UNIVERSITI PUTRA MALAYSIA

CARBON NANOTUBES, CARBON FIBERS AND CARBON NANOFIBERS FOR NATURAL RUBBER APPLICATIONS

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CARBON NANOTUBES, CARBON FIBERS AND CARBON NANOFIBERS FOR NATURAL RUBBER APPLICATIONS

By

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Thesis submitted to the School of Graduate Studies, Universiti Putra Malaysia, in partial fulfilment of the Requirements for the Degree of Master of Science

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This thesis is especially dedicated to Mak (Hjh Rosnah binti Johari and Allahyarhamah Hjh Hamsiah binti Hassim), ayah (Hj Tahir bin Hamzah and Ismail bin Saad), husband (Johary bin Ismail), son (Ghazaly bin Johary) and daughter (Nur Nafeesa binti Johary) Thank you very much for your love and support….

Abstract of thesis presented to the Senate of Universiti Putra Malaysia as fulfilment of the requirement for the degree of Master of Science

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Nanomaterials are well known to be used as reinforcing fillers. With its high surface area, addition of small amount of nanomaterials in the polymer matrix would increase the strength of the composite material. Carbon nanotubes (CNTs), carbon fibers (CFs) and carbon nanofibres (CNFs) are among the nanomaterials that are commonly used as fillers to improve the mechanical properties. These nanomaterials have excellent mechanical and thermal characteristics. The good characteristics of natural rubber such as heat built up, hysteresis, impact, and tensile strength, flexing and damping capability on service were expected to be enhanced when added with nanomaterials such as carbon nanotubes. The other method used to synthesize nanomaterials are based on laser ablation and arc discharge which limit the production size and produces a large amount of impurities. Chemical Vapor Deposition (CVD) was selected to encounter the problems mentioned. However, the most common problem faced in the production of nanocomposites is the blending of the matrix with the nanomaterial. This research project aims to find the suitable method to incorporate different nanomaterials with the natural rubber matrix. CNTs, CFs and CNFs were synthesized using the Floating Catalyst Chemical Vapor

Deposition (FC-CVD) method. Viscosity-stabilized natural rubber, SMRCV60, was used as the matrix. The blends were prepared using the solvent casting method. Test samples were then prepared using hot press. The nanomaterials are successfully synthesized by the Floating Catalyst Chemical Vapor Deposition (FC-CV) method. In this study, the production condition of pure CNTs has been fixed at reaction temperature 850° C with the hydrogen flow rate of 300 ml/min and reaction time 30 minutes. The nanomaterials then were used to prepare a rubber nanocomposites using solvent casting method. The tensile property shows that CNTs possess the highest strength with 1.21 MPa at 7% composition. The composite which contains 5% CNFs gave the highest strain at about 12.2% elongation. With 5 % CFs filling in the matrix, 7.75% elongation was achieved. From DMTA analysis, the damping characteristic, tan *d* proved the theoretical prediction of rubber nanocomposites, as the amount of filler increases the nanocomposites become rigid which is very significant from the storage modulus plot. The natural rubber nanocomposites possess the best tensile and thermal properties even without extensive purification on the nanomaterials produced and without any pretreatment on the natural rubber used.

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

KARBON TIUB NANO, KARBON SERAT DAN KARBON SERAT NANO, UNTUK APLIKASI GETAH ASLI

Oleh

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Bahan nano seperti seramik nano dan karbon tiub nano adalah terkenal sebagai bahan pengisi penguat. Luas permukaan yang besar, dengan jumlah yang sedikit sudah mencukupi untuk memberi kesan kepada kekuatan komposit. Karbon tiub nano (CNT), karbon serat (CF) dan karbon serat nano (CNF) adalah bahan nano yang digunakan dan dikenali sebagai bahan pengisi untuk menambahbaik sifat mekanik pada komposisi yang rendah. Bahan bersaiz nano tersebut mempunyai sifat mekanik dan sifat terma yang sangat baik. Bahan nano seperti namanya adalah gugusan zarah halus. Kaedah yang sering digunakan untuk menyediakan bahan nano adalah berasaskan kaedah ablasi laser dan nyahcas arka yang menghasilkan bahan nano dengan jumlah yang sedikit dan lebih banyak bahan yang tidak tulen. Masalah yang sering timbul dalam pembuatan bahan komposit nano adalah mencampurkan bahan matrik dan bahan nano. Tujuan kajian ini dilakukan adalah untuk mencari kaedah yang terbaik untuk mencampurkan bahan nano ke dalam bahan matrik getah. Sifat-sifat fizik kebanyakan bahan komposit hari ini lebih menumpukan kepada bahan plastik dan getah sintetik berbanding getah asli. CNT,CF dan CNF disintesis menggunakan kaedah pemangkin terapung –penguraian wap kimia (FC-

