CHAPTER 3

NANOFLUID PREPARATION AND CHARACTERIZATION

3.1 INTRODUCTION

The study of nanofluids begins from the preparation and characterization. While conducting the experiments to find the unknown parameters of nanofluid, it should be well-characterized. From the fundamental point of view, it is also important to understand the behavior of nanofluids in order to realize their benefits in any system. This chapter deals with an overview of the preparation and characterization methods used in this study.

3.2 PREPARATION OF NANOFLUID

Graphene nanoparticles purchased from SkySpring Nanomaterials, Inc., USA is used for preparing the nanofluid by two step method. De-Ionized water is used as base fluid. Surfactant, Sodium dodecyl Benzene Sulfonate (SDBS) of about 50% of the mass of Graphene nanoparticle is added to keep the nanoparticles stable in the base fluid. Nanofluids with 0.2%, 0.4% and 0.6% volume of graphene nanoparticles are prepared. The sonicator used for dispersing graphene nanoparticle and SDBS is shown in Figure 3.1. The dispersant (SDBS) was first added to the DI water and sonicated for about 15min. Sonication is continued when nanoparticles were added at a very slow rate. After the desired amount of particles, the mixture is sonicated for 45
minutes. The nanofluid prepared by this method is found to be stable without sedimentation.

3.3 CHARACTERISATION OF NANOFLUID

Characterisation of the sample is done by scanning electron microscope (SEM), X-Ray diffraction (XRD) and digital light scattering (DLS).

3.3.1 Morphology of graphene

The morphology of graphene nanoparticles was studied by scanning electron microscope (SEM) shown in figure 3.2 (SEM, JSM 6390, JEOL, USA). A SEM is a type of electron microscope that produces images of a sample by scanning the surface with a focused electron beam. The electrons interact with atoms in the sample, producing different signals that contain information on the topography and composition of the sample surface. The electron beam is scanned into a raster scan pattern, and the beam position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer.
Figure 3.2 Scanning Electron Microscope used for Characterization

The graphene nanoparticles at 10000 X Magnification taken by SEM is shown in Figure 3.3. The graphene are randomly distributed as a flake-like structure with less than 100 nm in thickness is clearly observed from Figure 3.3.

Figure 3.3 SEM image of graphene nanopowder
3.3.2 X-ray powder diffractometer

The shape, size and phase distribution of graphene nanoparticles are characterized by x-ray powder diffractometer (XRD 6000, Shimadzu, Japan). The XRD Spectra of the Graphene nanoparticle is shown in Figure 3.4. The Crystal Size, D is calculated from Scherrer’s formula (Timofeeva et al. 2007) and mention in equation 3.1. The average size of the graphene nanoparticle was 18 nm.

\[ D_{vol} = \frac{k\lambda}{\beta\cos\theta} \]  

(3.1)

Where, \( D_{vol} \) denotes volume-weighted crystallite size of the particles, \( \lambda \) is the wavelength of the x-ray source, \( k \) is sherrer constant, \( \beta \) is the value of the full width at half maximum (radian) and \( \theta \) is the half of the diffraction angle.

Figure 3.4 XRD spectra of graphene nanopowder
3.3.3 Particle size analysis

The particle size analyser (Nano ZS90 ZETASIZER Nanoseries, Malvern, USA) is used to analyse the particle size distribution of the nanoparticles in the nanofluid. The particle size analysis determines the range of particle sizes and their relative presence within the nanofluid sample. It works on the principle of dynamic light scattering method and it can measure the particle size from 0.3nm-5μm. The particle size distribution of the graphene-water nanofluid at 0.2% volume concentration is shown in Figure 3.5

![Size Distribution by Intensity](image)

**Figure 3.5 Particle size distribution of graphene-water nanofluids**

3.3.4 Visual observation study of Stability

The visual observation study is conducted in the prepared graphene-water nanofluid at 0.2% concentration to check the stability of nanoparticles. Surfactants are also used in nanofluids to enhance the stability of nanofluids. The prepared Nanofluid at 24, 48 and 72hrs are is shown in Figure 3.6. The first test tube has pure distilled water and second one has graphene nanofluid.
Similarly the visual observation study has been done for graphene water nanofluid and graphene ethylene glycol nanofluid. The images of graphene water nanofluid at 0.4%, and 0.6% concentration after 72 hrs are shown in Figure 3.7.