Rheological analysis of MWCNT, TiO$_2$, CuO based Nano–Fluids under varying temperatures and concentrations

M D Kathir Kaman*, A Sathishkumar, Kshitij Sharma, Chintan Trivedi, Abhishek Singhal

Department of Mechanical Engineering, SRM Institute of Science and Technology, Chennai 603 203, India

*Corresponding Author: kathirkaman.d@ktr.srmuniv.ac.in

Abstract. The present work aims to determine the effect of Nanoparticles in Water - Ethylene glycol mixture on contradictory results in literature, which includes thermo-physical property focusing on viscosity. The experimental analysis was carried out using Rheometer on Water – Ethylene glycol based Nano fluid containing 0.015 % and 0.15 % by weight concentration of cylindrical MWCNT, spherical CuO and TiO$_2$ at 10 °C, 20 °C, 30 °C and 40 °C. The Rheometer procured result of viscosity shows that the Newtonian to shear thinning transition takes place at high shear stress range in comparison to the base fluid. At 10 °C, highest increment in the viscosity was noted by MWCNT (0.015 % by mass) over (0.15 % by mass) for CuO, 139.3 % increment and TiO$_2$ recorded 64.73 % for (0.015 % by mass) over (0.15 % by mass). Moreover, the modified Maron and Pierce model predicted the viscosity of Nano–Fluid which in turn resulted in showing that viscosity ration of Nano-Fluid increased with increase in temperature due to the addition of high surface activity Nano particles into water-ethylene glycol mixture. The aspect ratio, Brownian motion, shear thinning and surface activity of Nano particles played a major role in the viscosity analysis.

Index Terms – Rheology, Shear thinning, Aspect ratio, Viscosity ratio.

Nomenclature

$\Phi$ - Volume fraction (%),

$\eta$ - Intrinsic viscosity (dL/g),

$\mu$ - Dynamic viscosity (Pa s),

$r$ - Aspect ratio of Nanoparticle

$T$ - Temperature (° C),
\( \rho \) - Density (kg/m³),

\( d \) - Diameter of particle (m),

\( \Phi_e \) - Effective volume fraction (%).

\( p \) - Particle,

\( \text{eff} \) - Effective,

\( \text{nf} \) - Nano fluid

\( a, b, c, n \) - Constants

\( f, m \) - Base fluid

\( \mu_{nf} \) - Dynamic viscosity of Nano fluid (Pa s)

\( \mu_f \) - Dynamic viscosity of base fluid (Pa s)

\( \Phi_{\text{max}} \) - Maximum volume fraction

\( D \) - Fractional Index

1. **INTRODUCTION**

Over the past few decades plenty of research has been carried out to develop the thermal application based on heating and cooling. In the recent techniques the conventional heat transfer fluids such as water, ethylene glycol and engine oil are improved by adding small amount of Nano-particles in them. The immediate need for energy efficient devices in thermal regime particularly in heat exchangers boosted the research made in Nano-particle based Nano fluid as the dispersion of the same increases the efficiency of base fluid, high emphasis is being made in this field. The development of Nano-particle and their dispersion into fluid Choi [1] was a first step in this field. The most useful studies carried out on the Nanoparticle properties were by Eastment et al. [2], Le and Choi [3, 4], Jang and Choi [5], Heris et al. [6] showing the enhancement of thermo-physical properties due to the addition of Nano-particle into the base fluid. The parameters affecting the stability of Nano fluid along with the physical and rheological properties emphasis on their heat transfer characteristics were studied by K.H Solangi, et al. [7]. An overview of the different heat transfer phenomenon in Nano fluid and most important parameters such as size, shape, concentrations, materials etc. and their influence to the thermos – convective and radiative experimental results were shown by Mouro, Lomascolo et al. [8]. Viscosity, a property occurring due to particle collision in a fluid that has different velocities or due to Vander waal forces along with the examination of rheological behavior of MWCNT – ZnO SAE 40 hybrid Nano fluid under various temperature and concentration were found out by Mohammad, Hemmat esfe et al. [9]. M.K.H Abdol Bagi et al. [10] studied thermal conductivity and viscosity of bio-glycol-water based TiO₂ Nano fluid at 30 – 80 °C temperature and 0.52 – 2.5 %
volume concentration was proposed with 5 % maximum deviation for the estimation of thermal conductivity and viscosity of Nano fluid. M.A Akhavan Behabadi et al. [11] investigated convective heat transfer and friction factor of MWCNT water Nano fluid inside a horizontal circular tube experimentally at particle weight concentration 0.05 %, 0.1 %, 0.2 %. The result obtained by his result explained the enhancement of heat transfer in Nano fluid with huge penalty in pumping power. Moreover, experimental and theoretical analysis was carried on ethylene glycol based TiO₂ spherical Nano fluid solution with 0.5 – 8 % weight percent concentration at 20 – 60 °C temperature by Haisheng Chen et al. [12] and stated that ethylene glycol based Nano fluid are Newtonian under the condition of this work with shear viscosity as a function of temperature and particle concentration. The Kreiger Dougherty equation [13] was used to predict high shear viscosity of Nano fluids.

