SYNTHESIS AND CHARACTERIZATION OF MULTI-WALLED CARBON NANOTUBE-NANOFUIDS

a thesis submitted by

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in partial fulfillment for the award of the degree of

DOCTOR OF PHILOSOPHY

under the supervision of

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DECLARATION

I, M. PREMALATHA hereby declare that the thesis, entitled “Synthesis and Characterization of Multi-Walled Carbon Nanotube-Nanofluids”, submitted to the Karunya University, in partial fulfillment of the requirements for the award of the Degree of Doctor of Philosophy in Physics is a record of original and independent research work done by me during the period 2012–2015, under the Supervision and guidance of Dr. A. Kingson Solomon Jeevaraj, Assistant Professor, Department of Physics, Arignar Anna Govt. Arts College, Namakkal. The work contained in this thesis has not been previously submitted to meet the requirements for a degree or diploma at this or any other higher education institution.

M. PREMALATHA
BONAFIDE CERTIFICATE

Certified that this Thesis titled “Synthesis and Characterization of Multi-Walled Carbon Nanotube-Nanofluids” is the bonafide work of M. PREMALATHA who carried out the research under my supervision. Certified further, that to the best of my knowledge the work reported herein does not form part of any other thesis or dissertation on the basis of which a degree or award was conferred on an earlier occasion on this or any other scholar.

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ABSTRACT

In many industrial applications, fluids are generally used as a cooling medium and the enhancement of heat transfer behavior of these fluids is of great importance in many potential sectors. Thermal fluids, such as water or industrial oil, are fluids that transport heat between different units of an industrial plant. Thermal properties of fluids play a decisive role in heating as well as cooling applications in industrial processes. Thermal conductivity of a liquid is an important physical property that decides its heat transfer performance. A new dimensional thermo fluid term is known as nanofluid has emerged after the pioneering work by Choi (1995). Conventional heat transfer fluids have inherently poor thermal conductivity which makes them inadequate for ultra high cooling applications. To increase the thermal conductivity of thermal fluids, particulate solids like metal and metal oxides with 1-3 order of magnitude larger thermal conductivity than base fluids may be utilized.

In this work, novel nanofluid systems were prepared using pure MWCNTs, -OH and -COOH functionalized MWCNTs as dispersants with silicone oil and Dowtherm A as base fluids. For the preparation of MWCNTs nanofluids, two-step method combined with ultrasonication technique has been used. Using ultrasonic and dielectric studies, molecular interactions between MWCNTs-
MWCNTs or MWCNTs-base fluids have been determined. To understand the thermal characteristics of nanofluids, heat transfer studies, like viscosity and thermal conductivity studies have also been studied.

This thesis comprises of six chapters. Chapter 1 gives a brief introduction to nanofluids, review of earlier research works done on the ultrasonic studies, dielectric studies, viscosity studies, thermal conductivity studies of MWCNTs nanofluids and their heat transfer applications, and also detailed objective of the research work. Chapter 2 demonstrates the characterization techniques used for MWCNTs and the characterization results of commercially purchased MWCNTs. X-ray diffraction (XRD) pattern illustrates well-defined peaks of pure MWCNTs, OH functionalized MWCNT and COOH functionalized MWCNTs and the diffraction peaks values corresponds to the hexagonal graphite structure and confirmed with the journal reference values. Surface morphologies of pure MWCNTs, -OH functionalized MWCNT and -COOH functionalized MWCNTs were analyzed using Transmission electron microscopy (TEM). From transmission electron microscopic (TEM) analysis, it is observed that the MWCNTs exhibit cylindrical (or rod) like structure and the average nanotube diameters of pure MWCNTs, -OH functionalized MWCNT and -COOH functionalized MWCNTs are found to be in nanometer and lengths in micrometer. The elemental compositions of the MWCNTs are analyzed using an energy dispersive X-ray (EDAX) analysis spectroscopy. From the FT-IR spectra of MWCNTs, type of functional groups (-OH and -
COOH) attached on the surface of the MWCNTs have been identified. From the FT-Raman spectra of MWCNTs, a well graphitized structure was confirmed from the G-band peak. Both weight changes (thermogravimetry) and heat flow (Differential Scanning Calorimetry) in MWCNTs as a function of temperature or time in a controlled atmosphere were obtained from the simultaneous TGA-DSC measurements. From the TGA/DSC measurements, the quantitative determination of the -OH and -COOH groups at the surface of MWCNTs were identified. Chapter 3 describes the preparation of MWCNT nanofluids. MWCNTs nanofluids of different nanotubes concentrations were prepared by two-step method. In this method, commercially purchased nanotubes were dispersed in base fluids (Silicone oil and Dowtherm-A) with the help of ultrasonication technique. To avoid the agglomeration of nanotubes and to produce well-dispersed stable suspension ultrasonication method was used. Totally thirty two MWCNTs nanofluid systems with different nanotube concentrations (0.001 g, 0.002 g, 0.003 g, 0.004 g and 0.005 g) in two different base fluids (Silicone oil and Dowtherm-A) were prepared for the analysis. Chapter 4 describes the experimental techniques for MWCNTs nanofluids characterization and the experimental results of MWCNTs nanofluids using ultrasonic, refractive index and dielectric studies. Ultrasonic and dielectric studies were carried out to determine the type of molecular interactions (nanotube-nanotube and nanotube-fluid) present in the MWCNTs nanofluids. Refractive index values of the MWCNTs nanofluids samples were
measured to study the optical properties of MWCNTs nanofluids. Chapter 5 describes the heat transfer analysis of MWCNTs nanofluids using viscosity and thermal conductivity studies. Viscosity and thermal conductivity of MWCNTs nanofluids were studied with respect to MWCNTs concentrations at different temperatures. The interaction among nanotubes increases due to denser solid surface and the fluid flow resistance also increases, that leads to increase in viscosity values. From the thermal conductivity measurements of MWCNTs nanofluids, it is observed that the dispersion of higher concentrations of MWCNTs in base fluids leads to enhanced thermal conductivity value which is due to greater percolation of heat through the carbon nanotubes to form tri-dimensional network because of the high aspect ratio of the nanotubes. Precisely, the increased quantity of nanotubes forms closely packed thermal interfaces, which in turn, bestowed to acquire improved thermal conductivity in the MWCNTs nanofluids. Among all the six MWCNTs nanofluids prepared, Dowtherm A based MWCNTs nanofluids exhibit higher thermal conductivity when compared with silicone oil based MWCNTs nanofluids. Chapter 6 summarizes the experimental findings, concluding remarks and future perspectives. It is concluded that Dowtherm A based -OH functionalized MWCNTs nanofluids has most favorable thermal performance and has the potential for using it as a heat transfer fluid.
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Figure 5.2 Variation in the viscosity values of (a) Pure MWCNT, (b) OH functionalized MWCNT and (c) COOH functionalized MWCNT-Silicone oil nanofluids with different concentrations of MWCNTs at different temperatures

