

THE EFFECT OF OIL PALM TRUNK FIBER ON CHARACTERIZATION OF
NATURAL RUBBER

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BORANG PENGESAHAN STATUS TESIS♦

JUDUL : THE EFFECT OF OIL PALM TRUNK FIBER ON
CHARACTERIZATION OF NATURAL RUBBER.

SESI PENGAJIAN : 2010/2011

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**THE EFFECT OF OIL PALM TRUNK FIBER ON CHARACTERIZATION OF
NATURAL RUBBER**

MOHAMAD AZLAN SYAH BIN DZULKAFLI

**A thesis submitted in fulfillment
of the requirements for the award of the Degree of
Bachelor of Chemical Engineering**

**Faculty of Chemical & Natural Resources Engineering
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DECEMBER 2010

“I hereby declare that I have read this thesis and in my opinion this thesis has fulfilled the qualities and requirements for the award of Degree of Bachelor of Chemical Engineering”

Signature :

Name of Supervisor : Mohd Bijarimi Bin Mat Piah

Date : 3 December 2010

I declare that this thesis entitled “*The Effect of Oil Palm Trunk Fiber on Characterization of Natural Rubber*” is the result of my own research except as cited in 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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Name : Mohamad Azlan Syah Bin Dzulkaflī

Date : 3 December 2010

*Dedicate to my beloved parents,
and also to my brothers and sisters for their
inspiration and encouragement*

ACKNOWLEDGEMENT

Alhamdulillah, praise to ALLAH SWT for His helps and guidance that I finally able to complete this Projek Sarjana Muda (PSM).

First of all, I would like to convey my appreciation towards my parents, Mr. Dzulkafli Bin Ali and Mdm. Norizan Bin Abu Hassan, for their prayers, encouragement and supports that inspiring me to complete this thesis. I really appreciate their patience, support and sacrifice throughout my studies and my research.

Special thanks also to my supervisor, Mr. Mohd Bijarimi Bin Mat Piah for his supervision and guidance during conducting the experiment and advice in order to complete this thesis. I appreciate his help in guiding me to overcome my research and other problems.

I grateful acknowledge the Faculty of Chemical and Natural Resources Engineering for the supports which enabled this work to be carried out. Thank all the technicians and staff in the Faculty of Chemical and Natural Resources Engineering Laboratory for their help in completing my research.

Last but not least, thanks to my dear friends for their encouragement, support and helps. Also for my family members that inspire me during completing this thesis.

ABSTRACT

The strength of a material to stress depends on the chemical bonds found in the chemical structure of the material itself. This thesis deals with the effect of different filler loading on the natural rubber vulcanizates so that the dynamic mechanical properties of vulcanized natural rubber can be obtained. The objective of this thesis is to study the effect of different filler loading or amount of filler loaded on natural rubber that is Standard Malaysian Rubber 10 (SMR-10). For this study, natural filler that is oil palm trunk fiber which is varies from 0 to 50 phr have been used as an additive to strengthen the chemical bonds of natural rubber as well as other chemicals that were added to natural rubber. Rubber compounds filled with natural fillers and other chemical additives is prepared using a two roll mill and the characteristics of the mechanical properties are studied and analyzed to determine the effect on the strength of natural rubber when natural filler is added as filler. In this thesis, there is description on background of the natural rubber and the characteristics of cured natural rubber after being treated by fillers. Most commonly used fillers by industry for curing natural rubber is carbon black and calcium carbonate which is can be categorized by black filler and non-black filler. But for this study, these materials will be used as a reference and oil palm trunk fiber is used as replacement and the effects of natural fillers are studied and analyzed. The functions for each material that is stated above might be vary based on their physical and chemical properties data but all fillers generally increase the tensile strength and modulus of vulcanized natural rubber into particular level.

ABSTRAK

Kekuatan sesuatu bahan terhadap tekanan bergantung kepada ikatan kimia yang terdapat didalam struktur kimia bahan itu sendiri. Tesis ini berkaitan dengan kesan perbezaan jumlah kemasukan bahan pengisi semula jadi pada getah asli yang divulkanasikan supaya sifat-sifat kekuatan dan sifat mekanik dinamik dari getah alam yang divulkanasikan boleh diperolehi. Tujuan tesis ini adalah untuk mempelajari kesan perbezaan jumlah kemasukan bahan pengisi semula jadi ke atas getah asli iaitu Standard Malaysian Rubber 10 (SMR-10). Untuk kajian ini, bahan penambah asli iaitu fiber batang kelapa sawit telah digunakan sebagai bahan penambah untuk menguatkan ikatan kimia pada getah asli serta bahan kimia lain yang turut ditambah bersama ke atas getah asli. Sebatian getah yang dicampur dengan bahan pengisi asli serta bahan penambah lain disediakan menggunakan 'two roll mill' dan ciri-ciri terhadap sifat-sifat mekanikal dikaji serta dianalisa untuk melihat kesan terhadap kekuatan getah asli apabila bahan pengisi semula jadi ditambah sebagai bahan pengisi. Dalam tesis ini, terdapat huraian tentang latar belakang getah asli dan ciri-ciri getah asli yang telah diperkuatkan selepas ditambah dengan bahan penambah asli iaitu fiber batang kelapa sawit. Antara bahan pengisi yang paling kerap digunakan oleh industri untuk memperkuatkan getah alam adalah karbon hitam dan kalsium karbonat yang boleh dikategorikan sebagai bahan penambah hitam dan bukan hitam. Tetapi untuk kajian ini, bahan ini akan digunakan sebagai rujukan dan fiber batang kelapa sawit digunakan sebagai pengganti dan kesan-kesan bahan pengisi asli akan dikaji dan dianalisa. Fungsi untuk setiap material yang dinyatakan di atas mungkin berbeza-beza dari segi sifat-sifat fizik dan kimianya tetapi semua bahan pengisi ini secara umumnya berfungsi meningkatkan modulus dan kekuatan tensile getah asli yang divulkanasikan ke suatu tahap tertentu.

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LIST OF ABBREVIATIONS

NR	-	Natural rubber
SBR	-	Styrene-Butadiene rubber
IR	-	Isoprene rubber
EPDM	-	Ethylene-Propylene rubber
ENR	-	Epoxidised natural rubber
SMR	-	Standard Malaysian Rubber
TCR	-	Technically Classified Rubber
phr	-	Part per hundred rubber
OPTF	-	Oil palm trunk fiber
RHP	-	Rice husk powder
WRHA	-	White rice husk ash
BRHA	-	Black rice husk ash
6-PPD	-	N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylene-diamine
CBS	-	N-cyclohexyl-2-benzothiazyl sulphenamide
TMQ	-	2,2,4-trimethylbutyl-1,2-dihydroquinoline
TMTD	-	Tetramethylthiuram disulphide
FKKSA	-	Fakulti Kejuruteraan Kimia & Sumber Asli
FKP	-	Fakulti Kejuruteraan Pembuatan
UMP	-	Universiti Malaysia Pahang

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CHAPTER 1

INTRODUCTION

1.1 Introduction of Natural Rubber

Natural rubber is a chemical compound that produced from latex. Usually latex is collected from the tapped rubber tree and also can be produced by synthetically. Both the natural and synthetic rubber is being used widely in many applications and products. But there is a different between natural rubber and synthetic rubber especially their strength or its elasticity. Natural rubber is known to exhibit numerous outstanding properties such as good oil resistance, and low gas permeability (Kaushik *et al.*, 2010). It is also more elastic than the synthetic rubber and because of its special properties made it preferred raw material for manufacturers that producing rubber products.

