INFLUENCE OF VOLUME CONCENTRATION RATIO ON THERMOPHYSICAL PROPERTIES OF GRAPHENE OXIDE-WATER BASED NANOFLUID

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Abstract.
This article sheds light upon the ideology of preparation and thermophysical properties of graphene oxide (GO)-water based nanofluid which is being used for performance evaluation for photovoltaic thermal system. GO in the form of powder or nanosheets were used to make tough composite materials, thin films and membranes. The nanofluids were prepared by two steps technique. Nanofluid with different volumetric concentration ratios specifically 0.1%, 0.2%, 0.3%, 0.4%, 0.5% and 0.6% were prepared with the aid of sonication and stirring process. X-ray diffraction analysis (XRD) and Raman analysis were conducted to investigate the structural characteristics of graphene oxide-based materials. The stability of nanofluids was assessed using zeta-potential test, high stability after 41th day and moderate to good stability after 90th day was found. Rheological behaviour results indicate that dynamic viscosity decreases with rise in temperature, but it increases with concentration ratio. Thermal conductivity measurement of the nanofluid was carried out in the temperature of 25 ^{0}C to 55 ^{0}C for volume concentration ratio ranges of 0.1% to 0.6%. The results revealed that the thermal conductivity of the nanofluid increased with both rising volume concentration and temperature. Although nanoparticle concentration has a greater impact than temperature, changes in thermal conductivity are more pronounced at higher temperatures. The enhancement in thermal conductivity of nanofluid was appeared about 7.8% to 11.58 %. overall drop of specific heat capacity up to 3.65%. Density of nanofluid is closest value to the density of most of base fluids, hence it provides the higher stability relative to other types of nanoparticles. There was adverse effect (12.5% increase) on viscosity has been reported.

Keywords:
Graphene oxide (GO), Nanofluid, Volume concentration ratio, Thermal conductivity, Dynamic viscosity.

1. Introduction
In recent decades, researchers have focused on developing the most efficient, effective and compact heat transfer equipment's. [1]. Heat transfer operations plays a very crucial role for different energy conversion and transport processes using heat transfer fluids (HTF). Despite modern technologies and multi-tasking system incorporated in the energy sector, scientist and engineers facing diverse types of problems, among that lesser thermal efficiency is the most common [1-5]. The low thermal efficiency of the heat exchanging devices is due to low thermal conductivity of heat transfer fluids [3]. To enrich the heat transfer aspects and to enhance the thermal performance methods such as waste heat recovery, material modification, use of extended surfaces, process parameters optimization has been attempted [7]. It has been observed that thermo physical behaviour of heat transfer fluids plays a vital role in the heat exchange processes. Water/steam is cheap, easily available and compatible with many heat exchange processes, but highest thermal conductivity is 0.62 W/mK, which is lower compared to metals [6]. Many research studies focuses on improvement of weak heat transfer ability of heat transfer fluid by dispersing higher thermal conductivity nanomaterials in conventional base fluids, known as nanofluids. Choi, Eastman et al. were pioneers in this field [9]. Since invention of nanoparticles, wide variety of nanofluids along with hybrid nanofluids have become prominent research domains. It has been observed that wide range of nanostructures more specifically nanoparticles have been

investigated as additives to a wide range of base fluids. This includes metallic nanoparticles (Zno, Al_2O_3, CuO, etc.) which have higher thermal conductivity. However, their availability and associated cost were significant challenges [11]. Graphene, a new form of an allotropy of carbon with a two-dimensional structure is attracting the significant researchers due to its outstanding properties [12]. Graphene structure ideally envisaged as a single-atom-thick sheet of hexagonally arranged, sp^2 bonded carbon atoms tightly packed into a honeycomb lattice [14]. Thermal conductivity of graphene was observed about 5000 W/mK which is much higher than the value presented by other carbon allotropes like MWNT, SWNT or diamond. Graphene Oxide (GO) is a mixture of C, O_2 & H group. GO sheets being used to make tough composite materials, membranes and thin films [16]. Compared to other nanoparticles graphene nanosheets possess larger surface areas, which allows bigger contact interfaces with the base fluid. This superior contact reduces Kapitza resistance at the graphene-fluid interface and consequently, helps to improve the effective thermal conductivity of the nanofluid [15]. Another advantage of graphene nanomaterial is relatively easy and cost-effective production at a large scale. Despite graphene's possess good material properties, it's use in heat transfer systems is limited [17]. This article focuses on preparation methods of nanofluids, surface morphology, stability of nanofluids, effects of concentration ratio on thermophysical properties like, thermal conductivity, density, specific heat, Viscosity etc.

