# Study of synthesis, characterization and thermo physical properties of $Al_2O_3$-$SiO_2$-$TiO_2$ / $H_2O$ based tri-hybrid nanofluid

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The prediction of behavior of thermo physical properties is essential to understand the heat transfer characteristics of any nanofluid. In the present work the tri hybrid oxide nanoparticles namely $Al_2O_3$, $SiO_2$ and $TiO_2$ of three different ratios are prepared at 0.1% concentration and their characterization and thermo physical properties are examined. The water is used as the base fluid and the sample is made for 100 ml. The nanoparticles are dispersed with the base fluid by magnetic stirring and followed by ultra-sonication. The stability is also found to be good under zeta potential test. The experimentation results showed that the thermo physical properties are better than that of base fluid. The thermal conductivity maximum enhancement was found to be 18.33% from the base fluid at just 0.1% concentration at 50$^o$ C. The maximum viscosity enhancement is found to be as 43.79% than that of base fluid. The experimental results are compared and validated with the available literature based mathematical models and similar works of other authors and found to be of good matching. The maximum deviation from the model correlation is below 1%. This novel tri hybrid nanofluid is effective alternative for many di-hybrid nanofluids and has better thermo physical properties than oxide based di hybrid nanofluids.

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*Keywords:* Oxide-nanofluid, Viscosity, Tri-hybrid-nanofluid, Tri-hybrid nanoparticles, Thermal conductivity

## 1. Introduction

Nanofluids play a major role in the process of energy conservation which is the need of the hour and the crux of the time. Over the years with the advent of nanofluids a new window of opportunity for researchers and environmentalist are open to reduce consumption and to effectively operate heat equipment. The nano fluids have transformed into a new level with the innovation of hybrid nanofluids. [1] Synthesized the hybrid nanofluid using thermo chemical method. Nanofluids at various volume concentrations were prepared by using two step methods. The thermo physical properties are evaluated and found to have better characteristics. The experimental results are found to be better than existing models.[2] Reviewed the progress of hybrid nanofluids which involves synthesis, characterization, stability mechanism and applications in various fields. It is observed that the application of nanofluid with low viscosity and high conductivity are promising and the stability analysis of nanofluid and intense research will lead to increase in the performance of nanofluids.

[3] Investigated hybrid g-$Al_2O_3$/mwcnt and observed that the stability of the nanofluid is essential to enhance the thermal conductivity which can even reach to 14.75% even for a volume concentration of 0.01%. [4] Investigated and found that the addition of surfactant along with

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sonication for 3 hours can bring stable nanofluid which can be utilized for any application for a month. [5] Examined and assessed that the Nano platelets can improve stability and the viscosity of hybrid nanofluid more than the unitary fluid. [6] Experimental results show that the thermal conductivity tends to increase with raise in temperature and volume fraction. The hybrid nanofluids have shown better thermal conductivity than unitary SIC or $TIO_2$ nanofluids. [7] Investigated on thermal conductivity and viscosity of $TiO_2$-$SiO_2$ nanofluids by blending water with ethylene glycol mixture. The thermal conductivity of the proposed sample tends to have a 22% increases in heat transfer for 3% volume concentration and experimentally found that the $TiO_2$ and $SiO_2$ based hybrid Nano fluid had enhancement of thermal conductivity up to 22.8% for 3% volume concentration and better relative viscosity with 62.5% increment for the same volume concentration They have also found that the regression equations are more satisfactory for 30 to 70 °C temperature limit for 3.0% concentration. [8] Determined using second order polynomial function that the viscosity is decreasing as temperature increases for all considered fluids and also the hybrid rheological behavior is close to the hybrid nanofluid. [9] Experimented and found that the $Al_2O_3$-$TiO_2$/$H_2O$ hybrid nanofluid has good conductivity and also hybrid nanofluid had reasonable stability due to addition of surfactant and sonication.

