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Preparation and Stability Analysis of Water Based Al2O3, TiO2 and ZnO Nanofluids

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Abstract

For nanofluids to be able to use practically, they must not cluster and precipitate. Namely, they have to become stable. The target of this study is to determine the parameters that are effective at preparing stable nanofluid and to obtain stable one.To follow nanofluid stability, its sedimentation state is determined by photo capturing and controlling continuously. It is verified by SEM images that the nanofluids, which do not precipitate and are determined as stable, are distributed homogeneously and do not constitute considerable agglomerates. The work fluid is made from Al2O3, TiO2, ZnO nanoparticles and deionized water as base fluid. The solutions are prepared with 0.1%, 0.3%, 0.5%, 0.7% and 1.0% volume concentration. They are mixed 30 minutes by probe type of ultrasonic homogenizer at environment conditions. Sodium Dodecyl Sulfate (SDS) was added to the solutions as surfactant to prevent instability occurred due to agglomeration and sedimentation. At this study, it is investigated that from where the contradictory data for stability experiments in the literature stems. Moreover, the various stable nanofluid preparation parameters that are not available in the literature are given.It is observed that Al2O3, TiO2 and ZnOnanofluids have stability up to 5 days, 7 days and 21 days without considerably sedimentation, respectively. It is ascertained that properties of nanoparticle and nanofluid preparation parameters are important to enable stability.

Key words

Al2O3, TiO2, ZnO, Nanofluid, Stability, Surfactant

1. INTRODUCTION

Heating and cooling demands needed at many sectors like transport, electronic, nuclear, military, space, energy production play a rather important role for appearing new technologies **Error! Reference source not found.**, **Error! Reference source not found.**. To meet these demands at present applications, various methods are used. Some of them are increment at surface areas that heat transfer occurs, higher temperature difference for more heat transfer and material usage having durable to high temperature. However, it is already reached to usage limits of these methods due to the causes like dimensional limits, durable limits of material, production costs. Moreover, due to performance limits of available work fluids i.e. antifreeze, engine oil, fluids that have particles with mili-micrometer sized are used as a solution. Yet, instability occurred because of agglomeration and sedimentation at these particles induces clogging in microchannels and desired developments not be able to be obtained **Error! Reference source not found.**. With time, thanks to production technology developed, particles with nanometer sized and nanofluids have obtained. Nanofluid usage has started as work fluid.

Nanofluid is a suspension obtained by dispersing particles with nanometer sized in a fluid. Nanoparticle sizes used in nanofluids are generally between 1 nm and 100 nm **Error! Reference source not found.**, **Error! Reference source not**

found. - Error! Reference source not found.. Nanofluids are prepared by one of the methods called 1-step or 2-step. At 1-step method, nanofluid is prepared by chemical reaction at one-step. As for 2-step method, firstly, particle is produced at nanometer size, and then nanofluid is obtained by mixing them in a base fluid. Nanofluids obtained by 1-step method are more stable than ones obtained by 2-step. However, at 1-step method, particle size cannot be controlled. At 2-step method, nanoparticles needed can be found at desired size and property from many producers **Error! Reference source not found.**, **Error! Reference source not found.**.At 2-step method, nanofluids can be prepared by numerous sub methods: Magnetic stirrer, high shear mixer, ball mill, ultrasonic bath, probe type ultrasonic homogenizer, adding surfactant, changing pH, surface modification of particle **Error! Reference source not found.**, **Error! Reference source not found.**, **Error! Reference source not found.**. In 2-step methods, the most efficient and effective one is determined probe type ultrasonic homogenizer in the literature **Error! Reference source not found.**, **Error! Reference source not found.**, **Error! Reference source not found.**.Nanofluids are desired the properties like high thermal conductivity, high heat transfer performance, long stability time. However, they are not demanded clogging at microchannels due to agglomeration and sedimentation, and increase at pumping losses due to viscosity increment and pressure drop. These unwanted results are generally related to nanofluid stability. Stability of nanofluids can be examined by various methods: Ultra Violet-Visible Spectrophotometer (UV-Vis), Zeta potential, SEM, TEM, DLS, XRay Diffraction, sedimentation method, 3-omega, centrifugation method, photo capturing **Error! Reference source not found.**, **Error! Reference source not found.**, **Error! Reference source not found.**.Nanofluid stability is enabled by the methods like using surfactant, pH changing, modification of nanoparticle **Error! Reference source not found.**, **Error! Reference source not found.**. To enable nanofluid stability, the researchers who do not want a change at thermal properties of nanofluid did not use surfactant **Error! Reference source not found. - Error! Reference source not found.**. However, those who use surfactant desired it to prevent stability problem **Error! Reference source not found.**, **Error! Reference source not found. - Error! Reference source not found.**, **Error! Reference source not found.** Pg.31, **Error! Reference source not found.** Pg.57, **Error! Reference source not found.**.

