SYNTHESIS AND CHARACTERIZATION OF POLYANILINE/MAGNETITE NANOCOMPOSITES FOR FABRICATION OF NANOFIBER TEXTILE

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DEDICATION

To my loving and caring Mother Hajiya Halima Isah and my late father Ibrahim Takai (May his magnanimous gentle soul continues to rest in perfect peace, amen) and my beloved wife Aisha Idris and my son Muhammad Zakiyyu Takai.

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ABSTRACT

Polymers nanocomposites containing inorganic fillers like metal particles dispersed in the polymer matrix are of great interest for the optical and dielectric application. Polymer/inorganic composites such as polyaniline/magnetite nanocomposites (PAni/Fe_3O_4) can be manipulated through various treatments in fabricating desire material such as nanofibers textile for many applications. The modified magnetite (Fe_3O_4) nanoparticles were successfully synthesized and incorporated into polyaniline at different weight ratio and blended with polyvinyl alcohol (PVA) to achieve a spinning solution, further PAni/Fe_3O_4 nanofiber composites solution were used for fabrication of nanofiber textile by an electrospinning method, and the composites nanofiber textile materials were investigated. The crystalline phase structure of PAni/PVA and PAni/Fe_3O_4 composites nanofibers textile was determined by XRD, shows the existence of peaks at $2\theta = 24.13^o$ and 35.63^o for PAni and Fe_3O_4 nanoparticles respectively. The FTIR analysis indicated a slight decrease in the intensity and broadening of the absorption bands at 3462 $cm^{-1}$and 3431 cm^{-1}, are due to vibration stretching –NH group. The disappearance of the peak for PAni/Fe_3O_4 composites nanofibers textile sample containing 25 wt% of Fe_3O_4 nanoparticles clearly indicated the interaction of nanoparticles with nitrogen, hydrogen, carbon, and oxygen atoms in the PAni and PVA chain. FESEM analysis of the composites nanofibers textile shows clearly no accumulation of nanoparticles on the surface of polymeric composites nanofibers. This implies that the growth of nanoparticles on the surface of polymeric composites has successfully been prevented. The decrease of electrical conductivity was observed due to insulating behaviour of Fe_3O_4 nanoparticles. The composites nanofiber textile exhibit hysteric loops under an applied magnetic field of -10000 HOe to 10000 Hoe, for PAni/Fe_3O_4 composites nanofibers textile. Finally, the nanofiber textile materials were successfully fabricated and found that electric and magnetic properties composite textile materials that could be used for many applications.

ABSTRAK

Nanokomposit polimer yang mengandungi pengisi bukan organik seperti partikel logam yang tersebar dalam matrik polimer adalah suatu yang sangat menarik untuk aplikasi optik dan dielektrik. Komposit polimer/bukan organik seperti polianilin/nanokomposit magnetik (($PAni/Fe_3O_4$) boleh dimanipulasikan melalui pelbagai kaedah dalam pemfabrikan bahan tertentu seperti tekstil nanofabrik untuk pelbagai aplikasi. Nanopartikel magnetik yang telah diubahsuai (Fe_3O_4) telah berjaya disintesis dan disatukan ke dalam polianilin pada nisbah berat yang berbeza dan diadun dengan polivinil alkohol (PVA) untuk mencapai larutan berputar. Kemudian, larutan komposit nanofiber $PAni/Fe_3O_4$ telah digunakan untuk pemfabrikan tekstil nanofiber mengunakan kaedah putaran elektron, dan bahan-bahan komposit tekstil nanofiber telah diselidiki. Struktur fasa kristal komposit tekstil nanofiber Pani/PVA dan $PAni/Fe_3O_4$ telah ditentukan oleh XRD, menunjukkan kewujudan puncak pada $2\theta = 24.13^o$ dan 35.63^o untuk nanopartikel PAni dan Fe_3O_4. Analisis FTIR menunjukkan sedikit penurunan dalam intensiti dan keluasan gelombang penyerapan pada 3462 cm^{-1} dan 3431 cm^{-1}, adalah disebabkan getaran regangan kumpulan -NH. Kehilangan puncak bagi sampel komposit tekstil nanofiber $PAni/Fe_3O_4$ yang mengandungi 25 wt% nanopartikel Fe_3O_4 jelas menunjukkan interaksi nanopartikel dengan atom-atom nitrogen, hidrogen, karbon, dan oksigen dalam rantai PAni dan PVA. Analisis FESEM komposit komposit tekstil nanofiber menunjukkan dengan jelas tiada pengumpulan nanopartikel pada permukaan nanofiber komposit polimer. Ini menunjukkan bahawa pertumbuhan nanopartikel pada permukaan komposit polimer telah berjaya dihalang. Penurunan kekonduksian elektrik adalah terlihat disebabkan oleh kelakuan penebat nanopartikel Fe_3O_4. Komposit tekstil nanofiber mempamerkan gelung histeris dibawah medan magnet yang diterapkan dari -10000 HOe hingga 10000 Hoe untuk komposit tekstil nanofiber $PAni/Fe_3O_4$. Akhirnya, bahan-bahan tekstil nanofiber berjaya dibuat dan didapati sifat-sifat elektrik dan magnet bahan tekstil komposit boleh digunakan untuk pelbagai aplikasi.