Rubber tree in Malaysia is brought from Brazil by Sir Henry Wickham in 1876. He gathered thousand of rubber tree seeds and these seeds were shipped to Sri Lanka, Indonesia, Singapore, and Malaysia. Then Malaya (Malaysia) was later to become the biggest producer of rubber at that time. New plantations areas also have been started in Thailand, Philippines, Africa, and Europe to support insufficient raw material in Malaysia and Indonesia. Today, Malaysia, Indonesia and Thailand are among the biggest rubber producers and produces almost 80% of world's natural rubber.

1.1.1 History and Development

It began in 1492 when Columbus sailed to the New World, he saw nothing unusual except for one ball. It bounces when dropped to the ground and made from the gum of a tree. Although they did not aware about it, but Columbus and his friends were the first Europeans to found this unique material that is rubber. The name was given by an eminent English chemist, Joseph Priestley when he found that the ability of the material to 'rub out' pencil marks and since it has been called rubber because of its function to 'rubbing out' pencil marks.

In early 1800's, a machine which is could soften, mix and shape the rubber was invented by Thomas Hancock in London. This effort has been made the rubber as a product that is useful for daily life by dissolving the rubber into the machine. Cloth can be functioning as raincoat by coating it with rubber solution and it will become waterproof. The most important discovery in developing the strength of rubber was made by Charles Goodyear in 1839. He accidently found that when raw rubber mix with sulphur in the presence of heat, the elasticity or its strength could be improved. This process called vulcanization process and the product produced called vulcanized rubber.

Among the first product to be made from rubber were conveyer belts, hose, flooring and footwear as the demand grew every year. In the middle of 19th century, rubber came from Brazil, where the hot wet climate suited to rubber tree to grow but it was found difficulty to collect it from the jungle. Then it makes the rubber producers are forced to grow rubber in a suitable place to meet the demands of the market.

Sir Henry Wickham in 1876 collected and brought about 70,000 seeds from the wild rubber tree and was shipped from Brazil to London. These seeds were rushed to Kew Gardens and planted there in specially prepared hot houses. The small amounts of survived seeds were taken in 1877 to Ceylon and Malaysia as well other countries around South-East Asia.

In order to increase the plantation area in Malaysia, large areas of forest were cut down so that rubber plantation can be expanded. Moreover, the rubber trees generally flourish and grow quickly. At the end of the 19th century, about 2500 hectares of rubber plantation were planted in Asia. During that time, Henry Ford was invented his motorcar and need supply of rubber to make the tyre. The supply from South America plantation could not able to produce enough rubber and the world depend the supply from plantation in Asia to produce as much as it can. In 1910, it was about half million hectares of rubber were planted around the world and Asia had become the biggest suppliers of raw rubber including Malaysia.

Motoring was introduced to enhance the productivity of rubber compound around the world. But the spread of motoring device still could not enhance the production of raw rubber and the supply still not enough to meet the market demand. Hence scientist have developed synthetic rubber which is rubber is made from petroleum industry and have similar characteristics with natural rubber. For certain product like tyre, only natural rubber can be used compared to the synthetic rubber because of the special properties that make natural rubber stronger and more elastic.

In Malaysia, almost all states have the rubber plantation including Sabah and Sarawak. Altogether Malaysia produces about 20% of the world's natural rubber production. Most of the rubber plantation in Malaysia is grown on big estates that were owned by various companies and each of it can cover over a thousand hectares. Overall, Malaysia has about 1.7 million hectares of rubber plantation today.

1.1.2 Properties and Elasticity

Natural rubber is a good example of an elastomer. Elastomer is materials that can readily stretched and return to its original shape when released without broken. This is because of its special properties that make it become different material compared to wood, plastic, and metal. The structure of natural rubber can be defined as combination of monomers 2-methylbutadiene and produce polyisoprene. The chemical structure is shown in figure below.

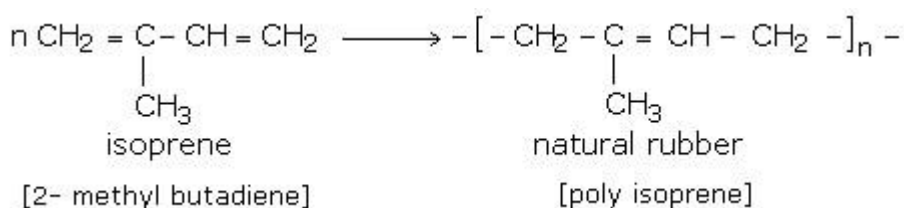


Figure 1.1: Chemical structure of isoprene and polyisoprene

This process called polymerization and 2-methylbutadiene as monomer and polyisoprene as the polymer or product but this process does not occur chemically since the natural rubber is a natural compound and is obtained from rubber tree. The synthetic rubber is produced chemically from petroleum industry and called styrene-butadiene rubber (SBR). It is understand that, without the addition of additives or reinforcing agent, natural rubber will not able to absorb such a huge amount of stress or pressure at high temperature. Besides that, natural rubber without addition of filler have very low physical strength and no practically useless (Bijarimi *et al.*, 2010).

The addition of filler leads to an increase of the mechanical properties, like elastic modulus and tensile strength. This reinforcement effect comes from the filler–filler and filler–rubber interactions, and it happened for both chemical and physical

(Omnès *et al.*, 2008). Table 1.1 and 1.2 below show the physical properties and general information of natural rubber.

Table 1.1: Physical properties of natural rubber

Quantity	Value	Unit
<i>Young's modulus</i>	1 - 5	MPa
<i>Tensile strength</i>	20 - 30	MPa
<i>Elongation</i>	750 - 850	%
<i>Thermal expansion</i>	6.7	e-6/K
<i>Thermal conductivity</i>	0.13 - 0.142	W/m.K
<i>Specific heat</i>	1880 - 1880	J/kg.K
<i>Glass temperature</i>	-70	°C
<i>Density</i>	910 - 930	kg/m ³

Rubber is different from other material or ordinary solids. The special characteristics of rubber are listed below:

- i. It is easily deformed by weak forces.
- ii. It can be deformed heavily: it can be elongated by two to three times its original length.
- iii. It becomes warmer when extended rapidly, and shrinks when warmed.

The reason why natural rubber and most elastic materials such as metal in springs is elastic and can return to its original shape because of its bond distortions. When force is applied, the bond length will stretched and deviate and this mechanism

will cause the strain energy is being stored. Hence, after the force is released, the strain energy stored will cause the bond attract each other and return to the basic shape

Table 1.2: Briefing description of natural rubber

Category	Description
<i>Specific Gravity</i>	Its specific gravity is 0.915.
<i>Physical</i>	Crude rubber is a tough and an elastic solid. It becomes soft and sticky as the temperature rises.
	Pure rubber is a transparent and amorphous solid
<i>Strength</i>	Natural rubber usually can be stretched because of its elasticity.
	Raw natural rubber has low tensile strength and abrasion resistant
<i>Absorption</i>	It absorbs large quantities of water
	It is insoluble in water, alcohol, acetone, dilute acids and alkalis
	It is soluble in ether, carbon disulphide, carbon tetrachloride, petrol and turpentine

1.1.3 Usage and Application of Rubber

Basically, natural rubber is a very versatile material. It can be stretched and compressed for certain limit according to their strength. This characteristic make the rubber as a special compound compared to the metal or wood. Among most popular rubber product are tyre, gloves, footwear, car parts like timing belt and gasket. Nowadays, the increasing of production of styrene-butadiene rubber (SBR) and has takeover the application of natural rubber slowly. But natural rubber becomes the preferable raw material in production of tyre especially for truck and aircraft. This is due to its poor conductor of heat and ability to absorb a huge amount of stress as well of its strength of tensile. This special properties has made the usage of natural rubber has been increase. Besides that, natural has been used widely in production of sanitary rubber

product and surgical gloves due to its self-reinforcing properties and strain crystallization.