CVD). Getah asli kelikatan stabil SMRCV 60 digunakan sebagai bahan getah matrik, campuran tersebut dihasilkan melalui kaedah pemeluwapan larutan dan pemampat panas digunakan untuk membentuk sampel ujian bahan getah komposit. Untuk kajian ini, penghasilan bahan nano telah ditetapkan pada suhu 850° C sebagai suhu tindak balas, kadar alir hidrogen pada 300 mL/min dan masa tindak balas ialah 30 minit. Penyediaan getah komposit meliputi 3 peringkat iaitu serakan bahan pengisi, pelarutan getah asli dan mencampurkan bahan pengisi dan getah terlarut. Sifat tegangan menunjukkan ada penambahbaikan dalam sifat mekanik. Jika dibandingkan diantara CNT, CF dan CNF, CNT memberikan nilai ujian tegangan tertinggi untuk 7 % komposisi iaitu 1.21 MPa. Pemanjangan pada tempat putus untuk getah komposit menunjukkan yang bahan getah komposit yang mengandungi serat memberikan nilai yang tinggi pada komposisi 5 % CNF iaitu 12.2 % pemanjangan dan pada komposisi 5 % CF ialah 7.75% pemanjangan. Daripada ujian dinamik mekanik terma, ciri-ciri serapan gegaran, tan d membuktikan ramalan teori untuk getah komposit, dengan bertambahnya komposisi bahan nano, bahan menjadi tegar dan dapat dilihat dengan jelas melalui plot modulus storan. Getah komposit memiliki ciri-ciri yang amat baik walaupun tanpa perlunya proses penulenan yang kritikal dan tanpa rawatan dilakukan kepada bahan matrik getah asli. Secara keseluruhannya getah asli komposit yang dihasilkan adalah sesuai untuk penggunaan lasak.

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I certify that a Thesis Examination Committee has met on 8 May 2009 to conduct the final examination of Hairani binti Tahir on her thesis entitled "Carbon Nanotubes, Carbon

Fibers, and Carbon Nanofibers for Natural Rubber Applications” 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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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.

HAIRANI TAHIR

Date:

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LIST OF ABBREVIATIONS

FCCVD Floating Catalyst Chemical Vapor Deposition

CNT	Carbon Nanotube
CNF	Carbon Nanofiber
CF	Carbon Fiber
SMRCV60	Standard Malaysia Rubber Constant Viscosity 60
TGA	Thermogravimetry Analyzer
DMTA	Dynamic Mechanical Thermal Analyzer
HNS	Hydroxylamine Neutral Sulphate
NR	Natural Rubber
MWNT	Multi-walled Carbon Nanotubes
SWNT	Single-walled Carbon Nanotubes
SEM	Scanning Electron Microscopy
TEM	Transmission Electron Microscopy
HRTEM	High Resolution Transmission Electron Microscopy
DSC	Differential Scanning Calorimetry
XRD	X-Ray Diffraction

CHAPTER 1

INTRODUCTION

At the beginning of this project, polyurethane was used as the starting material to incorporate with carbon nanotubes (CNTs). The production of polyurethane composite sample was found unsuccessful in the preparation stage due to the problems in controlling the foam formation and its removal from the glass substrate. A few attempts were made by applying the mold release agent underneath it however the problem still persists. Later, the SMRCV 60 was used to overcome the sticking problem and foam formation.

