UNIVERSITI TEKNIKAL MALAYSIA MELAKA

PREPARATION AND CHARACTERIZATION OF MODIFIED POLYANILINE – GRAPHENE NANOPLATELETS (PANI / GNPs) NANOCOMPOSITES VIA ACID PROTONATION METHOD

This report is submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)

by

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Date : 24th August 2020

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

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ABSTRAK

Sebelum ini, patch konduktif antenna diperbuat daripada tembaga, yang tidak fleksibel, mahal, fenomena berbilang pudar, besar, sensitif alam sekitar dan sukar untuk dihasilkan. Nanoteknologi ajaib graphene kerana kekonduksian elektrik luar biasa dan kekuatan unggul daripada logam, tetapi fleksibel, telah membolehkan mereka menjadi calon yang beroperasi menggantikan logam tembaga tanpa kompromi. Walaubagaimanapun, kelemahan utama graphene adalah kekurangan jurang band tenaga yang menutup banyak aplikasi potensi peranti (PANI). Manakala acid dodecylbenzenesulfonic (DBSA) digunakan sebagai penambah untuk meningkatkan sifat-sifat PANI. Kain yang boleh dipakai sebagai antena jenis penerima isyarat fleksibel, adalah topik penyelidikan yang hangat untuk komunikasi sentris badan dalam era revolusi industri 4.0 (IR4.0). Aplikasi antenna tekstil boleh pakai berkembang dengan pesat, terutamanya untuk pengkomputeran mudah alih, menjejaki navigasi dan keselamatan awam. Kajian ini adalah untuk menilai potensi penggubalan novel graphene nanoplatelets/polyaniline (GNPs/PANI) nanokomposits konduktif untuk generasi baru antena tekstil dapat dipakai fleksibel. Penyelesaian nanokomposits GNP/PANI telah disintesis oleh pempolimeran anilin dalam medium berasid. Kesan ke atas pemuatan GNP (0.25 wt %, 0.50 wt %, 0.75 wt % and 1.00 wt %) pada sifat radiasi antena untuk nanokomposit PANI/GNP/DBSA telah dicirikan dan dinilai, menggunakan kaedah pencirian morfologi, fizikal dan elektrik standard. Gabungan GNP dan PANI menyempurrnakan satu sama lain, meningkatkan sifat kekonduksian elektrik yang sedia ada, sambil membuka ciri-ciri berpotensi lain, terutamanya untuk pembangunan antena yang boleh dapat dipakai. Dari penyelidikan ini, bahan-bahan baru yang dirumuskan untuk nanokomposit PANI/GNP/DBSA tekstil dan pemahaman asas yang berkaitan untuk aplikasi sedemikian, dan pendekatan metodologi standard untuk fabrikasi antena yang dapat dipakai pakaian pintar telah dibentuk.

ABSTRACT

Previously, the conductive patch of antenna is made from copper, which are inflexible, expensive, multi-fading phenomena, bulky, environmental sensitive and difficult to manufacture. Miracle nanotechnology of grapheme owing to their extraordinary electrical conductivity and superior strength than metal, but flexible, has allowed them to be a potential candidate replacing uncompromising copper metallic. Furthermore, the main drawback of graphene is lack of energy band gap which close down many electronic device potential applications. Hence, in this research, graphene nanoplatelets (GNP) is incorporated with polyaniline (PANI) conductive polymers. While dodecylbenzenesulfonic acid (DBSA) use as doping to enhance the properties of PANI. Wearable fabrics as flexible body signal receiver type antennas, are overwhelmed research topics for body centric communication in an industrial revolution 4.0 (IR4.0) era. Wearable textile antenna applications are expanding quickly, especially for mobile computing, tracking navigation and public safety. This study is to evaluate the potential of novel formulation of graphene nanoplatelets/polyaniline (GNPs/PANI) conductive nanocomposites for the new generation of flexible wearable textile antenna. GNPs/PANI nanocomposites solution has been synthesized by aniline polymerization in an acidic medium. The effects on GNP loading (0.25 wt %, 0.50 wt %, 0.75 wt % and 1.00 wt %) on antenna radiating properties for PANI/GNPs/DBSA textile nanocomposite has been characterized and evaluated using standard morphological, physical and electrical characterization method. The combination of PANI and GNPs are perfecting each other, enhancing the existing electrical conductivity properties, while open up other potential characteristics especially for robust wearable antenna development. The simulated wearable textile antenna such as return loss, radiation pattern and antenna gain had been investigated. From this research, new materials formulation of PANI/GNPs/DBSA textile nanocomposites and related fundamental

understanding for such application, and standard methodological approach for smart clothing wearable antenna fabrication was established.

