# THE CURING CHARACTERISTICS AND MECHANICAL PROPERTIES OF WASTE TYRE DUST AND WASTE TYRE DUST HYBRID FILLED NATURAL RUBBER COMPOUNDS

**By**

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## LIST OF ABBREVIATIONS

| | |
|---|---|
| ASTM | American Standard Test Method |
| BKF | 2, 2'-Methylene-bis (4-methyl-6-tert-butylphenol) |
| BS | British standard |
| CB | Carbon black |
| CBS | N-cyclohexyl-2-benzothiazole sulfonamide |
| $CH_2$ | Methylene |
| CMF | Cellulose micro fibrils |
| $CO_3^{2-}$ | Carbonate |
| CV | Conventional Vulcanization |
| DOE | Design of experiments |
| EFC | Elementary fibrous cells |
| EV | Efficient Vulcanization |
| FBR | Fraction of bounded rubber |
| FEF | Fast extrusion furnace |
| FF | Fine furnace |
| FRIM | Forest Research Institute of Malaysia |
| FT | Fine thermal |
| FTFT | Fatigue-to-Failure Tester |
| FTIR | Fourier Transform Infra Red |
| FTIR-ATR | Fourier Transform Infra Red in attenuated total reflection mode |
| GPF | General purpose furnace |
| H | Hidrogen |

| | |
|---|---|
| HMF | High modulus furnace |
| IR | Infrared |
| ISAF | Intermediate super abrasion furnace |
| JIS | Japanese Industrial Standard |
| KBr | Potassium bromide |
| MDR | Moving die rheometer |
| MT | Medium thermal |
| MT-NS | Medium thermal non-staining |
| N | Nitrogen |
| N100-N990 | Normal cured carbon black |
| NBR | Acrylonitrile-butadiene rubber |
| NR | Natural rubber |
| O | Oxygen |
| RMA | Rubber modified asphalt |
| RRIM | Rubber Research Institute Malaysia |
| S | Sulphur |
| s.d. | standard deviation |
| SAF | Super abrasion furnace |
| SEBS-g-MA | Maleic anhydride-grafted styrene-ethylene-butylene-styrene |
| SEM | Scanning electron microscopy |
| SMR | Standard Malaysian Rubber |
| SMR-CV | Constant-viscosity Standard Malaysian Rubber |
| SMR-L | Light coloured Standard Malaysian Rubber |
| SMR LV | Lower-viscosity Standard Malaysian Rubber |

| | |
|---|---|
| SMR GP | General purpose Standard Malaysian Rubber |
| SMR WF | Darker coloured Standard Malaysian Rubber |
| SRF | Semi-reinforcing furnace |
| TC | Technical Classification |
| TMTD | Tetra methyl thiuram disulfide |
| TOR | *Trans*-polyoctylene rubber |
| WTD | Waste tyre dust |
| XCF | Conductive furnace |
| ZnO | Zinc Oxide |

## LIST OF SYMBOLS

| | |
|---|---|
| $^{o}C$ | Degree celcius |
| $d_{50}$ | Mean particle size |
| $D_{agg}$ | Aggregate diameter |
| $D_{pp}$ | Particle diameter |
| Eb | Elongation at break |
| $kgcm^{-2}$ | kilograms force per square centimeter |
| L/D | Length over diameter |
| $lbf/in^{2}$ | Pound-force per square inch |
| $m^{2}g^{-1}$ | Square meter per gram |
| M100 | Stress at 100% elongation |
| M300 | Stress at 300% elongation |
| $M_{HR}$ | Maximum torque |
| MPa | MegaPascal |
| phr | Part per hundred rubber |
| ppsi | pounds force per square inch |
| $Q_{f}/Q_{g}$ | Rubber-filler interaction |
| $revmin^{-1}$ | Revolution per minute |
| $t_{2}$ | Scorch time |
| $t_{90}$ | Cure time |
| Wd | Dried weight |
| Wi | Initial weight |
| Ws | Swollen weight |

