SYNTHESIS OF NATURAL RUBBER/PALYGORSKITE COMPOSITE VIA COMBINE LATEX COMPOUNDING AND MELT MIXING

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SYNTHESIS OF NATURAL RUBBER/PALYGORSKITE COMPOSITE VIA COMBINE LATEX COMPOUNDING AND MELT MIXING

by

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DECLARATION

I declare that the content presented in this dissertation entitiled "**SYNTHESIS OF NATURAL RUBBER/PALYGORSKITE COMPOSITE VIA COMBINE LATEX COMPOUNDING AND MELT MIXING**" is own my work which was done at Universiti Sains Malaysia unless onformed otherwise. This dissertation has not been previously submitted for any other degree.

Saya isytiharkan bahawa kandungan yang dibentangkan didalam disertasi ini bertajuk **"SINTESIS KOMPOSIT GETAH SEMULAJADI/PALYGORSKITE MELALUI GABUNGAN PENGKOMPAUNAN LATEKS DAN PENCAIRAN PENCAMPURAN"** adalah hasil kerja saya dan dijalankan di Universiti Sains Malaysia kecuali dimaklumkan sebaliknya. Disertasi ini juga tidak pernah disertakan untuk ijazah yang lain sebelumnya.

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Witness by,

Signature:

Supervisor's Name : **DR NADRAS OTHMAN**

Date :

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LIST OF ABBREVATIONS

ASTM	American Society for Testing Materials
BKF	2,2'-Methylenebis(6-Tert-Butyl-4-Methylphenol)
CRI	Cure Rate Index
CV	Conventional Vulcanization
FTIR	Fourier Transform Infrared Spectroscopy
HRTEM	High Resolution Transmission Electron Micros cope
METHA	3-(Trimethoxysily) propylmethacrylate-silane
MMT	Montmorillonite
NR	Natural Rubber
PAL	Palygorskite
SEM	Scanning Electron Microscope
SMR	Standard Malaysia Rubber
TESPT	Bis [3-(triethoxysilyl) propyl] Tetrasulfide-silane
TMTD	Tetramethyl Thiuram Disulfide
XRD	X-Ray Diffraction
ZDEC	Zinc Diethyldithiocarbamate
ZnO	Zinc Oxide

LIST OF SYMBOL

^{o}	Degree
^{o}C	Degree Celcius
θ	Tetha
δ	Delta
t_{90}	Cure Time
ts_2	Scorch Time
M_H	Maximum Torque
M_L	Minimum Torque
Tg	Glass Transition Temperature
E’	Storage Modulus
E”	Loss Modulus
%	Percentage
Phr	Part per Hundred Rubber
Mpa	Mega Pascal
dNm	Deci Newton Meter
kN	Kilo Newton
RH	Polymer Chain

SINTESIS KOMPOSIT GETAH ASLI/PALYGORSKITE MELALUI GABUNGAN PENGKOMPAUNAN LATEKS DAN PENCAMPURAN LEBURAN

ABSTRAK

Penghasilan nanokomposit berasaskan palygorskite (PAL) dan getah asli (NR) telah disediakan melalui kombinasi kaedah pencairan leburan dan pengkompaunan lateks. Objektif kajian ini dibahagikan kepada empat bahagian iaitu perbezaan masa pencampuran PAL, perbezaan masa sonikasi PAL, pembezaan pengisian PAL dan kesan rawatan permukaan menggunakan kaedah sililasi terhadap PAL. Keputusan ujian yang diperolehi dari siri sebelumnya adalah berkaitan dengan siri seterusnya. Ujian ciri pematangan nanokomposit NR/PAL, sifat-sifat mekanikal dan fizikal, dan ujian morfologi nanokomposit NR/PAL telah dijalankan untuk menguji sampel. Dalam siri yang pertama, 60 minit masa pencampuran PAL (PAL60) menghasilkan keputusan yang lebih baik untuk ciri-ciri pematangan, sifat mekanikal dan fizikal, dan peningkatan suhu peralihan kaca, Tg dimana ia menunjukkan interaksi antara getah dan pengisi adalah lebih baik berbanding sampel yang lain. Dalam siri kedua, 30 minit masa sonikasi PAL (S30) memapaparkan peningkatkan untuk ciri-ciri pematangan, menghasilkan keputusan sifat mekanikal dan fizikal yang baik dengan menghasilkan penyebaran PAL secara baik di dalam matriks disamping sokongan oleh struktur diinterkalasi dalam data XRD. Peralihan Tg kepada suhu yang lebih tinggi menunjukkan sekatan terhadap pergerakan rantaian berlaku di dalam mobiliti matriks. Seterusnya, di dalam siri yang ketiga, jumlah 4 phr PAL (PAL4) menunjukkan keputusan yang baik dalam sifat-sifat mekanikal dan ujian morfologi. Keputusan ini juga disokong dari pola XRD dimana PAL menunjukkan struktur diinterkalasi dan ia

