UNIVERSITI TEKNIKAL MALAYSIA MELAKA

PROCESSABILITY AND COMPATIBILY ANALYSIS OF THERMOPLASTIC ELASTOMER FILLED RECLAIMED RUBBER

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Material) (Hons.)

by

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2012

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: **Processability and Compatibility Analysis of Thermoplastic Elastomer Filled Reclaimed Rubber**

SESI PENGAJIAN: 2011/12 Semester 2

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Material) (Hons.). The members of the supervisory committee are as follow:

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ABSTRAK

Getah tebus guna terisi adunan PP/ENR adalah merupakan bahan elastomer termoplastik yang baru muncul. Kesan nisbah adunan, pemvulkanan dinamik, dan agen pematangan terhadap sifat adunan polipropilena (PP), getah asli terepoksida (ENR) dan getah tebus guna (RR) disiasat. Pemvulkanan dinamik disedia menggunakan asid stearik dan zink oksida (ZnO) sebagai bahan tambah pematangan dan diikuti oleh penambahan sulfur.Pemvulkanan dinamik ini meningkatkan kekuatan tegangan dan sifat terma. Sebatian elastomer termoplastik ini disediakan dengan penyebatian lebur dan pengacuanan mampatan. Campuran disediakan dengan menleburkan sebatian di dalam satu pencampur dalaman, Haake Rheomix pada suhu dan laju pemutar masing-masing bersamaan 180ºC dan 60 rpm. Komposisi getah tebus guna dibezakan pada 5, 25 dan 50 peratus. Pengaruh nisbah getah tebus,TMPTA dan Irganox1010 sebagai penstabil dan pengikat sebatian dikaji untuk menambahbaik ciri-ciri dan sifat-sifat mekanik adunan elastomer termoplastik. Ciri-ciri dan sifat-sifat mekanikditentukan menggunakan ujian tegangan, ujian hentaman Izod, dan kekerasan. Elastomer termoplastik dengan 50% getah tebus guna menunjukkan kekuatan tegangan dan mekanisme penyerapan tenaga yang tinggi iaitu 7.909 MPa dan 3 J/m. Mikrostruktur spesimen menggunakan mikroskop elektron imbasan (SEM) menunjukkan peningkatan kebolehcampuran antara plastik dan getah dengan kehadiran RR. Seperti yang dijangkakan, ciri-ciri tegangan dan kestabilan haba meningkat dengan peningkatan dalam kandungan RR disebabkan ciri-ciri unggul bahan ini.

ABSTRACT

Reclaimed rubber filled PP/ENR blend is an emerging new thermoplastic elastomer material. The effect of blend ratio, dynamic vulcanization, and crosslinking agent on the properties of polypropylene (PP), epoxidised natural rubber (ENR) and reclaimed rubber (RR) blends were investigated. Dynamic vulcanization was prepared using stearic acid and zinc oxide (ZnO) as additional crosslinking chemicals followed by sulphur curing. Design of experiment (DOE) was conceived and optimized response surface methodology (RSM). Thermoplastic elastomer compound was prepared by melt compounding and compression molding processes. Blends were prepared by melt mixing in an internal mixer, Haake Rheomix of temperature and rotor speed of 180ºC and 60 rpm, respectively. Reclaimed rubber composition was introduced in 0, 25 and 50 percentage. Effect of reclaimed rubber ratio, TMPTA and Iragnox1010 as stabilizer and binder to the cure characteristics and mechanical properties of thermoplastic elastomeric compounding was investigated. The characteristics and mechanical properties of the compound had been determined using tensile, Izod impact, and hardness tests. Thermoplastic elastomers filled 50% of RR showed highest tensile strength and energy absorption mechanism of 7.909MPa and 3 J/m. The microstructure of specimen observed using scanning electron microscopy (SEM) showed good miscibility between rubber and plastic due to the presence of RR. As expected, the tensile properties and thermal stability increase with the increases RR content due to superior characteristics of this material.

