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DEVELOPMENT AND CHARACTERIZATION OF GRAPHANE POLY(VINYL ALCOHOL) NANOCOMPOSITES

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By

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DEVELOPMENT AND CHARACTERIZATION OF GRAPHANE POLY(VINYL ALCOHOL) NANOCOMPOSITES

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Chairman : Associate Professor Suraya Binti Abdul Rashid, PhD
Institute : Advanced Technology

The present research aims to develop multifunctional nanocomposite material that has the adequate electromagnetic interference (EMI) shielding properties with minimal thickness as well as good mechanical flexibility and particularly was easily processed into films. Graphene nanoplatelets (GNP) with unique extraordinary properties were preferred as reinforcement agent in the multifunctional polymer nanocomposite films development. Strategic combination of composite analytical testing approaches was essential in determining optimum composite material formulation consequently enhanced the maximum properties of nanocomposite film as the GNP dispersed homogeneously in the poly(vinyl alcohol) (PVA) matrix prepared by both solution casting (SC) and solution-impregnated electrospun nanofibrous (SI) methods.

The first objective was to determine the tensile, thermal, and dynamic mechanical properties of resultant nanocomposite having different GNP size and loading content (1, 3, 5, 7wt%) prepared by solution casting (SC) method. Furthermore, second objective was to evaluate the microstructure of various GNP electrospun nanofibrous mat and to determine the thermal and dynamic mechanical properties of GNP nanofibrous mats/PVA (PVA/eGNP) nanocomposite films prepared by solution-impregnated electrospun nanofibrous (SI) fabrication method. The third objective was to compute the dielectric, attenuation and EMI shielding effectiveness (SE) values in the range of microwave frequencies.

In this research, both types of GNP (GNP-M15 and GNP-C750) that incorporated into the PVA have enhanced their tensile strength and modulus of the resultant nanocomposites at low GNP loading but decreased when GNP loading beyond 5wt%. Conversely, the elongation at the break of the nanocomposites decreased with an incorporation GNP content. Additionally, nanocomposite incorporated with 3wt% of GNP C750 grade (43.33MPa) show 13% higher tensile strength compared to M15 grade.

The storage modulus of PVA/GNP nanocomposites prepared by SC that incorporated with C750 and M15 GNP at 3wt% loading increased by 30% and 20% over the pure PVA film sample, respectively. The trend in dynamic mechanical properties (storage modulus) was in excellent agreement with the tensile characteristic. Moreover, the glass transition temperature, (T_g) in which significantly increased (10^oC) was observed attributed to the better interaction of the GNP nanofillers with the PVA matrix. It was observed that the degree of crystallinity evaluated by DSC for the PVA/GNP nanocomposites incorporated with 1wt% of GNP loading was slightly increased (15.5%) compared to pure PVA (13.2%) and this supported with the additional confirmation by the XRD characteristic.

Meanwhile, on the other hand the storage modulus of same GNP loading (3wt%) has shown an enhancement about 50% for the sample prepared by SI method. Furthermore, at the highest GNP loading (7wt%) of PVA/eGNP nanocomposite film has shown a comparable result to the optimum storage modulus (3wt%) obtained from PVA/GNP nanocomposite film. It was also found that the degradation temperature (T_d) of the PVA/GNP nanocomposite was appeared at about 340˚C and it was about 10˚C increment compared to pure PVA.

The PVA/GNP nanocomposites films show an enhancement up to 60% in dielectric properties at microwave frequencies range from 8GHz to 12GHz. The highest EMI SE_{Total} of approximately 7.5 dB was achieved from 7wt% of GNP electrospun nanofibers mat reinforced PVA nanocomposite film which prepared by solution-impregnated electrospun nanofibrous method. These nanocomposite films which exhibited appropriate dielectric constant and attenuate electromagnetic wave due to dielectric losses were promising candidature for various shielding applications by tuning their filler content. The reinforced GNP electrospun nanofibrous have successfully utilized as a scaffold for multifunctional components of the resultant hierarchically organization nanocomposite with enhanced multifunctional properties.

