

UNIVERSITI PUTRA MALAYSIA

IMPROVEMENT IN DISPERSEMENT AND AGGLOMERATION PROPERTIES FOR CARBON NANOTUBE INK

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IMPROVEMENT IN DISPERSEMENT AND AGGLOMERATION PROPERTIES FOR CARBON NANOTUBE INK

By

NUR HAZIQAH BINTI ABDUL AZIZ

Thesis Submitted to the School of Graduate Studies, Universiti Putra Malaysia, in Fulfilment of the Requirements for the Degree of Master of Science

December 2021

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Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfilment of the requirement for the degree of Master of Science

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Carbon nanotubes (CNTs) are developing in flexible, stretchable and sensitive electronic technologies for decades because of their impressive properties and performance in electrical and mechanical which are almost similar to metal. However, some of the current methods are costly as well as have issues with dispersion which will cause applomeration and sedimentation of the nanotubes. This study aims to produce improved dispersion of CNT-polymer conductive nano- ink for printing technology without agglomeration and sedimentation issues. The functionalized MWCNTs is dispersed in isopropyl alcohol (IPA) solution and compared with other organic solvents such as methanol, ethanol, acetone, chloroform and Tetrahydrofuran (THF) using Raman spectroscopy, Fourier Electron Scanning Electron Microscopy (FESEM), and current-voltage (I-V) characterisations. The conductive ink is synthesised via ultrasonication and magnetic stirring. Eight samples are prepared for various conditions which include the presence of ethylcellulose, types of chitosan and concentration of MWCNTs. The characterization analyses such as Energy Dispersive X-Ray Analysis (EDX), FESEM, Fourier Transform Infrared Spectroscopy (FTIR), rheological Raman spectroscopy, measurement via rheometer. thermogravimetric analysis (TGA), Ultraviolet-Visible (UV-VIS), and electrical testing's using 4-point probes as well as 2-point probes. EDX results show that the weight percentage of the Carbon (C) atom decrease with the presence of ethylcellulose. Meanwhile, the FESEM images show that the presence of ethylcellulose prevents the formation of nanotubes bundles. However, the higher concentration which is 5 mg/ml gives the better structure of nanotubes. The Raman study shows that the presence of cellulose increased the Peak Intensity Ratio for the carbon defect. The ethylcellulose also increased the viscosity of the conductive nano-ink, 11.13 mPA which is still in the range of the viscosity of commercial ink. Though the presence of ethylcellulose reduced the conductivity of the nano-ink, it successfully prevents the formation of the 'coffee ring' effect. Among all samples, Sample 7, the high concentration which is 5mg/ml with ethyl celluloses and chitosan solution is chosen as the best sample with better performance, stable and optimum conductivity on the glass which are in the range of 1.52×10^{-01} -01 to 2.15×10^{-01} .



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Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Master Sains

PENAMBAHBAIKAN DALAM SIFAT PENYERAPAN DAN PEMENDAPAN UNTUK DAKWAT NANOTUBE KARBON

Oleh

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Karbon nanotiub (CNT) berkembang dalam teknologi elektronik yang fleksibel, tegang dan sensitif selama beberapa dekad kerana sifat dan prestasinya yang mengagumkan dalam elektrikal dan mekanikal yang hampir serupa dengan logam. Walau bagaimanapun, beberapa kaedah semasa adalah mahal selain mempunyai masalah dengan penyebaran yang akan menyebabkan pengagregatan dan pemendapan nanotiub. Kajian ini bertujuan untuk menghasilkan penyebaran dakwat nano konduktif CNT-polimer yang lebih baik untuk pencetak inkjet yang ditambah baik daripada masalah pengagregatan dan pemendapan. MWCNT yang difungsikan disuraikan dalam larutan isopropil alkohol (IPA) dan dibandingkan dengan pelarut organik lain yang merangkumi metanol, etanol, aseton, kloroform dan Tetrahidrofuran (THF) melalui spektroskopi Raman, Mikroskopi Elektron Imbasan Fourier (FESEM), dan ciri voltan arus (I-V). Kemudian, dakwat konduktif disintesis melalui kaedah ultrasonikasi dan pengadukan magnetik. Lapan sampel disediakan untuk pelbagai keadaan termasuk kehadiran etilselulosa, jenis kitosan dan kepekatan MWCNT. Analisis pencirian dilakukan, Analisis X-Ray Penyebaran Tenaga (EDX), FESEM, Spektroskopi Inframerah Transformasi Fourier (FTIR), spektroskopi, pengukuran reologi rheometer, Raman melalui analisis termogravimetrik (TGA), Ultraviolet-Terlihat (UV-VIS) spektroskopi, dan ujian elektrik yang merupakan 4-titik siasatan dan juga 2-titik siasatan. Hasil EDX menunjukkan bahawa peratusan berat Karbon (C) atom menurun dengan adanya etilselulosa. Sementara itu, gambar FESEM menunjukkan bahawa kehadiran etilselulosa menghalang pembentukan ketulan nanotiub. Walau bagaimanapun, kepekatan yang lebih tinggi iaitu 5 mg / ml memberikan struktur nanotiub yang lebih baik. Dari kajian Raman, keputusan menunjukkan bahawa kehadiran selulosa meningkatkan Nisbah Intensiti Puncak untuk kecacatan karbon. Etilselulosa juga meningkatkan kelikatan tinta nano konduktif, 11.13 mPA yang masih dalam julat kelikatan dakwat komersial. Walaupun kehadiran etilselulosa mengurangkan kekonduksian nano-ink, ia berjaya mencegah pembentukan kesan 'cincin kopi'. Di antara sampel, Sampel 7, kepekatan tinggi 5mg / ml dengan etilselulosa dan larutan kitosan dipilih sebagai sampel terbaik dengan prestasi yang lebih baik, kekonduksian stabil dan optimum yang berada dalam lingkungan $1.52x\,10^{-01}\,\text{hingga}\,2.15x\,10^{-01}.$