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LIST OF SYMBOLS AND ABBREVIATIONS

3D	-	3 dimensional
%	-	Percentage
ε	-	Dielectric permittivity
Ω	-	Ohms
π	-	Pi
τ	-	Relaxation time
ε'	-	Dielectric constant
ε''	-	Dielectric loss
ρ_b	-	Bulk density
ε_o	-	Dielectric permittivity of free space
=	-	Equal to
$\leq$	-	Less than
°	-	Degree
°C	-	Degree celsius
β	-	Beta
θ	-	Theta
μ	-	Micro
σ	-	Conductivity
σ_{ac}	-	AC conductivity
σ_b	-	Bulk conductivity
σ_{gb}	-	Grain boundary conductivity
υ	-	Frequency
ω	-	Angular frequency

C_o	-	Capacitance of dielectric constant
A	-	Area
Å	-	Armstrong's
Fe	-	Iron
Fe_3O_4	-	Magnetite nanoparticles
PVA	-	Polyvinyl alcohol
PAni	-	Polyaniline
cm	-	Centimeter
CPE	-	Constant phase element
Cu	-	Copper
D	-	Diameter
EDX	-	Elemental dispersive x-ray
NPs	-	Nanoparticles
FESEM	-	Field emission scanning electron microscope
FTIR	-	Fourier transforms infra-red
FWHM	-	Full width at half maximum
g	-	gram
h	-	hour
Hz	-	Hertz
VSM	-	Vibrating Sample Magnetometer
IS	-	Impedance spectroscopy
JCPDS	-	Joint committee on powder diffraction standard
kg	-	kilogram
M	-	meter
MA	-	Mechanical activation
M″	-	Imaginary electric modulus
M′	-	Real electric modulus
MHz	-	Mega hertz
RT	-	Room temperature
S	-	Siemens
SC	-	Scandium
s.g	-	Space group
t	-	Thickness
XRD	-	X- Ray diffraction

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CHAPTER 1

INTRODUCTION

1.1 Background of the study

The fabrication of nanotechnology has tremendously attracted the attention of the world researchers toward the formation of nanomaterial in the range of 100-500 nm (Wang & Wang, 2014). Therefore, the applied scientists used the idea of polymeric composites to focused on the synthesis, characterization, design and application of several material and devices at nano level (Fakruddin, Hossain & Afroz, 2012). These materials show a considerable change in their properties such as electric and magnetic properties, thermal conductivity, absorbance, whenever they are designed at the nanoscale (Arivalagan *et al.*, 2011).

The discovery of intrinsically conducting polymer in the year 1960 by Alan Heeger, Alan MacDiarmid and Hideki Shirakawa, has hindered the perception that this polymer cannot conduct electricity (Batool *et al.*, 2015; Venkateswarlu *et al.*, 2014). Conducting polymer can be defined as an organic polymer that manages to conduct electricity and possesses combined mechanical properties of the polymer as well as the electronic and optical properties of metals or semiconductor. Conducting polymers such as poly(acetylene) (PA), polyaniline (PAni), poly(3,4-ethylene dioxythiophene) (PEDOT), polypyrrole (PPy) and polythiophene (PT) are some of the conjugated organic polymers, which contain π-electron backbone responsible for their unusual electronic properties such as electrical conductivity, low energy optical transitions, low ionization potential and high electron affinity (Al-Ibrahim *et al.*, 2005; Koul, Chandra, & Dhawan, 2000).