Even after only ultrasonic mixing, attraction forces existed between particles cause them to cluster. Nanoparticle groups at micrometer and bigger sized occurred due to that clustering start to behave like particles at macro sized. For they have bigger density than base fluid, they make instability by collapsing the bottom of base fluid. Surfactants are used to prevent that sedimentation. Surfactant covers surface of nanoparticle and make repelling force between them. So, clustering of particles is prevented considerably. Only surfactant usage is not enough to enable stability. Because, when nanoparticles are dispersed in base fluid for the first time, since they are clustered, surfactant cannot affect among them. These agglomerations can be broken by ultrasonication**Error! Reference source not found.**.In the literature, there are different results for same nanofluids whose stability changes from 1 hour to 1 year **Error! Reference source not found.**. There are limited number of studies that include stable nanofluid preparation parameters and indicate them to be standardized **Error! Reference source not found.** Pg.76. This study works to determine the parameters that are effective at preparing stable nanofluid, to standardize these parameters and to obtain stable nanofluid.

This study consists of four sections: Material and Method, Experiment, Results, Conclusions. Material and Method section includes properties of used materials and how to be prepared nanofluids. Experiment section includes two subsections in the way nanofluid stability parameters and nanofluid SEM/TEM images.

2. 2. MATERIAL AND METHOD

2.1. Propertiesof Nanoparticles

Al2O3, TiO2 and ZnO nanoparticles are used to prepare nanofluid. Average sizes of these particles are 20 nm, 10-25 nm and 18 nm, respectively. Nanoparticles are bought from "Nanografi Ltd. Company". All properties of nanoparticles are given in Table 1, their TEM images supplied by the producer are given in Figure 1.Sodium Dodecyl Sulfate (SDS) as surfactant was used to prevent sedimentation of nanoparticles by clustering and to make nanofluid stability keep on. SDS was bought from "Merck Inc." The density of this matter is 1.1 g/cm3 and its pH value is between 6 and 9.

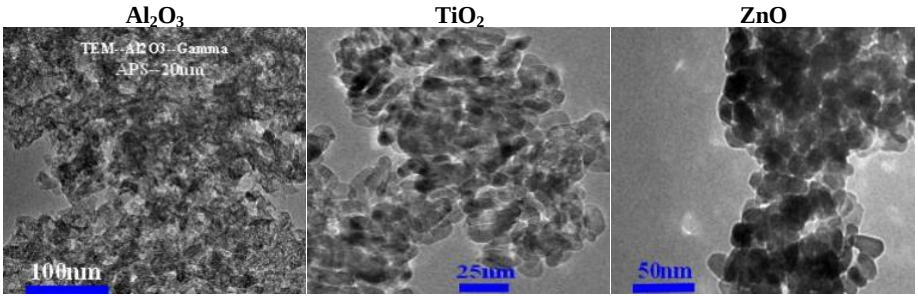


Figure 1. TEM images of nanoparticles

2.2. Preparationof Nanofluids

All nanofluids at this study were prepared by 2-step method. Probe type ultrasonic homogenizer was used to disperse nanoparticles in a deionized water (Ultrasonic Homogenizer Mark/Model: Optic Ivymen System / CY-500, Power: 500W, Frequency: 20kHz, Probe Diameter/Length: Ø5.6/60mm).Firstly, mass amounts of nanoparticle, deionized water and SDS were calculated in accordance with desired nanofluid volumetric concentration, nanofluid volume and SDS weight

concentration from

Figure 2. Preparation of nanofluid with temperature control

Table 2. These quantities were weighed by a precision balance (AND GX-600, Max Mass: 610g, Deviation: 0.001g). Then, nanofluids were prepared in a flask by paying regard to many parameters considered to be effective at nanofluid stability. The nanofluid taken from the bottom location of the flask by a pipet was filled in the glass tubes, which have Ø16x160 mm dimensions with screw thread. Stabilities of nanofluids were examined by photo capturing method according to time.

The most suitable parameters were determined by these images. According to these parameters, the most stable nanofluids were prepared.

