UNIVERSITI PUTRA MALAYSIA

PRODUCTION AND CHARACTERIZATION OF POLYPROPYLENE-CARBON NANOTUBE NANOCOMPOSITES

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By

JEEFFERIE BIN ABD RAZAK

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

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Emak, Ayah & All My Family Members

Uan, Omar, Fendi, Nana, Bee, Angah, Fauz,

KKA Postgraduates, FKP Staff & UTeM

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

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Chairman : Mohamad Amran Mohd Salleh, PhD

Faculty : Engineering

At the first stage in this research, the multi-walled carbon nanotubes (MWCNTs) were grown by using the floating catalysts chemical vapor deposition (FC-CVD) method. The produced MWCNTs were characterized by using the scanning electron microscopy (SEM), transmission electron microscopy (TEM) and the high resolution transmission electron microscopy (HRTEM). The MWCNTs was incorporated into polypropylene (PP) to produce the PP/MWCNTs nanocomposites through the direct melt compounding process using an internal mixer. The mixer parameters were varied to determine the best parameter to produce the nanocomposites. It was determined through the tensile test which performed on every nanocomposite which fabricated from the various combinations of parameters. The best parameters to produce the nanocomposites were at the temperature of 175°C, rotor speed of 60 rpm and the compounding time of 8 minutes. In the next stage, the effect of filler loading was studied. The filler loading was varied from 0, 0.25, 0.50, 0.75 and 1.00wt.%. The best tensile properties was observed in the nanocomposites with 0.75wt.% of MWCNTs, with the improvement of 42.82% and 126.90% of the tensile strength and tensile modulus, compared to the virgin PP matrix. The validation of the tensile test data was carried out by using the historical data design from the Response Surface Methodology (RSM) with the aid of the Design Expert Software 6.10. The PP/MWCNTs nanocomposites which compounded from the best processing parameter were further characterized for other properties. Physical test on the nanocomposites density was revealed that the density is decreased with the increasing percentage of MWCNTs addition. This condition gives benefit on the weight saving of the materials. Fourier Transform Infra Red (FTIR) and X-Ray diffraction analysis disclosed that the melt blending between the PP matrix and MWCNTs filler is entirely physical-mechanical blending, without involving any chemical interaction. This further explained the reinforcement behavior of the MWCNTs within the PP matrix. Furthermore, TEM images of the nanocomposites surface confirmed an excellent dispersion and distribution of the MWCNTs in the PP matrix. This condition was supported by the significant improvement of the flexural strength, flexural modulus, impact strength, and storage modulus and loss modulus properties of the fabricated nanocomposites. In overall, the proper selection of the melt blending processing parameter and the use of low filler loading was significantly helped to disperse and distribute the MWCNTs homogenously within the PP matrix, resulting major improvements to the many of the properties studied.

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

PENGHASILAN DAN PENCIRIAN NANOKOMPOSIT POLIPROPILENA-KARBON NANOTIUB

Oleh

JEEFFERIE BIN ABD RAZAK

2009

Pengerusi : Mohamad Amran Mohd Salleh, PhD

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Pada peringkat pertama penyelidikan, karbon nanotiub berbilang dinding (MWCNTs) telah ditumbuh melalui kaedah pemangkinan terapung secara pemendapan wap kimia (FC-CVD). MWCNTs yang dihasil telah diciri menggunakan mikroskop imbasan elektron (SEM), mikroskop pemindahan elektron (TEM) dan mikroskop pemindahan elektron resolusi tinggi (HRTEM). MWCNTs telah digabung dengan polipropilena (PP) bagi menghasil nanokomposit PP/MWCNTs melalui proses penyebatian lebur secara terus, menggunakan pencampur dalaman. Parameter pencampur dipelbagai bagi menentukan parameter terbaik bagi menghasilkan nanokomposit. Ia ditentukan melalui ujian tegangan yang dilakukan keatas setiap nanokomposit yang difabrikasi dari gabungan pelbagai parameter. Parameter terbaik bagi menghasil nanokomposit adalah pada suhu 175°C, kelajuan rotor 60 rpm dan tempoh penyebatian selama 8 minit. Pada peringkat seterusnya, kesan pembebanan pengisi telah dikaji. Pembebanan pengisi dipelbagai