2.Experimental section

2.1 Materials: Nanomaterial i.e. graphene oxide (GO) in the form of nanosheets was used in this experimental study. This material was purchased from PranaGraf Materials & Technologies Private Limited, Mumbai in the powdered form. Table 1 shows the properties of Graphene oxide nanoparticles.

Table 1. Properties of Graphene oxide nanoparticles.

Sr. No.	Parameters	Specification
1	Purity	99.99%
2	Colour	Black
3	Form	Powder
4	Bulk Density	0.39gm/cc
5	Thickness	0.5-0.6nm

Distilled water being used as base fluid for preparation of nanofluids of different volume concentration. Thermophysical properties of distilled water at 25°C, are as follows: density: 998 kg/m^3, specific heat: 4200 (J/kg.K), thermal conductivity: 0.607 (W/m.K), and viscosity: 0.89 (cP).

2.2 Nanofluid preparation: Carefully selection of methods for nanofluids preparation is very much important, as it may create direct impact on their characteristics. There are two most famous methods used for preparation of nanofluids i.e. one-step and two step preparation method. In one-step preparation of nanofluids several techniques can be employed such as direct evaporation, physical vapor deposition and chemical vapor deposition. Even though the one-step method provides uniform dispersion and good stability of nanoparticles in the base fluid, this method is limited to certain nanoparticles and base fluid, also it is costlier one. On the other hand, the two-step method of preparation of nanofluid covers wide range of nanoparticles. In this method the nanoparticles synthesized separately and then dispersed in the base fluid by violent agitation [20]. During these experimentation two-step preparation approaches was employed. Nanomaterial in the form of GO nanosheets was purchased from PranaGraf Materials & Technologies Private Limited, Mumbai. Graphene oxide (GO) was synthesized by chemical exfoliation of the graphite powder by a modified Hummers method. While distilled water used as base fluid. Before preparation of nanofluid samples, different volume concentration ratios for mixture graphene oxide-distilled were computed from Equation (01).

$$\emptyset = \left[\frac{\left(\frac{w}{\rho}\right)GO}{\left(\frac{w}{\rho}\right)GO + \left(\frac{w}{\rho}\right)water} \right] \qquad [1]$$

The volume concentration ratio of 0.1%, 0.2%, 0.3%%, 0.4%, 0.5% and 0.6% were obtained by considering 500ml water as base fluid for different weights (mg) of GO nanomaterial. GO powder was measured by digital Weighing Lab Balance with 1-mg precision, then these GO nanoparticles dispersed in the distilled water as a base fluid by proper dispersion approach. This mixture was stirred by magnetic stirrer (RCT basic IKAMAG® safety control, Staufen, Germany) for 30 min. at 45 ^{0}C and 500 rpm. After that this mixture was homogenized by ultrasonication to overcome the problem of agglomeration and aggregation. The sonication was performed for another 45 min using 6-liter ultrasonic bath (KQ221D Sonicator, 400W, 24 kHz, Hielscher Company, Germany), Proper dispersion and homogeneity were ensured to achieve good stability.
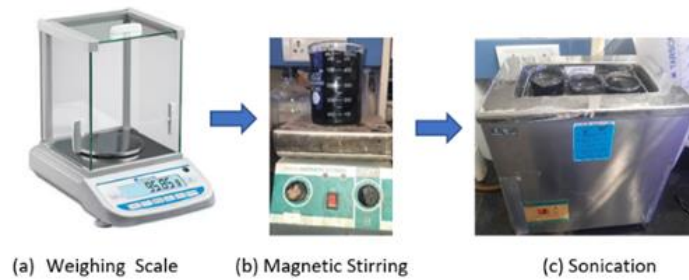

(a) Weighing Scale   (b) Magnetic Stirring   (c) Sonication

Figure 1. Nanofluid preparation steps

Fig.1 shows the step by step approach used while preparation of nanofluid samples.