The equations used in

Figure 2. Preparation of nanofluid with temperature control

Table 2 for nanofluids to be prepared are the following:

Volume concentration of nanofluid,

$$\phi = \frac{\forall_{np}}{\forall_{nf}} = \frac{\rho_{nf} - \rho_{bf}}{\rho_{np} - \rho_{bf}} \quad (1)$$

Volume of nanofluid,

$$\forall_{nf} = \forall_{np} + \forall_{bf} \quad (2)$$

Mass of nanofluid,

$$m_{nf} = m_{np} + m_{bf} \quad (3)$$

Density for nanofluid, nanoparticle and deionized water,

$$\rho = m/\forall \quad (4)$$

Weight concentration of SDS/Nanoparticle,

$$\phi = m_{SDS}/m_{np} \quad (5)$$

During mixing nanofluids by ultrasonic homogenizer, when it was not taken any precaution, too temperature increment was seen in the sample (nanofluid). Such that, this temperature increment reached 60 degrees in 10 minutes. During mixing, increased temperature affects both chemical-thermal properties of nanofluid and causes ultrasonic homogenizer to work unproductively **Error! Reference source not found.** Pg.32. At the experiments done in this study, it was seen that uncontrolled temperature increment decreased vibrations of ultrasonic homogenizer. This was noticed by change of ultrasonic sound and decrease at surge of the top surface of the sample. Therefore, a heat bath was used to hold nanofluid temperature constant (Mark/Model: Cole Parmer / EW-12108-25, Temperature: -20~200oC, Temperature Stability: ±0.01oC, Bath Capacity: 6L, Heating Capacity: 1kW, Cooling Capacity: 200W, Flow Rate: 11~24L/min). The flask

included nanofluid was put in the heat bath held at constant temperature as Figure 2. So, 100 mL of nanofluids were prepared. In addition, during ultrasonication, high temperatures were seen at the probe of ultrasonic homogenizer. To compensate it, the probe was cooled by a fan.

Table 1. Properties of nanoparticles

Nano Particle	Type	Density (kg/m3)	Purity	Average Size	Specific Surface Area	Shape
Al2O3	Gamma	3890	>%99	20 nm	138 m2/g	Close to spherical
TiO2	Anatase	3900	>%99.5	10-25 nm	200-240 m2/g	Close to spherical
ZnO	—	5606	%99.95	18 nm	40-70 m2/g	Close to spherical

Figure 2. Preparation of nanofluid with temperature control

Table 2.Nanofluids according to volumetric concentration at 20oC

Fluid	Volume Concent.	Nanofluid Volume	Base Fluid Density	Particle Density	Particle Volume	Base Fluid Volume	Particle Mass	Base Fluid Mass	SDS-Particle Weight Concent.	SDS Mass
	ϕ (%)	$\forall_{nf}$ (mL)	ρ_{bf} (kg/m^3)	ρ_{np} (kg/m^3)	$\forall_{np}$ (mL)	$\forall_{bf}$ (mL)	m_{np} (g)	m_{bf} (g)	$\phi_{w,SDS}$ (%)	m_{SDS} (g)
Al_2O_3	0,10%	100	998,0	3890	0,10	99,90	0,389	99,700	50,00%	0,195
	0,30%	100	998,0	3890	0,30	99,70	1,167	99,501	25,00%	0,292
	0,50%	100	998,0	3890	0,50	99,50	1,945	99,301	15,00%	0,292
	0,70%	100	998,0	3890	0,70	99,30	2,723	99,101	15,00%	0,408
	1,00%	100	998,0	3890	1,00	99,00	3,890	98,802	15,00%	0,584
TiO_2	0,10%	100	998,0	3900	0,10	99,90	0,390	99,700	50,00%	0,195
	0,30%	100	998,0	3900	0,30	99,70	1,170	99,501	25,00%	0,293
	0,50%	100	998,0	3900	0,50	99,50	1,950	99,301	15,00%	0,293
	0,70%	100	998,0	3900	0,70	99,30	2,730	99,101	15,00%	0,410
	1,00%	100	998,0	3900	1,00	99,00	3,900	98,802	15,00%	0,585
ZnO	0,10%	100	998,0	5606	0,10	99,90	0,561	99,700	50,00%	0,280
	0,30%	100	998,0	5606	0,30	99,70	1,682	99,501	50,00%	0,841
	0,50%	100	998,0	5606	0,50	99,50	2,803	99,301	25,00%	0,701
	0,70%	100	998,0	5606	0,70	99,30	3,924	99,101	15,00%	0,589
	1,00%	100	998,0	5606	1,00	99,00	5,606	98,802	15,00%	0,841

3. 3. EXPERIMENT

3.1. NanofluidStabilityParameters

The parameters that affect stability of nanofluids are classified as follows:adding surfactant, ultrasonic power intensity, ultrasonic mixing time, bath temperature, height of ultrasonic probe, flask diameter, nanoparticle type.

The effect of each parameter on nanofluid stability was investigated as subtitles. The information at the top row of the figures that show effect of these parameters includes (Figure 3-Figure 10): 1. Nanofluid volumetric concentration, 2. Nanofluid type, 3. Investigated parameter, 4. Time passed after preparing nanofluid.Stability was examined by the photos taken daily, according to be transparent from the top surface of the tube and sedimentation at the its bottom.

