



**STUDY OF POLYPROPYLENE/NATURAL RUBBER
THERMOPLASTIC ELASTOMER LOADED WITH PALM ASH**

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



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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:



ABSTRAK

Salah satu polimer serba boleh di pasaran hari ini ialah elastomer termoplastik (TPE), kerana mempunyai kualiti yang sama seperti plastik dan getah. Secara amnya, pengisi seperti zarah mikroskopik kerap ditambah kepada bahan elastomer untuk meningkatkan kebolehprosesan dan kualiti sebatian mekanikal sambil juga mengurangkan kos bahan. Abu kelapa sawit (OPA) adalah sisa industri minyak sawit yang berpotensi digunakan sebagai pengisi dalam TPE, sekaligus mengurangkan kesan alam sekitar sisa industri. Dalam kajian ini kesan Abu Kelapa Sawit (OPA) diisi Polipropilena/Getah Asli Termoplastik Elastomer polimer gabungan sifat fizikal, mekanikal dan morfologi telah diperiksa. Dua saiz zarah pengisi berbeza ($\leq 600 \mu\text{m}$) untuk CB dan ($\leq 100 \mu\text{m}$) untuk OPA telah digunakan dalam kajian ini. Sisa sawit PP/NR dengan 7 jenis sampel berbeza telah disediakan menggunakan teknik mencampurkan leburan bahan menggunakan Haake Rheomix OS pada suhu 200°C dengan kelajuan rotor 50 rpm selama 10 minit. Jumlah kandungan pengisi berbeza pada 0,10,20,30phr. Peningkatan dalam pemuatan abu sawit dalam komposit campuran polimer mengakibatkan peningkatan nilai kekuatan impak yang diserap, nilai kekerasan polimer komposit dan pemanjangan bahan tetapi mengurangkan kekuatan tegangan dan modulus. Mengimbas mikrograf mikroskop elektron mendedahkan bahawa pemuatan pengisi yang lebih tinggi mengakibatkan penggumpalan abu kelapa sawit dalam komposit campuran polimer. Oleh itu, penambahan sejumlah kecil pengisi sisa industri meningkatkan sifat fizikal dan mekanikal elastomer termoplastik PP/NR.

ABSTRACT

One of the most versatile polymers on the market today is thermoplastic elastomers (TPE), due to their having qualities that are like both plastics and rubbers. Generally, fillers such as microscopic particles are frequently added to elastomeric materials to improve processability and mechanical compound qualities while also lowering material costs. Oil palm ash (OPA) is the palm oil industrial waste that can be potentially used as filler in TPE, while reducing environmental impact of industrial waste. In this study the effects of Oil Palm Ash (OPA) filled Polypropylene/Natural Rubber Thermoplastic Elastomer polymer blend composite physical, mechanical, and morphological properties were examined. Two different fillers particle size ($\leq 600 \mu\text{m}$) for CB and ($\leq 100 \mu\text{m}$) for OPA were used in this study. The palm waste of PP/NR with 7 different types of samples were prepared using the melt mixing technique using a Haake Rheomix OS at 200°C with rotor speed of 50 rpm for 10 minutes. The total content of fillers varied at 0,10,20,30phr. Increase in palm ash loading in polymer blend composites resulted in increase the value of Impact toughness absorbed, hardness and elongation at break of the polymer blend composites but decreased the tensile strength and Young's Modulus. Scanning electron microscope micrographs revealed that the higher the filler loading resulted in agglomeration of palm ash in the polymer blend composites. Thus, addition of a small amount of industrial waste filler improves physical and mechanical properties of the PP/NR thermoplastic elastomer.

DEDICATION

I would like to dedicate this study to my parents, who gave the little they had to ensure I would have the opportunity of an education.

To people who supported me throughout my education, thanks for making me see this adventure through the end.



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LIST OF ABBREVIATION

TPE- Thermoplastic Elastomer

NR - Natural Rubber

PP - Polypropylene

OPA- Oil Palm Ash

MAGPP- Maleic anhydride grafted polypropylene

CB - Carbon Black

XRD - X-Ray Diffraction

SEM - Scanning Electron Microscopy

PSA - Particle Size Analysis

DSC- Differential scanning calorimetry

phr – parts per hundred



CHAPTER 1

INTRODUCTION

The background of study, problem statements, research objectives, and scope of the study are all covered and discussed in this chapter.