It is evident through numerous investigations [10–17] that heat transfer characteristics can be improved in thermal equipment with the usage of nanofluids. In the last decade the investigation migrated to hybrid Nano particles [3,10,18–23] focused on improving the thermo physical properties and heat transfer characteristics by blending two or more different nanofluids simultaneously with the base fluid. [20] Experimented on oxide based di-hybrid nanofluids and results shown better characteristics than that of unitary nanofluids and moreover new nusselt number correlation is suggested for alumina based nanofluids. [24] Examined graphene based hybrid nanofluids and tested the fluid with an experimental setup and results showed that these hybrid nanofluids have better heat transfer characteristics than unitary nanofluids. [5] Through experimentation suggested focusing more on economic aspects of nanoparticles is also essential. [25] Numerically found the thermo physical properties of $Al_2O_3$-$SiO_2$-$TiO_2$/$H_2O$ based di hybrid nano fluids. [26] Experimented on $SiO_2$-$Al_2O_3$ based di hybrid nanofluid to find the thermal conductivity of both unitary and hybrid nanofluids.[8] Experimented on $SiO_2$-$Al_2O_3$ based di hybrid nanofluid to find the viscosity of the unitary and hybrid nano fluids. [27] Experimented on $Al_2O_3$-$TiO_2$/$H_2O$ based di hybrid nanofluid and estimated the thermo physical properties.

[28] examined and found the stability of $Al_2O_3$-$SiO_2$-$TiO_2$/$H_2O$ based nanofluid and the same author [29] utilized the same oxide nanofluid and examined the heat transfer characteristics for automotive applications but have not experimented the thermo physical properties of nanofluid.

Therefore, In the present work, for the first time the focus is on the detailed study of synthesis, characterization and thermo physical Properties of tri hybrid oxide nanofluid at varied proportions of nanomaterial as mentioned in Table 1 and for the first time the mathematical models are validated which are exclusively modeled for tri hybrid oxide nanofluid suggested by previous authors but not tested yet.

## 2. Synthesis and characterization

### 2.1. Preparation of tri-hybrid Nano Material:

The Nano powders $Al_2O_3$, $SiO_2$ and $TiO_2$ were procured from the Nano manufacturer in India and the size of the Nano particles range from 10 to 50 nm (as per manufacturer data) with a purity of 99.5%.

The Nano powders procured were blended with each other at three different ratios (sample) as mentioned in Table 1 at 0.1% volume concentration.

*Table 1. Nanofluids with proportions and base fluid notations.*

| Sl.No | Sample Proportion | Sample name |
|---|---|---|
| 1 | Water ($H_20$) | W |
| 2 | 0.33 % $Al_2O_3$ + %0.33 $SiO_2$ + 0.33% $TiO_2$ | X |
| 3 | 0.50 % $Al_2O_3$+0.25% $SiO_2$ + 0.25% $TiO_2$ | Y |
| 4 | 0.70 % $Al_2O_3$ +0. 15 % $SiO_2$ + 0.15% $TiO_2$ | Z |

The Nano powders of the required weight concentration separately were found for each sample to bring 0.1% volume concentration. Then the samples were milled and grinded separately and later all the three samples were kept separately in a furnace and heated up to 900$^{o}$ C for perfect blending. The samples were allowed to cool in the furnace for effective heat treatment.

