CHAPTER 3

PREPARATION AND CHARACTERIZATION OF NANOFLUIDS

3.1 Preparation of mono/hybrid nanofluids

Both mono and hybrid nanofluids have been prepared by using a two-step method. In this method, the nanoparticles mixed with the base fluid is first stirred for proper mixing and then sonicated by using an ultrasonic vibrator to reduce particle agglomeration. Fig. 3.1 depicts the preparation of hybrid nanofluids using a two-step method.

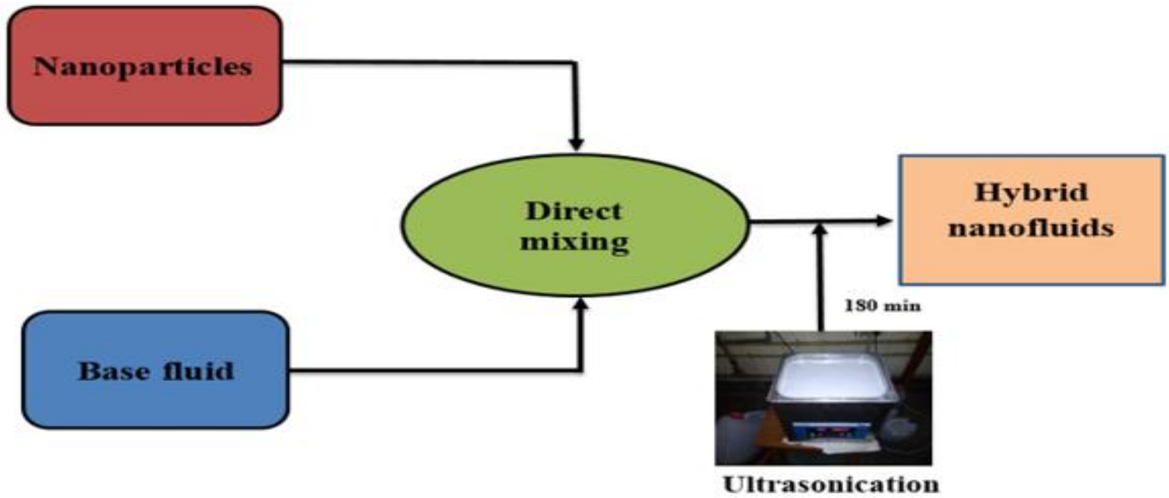


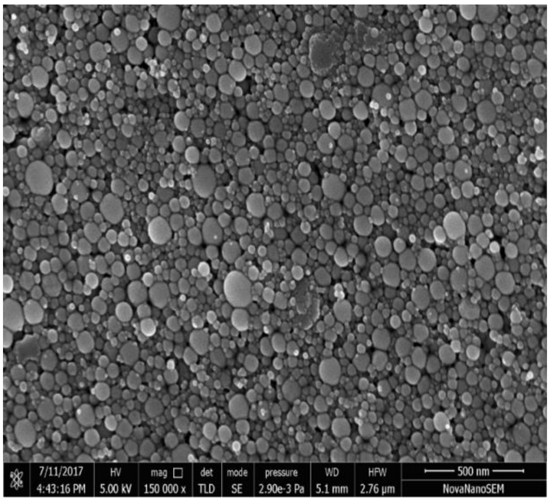
Figure 3.1: Preparation of mono/hybrid nanofluids