3.1.1. Adding Surfactant

In this section, it was investigated whether surfactant usage is necessary and if it is necessary, what its concentration value must be. For this purpose, the nanofluids included and not included surfactant were prepared (Figure 3). It was seen that the nanofluids not included surfactant were settled by agglomerating in 20 minutes (Figure 3-a,b,c). After that result was obtained, to enable nanofluid stability, SDS was selected as surfactant widely used in the literature **Error! Reference source not found.**, **Error! Reference source not found.**. At the nanofluids prepared by using SDS, it was seen that agglomeration was prevented and stability was enabled (Figure 3-d,e,f). As a result, it was decided to use SDS for all nanofluids. In order to determine SDS concentration, the nanofluids included SDS between 1% and 100% in the way SDS/Nanoparticle as mass were prepared (Figure 4). At Figure 4, for the nanofluid with 0.5% volumetric concentration, while stability was enabled for SDS with 15% and higher weight concentration, SDS with same 15% weight concentration was insufficient for the nanofluid with 0.2% volumetric concentration. From here, it was concluded that SDS having different weight concentration is necessary for nanofluids with different volumetric concentration. As general trend, it was seen that the more nanofluid volumetric concentration decreases, the more SDS weight concentration needed increases (Figure 4-d,e,f). By this way, the lower limit of SDS weight concentration was determined in a way to change from 15% to 50% for all nanofluids by some experiments (

Figure 2. Preparation of nanofluid with temperature control

Table 2). In the literature, SDS weight concentration is generally given as "Surfactant/Nanoparticle" (**Error! Reference source not found.**, **Error! Reference source not found.**, **Error! Reference source not found.**). Yet, in some studies, this ratio is also given as "SDS/Nanofluid" (**Error! Reference source not found.**, **Error! Reference source not found.**). Therefore, it must be paid attention which reference SDS concentration values are given according to.

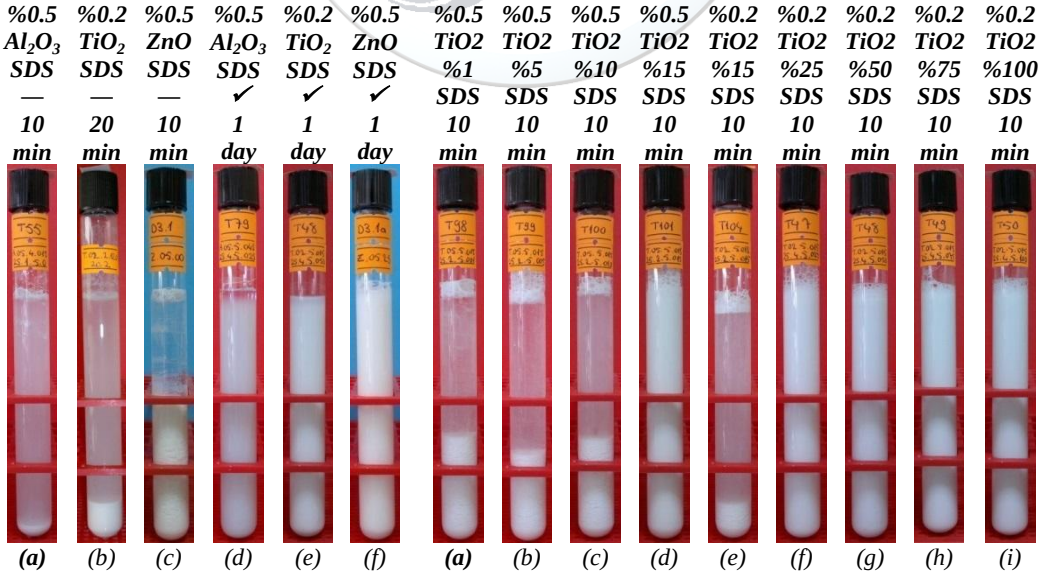


Figure 3. Controlling whether surfactant is necessary or not

Figure 4. Determining SDS weight concentration

It was worked to find a lower limit for using SDS. Because, SDS amount must be used many enough to enable nanofluid stability; it must be used little enough to decrease concretion, not to increase cost and not to affect thermodynamic properties.

3.1.2. Ultrasonic Power Intensity

Ultrasonic vibration power (W/mL) per nanofluid volume was investigated. For this purpose, three different nanofluids were prepared in a way to be their power intensity 3, 4, 5 W/mL (Figure 5). It was seen that the more bigger ultrasonic power intensity is, the more longer stability time is (Figure 5-d,e,f).As a result, it was decided that ultrasonic power must be applied to all nanofluids at full power (500 W) and this power must remain as a constant parameter.

3.1.3. Ultrasonic Mixing Time

It was examined for how much time it is necessary for the nanofluids to expose to ultrasonic vibration. For this, the nanofluids changed from 5 minutes to 240 minutes their mixing time were prepared (Figure 6). It could not be seen that mixing time lasting than 30 minutes had apparent effective on nanofluid stability time (Figure 6-e,f,g). Therefore, it was decided that all nanofluids must be exposed to ultrasonic vibration for 30 minutes. This time is same as numerous studies in the literature (**Error! Reference source not found.**, **Error! Reference source not found.** Pg.32, **Error! Reference source not found.**).

