



Faculty of Manufacturing Engineering

**MECHANICAL PROPERTIES AND MORPHOLOGICAL
CHARACTERIZATION OF THERMOPLASTIC ELASTOMER
POLYPROPYLENE/ EPOXIDISED NATURAL RUBBER FILLED
RECLAIMED RUBBER**

Nur Sharafina Binti Zainol

Master of Science in Manufacturing Engineering

2015

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NUR SHARAFINA BINTI ZAINOL

**A thesis submitted
in fulfillment of the requirements for the degree of Master of Science
in Manufacturing Engineering**

Faculty of Manufacturing Engineering

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2015

DECLARATION

I declare that this thesis entitled “Mechanical properties and Morphological Characterisation of Thermoplastic Elastomer Polypropylene/Epoxidised Natural Rubber Filled Reclaimed Rubber” is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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Date : 14th May 2015

APPROVAL

I hereby declare that I have read this thesis and in my opinion this thesis is sufficient in terms of scope and quality for the award of Master of Science in Manufacturing Engineering .

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Date : 14th May 2015

ABSTRACT

Thermoplastic elastomers (TPEs) has emerged its importance in engineering products due to combination properties prevailed by its components;vulcanized rubbers and thermoplastic. This research is an effort to explore the potential of polypropylene (PP) when incorporated with epoxidised natural rubber (ENR) and filled with reclaimed rubber (RR). RR has potential to be utilised as fillers in polymeric matrices to improve mechanical properties, to reduce prices as well as to increase sustainability of petroleum based products. The effect of blend ratio (PP:ENR), mixing parameters (rotor speed, temperature and mixing time) and chemical treatment (maleic anhydride grafted polypropylene) towards mechanical properties of PP/ENR blends were first investigated. Dynamic vulcanisation of PP/ENR blends was performed using sulphur system with help of stearic acid and zinc oxide as activators. Design of experiment (DOE) via statistical method was conceived and optimum process parameters were determined based on mechanical properties of thermoplastic elastomer using response surface methodology (RSM). Regression models for responses were selected from comparison between the actual data and predicted values by the *Design Expert* 6.0.10 software. The R^2 values of more than 0.900 from analysis of variance determined that the models are accurate to represent the actual systems. Then, PP/ENR filled RR blends with/without RR were prepared by melt compounding through an internal mixer, Haake Rheomix at temperature and rotor speed of 170°C and 60 rpm and followed by compression molding using hot press. Reclaimed rubber of composition 10, 20, 30, 40 or 50 percentage was introduced during the blending process. The characteristics and mechanical properties of the blends with or without RR had been determined using tensile test, Izod impact test and hardness test on compression molded specimens. The properties were further analysed and supported with swell measurements, Fourier Transform infrared spectroscopy, differential scanning electron calorimetry and scanning electron microscopy (SEM). The PP/ENR filled with 30% RR showed optimum result in flexibility for elongation at break (Eb) and impact strength of 18.5% and 33 J/m, respectively with minimum reduction in tensile strength of only 23%. The observed microstructure of specimens via SEM showed good miscibility between rubber and plastic due to uniform dispersion of rubber components, ENR and RR in PP matrices. The Eb and impact strength were observed to increase with increasing of RR content due to significant interaction between the materials. Through optimisation of formulation of PP/ENR filled RR constituents and compatibilisation techniques, a green material with improved toughness and flexibility with significant tensile and impact properties compared to PP is achieved. This new material has potential as green alternative for existing PP based product.