In the present study, the nanoparticles, i.e., Al_2O_3, CNT, TiO_2 and MgO have been chosen because of their low cost, easy availability and considerable thermal conductivity values. All the nanoparticles have been purchased from Alfa Aesar and Otto Chemika. Also, Capric acid has been used as a phase change material. The purpose of taking capric acid as PCM is its melting point (~32^{o}C), which is in between studied temperature range of cold fluid to take advantage of its latent heat. For the preparation, single or two different nanoparticles with a 50/50 volume ratio for 0.01% and 0.1 % total volume concentration were first dispersed in 10 liters of DI water and then it was sonicated for 3 hours using an ultrasonic bath of power 200W with 40 kHz. In the case of PCM dispersed nanofluid, PCM was added in the solid form and the temperature was maintained above its melting point temperature during the sonication to ensure that the PCM was homogeneously mixed in liquid state with base fluid. To avoid the particle agglomeration and maintain the stability of nanofluids, CTAB and SDS surfactants were used as per requirement. The volume fraction of nanoparticles in hybrid nanofluids were calculated by**,**

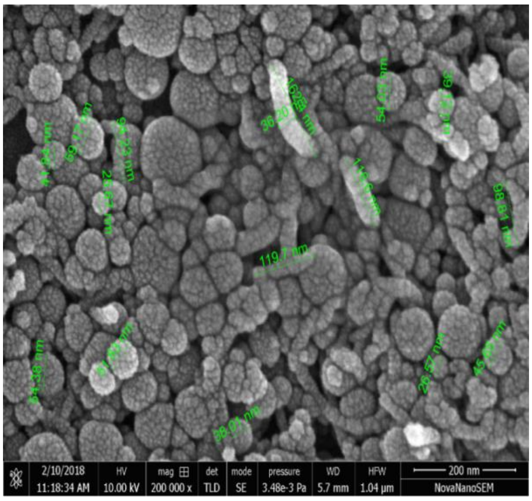
$$\varphi = \frac{\left(m/\rho\right)_{np1} + \left(m/\rho\right)_{np2}}{\left(m/\rho\right)_{np1} + \left(m/\rho\right)_{np2} + \left(m/\rho\right)_{bf}} \qquad (2.1)$$

3.2 Characterization of mono/hybrid nanofluids

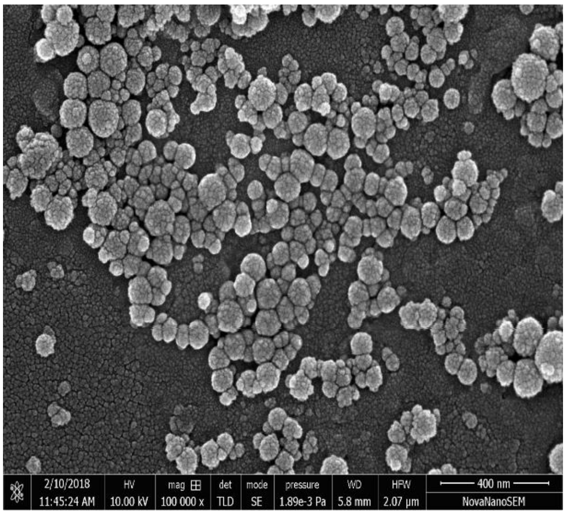
The characterization of nanofluids mainly consists of the morphology of the nanoparticles and analysis of the homogeneity and stability of the suspension. In this study, the scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images have been used to measure the morphology of the nanoparticles, as shown in Fig. 3.2. Fig. 3.2 (a) shows the SEM image of Al_2O_3 nanofluid and reveals that the Al_2O_3 nanoparticles are in a spherical shape with particle size ranging between 10-100 nm. The SEM image of Al_2O_3+CNT hybrid nanofluid is shown in Fig. 3.2(b) and exposes that CNT particles are in a cylindrical shape. Fig. 3.2 (c) shows the SEM image of Al_2O_3+TiO_2 hybrid nanofluid and reveals that the nanoparticles are spherical and of the average size of 40 nm with proper dispersion. The TEM image of Al_2O_3+MgO hybrid nanofluid is shown in Fig. 3.2(d) and found that the nanoparticles are in a spherical shape and of the average size of 50 nm with proper dispersion. In the case of PCM dispersed nanofluids, it has been characterized by measuring the homogeneity and stability of the fluids. The measurement of the thermophysical properties of different samples of the same PCM dispersed nanofluids has been performed to check the homogeneity of the fluids. The results found that all the values of different samples have no such variation. This ensured that PCM was properly dispersed in the base fluid. A similar homogeneity test has been performed for other proposed mono/hybrid nanofluids also.