1.2 Type of Natural Rubber

Generally, there are several types of rubber nowadays and each of it has being used for particular product according to the characteristic of the product. The most common elements in rubber compound are carbon and hydrogen and most of the chemical structure for all types of rubber are mainly built from these elements. For synthetic rubber, these elements were obtained from the petrochemical industry. Below is briefing on characteristic about several types of rubber and its usage.

1.2.1 Natural Rubber (NR)

Malaysia, Thailand and Indonesia are among the biggest natural rubber supplier which is contributing to produce 80% of world consumption. The tropical climate with hot and humid along year has made these countries the most suitable places to plant rubber trees. Rubber plantation also can be found in other countries like South America and Africa.

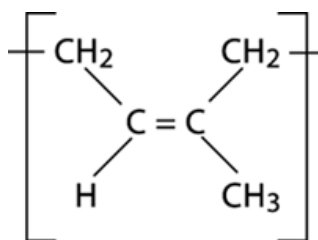


Figure 1.2: Chemical structure of natural rubber

In order to obtain the latex, a cut is made through the trunk in descending way so that the latex will flow down and drips into the cup. A tree with the best yield can give about 30-35 grams of rubber per day. The latex in the cup is accumulated into the containers and transported to the rubber factory. To promote the coagulation, acetic acid is added with latex and base is used for reversible process. Among all types of rubber, natural rubber has the best quality in terms of its elasticity and strength. It has a very good resistance to fatigue and abrasion compared to the synthetic rubber for example.

The most common and main product that was used natural rubber is tires especially heavy-duty tires. Besides that, natural rubber also is used to produce vibration dampers, springs and bearings. For special purposes it is used in hoses seals, conveyor belts, coated fabrics and other products.

1.2.2 Styrene-Butadiene Rubber (SBR)

In order to meet the demand from market especially in automobile industry, scientist trying to create alternative rubber compound that can be produce chemically. Starts since then, styrene-butadiene rubber (SBR) is produced to cover natural rubber as the main raw material. SBR the most cheaply and common synthetic rubber is derived from the example of the manufacturing principles. The raw material to produce SBR is derived from petroleum which is undergoing distillation process and produce styrene and butadiene. These two products were used as raw material to produce SBR. Figure below shows the chemical structure of SBR.

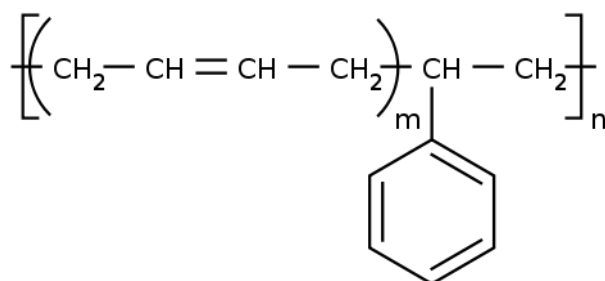


Figure 1.3: Chemical structure of styrene-butadiene rubber

For synthesis of SBR, styrene and butadiene is allowed to react together for some period of time. The new product produced consists of 25% of styrene and the remaining was butadiene. The result is a synthetic rubber called SBR that have similar properties as natural rubber. The only advantage of SBR compared to the natural rubber is heat resistance is better than natural rubber but low temperature flexibility and the tensile strength are weaker than natural rubber.

As natural rubber, styrene-butadiene rubber is used raw material for almost rubber product except for high requirement of chemical and physical conditions of product. Besides that, styrene-butadiene rubber is used to cover different types of hose, pneumatic tires, shoe heels and soles, gaskets and even chewing gum.

1.2.3 Isoprene Rubber (IR)

It almost similar to natural rubber from the chemical structure's aspect but there is differences between these two compounds. Isoprene rubber does not contain fatty acids, proteins and other elements that are present in natural rubber. Generally, these two compounds have slightly differences form the physical and chemical properties and isoprene rubber is weaker compared to the natural rubber, but in principle, these two compounds are very alike. The application of isoprene rubber is same as natural rubber and it is being used to cover the consumption of natural rubber.

<i>Volatile matter</i>	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8	0.8
<i>Wallace rapid plasticity</i>	-	-	35	30	-	-	30	-	30
<i>Plasticity retention index</i>	60	60	60	60	50	50	50	40	40

1.3 Problem Statement

The strength of vulcanizates natural rubber usually is determined by types of fillers used. Generally, carbon black and calcium carbonate is used as fillers and amount of filler loading can affect the chemical and physical properties of the end product and give different results. Then, in this research natural filler that is oil palm trunk fiber is used and amount of filler loading is manipulated so that it can give variety of result. The effect of natural filler is studied and analyzed and was compared to the result as carbon black functioned as filler.

1.4 Objectives of the Research

The objective of this research is to study the effect of filler loading on vulcanizates natural rubber and the range of filler loading varies from 0 to 50 phr. Generally, if the amount of filler loading is increased, the amount of material that would be reacted will be increased and this will cause the reaction become more efficient and faster. The function of addition of filler is to reinforce the strength of vulcanizates natural rubber and enhance its elasticity so that it will be more flexible. Below is the summary of the objective of this thesis.

- i. To study the characterization of natural rubber that filled with oil palm fiber and other additives

- ii. To analyze the mechanical properties of natural rubber after being treated with filler and other reinforcing agent

1.5 Scope of Study

This research is focus on the effect of filler loading used in rubber compounding and how this variable could influence the mechanical properties of natural rubber that is Standard Malaysian Rubber 10 (SMR-10). Oil palm trunk fiber is used as natural filler instead of carbon black as the standard filler. Five different parts of filler that is 0, 15, 30, 40 and 50 phr is used to diversify rubber compounding with the total compound of 250 grams. These five samples with different filler loading were incorporated with natural rubber and tested using several types of testing. The result which is cover the effect of filler loading will be recorded and analyzed. Numbers of parts of filler are manipulated and the other material part including SMR-10 remains unchanged so that the different outcomes of the samples can be obtained.

1.6 Rationale and Significant

The important of this research is specifications of natural rubber can be modified or adjusted according to the required or desired properties of end product. The specifications also can be determined based on type of filler used either natural filler or standard filler like carbon black. For example, a client need a rubber product such as tyre or footwear with particular specifications and the engineers will decide most suitable type of filler and amount of filler can be used to suit with desired specifications from the client. Besides that, the life span of the natural rubber product can be extended and improving the qualities.

CHAPTER 2

LITERATURE REVIEW

2.1 Fillers

Filler is a compound that is used to enhance the quality of rubber products. Generally, filler has no fixed classification but it can be categorized into two types according to their colour that are black filler and white filler. In order to improve the strength of natural rubber, filler is added while processing the natural rubber and the mechanism that took part in this process is that fillers could modify the properties of natural rubber (Chauyjuljit *et al.*, 2002). Most popular reinforcing filler is carbon black which is widely used in tyre manufacturing industry.