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LIST OF ABBREVIATIONS AND SYMBOLS

a	absorption coefficient
°C	Celsius
3	Strain
λ	wavelength
π	^{pi} PM
ρ	Resistivity
σ	Sigma, stress
σ	Conductivity
Ω	Ohm
Ω	angular motion
ý	shear rate
А	Absorbance
A	Ampere
a. u	Arbitrary unit
C	the speed of light
CIJ	continuous inkjet
cm	centimeter

	CNTs	Carbon nanotubes
	-СООН	Carboxyl group
	CVD	Chemical vapour deposition
	DOD	Drop-on demand
	E	Young's modulus
	EDX	Energy Dispersive X-Ray
	Eg	Optical bandgap energy
	eV	Electron volt
	F _{vw}	Van der Waals force
	FESEM	Field Emission Scanning Electron Microscopy
	FTIR	Fourier Transform Infrared
	FWHM	Full Width at Half Maximum
	h	plank constant
	hv	Photon energy
		Current
	I -V	Current voltage
	IPA	Isopropyl alcohol
	к	Kelvin
	I	length

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	m	meter
	Mg/ml	Milligrams per milliliter
	mm	millimeter
	MWCNTs	Multi-walled carbon nanotubes
	-OH	Hydroxyl group
	PDMS	Polydimethylsiloxane
	PVP	Polyvinylpyrrolidone
	R	Resistance
	RFID	Radio-frequency identification
	S	Siemen
	SDS	Sodium dodecyl sulfate
	SMU	Source Meter Unit
	SWCNTs	Single-walled carbon nanotubes
	t	Thickness
	TGA	Thermogravimetric analysis
	THF	Tetrahydrofuran
	UV-Vis	Ultraviolet-Visible
	V	Voltage

V Volts

Wt % Weight percent





CHAPTER 1

INTRODUCTION

1.1 Background of study

Carbon materials are widely involved in electronic and biomedical fields for the past few decades. The study and development have been actively running since then. Besides, research and development on carbon nanomaterials are renowned because carbon is non-metals with excellent electrical properties as well as better mechanical properties compared to metals. Kholgi stated in their paper, carbon nanotubes (CNTs) are very captivated nanomaterials because CNTs are low-cost nanomaterials with extraordinary electrical, mechanical, and optical properties[1]. The extraordinary properties mentioned are strong, lightweight, flexible, large aspect ratio of current capacity, rapid electron transfer, broad surface area, and ability to act as metallic or semiconducting [1]. Therefore, CNTs have become very high in demand material in the applications of nanotechnology, which are transistors[2], sensors [3], radio-frequency identification (RFID) tags[4], and many more.

Actuated from the extraordinary properties of CNTs, research and developments related to CNTs are actively running worldwide. There are a few deposition methods of CNTs which are dip coating [5], spray coating[6], electrophoretic deposition[7], and printing technologies are classified into two, which are contact and non-contact printing. The most enthusiastic and fastest method for printed electronic applications is inkjet printing under the non-contact printing method. Inkjet printing is a rapid low-cost printing method since it requires no prefabrication of templates and can produce multiple designs simultaneously with accurate measurement in thickness, edges of design[1]. The method used in this study is inkjet printing, which is a non-contact printing method.