**SIFAT-SIFAT PEMATANGAN DAN MEKANIKAL SEBATIAN GETAH ASLI TERISI SERBUK SISA TAYAR DAN HIBRID SERBUK SISA TAYAR**

## ABSTRAK

Ciri-ciri pematangan, sifat-sifat tensil, kelesuan, kelakuan pembengkakan dan morfologi getah asli (NR) terisi serbuk sisa tayar (WTD) telah dikaji. Sebatian-sebatian NR terisi WTD dibandingkan pada pelbagai pembebanan pengisi mulai dari 0-30 phr. Ciri-ciri pematangan seperti masa skorj ($t_2$), masa pematangan ($t_{90}$) dan tork maksimum ($M_{HR}$) menurun dengan peningkatan pembebanan WTD. Sifat-sifat mekanikal seperti sifat-sifat tensil dan hayat fatig berkurang secara umumnya dengan penambahan pembebanan WTD. Penambahan pembebanan WTD juga menyumbang kepada penurunan nilai interaksi getah-pengisi, $Q_f/Q_g$. Dalam kajian ini, WTD dihibridkan bersama tiga pengisi berbeza iaitu hitam karbon (CB), kalsium karbonat ($CaCO_3$) dan gentian kenaf. Sebatian NR terisi dengan 30 phr hibrid WTD disebatikan mengikut peningkatan penggantian separa WTD dengan CB, $CaCO_3$ dan gentian kenaf pada 0, 10, 15, 20 dan 30 phr. Keputusan dari sifat-sifat yang disebutkan di atas seperti tork maksimum, sifat-sifat mekanikal dan interaksi getah-pengisi secara umumnya menunjukkan peningkatan nilai dengan peningkatan pembebanan CB dan $CaCO_3$ serta pengurangan nilai dengan peningkatan pembebanan gentian kenaf. Mikrograf-mikrograf SEM untuk permukaan retak semua sebatian menyokong keputusan-keputusan bagi sifat mekanikal.

**Katakunci**: Getah asli, Serbuk sisa tayar, Hibrid, Sifat-sifat mekanikal, Morfologi

**THE CURING CHARACTERISTICS AND MECHANICAL PROPERTIES OF WASTE TYRE DUST AND WASTE TYRE DUST HYBRID FILLED NATURAL RUBBER COMPOUNDS**

## ABSTRACT

Curing characteristics, tensile properties, fatigue, swelling behaviour and morphology of waste tyre dust (WTD) filled natural rubber (NR) compounds were studied. The properties of WTD filled NR compounds were studied at various filler loading ranging from 0-30 phr. The curing characteristics such as scorch time ($t_2$) and cure time ($t_{90}$) and maximum torque ($M_{HR}$) decreased with increasing WTD loading. The mechanical properties such as tensile properties and fatigue life generally decreased with increasing amount of WTD loading. Increment of WTD loading also contributed to decreasing value of rubber-filler interaction, $Q_f/Q_g$. In this work, WTD is hybridized with three different fillers i.e. carbon black (CB), calcium carbonate ($CaCO_3$) and kenaf fibre. NR compounds filled with 30 phr of WTD hybrid are compounded with increasing partial replacement of WTD by the three fillers at 0, 10, 15, 20 and 30 phr. The results of the aforementioned properties like maximum torque, mechanical properties and rubber-filler interaction generally showed increment of values with increasing loading of CB and $CaCO_3$ as well as decreasing value after the addition of kenaf fibre. The SEM micrographs of fractured surface of all of the compounds supported the results for the mechanical properties.

**Keywords:** Natural rubber, Waste tyre dust, Hybrid, Mechanical properties, Morphology

# CHAPTER 1

# 1 INTRODUCTION

## 1.1 Overview

Ever since the Scottish born John B. Dunlop made a discovery for pneumatic tyre in 1887 (Mount & List, 1994), the sale and consumption of tyre show no sign of slowing down. Tyre became big necessity for people worldwide as the number of vehicles likewise growing. The record of car production in 2008 alone stated that 52,940,559 cars have been produced worldwide (Worldometers, 2009). The cars production phenomenon is closely related to the tyre sales and demands. Sumitomo Rubber Industries Limited, the Japanese-based and also the second biggest tyre maker in China had estimated 10% growth of tyre sales a year from almost 150 million tyres in 2009.

The demands of tyres are not focusing merely on the newly produced cars but also on replacements. The motorists in China replaced about 100 million tyres in 2009 (Agencies, 2010). These big numbers of tyre sales and demands contribute to big number of tyre consumption as well as big number of waste. Hence those wastes drive major problem of pollution since all the waste would be dumped into landfills. Since waste tyres are non-biodegradable items, the landfills will be packed with the ever-increasing number of waste tyres. Thus, this matter should be overcome by promoting tyre recycling purposely to reduce the amount of waste tyre as well as amount of pollution.

Moreover, ASEAN state members like Malaysia, Indonesia and some other countries have reinforced Waste Management Policies. In Malaysia, legislation such as Solid Waste and Public Cleansing Management Corporation Act 2007 was drafted purposely to enforce solid waste management laws and related matters. In Indonesia, the handling of waste is governed by the Act of the Republic of Indonesia Number 18 Year 2008 (ASEAN Secretariat, 2009). Therefore, in reinforcing the aforementioned legislations, many methods are promoted in recycling used tyres such as pyrolisis which is a process involving thermal decomposition of waste tyres. There are also companies who rethreaded used tyres and sold them. Besides, other method of waste tyres recycling is also performed by processing them into small chips or crumbs or even dust.

Waste tyres or waste tyre dust (WTD) have been widely used in industry lately with the growing interest in environmental consciousness among industrialists and researchers nowadays. As reported by Gotteland et al. (2007), waste tyres posses many good properties like hydrophobic nature, high strength, low thermal conductivity etc. However, recycled products have major drawbacks that they are not able to perform like the virgin materials. Therefore, there are works done in saving the WTD from being unused. One of those works is by applying shredded waste tyre or waste tyre crumbs in light applications for example playground mat in recreation application. Despite the drawbacks of waste tyre as waste or recycled material, there are still efforts done by researchers in utilizing the good properties in waste tyre as mentioned before.