**2.2. Synthesis of Tri-hybrid Nanofluid:**

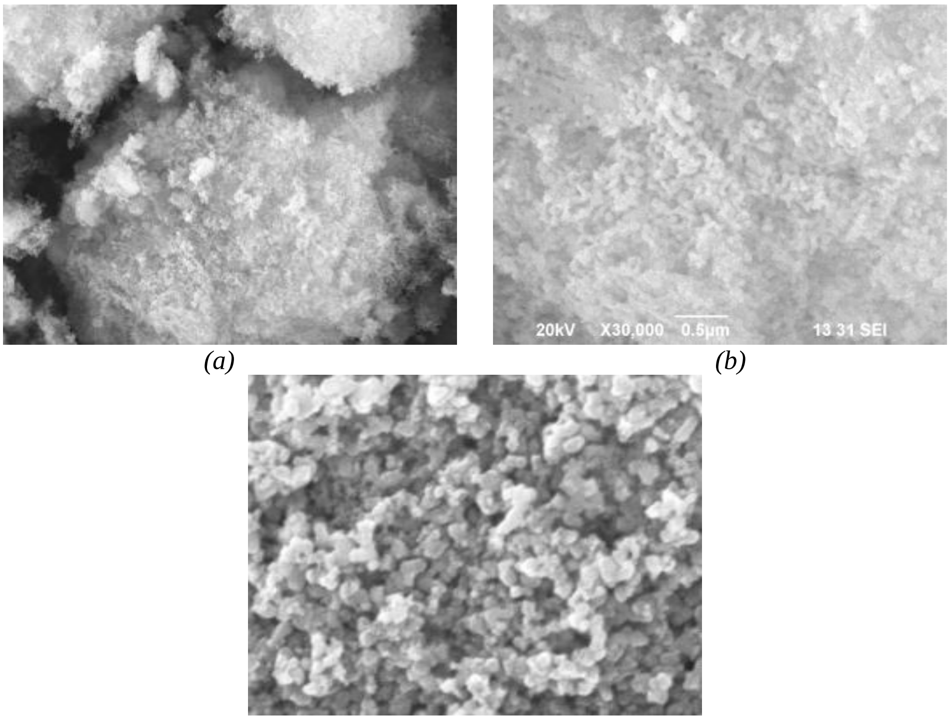
The preparation of tri-hybrid Nano fluid was done by two step method [1] . The prepared tri hybrid Nano powders were mixed with water by magnetic stirring. The stirring was done for two hours and then the same mixture was kept in an ultrasonic bath and sonicated for few hours. The volume of base fluid used for testing was of 100 ml at 0.1% concentration. The weight of nano particle required for 0.1% concentration was calculated using the "eq.1".

$$W_c = \left(\frac{\varphi}{100-\varphi}\right)\left(\%\, n_{p1}\ \frac{\rho_{np1}}{\rho_{bf}} + \%n_{p2}\ \frac{\rho_{np2}}{\rho_{bf}} + \%n_{p3}\ \frac{\rho_{np3}}{\rho_{bf}}\right)\left(W_{bf}\right) \quad (1)$$

**2.3. Material characterization**

***2.3.1. SEM Analysis***

The SEM analysis was done for all the three samples and it was observed that the particles were widely spread and the sizes were well under 100nm. The SEM images of all the three samples was examined and it was observed that addition of alumina leads to color change from dark to white as evident in the fig.1.



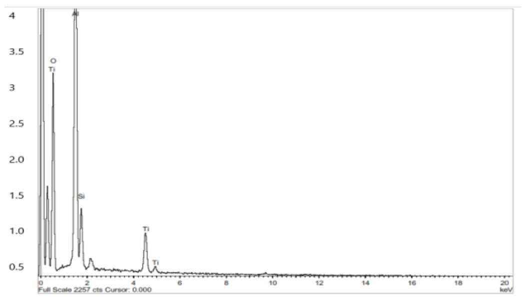

*(a)* *(b)*

*(c)*

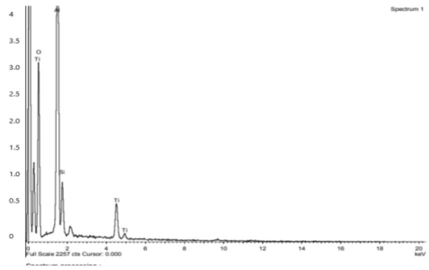
*Fig. 1. SEM images of samples a) X b) Y and C) Z.*

### *2.3.2. EDAX Analysis*

It is confirmed from all the three EDAX results in Fig.2 that the presence of Alumina, titanium and silica are at the required proportion for each sample, therefore proper blending of the nanoparticle has occurred as inferred from results inline to the blending as per table 1. The presence of oxide particles are more in the samples as evident by the spike of oxide line in graph.




