

**CHARACTERIZATION AND DETERMINATION
OF THERMAL PROPERTIES OF CARBON-
BASED NANOFLUIDS AS HEAT TRANSFER
MEDIA**

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**MASTER OF SCIENCE
(CHEMISTRY)**

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PROPERTIES OF CARBON-BASED NANOFLUIDS AS HEAT TRANSFER
MEDIA**

NORSHAFIQAH BINTI MOHAMAD SAIDI

Thesis submitted to the Centre for Graduate Studies, Universiti Pertahanan Nasional
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(Chemistry)

ABSTRACT

Nanofluid is a new potential fluid for cooling applications. It is a colloidal suspension of nanoparticles such as metals, metal-oxides and carbon-based materials in the conventional base-fluids such as water, ethylene glycol and oils. This new generation of nanofluid possess great thermophysical properties that can be considered for wide range of heat transfer applications. Carbon-based materials is a good candidates to prepare nanofluids due to their high thermal conductivity. However, the inability of carbon particles to disperse homogenously in most fluids imposes a major limitation on an effective application. Therefore, it is crucial to synthesis well dispersed nanoparticles in conventional based fluids. In this research, three type of carbon-based materials were used namely, Multi-walled Carbon Nanotube (MWCNTs), Carbon Nanofibers (CNFs) and graphite flakes. The commercial MWCNTs and CNFs were chemically modified using three different methods of acid treatment, whereas graphite flakes were chemically modified using Hummers method to introduce surface oxygen functional group (SOFG). The presence of SOFG on the carbon materials has been characterized by Fourier Transform Infrared (FTIR) spectroscopy. Morphology, structural and thermal properties were performed using Field Emission Scanning Electron Microscopy (FESEM), Raman spectroscopy and Thermogravimetric analysis (TGA), respectively. Based on the results obtained, modification with acid treatment has significant effect on both the degree of defects and surface group functionality of all samples. Among all, Method B of acid treatment was chosen as the best method to synthesize the commercial. Preparation of nanofluids was followed by using two different parameters: with and without polyvinylpyrrolidone (PVP). The experiment was conducted by setting variable concentration of carbon particle from range 0.1 wt% to 1.0 wt% and the amount of PVP is 10% concentration of carbon particles at different temperature (6 °C, 25 °C, 40 °C). Based on visual method, the dispersion of carbon particles was enhanced by the presence of PVP as the stabilizing agent. Whereas, for thermal conductivity test, graphene oxide without PVP gives the highest thermal conductivity enhancement with 23.28% for concentration of 1.0 wt% at temperature 40 °C with better stability. In overall, we successfully prepared carbon-based nanofluids with better stability and high thermal conductivity.

ABSTRAK

Bendalir nano merupakan satu bendalir baru yang berpotensi untuk aplikasi penyejukan. Ia adalah dispersi koloid partikel nano seperti logam, logam-oksida dan bahan berasaskan karbon dalam bendalir-asas konvensional seperti air, ethylene glycol dan minyak. Bendalir nano generasi baru ini mempunyai sifat termofizik hebat yang dapat dipertimbangkan dalam pelbagai aplikasi pemindahan haba. Bahan berasaskan karbon adalah calon yang sesuai untuk penyediaan bendalir nano disebabkan oleh mempunyai kekonduksian haba yang tinggi. Walau bagaimanapun, ketidakupayaan partikel karbon untuk tersebar secara homogen dalam kebanyakan bendalir menghadkan aplikasi yang berkesan. Oleh itu, adalah penting untuk mensintesis partikel nano yang tersebar dengan baik dalam konvensional berasaskan-bendalir. Dalam kajian ini, tiga jenis bahan karbon digunakan iaitu; MWCNT, CNF dan serpihan grafit. MWCNT dan CNF yang komersial diubah secara kimia menggunakan tiga (3) kaedah rawatan asid yang berbeza, manakala serpihan grafit diubah secara kimia menggunakan kaedah Hummers untuk memperkenalkan kumpulan fungsi oksigen dipermukaan (SOFG). Kehadiran SOFG pada bahan karbon telah dicirikan oleh spektroskopi Fourier Transform Infrared (FTIR). Morfologi, sifat struktur dan terma diuji dengan menggunakan Field Emission Scanning Electron Microscopy (FESEM), Raman spektroskopi dan analisis Thermogravimetric (TGA). Berdasarkan hasil yang diperolehi, pengubahsuaian dengan rawatan asid mempunyai kesan yang signifikan terhadap tahap kerosakan dan fungsi kumpulan dipermukaan bagi semua sampel. Di antara semua, kaedah rawatan B dipilih sebagai kaedah yang terbaik untuk mensintesis MWCNT dan CNF yang komersial. Bendalir nano disediakan menggunakan dua jenis parameter yang berlainan; tanpa dan dengan PVP. Eksperimen ini dijalankan dengan menetapkan pembolehubah kepekatan zarah karbon dari julat 0.1wt% hingga 1.0 wt% dan jumlah PVP adalah sebanyak 10% kepekatan partikel karbon pada suhu yang berbeza (6 °C, 25 °C dan 40 °C). Berdasarkan kaedah pemerhatian kasar, penyebaran zarah karbon meningkat dengan kehadiran polyvinylpyrrolidone (PVP) sebagai agen penstabil. Untuk ujian kekonduksian terma, graphene oxide tanpa PVP memberikan peningkatan kekonduksian terma tertinggi dengan 23.28 % untuk kepekatan 1.0 wt.% pada suhu 40°C dengan mempunyai kestabilan yang bagus. Secara keseluruhannya, kami berjaya menyediakan bendalir nano berasaskan karbon dengan kestabilan yang lebih baik dan kekonduksian terma yang tinggi.