Abstrak tesis yang dikemukakan kepada Senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN DAN PENCIRIAN GRAPHAN POLI(VINIL ALKOHOL) NANO KOMPOSIT

Oleh

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Penyelidikan ini bertujuan untuk membangunkan bahan nanokomposit termaju pelbagai fungsi yang mempunyai sifat pemerisaian gangguan elektromagnet (EMI) yang mencukupi dengan ketebalan minimum serta kelenturan mekanik yang baik dan terutamanya boleh diproses dengan mudah untuk membentuk filem. Nanoplatelet graphen dengan ciri yang unik adalah digemari sebagai agen pengukuhan dalam pembangunan nanokomposit polimer pelbagai fungsi. Gabungan strategik pendekatan analisis komposit adalah penting dalam menentukan rumusan bahan komposit optimum yang seterusnya meningkatkan sifat maksimum filem nanokomposit kerana GNP tersebar secara homogen dalam matriks poli (vinil alkohol) (PVA) yang disediakan oleh kedua-dua kaedah tuangan larutan (SC) dan kaedah elektrospun nanofibrous pengisitepuan-larutan (SI).

Objektif pertama adalah menentukan tegangan, terma, dan sifat mekanik dinamik nanokomposit yang terhasil yang mempunyai saiz GNP dan kandungan pemuatan (1, 3, 5, 7wt%) yang berbeza yang disediakan dengan SC. Selain itu, matlamat kedua adalah untuk menilai struktur mikro pelbagai kepingan nanofibrous elektrospun GNP dan menentukan sifat termal dan mekanik dinamik bagi filem nanokomposit kepingan nanofibrous GNP/PVA (PVA/eGNP) yang disediakan oleh kaedah SI. Objektif ketiga adalah untuk mengira nilai dielektrik, keberkesanan penyusutan dan pemerisaian EMI (SE) dalam julat frekuensi gelombang mikro.

Dalam kajian ini, kedua-dua jenis GNP (GNP-M15 dan GNP-C750) yang dimasukkan ke dalam PVA telah meningkatkan kekuatan tegangan dan modulus nanokomposit yang dihasilkan pada muatan GNP yang rendah tetapi menurun apabila muatan GNP melebihi 5wt%. Sebaliknya, pemanjangan pada pecahan nanokomposit menurun dengan kandungan GNP yang diperbadankan. Selain itu, nanokomposit yang digabungkan dengan 3wt% gred GNP C750 (43.33MPa) menunjukkan kekuatan tegangan 13% lebih tinggi berbanding gred M15.

Modulus storan nanokomposit PVA/GNP yang disediakan oleh SC yang mengandungi 3wt% muatan GNP C750 dan M15 meningkat sebanyak 30% dan 20% melebihi daripada sampel filem PVA tulen, masing-masing. Tren dalam sifat mekanik dinamik (modulus storan) adalah dalam persetujuan yang sangat baik dengan sifat tegangan. Selain itu, suhu peralihan kaca, (T_g) yang meningkat dengan ketara (10^0C) diperhatikan disebabkan oleh interaksi yang lebih baik dari nanofiller GNP dengan matriks PVA. Telah diperhatikan bahawa darjah kehabluran yang dinilai oleh DSC untuk nanokomposit PVA/GNP yang mengandungi 1wt% muatan GNP sedikit meningkat (15.5%) berbanding dengan PVA tulen (13.2%) dan ini disokong dengan pengesahan tambahan oleh ciri XRD.

Sementara itu, sebaliknya modulus storan muatan GNP yang sama (3wt%) telah menunjukkan peningkatan sebanyak 50% untuk sampel yang disediakan oleh kaedah SI. Tambahan pula, filem nanokomposit PVA/eGNP pada muatan GNP tertinggi (7wt%) telah menunjukkan hasil yang setanding dengan modulus storan optimum (3wt%) yang diperoleh daripada filem nanokomposit PVA/GNP. Suhu degradasi (T_d) nanokomposit PVA/GNP juga didapati muncul pada kira-kira 340˚C dan ia adalah kira-kira kenaikan 10˚C berbanding dengan PVA tulen.

Filem nanokomposit PVA/GNP menunjukkan peningkatan sehingga 60% dalam sifat dielektrik pada frekuensi gelombang mikro dari 8GHz hingga 12GHz. EMI tertinggi SE_{Total} kira-kira 7.5 dB dicapai daripada filem nanokomposit PVA bertetulang kepingan nanofiber elektrospun GNP pada muatan 7wt% yang disediakan oleh kaedah elektrospun nanofibrous pengisitepuan-larutan. Filem nanokomposit ini yang mempamerkan pemalar dielektrik yang sesuai dan penyusutan gelombang elektromagnetik kerana kehilangan dielektrik adalah calon yang baik untuk pelbagai aplikasi perisai dengan penalaan muatan pengisi. Elektrospun nanofibrous bertetulang graphene telah berjaya digunakan sebagai perancah untuk komponen pelbagai fungsi nanokomposit hasil daripada organisasi hierarki nanokomposit dengan menunjukkan peningkatan ciri-ciri pelbagai fungsi.