*(a)*

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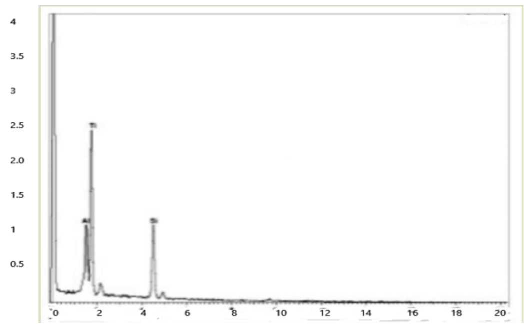


*(c)*

*Fig. 2 EDAX images of samples a) X b) Y and c) Z.*

### *2.3.4. XRD*

The Average crystalline size (D) of the samples was calculated using the Scherer formula [30], "Eq.2" and it was found that the size of the grain was 13.46 nm, 15.56nm and 16.86 nm for the tested sample (X),(Y) and (Z) respectively. λ, β and θ are the X-ray wavelength of radiation utilization.

$$D = \frac{0.9\lambda}{\beta cos\theta} \quad (2)$$

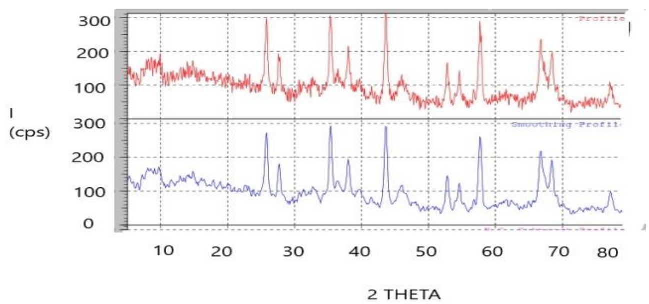
*** Basic Data Process ***

Group : Ka-XRD-915
Data : 1

# Strongest 3 peaks

| no. | peak no. | 2Theta (deg) | d (A) | I/I1 | FWHM (deg) | Intensity (Counts) | Integrated Int (Counts) |
|---|---|---|---|---|---|---|---|
| 1 | 41 | 43.5964 | 2.07439 | 100 | 0.50710 | 98 | 551 |
| 2 | 58 | 57.6760 | 1.59702 | 92 | 0.53670 | 90 | 507 |
| 3 | 34 | 35.3477 | 2.53723 | 87 | 0.49550 | 85 | 450 |

*(a)*




*(b)*

*Fig. 3. XRD images for Sample (Y) a) Peak values and b) Profile and smooth profile.*

***2.3.5. Zeta Potential***

The zeta potential test was performed for finding the stability of the nanofluid. It is observed that the fluid is found to be stable with the value of around 30mv for all the three samples and found to be of with reasonable stability. The stability may also be improved with the addition of surfactant if required. In the Fig.4 zeta potential for sample (Z) is depicted.

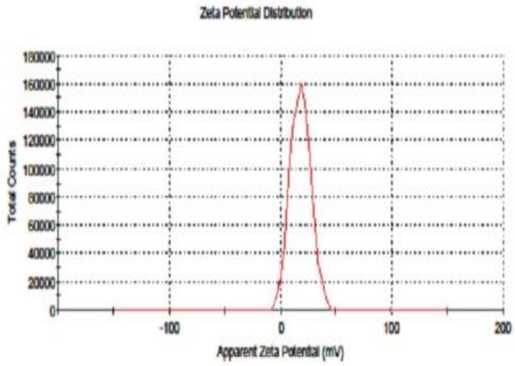



*Fig. 4. Zeta potential analysis of sample (Z).*

## 3. Testing Methods

### 3.1. Thermal Conductivity Measurement

Thermal conductivity can be measured by numerous ways and in the current work the measurement was made with KD2-Pro Analyzer. The principle of this apparatus is based on transient dynamic technique where the measurement is made with the rise of temperature in a linear hot wire which is coupled with the material to be tested. This instrument has an accuracy of 5% and supported with a probe of 1.3 mm diameter and a length of 60mm. The calibration of the needle was done by measurement of thermal conductivity under ambient condition using water as the testing fluid. The obtained value was 0.6 w/mk.