3.1.4. Bath Temperature

It was investigated at what temperature nanofluid must be during mixing it with ultrasonic homogenizer. For that purpose, during mixing, the nanofluids held their temperature constant at 20, 30, 40 and 50 degrees were prepared (Figure 7). It was not seen that temperature had significant effective on stability (Figure 7-e,f,g,h). Consequently, in order to prevent too temperature increase during mixing, it was seen that the heat bath is necessary to be held constant at any temperature. However, since high temperature causes efficiency of ultrasonic homogenizer to decrease and nanofluid volumetric concentration to change (increase) due to evaporating of water, it was made a decision that the heat bath must be held constant at 20-25°C environment temperature.

3.1.5. Ultrasonic Probe Height

Ultrasonic power that ultrasonic homogenizer gives fluid is mainly given from the top surface of the probe. Since conical volume that the tip surface of the probe makes and exposed vibrations changes, it was examined at how many height the probe must be from the bottom of the flask. To control that situation, the nanofluids were prepared by being held the probe at 1, 2, 3, 4 and 5 cm height (Figure 8). It was not seen that the probe height is effective on stability. As a conclusion, the probe can be held at any height from the bottom of the flask. Yet, even if it is little, to benefit from ultrasonic vibrations distributed from the side surfaces of the probe and to decrease noise during working, it was decided that the probe must be held at 1-2 cm height from the bottom of the flask.

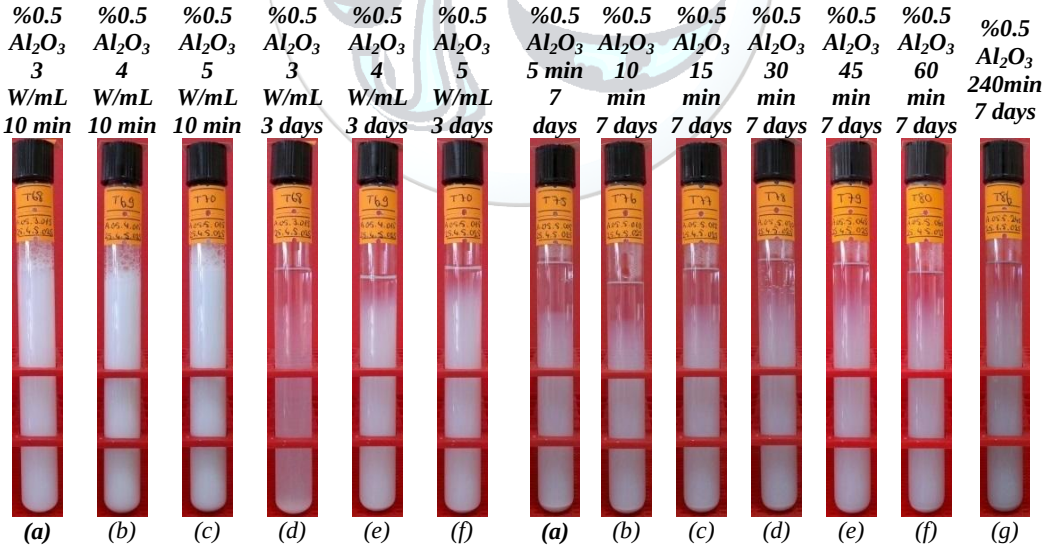
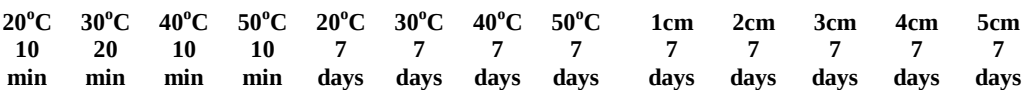
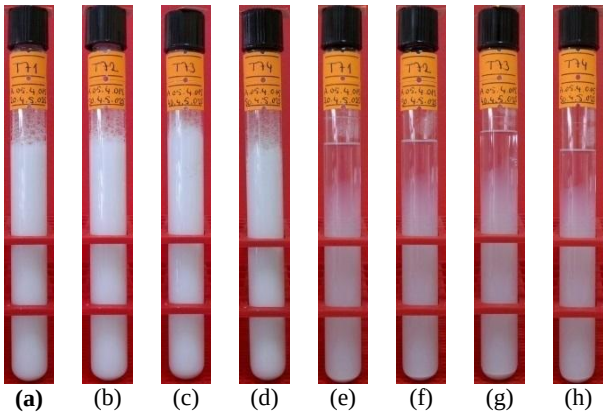


