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PRODUCTION OF POLYCARBONATE AND MULTI -WALLED CARBON NANOTUBES NANOCOMPOSITES AT LOW FILLER LOADINGS

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By

BUTHAINAH ALI ABED AL-TIMIMI

Thesis Submitted to the School of Graduated Studies, Universiti Putra Malaysia, in Fulfillment of the Requirement for the Degree of Master of Science

July 2015

DEDICATIONS

I would like to dedicate my thesis to my great family. A special feeling of gratitude

To

My loving parents, Ali and Zahra

Thank you for unconditional support with my studies. I am honored to have you as my parents.

My supportive brothers (Tahseen , Ahmed , Ammar, Sarmed , Jassim)

You were always there, as shields protecting me from the virulent life

My beloved sisters (Huda , Sajida , Baidaa)

You were always there to comfort me in a time of need.

My brother's wifes (Bushra , Huda , Hadeel)

My brothers kids (Ali , Yazin , Fatima , Abid alrhman , Maram)

Thank you my great family for believing in me and giving me a chance to prove and improve myself through all my walks life. Please do not ever change.

I would like to dedicate my thesis to the wonderful man

Wadhah

Your constant support and encouragement made this possible
I have learned so much from you and I strive to acquire the qualities that you possess.

I would like to dedicate my thesis to my spiritual friend

Wafaa

Thank you for allowing me to be your friend. Your strength and protection became my very own courage.

Abstract of thesis presented to the Senate of Universiti Putra Malaysia in fulfillment of the requirement for the Degree of Master of Science.

PRODUCTION OF POLYCARBONATE AND MULTI -WALLED CARBON NANOTUBES NANOCOMPOSITES AT LOW FILLER LOADINGS

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BUTHAINAH ALI ABED AL-TIMIMI

July 2015

Chairman: Dayang Radiah Binti Awang Biak, PhD
Faculty : Engineering

Polymer nanocomposites are the materials of the future, which will change the course of all industries, since polymers today find their applications in every industry. Multi-walled carbon nanotubes (MWCNTs) had been widely used for filler for various nanocomposites. Thus, this research focuses on the fabrication of polycarbonate (PC) / multi-walled carbon nanotubes (MWCNTs) nanocomposites. The objectives of the work were to study the physico-chemical properties of multi-walled carbon nanotubes produced by the modified Staudenmaier method. The mechanical, thermal, electrical and chemical properties of nanocomposites produced with different filler loadings were also assessed. The gasoline absorption capabilities of the nanocomposites were also conducted.

The modified Staudenmaier method was used to produce the multi-walled carbon nanotubes (MWCNTs) used in this work. The produced carbon nanotubes were characterized by scanning electron microscopy (SEM), transmission electron microscopy (TEM), X-ray diffraction (XRD), thermogravimetric analysis (TGA) and Raman analysis. The MWCNTs were then mixed with the PC to produce PC/ MWCNTs nanocomposites. The operating parameters of the mixer were varied for best operating conditions to produce the nanocomposites based on tensile data of the products. The effects of filler loading, ranges from 0–1 wt.% , on the properties of the nanocomposites were also investigated. The mechanical, thermal, and electrical properties as well as chemical resistance performance of the nanocomposites were evaluated. Multivariate analysis of variance (MANOVA) was applied to assess the significance improvement that had been introduced in this work.

The produced multi-walled carbon nanotubes (MWCNTs) have a diameter of 13.1nm. The yield is approximately 10% of the carbon source (graphite).