2. PREPARATION & MEASUREMENTS

2.1 MWCNT preparation

MWCNT Nano-particles were prepared using chemical vapor deposition technique. D. Madhesh [14] studied a preparation of MWCNT which was obtained by using a horizontal quartz tube reactor. The Multicomponent metal oxide as catalyst was used in the reactor with flexible graphite foil support. The method represented constant volume ratio of Argon to Propane Butane which yields MWCNT at constant feed rate of the same. Average size was found out within the range of 45 – 50 nm.

2.2 Synthesis of CuO

The copper oxide was synthesized using 10 g of copper sulphate with molecular weight 159.6 g/mol. The 10 g of copper sulphate was mixed with 25 ml of distilled water (D.I water) to form a saturated solution. Then, 24 g of Ascorbic acid (Vitamin – C) was added to the 60 ml D.I water to form a saturated solution. This was later added to copper sulphate solution and made to stand for few minutes. The obtained mixture was heated at 800 °C for 4 – 5 times until the solution boiled. The solution was filtered and washed with distilled water 2 – 3 times. The obtained powder was further washed with absolute ethanol two times, filtered and left for drying in drying oven. Nanoparticle was found within the range of 7 – 10 nm.

![Figure 1. TEM image of MWCNT](image_url)
2.3 TiO$_2$ preparation

TiO$_2$ Nanoparticles were prepared by hydrolysis and peptization of titanium isopropoxide solution. S. Mahshid [15] briefed about the hydrolysis of titanium isopropoxide solution along with the peptization of resultant suspension from various pH aqueous solutions for the preparation of TiO$_2$ Nano powder with narrow size distribution. The acidic solution provided Nano powder with fine particle size of 10 – 12 nm. Further the Nano powder obtained was ball milled and ultra-sonicated for 20 – 30 minutes for fine size and distribution of Nanoparticles.

2.4 Nano fluid preparation

The obtained Nanoparticles were set for ball milling for 6 minutes at 100 rpm using 4 balls. Nanoparticles after ball milling were kept for dry ultrasonification for 30 minutes. An Ethylene glycol – Water mixture was prepared with 4:6 ratio and was magnetic stirred for 10 minutes. The surfactant
SDBS (sodium dodecyl benzene sulphonate) was added in the mixture and further stirred for 10 minutes. The surfactant was added to increase the stability of Nano fluid. The Nanoparticle was then slowly added into the mixture and magnetically stirred for 15 minutes. The final solution was ultrasonicated in ultrasonicator for 90 minutes. Figure 1, 2, 3 shows the TEM image of MWCNT, CuO and TiO$_2$ Nano fluids respectively.