The light weight and recyclable properties of polymer are the best factors contributing to the increase of the number of research done in this area. Nanomaterial such as nanoclay and carbon nanotubes are well known as reinforcing filler; with its high surface area, addition of small amount of nanomaterials (one percent) could increase the strength of the composite material. Although rubbers are thermal and electrical insulators, incorporation of conductive fillers into these materials could produce composite materials with some electrical conductivity. Potential applications cover numerous fields such as rubber hoses, tire components, sensing devices to electrical shielding and electrical heating element (Shanmugaraj *et al.*, 2007)

Great interest has recently been developed in the area of nanostructure carbon material. Carbon nanostructure material is becoming of considerable commercials importance with interest growing rapidly over a decade since the discovery of buckminster fullerene, carbon nanotubes and carbon nanofibers (Dresselhaus *et al.*, 2001). Carbon nanotubes (CNTs) and carbon nanofibers (CNFs) are among most eminent material in the first rank of revolution nanotechnology. The most eye-catching features of these structures are their electronic, mechanical optical and chemical characteristics which open a way to future application (Atieh, 2005).

Chemical vapor deposition (CVD) technique has gained popularity as an alternative production method due to its potential to produce CNTs in a large scale. With this method, CNTs are produced via catalytic decompositions of hydrocarbons at relatively lower temperatures, normally between 427° C to 1400° C in a furnace. Although the technology has been applied to produce Vapor growth carbon fibers (VGCF) back in 1970s, the VGCF produced are often of low quality. Driven by the need to produce CNTs on a large scale, substantial and rapid progress was made in the development of CVD to establish the production of CNTs (Izawati, 2007).

Application of CNTs for natural rubber is one area of study in the field of polymer composites. SMR CV60 is one of the major polymers used in a wide variety of end products. SMR CV60 was a base material used in dry rubber product industry such as tires, engineering component and automotive part such as gasket, inner tube and lining (RRIM, 1981). The use of CNTs in SMR CV60 would be the new area for elastomeric composites because most of the study in polymer composites for carbon nanotubes, carbon fiber and carbon nanofibre has focused on plastics or thermoplastics materials as a base material but SMR CV60 is a thermosetting material that needs to be vulcanized before use. Nanocomposites are a blend of nanomaterials in the polymer. Nanocomposites nowadays are available in various grades and could be produced by various methods from dry blending to *in situ polymerization* techniques. Nanocomposites performance is normally studied for their physical or mechanical, thermal, and electrical properties.

1.2 Problem statement

The available methods to produce nanotubes and nanofibers are based on the arc discharge and laser ablation processes. Laser ablation produces small amount of nanotubes, whereas arc discharge methods generally produce larger quantities of impure material. In recent years work has focused in developing Chemical Vapor Deposition (CVD) technique using catalyst particles and hydrocarbon precursor to grow nanotubes. CVD seems to be the most promising method for industrial scale due to relatively low processing parameters.

Nanomaterial as the name implies are clusters of fine particle materials. The most common problem arises in the production of nanocomposite is the blending of matrix and the nanomaterial. This research project aims to determine the best method to incorporate nanomaterials with the polymer matrix. The physical properties of most of the composite materials prepared nowadays focuses on plastics material and synthetics rubber rather than natural rubber.

The good characteristics of natural rubber such as heat built up, hysterisis, impact and tensile strength and flexing and damping capability on service are expected to be enhanced when added with nanomaterial such as carbon nanotubes. The aims of this project is also to improve or modify the natural rubber properties to suit robust application like gasket for high pressure turbine or other engineering application. Limited shelf life of natural rubber and its flame retardant properties are also problems that sometime arise. Therefore thermal study was conducted to investigate these problems further.

1.3 Objectives of Study

This research work done is mainly on synthesizing nanomaterials and the produced materials then used to prepare rubber nanocomposites. The objectives of the works are as follows;

1. To produce Carbon fiber, nanofiber and nanotubes by FC-CVD method
2. To synthesize CNT, CNF and CF-rubber nanocomposites using different matrix-filler compositions.
3. To investigate the physical, thermal properties and microstructure of the resulting blend.

1.4 Scope of Study

In this research, floating catalyst chemical vapor deposition (FC-CVD) was used to produce a high purity and yield of CNTs, CFs and CNFs. The nanomaterials produced were characterized by using High Resolution Transmission Electron Microscopy (HR_TEM). Multi-walled carbon nanotubes and Carbon nanofibers were used to prepare natural rubber nanocomposites. Rubber nanocomposites were prepared by incorporating nanomaterials in a polymer solution and subsequently evaporating the solvents. Using this technique, nanotubes were dispersed homogeneously in the NR matrix.

The properties of the composites such as tensile strength, elongation ate beak and young modulus were investigated. The rubber nanocomposites were characterized using Thermogravimetric analyzer (TGA) and Dynamic Mechanical Thermal analyzer (DMTA).