The PC/MWCNTs nanocomposites were produced at mixing temperature of 245^{o}C with rotor speed of 55 rpm and the compounding time of 12 minutes. The density of the nanocomposites was improved in the range of 0.77% and 2.05% with different filler loadings. Increasing the content of MWCNTs will directly decrease the density of the material. The improvement in Young's modulus values ranged between 2.80% and 43.26%,whilst the thermal analysis data showed that the highest shift of degradation temperature onset was from 458.96^{o}C to 493.77^{o}C. The glass transition temperature (T_g) of the nanocomposites was also increased from 147^{o}C to 153.70^{o}C. Adding 1 wt.% of MWCNTs into the PC as fillers had increased the thermal diffusivity of nanocomposites to 35.46 % and improved the thermal conductivity and electrical conductivity of the PC/MWCNTs nanocomposites to 1.22 W. m^{-1}. K^{-1} and 1.02 x 10^{-8} S/cm respectively. The change in gasoline absorption resistance for the nanocomposites was found to be in the range of 10.13 % to 62.10 %. This indicates that the polymer did not absorb gasoline as the amount of filler increased. The MANOVA results indicate the correlation between the amount of filler used with the thermal, mechanical and electrical properties as well as the chemical resistance performance of the nanocomposites.

The work contributes knowledge on the effects of adding low filler (MWCNTs) loadings on the properties of polymer; in particular, PC/MWCNTs nanocomposites. It shows the potentials of PC/MWCNTs as suitable material for gasoline storage due to its high gasoline absorption resistance and can be a material of choice in the future.

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

PENGHASILAN NANOKOMPOSIT POLIKARBONAT DAN NANOTIUB KARBON BERDINDING PELBAGAI PADA MUATAN RENDAH

Oleh

BUTHAINAH ALI ABED AL-TIMIMI

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Nanokomposit polimer adalah bahan masa akan datang, yang mampu mengubah perjalanan semua industri. Ini kerana penemuan penggunaan polimer semakin meluas dalam setiap industri. Nanotiub karbon berdinding pelbagai (MWCNTs) telah digunakan secara meluas sebagai pengisi untuk pelbagai nanokomposit. Oleh itu, kajian ini memberi tumpuan kepada pembuatan nanokomposit polikarbonat (PC) / Nanotiub karbon berdinding pelbagai (MWCNTs). Objektif penyelidikan ini adalah untuk mengkaji sifat-sifat fiziko - kimia nanotiub karbon berdinding pelbagai yang dihasilkan melalui kaedah Staudenmaier yang telah diubah suai. Sifat-sifat mekanik , haba , elektrik dan kimia nanokomposit dihasilkan menggunakan beban pengisi yang berbeza juga dinilai. Keupayaan penyerapan petrol untuk nanokomposit juga dilaksanakan.

Kaedah Staudenmaier yang telah diubah suai digunakan untuk menghasilkan nanotiub karbon berdinding pelbagai (MWCNTs) yang digunakan dalam kajian ini. Nanotiub karbon yang dihasilkan dicirikan mengguna mikroskopi imbasan elektron (SEM), mikroskopi transmisi elektron (TEM), pembelauan sinar-X (XRD), analisis Termogravimetri (TGA) dan analisis Raman. MWCNTs kemudiannya dicampur dengan PC untuk menghasilkan PC/MWCNTs nanokomposit. Parameter operasi pembancuh telah diubah bagi mendapatkan keadaan operasi yang terbaik untuk menghasilkan nanokomposit berdasarkan data tegangan produk. Kesan kandungan pengisi, (antara 0-1% berat) keatas sifat nanokomposit juga disiasat. Ciri analisis mekanik, haba, dan elektrik serta prestasi rintangan kimia nanokomposit telah dinilai. Kaedah pelbagai varian telah diguna untuk menilai kesan pembaikan yang telah di laksanakan.

Nanotiub karbon yang dihasilkan adalah berbilang berdinding (MWCNTs) dan ber diameter 13.1 nm. Penghasilan produk adalah 10% daripada sumber karbon (grafit) yang digunakan.