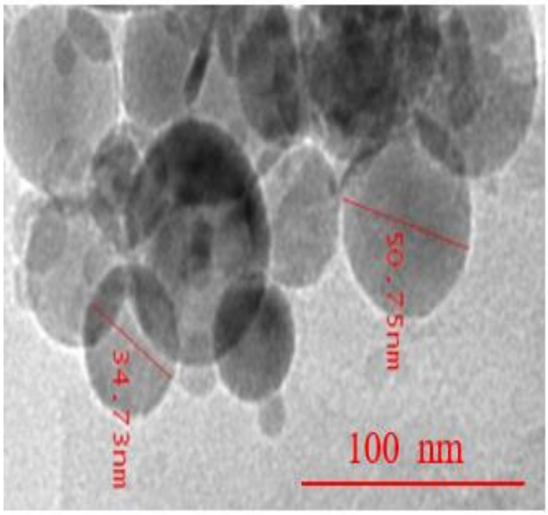
(a)



(b)



(c)



(d)

Figure 3.2: SEM image (a) Al_2O_3 nanofluid (b) Al_2O_3+CNT hybrid nanofluid (c) Al_2O_3+TiO_2 hybrid nanofluid and TEM image of (d) Al_2O_3+MgO

The stability of the hybrid nanofluids has been inspected by two methods, i.e, visual observation and its pH value. In this study, three samples, namely: Al_2O_3+CNT hybrid nanofluid of 0.01% vol. and Al_2O_3+TiO_2, Al_2O_3+MgO and Al_2O_3+PCM hybrid nanofluid of 0.1% vol. have been taken for visual observation, as shown in Fig. 3.3. The photographs have been taken only after preparation and after 10 and 20 days. The sedimentation was found after 10 days for Al_2O_3+CNT and Al_2O_3+PCM hybrid nanofluid and after 20 days for Al_2O_3+TiO_2 and Al_2O_3+MgO hybrid nanofluid as shown in Fig. 3.3 (a), (b) (c) and (d). Moreover, the pH value of synthesized hybrid nanofluid has been measured using a digital pH meter. Its value was found far away from the nanoparticle's isoelectric point (IEP) due to the large repulsion force between the particles **(Kumar and Sarkar, 2019)**. For example, the pH value of Al_2O_3+CNT of 0.01 vol% is 7.73 and the IEP of Al_2O_3 and CNT are 9.1 and 4.5, respectively, which is far away from its pH value **(Singh et al., 2005, Singh et al., 2012)**. Similarly, the pH value of Al_2O_3+MgO of 0.1 vol% is 9.09 and the IEP of MgO is 10.5 **(Wang et al., 2017)**. This ensured that the hybrid nanofluid had sufficient stability. Also, the thermo-physical properties have been measured before and after the experiment to check the homogeneity of hybrid nanofluid. No significant difference in measured properties has been found.