ABSTRAK

Termoplastik elastomer telah menampakkan kepentingannya dalam produk kejuruteraan kerana sifat gabungan yang ditonjolkan oleh komponennya; getah tervulkan dan bahan termoplastik. Kajian ini merupakan salah satu usaha untuk meneroka potensi polipropilena (PP) apabila digabungkan dengan getah asli terepoksida (ENR) dan diisikan dengan getah tebus guna (RR). RR mempunyai potensi untuk digunakan sebagai bahan pengisi dalam matriks polimer untuk meningkatkan sifat mekanik, menurunkan harga di samping menambahkan kemampuan produk berasaskan petroleum. Kesan nisbah campuran (PP:ENR), parameter pencampuran (halaju pemutar, suhu dan masa pencampuran) dan rawatan kimia (polipropilena tergraf maleik anhidrida) terhadap sifat mekanik adunan PP/ENR disiasat pada mulanya. Pemvulkanan dinamik PP/ENR dilaksanakan menggunakan sistem sulfur dengan bantuan asid stearik dan zink oksida sebagai bahan pengaktif. Rekabentuk eksperimen menggunakan kaedah statistik digunakan dan parameter proses optimum ditentukan berdasarkan sifat mekanik termoplastik elastomer menggunakan Metodologi Permukaan Sambutan (RSM). Model regresi untuk sambutan dipilih daripada perbandingan diantara data cerapan dan nilai ramalan oleh perisian Design Expert 6.0.10. Nilai R^2 yang melebihi 0.900 daripada analisis varians menunjukkan model adalah tepat untuk mewakili sistem sebenar. Kemudiannya, adunan PP/ENR terisi/tidak terisi RR telah disediakan melalui penyebatan lebur menggunakan alat pencampur dalaman, Haake Rheomix pada suhu dan kelajuan rotor 170°C dan 60 rpm dan diikuti proses pengacuan mampatan menggunakan alat penekan panas. Komposisi RR sebanyak 10, 20, 30, 40 atau 50 peratus dicampurkan semasa pengadunan tersebut. Ciri-ciri dan sifat-sifat mekanik adunan dengan/tanpa RR ditentukan menggunakan ujian tegangan, ujian hentaman Izod dan ujian kekerasan pada spesimen acuan termampat. Sifat tersebut dianalisa dan disokong dengan lebih lanjut dengan penentukuran pengampulan, spektroskopi penjelmaan Fourier infra-merah, permeteran kalori pengimbasan kebezaan dan kemikroskopan elektron imbasan. PP/ENR terisi 30% RR menunjukkan sifat kebolehlenturan yang optimum dengan pemanjangan pada takat putus (E_b) dan kekuatan hentaman masing-masing sebanyak 18.5% dan 33 J/m, dengan pengurangan minimum dalam kekuatan tegangan pada hanya 23%. Mikrostruktur spesimen yang diperhati menggunakan SEM menunjukkan kebolehcampuran yang baik antara getah dan plastik kerana penyebaran seragam antara komponen getah, ENR dan RR dalam matriks PP. Melalui pemerhatian, E_b dan kekuatan hentaman meningkat dengan peningkatan kadar RR kerana interaksi yang bererti antara bahan. Melalui pengoptimuman formulasi komponen PP/ENR terisi RR dan teknik penserasian, suatu bahan hijau yang menunjukkan peningkatan sifat ketahanan dan kebolehlenturan yang lebih baik berbanding PP dicapai. Bahan ini mempunyai potensi sebagai alternatif hijau untuk menggantikan produk berasaskan PP yang sedia ada.

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LIST OF ABBREVIATIONS

ABS	-	Acrylonitrile-Butadiene-Styrene Rubber
DV	-	Dynamic Vulcanisation
ENR	-	Epoxidised Natural Rubber
EOC	-	Ethylene Octene Copolymer
EPDM	-	Ethylene Propylene Diene Monome
EVA	-	Ethylene Vinyl Acetate
EPR	-	Ethylene-propylene copolymer
Mah-PP		Maleic Anhydries Grafted Polypropylene
NBR	-	Nitrile Rubber
NR	-	Natural Rubber
PE	-	Polyethylene
PP	-	Polypropylene
PP-g-MAH	-	Maleic Anhydries Grafted Polypropylene
PC	-	Polycarbonate
PhR	-	Part perhundred rubber
PA-6	-	Polyamide-6
PBT	-	Polybutylene terephthalate
PVC	-	Polyvinyl Chloride
RR	-	Reclaimed Rubber

RSM	-	Response Surface Methodology
T _g	-	Glass transition temperature
TPE	-	Thermoplastic Elastomer
TPO	-	Thermoplastic Elastomeric Olefin
TPU	-	Polyurethanes
TPV	-	Thermoplastic Vulcanizates
RIPS	-	Reaction Induced Phase Separation
PCL	-	Polycaprolactone
PPO	-	Polypropylene Oxide
TETA	-	Triethyl Tetra Amine
iPP	-	Isotactic Polypropylene
SBS	-	Styrene Block Copolymer
MAH	-	Maleic Anhydride
MAH-PP	-	Maleic Anhydrides Grafted Polypropylene
HDPE	-	High Density Polyethelene
SEM	-	Scanning Electron Microscopy
DSC	-	Differential Scanning Calorimetry
FTIR	-	Fourier Transform Infrared Spectra
T _m	-	Melting Temperature
Ph-PP	-	Phenolic Modified Polypropylene
T _s	-	Tensile Strength
E _b	-	Elongation at Break

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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, lightweight, 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 (Rader, 2003). 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/cm^3 . 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 (Kenzo & Mitsuamasa, 2001).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., 2001). 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 vulcanisation of the rubber phase through dynamic vulcanisation.

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) and epoxidised natural rubber (ENR) involving the effort to reuse the reclaimed rubbers that had been vulcanized. This kind of blend could reduce the manufacturing cost and contribute to greener environment due to use of reclaimed rubbers. 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.

