressibility also decreases with an increase in the temperature of the system [33][34].

4.3.5 Acoustic Impedance

In figure 12 there is an increase in the value of acoustical impedance in the nanosuspensions with the rise in the concentration of the particles. The higher values of the acoustical impedance suggest that there is a substantial interaction between the nanoparticles and base liquid molecules which may affect the structural arrangement. On the other hand, the acoustical impedance of both nanosuspensions declines with the rise in the temperature that may be due to an increase in the Brownian motion of the
base liquid molecules. The increasing particle-fluid interactions at lower concentrations increase the intermolecular distance which creates the resistance in the propagation of ultrasonic waves [35][36].

4.3.6 Ultrasonic attenuation

Figure 13 represents the attenuation of the ultrasonic waves while passing through prepared nanosuspensions. Ultrasonic attenuation increases dramatically with the addition of the nanoparticle to the base fluid because the network of these particles may block most of the ultrasonic waves from reaching to the other end. Further, the attenuation increases very slightly with the increase of concentration of the particles. Further, the dependency of attenuation on temperature can be seen in both prepared nanosuspension. It can be seen that as the temperature rises the value of the attenuation declines because with temperature the Brownian motion the base fluid increases [37] [38].

4.3.7 Bulk Modulus:

Bulk modulus Can be defined as the resistance of the liquid or fluid to the applied stress. In figure 14 bulk modulus increases for the Water-RGO nanosuspension with the increase in the concentration of the particles. Bulk modulus also increases with the rise in temperature. An rise in bulk modulus or a decrease in compressibility is attributed to the fact that powerful, cohesive interaction forces work among molecules / atoms after the dispersion of RGO nanoparticles in liquid [39] [40].
4.3.8 Relaxation Time

Figure 15 represent the relaxation time of the water-RGO nanosuspension. Relaxation time is the molecular rearrangement time during the process of transmission of ultrasonic waves. It can be seen clearly in figure 15(a) and 15(b) that relaxation time increase from $5.3 \times 10^{-13}$ s to $9.28 \times 10^{-12}$ s because of the addition of RGO nanoparticles in the pure water. Further, a slight increase in the relaxation time is seen in both nanosuspension. Relaxation time decreases with the increase in the temperature as the Brownian motion of the water molecules increases with the rise in the temperature [40] [41].

4.3.9 Intermolecular free length

In figure 16 it can be understood clearly that intermolecular free length is a function of temperature. As the temperature rises the intermolecular free length declines as the relaxation time increases. Further, the intermolecular free length decreases with the rise in the concentration of the nanoparticles in the base fluid. This behavior is the response of fluid and particle interaction in the prepared nanosuspension [35] [36].
5. Conclusion

RGO was prepared with Original Hummer’s Method. Various characterization techniques like FESEM, XRD, FTIR, EDS, and RAMAN were used to confirm the conversion of GO and RGO. Prepared GO and RGO powder were dispersed directly into distilled water using the ultrasonication technique. Samples with various compositions were prepared for ultrasonic studies. The ultrasonic study of Water-RGO nanosuspension shows the dominance of intramolecular interaction over intermolecular interaction. The ultrasonic velocity increases with an increase in concentration which is due to the decrease in intermolecular free length of the mixture. The density of both of the nanosuspension decreases and viscosity increases with an increase in the concentration of RGO particles. Adiabatic compressibility, intermolecular free length, decreases with an increase in concentration. This decrease in acoustical parameters indicates that there is a weak interaction between the molecules of the Particles. Acoustical impedance, Bulk Modulus, attenuation, and relaxation time shows an increase with the increase in RGO concentration, which indicates the absence of complex formation in the mixture.

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