DEDICATION

This thesis is gratefully dedicated to my family, all my friends, my supervisor, Puan Toibah Binti Abd Rahim and and my co-supervisor Dr Noraiham Binti Mohamad. Thank you for all your supports, guidance, help and co-operation, directly or indirectly.

ACKNOWLEDGEMENT

Bismillahirrahmanirrahim, I would like to thank to Allah for His blessing because I have accomplished this research. Firstly I would like to extend my heartiest gratitude to Puan Toibah Binti Abd Rahim and Dr Noraiham Binti Mohamad as my supervisor and co-supervisor who had given me guidance and support during the research. Not to be forgotten to other lecturers, my friends and other person whose name is not mention here.

Secondly, I want to thank my loving mother and father for supporting me to continue this study. With prayers and moral support from both of them, I have gained strength to endure this study. I also want to thank to UTeM especially for Faculty of Manufacturing Engineering for giving me the opportunity to gain experience and knowledge during my studies.

Finally, I sincerely thank to all people who directly or indirectly helped to finish my final year project.

Thank you.

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LIST OF ABBREVIATIONS

ASTM	-	American Standard Test Method
TPNR	-	Thermoplastic natural rubber
SEM	-	Scanning Electron Microscope
TPNR	-	Thermoplastic natural rubber
NBR	-	Nitrile butadiene rubber
PP	-	Polypropylene
RSM	-	Response surface methodology
SEM	-	Scanning electron microscopy
DSC	-	Differential scanning calorimetry
FTIR	-	Fourier transform infrared
TPO	-	Thermoplastic polyolefin
TPV	-	Thermoplastic vulcanizate
TPE	-	Thermoplastic elastomer
RR	-	Reclaimed Rubber
TMPTA	-	Trimethylolpropane triacrylate
ENR	-	Epoxidized Natural Rubber
Irganox1010	-	Phenolic Primary Antioxidant

CHAPTER 1

INTRODUCTION

1.1 Research Background

Thermoplastic elastomers (TPEs) have emerged as a highly demanding class of polymeric materials and already started replacing many other conventional materials in various applications. With the exception of their dual characteristics of vulcanized elastomer and thermoplastic properties, the possibility of adjusting their properties by different routes has caused TPEs to be a versatile class of materials. TPEs have low cost and attractive properties such as superior mechanical strength, light weight, corrosion resistance, applicability at elevated temperatures, ability to be tailored for specific engineering applications are some of the properties of TPEs which may not be found in any other materials. The most important feature of TPEs is the repeated recyclability up to several times without significant loss of properties (Naderi *et al*., 1999).

Basically, TPES consists of at least two polymeric phases. A hard thermoplastic phase combined with a softer elastomer phase, and the properties of the resultant TPES will be derived from the properties of each of the two phases individually and from the extent of interaction between these phases (Arnold and Rader, 1992). There are many possible combinations of plastics and elastomers of TPEs that could be developed in accordance with the expected properties of the final material. Polypropylene (PP) is a linear hydrocarbon polymer and the typical density of PP is 0.9 g/cm3.The products based on PP are very significant commercially due to the advantages of being low in both density and the cost. Additionally, its crystalline structure and high melting point results in resistance to solvent and high temperature (Holden, 2000). The importance of recycling of waste materials (reclaimed rubber) generated from industries worldwide has become significantly important in the recent past mainly due to environmental reasons. The rubber manufacturing industry also faces a major challenge in this regard. A study to find the satisfactory ways and means to deal with the enormous quantity of waste rubber goods generated by the industry which may lead to severe environmental problems unless they are disposed properly. Reclaimed rubber wastes are usually generated during the processing of the products and from the disposal of post-consumer products.