DEDICATION

Only

My beloved father, Ab. Majid Bin Sakimin,

My appreciated mother, Rosidah Binti Salikan,

My supervisor, Ts. Dr. Jeefferie Bin Abd Razak,

My appreciated families and all my friends and colleagues

for giving me moral support, cooperation, encouragement and also understandings.

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LIST OF ABBREVIATIONS

PANI	-	Polyaniline
GNPs	-	Graphene Nanoplatelets
DBSA	-	Dodecylbenzenesulfonic Acid
CNT	-	Carbon nanotube
CVD	-	Chemical vapour deposition
2D	-	Two dimensional
XRD	-	X-ray Diffraction
SEM	-	Scanning Electron Microscope
PNC	-	Polymer nanocomposites
FTIR	-	Fourier Transform Infrared Spectroscopy
VNA	-	Vector Network Analysis (VNA)
DUT	-	device-under-test

LIST OF SYMBOLS

TPa	-	Terapascal
µm	-	Micrometer
%	-	Percent
g/m^3	-	Gram/cubic meter
wt. %	-	Weight percent
mm	-	Millimetre
°C	-	Degree Celsius
W/mK	-	Watt per metre per Kelvin
nm	-	Nanometre
r_f	-	Fibre radius
S/m	-	Siemens per meter
g/mL	-	Gram/millilitre
dBi	-	Decibels-isotropic
W/m.K	-	Watt per metre kelvin
RH	-	Relative humidity
mA	-	miliamperes
Hz	-	Hert

CHAPTER 1
INTRODUCTION

In Chapter One explain a full description of study context, the problem statement of project, objectives, study scope, rationale and research, and thesis organization. This work was fully justified in this chapter because it is important for the research study. In addition, the research scopes cover the depth of the investigation in this chapter. For research background, brief introduction on polyaniline (PANI), graphene nanoplatelets (GNPs), dodecylbenzene sulfonic acid (DBSA), wearable antenna fabrication and application. Where, for problem statement was highlighted few problems related with this work.

1.1 Research Background

The aim of this research is to create preparation and characterization of modified polyaniline – graphene nanoplatelets (PANI/GNPs) nanocomposites with dopant dodecylbenzene sulfonic acid (DBSA) materials. Superb electrical conductivity, thermal conductivity, mechanical properties, and layers of gas barriers attributes are the key properties of GNPs. The properties of GNPs can be affected by the methods of production. The most promising characteristic of wearable antenna made of PANI/GNPs is electrical conductivity, as it has provided the most common antenna in field such as deformable electronics and flexible wearable. Previously, an incorporation of the conductive nanoparticles inside the conductive polymer nanocomposites was performed, but this approach is expensive. In this study, to further increase the chemical conductivity. Properties of PANI/GNPs nanocomposites, the produced composites were doped with DBSA. For applications related to supercapacitors, electrochemical sensors,

generators, impressive results are obtained using such an approach. However, in order to create conductive wearable antenna with stable electronic efficiency, further research is needed. In particular, wearable electronics requires the re-creation of category materials that are at the same time flexible and foldable, while preserving a sufficient degree of electrical conductivity. The polymeric nanocomposite materials are useful for the conductive wearable due to the inherent mechanical properties of polymers and simple to produce, as well as the wide spectrum of properties accessible with different nanoparticles added. At the same time, polymer offer flexibility in shaping the wearable antenna, not like copper metallic.