2.3 Characterization: Thermophysical property analysis of graphene oxide based nanofluid were carried using different tools. XRD was performed using D8 Advance, Bruker, X-ray diffractometer. While morphology was examined using field emission electron microscopy (FESEM, SUPRA-52, ZEISS, Germany), structural characteristics of GO materials examined using Raman spectroscopy (Renishaw, GmbH, Pliezhausen, Germany). Stability was assessed after 41th day and 90th day using Zetasizer, ver. 7.13, Malvern Instrument. Hot Disk, Thermal analyzer, (TPS 2500S, K-Analysis AB Pvt Ltd Sweden) was used to measure the thermal conductivity and specific heat capacity, Viscosity was measured as a function of temperatures using viscometers (DV2THA, Brookfield, USA).

Figure 2. Image of Nanofluid Samples

3. Results and Discussion:

3.1 X-ray diffraction analysis: XRD technique was used to investigate the crystalline structure of the GO samples. A typical XRD pattern of GO represented in Fig. 3. It displays the characteristic diffraction peak located at $2\theta = 11.66°$, which was attributed to the (001) crystalline plane. The relative inter-layer spacing (d_{space}) of powdered samples were calculated using Eq. (1) below:

$$d_{space} = \frac{\lambda}{2\sin\theta} \qquad [1]$$

Where, λ is the X-ray source wavelength, and θ is half of the corresponding peak diffraction angle. The intense peak was recorded as the (002) peak for graphite, and the subsequent inter-layer spacing was calculated to be 0.359 nm, which is likely to the inter-layer spacing for pure graphite (0.334 nm). The larger inter-layer spacing of 0.758 nm for the GO sample results from the inclusion of oxygen-bearing functional groups and water molecules between the graphene layers produced during oxidation. The difference in 2θ (10°–12.5°) also represents the variation in the amount of oxidation. The average crystalline size or perpendicular dimension ($D_{(hkl)}$) that contains the graphitic sequence is expressed by the Debye–Scherer equation as follows (2)[21]

$$D_{(hkl)} = \frac{k\lambda}{\beta Sin\Theta_{(hkl)}} \qquad [2]$$

Where k is crystallite shape constant (0.89) and β is FWHM (i.e. Full Width at Half Maximum of the peak at the maximum intensity), θ(hkl) is peak diffraction angle.

An estimate of the mean GO crystallite size for the (001) peak was calculated to be 12.53 nm. The average number of graphene layers (n) per domain were calculated as 18 using results produced from Eqs. (1) and (2) using the expression (3) below:[21]

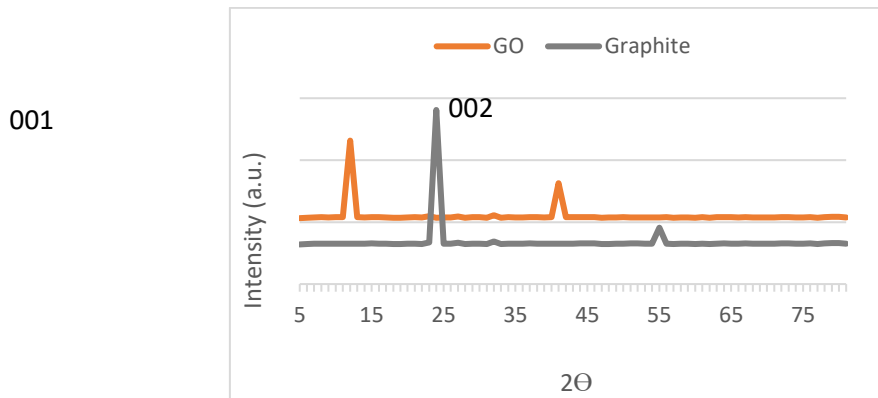$$n = \left(\frac{D_{(hkl)}}{d_{(hkl)}} + 1 \right) \qquad [3]$$

