CHAPTER 3

NANOFLUID PREPARATION AND CHARACTERIZATION

Preparation of nanofluid is an important step for changing heat transfer performance of conventional base fluids. Nanofluid refers to a new class of heat transfer fluids prepared by dispersing nanoparticles into conventional liquids and should have stable suspension, minimal agglomeration of particles and no chemical change of the fluid. So, different methods are used for preparation of effective suspensions such as changing the pH value, using surfactants and using ultrasonic vibrations [11].

3.1 Preparation of Nanofluids

A nanofluid is by definition a stable suspension of nanoparticles. Nanofluids are colloidal suspensions of nanoparticles in water and exhibit good thermal properties at modest concentrations preferably less than 0.1% volume concentration. Many solid nanoparticles with relatively large thermal conductivity can be used as additives of nanofluids. Some common nanoparticles used are, metallic particles (Cu, Al, Fe, Au, and Ag), metal compounds (Al_2O_3, CuO, TiO_2, Fe_3O_4 and SiC) and carbon nanotubes. The liquids generally used are water, oil, acetone and ethylene glycol. As the nanofluid contains solid particles of high density and liquid phase of low density, there is always a probability for the nanoparticles to settle down. The time could be shorter if the particle material and the base fluid react with each other to make compounds and hence bring out chemical change to the solution.

For the present experimental study, Al_2O_3-water, TiO_2-water and SiO_2-water nanofluids of three different concentrations, 0.001%, 0.005% and 0.01% by volume were prepared. Concentrated water based dispersion of alumina, titanium and silica

nanoparticles were acquired from Alfa Aesar, a Johnson Matthey company. The specified weight concentration was 23% for Al_2O_3, 27.5% forTiO_2 and 31% for SiO_2. An Ultrasonic Bath (MJL Laboratory Instruments & Equipment, Fig. 3.1) was used for 4 h of sonication of nanofluids after diluting with distilled water up to the required concentration. The specifications of the ultrasonicator have been presented in Table 3.1.

Fig. 3.1 Ultrasonicator

Table 3.1 Specification of ultrasonicator

Frequency	40 kHz
Power	50 W
Maximum Processing Volume	2.5 ltr
Power supply	AC 220 V 50 /60 Hz
Heating	0-80 ^oC
Dimensions (mm)	175x165x250 mm
Digital timer	5-60 minutes
Material	Stainless steel

Table 3.2 Nanofluid specifications

Nanoparticle Material	As purchased concentration (wt. %)	Transparency
Al_2O_3	23	Opaque
TiO_2	27.5	Opaque
SiO_2	31	Transparent

As can be seen in Table 3.2, nanofluid specification has been provided in weight percentage (wt. %). This was converted into volume percentage (vol. %) for the present study. A formula has been derived for the amount of distilled water to be added for the required concentration of nanofluid (vol. %). For making a nanofluid of φ vol. % concentration from x ml of γ wt. % nanofluid, the amount of distilled water to be added has to be determined. Supposing that x ml of γ wt. % nanofluid contains y ml of nanopowder of density ρ_P and z ml of distilled water of density ρ_f, the required amount of distilled water to be added, D ml, for making φ vol.% nanofluid can be calculated as follows:

$$x = y + z \tag{3.1}$$

$$\gamma\ wt\% = \frac{y\rho_P}{y\rho_P + z\rho_f} \cdot 100 \tag{3.2}$$

$$\varphi\ vol.\% = \frac{y}{y + z + D} \cdot 100 \tag{3.3}$$

As γ and φ are known, assuming a value for x, D can be calculated using above 3 equations. In order to prepare 0.01 vol. % Al_2O_3-water nf, 1ml of the purchased nanofluid requires 699 ml of distilled water.