The sample of Nano fluid of about 45ml was poured in a glass vial which has a diameter of 30mm. The cap of the vial was enabled with a septum to allow the sensor needle to enter into the fluid with the orientation at which it will not touch the walls of the vial. The thermal conductivity of all the samples was measured with a periodic interval of 15 minutes and only after few hours of sonication. The time taken for completing a cycle was about 90 secs which includes the time for heating and cooling of the needle. 3 sets of readings were taken for each sample and average of three values was used for analysis. The obtained results were compared with the mathematical model suggested by [25] of "Eq.3".

$$K_{eff} = K_{bf}\left[1 + \frac{K_{P1}\varphi_1 r_m}{K_m(\varphi_1+\varphi_2+\varphi_3)r_{p1}} + \frac{K_{P2}\varphi_2 r_m}{K_m(\varphi_1+\varphi_2+\varphi_3)r_{p2}} + \frac{K_{P3}\varphi_3 r_m}{K_m(\varphi_1+\varphi_2+\varphi_3)r_{p3}}\right] \quad (3)$$

### 3.2. Viscosity Measurement

The viscosity is a very crucial parameter in the thermo physical properties of the Nanofluids. The viscosity in the current work was measured with the Brookfield viscometer. The viscometer was calibrated using water measured at room temperature at 30°C and value obtained was 0.767 mPa-secs. The viscosities of the given samples were measured by allowing the fluid to flow in a small gap which has a cone and plate arrangement. The viscometer can measure the viscosity at a range of 0.3 mPa-secs to 1000 mPa-sec. The angular movement of the spindle is measured with the support of deflecting spring and thus the viscous drag of the flowing fluid with respect to the spindle is measured. 3 sets of readings were taken for each sample and average of three values was used for analysis. The obtained results were compared with the mathematical model. The viscosity of the blend of two or more liquids can be estimated using the Refutas equation suggested by [20] Where $Y_i$ is the mass fraction of each component and VBNi is the viscosity based number for each constituent. "Eqs. (4), (5) and (6)" are utilized in the present work to calculate theoretical viscosity of the samples.

$$VBN = 14.534\ ln\,[ln\,(\nu + 0.8)] + 10.975 \tag{4}$$

$$VBN_m = \sum_{i=1}^{n} [Y_i . VBN_i] \tag{5}$$

$$\nu_m = exp\left(exp\left(\frac{VBN_m - 10.975}{14.534}\right)\right) - 0.8 \tag{6}$$

### 3.3. Density Measurement

The density of the Nano fluids were measured with a weighing machine loaded with a measuring jar where the weight of the nanofluid was measured for the given volume and a trial of 5 readings were taken and the average value was obtained. The calibration was done using distilled water with an obtained value of 0.997 kg/m3 which was very close to the standard value (1000 kg/m3). The "Eq.7" was suggested by [25] for ternary fluid and it was used for validating the experimental results and can be used for future research.

$$\rho_{hnf} = \left(\varphi_{np1}\rho_{np1} + \varphi_{np2}\rho_{np2} + \varphi_{np3}\rho_{np3}\right) + \left(1 - \varphi_{np1} - \varphi_{np2} - \varphi_{np3}\right)\rho_{bf} \tag{7}$$

### 3.4. Specific heat Measurement

The specific heat of the given samples was calculated using a Differential Scanning Calorimeter. The readings were taken after the calibration. The average of 5 readings for each cycle of all the three samples was obtained. The "Eq.8" was suggested by [25] for ternary fluid and it was matching with the experimental results and can be used for future research.

$$C_{phnf} = \frac{\varphi_{np1}\,\rho_{np1}C_{p,np1} + \varphi_{np2}\,\rho_{np2}C_{p,np2} + \varphi_{np3}\,\rho_{np3}C_{p,np3} + \left(1 - \varphi_{np1} - \varphi_{np2} - \varphi_{np3}\right)}{\rho_{hnf}} \tag{8}$$