Figure 3. XRD Analysis of GO

3.2 Raman spectroscopic Analysis: Raman spectroscopy is an extensively used technique to investigate the structural characteristics of graphene-based materials. Raman spectrum of graphene oxide (GO) nanosheets illustrated in Fig.4. GO sample spectra is characterised by two main prominent peaks, G band (1575 cm^{-1}) and disorder-induced D band (1217 cm^{-1}). The G band is always lying between 15001 and 1630 cm^{-1} for all poly-aromatic hydrocarbons and results from the in-plane vibration of sp^2 carbon atoms (E$_2$g symmetry mode) while the "D band" associates with the defects in the graphite lattice. The G band in graphene oxide shifted towards higher wavenumber which is due to the oxygenation of graphite which results in the formation of sp^3 carbon atoms. The D band in GO is broadened due to the reduction in size of plane sp^2 domains by the creation of defects, vacancies and distortions of the sp^2 domains during oxidation.
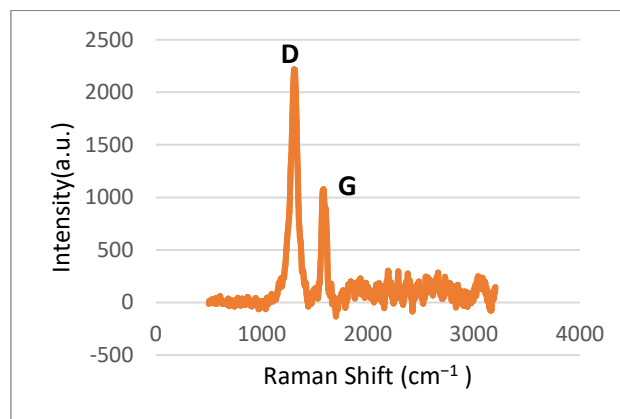
Figure 4. Raman Spectra of GO

3.3 Scanning Electron Microscopy (SEM):

The Surface morphology of GO was studied by using field emission scanning electron microscope (FESEM). To inspect the layered structure of material significant number of layers were examined. Thickness of structure layer was recorded about 10nm. As per the observations recorded in fig. 5, FESEM image of GO reflects the disordered and amorphous 2D structure of synthesized.
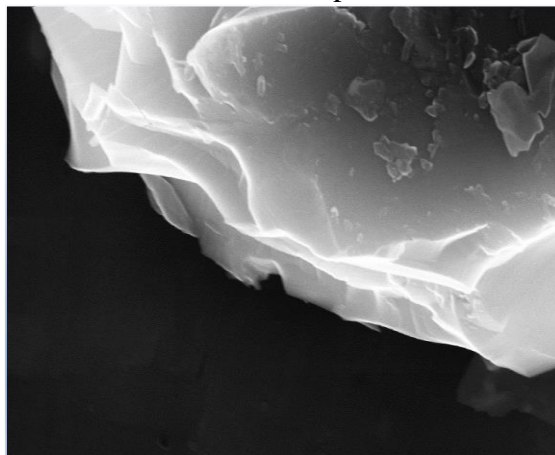

Figure 5. FESEM image

The image shows an agglomeration of large micrometer scale sheets displaying surface wrinkling and folding. The thick structure of GO is because of covalently bound oxygen presence, and it is easy to determine sheets edges.

3.4 Thermal Stability of nanofluids:

Thermal stability of all the samples were measured as a function of temperature using Zetasizer. The Stability test was performed after 41th day and 90th day of sample preparation. In this technique the thickness of the layer surrounding the particle i.e. Zeta Potential (ZP) value either positive or negative was measured. As reported by ASTM, ZP ranges of 20 to 30 mV are moderately stable and $>\pm$ 30 mV are highly stable [22]. After 41th day the ZP values were found in the range of 36 mV to 45.2 mV for all the samples of concentration ratio of 0.1 to 0.6%, that indicates the high stability of all the samples. fig. 6(b) shows the results obtained by for 0.6% sample. Whereas for the same samples these ZP values were recorded as 23.5 to 30.36 on 90th day which shows moderate to good stability.
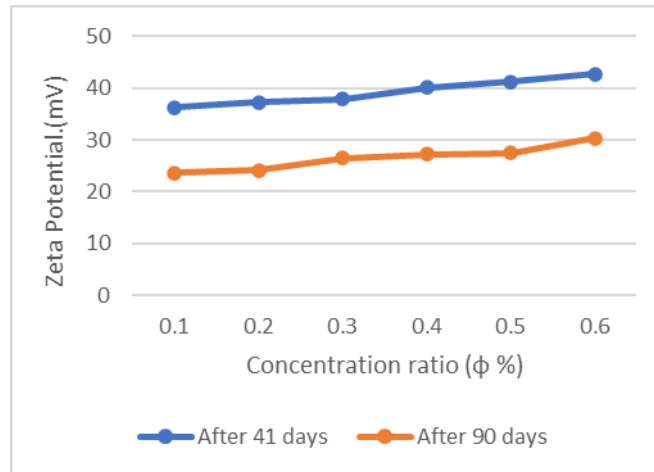
Figure 6. Stability analysis