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This thesis was submitted to the Senate of the Universiti Putra Malaysia and has been accepted as fulfillment of the requirement for the degree of Doctor of Philosophy. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ADF-STEM	annular dark-field scanning transmission electron microscopy
AFM	atomic force microscopy
AR	aspect ratio
CA	cellulose acetate
CB	carbon black
CMT	combine motor and transducer
CNF	carbon nanofibers
CVD	chemical vapor deposition
DMA	dynamic mechanical analysis
DMF	dimethylformamide
DP	degree of polymerization
EG	expanded graphite
EM	electromagnetic
EMC	electromagnetic compatibility
EMI	electromagnetic interference
EMW	electromagnetic wave
ESD	electrostatic discharges
f-(PVA)	PVA functionalised GO flakes
f-graphene	L-phenylalanine-functionalized graphene
FLG	few-layer graphene
FRPC	fiber-reinforced polymer nanocomposites
GICs	graphite intercalation compounds
GNP	graphene nanoplatelet

GO	graphene oxide
HD	hydrolysis
HPOG	highly oriented pyrolytic graphite
LEEM	low-energy electron microscopy
MIR	multiple internal reflection
Mw	molecular weight
NMP	N-methylpyrrolidone
PC	propylene carbonate
PET	polyethylene terephthalate
PNC	polymer nanocomposite
PVA	polyvinyl alcohol
PVA-g-GO	PVA-grafted graphene oxide
PVAc	polyvinyl acetate
RFID	radio frequency identification
rGO	reduced graphene oxide
SAED	selected area electron diffraction
sccm	standard cubic centimeters per minute
SE	shielding effectiveness
SEA	absorption shielding efficiency
SEM	multiple reflection shielding efficiency
SET	total shielding effectiveness
SFRC	short-fiber fortified composites
SHF	super-high frequency
SLG	single-layer graphene
SNA	scalar network analyser

TEM	transmission electron microscopy
Tg	transition temperature
TGA	thermogravimetric analysis
UHF	ultra-high frequency
UHV	ultra-high vacuum
VNA	vector network analyser
WAIC	Wireless Avionic Intra-Communications Specifications
XRD	X-ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Overview

In a time that is buzzing with technological development, new and more sophisticated classes of smart materials are constantly being developed. Given the amazing complexity and successful designs of natural materials that biology is capable of producing, the modern materials engineer or scientist are creating an incredible array of materials which may emulate biological system with the capability to select and execute specific functions intelligently and respond to variability in the environment. “Smart” multifunctional properties are the key component of the next generation advanced materials, whereby the materials have the ability to go beyond their existing capabilities, including adaptation to environmental cues, the ability to dynamically switch between different material states, and self-healing. In parallel with the extensive growth in modern gigahertz (GHz) electronic systems and multifunctional telecommunication devices with greatly enhanced data transfer speeds that operate at higher frequencies has raised the electromagnetic (EM) pollution to a level never attained before (Qin et al, 2012). Several studies have reported the hazardous effect of EM waves which include the increase risks of health issues related to skin problems, cancer, heart problems, headache and several other minor and acute diseases (Sowmya et al., 2018).

Nanotechnology has greatly contributed to the development of bioinspired advance nanocomposites that produced by hierarchically organized nano-components whereby each component exhibits a unique and greater functional capability and performance (Qian et al., 2010). Recently, tuneable nanofibers is explored as a scaffold for multi-functional components of the hierarchically organization (Wee-Eong et al.,2009). Consequently, the visions of developing material that responds to environmental changes would become a reality (Adnan et al., 2015).

In this research, graphene nanoplatelets (GNP) with retaining the single-layer graphene (SLG) extraordinary properties were preferred nanomaterial due to their economically feasible and have highly potential to form graphene nanofibrous mats by electrospinning fabrication method. These high aspect ratios of electrospun nanofibers with finite length and the absence of fiber edges (ends) that act as obvious stress concentration zones have played an important role in designing the qualified conductive and good mechanical flexibility of nanoreinforcement (Pillay et al., 2013). Moreover, the superior interfacial bonding strength between matrices to the nanoreinforcements can dramatically improve due to nanofibrous high specific surface area properties

(Zheng-Ming at al., 2003). Therefore, graphene derivatives itself have various extraordinary capabilities can be incorporated into the nanofibers to form the first level of the hierarchical organization. In the next level, the graphene based nanofibrous mat were impregnated within a matrix to become the reinforcement appliance as well as to be used to control and manipulate electromagnetic radiation over a wide range of wavelengths (Qin et al, 2012).