CHAPTER 2

LITERATURE REVIEW

In this study, carbon nanotubes, carbon fiber and carbon nanofibre were synthesized using Floating Catalyst Chemical Vapor Deposition method (FC-CVD). Besides CVD, other methods such as Electric arc discharge, laser ablation, and solar energy can also be used to synthesize the nanomaterials but in CVD, the carbon source is deposited with the assistance of a catalyst at temperatures lower than 1200° C (Wei *et al*, 2007). CVD is defined as a chemical process for depositing thin films of various materials. In a typical CVD process the substrate is exposed to one or more volatile precursors, which react and decompose on the substrate surface to produce a desired deposit. There are various CVD method used currently and this report focuses on the Floating Catalyst Chemical Vapor Deposition (FC-CVD) method.

SMR CV 60 was in liquid form before being mixed with carbon nanotubes, carbon fiber and carbon nanofiber. The method used to incorporate these nanomaterials into the SMR CV60 was chosen after a few trials were made. The selectivity of the mixing equipment as a method to incorporate the nanomaterials was found unsuccessful due to its high composition capacity (i.e. 40g) since the nanomaterials produced is very limited. The other mixing methods, such as the use of two roll mills were also found to be not suitable because fine particle might be released to the environment. The possible and manageable method is to incorporate nanomaterials with SMR CV 60 is to mix the materials in liquid form, also called the solvent casting method.

2.1 Natural Rubber

Natural rubber (NR) is obtained from the tree *Hevea Braziliensis*. It is a polymer with long chain-like molecules. Structurally, the chain is made of isoprene units as monomers. Natural rubber is made up of many non-rubber chemical constituents. These constituents may or may not play any part in the properties of natural rubber. Typical examples of non-rubber constituents are amines and phospholipids which can act as antioxidants. Other non-rubber contituents may act as accelerator and stabilizers for latex. It is believed that there are carbonyl groups present in natural rubber in the form of aldehyde group; this aldehyde group will give rise to storage hardening. The location of aldehyde group in natural rubber is still being investigated (RRIM, 1981).

2.2 Viscosity Stabilized Rubber, SMRCV60

The variation of hardness and viscosity of rubber is due to the formation of gel. Gel in rubber industry refers to ‘storage hardening’ and its spontaneously formed crosslinked raw polymer. The crosslinks are formed due to the natural occurrence and reactivity of abnormal aldehyde groups in rubber. Storage hardening is prevented by reacting a mono-functional reagent with the aldehyde carbonyl groups or is accelerated by reacting the aldehyde carbonyl groups with a bi-functional reagent; thus creating crosslinks with itself. One or other of these two reactions take place in the drying period to gives two types of viscosity stabilized rubber. The two reagents used are hydroxylamine NH_2OH

and hydrazine $NH_2.NH_2$. (RRIM, 1981). Natural rubber used is SMR CV 60. SMR CV 60 is a viscosity stabilized grade of natural rubber produced from natural rubber latex which has undergone SMR processing techniques such as coagulation, washing, machining (prebreaker, creper and shredder) and is dried in the drier, then baled into SMR standard shape and size and packed (RRIM, 1981). SMR CV 60 is used due to its viscosity and longer storage compared to other SMR grades. The problem of storage hardening is normally due to the reaction of aldehyde groups in the rubber itself which lead to changes in viscosity value. Selective blending of field latex was done before starting SMR CV production; a constant viscosity survey must be carried out on all available sources of latex. Selective blending of field latex can then be carried out so that the calculated viscosity of the blend is well within the specified limit. Effective viscosity stabilization; can be achieved by treating the field latex with 1.5 kg of hydroxylamine neutral sulphate (HNS) per tonne of SMR CV (RRIM ,1981).

2.3 Nano material

Nanomaterials are by definition, materials that have at least one of their dimensions below 100nm (Lake *et al.,* 1999). Nanoparticles have very special properties due to their very small dimensions. These properties include optical properties, diffusion properties, electrical properties, catalytic activity and many others. Nanomaterials that are widely used are silica, carbon nanotubes, titanium dioxide, zinc oxide and nanoclays.Carbon nanotubes (CNT) are available in the market commercially with two main grades; multiwalled CNT (MWNT) and single walled CNT (SWNT). Single walled nanotubes is consist of a hollow tube graphitic carbon, normally capped at each end with a nanoscale