Figure 1.1: Printing technologies

The low-cost and superior properties of CNTs made the use of CNTs via inkjet printing in these nanotechnology inventions are the best combination of the method. In the preparation of ink for an inkjet printer, there are three major elements which are colourant (also known as filler), vehicle (also known as solvent), and additives. Those elements depend on significant applications. In the production of conductive nano-ink, a suspension of carbon nanomaterials, MWCNTs, and polymer are included as the colourant or filler. Besides, to achieve the structure of the industrial ink, MWCNTs are dispersed in an organic solvent with the help of additives. Similarly, additives are also used to stabilise the properties of MWCNTs. MWCNTs are chosen over SWCNTs because MWCNTs offer better conductivity with a lower cost of purchase.

Few factors need to be concerned while the making of MWCNTs conductive nano-ink for an inkjet printer and the most vital factors to examine are material quality, degree of dispersion, and stability. However, most researchers found issues of stability and dispersion of MWCNTs in the solvent. CNTs are difficult to disperse. The problems such as agglomeration and sedimentation may occur because of the strong Van der Waals forces between CNTs, which can lead to clogging in the inkjet nozzle [8]. Therefore, to resolve the issues, there are studies on methods and materials that can help in developing a good dispersion of MWCNTs conductive carbon nano-ink.



As mentioned earlier, there are issues in developing MWCNTs conductive nano-ink, especially the dispersion of MWCNTs in a solvent. The issues are agglomeration, maintaining suspension along with dispersion as well as concerns of health, safety, and environment[9]. To counter the agglomeration issue, a study reported, there are three methods which are functionalization of the sidewalls of the CNTs, ultrasonication of MWCNTs into the solvent, and lastly, use of organic solvents to overcome the hydrophobicity of MWCNTs. Apart from that, a wise choice of solvents[10], ultrasonication[8] and addition of copolymers[11] able to maintain suspension as well as dispersion of aqueous

solution with time. Over and above, regarding health, safety, and environmentally friendly, carbon nanoforms are less hazardous compared to heavy metals.

In this project, there are two stages of study which are the study of the dispersion of MWCNTs in organic solvents and the study of the synthesised conductive nano-ink. The studies involved, electrical properties, the morphology of samples, chemical properties and optical properties of the samples. The purpose of the first stage is to choose the best organic solvent for synthesising conductive nano-ink in the second stage.

1.2 Problem statement

The involvement of nanotechnology in daily life is growing rapidly day by day. This matter is also involved in the development of a flexible sensor. The flexible sensor is being widely used and studied in the most field, which is industrial, safety, medical as well as research in the laboratory. Nowadays, the high demand for flexible sensors has led to inkjet printing technology[12] is being explored to fulfil the demand.

The motivation of this study is to synthesize a well-dispersed and stable CNTs/polymer conductive nano-ink that can be used in a commercial inkjet printer. The inkjet printing method is low in cost and requires a short time in fabrication compared to another printing method. Inkjet printing offers fast fabrication as sensors can be printed directly after they are designed.

However, the nature of CNTs having a hydrophobic surface and strong Van der Waals forces poses major challenges to synthesize the ink for inkjet printers. The behaviours of CNTs are the major reason which causes CNTs to hardly be dispersed, as well as the formation of agglomeration and sedimentation. These issues will be the biggest factor of clogging in the nozzle as well as unstable conductivity. The poor dispersion also may affect the viscosity of the ink which the ink will be high viscosity since there are undispersed MWCNTs in the ink solution. Besides, in the formulation of conductive inkjet printing, a few other aspects that influence the conductivity and performance of nano-ink need to be taken care such as the 'coffee ring' stain effect[13][14], porosity[15], and wettability. The 'coffee ring' effect stain is presented if the CNTs are not well dispersed and will also affect the conductivity of the nano-ink. Aurore Denneulin stated that to get the optimum electrical conductivity, a few defaults such as the 'coffee ring' stain effect should be avoided[16]. The range of conductivity of MWCNTs in current studies are between x 10^{-4} to the x 10^{-3} [17][18].

Few methods have been applied to solve the problems including using functionalised CNTs. The involvement of an alcohol solution, which is helping to increase the dispersion and wettability of the conductive ink. The better dispersion and improvement on agglomeration and sedimentation of conductive ink are the main contributions to this project. Besides, the dispersion and other properties of CNTs can be maintained by using additives and optimum mechanical reaction.