Nanokomposit PC/MWCNTs telah dihasilkan pada suhu 245^{o}C dengan kelajuan pemutar 55 rpm dan masa pengkompaunan selama 12 minit. Ketumpatan nanokomposit yang dihasilkan dengan kandungan pengisi yang berbeza telah ditambahbaik antara 0.77% - 2.05%. Ini menunjukkan bahawa peningkatan kandungan MWCNTs secara langsung akan mengurangkan ketumpatan bahan. Peningkatan dalam nilai modulus Young adalah di antara 2.80% dan 43.26%, manakala data analisis terma menunjukkan peralihan tertinggi bermulanya suhu degradasi adalah dari 458.96^{o}C ke 493.77^{o}C. Suhu peralihan kaca (T_g) nanokomposit juga meningkat daripada 147^{o}C ke 153.70^{o}C. Penambahan 1% berat MWCNTs ke dalam PC sebagai pengisi telah meningkatkan kebolehan kemeresapan terma nanokomposit kepada 35.46% dan memperbaiki sifat keberaliran haba PC/MWCNTs nanokomposit kepada 1.22 W. m^{-1}. K^{-1} dan kekonduksian elektrik kepada 1.02 x 10^{-8} S/cm. Perubahan dalam rintangan untuk penyerapan petrol untuk nanokomposit didapati dalam julat 10.13% hingg 62.10% . Ini menunjukkan bahawa polimer tidak menyerap petrol apabila jumlah kandungan pengisi meningkat. Keputusan MANOVA menunjukkan hubungan antara kandungan pengisi dan sifat haba, mekanikal dan elektrik serta prestasi rintangan kimia nanokomposit.

Kajian ini menyumbang pengetahuan berkaitan kesan muatan rendah pengisi ke atas sifat polimer khususnya PC/MWCNTs. Ia menunjukkan potensi PC/MWCNTs sebagai bahan yang sesuai untuk penyimpanan petrol kerana rintangan penyerapan petrol yang tinggi dan boleh menjadi bahan pilihan yang utama pada masa akan datang.

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I certify that a Thesis Examination Committee has met on 10 July 2015 to conduct the final examination of Buthainah Ali Abed on her thesis entitled "Production of Polycarbonate and Multi-Walled Carbon Nanotubes Nanocomposites at Low Filler Loadings" in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded the Master of Science.

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

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I hereby confirm that:

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LIST OF ABBREVIATIONS

CNTs	Carbon Nanotubes
PC	Polycarbonate
MWCNTs	Multi-Walled Carbon Nanotubes
SWCNTs	Single-Walled Carbon Nanotubes
DWCNTs	Double-Walled Carbon Nanotubes
MNC	Metal Matrix Nanocomposites
CNC	Ceramic Matrix Nanocomposites
PNC	Polymer Matrix Nanocomposites
EDP	Electrostatic Discharge Protection
EMS	Electromagnetic Shielding
Wt.%	Weight Percent of Filler
Vol.%	Volume Percent of Filler
TEM	Transmission Electron Microscopy
T_g	Glass Transition Temperature(K)
UTS	Ultimate Tensile Strength
l_{sp}/d	Mesoscopic Shape Factor
l_{sp}	Static Bending Persistence Length
d	Outer Diameter of Carbon Nanotubes (cm or nm)
EMI SE	Electromagnetic Interference Shielding Effectiveness

DMA	Dynamic Mechanical Analysis
DSC	Differential Scanning Calorimetric
$p_c s$	Electrical Percolation Thresholds
tan δ	Loss Tangent
V	Voltage
A	Electrical Current
DC	Direct Current(A)
^oC	Temperature(centigrade)
CVD	Chemical Vapor Deposition
CCVD	Catalytic Chemical Vapor Deposition
MPECVD	Microwave Plasma-Chemical Vapor Deposition
HFCVD	Hot-Filament-Chemical Vapor Deposition
RF-CVD	Radiofrequency-Chemical Vapor Deposition
ST	Staudenmaier Method
HO	Hofmann Method
HU	Hummers Method
BPA	2, 2-bis-(4-hydroxyphenyl) propane
PS	Polystyrene
PMMA	Poly (Methyl Methacrylate)
HDPE	High-Density Polyethylene

