Settling Characteristics of Alumina Nanoparticles in Ethanol-Water Mixtures

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Abstract. Nanofluids are considered as promising heat transfer fluids due to enhanced heat transfer ability as compared to the base fluid alone. Knowledge of settling characteristics of nanofluids has great importance towards stability of nanosuspensions. Sedimentation behavior of Alumina nanoparticles due to gravity has been investigated using different proportions of ethanol-water binary mixtures. Nanoparticles of 40 nm and 50 nm are used in this investigation at 23°C. Sediment height with respect to time is measured by visualization method in batch sedimentation. The effect of sonication on the sedimentation behavior is also studied using ultrasonic agitator. The effect of particle diameter, nanoparticle concentration and ethanol-water proportion on sedimentation behavior of nanofluids has been investigated and discussed.

Introduction

Suspension of nanoparticles in liquid is termed as nanofluid. Addition of nanoparticles in fluids at low concentration can alter the fluid properties, thus enhance the heat transfer process [1-3]. The settling of nanoparticles in a nanofluid after a certain period of time is a natural phenomenon due to gravity. This settlement of solid particles is the only predicament towards their usage at industrial level. The knowledge of the settling behavior of nanoparticles is of great importance towards stability of nanofluids as well as design and operation of heat transfer equipment. In settling process, aggregation takes place due to interaction among particles to form a cluster of colloidal particles. These aggregates are then settled down forming sediment layers. These sediment layers are increased and further consolidate under its own weight with the passage of time [4]. Agglomeration can cause clogging and settling in micro channels and can decrease overall thermal conductivity of nanofluid.

Yu and Xie [5] gave brief overview of evaluation methods for stability and techniques to increase stability of nanofluids. Murshed *et al.* [6] prepared TiO₂/water based nanofluids for the investigation of effective thermal conductivity of nanofluids using ultrasonic dismembrator to ensure proper mixing. The settled particles in the test tubes were considered to be big agglomerates, which can be reduced by applying any mechanical mixing technique or using surfactant or stabilizer [7-9]. Iritani *et al.* [4] have performed shown similar kind of investigation using $TiO₂$ particles. Consolidation-sedimentation behaviors of consolidated sediment under the action of gravity were studied for various conditions such as different pH, initial heights, and initial concentrations. Tiller and Khatib [10] investigated the state of aggregation in suspensions depends on the particle concentration and sediment volume. Li *et al*. [11] summarized recent research progress on the characterization and sedimentation of nanofluids. They used photographic technique to observe stability of nanofluid. Many studies were shown that the stability of nanosuspensions is directly affected by the amount of nanoparticles in nanofluid and the interaction between dispersant and nanoparticles [12]. Palabiyik *et al*. [13] studied sedimentation behavior of glycol nanofluids. Propylene glycol (PG), ethylene glycol (EG) and water-ethylene glycol (50:50 wt %) based nanofluids were used to investigate the surfactant, particle size and sunlight effect on the stability of nanofluids. Shirato *et al.* [14] proposed a model for the mechanism for settling of sediments

relevant to consolidation theory. Settling behavior of solid liquid mixtures can be classified into flocculation, dispersion and mixed settling (Fig. 1). In flocculated sedimentation, particles stick together to form clumps and settle down, which occurs in high concentrations of solids. Discrete or dispersed settling occurs in very low solid concentrations, in which the individual particles settle independently. In mixed settling, particles follow both flocculated and dispersed trend simultaneously.

In the present study sedimentation behavior of Al_2O_3 nanoparticles in mixtures of ethanol-water has been studied with and without ultrasonic agitation. During settling, sedimentation height is measured using visualization method in batch sedimentation. There is no much literature available on the sedimentation behavior of nanoparticles in liquids at low concentrations, hence the motivation.

Fig. 1 Three different types of settling behavior of suspensions with solid particles at different time intervals, where $t_0 < t_1 < t_2 < t_f$.

Materials and Methods

Aluminum oxide (alpha) nanoparticles of 40 nm (99.5% pure, MK-Nano, Canada) and 50 nm (99.9% pure, Sky Spring Nanomaterials, US) are used to study the sedimentation behavior. Ethanol and distilled water are used for the preparation of nanofluids. 90 samples of nanofluids are prepared with varying proportions of ethanol and water. All the experiments are conducted at 23^oC. All samples are prepared in wt/wt percentages of nanoparticles. The conventional batch sedimentation technique and photographic method is used to observe the settling characteristics of alumina suspensions. The investigation is conducted in three different concentrations $(0.1, 0.3 \text{ and } 0.5 \text{ wt%)}$ of alumina nanoparticles in water-ethanol mixture. The most common technique to study sedimentation behavior is visual observation, which was used by Wei and Wang [15] using micro fluidic copper nanofluids to study the sedimentation characteristics. Li *et al.*[12] used the same method for the study of stability of Cu-H₂O nanofluids with and without addition of dispersant. In the current study, two-step method has been used to prepare different samples of nanofluids. These mixtures are allowed to settle under gravity and observed the settling behavior. An ultrasonic agitator is used to mix the nanoparticles. The samples are sonicated for 24 hours and allowed to settle down under gravity. Ultrasonic agitator operates on the principle of mixing through ultrasonic waves. When ultrasonic waves travel through the fluid, the attenuation of waves results in agitation. The stability of nanofluids is observed with and without ultrasonic agitation. No surfactant or stabilizer is used in this study.