From the fig. 6, it can be concluded that thermal stability increases as concentration increases but decreases over the time. From above values of ZP, it can also conclude that GO has aggregation behavior by increasing the mass fraction. By increasing mass fraction, negative electrical surface charge rose which is due to the ionization of carboxylic acid groups [26].
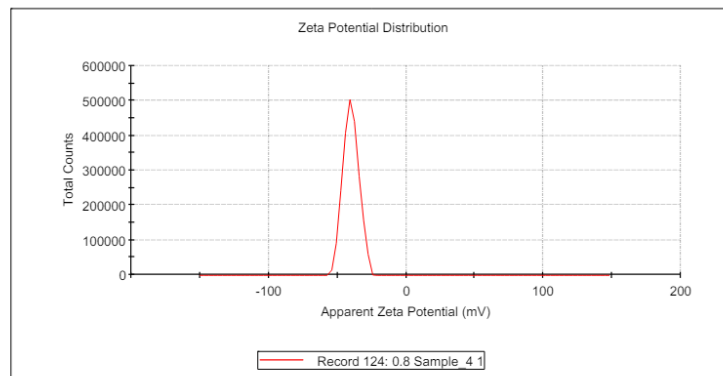

Figure 6(b). Zeta potential of GO ($\phi = 0.6\%$)

3.5 Thermal Conductivity:

Hot Disk, Thermal analyzer, (TPS 2500S, K-Analys AB Pvt Ltd Sweden) is the most appropriate instrument to measure thermal conductivity of different materials in the range from 0.005 to 1800 W m^{-1} K. This instrument was employed to measure the thermal conductivity of GO nanofluids samples in the temperature range of 25^{0}C to 55 ^{0}C. The precision of measurement was ensured by repeating each measurement by 4 times and taking average value. These results were validated using the Maxwell-Garnet model as per Eq. 5 [27]

$$K_{nf} = \left[\frac{K_{np} + 2K_{bf} + 2(K_{np} - K_{bf})(1+\beta)^3 \emptyset}{K_{np} + 2K_{bf} - 2(K_{np} - K_{bf})(1+\beta)^3 \emptyset} \right] K_{bf} \quad [5]$$

Where k_{bf} is the thermal conductivity of the base fluid, k_{np} is the thermal conductivity of nanoparticles, φ is the volume concentration of nanoparticles, and β indicates the ratio of the nanolayer thickness to original particle radius.

Fig.7 represents the values of thermal conductivity of GO nanofluids were obtained for multiple experiments. It seems that the thermal conductivity nanofluid was improved with increasing

temperature. Moreover, further increase in nanofluid's thermal conductivity occurred at higher temperatures. Overall increase in the thermal conductivity being reported about 3.68%.
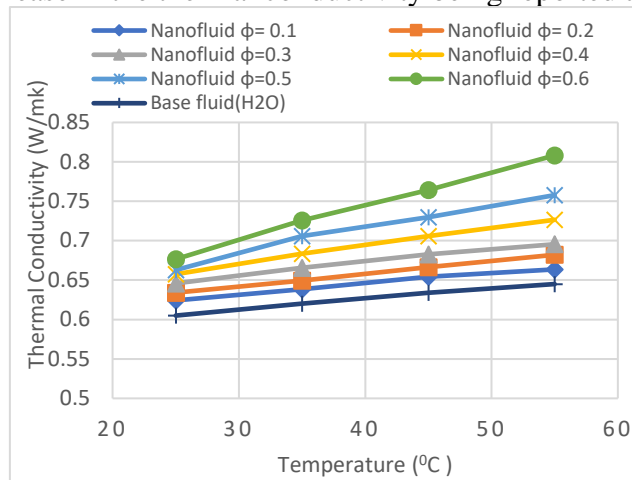

Figure 7. Effect of temperature on thermal conductivity

An effective factor in improving nanofluid thermal conductivity under increasing temperature may be energy interaction among nanofluid layers. Energy interaction increases with increasing particle volume in the base fluid; on the other hand, molecular movement also increases with raising the temperature. These factors govern the improvement in heat transfer.
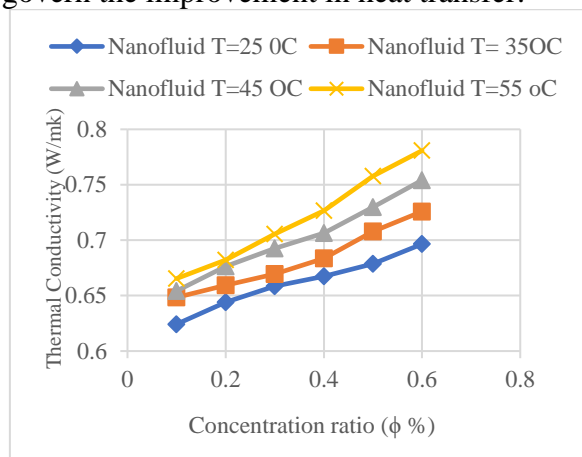