1.1 Background of study

A polymer is a natural or synthetic substance made up of macromolecules, which are multiples of monomers and are smaller chemical units. Polymers, such as proteins, cellulose, and nucleic acids make up many of the components present in living organisms. They are also the foundation for minerals such as diamond, quartz, and feldspar, as well as man-made materials such as concrete, glass, paper, plastics, and rubbers.

A multitude of techniques are used to make synthetic polymers. Many simple hydrocarbons, such as ethylene and propylene, may be transformed into polymers by adding one monomer after another to the growing chain. Polyethylene is an additive polymer made up of ethylene monomers that are repeated. It might have up to 10,000 monomers linked together in long coiled strands. Polyethylene is crystalline, transparent, and thermoplastic, which means that when heated, it softens. Coatings, packing, moulded components, and the production of bottles and containers all employ it. Polypropylene (PP) is crystalline and thermoplastic, although it is more difficult to work with than polyethylene. Its molecules might be made up of anything from 50,000 to 200,000 monomers. This substance is employed in the textile sector as well as in the production of moulded products.

One of the most versatile polymers on the market today is thermoplastic elastomers (TPE). A physical composition of polymers creates thermoplastic elastomeric materials (a plastic and a rubber). They have qualities that are like both plastics and rubbers. Because TPE materials are

generated only by physical mixing of a thermoplastic and an elastomer, there is no chemical or covalent linkage between the two, hence have distinct features. This characteristic has ushered in a new era in polymer research. Thermoplastic elastomers have established themselves as an important component of the polymer industry. They are applied in a variety of products, including adhesives, footwear, medical equipment, vehicle parts, and household items (Amin & Amin, 2011). TPE manufacturers' continuing product innovation has aided the development of the downstream application market in recent years. They have largely replaced traditional synthetic rubber in a variety of applications, and their use has grown dramatically.

Synthetic rubber and natural rubber (NR) are the two primary forms of rubber. Natural rubber is harvested from mature rubber trees that must be grown in damp, hot conditions. Natural rubber is made up of polymers of the chemical compound isoprene with tiny impurities of other organic compounds when it is first made. Thailand and Indonesia are two of the world's most important rubber producers. Elastomers are polyisoprenes that are used as natural rubbers in a variety of applications. Latex from rubber plants or other sources is the most common kind of rubber presently available. Latex is a thick, chalky white colloid that is removed from the bark by making slits in the bark and collecting the fluid in vessels, a process is termed as "tapping". The latex is next developed into rubber, which is suitable for industrial use. In the collecting cup, latex is left to coagulate. Before being marketed, the coagulated chunks are collected and dried. Natural rubber is used in a broad range of goods and applications, both alone and in combination with other materials. In most of its practical forms, it has a high stretch ratio and exceptional durability, as well as being waterproof.

Fillers, such as microscopic particles and short fibres, are frequently added to polymeric materials to improve processability and mechanical compound qualities while also lowering material costs. The particle size distribution, surface area, shape, and surface chemistry all had an impact on the filler behaviour in the compound. Filler represents the largest volume category of material added to polymers and may be subdivided into reinforcing or extending type. Reinforcing fillers increase hardness and modulus, improve tensile and tear strength, and provide abrasion resistance. The purpose of extending filler is to lowest cost without sacrificing key performance properties. Many compounds use a mixture of both reinforcing and extending fillers. Ash is one potential component that has recently gotten a lot of attention. Numerous studies have been conducted using ash as a filler in polymer composite, including studies of fly ash (Khalaf, 2015), rice husk ash (M. S. Ismail & Waliuddin, 1996) Kenaf bast (Anuar & Zuraida, 2011) and oil palm ash (Bhat & Abdul Khalil, 2011).

1.2 Problem statement

Because scrap and rejects may be recycled, TPEs allow for higher material usage. Furthermore, because no compounding or vulcanization is necessary, productivity is excellent, and they may be simply prepared by internal mixers or extrusion. Blend compositions vary, resulting in materials with a wide range of properties. Polypropylene (PP) has been widely employed in the form of moulded products, films, and sheets due to its excellent moulding processability and strong mechanical qualities. Due to its distinctive features, such as a high melting point, a low density, a high chemical solvent capacity, and a strong resistance to heat, PP offers a wide range of uses. Additionally, PP has a low impact strength, limiting its applicability. By adding certain elastomers to PP, it may be toughened while keeping stiffness, strength, and processability. The current polymer industry relies heavily on the incorporation of recycled rubber (RR) into polymers, such as rubber powder combined with PP, not only for the creation of new materials but also for practical recycling (Anuar & Zuraida, 2011).