## 4. Results and discussion

### 4.1. Thermal conductivity of hybrid nanofluids

The thermal conductivity of tri hybrid oxide nanofluids were measured for all the three samples at 0.1% volume concentration and among the three samples, sample (Z) had better thermal conductivity than the other two samples. The reason for the better thermal conductivity of sample (Z) was due to increase in the presence of $Al_2O_3$ which had better thermal conductivity than the other two particles (X) and (Y). The thermal conductivity of the nanofluid increases with the raise in temperature due to Brownian motion of the Nano particles. The Experimental results of [18] and [24] were matching with the base fluid result of the present work and thus experimentally validated. The maximum enhancement of thermal conductivity is 18.33 % more than the base fluid for sample (Z) at $50^{\circ}$ C.

### 4.2. Viscosity of hybrid nanofluids

The viscosity of the nanofluids decreases with the increase in temperature. The viscosity decreases with the increment of temperatures because of the weakening of inter-molecular and inter-particle adhesion forces. There was about 43.7 % of increase in viscosity at just 0.1% weight concentration of nanofluid compared to the viscosity of the water at 50°C for sample (Z). The viscosity of nanofluid plays a major role in the fluid properties. The viscosity based numbers were used to find the theoretical viscosity. The effect of viscosity on nanofluids was more than the base fluid, due to the addition of particles and the effect of viscosity is more for sample (Z) than sample (Y) and followed by Sample (X) due to increase in concentration of alumina.

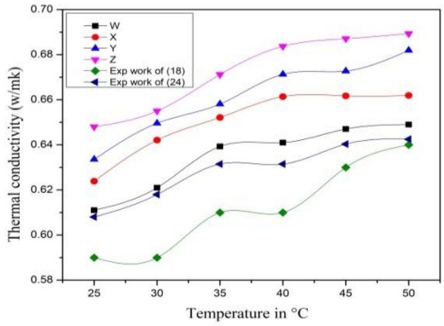



*Fig. 5. Variation of thermal conductivity with the increase in temperature.*

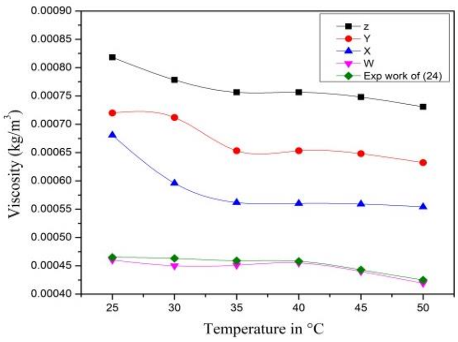



*Fig. 6. Variation of viscosity with the increase in temperature.*

### 4.3. Density of hybrid nanofluids

The density of nanofluids decreases with the increase in temperature and found to be matching with the density model proposed for calculating density. The density for the sample (Z) was higher than the other two samples (Y) and (X) and also it is to be noted that this may be due to the higher concentration of $Al_2O_3$. The further addition of $Al_2O_3$ tends to increase the density of the nanofluid. The results obtained were compared with the results of [18,24] and found to be of good matching.

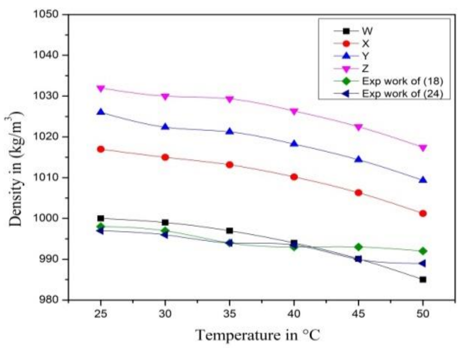



*Fig. 07. Variation of Density with the increase in Temperature.*

### 4.4. Specific heat of hybrid Nanofluids

The specific heat of nanofluids was higher than the base fluid. The sample (Z) shows lower specific heat value than the other two samples (X and Y). The base fluid tends to have better specific heat than all the three tested nanofluid samples. The experimental values were matching with the previous experimental work of [18,24] as shown in Fig.08. The specific heat remains to be almost a constant for the measured temperature range for all the three samples.