Besides as mentioned above, there are particular types of filler that were used to enhance rubber properties and strength. Resin filler is usually used in shoe soles because of the hardness imparted when compounded with rubber vulcanizates. There are two types of resin filler that are phenolics and high styrene resin. Other than that, there is filler that was used widely in the plastics industry rather than in the rubber industry that is fibrous filler. It can be categorized based on its shape either long or short. Asbestos and cellulose are among natural fibrous fillers while glass, carbon and aramid are among synthetic fibrous fillers. Both natural and synthetic fibrous fillers can be classified as short type of fibrous filler while for long synthetic fibrous fillers are glass, carbon, aramid, nylon, rayon and

polyester (Bijarimi *et al.*, 2009). Below is the classification of fillers that is being used as reinforcing agent nowadays.

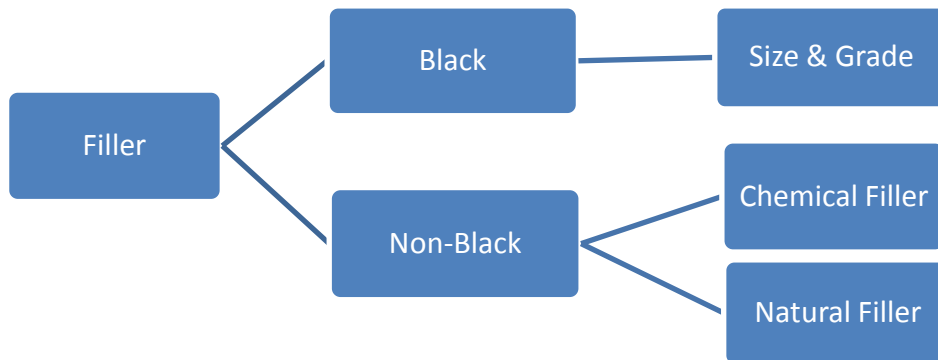


Figure 2.1: Classification type of fillers

2.2 Carbon Black

Carbon black is material that produced from incomplete combustion of gaseous or liquid hydrocarbon in the form of colloidal particles. Usually, its physical appearance is in form of black powder and widely used in rubber and plastic industry. It is also available in various sizes and specifications. In tyre manufacturing industry, carbon black is used as reinforcing agent which is it enhance the strength and improve the properties of natural rubber. Besides being as reinforcing agent, carbon black also can conduct heat away from tyre and make it become long-lasting. Smaller size of the carbon black used in rubber processing will cause bigger surface area of the filler. This will contribute great and high degree of reinforcement towards natural rubber. (Peng, 2007).

The increasing amount of carbon black also could result the increase of strength of rubber filled with carbon black. Besides that, the durability of the product that was filled with carbon black like tyre is influenced by the effect of incorporated carbon black

and natural rubber. Usually, carbon black is produced from petroleum and has been commercialized since the existence of automobile industry because of the demand to use natural rubber as main raw material to make tyre and carbon black as the reinforcing agent. The addition of carbon black is not just as reinforcing agent, but it also changes the physical appearance of natural rubber into black colour. Most of the tyre that was produced nowadays is in black colour and it is shows that how huge influence of filler towards rubber compounding or elastomer. Rubber technologist will decide the grade of carbon black based on the end product like tyre for aeroplane or for truck. Greater the load on the transport basically need stronger tyre that can absorb pressure and cope with high temperatures.

2.2.1 Carbon Black Classification

Carbon black can be categorized into several classes based on their size and specification. Table below shows the list of types of carbon black and their specifications. They can call up based on the abbreviation or size like HAF or N330 and each of these fillers has their own advantages and can be suit according to the specifications of end product.

Table 2.1: Types and specification of carbon black

Name	Abbrev.	ASTM Design	Particle Size (nm)	Tensile Strength (MPa)	Relative Lab. Abrasion	Relative Roadwear Abrasion
<i>High Abrasion Furnace</i>	HAF	N330	28-36	22.4	1	1
<i>Easy Processing Channel</i>	EPC	N300	30-35	21.7	0.8	0.9
<i>Fast Extruding Furnace</i>	FEF	N550	39-55	18.2	0.64	0.72
<i>High Modulus Furnace</i>	HMF	N683	49-73	16.1	0.56	0.66

<i>Semi-Reinforcing Furnace</i>	SRF	N770	70-96	14.7	0.48	0.6
<i>Fine Thermal</i>	FT	N880	180-200	12.6	0.22	-
<i>Medium Thermal</i>	MT	N990	250-350	9.8	0.18	-

In order to produce the high quality of rubber product, the processing part of uncured natural rubber must be conducted properly. The selection of the suitable additives is significantly influence the properties of rubber products. Generally, the structure and specific area of the carbon black will influence the yield products. The higher the surface areas of the carbon black, the higher levels of reinforcement and properties. Basically, rubber technologist or engineer will decide the formulation and choose the suitable type of filler and the properties of the end product will be tested and analyzed so that it meets the market demand.

2.2.2 Mixing of Carbon Black and Rubber

A lot of studies have been conducted to analyze effect of carbon black on natural rubber or synthetic rubber. Most of them has varies the carbon black structure to either size or grade to vary the results and comparison can be made. Among the studies conclude that natural rubber compound that containing high structure of carbon black showed higher torque and shorter cure time while low structure of carbon black showed a little influence on physical properties (Bijarimi *et al.*, 2010). Omnès *et al.*, (2008) found that the Young modulus is increased as the increasing amount of carbon black used. From that study also found that the Young modulus also increased if lower structure or smaller size of carbon black is used. The addition of carbon black into rubber compounding has its significant because of the influence and impart towards rubber strength and physical properties.

2.3 Non-Black Fillers

Beside the widely used carbon black fillers, there is another type of filler that is non-black filler. Non-black filler or white filler is can be differentiate by its colour and label. Most popular white fillers that are used in rubber processing are calcium carbonate, precipitated silica and clay. Like carbon black, white fillers also can give different effect based on their particle size. The smaller size of the white filler and then more big surface area of the white filler which is the bigger surface area give greatest degree of reinforcement towards natural rubber. Non-black fillers like natural fillers such as rice husk, empty fruit bunch and oil palm trunk fiber together with clay and asbestos is being used as replacement for black filler to increase the production as well diversify of rubber product. The addition of these reinforcing agents also functioning as additives that will be enhance the strength, increase hardness and reduce the cost of compound (Peng, 2007). Another example of non-black filler are organoclay and alumina nanoparticles.

2.3.1 Non-Black Fillers Classification

Non black fillers generally have no universal classification but it can be categorized as chemical filler and natural filler. In recent years, there a lot of studies that use non-black as filler instead of using carbon black. The effect on rubber compound physical properties can be classified into three main categories:

- i. Non-reinforcing or degrading fillers
- ii. Semi-reinforcing or extending fillers
- iii. Reinforcing fillers

Among all non-black fillers that were produced in industry, most used and popular white fillers are listed below. These non-black filler is used as replacement for carbon black and to produce middle range of strength of end product like footwear and insulator product.

- i. Calcium carbonate
- ii. Ground crystalline silica
- iii. Kaolin clay (china clay)
- iv. Alumina trihydrate
- v. Baryte
- vi. Natural fillers (rice husk, oil palm fiber and empty fruit bunch)

These are the most used non-black filler in rubber processing and were obtained from mineral or natural resources. Usually, all fillers increase the hardness and modulus of natural rubber vulcanizates into particular level but not all the fillers can strengthen the properties of rubber compound. There also has non-black filler that was produced by chemically and called synthetic fillers. Among the synthetic fillers are listed below.