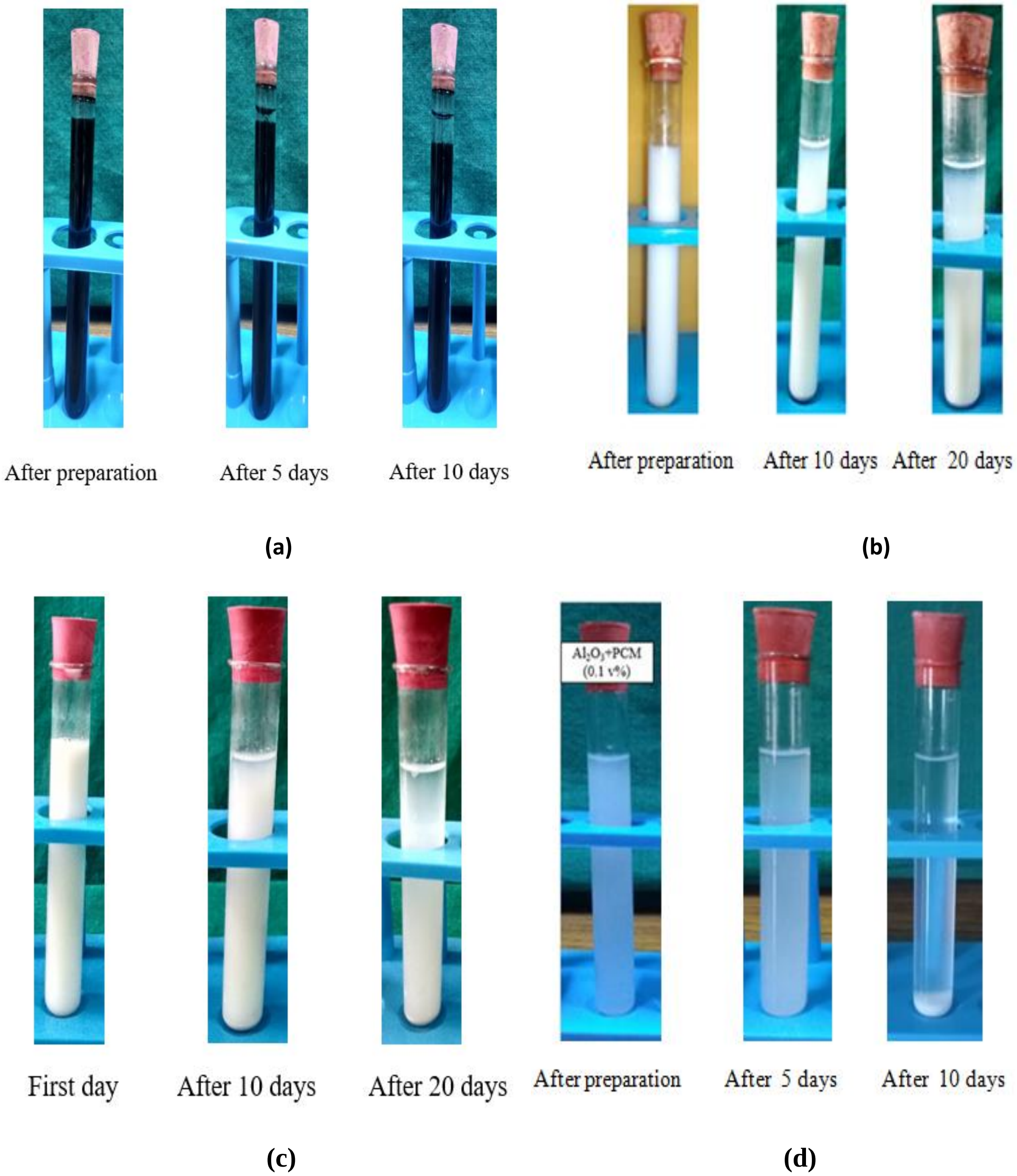


Figure 3.3: Sedimentation observation of (a) Al_2O_3+CNT (0.01 vol %.) (b) Al_2O_3+TiO_2 (0.1% vol.) (c) Al_2O_3+MgO (0.1% vol.) (d) Al_2O_3+PCM (0.1% vol.)