dari 0, 0.25, 0.50, 0.75 dan 1.00 peratus berat. Sifat tegangan terbaik diperhati pada nanokomposit terisikan 0.75 peratus berat MWCNTs, dengan penambahbaikan sebanyak 42.82% dan 126.90% bagi kekuatan tegangan dan modulus tegangan berbanding matriks PP dara. Pengesahan keatas data ujian tegangan dilaksana dengan menggunakan kaedah permukaan sambutan (RSM) dengan bantuan perisian *Design Expert* 6.10. Nanokomposit PP/MWCNTs yang disebati menggunakan parameter pemprosesan yang terbaik seterusnya diciri bagi sifat-sifat yang lain. Ujian fizikal bagi ketumpatan nanokomposit menunjukkan ketumpatan adalah mengurang dengan peningkatan peratusan penambahan MWCNTs. Keadaan ini memberi kebaikan kepada pengurangan berat bahan. Analisis perubahan *fourier* infra merah (FTIR) dan pembelauan sinar-X (XRD) mendedahkan bahawa penyebatian lebur antara matriks PP dan pengisi MWCNTs secara keseluruhannya adalah penyebatian fizikal-mekanikal, tanpa melibatkan sebarang interaksi kimia. Ini selanjutnya menerangkan kelakuan penguatan MWCNTs dalam matriks PP. Sebagai tambahan, imej-imej TEM bagi permukaan nanokomposit mengesahkan taburan dan serakan MWCNTs yang sangat baik didalam matriks PP. Keadaan ini disokong oleh penambahbaikan signifikan bagi sifat-sifat kekuatan pelenturan, modulus pelenturan, kekuatan hentaman, modulus simpanan dan modulus lesapan bagi nanokomposit yang difabrikasi. Secara keseluruhan, pemilihan parameter pemprosesan pencampur lebur yang betul dan penggunaan pembebanan pengisi yang rendah akan secara signifikannya dapat membantu serakan dan taburan MWCNTs secara seragam, menyebabkan penambahbaikan yang major bagi kebanyakan sifat yang dikaji.

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

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DECLARATION

I hereby declare that the thesis is based on my original work except for quotations and citations, which have been duly acknowledged. I also declare that it has not been previously or concurrently submitted for any other degree at UPM or other institutions.

JEEFFERIE BIN ABD RAZAK

Date: 9 February 2009

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS

°C	Degree Celsius
ABS	Acrylonitrile Butadiene Styrene
ANOVA	Analysis of Variance
ASTM	American Society for Testing and Materials
CASOS	Centre for Computational Analysis of Social and Organizational Systems
CNTs	Carbon Nanotubes
CVD	Chemical Vapor Deposition
DMA	Dynamic Mechanical Analysis
DSC	Differential Scanning Calorimetry
DWCNTs	Double Walled Carbon Nanotubes
E/ρ	Specific Modulus
ESEM	Environmental Scanning Electron Microscope
FC-CVD	Floating Catalyst Chemical Vapor Deposition
FTIR	Fourier Transform Infra-Red
HRTEM	High Resolution Transmission Electron Microscopy
ID	Internal Diameter
MA-SEBS	Maleic anhydride grafted styrene-(ethylene-co-butylene)-styrene
MWCNTs	Multi-Walled Carbon Nanotubes
OD	Outer Diameter
OFAT	One Factor at Time
PE	Polyethylene

PEN/MWCNTs	Poly(ethylene 2, 6-naphthalate/Mutiwalled carbon nanotubes
PMC	Polymer Matrix Composites
PMMA	Polymethyl Metacrylate
PNC	Polymer Nanocomposites
PP	Polypropylene
PP/MWCNTs	Polypropylene-Multiwalled Carbon Nanotubes Composites
PVC-U	Unplastisized Polyvinyl Chloride
PVCv/NBR	Virgin Polyvinyl Chloride / Natural Butadiene Rubber
PVCw/NBR	Waste Polyvinyl Chloride / Natural Butadiene Rubber
RHA	Rice Husk Ash
rpm	rotation per minute
RSM	Response Surface Methodology
SEM	Scanning Electron Microscopy
Si	Silicon
SWCNTs	Single Walled Carbon Nanotubes
TEM	Transmission Electron Microscopy
Tg	Glass Transition Temperature
TGA	Thermogravimetry Analysis
TPa	Tera Pascal
UHMWPE	Ultra High Molecular Weight Polyethylene
wt.%	weight percentage
XRD	X-Ray Diffraction