PA6	Polyamide-6
PA66	Polyamide-66
PBT	Polybutylene Terephthalate
CB	Carbon Black
TGA	Thermogravimetric Analysis
T_{onset}	Onset of Degradation Temperature
T_{max}	Temperature at Maximum Weight Loss
PP	Polypropylene
M_t	The Relative Weight Gain
t	Time
M_∞	The Equilibrium Relative Weight Gain
k	Constant
n	Constant
D	Diffusion Coefficient
b	Thickness of Sample
K	The Slope of The Linear Portion of The Plot of M_t versus $t^{1/2}$
ASTM	American Society for Testing and Materials
SEM	Scanning Electron Microscopy
XRD	X-Ray Diffraction
rpm	Roller Rotor Speed

ρ	Specimen Density (kg/m^3)
m_{dry}	The Dry Mass (kg)
m_{wet}	The Submerged Mass (kg)
ρ_{water}	The Density of The Test Water (kg/m^3)
E'	Dynamic Storage Modulus
tan δ	Damping Factor
α	Thermal Diffusivity($m^2.s^{-1}$)
λ	Thermal Conductivity ($W.\ m^{-1}.\ K^{-1}$)
C_p	Specific Heat Capacity ($J.\ kg^{-1}.\ K^{-1}$)
w_1	Initial Weight (g)
w_2	Final Weight (g)
MANOVA	Two Way Multivariate Analysis of Variance
IV	Independent Variable
DVs	Dependent Variables
nm	Nanometer
DTG	Derivative Weight Loss
ID/IG	The Intensity Ratio of the G–band and D–Band
OV	Overall performance
μ	Mean
H	Hypothesis

A	Wilks' lambda

CHAPTER 1

INTRODUCTION

1.1 Background of Study

The modern applications of plastics and their associate composites can be regarded as universal and wide-ranging, from basic household wares and appliances to equipments used in avionics and aviations right to the fields of military and defence (Michael *et al*.,2013; Pfaendner, 2010). Their exclusive properties such as light in weight, economical, and the fact that they are easy to process made them an untenable choice in many everyday applications compared to other metallic materials like steels and copper (Chung, 2010).

Researchers had unraveled multifarious methods to launch further into the use of plastics (Fried, 2003). This includes formulating or forming new composite with new or improved properties that have proven to be better than the properties of the parent materials. Two decades have passed, and scholars have devoted their time on producing materials (nanomaterials) that could be used to enhance the strength of polymers; and the subsequent distinctive manifestations of carbon fiber (Guduri & Luyt, 2006; Sushant *et al*., 2008), fiberglass (Yung-Kuang, 2006; Gautam *et al*., 2010) and aramid (Sarawut *et al*., 2012) were birthed.

The size, length and the orientation of such fiber have become integral components that can determine the properties of the composite created (Wool & Sun, 2005). The more recent body of literature on nanotechnology has given emphasis on the incorporation of MWCNTs into polymers to produce polymer nanocomposites deemed appropriate for high value-added and customized applications (Kenan *et al*., 2013).

The production of polymer nanocomposites and any manipulations thereof are in the pursuit of developing the research field in the advanced material sector. Researchers have made improvements on the properties of typical plastic resin by adding filler materials, *e.g.* carbon nanotubes (Anselm *et al*., 2014; Maiti *et al*., 2013; Via *et al*., 2012; Mehdi, 2013; Jogi *et al*., 2012), graphene oxide (Lee *et al*., 2010), strands of carbon fibers (Sushant *et al*.,2008; Guduri & Luyt, 2006), and nanoparticles(Shilpa *et al*., 2013), merely to boost their potential.