3.3 Thermophysical properties of mono/hybrid nanofluids

In the present study, all the thermophysical properties such as thermal conductivity, viscosity, density and specific heat have been measured using various instruments. Hot disk

TPS-500 analyzer has been used to measure the thermal conductivity and specific heat of the mono/hybrid nanofluids, as shown in Fig. 3.4 (a). Before collecting data, the instrument was calibrated and measurements were repeated three times. The thermal conductivity of nanofluids in the range of 0.2-2W/m.K with an accuracy of ± 1% can be measured. The dynamic viscosity of mono/hybrid nanofluids has been measured by LVDV-II+ Pro Brookfield digital viscometer, as shown in Fig. 3.4 (b). The experiments were performed at different shear rates (different RPM) from 76.8 s^{-1} to 384 s^{-1} for each temperature (30^{o}C-70^{o}C) to study the rheological behavior of the nanofluid. The results indicate that the hybrid nanofluid behaved like Newtonian fluid as the nanofluid has the same viscosity at different shear rates. Moreover, the open literature shows that the nanofluids with a concentration lower than 1% can be considered as Newtonian. Here, the concentration is ≤ 0.1% and hence the prepared mono/hybrid nanofluids have been considered as a Newtonian fluid. Also, the viscosity of the hybrid nanofluid decreases with a rise in temperature.

The density of mono/hybrid nanofluids has been estimated by measuring the mass of certain volume using a high precision digital weighing balance (model: ATX224, Shimadzu, Japan) as shown in Fig. 3.4 (c), followed by the relation, density = mass/volume. For measuring, the known volume of nanofluid has been taken in measuring beaker and weighted it in digital weighing balance. From obtained weight, the weight of the empty measuring beaker has been subtracted to get the weight of the nanofluid. The density of the nanofluid has been estimated by dividing this weight to the known volume of the nanofluid.

(a)

(b)

(c)

Figure 3.4: Photograph of (a) Hot disk TPS-500 analyser, (b) Brookfield DV1 digital viscometer and (c) Digital weighing balance

3.4 Results and discussion

The thermophysical properties of different nanoparticles and PCM are given in **Table 3.1.** Among all the nanoparticles, CNT exhibits higher thermal conductivity, so it may be useful for better heat transfer enhancement. Since it is costlier among all nanoparticles, so it has been taken in low quantity (0.01% vol.). PCM possesses the lowest thermal conductivity in comparison to all the studied nanoparticles. However, its latent heat may enhance the overall heat capacity of the fluid. In term of density, TiO_2 possesses the highest and PCM possesses the lowest density.

Table 3.1 Thermo-physical properties of nanoparticles and PCM at ambient temperature.

Types of nanoparticles	**Thermal conductivity (W/m.K)**	**Density (kg/m^3)**	**Specific heat (J/kg.K)**	**Melting point (^oC)**	**Latent heat (kJ/kg)**
Al_2O_3	40	3900	880	-	
TiO_2	11.7	4260	697	-	
MgO	48.4	3580	877		
CNT	3000	2600	740		
PCM	0.372 (solid) 0.151 (liquid)	1004 (solid) 853-886 (liquid)	2100 (solid) 2090 (liquid)	31.6	157.8

The thermophysical properties of different hybrid nanofluids of 0.01% volume concentration with a 50/50 ratio at ambient temperatures are listed in **Table 3.2.** From the table, it is observed that CNT/water nanofluid exhibits the highest thermal conductivity due to the higher thermal conductivity of CNT nanoparticle. The enhancement of thermal conductivity for CNT/water and PCM/water are observed around 1.15% and 0.09%, respectively, as compared to that of the base fluid. The density of Al_2O_3+CNT and Al_2O_3+PCM hybrid nanofluids are nearly the same as their volume concentration is very low. All the working fluids are considered as an incompressible fluid in this study. In the case of hybrid nanofluids, Al_2O_3+CNT possesses the highest value of thermal conductivity, followed by Al_2O_3+PCM. Density and viscosity are found maximum for Al_2O_3/water and minimum for PCM/water.

Table 3.2 Thermo-physical properties of the base fluid and different mono/hybrid nanofluid (0.01 vol.%) in equal proportion at ambient temperature (30^{o}C).