3.2 Nanofluid Characterization

3.2.1 X-ray Diffraction Technique (XRD)

X-ray Diffraction (XRD) is one of the classical methods for identification and characterization of crystalline solids. Each crystalline solid has its unique characteristic X-ray powder pattern which is used as a "fingerprint" for its identification. The method is based on the diffraction of X-rays by the sample indifferent directions. Waves of wavelength comparable to the crystal lattice spacing are strongly scattered (diffracted). The purchased nanofluids were diluted with distilled water followed by centrifugation at 4000 rpm for 90 min. The settled nanoparticles were then washed with absolute ethanol and acetone. Further these were vacuum dried at 80°C for 2 hour in the oven. The obtained nanopowder was characterized by (X Ray Diffraction) XRD with a Rigaku X-ray Diffractometer and Cu-kα1 radiation in the range of 20 to 80^o. The X-ray diffraction test was carried out with a scan speed of 3^o/minute. The average grain size was estimated by using Debye-Scherrer formula. The full width at half maximum (FWHM) was taken from the XRD patterns (Fig. 3.2)

$$d = \frac{0.9\lambda}{(FWHM)Cos\beta} \tag{3.4}$$

where d, λ, and β are the average particle grain size, wavelength of the Cu-kα1 X rays (1.5418A^o) and Braggs angle respectively.