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APPROVAL

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LIST OF ABBREVIATIONS

MWCNTs	-	Multi-walled carbon nanotubes
SWCNT	-	Single walled carbon nanotubes
CNFs	-	Carbon nanofibers
HHT	-	High heat treatment
GO	-	Graphene oxide
SOFG	-	Surface oxygen functional group
FESEM	-	Field emission scanning electron microscopy
FTIR	-	Fourier transform infrared spectroscopy
TGA	-	Thermogravimetric analysis
DI	-	Deionized water
PVP	-	Polyvinyl pyrrolidone
TiO ₂	-	Titanium oxide
CVD	-	Chemical vapour deposition
HNO ₃	-	Nitric acid
H ₂ SO ₄	-	Concentrated sulphuric acid
KmMNO ₄	-	Potassium permanganate
PANI	-	Polyaniline
PVA	-	Polyvinyl alcohol
PAN	-	Polyacrylonitrile
Te	-	Tellurium
CoFe	-	Cobalt-iron
CaCO ₃	-	Calcium carbonate
SAN	-	Styrene-acrylonitrile
Al ₂ O ₃	-	Aluminium oxide
CNC	-	Carbon nanocoil
C ₂ H ₅ OC ₂ H ₅	-	Ethoxy ethyl
DDA	-	Dodecylamine
EDA	-	Ethylenediamine

NGQNs	-	N-doped graphene quantum dots
N-CNF	-	N-doped carbon nanofibers
SDBS	-	Sodium dodecylbenzene
SDS	-	Sodium dodecyl sulphate
OA	-	Oleic acid
SAAS	-	Polyacrylic acid sodium
DTG	-	Differential thermogravimetric
NC	-	Nanocarbon
TPD	-	Thermal desorption spectroscopy
XPS	-	X-ray photoelectron spectroscopy
BET	-	Brunauer-Emmett-Teller
XRD	-	X-ray diffraction
EG	-	Ethylene glycol
nm	-	Nanometer
<i>t</i>	-	Time
$k_{\text{enhancement}}$	-	The enhancement percentage of nanofluid
k_{nf}	-	Thermal conductivity of nanofluid
k_{f}	-	Thermal conductivity of pure base fluid
wt%	-	Weight percentage

CHAPTER 1

INTRODUCTION

1.1 General Introduction

Thermal properties of materials are referring to the response of materials which is related to its changes in their temperature and to the application of heat. These properties exhibited when heat is passed through a material. The thermal properties of materials are important whenever heating and cooling devices are designed. Thermally induced expansion of materials must be considered in the construction industry as well as in the design of precision instruments. The major component of thermal properties is thermal conductivity, heat capacity, thermal expansion and thermal stress. For an example, heat transfer occurs at a lower rate in materials of low thermal conductivity than in materials of high thermal conductivity.

