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# Synthesis and characterization of Aluminumsilicon oxide hybrid nanofluid

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## ABSTRACT

Nanofluid is primarily used for enhanced performance due to thermal properties, chemical stability, less toxicity, economical and ease of availability in various applications in day-to-day life. The nanofluid is prepared with colloidal suspension of nanoparticles in base water. This paper explained synthesis of  $Al_2O_3$ ,  $SiO_2$  and hybrid nanofluid of  $Al_2O_3$  and  $SiO_2$  in 90:10 ratio using sol-gel method. The steadiness of nanofluid is investigated using visual analysis technique. The sedimentation is done for 0 hours, 24 hours, 1 week, 2 weeks and 4 week of period. The SEM and EDS analysis is performed to analyze nature of  $Al_2O_3$ ,  $SiO_2$  and hybrid nanofluid of  $Al_2O_3$  and  $SiO_2$ . The XRD analysis is done to understand phases of  $Al_2O_3$  and  $SiO_2$  at peak positions of 20.

Keywords: Nanofluid, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Sol-Gel Method

## 1. INTRODUCTION

Nanotechnology has wide applications in refinery and petroleum industry for extraction of oil which is not done with primary or secondary oil retrieval method. The oil extraction increases with enhancement of surface tension of the oil with use of nanofluid. Nanofluid is prepared from base oil with colloidal suspension of nanoparticles (1),(2),(3),(4),(5),(6). The silicate nanomaterials and metal oxides have used for applications in nanofluids due to better performance including thermal behaviour, chemical stability, less deadliness, economical and ease of availability (7). Nanofluids are classified into single-material and hybrid nanofluids (8). Single-material nanofluid is the traditional nanofluid with suspension of single type of nanoparticles. Hybrid nanofluid is produced from mixture having suspension with multiple variety of nanoparticles in a base liquid(9). The Al<sub>2</sub>O<sub>3</sub> nanoparticles are preferred due to the better phase stability with dispersion in water, better dimensional stability and costeffectiveness(10),(11). The SiO<sub>2</sub> nanoparticles are useful because of low toxicity, better thermal behaviour, and ease of synthesis. It can be precisely controlled particle size, crystallinity, porosity, and shape (12), (13). The thermal performance is enriched with Copyrights @Kalahari Journals Vol. 6 No. 2(September, 2021)

nanoparticles in the nanofluid at a very low concentration of 0.1%. The hybrid nanofluid of Al<sub>2</sub>O<sub>3</sub>-Cu/water shows marginally more frictional factor than nanofluid of Al<sub>2</sub>O<sub>3</sub> and water owing to the high viscosity(14). The surfactant has significance in dispersion of nanoparticles to make nanofluids more stable. Further it is observed that the Al<sub>2</sub>O<sub>3</sub> get better dispersed in water due to the lower concentration, though it tends to aggregate with more amount. Thus, the mixing of nanoparticles enhances the rheological properties of hybrid nanofluids(15). The shape of the nanoparticles made for Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> is spherical with a size of 14 nm for Al<sub>2</sub>O<sub>3</sub> and 43 nm for TiO<sub>2</sub> nanoparticles. The absorption spectra indicate strong peaks at 344 nm for Al<sub>2</sub>O<sub>3</sub> and 483 nm for TiO<sub>2</sub> (16). It was observed better dispersion of nanoparticles at a less and average Al<sub>2</sub>O<sub>3</sub> concentration in water base liquid. The higher surface to volume ratio of NPs forms aggregation at a high level mass fraction owing to more surface energies of the NPs that promotes formation of aggregate which reduces the surface energies(17). The sol-gel method is used for synthesis of silica nanospheres with the optimum parameters at calcination temperature of 700 °C and aging time of 2 hrs. The range of average size of silica nanoparticles was achieved between 79.68 nm to 87.35 nm (18). The thermal behaviour, chemical stability and properties of hybrid nanofluids are enhanced than the traditional single material nanofluid(19). The SiO<sub>2</sub>-TiO<sub>2</sub>/polyacrylamide (PAM) nanofluids have more stability about a month or more. But a small decrease of viscosity at higher temperature shows that the nanofluids are useful for elevated thermal applications(20). Most of the researchers have studied effect of blending Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> particles in nanofluid as an individual component. But limited studies are done with use of hybrid nanofluid of Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub>. The synthesis of hybrid nanofluid of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> with sol-gel method in ratio of 90:10 is explained in this study.