- i. Precipitated calcium carbonate
- ii. Metal oxides
- iii. Precipitated silica

2.3.2 Mixing of Non-black Fillers and Rubber

There a lot of research on using the non-black filler as reinforcing agent instead of using carbon black. Among the studies is done by Teh *et al.*, (2003) which is found that cure characteristics of the natural rubber/organoclay compounds such as cure time decreased with increasing filler content from 0 to 10 phr but showed slight increases in maximum torque. Besides that, alumina nanoparticles is used as filler to incorporate with epoxidised natural rubber (ENR) which is the filler loading is varied and was found that the incorporation of alumina nanoparticles in ENR accelerated curing process and improved curing characteristics but increasing amounts of alumina nanoparticles did not cause pronounced effect to the tensile properties. The tensile strength and elongation at

break decreased with increasing of alumina content in composites but increased the tensile modulus (Noraiham *et al.*, 2008).

2.4 Natural Fillers

Natural fillers generally can be described as reinforcing agent that was produced from natural resources like rice husk, empty fruit bunch and oil palm trunk fiber. Basically, the function of these natural fillers might be same as chemical fillers that are to enhance the mechanical properties of rubber compound so that it reaches certain level of improvement. There is a question about how natural fillers that was produced from the plantation waste can be used as fillers in rubber processing. In recent years, a lot of study has been done to investigate the effectiveness of natural fillers on characterization of natural rubber. The contribution towards enhancing or improving the mechanical properties might be become significant and the main target, hence natural fillers is used in research and the outcomes are discussed in section 2.5 below. The advantages of using the natural filler instead of chemical fillers like carbon black is it is not harmful to human health compared to the carbon black for example. Besides that, they are renewable nature, easy availability and ease of chemical and mechanical modifications (Ismail *et al.*, 1997). The most significant part is the cost might be lower than chemical filler but can give quite same level of reinforcement of rubber compounds.

2.5 Natural Filler as Reinforcing Agent

In order to produce high quality of rubber product, reinforcing fillers, along with other necessary additives, are combined into natural rubber before to the vulcanization process. In tyre industry, carbon black is generally used as reinforcing filler but recently it has been found that the use of natural filler as reinforcing agent for thermoplastic–polymer composite materials (Khalf *et al.*, 2010). Besides that, it was found that as the

particle size of carbon black is decreased and amount of filler loading is increased, the mechanical properties of the rubber product are proportionally increased. The extent of the reinforcing effect is also increased when a larger amount of carbon black is incorporated (Chuayjuljit *et al.*, 2002).

Recently there a lot of studies that conducting the effect of oil palm fibre on mechanical properties of natural rubber. It was found that the incorporation of oil palm fiber decrease the tensile strength and elongation at break (Ismail *et al.*, 1997). Natural rubber naturally possesses higher strength because of the strain induced by crystallization. When fibers are incorporated into natural rubber, standard arrangement of rubber molecules was disturbed and caused the crystallization ability reduced resulting decreasing in tensile strength (Shinoj *et al.*, 2010).

Table 2.2: Effect of oil palm fiber (OPF) on mechanical properties of natural rubber

Property	Fiber content (phr)	Composite	
		OPF-sisal-NR ^a (Jacob <i>et al.</i> , 2004)	OPF-NR (Joseph <i>et al.</i> , 2006)
Tensile strength (MPa)	5	–	19.2
	10	1.75	–
	30	7.5	–
	50	3.25	7.28
Elongation at break (%) ^a	0	875	–
	5	–	1082
	10	650	–
	30	800	496
	50	–	–
Tear strength (MPa) ^b	0	20	–
	50	30	–

Source: Shinoj *et al.*, (2010)

Ishak *et al.*, (1994) reported that the tensile strength on expoxidized natural rubber (ENR) filled with white rice husk ash (WRHA) and black rice husk ash (BRHA) is increased as the increasing of filler content until 20 phr but further increasing of the filler content caused the reduction on both tensile strength. This is caused by the

formation of agglomerate of filler as the increasing amount of filler and the decreasing amount of ENR. Hence the formation of agglomeration of filler effect the filler is no longer wetted by rubber phase. Figure below shows the behaviour of tensile strength against filler content.

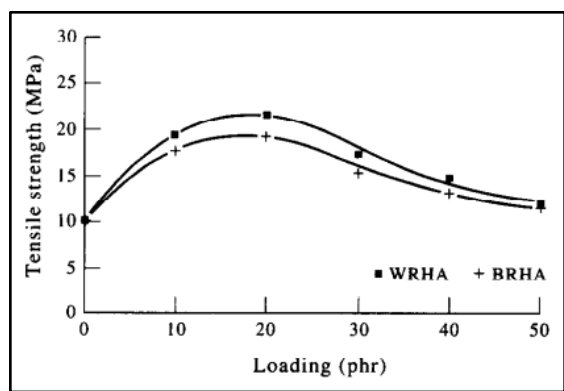


Figure 2.2: Tensile strength of WRHA and BRHA on ENR vulcanizates

Source: Ishak *et al.*, (1994)

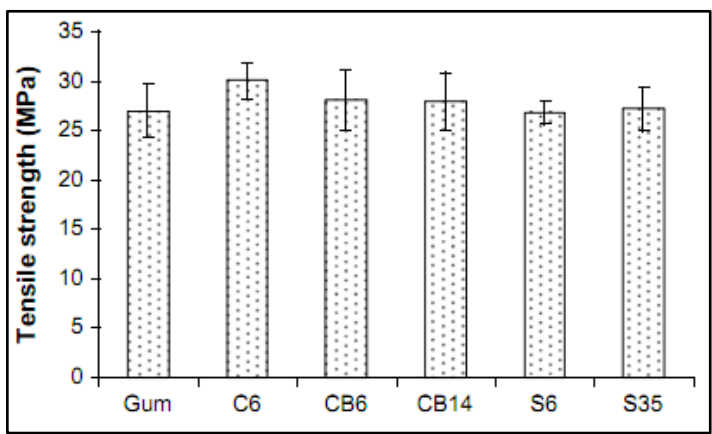


Figure 2.3: Tensile strength of NR vulcanizates filled with various fillers at various contents

Source: Rattanasom *et al.*, (2009)

Figure 2.3 above show the results of natural rubber that filled with various types fillers. The fillers that were used are clay (C), carbon black N330 (CB6 & CB14) and silica (S6 & S35). It found that natural rubber that was filled with clay exhibits the highest tensile strength whiles the other samples shown the similar value of tensile strength (Rattanasom *et al.*, 2009). Sarkawi *et. al.*, (2003) stated that natural rubber that filled with rice husk powder (RHP) and silica at 50 phr has the tensile strength above 10 MPa. Hence there is a evidence that can be used to prove that natural fillers is suitable to be used as reinforcing agent for natural rubber instead of carbon black, calcium carbonate or other chemicals filler.

2.6 Rubber Compounding

The history of rubber compounding began in 1839 when Charles Goodyear found that the addition of sulphur could change the chemical and physical properties of the vulcanized rubber. This process can be done in the present of heat of the hot stove and since that Goodyear had proposed a lot of new product from his discovery. Below is the description on rubber compounding and example of the chemical that is used.

2.6.1 Rubber Compounding Ingredient

Usually, rubber compounding ingredient is prepared by formulate the ingredient with the part per hundred rubber (phr). Table below is the example of formulation of effect of effects of particle size and amount of carbon black and calcium carbonate on natural rubber (Chuayjuljit *et al.*, 2002). The formulation must contain elastomer, activator, plasticizer, antidegradation, accelerator, vulcanizing agent and of course fillers as reinforcing agent. There also was processing aids that can be used to the rubber compounding as an option.