Results and Discussion

The height of the sediment for 90 samples are measured by photographic method with and without sonication. More amount of sediment is observed in higher concentrations of alumina nanoparticles. The sediment ratio in terms of volume is measured for each sample.

Sediment ratio = V_S/V_T (1)

where V_S is the volume of sediment and V_T represent total volume of sample.

Effect of Ethanol-Water Concentration. It is observed that the settling of nanoparticles in the ethanol-water suspensions is due to gravity and agglomeration effect. The height of sediment in the bottom is observed to be increasing with the passage of time which is evident from Fig. 2 (a). It is due to dispersed or discrete settling behavior for 8 hours, which is observed in all the samples of nanosuspensions. A slight decrease in the volume of sediment after 8 hours is observed due to compression settling.

Fig. 2 (a) Effect of time on sediment ratio using different volume fractions of Alumina nanoparticles at Ethanol=0%, (b) Effect of ethanol concentration on sedimentation behavior (0.5% Alumina 40 nm).

Fig. 3 (a) Effect of ethanol concentration (1-5%) on sedimentation behavior at 5 min and 15 min, (b) Effect of ethanol concentration (95%-100%) on sedimentation behavior at 30 min and 1 hour.

It is observed that the proportions of binary mixture of liquids have great influence on the sedimentation behavior. Fig. 2 (b) shows the relation of ethanol-water concentration with respect to sediment volume. At 25% and 50% of ethanol in water, the height of sediment is more than other concentrations. Experiments are conducted to see sedimentation behavior at low concentration $(0-5\%)$ and high concentration $(95-100\%)$ of ethanol, which are shown in Figs. 3 (a) and (b), respectively. In high concentrations of ethanol, height of sediment was observed to be small as compared to other concentrations. The reason may be that the viscosity and interactions with nanoparticles can change with proportions of ethanol-water mixtures.

Effect of Alumina Nanoparticle Diameter. Two different diameters of alumina nanoparticles (40 nm and 50 nm) are used in these experiments. It is observed that lower diameter i.e. 40 nm have shown slight better stability than 50 nm because nanosuspensions of alumina 40 nm have slightly less sediment height as compared to alumina 50 nm which is shown in Figs. 4 (a) and (b). In overall, the settling behavior of nanosuspensions is dispersed type sedimentation. The aggregation in nanofluids with the time elapsed is due to higher surface activity of nanoparticles.

Fig. 4 (a) Comparison of Alumina 40 nm and Alumina 50 nm particle concentration of 0.5%, 0.3% and 0.1% at 1% ethanol in water, (b) Comparison of Alumina 40 nm and 50 nm in 5% ethanol at 2 minutes and 1 hour.

Effect of Ultrasonic Agitation. It is observed that sonication has great influence on settling characteristics. The nanofluid samples are sonicated for 24 hours using ultrasonic agitator and then allowed for settling and measured the sediment volume. It has been observed that most of the samples show more stability with sonication than without sonication. It is also observed that most of the nanosuspensions of particle concentration of 0.1% and 0.3% have no sediment for first 15 minutes. After sonication, the sediment behavior of 25%, 50% and 75% ethanol-water mixtures turned to flocculated sedimentation behavior. The particles are tending to remain suspended for longer time after sonication. Figs. 5 (a) and (b) represents the comparison of with and without sonication in nanosuspensions in different proportions of ethanol-water mixtures.

Fig. 5 (a) Effects of with and without ultrasonic agitator (UA) on sedimentation behavior in 98% ethanol concentration at 4 hours and 24 hours, (b) Effect of ethanol concentration on sedimentation behavior with comparison of with and without sonication in nanosuspensions of 0.5 % of Alumina (40 nm).

Fig. 6 Comparison of 0.5% Alumina nanosuspensions in 50% ethanol using ultrasonic agitator (left) and without sonication (right).

Dispersed type sedimentation is observed in all samples except 25%, 50% and 75% ethanolwater mixtures. Ethanol-water mixtures have high viscosity than pure liquids and it peaks at 50% proportions, however, pure ethanol has less viscosity than water. Settling behavior of nanosuspensions with and without sonication are similar, however sedimentation height is lower in sonicated nanosuspensions (Fig. 5 (a)). Fig. 6 represents sedimentation behavior of 50% ethanol in water with and without sonication. It can be seen that the height of sediment is higher in sonicated sample and it can be attributed to dispersed type of sedimentation.

Conclusions

This experimental study presents the settling behavior of alumina nanoparticles in ethanol-water mixtures. Dispersed settling behavior has been observed in all samples without sonication. Alumina nanoparticles of 40 nm diameter exhibit slightly better stability than Alumina 50 nm. It is found that sonication can increase the stability of nanofluids. The decrease in sediment height is also observed after sonication. In 25, 50 and 75% ethanol-water proportions, the settling behavior turns to flocculated sedimentation when treated with sonication process. Suitable surfactants can be added to decrease the agglomeration and to increase the stability of nanofluids.

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