One of the efforts to apply the good properties of WTD is by fitting it in a hybrid system. Hybrid system, which comes from the word meaning something that is the product of different things is a system with combination of more than one substance or material. In fact, hybrid system is beneficial for a compound because there are contributions of properties from every substances or materials in the hybrid. The usage of hybrid fillers has proven improvements on composite performance. In addition, there are many studies (Ghosh et al., 2008; Jian et al., 2010 & Kudus et al., 2011) done regarding the usage of hybrid compounds. Through such studies, there are many discoveries of superiority of hybrid system such as the enhancement of thermal conductivity (Yu et al., 2008 & Lee et al., 2006), reduction of environmental degradation (Leong et al., 2003), improvement of mechanical properties (Kundu et al., 2004), chemical resistance (Kudina & Burya, 2010) and others.

In this project, WTD is paired with three well known, different reinforcement materials i.e. carbon black (CB), calcium carbonate ($CaCO_3$) and kenaf fibre. These three materials are highly available in Malaysia. Besides availability, they are also well known as filler or reinforcement materials in industry. Thus, these three materials are suitable to be chosen to be paired with WTD as hybrid. In addition, WTD and WTD hybrid will be compounded with virgin natural rubber (NR) in this work where the curing characteristics and mechanical properties of the compounds will be investigated.

## 1.2 Problem statement

In Singapore, there were around 25,100 tonnes waste tyres generated in 2008 alone (Tay, 2008). Waste tyres create environmental problems because of their non-biodegradable properties (El- Gammal et al., 2010). Therefore, one way to solve the problems is by recycling or reutilizing the waste tyre. As previously mentioned, waste tyres became well known among industrialists and researchers as the increase of environmental conscious occurs globally. Moreover, recycled materials are cheaper in term of price. Thus, the production cost of products will be reduced with the usage of recycled materials. Besides using waste tyre, cost reduction may occur through utilization of hybrid materials or hybrid fillers. For example, some industrialists use calcium carbonate in most part of the hybrid filler as the cost reducer (Bregg, 2006).

Hybridization also functions in upgrading the performance of composites or engineering materials. This concept has been studied by Lee et al. (2006) where aluminium nitride/wollastonite hybrid filler was used in enhancing the thermal conductivity for microelectronics application. Besides Lee et al. (2006), Nigam et al. (2001) had utilized the concept of hybridization through the usage of carbon black/phenolic resin hybrid filler in their nitrile rubber vulcanizates in their study. They had discovered that the usage of hybrid filler had increased the physico-mechanical properties of the vulcanizates. Besides the enhancement of the aforementioned properties, the utilization of hybrid filler is capable in optimizing weight reduction, swelling reduction and other desirable properties.

### 1.3 Research objectives:

The objectives of this project are listed below:

1. To study the effect of WTD loading on the curing characteristics, mechanical properties which are inclusive of tensile properties and fatigue life, rubber-filler interaction and morphological study of WTD filled NR compounds.

2. To study the effect of CB, $CaCO_3$, and kenaf fibre loading on the curing characteristics, mechanical properties which are inclusive of tensile properties and fatigue life, rubber-filler interaction and morphological study of WTD/CB hybrid filler filled NR compounds.

3. To study and compare the effect of CB, $CaCO_3$ and kenaf fibre loading on the curing characteristics, mechanical properties which are inclusive of tensile properties and fatigue life, rubber-fibre interaction and morphological study of WTD/kenaf fibre hybrid filler filled NR compounds.

# CHAPTER 2

## 2 LITERATURE REVIEW

## 2.1 Tyre

### 2.1.1 Tyre recycling

The growth of development happens throughout the globe turn out to be the reason of the big necessity for raw materials especially materials from natural resources. The natural resources well known to equip the industrial needs are natural rubber, crude petroleum oil, mineral resources (like limestone etc.), natural fibres (like jute etc.) etc. The number of those natural resources that are previously mentioned is sometimes not growing but keep lessening like crude petroleum oil which are obtain through decomposition of fossil that takes million years of process. Furthermore, there are products from natural sources like natural rubber that cannot be biodegraded.

Therefore to prevent growing number of waste and rubbish and also to avoid the natural resources from running out, people have discovered ways like reusing, retreading, recovery or recycling. Those aforementioned methods can be considered a step in environmental saving, by reducing the consumption of virgin or natural rubber. In addition, tyre industry occupied one half of global natural rubber production (Food & Agriculture Organization of the United Nations, 1989). A few regions like Western Europe, Central Europe and North America are dominant consumers of natural rubber and at least half of the world's total rubber productions are from tyre industry (Booth, 1999).