Nowadays, among all, heat transfer media in a liquid has attracted many researchers' attention. Heat transfer media can be defined as a medium that able to transport heat from one component to another component in any process. It also can be used to store heat in a reversible form which can be circulated within the installation. It may be present in a solid, liquid, and/or vapor phase. Each application needs a suitable phase of heat transfer media.

Heat transfer media is the crucial component in order to avoid all the electronic or automobile applications from overheating which could lead to damage in their system. For example, the car engine's cooling system needs transfer media in a liquid form. Their job is to absorb the heat generated by the engine and transport it away, hence keeping the engine cool. Like other applications such as air conditioning system and heating.

Nanotechnology has played an important role in multifield of the heat transfer process and has made significant progress in energy applications. One of the most reasonable applications of nanotechnology is to produce nanoparticles with high thermal conductivity and mixing them with a basic fluid that transfers energy forming what is called nanofluid. Liquid/fluid-based heat transfer is widely used by many researchers such as water, ethylene glycol and engine oil. But they have low thermal conductivity. Their performance can be enhanced by incorporating them with a high thermal conductivity nanoparticle. Nanoparticles are tiny materials that have various sizes ranging from 1 to 100 nm. They can be classified into various types, according to their properties, shapes or sizes [1]. Nanoparticles possess unique physical and chemical properties due to their high surface area and nanoscale size.

The incorporation of nanoparticle in a liquid/fluid is known as nanofluid. However, this modification has a few drawbacks where it can cause contamination in the heat transfer media. The nanoparticle agglomerate and may cause blockage of the device's channel. This could reduce the practical application of incorporation of nanoparticle in a base fluid for heat transfer application.

There is an alternative to improve the capability of heat transfer in nanofluid which is by introducing surface oxygen functional group (SOFG). Oxidation by acid treatment or acid solution is one of the surface modification methods used to introduce SOFG [2]. This method was used to insert oxygen functional groups [2]–[5] such as carboxyl group (COOH), carbonyl group (C=O) and hydroxyl group (OH) on the sidewall of carbon particles and create defects on the surface walls. This method is able to enhance thermal conductivity of heat transfer media which can be used in many applications.. SOFG able to increase the interaction between carbon particles and water molecules, and as a result the hydrophobic carbon particles becoming more hydrophilic, which greatly improves their dispersibility

1.2 Problem Statement

The dispersion of carbon particles in liquid/fluids can be challenging. This is due to the strong van der Waal forces between carbon surfaces. For example, multi-wall carbon nanotube (MWCNTs), carbon nanofibers (CNF) and graphite flakes are hydrophobic in nature, which makes them hard to disperse in water solution under any ambient conditions [6], [7]. Based on the published data to-date, many researchers have started to use dispersant agent such as surfactants to prevent the sedimentation or agglomeration of nanoparticles in which the surfactant produces an efficient coating and induces electrostatic or steric repulsions that could counterbalance van der Waals attractions [7]. However, the usage of surfactant in larger amounts has a few drawbacks, such as it can cause contamination in heat transfer media. Moreover, surfactant molecules will react and attach to the surface of carbon particles which could increase the thermal resistance between the carbon particles and the base liquid. This

could reduce the thermal conductivity of carbon particle due to an increment in the density and viscosity of nanofluid [8], [9], [10]. As we know, heating and cooling are routine processes in heat transfer media. However, the presence of surfactants can lead to the production of foams especially during heating [11]. All these factors could hinder the performance of heat transfer media.

Therefore, it is important to synthesize carbon particles without or with the use of small amount of surfactant in order to achieve outstanding thermal performance which could help to extend its application. In this work, surface modification via acid treatment has been used. This treatment introduced surface oxygen functional group (SOFG) on carbon walls and had a significant effect on the degree of defect. SOFG is able to increase the interaction between the carbon particle and water molecules via hydrogen bonding. As a result, it has increased the hydrophilicity of the carbon particles and prevented their settlement in water over time. The thermal conductivity of carbon-based nanofluids is best associated with functionalized nanofluids. In addition, the usage of polyvinylpyrrolidone (PVP) in a small amount which is 10% of carbon particles, as a dispersant agent in nanofluid will improve the stability or dispersibility of nanoparticles in fluids. Moreover, combination of SOFG based nanocarbon combined with PVP as surfactant in the preparation of nanofluid was less explored by many researchers.