2.5 Viscosity measurement

The viscosities of Nano fluid and rheological behavior was measured by employing Rheometer at 10 °C, 20 °C, 30 °C, 40 °C, the temperature was maintained using a heating element and cooler attached to the Rheometer. The Nano fluid sample was placed on the lower shear plate. The upper shear plate used was of 2 °/55 configurations which is commonly used for liquid samples. Viscosity parameter such as sweep time was set to 120 seconds and shear rate between 0.1 to 20 m/s$^2$ was applied. Bohlin Gemini 260 software was used to get the rheological data along with the instantaneous viscosity of fluid.
3 RESULTS & DISCUSSIONS

3.1 Viscosity of Nano fluids

There has been not a single mathematical model derived as of today which truly relinquishes the viscosity coefficient of a given Nano fluid. Several groups conducted studies on the same and have not been able to establish rationale as of why viscosity varies among Nano fluids. A valid logic would be, as conventional suspensions, reliance of viscosity on concentrations of Nanoparticles. More nanoparticle concentration, results in Nano fluid being more viscous in nature. From the figure 5, we conclude TiO$_2$ of (0.15 % by mass) shows the highest viscosity. Even at higher temperatures its value remains above the other compared ones. He, Jin, et al. [16] adduce dependence of viscosity on particle size. With increase in size, viscosity should increase. But, Rudyak et al. [17] suggests opposite, for a given concentration, decrease in Nano particle size offers increasing viscosity. Measurements taken from Figure 4 SEM images of all the Nanoparticles at both the concentrations show the following data in Table 1. The values are tabulated after taking average of 5 sets of measurement of diameters. Since, sampling concentrations were very low, with respect to our data, size plays a non – significant role in determining viscosity.
Another way to look for explanation can be going into the roots of the Nano fluid, examining each compound of the Nanoparticle. Strength of the intermolecular forces present amid components direct viscosity in a fluid. And these forces also play a role in determining density of the same. Generally, talking points on viscosity do not factor in the importance of density much like its allegiance with temperature. Unless, we compare multi-component fluids where, different components enjoy variety of both physical and chemical properties, we do need to include density into the picture. Closely all viscosity models have correlations with temperature and concentration but, Sunderland’s law [18] gives a relation between density and temperature. Even if we consider this argument, CuO is denser than other elements in consideration. But, there is no thumb rule whatsoever for denser element/compound to be more viscous. Considering, ethanol whose density is 789 kg/m$^3$ with viscosity of 0.001095 Ns/m$^2$ and for water, density is 1000 kg/m$^3$ and viscosity is 0.00089 Ns/m$^2$.

**Figure 5.** Variation of viscosity with temperature
Table 1. Average sample size of Nano fluid

| Sample             | Average size (nm) |
|--------------------|-------------------|
| MWCNT (0.015 %)    | 117.67            |
| MWCNT (0.15 %)     | 120.345           |
| TiO₂(0.15 %)       | 67.318            |
| TiO₂(0.015 %)      | 63.411            |
| CuO (0.15 %)       | 69.448            |
| CuO (0.015 %)      | 66.318            |

It is also well known that liquids having polar natured molecules or presenting more affinity to form hydrogen bonds are usually more viscous than similar nonpolar substances. Going further into the physical and chemical properties of the individual compounds, we observe TiO₂ has hydrogen bond acceptor count as 2 whereas, CuO has only 1. Intermolecular forces also affect the resistance offered to a flow, higher the bond strength more resistance resulting in it being more viscous. Boiling point of TiO₂ lies in the range of 2500 – 3000 °C and for CuO its closely 1026 °C. Clearly, bond strength of former is much more than that of latter. Also, TiO₂ having superior chain length as compared to CuO [19] can be a deciding factor in it being more viscous. Now let us see the variations observed for same samples, different concentrations. At lower temperature, (say 10 °C) increment in MWCNT (0.015 % by mass) over (0.15 % by mass) is recorded highest, viscosity of latter being more than 5 times of the former. Whereas, for CuO it is 139.3 % increment and for TiO₂ it remains at 64.73 %. As we observe further for higher temperatures the gap amongst the viscosities for the same Nano fluid narrows down. But, that is not the case for TiO₂. At 20 °C the number becomes 358.47 %, nearly 4 times the viscosity of (0.015 % by mass), at 30 °C it is 200.36 % and for 40 °C, 307.54 %. As observed, the variation is not linear in nature. Slight bump evident from the graph for TiO₂ (0.015 % by mass) is the reason behind this variation. Interestingly, all the samples along with their concentrations follow an upward trend in the 20 °C to 40 °C range.