The importance of inexpensive items in achieving long-term economic growth and greater human development is underscored by rising costs. The use of bioresource materials, particularly those derived from clean indigenous resources, has become more important than ever. In Malaysia and Thailand, oil palm ash (OPA) is a common agricultural solid material. According to Foo and Hameed (2009), the potential output of oil palm ash in Malaysia alone has been estimated at four million tonnes per year, a figure that has sparked widespread criticism and protests, owing to its persistent, carcinogenic, and bio-accumulative impacts. Palm oil waste may be converted into an alternative fuel for steam generation in palm oil factories, resulting in the creation of oil palm ash (OPA) as a by-product. Following the combustion process, the OPA is often disposed of in a landfill or field, creating environmental concerns. To resolve this concern, oil palm ash should be utilised and converted into a valuable product.

Therefore, this research is carried out to reduce the cost to handle the industrial waste. The idea is by filling the matrix with available inexpensive waste derived materials such as oil palm ash. By utilizing the waste, it will eventually give added value to the waste and reduce cost to handle the industrial waste. Besides that, it also increased the properties of PP/NR thermoplastic elastomer.

1.3 Objective

The purpose of this study is to:

- i. To evaluate the effect of NR content to the physical and mechanical properties of PP/NR blend composite.
- ii. To study the influence of oil palm ash (OPA) addition on the physical and mechanical properties of the polymer blend composite.
- iii. To correlate the morphological properties of the waste derived filled PP/NR blend composites with their mechanical behaviour.

1.4 Scope of research

In this study, the effects of the OPA loading have on the processability, physical and mechanical properties of PP/NR thermoplastic elastomer are investigated. The explanation can be described as below:

- i. Thermoplastic elastomer composites are prepared using various composition of materials consists of polypropylene, natural rubber, carbon black and oil palm ash.
- ii. Samples are prepared by using melt mixing method via internal mixing and hot compression moulding.
- iii. Raw ashes underwent Particle Size Analysis and X-Ray Diffraction (XRD), Samples of polymer blend composite underwent various types of tests which are Tensile test, impact test, hardness test, and Scanning Electron Microscopy (SEM) to study the topography of the sample surface.

CHAPTER 2

LITERATURE REVIEW

The section revises the scope of earlier research works related on polypropylene/natural rubber thermoplastic elastomer filled with different types of fillers from a few years back.

2.1 Thermoplastic

Thermoplastics are readily melted and softened with heat, moulded and reformatted, and hardened with cooling. This technique is reversible or repeatable. Thermoplastics, unlike thermosetting materials, do not form cross-links. This demonstrated that they could reform their shape. Additionally, it may be amorphous or semi-crystalline, each of which has distinct features. Amorphous thermoplastics lack order, and their lengthy chains of molecules may become completely intertwined and irregularly structured. Additionally, amorphous thermoplastics have a temperature range that varies between a hard and moderately brittle state and a fluid, rubbery state. The temperature at which the states shift is referred to as the glass transition temperature (T_g), which is the optimal temperature for amorphous plastics materials to be above the polymer chains. This enables information to pass over one another for processing. Table 2.1 shows the type, properties, and uses of thermoplastics.

Table 2. 1: Classification of thermoplastics

Name	Properties	Uses
Polypropylene	Lightweight, durable yet readily scratched, tough, excellent resistance.	Medical equipment, laboratory equipment, rope, and cooking equipment

Polymethyl methacrylate (Acrylic)	Stiff, tough yet readily scratched, resilient, brittle, excellent electrical insulator, polishes well.	Canopies and windows for aircraft, as well as coverings for automobile headlights.
Polystyrene	Lightweight, rigid, opaque, brittle, and resistant to water.	Toys, particularly model kits, packing and storage container.
Low density polythene (LDPE)	Tough, chemically resistant, flexible, and rather soft.	Packing, particularly bottles, toys, and film packaging.
High density polythene (HDPE)	Sturdy, hard, and sterilizable.	Bottles, tubes, and domestic equipment made of plastic.

2.2 Natural Rubber

Natural rubber is an elastic substance contained in the milky juice (latex) of any various plants of the genus *Hevea*, especially the rubber tree (Raghavan Vijayaram, 2009). Latex consists of an aqueous dispersion of cis-1, 4-polyisoprene (C_5H_8)_n an unsaturated, high molecular weight hydrocarbon for commercial purposes. This latex is coagulated by adding acetic or formic acid or sodium hexafluorosilicate and subsequently concentrated by evaporation or centrifugation. The processed latex is usually dried and converted into sheets of crude rubber. Besides, unvulcanised natural rubber has poor mechanical properties and chemical and environmental resistance, but these are significantly improved by crosslinking (or vulcanizing). Usually, the improvement through treatment with sulphur or special chemicals. Natural rubber (NR) is the second largest type of rubber utilized in the world (Raghavan Vijayaram, 2009).