Figure 8. Effect of concentration ratio on thermal conductivity

The effect of change concentration ratio on thermal conductivity of nanofluids in temperature range of 25 to 55 ^{0}C has been represented by fig.8 According to these results, thermal conductivity increases with increasing the concentration ratios (ϕ). It was reported that overall 7.8 to 11.58 % increase in thermal conductivity for this range of nanofluids. Experimental results reveled that the thermal conductivity of the nanofluids in the current study was very higher than that of the base fluid, these positive changes noted at higher concentration could be related to the thermal conductivity of nanomaterials. There secondary reason for improvement in the thermal conductivity nanofluid was may be its instability caused by the Brownian motion, molecular-level layering at the particle/liquid interface was the main mechanism of enhancement in thermal conductivity [29]. Experimental results slightly deviated from empirical correlations due to the inherent assumptions made for ideal particles shape, uniform single particle size, and absence of mutual interaction among nanoparticles and between base fluid and nanoparticles, which is not quite accurate.

3.6 Specific heat capacity: The specific heat capacity (J/g.k) of nanofluids plays a crucial role in heating and cooling systems. Hot Disk, Thermal analyzer, (TPS 2500S, K-Analys AB Pvt Ltd Sweden) was used to measure the specific heat capacity. The specific heat was observed as 4.18 J/g.K for distilled water, while these values for graphene oxide was between range of 0.643-2.10 J/g.K. As per

experimental results the specific heat capacity of GO-water nanofluid was varies 3.915 J/gK to 3.768 J/gK. As we know specific heat capacity is the product of mass flow rate of fluid and specific heat. Hence nanofluids with higher specific heat required less fluid flow rate for a certain heating/cooling duty, which also reduces the pumping power requirements. unfortunately, there was decrease of specific heat capacity about 3.55% was reported that when the volume concentration of samples changed from 0.1% to 0.6%. after comparing the results with other nanofluids it was observed that GO nanoparticles has highest specific heat capacities among NPs this provides additional advantage compare to other nanofluids. For different volume fractions the specific heat capacity of nanofluid corelated to the specific heat capacities of the NPs and BF using expression as follows.[31]

$$C_{p(nf)} = (1-\phi)\, C_{p(bf)} + \phi C_{p(np)} \qquad [5]$$

3.7 Density:

Being a porous in nature, GO has both true density and bulk density. The true density of GO is close to that of graphite of 1.8-2.50 g/ml, while the bulk density of is much less in the range of 0.2-0.4 g/ml. The density of naofluid was increased from 0.9805 g/ml for pure water to 1.0048 g/ml at 0.6.%. following empirical correlation was used for calculating density[31].

$$\rho_{nf} = (1-\phi)\rho_{bf} + \phi \rho_{np} \qquad [6]$$

The increase in density along with the viscosity of nanofluid is not desirable as it creates a significant impact on increasing pressure drop and pumping power. The increase in density of nanofluid has another adverse effect on their stability. Due to Higher density of nanoparticles causes higher settling or sedimentation velocity which results in loss of nanoparticles in the heat transfer loop over time, which causes degradation of thermal performance of nanofluid. In this regard, it seems that the density of graphene is closest value to the density of most of BF, hence it provides the higher stability relative to other types of NPs, which is an additional benefit of Gr based nanofluid. relative to other types of nanofluid.