3.2 Shear thinning effect

From Figure 6. (a), we can tell shear stress does not vary at constant rate with increase in shear rate. Hence, this relation clearly states our Nano fluid being a non-Newtonian fluid with downward trend after 10 s⁻¹ (approx.) value of shear rate. This trend later proves the fluid to observe shear-thinning effect. The viscosity depends on the cross-sectional area in the direction of flow. At low flow rates, molecules which are thin and long with preferred conformations have effectively large cross-sections and surface activity due to them dipping in solution but, at high rates of shear the molecules seem to align with the flow, giving much smaller effective cross-sections and hence much lower viscosities, much like in the case of MWCNT. Now, dense molecules do not get affected by their orientations in relation to flow and hence their viscosity changes little with shear rate as in the case of CuO Nano fluid (both concentrations).

As depicted in the Figure 6 (b), concentration do influence shear-thinning, all the three different Nano fluids with higher concentration (0.15 %) show high amount of shear-thinning effect.
TiO$_2$ (0.15 %) concentration fluid shows the largest negative slope, CuO (0.15 %) follows next. Decrease in viscosity might be the result of microscale structural rearrangements within the fluid.

**Figure 6 (a).** Rheological behavior of Nano-Fluids at 30°C
Figure 6(b). Rheological behavior of Nano-Fluids at 30°C

3.2.1 Comparison of Experimental Data with Data from Viscosity Models

In recent years, a large number of equations have been developed from the classical theoretical viscosity models to forecast the viscosity of Nano fluids at various temperatures and concentrations. The very first theoretical model for predicting the viscosity was given by Einstein [20]. The viscosity ratio determined from Einstein’s Equation is valid for spherical particles of low particle volume fraction $\Phi < 0.02$ and is given by formula,

$$\frac{\mu_{nf}}{\mu} = 1 + 2.5\Phi$$

Other famous Classical theoretical model was given by Batchelor [21] by extending the Einstein’s equation considering the effect of Brownian motion and is given by formula,

$$\frac{\mu_{nf}}{\mu} = 1 + 2.5\Phi + 6.5\Phi^2$$

Saito [22] presented another derived viscosity model from the classical models which shows that viscosity depends on particle volume fraction and is given by,
$$\frac{\mu_{nf}}{\mu} = 1 + 2.5\Phi + 2.5\Phi^2$$  \hspace{1cm} (3)

Brinkman [23] formulated a theoretical viscosity model by two corrections of Einstein’s model (eq.1) for the fluids having low concentration of solid particles and is given by,

$$\frac{\mu_{nf}}{\mu} = \frac{1}{(1 - \Phi)^{2.5}}$$ \hspace{1cm} (4)

Nguyen [24] presented the temperature dependent viscosity models for Nano fluids having particle concentrations between 1 – 4 % and is given by,

$$\frac{\mu_{nf}}{\mu} = 2.1275 - 0.0215T + 0.00027T^2$$ \hspace{1cm} (5)

Brenner and Condiff [25] considered the shape effect for developing the viscosity model for Nano fluids and is given by,

$$\frac{\mu_{nf}}{\mu} = 1 + \eta\Phi$$ \hspace{1cm} (6)

Where,

$$\eta = \frac{0.312r}{ln(2r - 1.5)} + \frac{0.5}{ln(2r - 1.5)} - \frac{1.872}{r}$$ \hspace{1cm} (7)

Thus, Maron and Pierce [26] developed an equation considering the above equation for viscosity of spherical particles in suspension considering the Ree-Eyring [26] flow as follows:

$$\frac{\mu_{nf}}{\mu} = \left[1 - \frac{\Phi_{p}}{\Phi_{m}}\right]^{-2}$$ \hspace{1cm} (8)