## 2. MATERIALS AND METHODS

#### 2.1 Chemicals

The synthesis of nanoparticles is carried out using the top-down and bottom-up methods. The top-down method is where bulk material is physically broken into various nanosized particles or structures. Whereas, the bottom-up method, also referred to as the 'wet' method is a synthesis of nanoparticles through chemical reactions among the atom, ions, or molecules. The preparation of  $Al_2O_3$ -SiO<sub>2</sub> nanoparticles was done with the help of the Sol-Gel method which required numerous lists of chemicals, which are  $AlCl_3$  (aluminium trichloride) of 99%, purity level was used during the synthesis of  $Al_2O_3$  as the precursor. The C<sub>2</sub>H<sub>5</sub>OH (Ethanol) of purity at 95%, NH<sub>3</sub> (ammonium hydroxide) solution of 28%-30%, TEOS (tetraethyl orthosilicate), CH<sub>3</sub>COOH (acetic acid), and distilled water solvent used in this synthesis. The SDS Anionic surfactant used for scattering the particles and enhances the suspensions to make more stable.

#### 2.2 Synthesis of Al<sub>2</sub>O<sub>3</sub> Nanoparticles

The precursor made up of 0.1M AlCl<sub>3</sub> is combined with an ethanolic solution is used. The mixture is solubilized by stirring solution for 30 minutes at 40<sup>o</sup>C.Further 28% of NH<sub>3</sub> drops is mixed in the solution mixture with speed of 2.5 mL/minute at constant stirring to get a gel-like substance. This gel-like substance is kept for the finishing reaction for 30 hours at room temperature. Then, it is soaked at 100<sup>o</sup>C by keeping 24 hours in an oven. The gel is calcined at  $100^{\circ}$  C for 2 hours in a furnace to form Al<sub>2</sub>O<sub>3</sub> nanoparticle powder as shown in Fig.1.

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Fig.1 Synthesis of Al<sub>2</sub>O<sub>3</sub> nanoparticle powder

# 2.3 Synthesis of SiO2 Nanoparticles

The tetraethyl orthosilicate (TEOS) is used as a precursor in synthesis of SiO<sub>2</sub> nanoparticles. Initially, TEOS (18 ml) is mixed with acetic acid (36 ml) and distilled water (6.4 ml). Further, the blend solution is stirred for 10 min. Then the compound solution is centrifuged. This centrifuged solution is washed with ethanol (20 ml) further it is again centrifuged for precipitate separation. The precipitate was soaked at 60<sup>o</sup>C for 24 hours. Lastly, it is calcined at700<sup>o</sup>C for 90 min to get SiO<sub>2</sub> nanoparticle powder as shown in Fig.2.

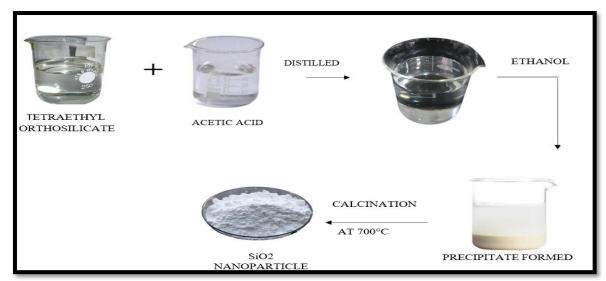


Fig.2 Synthesis of SiO<sub>2</sub> nanoparticle powder

# 2.4 Preparation of Al<sub>2</sub>O<sub>3</sub>/SiO<sub>2</sub> Hybrid Nanofluid

The Nano-powder aluminum oxide  $(Al_2O_3)$  with diameter of particle 30 nm and silicon dioxide  $(SiO_2)$  with a diameter of particle 20 nm are mixed to form a hybrid nano powder of  $Al_2O_3+SiO_2$  in a ratio of 90:10 as shown in Fig.3.This hybrid nanoparticle powder further gets blended with water as a base fluid to form the hybrid nanofluid. With two step technique, which is obtaining nanoparticles and then adding them to the base fluid, the nanoparticle powder was dispersed into 150 ml distilled water with 1.0% w/v inside a beaker with the help of sonication which was done at the frequency of 50 Hz for a time period of 3 hours at room temperature. Moreover, for this volume of nanofluid, approximately 1.5 grams of hybrid nanopowder was consumed which was in the ratio of 90:10.