Due to the environmental stability, different higher conductivity, polyaniline (PANI) is the one of preferred technically effective polymers. PANI has also been the subject of significant recent attention due to its diverse electrical behavior, high environmental stability in doped and neutral states, ease of synthesis and wide-ranging applications in various fields. In addition, the surface of modification could be an effective way of introducing desired functional groups and controlling their final properties while maintaining the bulk characteristics of PANI polymer macromolecules. Composite materials are based on PANI that have the ability for multiple applications such as various sensors, gas sensing and inductors. In addition, PANI get involves in integrating electrical properties, usually as typical semiconductors with polymer-specific material parameters, such as the production of electrochromic instruments, plastic microelectronics, and smart fabrics. Modification of the PANI with GNPs and DBSA to be carried out in this report. Most certainly, the PANI properties would be improved.

The amounts of GNPs used for this analysis being will be varied by 0.00 wt %, 0.25 wt %, 0.50 wt %, 0.75 wt % and 1.0 wt %, respectively. In this study, nanocomposites from PANI and PANI-GNPs are synthesized at the first stage by aniline oxidative polymerization in acid medium process. The PANI content in this composite was determined by the GNP weight gain after modification was performed by using DBSA and its percentage results were further determined. In terms of morphological analysis, physical and electrical conductivity testing, several experiments are being carried out. The morphological analysis is conducted by using Scanning Electron Microcopy (SEM). In addition, X-Ray diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) were used for physical properties analysis while the

electrical properties testing was used to determine the conductivity properties of the produced samples.

The results obtained will be further compared with other proven results from other previous researchers. There are several promising applications that include wearable antenna form such as military, medical, mobile devices, networking, and so on. As a result, given the vast potential and in line with the material revolution in the industrial 4.0 (IR4.0) era, it was indeed quite feasible to undertake this research.

1.2 Problem Statement

Researchers around the globe have been actively investigating the preparation material that can use in wearable antennas. The concept of wearable technology has been debated by futurists and antenna designers for wide-band interaction for almost two decades. This thesis provides a fundamental in exploring and understanding the potential of formulating material for versatile wearable textile antenna based on PANI-GNPs/DBSA based nanocomposites using cotton fabric as the substratum model. This investigation is novel and has not similar with other previous work conducted by other investigators.

The first problems that may arises for this work are related to no availability of usable wearable type of fabric antenna that has good conductivity. In this research will therefore include basic information and important data for preparation and characterization of modified polyaniline – graphene nanoplatelets (PANI/GNPs) nanocomposites via acid protonation method. The materials used are the materials that are capability in wearable textile antenna application for further research.

In addition, this work aimed to propose a novel modified preparation materials made from the combination of PANI/GNPs nanocomposites via acid protonation method. Eventually, there is currently no standard understanding of the properties of the wearable fabric antenna made up of polymer nanocomposites. In addition, this work takes the initiative to understand the effects of GNP filler loads, DBSA doping strategy. The most common problem in dealing with polymer nanocomposites was the agglomeration classification of GNP nanofillers. In this analysis, the results of the

addition of nanofillers to the specific loading percentage will be measured taking into account the condition of agglomeration that may occur due to the larger graphene surface region. This work is intended to use traditional solution casting technique to synthesize free standing films of doped PANI- GNPs nanoplatelet /DBSA nanocomposites with higher conductivity. The existence of GNPs in the PANI matrix has enhanced the composite film's electrical conductivity due to the development of three-dimensional conductive networks and the production of additional charging carriers. In addition, what new for this study is the influence of DBSA as protonation agent to further enhance the performance of PANI/GNPs nanocomposites.

1.3 Objectives

The objectives of this research are as follows:

a) To modify the PANI-GNPs nanocomposites using DBSA acid protonation method during the oxidative aniline polymerization process.

b) To characterize the physical and chemical properties of PANI-GNPs/DBSA nanocomposites using XRD, FTIR and electrical conductivity tests.

c) To evaluate the morphology characteristic of PANI-GNPs/DBSA nanocomposites powder using SEM test observation.