This equation was further modified [27] for both spherical and cylindrical nanoparticles as follows:

$$\frac{\mu_{nf}}{\mu} = \left[1 - \frac{\Phi_{a}}{\Phi_{m}}\right]^{-2}$$ \hspace{1cm} (9)

Where,

$$\Phi_{a} = \Phi\left(\frac{a_{a}}{a}\right)^{3-D}$$ \hspace{1cm} (10)

In the above equation the value of $a_{a} \approx 84.085$ nm is taken average for all nanoparticle(s) from SEM images of Nano fluids at 0.15 % and 0.015 % concentrations. The average agglomerate size of all samples taken from SEM images is shown in Table 1 above. The value of ‘a’ and ‘D’ are taken as 40 nm and 2.1 respectively for all the three nanoparticles. The above equations were used for the comparison of experimental data obtained from Rheometer (Bohlin CVO, Malvern Instruments, UK)
with the predicted results of viscosity from these models and the comparison is shown below in the form of graph.

The above graph in the figure 7 shows the Comparison of Experimental Data of viscosity ratio with Data from Viscosity Models given in equations (1 – 5) and equation (9 & 10) at a temperature of 20 °C. As represented by the graphs all the viscosity models show good agreement with the viscosity ratio of TiO₂, CuO and MWCNT Nano fluids at low concentration of $\Phi < 0.05$ with an average deviation of 0.5 % but, when the concentration increases from $\Phi > 0.05$ the viscosity models under predicted the viscosity of Nano fluids.

![Figure 7. Comparison of theoretical and experimental data at 20 °C](image)

The reason behind the correct prediction of viscosity ratio by several models at low concentration is that at low concentration various effects such as Brownian motion and agglomeration does not take place in the Nano fluid leading to the prediction of viscosity just based on the concentration of the nanoparticle but, as the concentration increase, various effects such as aspect ratio because of agglomeration and Brownian motion come into existence. Thus, modified form of Maron and Pierce equation which includes the effect of aspect ratio of nanoparticles and takes into account the agglomerates formed at higher concentration shows good
prediction of viscosity of CuO Nano fluid with a very small deviation of 0.03 %. Also Modified Maron and Pierce equation shows good agreement with other Nano fluid solutions with a maximum deviation of not more than 0.55 % even at high concentrations. Hence, the above comparison shows it’s very important to include the effects arising from aspect ratio, agglomeration, shape and size of Nano particle along with the concentration of nanoparticle in the base fluid.

4 CONCLUSION

The measured viscosity of the three Nano fluids used for this research decreases with increase in temperature as cohesion plays a main role with increase in temperature this cohesive force decreases as the energy of particles become more, hence movement of particles become free and easy thus, decreasing the viscosity with increase in temperature.

Among the three nanoparticles TiO$_2$ nanoparticle shows highest viscosity at 0.15 % concentration in Water – Ethylene glycol base fluid as the viscosity increases with decrease in size of the nanoparticles and TiO$_2$ being the smallest shows highest viscosity. At 10 °C increment in MWCNT (0.015 % by mass) over (0.15 % by mass) is recorded highest, viscosity of latter being more than 5 times of the former. Whereas, for CuO it is 139.3 % increment and for TiO$_2$ it remains at 64.73 %. Further, at higher temperatures the gap in viscosities for the same Nano fluids tapers down. The comparison of shear rate vs shear stress and shear rate vs viscosity shows the non-Newtonian nature and shear thinning effect in Nano fluids prepared for the research. Further, the modified Maron and Pierce equation shows nearly accurate prediction of viscosity at both concentrations which depicts that nanoparticle shape, size along with agglomerate size plays a significant role in the viscosity of Nano fluids.

Also, as most of the theoretical models were able to predict the viscosity of all the Nano fluids at low concentrations, it shows that only the concentration of nanoparticle plays a significant role in the viscosity of Nano fluid at low concentrations. Thus, at low concentration the amount of Nano particle in the base fluid is responsible for viscosity but, as the concentration increases various effects such as shape, size, agglomeration, shear thinning and Brownian motion plays an important role in the viscosity of Nano fluid and hence, predicted accurately by modified Maron and pierce equation.

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