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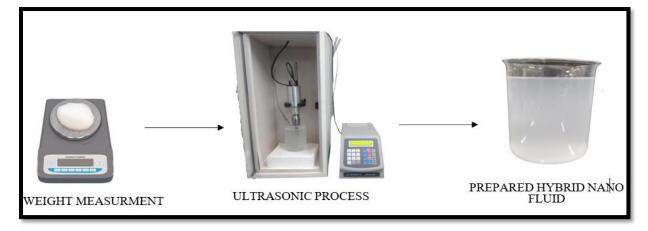


Fig.3 Preparation of hybrid-nano fluid Al<sub>2</sub>O<sub>3</sub>+SiO<sub>2</sub> with the help of ultrasonic probe sonication

## 3. RESULTS AND DISCUSSION

From visual observation, the prepared hybrid Nanofluid of Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> of 90:10 ratio and volume fraction of 1.0% was white in color. Further, it was evaluated for stability using the visual analysis technique. The visual analysis was carried out for periods of 0 hours, 24 hours, 1 week, 2 weeks, and 4 weeks are as shown in Fig.4. Based on figure 4(a), the hybrid Nanofluid was freshly prepared and was completely stable with no sign of phase separation or sedimentation. In figure 4 (b), the hybrid nanofluid had completed 24 hours of preparation and indicating slight separation of phase, with very little formation of a clear layer in the test tube. In figure 4 (c), the hybrid nanofluid had completed 1 week of preparation and revealed that the phase separation was more evident and it resulted in the formation of the clear layer in a test tube. Besides, we can observe some sedimentation at the lowermost inside the test tube. In figure 4 (d), the hybrid Nanofluid had completed 2 weeks after the preparation and the phase separation happens more dynamically in this period. The clear layer of fluids appeared to be better perceptible in the test tube and sedimentation had occurred largely. In figure 4 (e), the hybrid Nanofluid had completed a total of 4 weeks' time period from the preparation, and, notably, maximum phase separation had appeared with a clear layer of fluids significantly visible in the test tube. Moreover, the sedimentation height of nanoparticles in a test tube has increased greatly.

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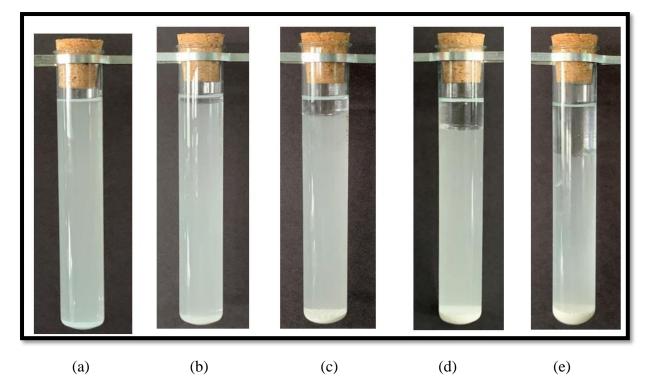


Fig.4 Sedimentation Analysis (a) 0 Hrs (b) 24 Hrs (c) 1 Week (d) 2 Week (e) 4 Week

The theoretical thermal conductivity model was used to find the thermal conductivity of the hybrid nanofluid. The Maxwell model which is employed for the hybrid nanofluids is ,

$$Khnf = Kbf \frac{(\frac{(\phi p1Kp1 + \phi p2Kp2)}{\phi tot} + 2Kbf + 2(\phi p1Kp1 + \phi p2Kp2) - 2\phi totKbf)}{(\phi p1Kp1 + \phi p2Kp2)} + 2Kbf - (\phi p1Kp1 + \phi p2Kp2) + \phi totKbf}$$

Here,  $\Phi$  – Volume fraction of nanoparticles

 $\operatorname{Tot}-\operatorname{Total}$ 

 $K1-Thermal\ conductivity\ of\ Al_2O_3$ 

 $K2-Thermal\ conductivity\ of\ SiO_2$ 

Kbf – Thermal conductivity of the base fluid (water)

Khnf – Thermal conductivity of hybrid Nanofluid.

## Given Data -

- 1.  $\Phi 1 0.09$
- 2.  $\Phi 2 0.01$
- 3. K1 40 W/mK
- 4. K2 1.4 W/mK
- 5. Kbf 0.598 W/mK

The Maxwell equation model for thermal conductivity of hybrid nanofluid becomes,

Khnf – 0.598 \* (39.4685/36.2697) W/mK

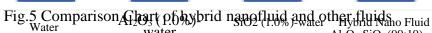
## Hence, Khnf = 0.6507 W/mK

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0.7 0.68 0.66 0.64

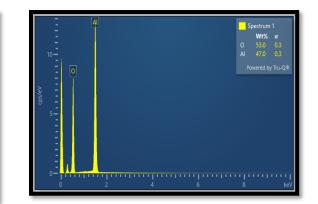
The prepared hybrid Nanofluid has also undergone a thermal conductivity test using the KD2 Prodevice. The device measured thermal conductivity in three tests and the final test output for thermal conductivity was found to be 0.6315 W/mK.