PP is well known of its outstanding properties in terms of electric as well as high dielectric properties under high voltage and high frequency condition of up to 30khz (Khachen et al., 1992). It is a suitable material for electrical insulator except for increasing

stiffness at high thickness. The ENR is potential candidate to increase the flexibility of PP. These properties are reflected in vulcanizates with increased oil resistance, enhanced adhesive properties, high degree of damping and reduce gas permeation (Gelling, 1991). Response surface methodology (RSM) is reported to be an effective tool for optimizing a process. (daCosta et al., 2010). RSM could save cost and time by reducing number of experiments required. Due to the high generated heat in electrical cable, a suitable heat dissipation mechanism could increase the life-time of the cable. The presence of carbon black in reclaimed rubber is hypothesised to increase the heat dissipation from the system as well as play a role as reinforcement to strengthen the material since it is targeted for outdoor's electrical cable. This research is a preliminary study to study the feasibility of PP, ENR and RR for a novel material with significant physical and mechanical properties.

1.3 Objectives

The main focus of this study is to develop a new thermoplastic elastomer (TPEs) using polypropylene (PP), epoxidised natural rubbers (ENR) and reclaimed rubber with outstanding mechanical properties. Hence, being one of the suitable solutions to use local produced and solve 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 PP/ENR filled reclaimed rubber using melt compounding and dynamic vulcanisation technique.
- b) To determine the suitable ENR to PP ratio and mixing parameters based on mechanical properties using Response Surface Methodology.

- c) To study the effect of Mah-PP as compatibiliser of PP/ENR blend.
- d) To prepare and characterise the mechanical, thermal, chemical and morphological properties of PP/ENR filled reclaimed rubber at various RR loading.

1.4 Scope of Project

This study focuses on producing a new thermoplastic elastomer material with material from the blend of polypropylene (PP), epoxidised natural rubber (ENR) and reclaimed rubber (RR). The study also focuses on the effect of blend ratios, mixing parameter and additional of maleic anhydrides grafted polypropylene (Mah-PP) as compatibiliser to the materials. The effects of earlier mentioned parameters were optimised using response surface methodology (RSM). In this study, zinc oxide and stearic acid were used as activator whereas sulphur acted as curing agent. During melt compounding, materials in the form of pallet were blended together in internal mixer to produce compound materials at parameters optimised in the first stage of the study. Crusher machine was used to refine the blends before vulcanisation and fabrication using hot press. 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 Polymer Blends

The definition of polymer blends has been described in various ways from a brief explanation to a specific one. Utracki defined polymer blend as mixture of two or more polymer or copolymer materials and stated that polymer blend as a mixture of two or more macromolecular species (Utracki, 2002). Deanin (1977), in the 70's defined polymer blends as mixture of two or more polymers intimately in single continuous solid phase. The latest, Kumar and Gupta (1998) noted that polymer blends are physical mixture of two or more polymers that are commercially prepared by mechanical mixing which can be achieved through rotor-cam compounder and screw extruder.

Generally, polymer blend could be derived into few types of polymer materials mixture through various type of mechanical compounding or solution mixing such as plastic – plastic mixture', rubber – rubber mixture, plastic – rubber mixture.

In the development of new polymeric blends, the target of combining the approving properties from each of individual material is not an easy task. This is caused by low combinatorial of entropy of mixing and immiscible of blend will increase to two-phase system, It is mostly characterised by a coarse and unstable phase morphology and poor interfacial adhesion between the phases. The poor interfacial adhesions could also be affected by differences in material characteristic such as polar-polar and non-polar attraction, amorphous or crystalline of material phase as well as the suitable blend ratio of

blend composition will result in different compounded properties. There is a number of polymer pairs that were found completely miscible to give homogeneous single phase, with properties proportional to the ratio of the two polymers in the blend, (George et al., 1999) and several of these blends were exhibited commercial importance. For example, PVC/ENR-50 blends (Chantara et al., 2001) and blends of polyphenylene-ether with polystyrene (Duff et al., 2001); (Stack et al., 2003) were reported. When two polymers are miscible to segmental level, single homogeneous phase are formed, it will exhibit a single glass transition temperature, T_g . This will give the compounder economic control over the balance of properties for different applications. Nowadays there are a few plastic technologists who develop large number of polymer blends that are immiscible but very useful, combining some of the good application properties of each polymer in the blend; they tend to use the term compatible blend. In contrast, there still are a lot of blends which are totally immiscible and incompatible when blended together. It is due to differences in material characteristics in terms of molecular weight, crystallinity, polarity and etc. As a result, these incompatible blends will exhibit poor properties in physical and mechanical. These problems can be solved by the addition of compatibiliser or filler, which consists of modification of the interfacial properties of the blend phases by using a suitable block or graft copolymer which is located at the interface between the phases of an immiscible blend and act as an emulsifying agent (Dedecker and Groeninckx, 1998).

2.2 Thermoplastic Elastomer (TPE)

Thermoplastic elastomer (TPE) is a unique class of materials which combine properties of elastomer vulcanized rubber and also able to be processed as thermoplastic (Lopez and Arroyo, 2000; Ismail and Suryadiansyah, 2002; Azman et al., 2003). This