menunjukkan bahawa penyebaran PAL berlaku secara baik di dalam matriks. Akhirnya di dalam siri terakhir, PAL dirawat dengan dua jenis silana; 3- (Trimethoxysily) propyl methacrylate-silane (METHA) dan Bis [3-(tri ethoxy silyl) propyl] Tetrasulfide-silane (TESPT). Ciri-ciri PAL dengan dan tanpa rawatan silana telah ditentukan dengan menggunakan Spektroskopi Tranformasi Inframerah Fourier (FTIR). Kehadiran kumpulan hidroxil dan karbonil dalam PAL selepas menjalani rawatan silana membantu dalam penghasilan penyebaran PAL yang baik dalam matriks. NR/PAL-TESPT menunjukkan sifat-sifat mekanikal yang lebih baik, penyebaran PAL yang baik dengan adanya ketebalan garisan lusuh dengan permukaan yang kasar dan peningkatan keluasan XRD berbanding NR/PAL-METHA.

SYNTHESIS OF NATURAL RUBBER/PALYGORSKITE COMPOSITE VIA COMBINE LATEX COMPOUNDING AND MELT MIXING

ABSTRACT

The fabrication of nanocomposite based on palygorskite (PAL) and natural rubber (NR) was prepared by a combination of melt mixing and latex compounding methods. The objectives of this study was divided into four which are the mixing time variation of PAL, the sonication time of PAL, the different filler loading of PAL and the effect of surface treatment using silylation method of PAL. The obtained results from previous series was related to the next series which the characterization of NR/PAL nanocomposites, the mechanical and physical properties, and the morphology testing of NR/PAL nanocomposites were conducted to test the samples. In the first series, 60 minutes of PAL mixing time (PAL60) produced a better cure characteristics, good mechanical and physical properties, and an increasing in glass transition temperature, Tg which it indicates the better rubber-filler interaction than other samples. For the second series, 30 minutes of PAL sonication time (S30) displayed an improving in cure characteristics, good mechanical and physical properties with producing good dispersion of PAL in the matrix by supporting with intercalated structure as shown by XRD data. The shifting of Tg towards higher temperature indicated a restriction in chain mobility. Next, in the third series of study, 4 phr of PAL loading (PAL4) demonstrated good results in mechanical properties and morphological testing. The good results of PAL4 was also supported from XRD patterns which is PAL 4 shows the intercalated structure and it represents a well dispersion of PAL in the matrix. Finally, in the last series, the PAL is treated with two type of silane; 3-(Trimethoxysily) propyl methacrylate-silane (METHA) and Bis [3- (tri ethoxy silyl) propyl] Tetrasulfide-silane

(TESPT). The characterisation of PAL with and without silane treatment was determined using Fourier Transform Infra-Red (FTIR) spectroscopy analysis. The present of hydroxyl and carbonyl groups in the PAL after underwent silane surface treatment assisting in producing well dispersion of PAL in the matrix. NR/PAL-TESPT showed better mechanical properties, good dispersion of PAL with thicker tear line and rougher surface and a broad peak of XRD patterns than NR/PAL-METHA.

CHAPTER ONE

INTRODUCTION

1.1. Background of study

Polymer possesses a great change of material characteristics such as flexibility, transparency, biocompatibility, and chemical stability compared to other materials. These properties enable them to be utilized in varied applications. Polymer modification and polymer blending have conquered a place in the polymer technology which the scientist realized that the synthesis of new polymers is always given outstanding output products. In recent years, researchers both in industry and academia have focused their interest on reinforcement of any inorganic or organic particles in order to improve the properties of polymeric composites. Polymer nanocomposites have caught the attention because they can achieve a higher degree of strength, high thermal stability (fire retardance applications) and excellent in barrier properties with very low nanofiller content (generally lower than 5%) (Cassagnau & Barres, 2010). In comparison with conventional composites, there are several advantages of filler reinforcements of polymer nanocomposites summarized as follows (Ma et al., 2010):

1. The smaller fraction of nanomaterials can be more effective reinforcements of the polymer nanocomposites.
2. The bigger surface area of the nanomaterials can increase the load transfer from the matrix to the reinforcing nano filler assuming good adhesion at the interface.
3. The crack propagation length at the interface of the nanocomposites becomes longer because of the size reduction of nanomaterials. This result improves the