3.8 Viscosity: Viscosity of the nanofluid samples were investigated as a function of temperatures using viscometers (DV2THA, Brookfield, USA). The multiple experiments of samples were conducted by changing the temperature at 25, 35, 45 and 55 ^{0}C nanofluids samples at a high shear rate of 500 s^{-1}. There was a highest increase in viscosity at 25 ^{0}C. Viscosity is nothing but a simply the resistance of the fluid to flow due to inter-layer fluid interaction. As like to density, the viscosity also has two detrimental effects on pressure drop and pumping power requirements. The presence of nanoparticles in the nanofluids increases the friction at the fluid-surface interface due to nanoparticles-surface collisions and other inter-layer resistance and interfacial forces. These interfacial resistances result in increasing the viscosity of the nanofluid relative to the base fluid. There are several models available to analyses viscosity of nanofluid, one of them Brinkman model [32] which already been successfully utilized with different aspect rations such as fibers or platelets or flakes is expressed as follows:

$$\mu_{nf} = \frac{\mu_{bf}}{(1-\emptyset)^{2.5}} \quad [7]$$

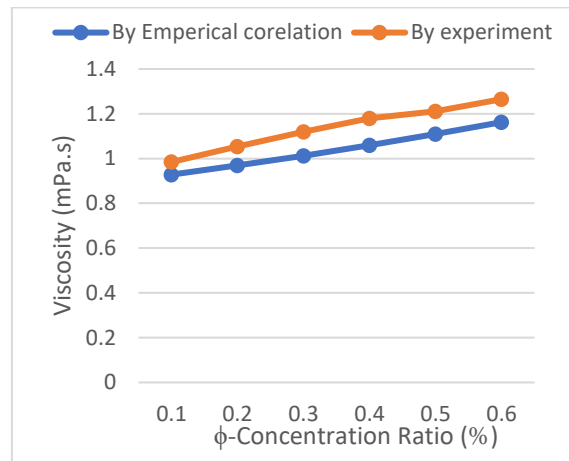Where, ϕ is the volume fraction of nanoparticles.

Figure 9. Effect of concentration ratio on Viscosity

Fig. 9 represents the graphical comparison between experimental results and the values obtained using Brinkman model at a temperature of 25ºC. It Seems that viscosity increases with increase in vol concentration ratio. At a lower value (0. 1%) of nanofluid, 5.8% increase in the viscosity were noticed as compare to water. As like to thermal conductivity the increase in the viscosity is much higher than another NF at higher concentration ratio. As per Einstein assumption for low concentration the particles to be rigid, uncharged and devoid of any attractive forces, the influence of aggregation and the formation of interfacial layers. It was observed that dynamic viscosity decreases considerably with temperature, Modified Andrade's equation used to describe temperature dependence of viscosity[33].

$$\ln \mu\,(T) = \ln \mu_0 + \frac{D.T_0}{(T - T_0)}$$

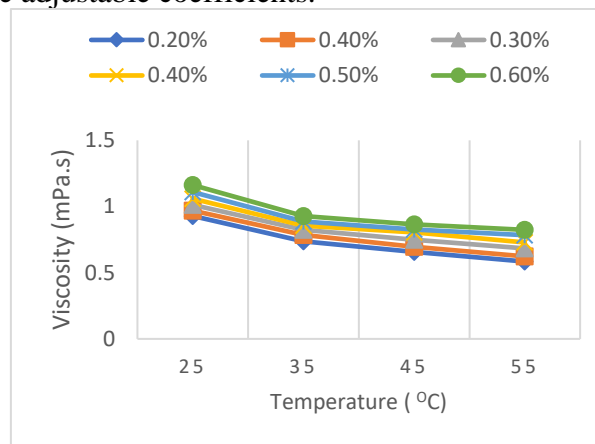where μ_0, D, and T_0 are the adjustable coefficients.

Figure 10. Effect of temperature on viscosity

Conclusion:

In this study, experimental analysis of thermophysical properties of GO/Water based nanofluid has been performed in the temperature range of 25 ^{0}C to 55 ^{0}C and volume fractions of 0.1%, 0.2%, 0.3%, 0.4%, 0.5% and 0.6%.

1) During preparation of nanofluids of various concentrations using oxide type of nanoparticles, mechanical and ultrasonic preparation processes were applied this results in homogeneous nanofluid.

2) Zeta Potential test confirmed that stability increases with vol. concentration upto certain limit, all sample reported good to moderate stability till three months.

3) The effect of an increase in vol. concentration on thermal conductivity of the nanofluid was significantly greater than that of temperature variations, the greatest increase in thermal conductivity

of nanofluid was appeared as 11.58 % for a volume concentration of 0.6 % at a temperature of 55 ^{0}C. However, there was an associated drop in specific heat of up to 3.65%.

4) A considerable increase in density and much higher increase in viscosity (5.8-12%) was observed with increase in nanoparticle concentration. Conversely both density and viscosity deceases with increases in temperature.

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