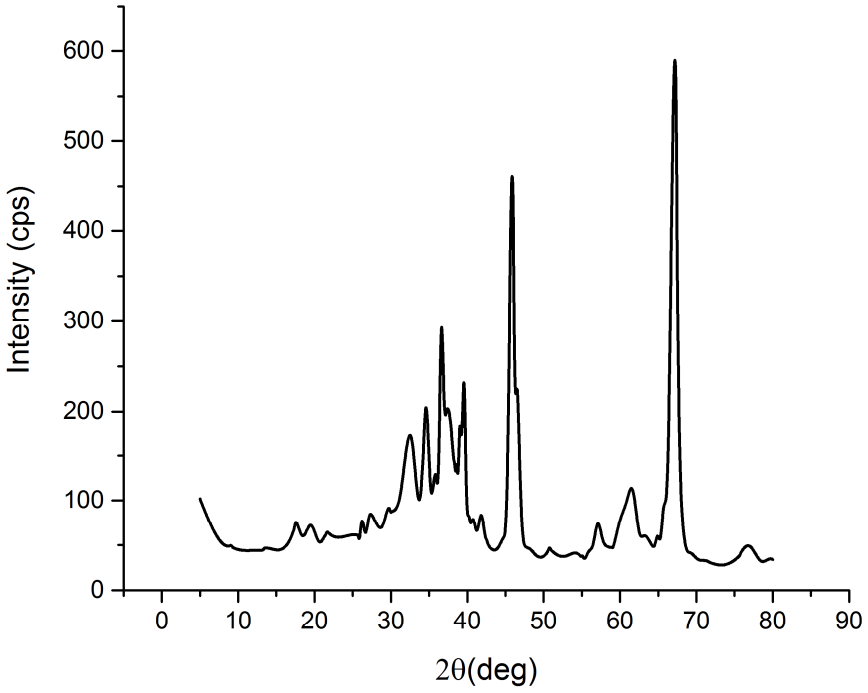

Fig. 3.2a XRD pattern of Alumina nanoparticles

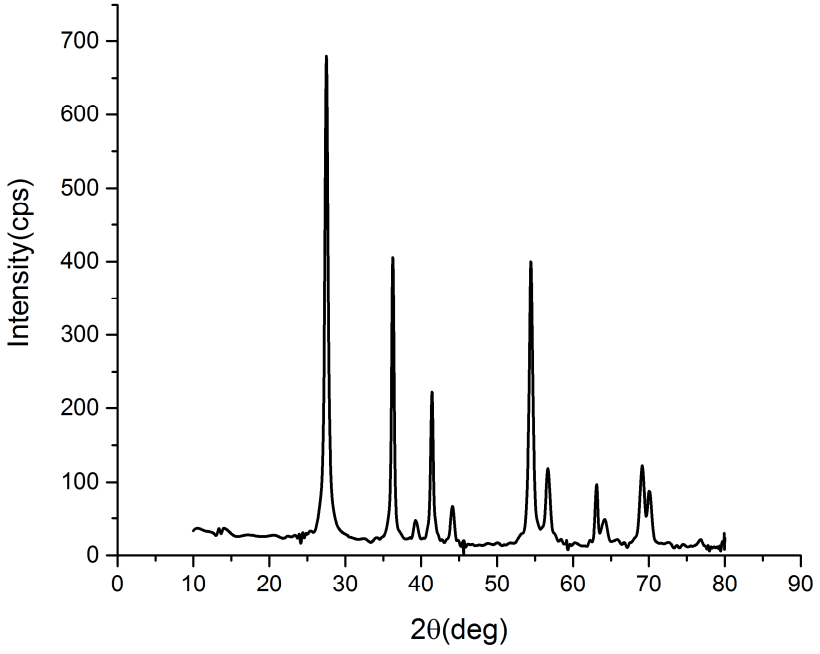

Fig. 3.2b XRD pattern of Titanium nanoparticles

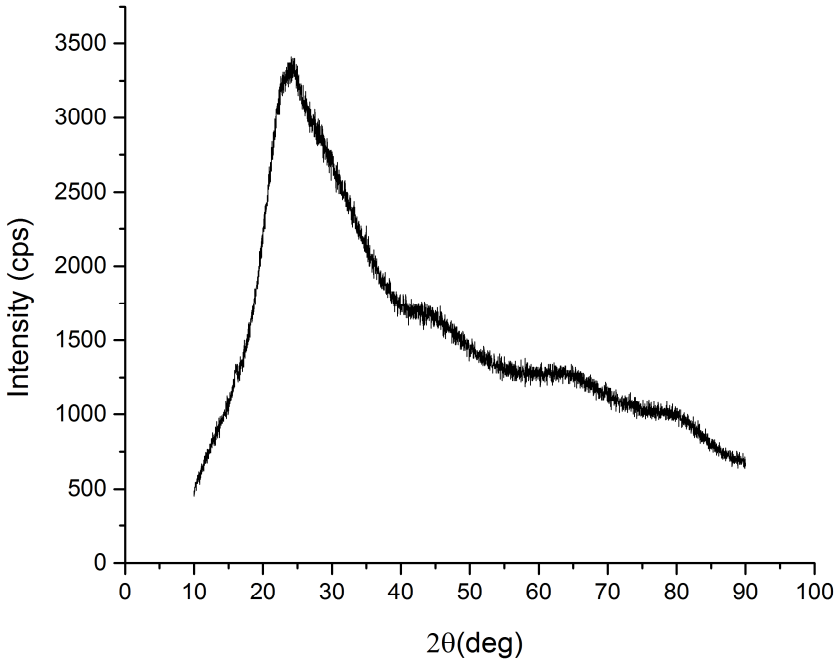

Fig. 3.2c XRD pattern of silica nanoparticles

The average grain size of alumina nanoparticle was found to be between 20 and25 nm, titanium oxide nanoparticle was found to be between 30 and38 nm and silica nanoparticle was found to be between 8nm and 13nm by using the Scherrer formula.

3.2.2 Scanning Electron Microscopy (SEM)

This electron microscopy based technique determines the size, shape and surface morphology with direct visualization of the nanoparticles. Therefore scanning electron microscopy offer several advantages in morphological and sizing analysis. In SEM, high spatial resolution microanalysis of materials is possible. The nanoparticles size was verified by the scanning electron microscope. The size of the nanoparticles was fairly close to the value obtained from XRD pattern. The SEM images (Fig. 3.3) demonstrate that the particles were in aggregates and somewhat spherical in shape.