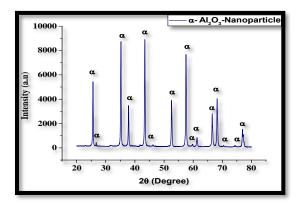
The chart shown in Fig.5 reveals to us the thermal conductivity  $O_1^{Al_2O_3}$ ,  $SiO_2^{(90:10)}$ ,  $Al_2O_3$ water-based Nanofluid with 1% w/v concentration, SiO<sub>2</sub>-Water-based Nanofluid with 1% w/v concentration, and Hybrid Nanofluid (Al<sub>2</sub>O<sub>3</sub>-SiO<sub>2</sub> in 90:10 ratio) with 1% w/v concentration. Moreover, we can observe that the hybrid nanofluid has better thermal conductivity compared to pure water, and only Al<sub>2</sub>O<sub>3</sub> water-based nanofluid, while the single element SiO<sub>2</sub> water-based nanofluid has a slight edge over the hybrid nanofluid but the cost and availability of SiO<sub>2</sub> does not serve the purpose to be as efficient as the hybrid nanofluid. The morphology of Al<sub>2</sub>O<sub>3</sub> is shown with SEM analysis in Fig.6 (a). SEM analysis of Al<sub>2</sub>O<sub>3</sub> reveals white non- spherical structured and agglomerated nanoparticles with average primary size between 30-50 nm. The EDS analysis for  $Al_2O_3$  is shown Fig. 6 (b) and (c) showing Wt % 53 % of O and 47 % of Al at spectrum 1 while 51.2 % of O and 48.8 % of Al at spectrum 2. The X-ray diffraction analysis of  $Al_2O_3$  is revealed in Fig.6 (d). The synthesis of  $Al_2O_3$  shows the distinct peak positions of  $\alpha$ -  $Al_2O_3$  in the diffraction pattern. The peaks at the different  $2\theta$  positions shows similarities while peak at  $44^0$  shows maximum higher reflection revealing crystalline form. The morphology of SiO<sub>2</sub> is shown with SEM analysis in Fig. 9. The SEM analysis of SiO<sub>2</sub> indicated that white colored spherical structured nanoparticles of average primary size around 40-70 nm. The EDS analysis for SiO<sub>2</sub> showed in in Fig. 7(b) and (c) revealing Wt % 63.1 % of O and 36.9 % of Si at spectrum 3 while 59.6 % of O and 40.4 % of Si at spectrum 4. The X-ray diffraction analysis of SiO<sub>2</sub> is revealed in Fig.7 (d). The highest peak at  $23^{0}$  of  $2\theta$  position shows that amorphous nature without crystalline form. The SEM analysis of hybrid nanofluid of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> shows mixed nature of non- spherical structured with agglomerated of Al<sub>2</sub>O<sub>3</sub> nanoparticles and spherical with regular shape of SiO<sub>2</sub> nanoparticles. The EDS analysis of hybrid nanofluid in Fig. 8 (a) to (d) shows Wt % 48.6 % of O,42.2% of Al ,9.2 % of Si at spectrum 5, 58.0 % of O,34.2 % of Al and 7.8 % of Si at spectrum 6, 53.7% of Al,37.2 % of O and 9 % of Si at spectrum 7. The X-ray diffraction analysis of hybrid nanofluid shown in Fig.8 (e) shows large no. of  $Al_2O_3$  peaks than the SiO<sub>2</sub>. The height of  $Al_2O_3$  peaks is comparatively higher than SiO<sub>2</sub> peaks.

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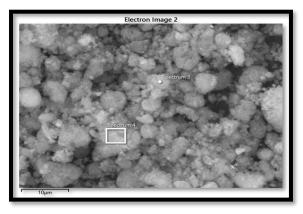