The demand for NR-based products is considerable owing to the amazing features of NR that are not found in other materials. NR maintains superior mechanical qualities by the inclusion of fillers, a vulcanizing system, and other specialist compounds. NR was a widely utilised polymer in a variety of commercial and technical applications. NR is unique among elastomers in terms of strength qualities due to its strain-induced crystallisation behaviour. NR is stronger, generates less heat, and performs better at low temperatures. Natural rubber is well-known for its crystallisation behaviour when subjected to strain (H. Ismail et al., 2015). It then demonstrates exceptional qualities, including green strength. Several studies have reported that

blending of NR with PP resulted in characteristic improvement, such as increased thermal stability, decreased fracture elongation, and increased Young's modulus (Ginting et al., 2019). Additionally, NR is resistant to abrasion and has high hysteric characteristics. However, NR must be enhanced in order to attain the improved hardness characteristics with the filler.

2.2.1 Properties of natural rubber

Natural rubber mixes extreme tensile strength with exceptional fatigue resistance. It has exceptional green strength and tack, which means it can adhere to itself and to other materials, making fabrication simpler. One of its disadvantages is that it is somewhat resistant to environmental deterioration caused by heat, light, and ozone.

Natural rubber adheres very well to brass-plated steel cable, making it perfect for use in rubber tyres. It has a low hysteresis, which results in less heat production, hence preserving the integrity of new tyres and extending their retreadability. Natural rubber has a low rolling resistance, which contributes to its increased fuel efficiency. It resists slicing, chipping, and ripping well (Raghavan Vijayaram, 2009).

2.2.2 Rubber processing methods

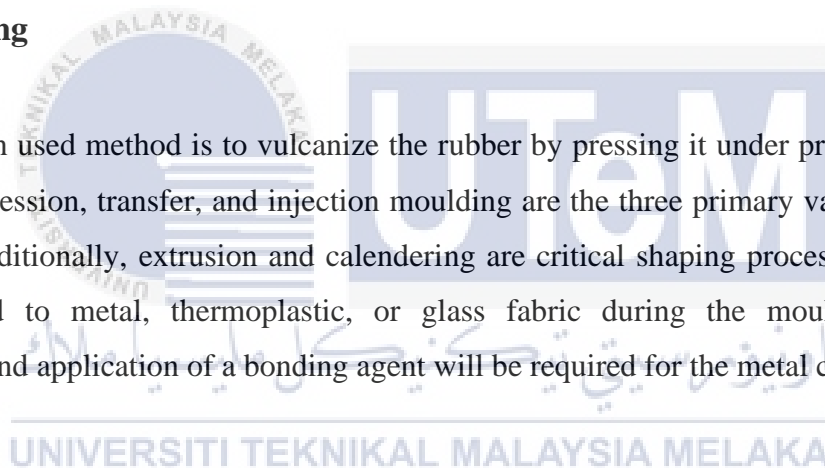
The manufacturing procedures for rubber goods made from latex concentrate and dry rubber are fundamentally different, even though both materials need shape and vulcanization, and parts of the shaping operations are shared between the two materials. By and large, the latex and dry rubber sectors are diametrically opposed. The manufacture of dry rubber products is separated into three main operations which are mixing, shaping and vulcanization (Raghavan Vijayaram, 2009).

2.2.2.1 Mixing

The mixing procedure is often carried out in large internal mixers capable of handling batches weighing up to 200 kg in two minutes. This process has two purposes: first, it softens the rubber, a process often referred to as mastication; and second, it admixes the rubber with compounding components such as fillers, vulcanizing agents, protection agents, and blends with other synthetic rubbers. This is referred to as compounding. Following mixing, the compounded rubber transforms into plastic and is ready to shape. This may be accomplished in several methods and is commonly paired with vulcanization, a process in which rubber is changed from its plastic state to a strong, highly elastic substance by a chemical reaction at a high temperature (Raghavan Vijayaram, 2009).

2.2.2.2 Shaping

The most often used method is to vulcanize the rubber by pressing it under pressure in a hot mould. Compression, transfer, and injection moulding are the three primary variations of the procedure. Additionally, extrusion and calendering are critical shaping processes. Rubber is often attached to metal, thermoplastic, or glass fabric during the moulding process. Pretreatment and application of a bonding agent will be required for the metal component.