Recently, the importance of recycling waste materials has been increasing for all industries worldwide. For rubber products, the automotive and transportation industries are the biggest consumers of raw rubber. Rubber waste is usually generated during the manufacturing process of products for these industries and by disposal of post-consumer (retired) products, mainly including scrap tires. For example, in Japan, about one million tons of scrap tires are generated annually. Blending the reclaimed rubber with another polymeric material to form a blend is one of the most effective methods of utilizing the reclaimed rubber for any industrial applications. Among various thermoplastic elastomers (TPEs), blends based on polypropylene are an important class of engineering materials (Rajalekshmi *et al*., 2005). Therefore this study is focused on the development of a new class of TPES material by blending PP, ENR and reclaimed rubber with different composition ratio generating from rubber manufacturing industry. It is hopeful at the end the final product developed would find useful applications in the automotive and rubber industries. The blending of PP, ENR and reclaimed rubber looks to be a very attractive as a way to obtain new TPEs with good mechanical properties and easy processability, these blends are found to be highly compatible. To improve the properties of TPES blends is to introduce crosslinks in the rubber phase of the blend. This can be done by vulcanization of the rubber phase through dynamic vulcanization

1.2 Problem Statement

Prior to the existence of thermoplastic elastomers (TPES), there were individual recyclable thermoplastic and non-recyclable elastomers. Nowadays, thermoplastic elastomers are getting encouraging response due to its unique properties, combining individual properties of plastic and elastomer, characteristics of vulcanized elastomer and thermoplastic properties .They exhibit the properties of both plastics and rubbers. The unique properties of both materials exist because TPES materials are created only by physical mixing of a thermoplastic and elastomer and no chemical or covalent bonding exists between the two. Thermoplastic elastomers have become a significant part of the polymer industry. Further research of thermoplastic currently lies on the polypropylene (PP), epoxidised natural rubber (ENR) involving the effort to reuse the reclaimed rubbers that had been vulcanized. This kind of rubber could reduce the manufacturing cost and contribute to greener environment due to the reclaimed rubbers used. The latest TPEs blend filled reclaimed rubber present good mechanical strength and compatible performance like other thermoplastic elastomer properties. The compatibility of recycled rubber to be the constituents of thermoplastic elastomers obviously could cut the production cost.

1.3 Objectives

The main focus of this study is to develop a new thermoplastic elastomer (TPEs) using polypropylene (PP), epoxidized natural rubbers(ENR) and reclaimed rubber and thereby to find a suitable solution to the existing problem of disposing the reclaimed rubber waste material generated from rubber based product industries.

The objectives of this study are :-

a) To prepare TPE filled reclaimed rubber using melt compounding and dynamic vulcanization technique.
b) To characterize the physical, mechanical and thermal properties of thermoplastic elastomer filled reclaimed rubber.
c) To identify the compatibility between thermoplastic, epoxidised natural rubber and reclaimed rubber.

1.4 Scope of Project

This study focuses on to produce a new thermoplastic elastomer material with blending of polypropylene (PP), epoxidised natural rubber (ENR) and reclaimed rubber (RR). It investigated the effect of blend ratios and some process modification such as dynamic vulcanization (with sulphur, stearic acid and zinc oxide) and additional of TMPTA and Irganox1010 as stabilizer and binder. The effect of ratio on blends is evaluated from characteristic and properties of PP/ENR filled reclaimed rubber, zinc oxide and stearic acid are used as activator, then sulphur act as curing agent. The melt compounding process involved materials in pallet form were blended together in an internal mixer to produce compound at operating temperature of 180 ^{o}C and rotor speed of 60 rpm. Crusher machine is used to crush compound into smaller particle size before placed into a mold (Hydraulic hot moulding machice) for compression molding process. Lastly, the sample materials were cut into specific size for mechanical testing, physical testing, thermal testing, composition and morphology analysis.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

Since the beginning of the plastics industry, it has been recognized that blending yields materials with property profiles superior to the features of the individual components. The blending of polymers provides a means of producing new materials, which combine the useful properties of all of the constituents (Utracki, 2002). The technology of blending is now advancing at a rapid pace. The ability to combine existing polymers into new compositions with commercial utilities offers the advantage of reduced research and development expense compared to the development of new monomers and polymers to yield a similar property profile. An additional advantage is the much lower capital expense involved with scale-up and commercialization. Another specific advantage of polymer blends versus new monomer polymer compositions is that blends often offer property profile combinations not easily obtained with new polymeric structures. Blending technology is more useful in the field of plastics recycling. It is estimated that about one third of all commercially produced polymer materials are blends of two or more polymers.