Working fluids	**Thermal conductivity (W/m.K)**	**Density (kg/m^3)**	**Specific heat (J/kg.K)**	**Viscosity (Pa.s)**
DI Water	0.6065	995.7	4183	0.000768
Al_2O_3/water	0.6069	996.0	4182	0.000781
CNT/water	0.6135	995.8	4182	0.000776
PCM/water	0.6071	995.7	4183	0.000764
Al_2O_3+CNT/water	0.6089	995.9	4181	0.000813
Al_2O_3+PCM/water	0.6083	995.8	4182	0.000788

Table 3.3 shows the thermophysical properties of different hybrid nanofluids of 0.1% volume concentration with a 50/50 ratio at ambient temperature. From the table, it is found that the thermal conductivity of mono/hybrid nanofluids is greater than that of the DI

water. Also, in the case of PCM dispersed nanofluids, while increasing the volume concentration, the thermal conductivity increases and specific heat decreases. Among all hybrid nanofluids, Al_2O_3+MgO possesses the highest and Al_2O_3+PCM possesses the lowest thermal conductivity. The enhancements of thermal conductivity for Al_2O_3+MgO, Al_2O_3+TiO_2 and Al_2O_3+PCM are observed around 1.23%, 0.72% and 0.47%, respectively, as compared to that of the base fluid. The density and viscosity of mono/hybrid nanofluids are greater than that of the base fluid. Al_2O_3+MgO hybrid nanofluid possesses the highest and PCM/water possesses the lowest viscosity. On the other hand, by adding the nanoparticles in the DI water, the specific heat of the mono/hybrid nanofluids is lower than that of the DI water. The specific heat is found maximum for Al_2O_3+PCM and minimum for Al_2O_3+TiO_2. In the case of mono nanofluids, Al_2O_3/water shows a higher value of thermal conductivity, density and viscosity than that of the PCM/water nanofluid. However, PCM/water possesses the highest value of specific heat.

Table 3.3 Thermo-physical properties of different mono/hybrid nanofluid (0.1 vol.%) in equal proportion at ambient temperature.

Working fluids	**Thermal conductivity (W/m.K)**	**Density (kg/m^3)**	**Specific heat (J/kg.K)**	**Viscosity (Pa.s)**
DI Water	0.6065	995.6	4183	0.000768
Al_2O_3/water	0.6120	997.6	4170	0.000788
PCM/water	0.6088	995.7	4181	0.000769
Al_2O_3+TiO_2	0.6109	997.9	4169	0.000799
Al_2O_3+MgO	0.6140	997.3	4170	0.0008187
Al_2O_3+PCM	0.6094	996.2	4175	0.000796

3.5 Highlights

In this study, all the considered mono/hybrid nanofluids have been prepared by using a two-step method and characterized. With the help of SEM and TEM images, the morphology of the nanoparticles has been examined. A property-based homogeneity test has been performed for all prepared mono/hybrid nanofluids. The visual observation and pH value determination have been performed to inspect the stability of the mono/hybrid nanofluids. The thermophysical properties of hybrid nanofluids for different volume concentrations have been measured and tabulated. The main conclusions are as follows:

- From the SEM and TEM images, all the nanoparticles are in a spherical shape with the particle size ranging from 10-100 nm except CNT nanoparticles, which is found cylindrical in shape.
- Since the sedimentation of hybrid nanofluid was found a minimum of 10 days, which was enough time interval for conducting the various experiments.
- All the nanoparticles were properly dispersed in the base fluids, as confirmed by the homogeneity test.
- At 0.01% volume concentration, the Al_2O_3+CNT (in case of hybrid nanofluid) exhibits higher thermal conductivity and viscosity, which implies that dissimilar shaped particle combination exhibits higher flow resistance than similar shaped particle in the fluid.
- At 0.1% volume concentration, Al_2O_3+MgO/water nanofluid possesses higher thermal conductivity and viscosity, while PCM/water nanofluid possesses lower thermal conductivity and viscosity.