Figure 5.3 Variation in the viscosity values of (a) Pure MWCNT, (b) OH functionalized MWCNT and (c) COOH functionalized MWCNT-Dowtherm A nanofluids with different concentrations of MWCNTs at different temperatures

Figure 5.4 KD2 Pro Thermal properties analyzer

Figure 5.5 Variation in the thermal conductivity values of (a) Pure MWCNT, (b) OH functionalized MWCNT and (c) COOH functionalized MWCNT-Silicone oil nanofluids with different concentrations of MWCNTs at different temperatures

Figure 5.6 Variation in the thermal conductivity values of (a) Pure MWCNT, (b) OH functionalized MWCNT and (c) COOH functionalized MWCNT-Dowtherm A nanofluids with different concentrations of MWCNTs at different temperatures
LIST OF SYMBOLS AND ABBREVIATIONS

\( \beta \) - Adiabatic compressibility
\( \theta \) - Angle of diffraction
\( D \) - Average crystallite size
\( N_A \) - Avogadro’s number
\( K_B \) - Boltzmann constant
\( C_O \) - Capacitance of air
\( C_R \) - Capacitance of reference fluid
\( C_X \) - Capacitance of unknown fluid
\( X_2 \) - Concentration of solute
\( X_1 \) - Concentration of Solvent
\( D \) - Debye
\( \rho \) - Density of nanofluids
\( \varepsilon_R \) - Dielectric constant of reference fluid
\( \varepsilon_x \) - Dielectric constant of unknown fluid
\( \mu \) - Dipole moment
\( \delta \) - Dislocation density
\( \varepsilon^E \) - Excess dielectric constant
\( \mu^E \) - Excess dipole moment
\( g \) - Grams
\( d \) - Interlayer spacing
\( L_f \) - Intermolecular free length
\( K \) - Kelvin
\( f \) - Known frequency of ultrasonic waves
\( P \) - Molar Polarization
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
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<tbody>
<tr>
<td>$M_1$</td>
<td>Molecular weight of base fluids</td>
</tr>
<tr>
<td>$M_2$</td>
<td>Molecular weight of nanotubes</td>
</tr>
<tr>
<td>MWCNT</td>
<td>Multi-walled carbon nanotube</td>
</tr>
<tr>
<td>$n$</td>
<td>Order of diffraction peak</td>
</tr>
<tr>
<td>$a_0$</td>
<td>Slope of static dielectric constant with</td>
</tr>
<tr>
<td></td>
<td>concentrations</td>
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<tr>
<td>$a_{\infty}$</td>
<td>Slope of optical dielectric</td>
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<tr>
<td></td>
<td>constant with concentrations</td>
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<tr>
<td>$Z$</td>
<td>Specific acoustic impedance</td>
</tr>
<tr>
<td>$\varepsilon$</td>
<td>Static dielectric constant</td>
</tr>
<tr>
<td>$K_T$</td>
<td>Temperature dependant Jacobson constant</td>
</tr>
<tr>
<td>$T$</td>
<td>Temperature of the medium</td>
</tr>
<tr>
<td>$k$</td>
<td>Thermal conductivity</td>
</tr>
<tr>
<td>$v$</td>
<td>Ultrasonic velocity of nanofluids</td>
</tr>
<tr>
<td>$\eta$</td>
<td>Viscosity of nanofluids</td>
</tr>
<tr>
<td>$\lambda$</td>
<td>Wavelength</td>
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