Polymer nanocomposites comprise of fillers with the dimension on the nano-scale, (Paul & Robeson, 2008). The filler addition can adjust the mechanical, electrical, thermal and chemical resistance properties of the base resin (Pham *et al*.,2008; Mi *et al*.,2009; King *et al*.,2010; Abbasi *et al*.,2010; Jansen,2011; Ayman,2012; Choi & Ryu,2013; Rabiatul & Kamal,2014; Shailaja *et al*.,2014; Jiaxi *et al*.,2014).

The preparation methods also have some striking influences on the composite material's morphology and thermal properties. The addition of fillers may also leave an impact on the viscosity (Anselm *et al*., 2014) as well as the glass transition temperature, T_g, of the polymer (Jogi *et al*., 2012). This will give a direct effect on the conversion temperatures and lead to a situation where the polymer nanocomposites are irrecoverable.

The processing stages pose the main challenge to the researcher and the industrial players seeking to make a uniform dispersion onto the polymer matrix (Brooks, 2013; Kenan *et al*., 2013; Marcin, 2014). The typical effect of non-homogenized dispersion of fillers onto the matrix includes inconsistent properties and the characteristics of the polymer nanocomposites. This will have some primary direct effects on the end-use products, further causing them to fail at random (Via *et al*., 2012; Maiti *et al*., 2013; Anselm *et al*., 2014).

Polycarbonate is a common thermoplastic possessing exclusive properties, simultaneously rendering it an unbeatable contender for various end-use applications. Having the qualities that include glass-like transparency, good impact resistance, high heat tolerance, dimensionally stable, outstanding optical properties, great colourability, good electrical properties, balanced mechanical performance and ease of processing (Gallucci & Derudder, 2009), the polymer is predominantly utilized to yield composites.

There is an undeniable pressing, if not increasing, need to manufacture smart and strong materials. Therefore, it is regarded as integral to improve the properties of plastics or natural materials using new and advanced materials. Nanosized materials, for example carbon nanotubes, graphene and graphene oxide or any other types of nanoparticles, had been subjected to a synthesis and their functionalities applied and exploited. With their special sizes, *i.e*. nanosize, small manipulations will directly generate a high impact.

Nonetheless, mixing nanomaterials to polymer can pose a great challenge. The nanomaterials can easily agglomerate or the polymers might not be fully melted, making the blend inhomogeneous, and thus the properties of the blend do not really depict the exact properties. For instance, a work on the production of Polycarbonate /MWCNTS composite utilizing melt blending technique had been conducted where a good dispersion with the improved electrical conductivity was accounted for a loading level of 2 wt. % of MWCNTs. Nonetheless, the high loading and good dispersion had been lacking in the mechanical properties, glass transition temperature (T_g) and chemical resistance of the nanocomposites (Sathpathy *et al*., 2007).

This study will dwell into the inter-relation and inter-dependence of the Young's modulus, density, specific heat, thermal diffusivity, thermal conductivity, glass transition temperature (T_g), the electrical conductivity and the chemical diffusivity capacity of the produced polymer with the loading amount of MWCNTs used as the nanocomposite filler.

1.2 Problem Statement

Since the pioneering research of MWCNTs in the early 1990s, a significant progress has been made in their synthesis. Considering the wide range of applications and the increasing demands of MWCNTs in various fields, the superb mechanical, electrical and thermal properties of MWCNTs suggest their increasing potential as fillers for a large variety of polymer nanocomposites, where a high purity and low filler loading are important considerations. If the chemical properties of the nanocomposites are the main interest, then contamination of other elements besides the polymer and the filler themselves are not desirable. Therefore, it is important to choose a suitable synthesis method to produce the filler, *i.e.* MWCNTs, which did not utilize metal catalyst in its reaction.