2.3 Fillers

Reinforcements, fillers, and reinforcing fillers are all terms used to describe additives for polymer composites (Fillers et al., 2010). The modulus and strength of the polymer are frequently increased by the reinforcements, which are significantly stiffer and stronger than the polymer. As a result, their principal job may be to modify mechanical properties, even though their presence might have a considerable impact on thermal expansion, transparency, thermal stability, and other factors.

Filler is a relatively inert, finely split component such as calcium carbonate, carbon or graphite, glass spheres, kaolin, silica, or wood flour in polymer engineering (Harald, 2004). To enhance bulk, the filler is added into a polymer in relatively large concentrations. It has also been proved to reduce costs and alter physical, mechanical, thermal, electrical, and other qualities. To be

successful as a reinforcing agent in rubber, a filler must be properly dispersed and provide a huge polymer filler interface as well as enhanced surface adhesion between rubber and filler (Munirah et al., 2015).

Chemical compatibility is required for filler dispersion. Rubber processing characteristics and network structure will be influenced by the presence of reinforcing fillers. Particulate fillers are frequently used in rubber to improve a variety of desired qualities for a variety of applications while also lowering costs. Rubber reinforcement by rigid entities such as carbon black, clays, silicates, and calcium carbonate are one of the most important phenomena in material science.

These fillers or reinforcing aids are added to rubber formulations to maximise qualities that meet a certain service application or set of performance standards. The selected active fillers and their amount, which generate changes in the physical characteristics of rubber, are now of vital importance, notwithstanding the initial purpose of lowering the cost of the moulding compounds.

2.3.1 Conventional filler

Conventional fillers, which are precisely layered silicates, have been utilised as efficient reinforcing agents in rubber formulation to generate conventional composites for a range of purposes since the turn of the twentieth century (Jayaraj et al. n.d.). Layered silicates as nanofillers are currently being used in rubber with significant improvements in physical properties thanks to new technology. Filler is also known as reinforcing agent which defined as a material such as carbon black, kaolin or zinc oxide in finely divided form is added to elastomers or plastic usually in relatively high percentages to increase strength, hardness and abrasion resistance (Harald 2004). Particulate fillers have been extensively utilized as additives in many rubber products (Pangamol et al., 2017).

Rubber may be reinforced with two kinds of fillers: reinforcing and non-reinforcing fillers. A reinforcing filler is favoured over a non-reinforcing filler in engineering applications due to its ability to improve rubber characteristics. Silica and carbon black are two reinforcing fillers that are used in the rubber industry to enhance the mechanical qualities of rubber products. Rubber containing these fillers has increased tensile strength, excellent abrasion resistance, a high

modulus of elasticity, and a high degree of hardness. Silica used in the rubber sector is precipitated silica, which is made by precipitating an aqueous sodium silicate solution, and fume silica which is formed by a pyrogenic process.

2.3.1.1 Carbon Black

Rubber compound fillers typically have multipurpose. Some fillers like carbon black are used to improve the properties of rubber compounds by acting as reinforcement agent. On the other hand, some fillers such as calcium carbonates cannot increase the properties of rubber compounds but are still used to reduce the cost of the tire. Absence of any fillers in the rubber compound, would result in soft and easy to break rubber material. Filler addition typically results in stiffer rubber resistant to abrasion and deformation. Among all kinds of rubber fillers, carbon black is the most traditional and commonly used reinforcing filler. Carbon black is collected from the carbon black oil furnace process.

The standard D1765, published in the "Annual Book of ASTM Standards, Volume 9.01" in 2000, was used to classify carbon black. The ASTM D24 Committee on Carbon Black recommended using a four-character nomenclature scheme to designate carbon blacks used for rubber fillers. The first letter in this scheme denoted the effect of carbon black on the cure rate of a typical rubber composite including that carbon black as a filler. The second character in the naming scheme denoted the carbon black's average surface area, which was determined using the nitrogen surface area technique. Usually, the third and fourth characters ranged from 0 to 4. They were given digits at random. Take the example of N234, the first letter "N" meant that this carbon black does not have specific influence on the cure rate of the rubber compound. The second character "2" indicated that the surface area is about 100 to 120 m^2/g (Table 2.2) as given by the D1765 standard.

Table 2. 2: The relations between second character and average N^2 surface area (m^2/g) in the carbon black nomenclature system.

Second Character	Average N^2 surface area (m^2/g)
0	>150
1	121 to 150