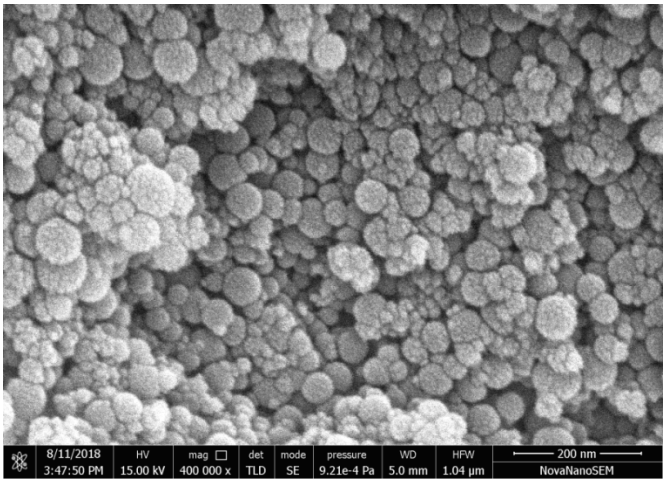

Fig. 3.3a HRSEM of Al_2O_3 nanoparticle

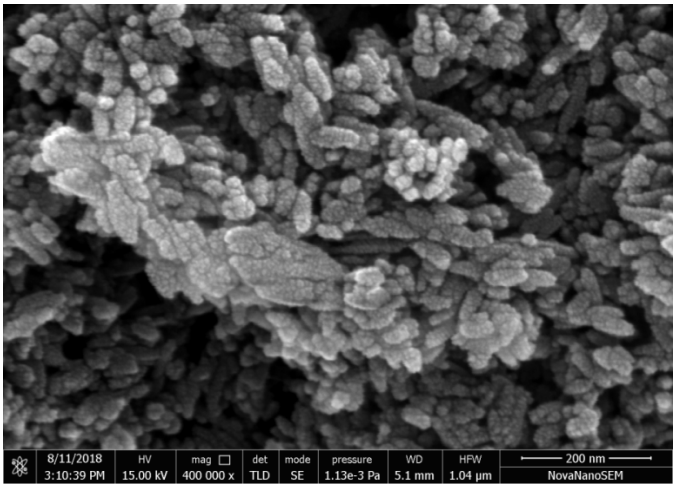

Fig. 3.3b HRSEM of TiO_2 nanoparticle

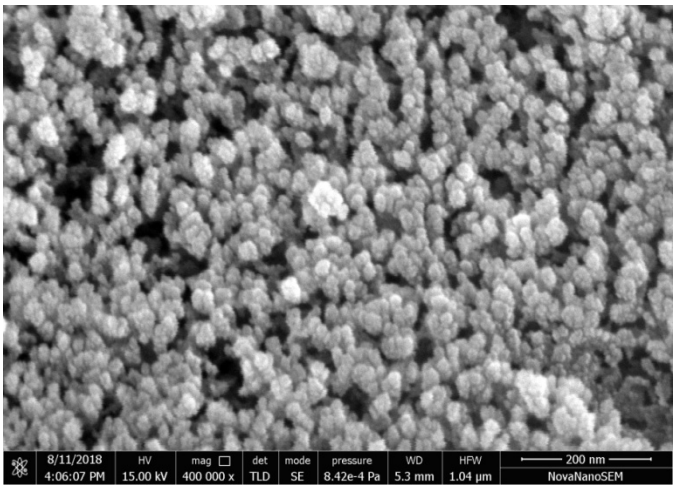

Fig. 3.3c HRSEM of SiO_2 nanoparticle

3.2.3 Energy Dispersive Spectroscopy (EDS)

Energy dispersive spectroscopy is a technique to analyze near surface elements and estimate their proportion at different positions. This technique is used in conjunction with SEM. In order to analyze the chemical composition of the surface of a mixture, EDS was employed. The EDS spectroscopy showed the presence of aluminum and oxygen in the synthesized aluminum oxide nanofluids by the distinct peaks as observed in the EDS spectrum shown in Fig. 3.4a. Similarly, the presence of titanium and oxygen is observed in Fig. 3.4b and the presence of silicone and oxygen is observed in Fig. 3.4c.