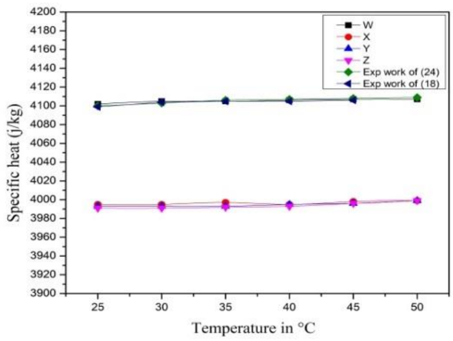
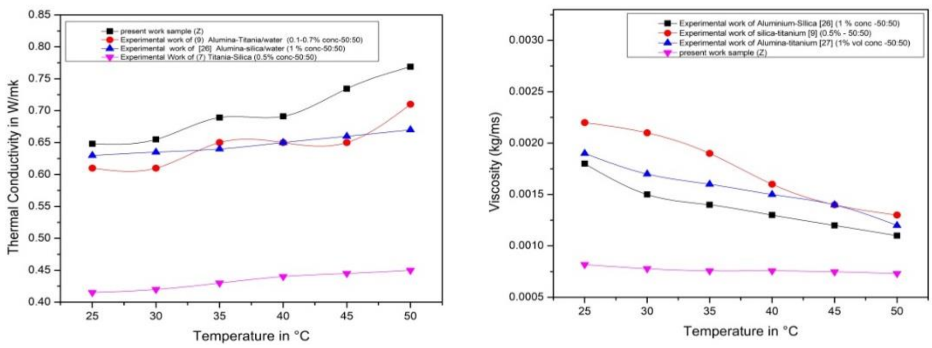



*Fig. 08. Variation of Specific heat with the increase in temperature.*

### 4.5. Validation and Comparison:

#### *4.5.1. Validation and comparison of results*

The obtained tri-hybrid oxide nanofluids results are compared with the di hybrid oxide nanofluids results of previous authors (refer fig.09). The thermal conductivity of present work was more than all the compared oxide di hybrid nanofluids. The thermal conductivity was more because of the improved Brownian motion of particles, augmentation of the particles and the inter molecular forces. Though the concentration is more in comparison for di hybrid nanofluids the thermal conductivity is more for the present tri hybrid oxide nanofluid due to the above said reasons. The thermal conductivity peak values in comparison with present work sample (z) were 13 % more in comparison with the previous work [9], 14% more in comparison with [26] and 70 % more in comparison with [7]. The reason for 70 % enhancement in the present work was due to the raise in thermal conductivity with the addition of $Al_2O_3$ in comparison with low thermal conductivity values of $SiO_2$ and $TiO_2$ combination. In comparison the viscosity values were less for present work but it was due to the low concentration of nanoparticles than the compared previous di hybrid nanofluid work and moreover better tradeoff is guaranteed with the tri-hybrid nanofluid.

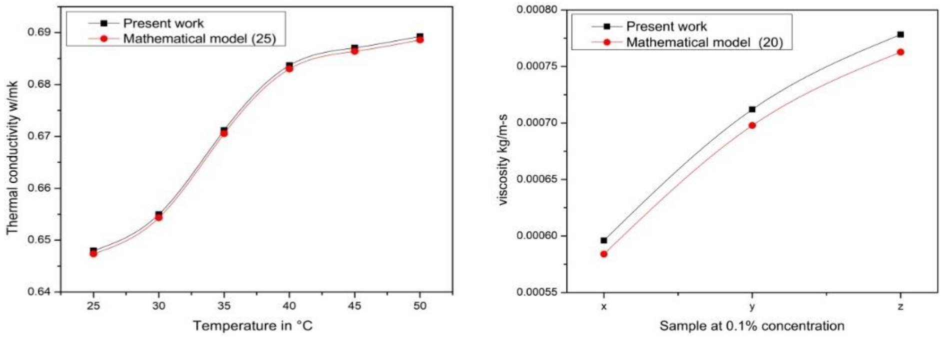



*(a)* *(b)*

*Fig. 09. Comparison of di hybrid nanofluid (previous authors work) with tri hybrid nanofluid (Present work) a) Thermal conductivity and b) viscosity.*