(b) EDS Analysis at Spectrum 1 of Al<sub>2</sub>O<sub>3</sub>



(d) XRD Analysis of Al<sub>2</sub>O<sub>3</sub>

Fig.6 SEM , XRD and EDS Analysis of  $Al_2O_3$ 

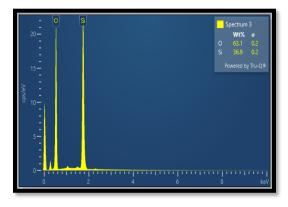


(c) EDS Analysis at Spectrum 2 of Al<sub>2</sub>O<sub>3</sub>

Electron Image 1

(a) SEM analysis of Al<sub>2</sub>O<sub>3</sub>

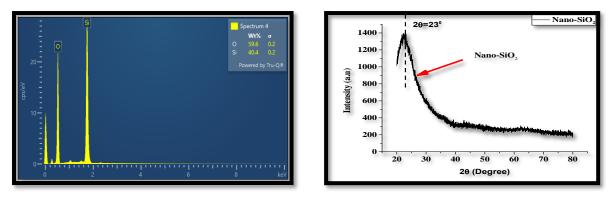
(a) SEM analysis of  $SiO_2$ 



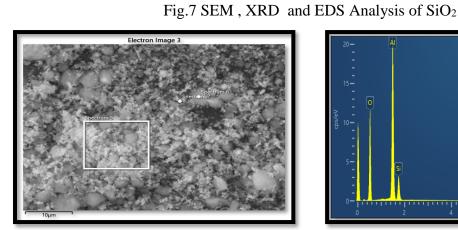
(b) EDS Analysis at Spectrum 3 of  $SiO_{\rm 2}$ 

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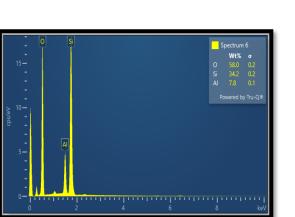
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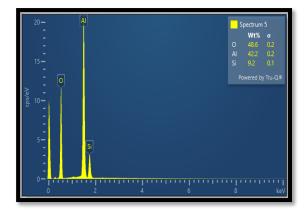
- (c) EDS analysis of Spectrum 4 of  $SiO_2$
- (d) XRD Analysis of SiO<sub>2</sub>



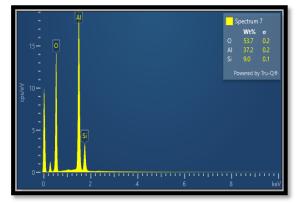
(a) SEM analysis of Hybrid Nanofluid



(c) EDS Analysis at Spectrum 6 of hybrid nanofluid



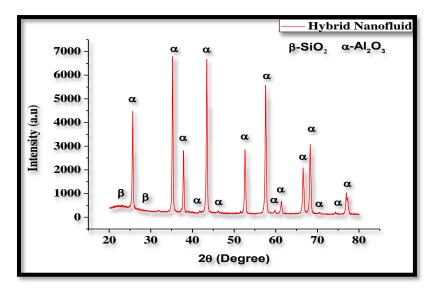
(b) EDS Analysis at Spectrum 5 of hybrid nanofluid



(d) EDS Analysis at Spectrum 7 of hybrid nanofluid

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(e)XRD analysis of hybrid nanofluid

Fig.8 SEM , XRD and EDS analysis of Hybrid Nanofluid  $(Al_2O_3 + SiO_2)$ 

# 4. CONCLUSIONS

This study discussed synthesis of Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub> and hybrid nanofluid of Al<sub>2</sub>O<sub>3</sub> with SiO<sub>2</sub>.The Al<sub>2</sub>O<sub>3</sub> nanoparticles have better properties such as better phase stability with dispersion in water, better dimensional stability while SiO<sub>2</sub> nanoparticles have properties like low toxic, better thermal behaviour. The stability of nanofluid is evaluated with visual analysis technique. The slight separation of phase indicated, with very little formation of a clear layer was observed after 24 hours of preparation. the phase separation was more evident and it resulted in the formation of the clear layer after 1 week of preparation. The phase separation occurs more vigorously after 2 weeks of preparation. The maximum phase separation had appeared with a clear layer of fluids significantly after 4 weeks of preparation. The hybrid nanofluid has better thermal conductivity than pure water, and only Al<sub>2</sub>O<sub>3</sub> water-based nanofluid, while the single element SiO<sub>2</sub> water-based nanofluid has a slight edge over the hybrid nanofluid but the cost and availability of SiO<sub>2</sub> doesn't serve the purpose to be as efficient as the hybrid nanofluid. SEM analysis of Al<sub>2</sub>O<sub>3</sub> reveals white non- spherical structured and agglomerated nanoparticles with average primary size between 30-50 nm. SEM analysis of SiO<sub>2</sub> indicated that white coloured spherical structured nanoparticles of average primary size around 40-70 nm. SEM analysis of hybrid nanofluid of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> shows mixed nature of non-spherical structured with agglomerated of Al<sub>2</sub>O<sub>3</sub> nanoparticles and spherical with regular shape of SiO<sub>2</sub> nanoparticles. The XRD analysis shown peak at 44<sup>0</sup> shows maximum higher reflection revealing crystalline form. The highest peak at  $23^{\circ}$  of  $2\theta$  position shows that amorphous nature. The X-ray diffraction analysis of hybrid nanofluid indicates mixed nature of form.

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