Polymer blend could be defined as a mixture of at least two macromolecular substances, polymers or copolymers, in which the ingredient content is higher than 2 wt% (Utracki,1998).Preparation of polymer blends can be done by melt mixing, latex blending, solution blending, partial block or graft polymerization as well as interpenetrating polymer network (IPN) technology. Melt mixing is the most widespread method of polymer blend preparation in practice. It is important that the size of the dispersed phase be optimized, considering the final performance of the blend (Horak *et al.,* 2007).

Polymer blends can be classified with many parameters such as number of components (binary, ternary etc), type of constituent (thermoplastic, thermosetting or elastomer), nature of the polymer architecture (graft or block polymer), compatibility among the constituent (compatible, incompatible), and method of producing (physical or chemical blending). Among the listed parameters, type of constituent is the most commonly used indicator. The polymer blends is classified into three main categories that is plastics-plastics blends, elastomer- elastomer blends, plastics elastomer blends As this study is more about the plastics elastomer blends.

2.1.1 Thermosetting Elastomer (TSE)

Thermoset is a polymer that is cured by heat or chemical reaction and becomes infusible and insoluble material. Thermoset polymers posses a cross linked molecular structure and are formed in two stage polymerization. The first stage is formation of a polymer with linear chains. The second stage of polymerization results in final cross linked structure. The end product can be made rigid or flexible. The Polymerization is controlled to result in heavily cross linked short chains for hard product and lightly cross linked long chains for soft and flexible products (Kear, 2003). Thermosetting polymers are network polymers (Callister, 2000). They become permanently hard during their formation, and do not soften upon heating. Thermosets have been chemically linked chains by covalent bonds during polymerization or by subsequent chemical or thermal treatment during fabrication. Others thermosets (such as epoxy, polyester, and urethane) cure at room temperature, because the heat produced by the exorthermic reaction is sufficient to cure the plastics (Kalpakjian and Schmid, 2006). The high density of crosslinking between the molecules makes the thermosetting material stiff and brittle and has better stability (Osswald and Menges, 2003). Thermoset is a polymer that is cured by heat or chemical reaction and becomes infusible and insoluble material. Thermoset polymers have a permanent irreversible polymerization. It is form permanent crosslinks between chains when cured and are comparatively strong and stiff but can't be recycled (Callister, 2006). Thermoset useful physical property of a thermoplastic is its glass transition temperature at which it begins to soften. Glass transition temperatures of different thermoplastics can be seen in (Legge, 1987).

2.1.2 Thermoplastic Elastomer (TPEs)

Holden (2000) and Walker (1986) have both shown that thermoplastic elastomers (TPEs) have become a technologically important class of material in recent past years. TPEs have many of the elastomeric physical properties of rubbers such as softness, flexibility, resilience but they are processable as thermoplastics. TPEs can be able to process using conventional thermoplastic processing techniques such as injection molding, extrusion. TPEs can also be completely reprocessable without any considerable loss in properties or processing characteristics due to recyclability of their scrap and rejects. Charles (2002) state that thermoplastic elastomers one of synthetic rubber aren't crosslinked, however. They can be molded and remolded again and again. Thermo- means "heat", and plastic means "moldable". Elastomer is just a fancy word that means "rubber." So a thermoplastic elastomer is a rubber that can be molded when it is heated.

Abdullah and Dahlan (1998) classify TPEs are a new of material that combine the properties of vulcanized rubbers with the ease of processability of thermoplastics Thermoplastic elastomers can be prepared by blending thermoplastic and elastomers at high shear rate. Thermoplastics, for example, polypropylene (PP), polyethylene (PE) and polystyrene (PS), and elastomers, such as ethylene propylene diene monomer (EPDM), natural rubber (NR) and butyl rubber (BR), are among the materials used in thermoplastic elastomer blends.