1.2 Problem Statement

The presence of numerous exceptional properties owing by graphene nanoplatelets (GNP) in the commercial polymers that lead to significant reinforcement has contributed to a vast amount of research focused on the development of graphene-based multifunctional polymer nanocomposite materials (Dimitrios et al., 2017). However, there were only few of the researcher work on incorporated the GNP into high mechanical flexibility polymer matrices films.

There is high interest and motivation to develop multifunctional polymer nanocomposite films with minimal thickness as well as good mechanical flexibility for electronic component application as it is essential due to the global demand and staggeringly rapid advances in massive development of high speed Gigahertz (GHz) wireless technology, as well as the on-going miniaturization of electronic devices evolution (Sowmya et al., 2018).

This approach can be made possible with the incorporation of GNP into the light weight and superior mechanical flexibility polyvinyl alcohol (PVA) matrix as reinforcement agent for the first time. There is much need to evaluate and understand the fundamental behaviour on incorporating various GNP size and loading into PVA as well as its effect on tensile, thermal, crystallinity and dynamic mechanical properties for their potential application in EMI shielding industry.

The applications of dynamic mechanical analysis (DMA) show extreme importance in the field of nanocomposite and hence it is potentially useful tool for designing materials in films processing applications. Furthermore, DMA provide remarkable insight into the different chemistries associated with film formation of the solvent-based and water dispersible formulations. Moreover, DMA offers an important test method in evaluation of the interfacial bonding in the temperature and strain rate ranges of interest for polymer nanocomposite applications. In addition, using a strategic combination of polymer crystallization analytical testing approaches is essential for improving fabrication processes, optimizing material properties in developing new polymer composite films and obtaining their details failure analysis. Additionally, these analyses are very useful in material formulation and quality control procedure.

The EMI shielding specialists are facing new challenges to figure out new ways to use less space, thinner and lighter materials for creating shielding films with tighter tolerances such as strong absorption and weak secondary reflection of EMI pollution. The general trend for composites with different carbonaceous nanofillers (Qin and Brosseau, 2013; Jean et al., 2013; Adohi et al., 2010) shows that EMI shielding effectiveness (SE) was enhanced by incorporating higher filler loadings (>20wt%) that only demonstrated reflection-dominant mechanism. However, this condition is economically not feasible and not viable for films processing.

In this research, for the first time the low loading of graphene electrospun nanofibrous possess high aspect ratio has been used as reinforcement agent into flexible PVA matrix in the hierarchically organized nanocomposite materials. Production of this multifunctional nanocomposite material that have an equivalent electromagnetic interference (EMI) shielding properties with minimal thickness as well as good mechanical flexibility were highly demand, particularly it was easily processed into films.

1.3 Objectives

The aim of this research focuses on design and constructs a novel formulation of a multifunctional advanced nanocomposite material by combining the light weight and superior mechanical flexibility of polyvinyl alcohol (PVA) with the extraordinary properties of the graphene nanoplatelets (GNP) at far lower reinforcement concentrations. The PVA/GNP nanocomposites films that fabricated by both solution casting and solution-impregnation electrospun nanofibrous processing methods which then further characterise and evaluate in term of microstructural features, tensile, dynamic mechanical, thermal and dielectric properties in detail for the first time. The deep investigation on the effect of incorporating various GNP types and content on the PVA/GNP nanocomposites is taking into account for the potential application in EMI shielding industry.

In order to explore these process-structure-property relationships, the following objectives have to be carried out:

To evaluate the effect of GNP filler size (M15 and C750 grade) and loading content (1,3,5,7wt%) on the tensile, thermal, crystallinity and dynamic mechanical properties of GNP/PVA nanocomposite films prepared by using solution casting method.

To evaluate the effect of impregnating GNP electrospun nanofibrous mats on the thermal and dynamic mechanical properties of GNP nanofibrous mats/PVA nanocomposite films prepared by solution-impregnation electrospun nanofibrous prosessing method.

To determine the attenuation performance of graphene-based poly(vinyl alcohol) nanocomposite for Electromagnetic Interference (EMI) Shielding application at microwave frequencies range .