Table 2.3: Example of rubber formulation

Ingredient	Formulation (phr)				
	1	2	3	4	5
STR L	100	100	100	100	100
Stearic Acid	2	2	2	2	2
Zinc Oxide	10	10	10	10	10
TMQ ^a	2	2	2	2	2
6-PPD ^b	2	2	2	2	2
CBS ^c	1.2	0.5	0.5	0.5	0.5
TMTD ^d	1.2	1.2	1.2	1.2	1.2
Sulphur	0.2	0.2	0.2	0.2	0.2
Paraffinic Oil	-	6.7	6.7	6.7	6.7
N330	-	30	-	-	-
N550	-	-	30	-	-
N660	-	-	-	30	-
Calcium Carbonate	-	-	-	-	30
Total	118.6	154.6	154.6	154.6	154.6

^a 2,2,4-trimethylbutyl-1,2-dihydroquinoline

^b N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylene-diamine)

^c N-cyclohexyl-2-benzothiazyl sulphenamide

^d tetramethylthiuram disulphide

Source: Chuayjuljit *et al.*, (2002)

2.6.2 Elastomer

Elastomer is described as the material that can be stretched into certain elongation and back into its original form when the force is released. It is the main ingredient or raw material that is used in rubber processing to enhance its mechanical properties and its strength. Common used elastomer is natural rubber and other type of rubber can be used like styrene butadiene rubber (SBR) and epoxidised natural rubber (ENR). Usually, the selection of elastomer for processing is depends on the

specifications and cost of the rubber product. Natural rubber are the most common used elastomer in rubber industry because of the expanded of automobile industry that need the use of tyre and natural rubber as the raw material.

2.6.3 Fillers

Fillers are an ingredient that is incorporated with rubber compound to improve the mechanical properties of rubber compound. Generally filler can be classified as black and non-black fillers as well as chemical and natural fillers. Among the most popular filler is carbon black that was used widely in tyre industry and it is among the best filler in enhancing the strength of rubber compound. Like carbon black, it can be obtained in variety of sizes and grades. Natural fillers also can be obtained from plantation waste like rice husk, coconut fiber, empty fruit bunch and oil palm fiber. But in order to obtain the ash of the fiber, it must undergo sieving process and can be sieved according to the desired size.

2.6.4 Accelerator

Accelerator is used to increase the rate of cross-linking reaction in curing or vulcanization process. Most commonly used accelerator is N-cyclohexyl-2-benzothiazyl sulphenamide (CBS) and tetramethylthiuram disulphide (TMTD). These two ingredients are used widely in rubber processing as the curing time in vulcanization process might take a long time. The present of accelerator can decrease the cure time so that the efficiency can be enhanced. The type of cross-linking that might formed is depends on the accelerator used.

2.6.5 Activator

Activator is an ingredient that is used as activating system after is being added by fillers. Most commonly used activator is zinc oxide and was an important inorganic accelerator but other metallic element such as magnesium oxide and lead oxide also used. Among the organic compounds that can be used as activator are fatty acids, weak amines and polyalcohol. Stearic acid is used alongside with zinc oxide and the combination of these two chemicals would activate the system. Basically, the addition of zinc oxide to the rubber compounds is to function as activator to accelerate the reaction and provide the well cured stock of rubber compounds.

2.6.6 Vulcanization Agent

Vulcanizing agent is used to change the chemical and physical properties by make cross-linking between the polymer chains. Sulphur is used as vulcanizing agent as stated by Charles Goodyear in 1839 and generally in the form of powder. The chemical bonding that was formed usually is stronger than uncured elastomer. The addition of sulphur must be added alongside with accelerator so that it can reduce cure time by increase the rate of cross-linking reaction. The new chemical bonding that was formed after the addition of sulphur or sometimes was called sulphur bridges has new chemical and physical properties and more elastic as well stronger than uncured rubber compound.

2.6.7 Antidegradation

Antidegradants are generally can be classified into two main category with two characteristics function features. Antidegradants are considered either staining and discoloring, or non-staining and non-discoloring. Basically, function features either

antioxidant or antiozonant that was used to reduce aging properties for the end product by slow down the deterioration of rubber products. Deterioration generally occurs during reaction that take place in rubber processing such as oxygen, ozone, light, radiation and heat.

2.6.8 Processing Aids

Processing aids are commonly known as processing oils can either be added to the polymer itself during manufacture it or while running the rubber processing. These kinds of oils are added to the rubber compound during rubber processing to lowering the viscosity and to reduce the strain resistance as well the hardness of end product. Generally, the cost of processing aids is not too expensive compared to the cost of elastomer or fillers like carbon black but the excess amount of processing oils in rubber compound might reduce the hardness, modulus and caused the long elongation. Processing aids and carbon black tend to affect in opposite direction of direction such as viscosity, hardness, modulus and elongation. For example, carbon black functioning to increase the viscosity but processing oils reacts to reduce the viscosity of rubber compound. As a result, both chemical are simultaneously reacts and caused in neutral or no change of starting level of rubber compounds (Dick, 2001).

2.7 Rubber Processing

Elastomers like raw natural rubber that is without addition any reinforcing agent or filler is weak and easy to breakdown. The unfilled or uncured rubber has poor physical and chemical properties and low resilience, too soft, low of tensile strength as well low abrasion resistance. The uncured rubber is not suitable to be used as raw material to produce end product like tyre, fuel hose or radiator hose that will be interact with high temperature and pressure from surrounding because of the weak mechanical

properties and could be fatigue if being used. In order to enhance the ability and the strength to some higher level, rubber need to undergo processing and need to incorporated with compounding ingredient and reinforcing agent. After that it will be shaped and cured so that it will be become rubber that was filled and cured and ready to use as end product.

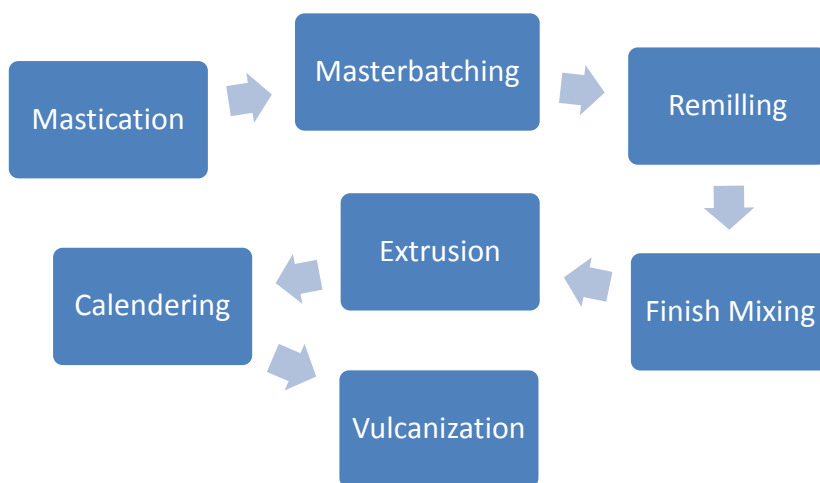


Figure 2.4: Flow diagram of rubber processing method

Rubber processing generally can be classified into three main processes that are mixing, shaping and curing. Mixing is the critical part among of the three processes because of the good incorporated of the raw material and additives will contribute to another process that is shaping. If the raw rubber is not incorporated well with fillers, oil, and chemical will be disturb the shaping and curing and the sample might be rejected (Johnson, 2001). Below is the description of each process of rubber processing until the rubber is cured and figure above is the flow diagram of rubber processing from uncured rubber until become it has been cured with sulphur.