Figure 5. Determining ultrasonic power intensity

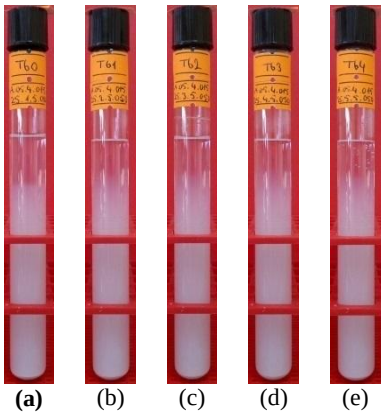
Figure 6. Determining ultrasonic mixing time





(a) (b) (c) (d) (e) (f) (g) (h)

Figure 7. Determining bath temperature (%0.5 Al2O3)



(a) (b) (c) (d) (e)

Figure 8. Determining height of ultrasonic probe

3.1.6. Flask Diameter

Due to the reasons described at the probe height section, it was examined what flask diameter must be. For this purpose, the nanofluids were prepared by using 5, 7 and 9 cm diameter of the flask (Figure 9). As determined at the probe height, similarly it was not seen that the flask diameter is effective on stability (Figure 9-d,e,f). As a result, a flask with any diameter can be used.

3.1.7. Nanoparticle Type

For nanofluids included different nanoparticles have different stability time, nanoparticle type was investigated effect on stability. The aim is to determine the nanofluid that had the longest stability time trend between different nanofluids. Al_2O_3, TiO_2 and ZnO nanoparticles were used to test stability time. The stability times of the nanofluids prepared were determined as ZnO, TiO2 and Al2O3 from the longest stability time to the least, respectively (Figure 10)

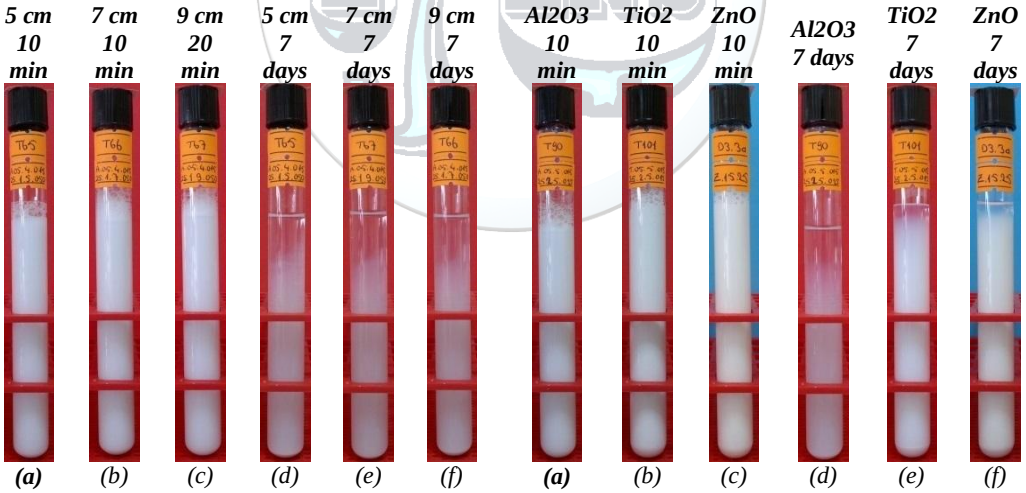


Figure 9. Determining flask diameter (0.5% Al2O3)

Figure 10. The effect of the nanoparticle type on the stability time (Vol. Cont. 0.5 %)

3.2. StabilityControl of Nanofluid

The nanofluids were prepared by considering the optimum values of the parameters that affect nanofluid stability time. The optimum parameters can be summarized as follows: Surfactant (SDS (

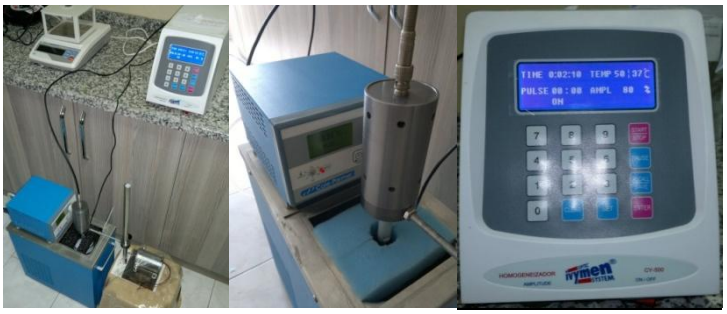


Figure 2. Preparation of nanofluid with temperature control

Table 2)), Ultrasonic power (500 W), Mixing time (30 min), Bath temperature (25^{o}C), Probe height (1-2 cm), Nanofluid volume (100 mL (