1.4 Scopes of the Research

The scopes of research are as follows:

a) In order to achieve the objectives set out for this study, several steps have been considered to achieve the modified of the PANI-GNPs/DBSA nanocomposites using acid protonation method. It includes a comprehensive review of the literature, which is used to research the previous antenna design and materials. It also important to

develop the basic understanding to identify the expected result and limitation within the preparation and characterization of modified materials used.

b) The volume of GNPs consumed in this sample are 0.00 wt %, 0.25 wt %, 0.50 wt %, 0.75 wt %, and 1.0 wt %. The modification of GNPs with PANI was performed by oxidative aniline polymerization in acidic medium for sample preparation of pristine PANI and PANI-GNPs/DBSA nanocomposites.

c) Unfilled PANI/DBSA to control sample and being compared with PANI/DBSA with filled GNPs.

d) The final content of pure PANI and PANI – GNP/DBSA nanocomposites is being checked for the morphological, electrical and physical properties are being characterized. The morphological study is done under the Scanning Electron Microcopy (SEM). X-Ray diffraction (XRD) and Infrared Spectroscopy Fourier Transform (FTIR) were performed for the physical properties characterization. Electrical analysis was carried out for electrical testing to determine the electrical conductivity of the produced sample.

e) Two main tests in characterizing the powder are consisting of an electrical test and physical testing. Electrical analysis is used to study the electrical conductivity of the PANI-GNPs/DBSA nanocomposites powders.

1.5 Rationale of Research

The rational of research are described as follows:

i. Improving knowledge of PANI-GNPs/DBSA nanocomposites as a possible advanced material by performing some relevant research and characterization for properties improvement.

ii. To achieve understanding of polyaniline modification in acidic medium of PANI-GNPs/DBSA naocomposites.

iii. To generate higher response for specific standard antenna attributes due to improved electrical and physical properties materials of PANI-GNPs/DBSA nanocomposites.

1.6 Thesis Organization

In this research consists of the various sub-topics and chapters. The introduction begins as Chapter One, which includes the background of the research study, the problem statement, the objectives, the scope of the thesis, the rationale for the research and also the thesis organization.

Thus for Chapter Two, briefly discussed about the literature review of the research including related theories and investigations related on antenna, the wearable antenna, polymer nanocomposites, polyaniline, graphene nanoplatelets, dodecylbenzene sulfonic acid and characterization related to wearable antenna by other previous researchers. All this knowledge is necessary to understand the working theory of PANI-GNPs / DBSA nanocomposites preparation and modification.

Chapter Three describes the basic research methodology on conducting the chosen method and the characterization used for variety of important experiments. Also included in this chapter was the flowchart of the overall research methodology from start to finish.

Chapter Four will present the findings and discussions of this study. In addition, all data and related figures on testing and characterization of pristine PANI and PANI-GNPs/DBSA nanocomposites have been put and discussed further in this chapter.

Finally, the conclusion and recommendation of this work is clarified in Chapter Five. The entire discussion on this research will be summarized and concluded exclusively in this final chapter. The suggestion section recommends the enhancement of studies for the next generation of research studies.

CHAPTER 2
LITERATURE REVIEW

Chapter two outlines the theory and work that was developed and carried out by a group of researchers a few years ago. Similar knowledge from previous studies is extracted as references and discussion based on their work on polyaniline (PANI) modification, graphene (GNP) nanoplatelets and specific nanocomposites, conductive polymer material, dodecylbenzene sulfonic acid (DBSA) and wearable antenna, as well as some of the relevant performance tests.

2.0 Introduction

This chapter discusses on the review of related work on this project. This chapter consists of review on the wearable antenna made from electro-conductive polymer material, review on the modification materials of Polyaniline (PANI), Graphene nanoplatelets (GNPs), PANI/GNPs nanocomposites and Dodecyl Benzene Sulfonic Acid (DBSA).

The reviews are important as it will be showcase the feasibility of conducting work. Nevertheless, in this research the new formulation of advanced materials using conductive polyaniline polymer (PANI) containing various graphene nanoplateles (GNP) through acid protonation has already to be developed for modification of the previous preparation and characterization of flexible wearable textile antenna applications. The preparation of modified PANI/GNPs nanocomposites will be proposed due to acid protonation method DBSA. Therefore, this chapter was undertook