1.3 Objectives

This study is to formulate a stable conductive nano-ink. The design of research has the following objectives:

- 1. To improve the dispersion of CNTs/polymer conductive ink by reducing the agglomeration and sedimentation of conductive ink using chemical reaction which is by involving additives and solvent as dispersant agent, also mechanical reactions which are ultrasonication and magnetic stirring.
- To synthesise CNTs/polymer conductive ink in an alcohol solvent, Isopropyl Alcohol (IPA) with the addition of additives which are Sodium dodecyl sulphate (SDS), Polyvinylpyrrolidone (PVP), a-Terpineol, chitosan and ethylcellulose.
- 3. To characterise the electrical and physical properties of the synthesised conductive ink which includes the I-V characteristics and morphology characterizations to get conductive ink with improved dispersion.

1.4 Scope of work

The scope of work is primarily on the synthesis of Carbon Nanotubes (CNTs) conductive nano-ink. The major problem to solve is to homogeneously disperse the CNTs in the solvent to form an ink as well as to maintain the stability and conductivity of the nano-ink without agglomeration and sedimentation. This study involves three major parts which are the study of theoretical data and running of simulation for electrical data, study the dispersion of MWCTs in organic solvents and the study of synthesised MWCNTs nano-ink. Firstly, the study of theoretical data by referring to the research papers and the study of the behaviour of MWCNTs via Energy Dispersive X-Ray Analysis (EDX) as well as the simulation of COMSOL to study the electrical properties of MWCNTs. The results are then as the reference for the whole experiment. Then, two types of MWCNTs, MWCT-COOH and MWCNT-OH are dispersed into six different organic solvents which are acetone, chloroform, ethanol, propanol, methanol and THF. The characteristics of the dispersion solution are then studied by running a few characterisations which are Raman spectroscopy, Field Emission Scanning Electron Microscope (FESEM) and I-V characteristics. The last part of this study is the synthesize of MWCNTs nano-ink. The

synthesis MWCNTs nano-ink is analysed based on physical and electrical characterisations which are the ultraviolet-visible spectroscopy (UV-Vis) for the dispersion of the conductive ink solution, Fourier Transform Infrared spectroscopy (FTIR) is for determining the functional groups and materials in the ink solution, FESEM and Energy Dispersive X-Ray analysis (EDX) for the study of surface morphology and elemental composition of the ink solution, Raman spectroscopy which is to determine the chemical structure of ink. solution as well as rheometer for the viscosity and flow rate the ink. Besides, thermogravimetric analysis (TGA) and electrical properties are also involved to characterise the conductive ink which is to study the condition of the ink in different environments and conductivity of the ink respectively. In addition, the conductivity of the ink solution is also studied. For the synthesis of MWCNTs nano-ink, isopropanol is chosen as the organic solvent due to the optimum performance and stability of the MWCNTs in the solvent after being dispersed. The ink solution needs to meet the range of viscosity of the commercial ink, have conductivity and better dispersion which is no agglomeration and sedimentation.

1.5 Contribution of Work

This research study focuses on dispersion improvement of the MWCNTs conductive nano-ink. Problems such as agglomeration and sedimentation issues were successfully solved. The enhanced nano- ink has better dispersion and more stable compared to previous studies. The nano-ink is able to remain conductive, stable and well dispersed for a longer duration (more than two weeks This research contributes to the improvement of ink stability benefits the development of flexible electronics and are very useful for printing electronic devices such as chemical sensors that require stable inks with moderate conductivity. The nano-ink has been studied in several characteristics. The data have been recorded and discussed in detail in this thesis.

1.6 Thesis outline

This thesis comprises five chapters that contain the required information in understanding the process and study of the synthesis of conductive nano-ink.

Chapter 1, the introductory chapter, presents the background of study related to the sensor technology, printing technology and composition of the ink. Chapter 1 also included the problem statement, objectives of the research, scope of work and thesis outline.

Chapter 2 discusses in detail of literature review. This chapter includes a review on sensor technology, materials of a sensor, printing technology, preparation of carbon nanotube for the synthesis of ink, and synthesis of

conductive ink. Besides, this chapter also stated a review on the resistivity and piezo resistivity, parameters of a sensor, testing, and characterisation and lastly, the summary of techniques, materials, and characterisation.

Chapter 3 presents the flow of work to achieve the objectives of the research, materials used throughout the research, method in the dispersion of CNTs, the method in the synthesis of CNTs conductive nano-ink, the design involved, printing process as well as the characterisation and data analysis.

Chapter 4 comprises results and discussion based on the characterisation of the CNTs conductive nano-ink. The characterisations involved are conductivity, FESEM, FTIR, EDX, SEM, UV-Vis and TGA, rheology and Raman Spectroscopy. The results show the study of the electrical and physical properties as well as the morphology of the conductive nano-ink.

Finally, Chapter 5 concludes the research works related to the objectives and methodology as well as provides the recommendation for future work.

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