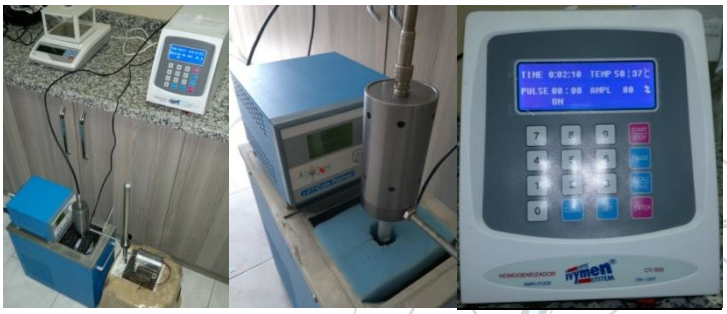


Figure 2. Preparation of nanofluid with temperature control

Table 2)).The nanofluids were prepared according to the values in

Figure 2. Preparation of nanofluid with temperature control

Table 2 under above conditions. Their images are given in Figure 11 three hours after preparing.Thenanofluids that have the longest stability time at Figure 11 were determined as 0.5%, 0.7% and 1.0% volumetric concentrations for Al_2O_3; 0.3% for TiO_2; all concentrations for ZnO. The stability time without apparently sedimentation for these concentrations was enabled up to 5 days for Al_2O_3, up to 26 days for TiO_2, up to 21 days for ZnO (Figure 12). It changed from some days to 2 weeks for other concentrations. As average, it can be said that the stability time continued up to 5 days for Al_2O_3nanofluid, 7 days for TiO_2nanofluid, 21 days for ZnOnanofluid.

SEM images were taken in order to confirm that the stable nanofluids prepared were distributed homogeneously and did not include big clusters according to the original nanoparticle size. Moreover, TEM images were taken in order to verify that they were at the desired size. SEM and TEM images of the Al_2O_3, TiO_2 and ZnOnanofluids with 0.5% volumetric concentration were given in Figure 14 and Figure 15.For SEM images, SEM device (FEI Quanta FEG 450, STEM Detector, 30 kV) in Bülent Ecevit University, Science and Technology Application and Research Center (ARTMER) was

used (Figure 13). As for TEM images, TEM device (FEI Tecnai G^2 Spirit BioTwin, CTEM, 120 kV) in Middle East Technical University, Central Laboratory, R&D Education and Measurement Center was used.

Figure 11. The nanofluids prepared with the optimum parameters

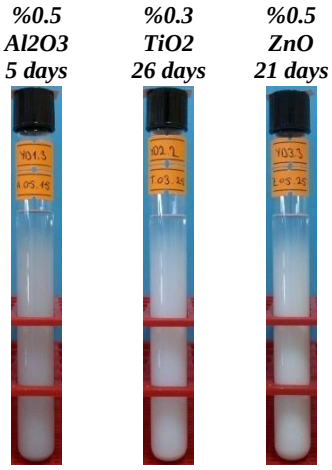


Figure 12. Stability time of the stable nanofluids

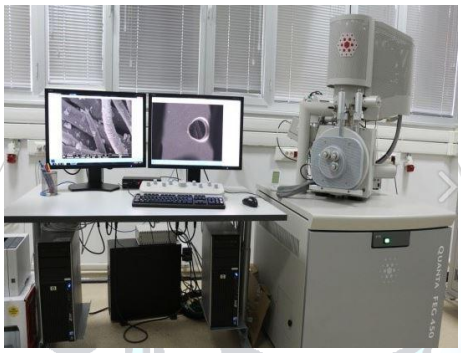


Figure 13. STEM device in ARTMER

4. RESULTS

To prevent sedimentation of nanofluids by agglomerating, SDS as surfactant is necessary. For this study, SDS weight concentrations are given in

Figure 2. Preparation of nanofluid with temperature control

Table 2.It is required to run the ultrasonic homogenizer at full power. In this study, the device was run at 500 W powers.The ultrasonic mixing time was determined as 30 minutes.The bath temperature was chosen as 25°C.

The tip of the ultrasonic probe was held at 1-2 cm height from the bottom of the flask.It was seen that Al_2O_3, TiO_2 and ZnOnanofluids prepared with the optimum parameters at 0.1%, 0.3%, 0.5%, 0.7% and 1.0% volumetric concentrations didn't show apparently sedimentation up to 5, 7 and 21 days as average, respectively.It was found that the nanofluids prepared with the optimum parameters remained stable max. 5 days for 0.5% Al_2O_3, 26 days for 0.3% TiO_2 and 21 days for

ZnO.SEM and TEM images were taken for the stable nanofluids. It was found that homogeneous distribution obtained and the nanoparticles were at the specified size from the images.