In order to make conducting polymers act as a conductor, the main requirement is the conjugated double bonds, which is alternating single and double bonds in the polymer chain. However, it is insufficient for the conducting polymer to be highly conductive. Thus, the dopant for the formation of a charge carrier in the electric field is required to achieve high conductivity. Dopant, which is either add (n-doping) or remove (p-doping) electron from the polymer chain will create an extra holes (positive charge) or electrons (negative charge) in order for the lonely electron to move easily and thus enhance the electrical conductivity (Hecht, Hu & Grüner, 2007; Noh, 2016).

Therefore, a high density and mobility of charge-carriers along the polymer chains are the main factors for doped conjugated polymers act as a good conductor (Abdi & Sedaghat, 2016; Araújo *et al.,* 2010a; Hanemann & Szabó, 2010). Various aspects can be manipulated including polaron length, the conjugation length, overall chain length and by the charge transfer to adjacent molecules, which can influence the conductivity of conducting polymers (Hossein & Asadnia, 2013; Tayebi *et al.*, 2016). Basically, all these properties are influenced by the synthesis parameters such as different reaction temperature, the effect of dopants, different alkyl substituted monomers and etc.

Among the conducting polymers, PAni is perhaps the universal conducting polymer due to the straightforward synthesis method, stable in air, environmentally friendly and sensing application. Also, they have desire features like chemical stabilities, low density, and adjustable conductivities at a microwave frequency (Philippova *et al.,* 2011; Berglin, 2013). PAni is fitted for the covalent bond of molecules because it has active functional groups in the molecules. Besides, the size and shape of PAni can be controlled by varying synthesis or processing condition which usually obtained with the required electrochemical and physical properties (Xiao & Wang, 2007). The PAni features can be fitted through altering oxidation state, doping or combine it with another form of organic or inorganic nanoparticles (Batool *et al.,* 2015; Liu *et al.,* 2006). Some metal oxide nanoparticles are added to the conducting polymers to form nanocomposites that can be used for fabrication of nanofibers textile materials for many application (Bagheripour *et al.,* 2016; Yu, *et al.*, 2008).

Highly magnetic metals such as cobalt and nickel are toxic and susceptible to oxidation and cannot be used for biomedical applications, iron oxide particles such as magnetite (Fe_3O_4) or its oxidised form maghemite (γ-Fe_2O_3) and hematite (α- Fe_2O_3) are most commonly used (Bonilla *et al.*, 2017; Fajaroh *et al.*, 2012; Sun *et al.*, 2014a; Wilson *et al.*, 2007). Iron oxide nanoparticles have different crystal structures depending on the oxidation state Fe (II) or Fe (III), Fe_3O_4 has a cubic inverse spinel structure.

Fe_3O_4 nanoparticles were used as the reinforcing phase embedded inside the polymer matrix, in the current research. Beside, magnetite nanoparticles have the highest saturation magnetization (74-100 emu/g) (Toomey *et al.*, 2015) and usually low bulk resistivity (1.068 x 10^{-2} Ω·cm) (Farias *et al.*, 2017). Fe_3O_4 nanoparticles have the ability to provide the desired magnetic, electrical, and mechanical properties to the final composite. It is expected that the reinforcing effect of nano-sized fillers will be noticeable at lower volume fractions than their micron-sized counterparts (Sam *et al.*, 2013).

Several research reported that the electrical and magnetic properties of nanoparticles are dependent on the size and shape of the magnetic materials (Farias-Mancilla *et al.*, 2016; Mustafa & Ruslan, 2016). This size dependence can be leveraged to adjust the electrical, magnetic, and mechanical properties of the composite without changing the particles content and/or type. There is a great potential application in using magnetite nanoparticles as fillers in a composite not only from the multifunctional aspect but also from the ability to adjust the magnetic, electrical, and mechanical properties of a composite by changing the content in the composites (Fayemi *et al.*, 2016; Fakruddin *et al.*, 2012).

Coating Fe_3O_4 nanoparticles with a conductive polymer, such as PAni, has been reported in the literature (Cheng *et al.*, 2010). Fe_3O_4 nanoparticles are relatively stable in atmospheric conditions, but the surface of the particles is susceptible to oxidation.

However, the conductivity of the polymer composite may be decreased with increased of Fe_3O_4 nanoparticles content and consequently, the weight penalty will be minimal (Tayebi *et al.*, 2016). It is also a way to decouple magnetic and electrical properties and allows for a wider range and flexibility in controlling those properties (Masotti & Caporali, 2013). As reported by Khiew *et al.*, (2004) earlier PAni/Fe_3O_4 nanocomposites are used to fabricate multifunctional material for different purposes, polyvinyl alcohol (PVA) can be used as the matrix material, Fe_3O_4 nanoparticles and

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