Despite utilizing the amazing properties of MWCNTs based polymer nanocomposites, the easy and inexpensive method to produce high purity MWCNTs is still a great challenge. The most common methods to produce carbon nanotubes are arc-discharge, laser ablation and chemical vapor deposition (Ali *et al.*, 2014). However, the requirements of high synthesis temperature and pressure, used of metal based catalysts, complicated processing steps, longer synthesis period or expensive costs may still be the major drawbacks that arise from the above mentioned techniques. Therefore, it is necessary and meaningful to explore new processes for the preparation of MWCNTs, which could overcome the disadvantages of these techniques and is crucially important to the future application of MWCNTs based polymer nanocomposites. The physico-chemical data of MWCNTs produced via the modified Staudenmaier method is also still lacking. Thus, getting information on these properties will assist in understanding the behaviors of nanocomposites that utilized the MWCNTs as fillers.

The main problem that had propelled the research efforts to place emphasis on the polymer/ MWCNTs nanocomposites development is the search for lighter materials with a better performance to serve in various applications. Today, polycarbonate (PC) stands to be one of the most commonly used plastic materials. Nevertheless, polycarbonate has still yet to be explored in detail to enhance its overall performance through the low filler loading of MWCNTs. In addition, the utilization of pure MWCNTs which are metal free as filler reinforcement in nanocomposites industries is quite limited due to the high cost of nanofiller (Choudhary *et al.*, 2014). Most works on PC/MWCNTs nanocomposites utilized high loadings of MWCNTs, *i.e.* in the range of (Jindal *et al.*, 2014; King *et al.*,2010; Pötschke *et al.*, 2012).

Moreover, the typical properties that were investigated by researchers when producing PC/MWCNTs are the electrical properties and/or tensile strength only. The chemical diffusivity, thermal diffusivity, thermal conductivity, glass transition temperature(T_g) and Young's modulus properties which strive as the vital performance criteria for nanocomposites especially with low filler loadings were lacking, or only partially reported in the established body of literatures (Mahmoodi *et al.*, 2013; King *et al.*, 2012; Tarek *et al.*, 2014; Maiti *et al.*, 2013).

This work will attempt to investigate the properties of PC/MWCNTs using MWCNTs produced via modified Staudenmaier method. By utilizing the in house produced and fully characterized MWCNTs, the impact of filler loadings on the characteristics and properties as well as performance of the nanocomposite can be better understood.

1.3 Objective of Study

This study mainly aimed to produce PC/MWCNTs nanocomposites with enhanced mechanical, thermal, electrical properties and chemical resistance performances by varying the loading of MWCNTs. The work will be further executed based on the following objectives:

1. To investigate the physico-chemical properties of Multi-Walled Carbon Nanotubes produced by the modified Staudenmaier method.

2. To assess the mechanical, thermal, electrical and chemical properties of nanocomposites produced with different filler loadings.

1.4 Scope of Research

The following research scope had been identified, to ensure that the objectives of the research are achieved:

A. Synthesis (Production and Characterization) of Multi-Walled Carbon Nanotubes using a modified Staudenmaier method.

B. Preparation of PC/MWCNTs nanocomposites using the melt mixing technique. The morphology, Young's modulus, density, thermal diffusivity, thermal conductivity, glass transition temperature (T_g), the electrical conductivity and the chemical diffusivity capacity of the nanocomposites were assessed.

C. Application of statistical tools to evaluate the correlation between various processing variables with product quality.

1.5 Thesis Organization

This thesis can serve as a platform that supplies information on techniques used in PC/MWCNTs nanocomposite preparation, characterization, and the corresponding studies. This thesis was organized into five chapters. Chapter one introduced the work and the associated problem statement, the objectives, the research scope and the thesis organization. Chapter two offered the literature review and critical analyses of some of the works that had been conducted on the subject matter. It detailed the overview of polymers nanocomposites and establishes an overview of polymers reinforcement. In the third chapter, the experimental methods were conducted to answer the objectives that had been outlined. Chapter four elaborated on the findings of the research which significantly highlighted the influence of different carbon nanotubes loading on the thermal, mechanical, electrical and physiochemical properties of the material. Chapter five concluded the findings that were obtained in the study.

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