2.7.1 Mastication

Before the raw rubber is introduce to compounding ingredient, it is necessary to break its polymer chain by undergo the mastication process. It is because of the high

viscosity of the rubber material after undergo coagulation and it found that natural rubber contains up to 5% by weight of non-polymeric constituents (Johnson, 2001). After is being processed as raw material, natural rubber is placed in storage and could be export by shipping and at this time, there might be interactions between the constituents and polymer chain and increase the viscosity and molecular weight. Before of this incident, raw rubber can be directly use as raw material will be effectively mixed but after the long being stored it is necessary to reduce its viscosity by mastication process.

Mastication can be done in the present of heat and shear on open mill or two-roll mill and the combination of both heat and shear can be result the changes on chemical and physical properties of the natural rubber. Usually this process is necessary for elastomer compound that contain of mix of polymer so that the mixture is distributed uniformly to the compounding.

2.7.2 Masterbatching

Masterbatching also means incorporation is the process which is elastomer or natural rubber is mixed with all incorporation ingredients except for curing process. This process was done to create a homogenous mixture of incorporated compound that blending all chemical additives and filler with elastomer and certain level of viscosity. Usually, the masterbatching process can separate into two parallel mechanisms. First, the elastomer undergoes deformation which is it provide larger surface area of the elastomer. This condition will promote the efficiency when the elastomer is mixed with the filler and chemical additives. Elastomer deformation can be done on mill mixing and cutting and blending on the mill also can produce same result. The second mechanism is the step which is elastomer is deformed beyond of its breaking strain limit.

Masterbatching process usually can be done either open mill or internal mixer and for this research two roll mill is used as mixer. During the addition of filler like carbon black to the elastomer, the pressure is applied on filler compound by elastomer and the agglomerate is cracked by the compressive forces. At this stage the interactions

within the agglomerate are still filled with air and make the sample become very weak. As the mixing process continuing, the rubber is forced to void region in the filler and the air is released (Johnson, 2001).

2.7.3 Remilling and Plasticization

Remilling is required if the compound from masterbatching process shows inadequate dispersion or need more milling process because of the high viscosity of rubber compound. After the compound is incorporated, the compound is ready to undergo next step.

Plasticization is a process which is plasticizers or internal lubricant is added to rubber compounds to modify their viscosities. The addition of plasticizers towards rubber compounds might contribute to the slightly change in reduction in viscosity.

2.7.4 Finish Mixing

Finish mixing is done when the rubber compound completes the incorporation process until curing system. Usually curing system or vulcanization process was done in internal mixer where mixing rate and time are balanced so that the homogeneity is uniform without regenerating heat that will cause premature curing.

2.7.5 Extrusion

Extrusion is a process which is rubber compound is forced by the pressure into the die and form the final product in particular required shapes. Basically the extruder has two main functions, first is to force the rubber compound through the die and

generate sufficient pressure in order to push the rubber pass through the die to form pellets or through a pair of roller to form a sheet. There are many types of extruders but most common used is screw type extruder and the rubber compound usually is extruded directly from masterbatching process while the compound still hot.

Extrusion process basically can be done by several parts that is feeding, conveying and shaping. Feeding part is conducted by feed the rubber compound which is in form of strip and the dimension must be in consistent width and thickness. The feed section must be feed in sufficient amount and cannot be more until flooding or not enough. Then the process is continued with conveying which is the pressure is applied by the moving screw and push the rubber compound through at certain temperature. After that the compound is allowed to undergo shaping part and the desired shape is determined by die that is being used (Johnson, 2001). Curing process is conducted after the extrusion process and rubber compound can be continuously cured after extruded.

2.7.6 Calendering

After being extruded, rubber compound can be shape into particular thickness by undergoes calendering process. This process is conducted to produce a continuous sheet of rubber and also for incorporated the rubber sheet with reinforcing material such as textile or wire cord. To ensure that the end product is perfectly formed with desired thickness, the calender needs at least two or more mills and usually the rubber compound is compressed up to four mills. High pressure is significant for this process in order to gain desired thickness of sheet and to release the trapped air. Basically, the calendering process can be separated into several parts as listed below:

- i. Feeding
- ii. Sheeting
- iii. Frictioning
- iv. Coating

The process starts with feed the rubber compound to the feed section. Before that, the compound is heated to temperature around 90°C and was plasticized. This is to ensure the smooth process without any problems while sheeting in progress. After that, sheeting part take place and the gap between the rolls is controlled so that the thickness for end product is under control and in good shape. Then frictioning is conducted which is textile or metal fabric is feed to the calender with different speed and rubber sheet is forced to interstices of the substrate. Finally the coating part takes place and the process same as frictioning part but same speed is applied. The rubber sheet will lay down under the coating material and pressed into it (Johnson, 2001).

2.7.7 Vulcanization

In 1893, Charles Goodyear found that the properties of natural rubber can be modified by using vulcanization process. The rubbers that have been mixed but uncured with sulphur have poor chemical and physical properties. Vulcanization process is the process where sulphur will be added to natural rubber to impart high elasticity, tensile strength and resistance to abrasion. The addition of sulphur as vulcanizing agent into rubber compound is to create chemical bonds with polymer chain that already form before the process. The new chemical bond formed are generally stronger than uncured one and give elastic three-dimensional network. This reaction is called cross-linking process as sulphur functioning as connector between polymer chain and tied it so that it become new chemical bonds.

Usually curing or vulcanization process is last stage in rubber processing or after the final shape of end product has been obtained. In order to complete the process, vulcanizing agent that is sulphur is added to rubber compound with accelerator, activator and retarder. Among the accelerator that commonly used is N-cyclohexyl-2-benzothiazyl sulphenamide (CBS) and functioning to increase the rate of the cross-linking reaction. Table below is the comparison between the uncured rubber with cured rubber from the chemical and physical properties aspect.

Table 2.4: Comparison between raw natural rubber and vulcanized natural rubber

Raw Natural Rubber	Vulcanized Natural Rubber
Soft and sticky	Comparatively hard and non-sticky
Low tensile strength and not very strong	High tensile strength and very strong
Low elasticity	High elasticity
Can be used over a narrow range of temperature from 10 to 60 °C	Can be used over a wide range of temperature from -40 to 100 °C
Low abrasion resistance	High abrasion resistance
Soluble in solvents like ether, carbon tetrachloride, petrol and turpentine	Insoluble in all the usual solvents

Hani Aziz Ameen, (2008) stated that the hardness and tensile stress between vulcanized and unvulcanized natural rubber filled with epoxy resin shows that vulcanized natural rubber compound have higher tensile stress and hardness rate. Hence the incorporation of vulcanizing agent should increase the mechanical properties of natural rubber.

2.8 Mixer

Generally, rubber processing is conducted by using the mixer that can mix all the chemicals and fillers until all become a compound that well mixed. Uncured rubber, chemicals additives and fillers are loading into the mixer to form uncured rubber compound. There are two basic types of mixers that was used to compound rubber, first is internal mixer and the other one is two roll mill. The description about both equipments are describes below.

2.8.1 Internal mixer

Internal mixer was first developed by Fernley H. Banbury in 1916 because of the large amount of rubber compound in rubber processing. It contain two blades that turning toward each other and either can be operate in tangential or not. There is a loading area which is all the chemicals and fillers as well natural rubber is loaded and is allowed to enter mixing area. After the mixing completed, the end product will be discharged on the bottom of the internal mixer and basically, the period of mixing depend on the type of rotor is used and the horsepower of the mixer.