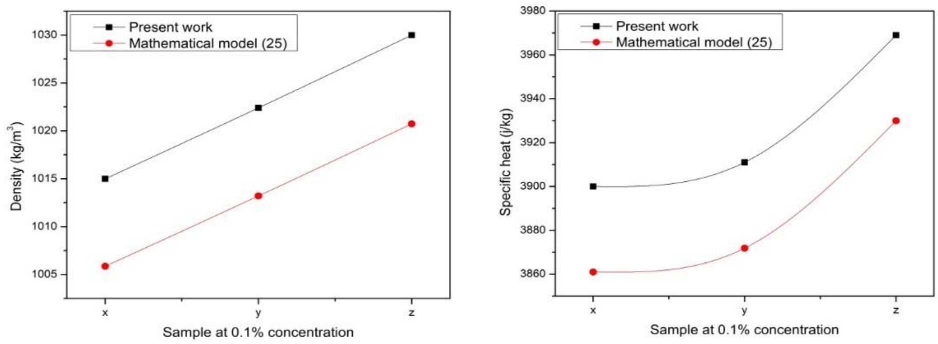
### *4.5.2. Mathematical model validation:*

The obtained results through the experimental work were compared with the mathematical models. Fig 10 and Fig.11 suggested by previous authors thermal conductivity, Specific heat, Density [25] and Viscosity [20] for tri hybrid nanofluid. In comparison the deviation was only about less than 1% for all the respective experimental values. Therefore the models were valid and can be used for future research on tri hybrid nanofluids




*(a)* *(b)*

*Fig. 10. Comparison of present work and Mathematical Models for a) Thermal conductivity and b) Viscosity.*




*(a)* *(b)*

*Fig. 11. Comparison of present work and mathematical models for a) Density and b) Specific heat.*

## 5. Conclusion

To conclude, in the current work, the tri hybrid oxide nanoparticles were prepared using two step methods. The Characterization of nanoparticles were examined and found to be of Nano particle in the required composition and also with a stability of 30mv. The EDAX testing confirms the presence of $Al_2O_3$, $TiO_2$ and $SiO_2$ at the required proportions. The nanoparticle tested samples were sized to be around 16.85nm by the XRD analysis. The thermal conductivity, viscosity, specific heat and density were measured using the testing equipment suggested by the authors of previous works respectively and compared with the previous results. The correlation models for validation were found to be of good matching. The nanofluids tend to have better thermal conductivity than the base fluid. The thermal conductivity maximum enhancement is found to be 18.33% from the base fluid for sample (Z) at 50 $^{o}$C and the maximum viscosity enhancement is found to be as 43.79%. . The thermal conductivity corresponding peak values of present work of sample (z) were 13 % more in comparison with the previous work of [9], 14% more in comparison with that of [26] and 70 % more in comparison with that of [7].The specific heat is less for nanofluids than the base fluids. The density of nanofluids decreases with the increase in temperature.

In the current work, the nanofluids are tested for volume concentration of 0.1% dispersed in 100ml of base fluid. The characteristics indicate that the tested samples were more suited for heat transfer applications as they tend to have better thermo physical properties. The heat transfer rate may further increase with the increase in volume concentration and temperature. For the first time ever in the present work all the exclusive mathematical models for the tri hybrid nanofluids suggested were validated by comparing with the experimental results. The utilization of tri-hybrid nanofluids will enhance thermo-physical properties better than oxide based di-hybrid nanofluids [7,9,26], therefore further investigations are needed to find the heat transfer characteristics for implementing the nanofluid as a working fluid in heat transfer equipment and also rheological properties are yet to be examined and are suggested for future work.

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