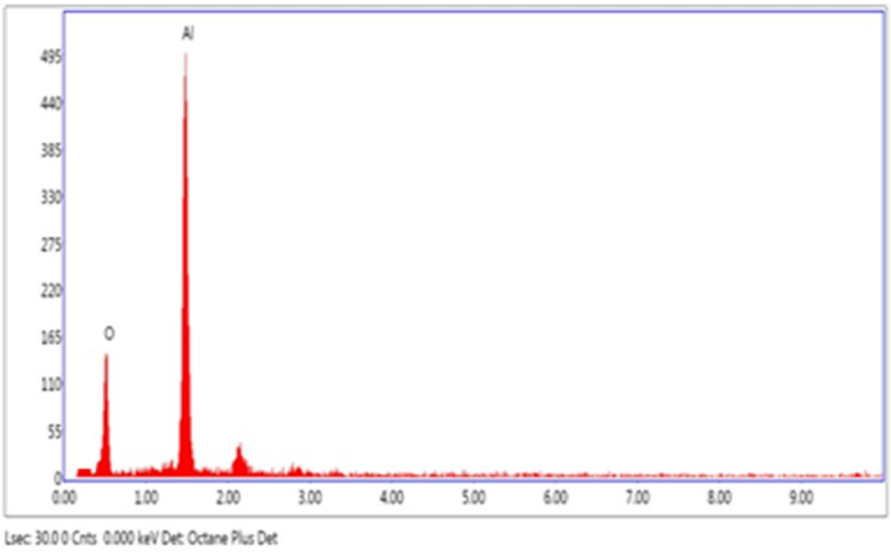

Fig. 3.4a EDS of Al_2O_3 nanoparticle

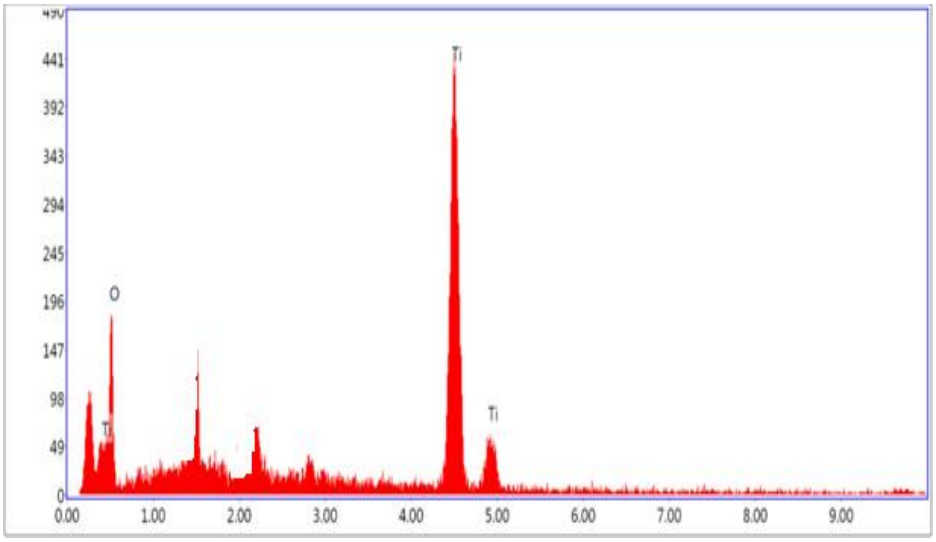

Fig. 3.4b EDS of TiO_2 nanoparticle

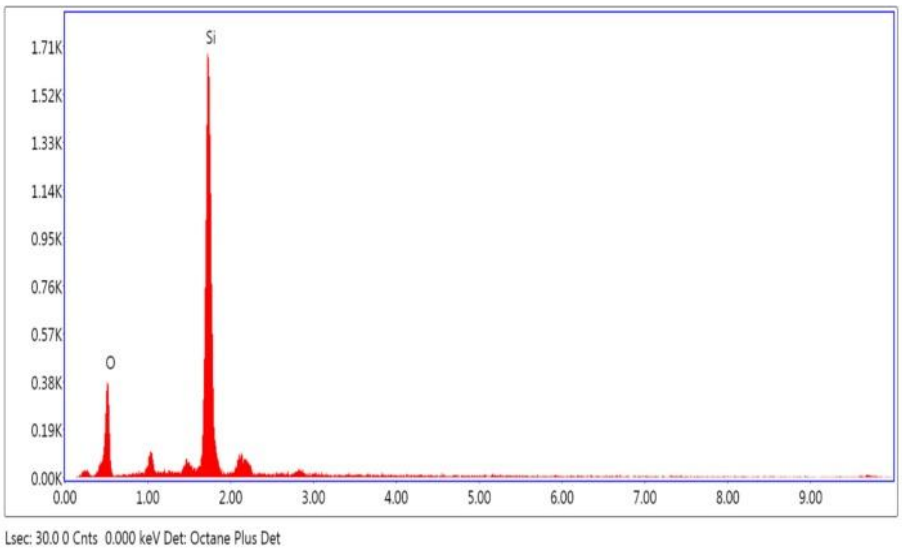

Fig. 3.4c EDS of SiO_2 nanoparticle

3.2.4 Stability of Nanofluid

Nanoparticles have a tendency to aggregate with time for its high surface-activity. The agglomeration of nanoparticles results not only clogging of channels but also decreasing of thermal conductivity of nanofluids. So the investigation on stability is a fundamental issue that influenced the properties of nanofluids. In general, there are three

effective methods used to attain stability of suspension against sedimentation of nanoparticles and are as follows:

- Control of pH value of the suspension
- Addition of surfactants
- Use of ultrasonic vibration

All of these techniques aim at changing the surface properties of suspended nanoparticles and suppressing formation of the cluster of particles in order to obtain stable suspensions [126]. The addition of surfactants was already done by the manufacturer. The formulated nanofluids after ultrasonication exhibited good colloidal stability without any visual sedimentation for almost seven days, which was sufficiently long for the experiments. To avoid particle agglomerations, nanofluids were homogenized before each experiment [42]. An important parameter for the colloidal stability of oxide nanoparticles is pH, which determines the electrostatic charge on the particles surface. Values of the pH for our dilute alumina, titanium and silica nanofluids were measured to be 4–5, 3–5 and 7–10, respectively, which are far from the IEP (Iso Electric Point) of alumina (~9), titanium (~8) and silica (~3). All nanofluids used in our experiments were found to be colloidally stable (i.e., didnot sediment) at the reported nanoparticle concentrations in the reported pH ranges, with no surfactant addition.