1.4 Scope of Study

PVA with excellent chemical resistance, physical properties, biodegradability and light weight features was used as the matrices. The incorporation of GNP which owing multi extraordinary properties into the PVA has offers new opportunities in tailoring the matrices properties as well as introduce new properties to the resultant nanocomposite. Moreover, both of these polymer solution cast films technique could replace traditional film extrusion processing method thus deliver a better cost effectiveness as well as high quality films with superior mechanical, thermal and electrical film properties. This obtained by their capability of processing condition at low temperatures, which suitable for thermally activated films or incorporating temperature-sensitive active ingredients. Furthermore, it has a facility for producing high-temperature resistant films from thermoplastic of soluble raw materials. The ability for single pass manufacturing of multi-layer films and quicker changeovers for platforms with many part numbers having differentiated formulation has make this fabrication method suitable for a large scale production. The scope of study for each objective was performed as follows:

1. The graphene based PVA nanocomposites in the form of films were prepared by solution casting processing methods. The tensile, thermal, crystallinity and dynamic mechanical properties of resultant nanocomposite can be tuned by the incorporation of different GNP filler size and loading. Furthermore, the structural fractographic characterization of PVA/GNP nanocomposites is crucial to support the fundamental understanding in the structure to property relationship mechanism.
2. In solution-impregnation electrospun nanofibrous method, GNP were incorporated into the nanofibers by using elctrospinning technology to form the first level of the hierarchical organization. In the next level, the graphene based nanofibrous mat was impregnated within a PVA matrix. The effect of electrospinning variables and parameter on the morphology of GNP electrospun nano fibrous mat was evaluated. Furthermore, the impregnation of different GNP nanofibrous mats morphological structure and size can be used to tune the thermal and dynamic mechanical properties of GNP nanofibrous mats/PVA nanocomposite films.

3. The graphene-based poly(vinyl alcohol) nanocomposite films attenuation performance was evaluated in microwave frequencies range for EMI shielding application.Firstly, the dielectric properties of graphene-based poly(vinyl alcohol) nanocomposite at X-band frequency range were determined. Next, the Scattering Parameters [S] of graphene-based poly(vinyl alcohol) nanocomposite were determined by using vector network analyser (VNA). Finally, the attenuation and EMI shielding effectiveness (SE) value of graphene-based poly(vinyl alcohol) nanocomposite at X-band frequency range were computed.

In general, the evaluation of graphene-based poly(vinyl alcohol) nanocomposite films were tensile properties (tensile strength, modulus and elongation at break), thermal properties (thermogravimetric and differential scanning calorimetric analysis), morphology (scanning electron microscopy and field emission scanning electron microscopy), crystallinity (X-ray diffraction), dielectric and EMI shielding effectiveness (waveguided vector network analyser).

1.5 Significance of Research

Electromagnetic interference (EMI), a novel kind of pollution is receiving a lot of scientific attention all over the world due to their capability to produce deleterious effects to the human health. There is an urgency to develop advanced and sophisticated classes of EMI shielding materials which can satisfy the need of next-generation portable equipment and wearable devices. Executing this strategy requires the progress in science, technology and business because novel materials often require complementary new requirements on material properties, new demands on performance and cost, and also new markets. Specifically with every advance in electronic technology such as GPS, 4G/LTE, RFID and more intricate cell phones, it comes with a new demand for EMI shielding mechanism and strategies. It has been noted that the biggest obstacles for the EMI shielding industry are size, flexible design and cost. However, with the used of right materials along with an optimum formulation of materials design together with effective manufacturing procedure, a successful EMI shielding can be achieved across even the most complex applications. Thus having adequate conductivities nevertheless limiting the fraction of GNP by compositing these conductive nanofiller with low dielectric constant matrices (PVA) being a smart strategy for restraining reflection and enhance absorption as the electromagnetic wave (EMW) can perfectly transmitted into the shielding material due to its low surface impedance. The enhancement of EMI shielding in these structures has been frequently attributed to favoured re-reflection and subsequent dissipation of the absorbed portion of the wave in the hierarchically organized nanocomposite material. Finally, few advantages of the resulting film from polymer solution cast fabrication technique were broader range of film thickness with greater film flatness and thickness uniformity as well as the absence of typical extrusion process lubricants as compared with traditional manufacturing procedure.

1.6 Organization of the thesis

Chapter 1 is the introduction of the thesis which given general overview on the global issues of pollution created by electromagnetic interference (EMI) due to high speed and frequencies demanded from the electronic and industry evolutionary. Besides, the problem statement regarding the conventional EMI shielding materials is mentioned together with the objectives of the study.

In chapter 2, a comprehensive review of literatures on related topic toward this research such as the polymer nanocomposite (PNC), the production and properties of graphene and its derivative, the polymeric matrices in details, electrospining technology and the fundamental of EMI shielding design mechanism and characterization.

Chapter 3 is the methodology section which discusses the methods and materials used in the research. Chapter 4 presented the results followed by discussion on the obtained result. Development of optimum mutlifunctional graphene based PVA nanocomposites also discussed in this section. The last chapter is the overall conclusion of the thesis and recommendation for future research based on the understanding and knowledge generated in the present study.

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PUBLICATION

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