5. CONCLUSIONS

To minimize some controversial results (i.e. instability, stability time up to one year, anomalous thermal conductivity increase, too heat transfer increase) seen at different studies, nanoparticle properties used have to be same. Some of these properties are producer, nanoparticle size (20nm, 50nm), nanoparticle shape (spherical, cylindrical, porous), nanoparticle purity (99%) and quality (homogeneous size distribution, specified size and shape).

At nanofluid stability researches, zeta potential of nanofluid can be compared with images showed it remained stable. By this way, it can be appeared physically whether 30 mV of zeta potential limit value is enough (**Error! Reference source not found.**, **Error! Reference source not found.**, **Error! Reference source not found.**).

At SEM/TEM images given for nanofluid stability analyses, not only nanoparticle size images, but images at scale showed suspension distributed homogeneously must be also given.

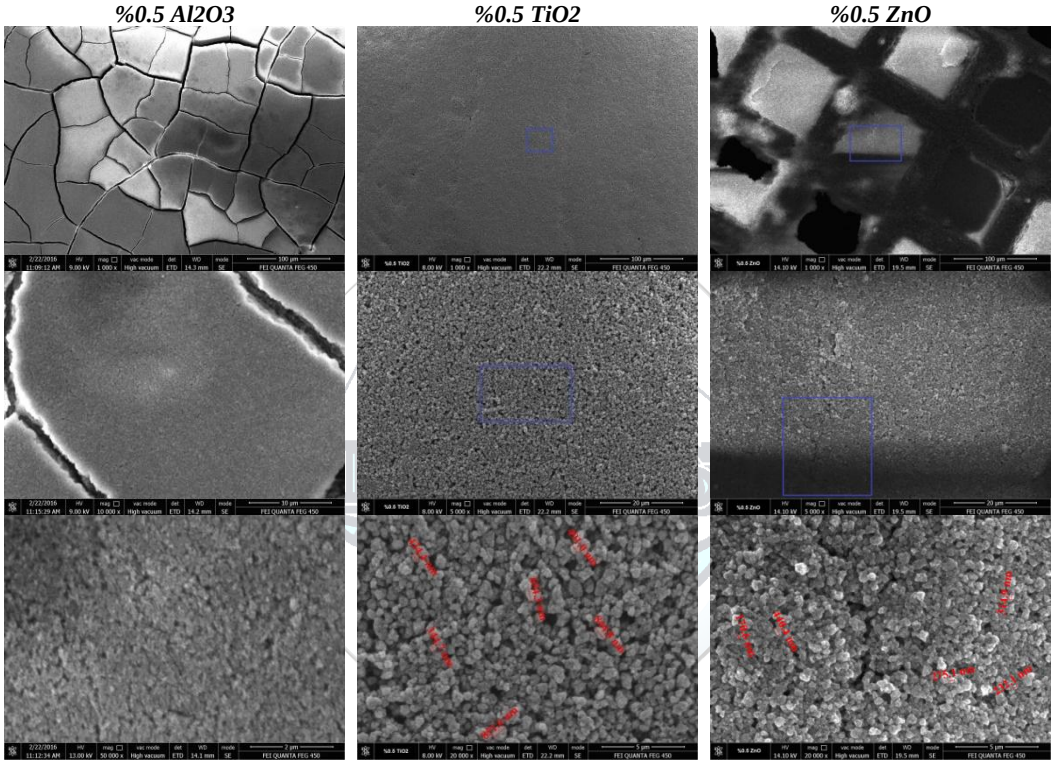


Figure 14. *SEM images the nanofluids with 0.5% vol. concentration*

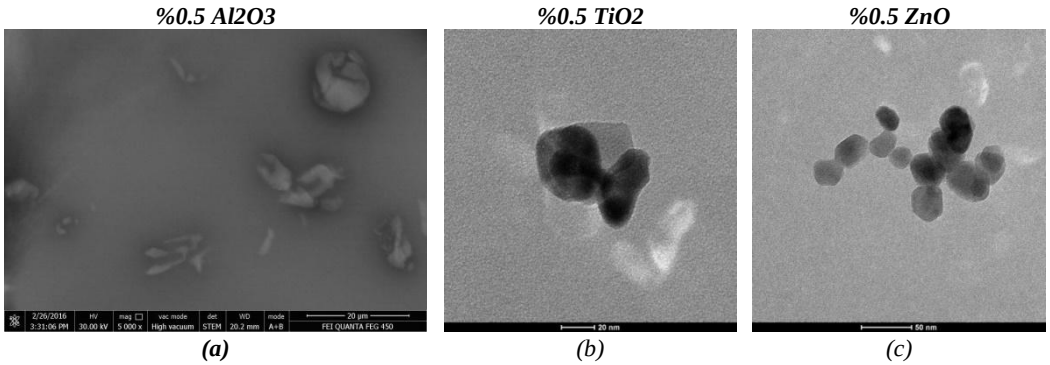


Figure 15. TEM images the nanofluids with 0.5% vol. concentration

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