3.2.5 Thermophysical properties of Nanofluids

For flow boiling experiments, it is important to know the properties of the fluids that can have effects on heat transfer. These properties include density, viscosity, thermal conductivity and specific heat.

3.2.5.1 Density

Density of nanofluid is very important because any change in thermo-physical properties can affect the heat transfer of the nanofluid boiling. In fact, change of density of nanofluid is negligible compared to water because of low concentration of nanoparticles. The nanofluid density can be calculated by using

$$\rho_{nf} = \rho_P \varphi + \rho_f (1 - \varphi) \quad (3.5)$$

Where, φ is the nanoparticles concentration, ρ_f and ρ_P are the density of the base fluidand nanoparticles respectively.

3.2.5.2 Viscosity

The rheological attributes of DI water and nanofluids were measured using a Brookfield DVI rotational viscometer (shown in Fig.3.5) with a temperature-controlled bath. The viscometer allows changes in rotational speed such that required torque can be attained for varying viscosities. Generally, low viscosity nanofluids require spindle with larger surface area and high rotational speed. The minimum amount of nanofluid required for viscosity measurements is 1 ml. As the spindle is rotated, the viscous drag of the fluid against the spindle is measured by the deflection of the calibrated spring. The spindle type and speed combination will produce satisfactory results when the applied torque is above 50% of the maximum possible torque. Before any set of measurement, the viscometer was calibrated using Brookfield viscosity standard fluid and DI water, respectively. Each measurement was repeated ten times to compute the mean value of this data. The uncertainty of these measurements was found to be within ±1.0%. The measured values of dynamic viscosity are listed in Table 3.3.

