PREPARATION AND PROPERTIES OF SEPIOLITE-FILLED ETHYLENE PROPYLENE DIENE TERPOLYMER COMPOSITES

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by

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LIST OF SYMBOLS

%	Percentage
°C	Degree Celsius
°C/min	Degree calcium per minute
µm	Micrometer (10^{-6})
Å	Angstrom (10^{-10} meter)
dNm	Deci Newton meter
DTG	Derivative thermogravimetric
E’	Storage modulus
g	Gram
g/cm^3	Gram per cubic Centimeter
Hz	Hertz
kN	Kilo Newton
m^2/g	Square meter per gram
M_d	Dry mass
M_H	Maximum torque
M_i	Initial Mass
M_L	Minimum torque
MPa	Mega Pascal
M_s	Swollen mass
nm	Nanometer (10^{-9})
Q_f	Interaction index of filled compound
Q_f/Q_g	Rubber-filler interaction index
Q_g	Interaction index of gum compound
T	Temperature

$T_{25\%}$	Temperature at 25% weight loss
$T_{5\%}$	Temperature at 5% weight loss
$T_{50\%}$	Temperature at 50% weight loss
t_{90}	Optimum cure time
tan δ	Tan delta
T_g	Glass transition temperature
t_{s2}	Scorch time
wt %	Weight percent
γ	Gamma

LIST OF ABBREVIATIONS

APTMS	3-aminopropyltrimethoxysilane
$CaCO_3$	Calcium carbonate
CB	Carbon black
CRI	Cure rate index
DCP	Dicumyl peroxide
DMA	Dynamic Mechanical Analysis
E_b	Elongation at break
ENB	5-ethylidene-2-norbonene
EPDM	Ethylene propylene diene monomer
EPDM/Sep	Ethylene propylene diene monomer/sepiolite
FTIR	Fourier Transform Infrared
GP	Glut Palmitate
M100	Tensile modulus at 100% elongation
MBT	2-Mercaptobenthiazole
MDR	Moving Die Rheometer
MMT	Montmorillonite
N330	N330 Grade of Carbon black
NR	Natural Rubber
phr	part per hundred rubber
SEM	Scanning electron microscopy
Sil	Silica
TGA	Thermogravimetric Analysis
TMTD	Tetramethylthiuram disulphide
XRF	X-ray Fluorescence

PENYEDIAAN DAN SIFAT-SIFAT KOMPOSIT TERPOLIMER ETILENA PROPILENA DIENA TERISI SEPIOLIT

ABSTRAK

Peranan tanah liat sebagai pengisi dalam komposit getah mempunyai kepentingan yang tidak boleh diragui disebabkan oleh sumber tanah liat yang banyak, murah, warna yang cerah, dan kebolehan untuk meningkatkan kekuatan dan kekakuan getah. Walau bagaimanapun, ketidakserasian antara sifat hidrofilik tanah liat dan sifat hidrofobik getah yang akan mengakibatkan penyebaran yang kurang baik, kekal menjadi masalah yang mencabar dalam kegunaan industri. Kajian ini telah dilakukan untuk mengenal pasti potensi penambahan sepiolit (Sep) sebagai pengisi di dalam getah terpolimer etilena propilena diena (EPDM). Komposit EPDM/Sep telah dihasilkan menggunakan kandungan sepiolit sebanyak 0 hingga 70 bahagian perseratus getah (bsg) menggunakan mesin penggiling bergulung dua. Keputusan kajian menunjukkan peningkatan dalam sifat-sifat tegangan dan sifat-sifat mekanik dinamik, rintangan pembengkakan dan ketumpatan silang, kestabilan terma, dan rintangan kebolebakaran bagi komposit terisi sepiolit berbanding komposit tidak terisi sepiolit. Penyebaran homogen partikel sepiolit dalam matriks EPDM dan pembentukan struktur zig-zag, terutamanya pada 60 bsg, ialah penyebab utama peningkatan sifat-sifat mekanik komposit ini yang telah dikenal pasti melalui kajian morfologi. Pembentukan suatu lapisan perlindungan yang berperanan sebagai penghalang kepada pemindahan haba kepada lapisan yang lebih dalam telah meningkatkan rintangan kebolehbakaran komposit ini. Dalam kajian sepiolit yang dihibrid dengan karbon hitam (CB), silika, atau kalsium karbonat ($CaCO_3$), lima komposisi yang berbeza telah disediakan dengan EPDM/Sep pada 30 bsg sebagai komposit kawalan. EPDM/Sep/CB mempamerkan

kekuatan tegangan, kestabilan terma yang tertinggi, dan peningkatan rintangan kebolehbakaran. Keputusan kajian ini dipengaruhi oleh kebolehan karbon hitam untuk tersebar dengan baik dan membentuk lapisan perlindungan yang berperanan sebagai penghalang pengangkutan jisim dalam matriks. Penemuan penting dalam kajian ini menunjukkan bahawa silika dan CB boleh dihibridkan dengan sepiolit, malah sepiolit juga berpotensi sebagai pengganti $CaCO_3$ dalam komposit EPDM. Sementara itu, penggunaan garam glut palmitat (GP) dan (3-aminopropil)trimetoksisilana (APTMS) sebagai agen pengganding dalam komposit EPDM/Sep berjaya meningkatkan kekuatan tegangan dan pemanjangan putus, interaksi getah-bahan pengisi dan sifat-sifat mekanik dinamik. Penambahan interaksi getah-bahan pengisi menunjukkan bahawa kehadiran GP and APTMS telah meningkatkan kesesuaian dan interaksi di antara sepiolit dan matriks EPDM, seperti yang disokong oleh keputusan dari spektroskopi inframerah transformasi Fourier (FTIR). Kajian ke atas sistem pemvulkanan yang berbeza menunjukkan bahawa sifat-sifat kekuatan tegangan dan sifat-sifat dinamik mekanik yang tertinggi dipamerkan oleh sistem sulfur, diikuti oleh sistem campuran kuratif dan sistem peroksida, dan sebaliknya kestabilan terma dan rintangan kebolehbakaran tertinggi pula ditunjukkan oleh sistem peroksida.

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ABSTRACT

The role of clays as fillers in rubber composites is unquestionably important due to their abundance, low cost, light colour, and ability to enhance the strength and stiffness of rubber. However, differences between the compatibility of the hydrophilic clay and the hydrophobic rubber, which would result in a poor dispersion, remains as a challenging problem in industrial applications. This study was conducted to determine the potential of incorporating sepiolite (Sep) as a filler in ethylene propylene diene terpolymer (EPDM). EPDM/Sep composites were prepared with Sep loadings of 0–70 parts per hundred of rubber (phr) using a two-roll mill. The results indicated improvements in the tensile and dynamic mechanical properties, swelling resistance and crosslink density, thermal stability, and flammability resistance of the sepiolite-filled composite compared with the unfilled composite. The homogenous dispersion of sepiolite particles in the EPDM matrix and the formation of zigzag structures, especially at 60 phr, were the main reasons for the improved mechanical properties of the composite, which were confirmed by the morphological studies. The formation of a protective layer, which acted as a barrier against heat transfer into the deeper layers, had enhanced the flammability resistance of the composites. In the study of sepiolite hybridised with carbon black (CB), silica, or calcium carbonate ($CaCO_3$), five different compositions were prepared, with EPDM/Sep at 30 phr as a control composite. EPDM/Sep/CB displayed the highest tensile strength, thermal stability, and improved flammability resistance. These results were influenced by the ability of CB to disperse well and form protective layers that could act as a mass transport barrier in the matrix.