Fig. 3.5 Brookfield Viscometer (CPE-42)

Rheological properties play a very important role in fluid flow. As the nanofluids are likely to flow either by forced or natural convection, the flow properties such as viscosity are therefore essential in the study of suspensions containing nanosized particles**.**

The published rheological studies of nanofluids are limited and Newtonian behaviour of nanofluids has not been completely clarified. However, some researchers have reported Newtonian behaviour of nanofluids [122, 127-133]. Alphonse et al. [134] studied on viscosity of TiO_2/water nanofluid and observed Newtonian behaviour. Chandrasekar et al. [135] observed Newtonian behaviour in Al_2O_3/water nanofluid. Longo et al. [136] observed Newtonian behaviour in oxide based nanofluids (1- 4% particle volume fraction for Al_2O_3/water nanofluid and 1- 6% particle volume fraction for TiO_2/water nanofluid) for all the investigated ranges of temperature. Fedele et al. [137] conducted an experimental study and found that TiO_2/water nanofluid have Newtonian behaviour. Namburu et al. [138] demonstrated that SiO_2/water nanofluid (0- 10% particle volume fraction) displayed Newtonian behaviour. Hence, from the above literature, the Newtonian behavior of the nanofluids (Because of low concentration i.e. 0.001%, 0.005%

and 0.01% v/v, viscosity values are nearly similar to that of water.) in the present work have been verified.

3.2.5.3 Thermal Conductivity

The thermal conductivity of the suspensions were measured using a Hot Disk TPS500 device (Fig. 3.6) based on the transient plane source method. The transient plane source (TPS) technique is a rapid and precise method for studying thermal transport properties. The TPS technique is based on the use of a transiently heated plane sensor consisting of an electrically conductive pure nickel pattern in the shape of a double spiral. The spiral is sandwiched between two thin insulating sheets of Kapton. The temperature coefficient of resistivity (TCR) of the nickel is such that the temperature increase of the element can be precisely deduced from its resistance. The voltage change over the TPS sensor element is recorded simultaneously with the resistance (temperature) increase as a function of time.

To perform the thermal transport measurements, the Hot Disk sensor was fitted between two pieces of sample: each one with a plane surface facing the sensor. By passing electrical current high enough to raise the temperature of the sensor between fractions of degrees up to several degrees, thermal conductivity was determined. Thermal properties are calculated by recording the temperature increase as a function of time. The Hot Disk sensor is used both as a heat source and as a dynamic temperature sensor. The solution of the thermal conductivity equation assumed that the Hot Disk sensor is located in an infinite medium, which means that the transient recording must be interrupted as soon as any influence from the outside boundaries of the two sample pieces is recorded. To reduce the measurement errors, each experiment was repeated at least ten times to

compute the mean value of this data. The uncertainty of these measurements was found to be within ±1.50%. The measured values of thermal conductivity are listed in Table 3.3.

Fig. 3.6. Thermal conductivity measurement apparatus (Hot Disk TPS 500)

Table 3.3 Thermal Conductivity and viscosity values at 30°C

Fluid used	Volume concentration	Dynamic viscosity (cP)	Thermal Conductivity(W/mK)
Water	-	0.70	0.631
Al_2O_3-water	0.001%	0.74	0.662
	0.005%	0.77	0.670
	0.01%	0.82	0.688
TiO_2-water	0.001%	1.05	0.657
	0.005%	1.15	0.664
	0.01%	1.21	0.672
SiO_2-water	0.001%	0.79	0.629
	0.005%	0.82	0.633
	0.01%	0.88	0.641

3.2.5.4 Specific heat

The specific heat capacity is one of the important thermal properties that will affect the thermal performances of the nanofluid. It can be calculated using the same theory of mixtures used to determine the density. This theory of mixture yields:

$$C_{nf} = \left[\rho_p C_p \varphi + \rho_f C_f (1-\varphi)\right] / \left[\rho_p \varphi + \rho_f (1-\varphi)\right] \qquad (3.6)$$

Where C_p and C_f are the specific heat of the nanoparticle material and base fluid, respectively. Because of low concentration of nanoparticles used in this work, the thermo-physical properties of the working fluid are not meaningfully different from distilled water.

3.2.6 Summary

The experimental task associated with this chapter was the preparation and characterization of nanofluids. It was observed that the nanofluids prepared at different concentrations remained stable for several days. Finally, the thermo-physical properties of nanofluids were characterized, showing very few differences compared to the base fluid. This was expected in regards with the low load of particles in the prepared nanofluids.