2.8.2 Two roll mill

Two roll mill consists of two horizontal metal rollers that can be heat up to 100 °C and is used for rubber processing. Mixing process that includes incorporation of fillers and chemicals can be done by using two roll mill. The gap between two metal rollers can be adjusted so that the thickness of rubber compound will be in uniform thick. Basically, this equipment only can support a small amount of rubber compound and for large amount of rubber compound; it can be done with internal mixer.

CHAPTER 3

METHODOLOGY

3.1 Equipment

There are two main equipments that were used to conduct the rubber processing. They are two roll mill that was used in rubber processing to mix rubber with additives and hot and cold molding press that was used to mold the rubber compounding. These two equipments are available at FKKSA's laboratory at UMP Kampus Gambang. Beside as mentioned before there is equipment that was used but was conducted manually that is siever. Siever is used to sieve the oil palm trunk fiber until become in powder form. There is equipment that should be used in rubber processing that is extruder but was ignored because of the availability of that equipment in UMP.

3.1.1 Two Roll Mill

Two roll mill in FKKSA's laboratory was used in rubber processing for research of effect of oil palm trunk fiber on characterization of natural rubber. This equipment consists of two metal rollers and a heater so that the metal roller can be heat up to 100 °C. During the rubber processing is conducted, some of the fillers or chemicals might stick to the rolling roller and after the rubber is repeated compressed by the rollers, it

will well mixed with the rubber compounds. Figure 3.1 below shows the two roll mill in FKKSA's laboratory.



Figure 3.1: Laboratory two roll mill

3.1.2 Hot and Cold Molding Press

Hot and cold molding press in FKKSA's laboratory is used to prepare the rubber compound in the form of homogenous sheet so that the sheet can be cut into dumbbell shape for testing procedures. The details procedure for conducting this equipment is describe section 3.5 and figure 3.2 below shows the hot and cold molding press that available at FKKSA's laboratory.



Figure 3.2: Hot and cold molding press

3.1.3 Tensile Testing Unit

Tensile testing was conducted by using the tensile testing unit at FKP's laboratory at UMP Kampus Gambang. This machine can be loaded until maximum of 50 kN and can be use for various types of material such as metal, nanocomposite, polymer and others materials. The procedure for tensile testing is described in section 3.6.1. Figure 3.3 below shows the tensile testing unit that was available at FKP's laboratory.



Figure 3.3: Tensile testing unit

3.2 Preparation of Raw Material

The natural rubber that is Standard Malaysian Rubber 10 (SMR-10) and oil palm trunk fiber are obtained from the supplier in a bulk package. In order to prepare the raw material that need small amount that is 250 grams per sample, it is necessary to cut the natural rubber into small amount or in the form of slice with the thickness of around 1

centimeter. The oil palm trunk fiber also needs to be sieve and the ashes are collected while the fiber waste is eliminated. Table 3.1 below is the shows the rubber formulation for this research.

Table 3.1: Rubber formulation of effect of oil palm trunk fiber on natural rubber

Ingredient	Formulation (phr)				
	1	2	3	4	5
SMR-10	100	100	100	100	100
Stearic Acid	2	2	2	2	2
Zinc Oxide	5	5	5	5	5
6-PPD ^a	1	1	1	1	1
CBS ^b	0.5	0.5	0.5	0.5	0.5
Sulphur	2.5	2.5	2.5	2.5	2.5
OPTF ^c	0	15	30	40	50
Total	111	126	141	151	161

^a N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylene-diamine)

^b N-cyclohexyl-2-benzothiazyl sulphenamide

^c Oil Palm Trunk Fiber

The weight of each ingredient is determined by percentage by parts times with total weight of a sample that is 250 grams and the calculations are shown at section 3.2.4. Below is description of preparation of raw material for natural rubber, fillers and chemicals including the step that was taken to prepare the raw material that readily use for rubber processing also the example of calculation of weight based on rubber formulation in Table 3.1.

3.2.1 Natural Rubber

Natural rubber is obtained in bulk package and to prepare into small amount, it is necessary to cut the rubber into slice using a small saw. The compound it is in form of high viscosity and has high elasticity hence it is hard to cut the rubber into smaller size.

Then the sample of rubber is weighed according to the rubber formulation and was kept as well labeled. After that, the rubber slice will undergo mastication to prepare rubber compound that well homogeneous and low viscosity.



Figure 3.4: Sample of natural rubber

3.2.2 Fillers

Oil palm trunk fiber is in the form of coagulation of waste palm oil fiber. In order to obtain the powder from the fiber, it is necessary to sieve the filler using siever and was sieved manually using size of 400 μm . the filler has a light weight and for every session of sieving, only produce around 20% from the coagulate fiber. Hence the sieving process was taking a long time in order to yield around 226.82 grams of fillers for all samples. Below are the figures of siever that was used and conditions during the sieving process.



Figure 3.5: Siever with size of 400 μm

The sieving process started by taking some amount of coagulates oil palm trunk fiber into siever and was shaken so that the powder will fall down with the uniform size. The process is repeated for large amount of filler in order to obtain total amount of filler for each sample. Figure 3.6 below shows the physical appearances of fillers before and after being sieved.



Figure 3.6: Physical appearances of fillers before and after sieving process

3.2.3 Chemicals

Chemicals are used as additives for the rubber processing and five chemicals were used and can be separate into two groups. First is the activator and antidegradant

group and second is vulcanizing group that represent stage 1 and stage 2. Summary of the chemicals used is shows in table 3.2 below.

Table 3.2: Summary of chemical in rubber compounding

Stage 1	Stage 2
Zinc oxide	Sulphur
Stearic acid	CBS
6-PPD	

All chemicals are weighed according to the rubber formulation and were packed according to the stages. Zinc oxide, stearic acid and 6-PPD are packed together and were shacked so that it well mixed in a mixture. The rubber processing is conducted by addition of chemicals by stages. Each of the stage is added to the rubber compound and for each interval of the stages will take some period in order to ensure the rubber compound is well mixed with the chemicals. Figure 3.7 below shows the chemicals were used according to the stages.



Figure 3.7: Chemicals for mixing process

3.2.4 Weight Calculation of Each Ingredient

The rubber formulation in table 3.1 shows the part per hundred rubbers (phr) for each ingredient. In order to proceed to the rubber processing, it is necessary to calculate the weight of each compound based on the phr as listed in the rubber formulation. The equation that can be used to calculate the weight is shown below.

$$\text{Weight of ingredient (g)} = \frac{\text{number parts of ingredient}}{\text{total parts of a compound}} \times 250 \text{ g} \quad (3.1)$$

3.2.4.1 Example of Calculation for First Sample

Below is the detail weight calculation of the first sample with 0 phr of filler. The other sample weight calculation is shown at Appendix A for the remaining four samples.

- a) Natural rubber (NR) with 100 phr from total part of 111

$$\begin{aligned} \text{Weight of NR (g)} &= \frac{100}{111} \times 250 \text{ g} \\ &= 225.23 \text{ g} \end{aligned}$$

- b) Stearic acid with 2 phr from total part of 111

$$\begin{aligned} \text{Weight of Stearic acid (g)} &= \frac{2}{111} \times 250 \text{ g} \\ &= 4.50 \text{ g} \end{aligned}$$

- c) Zinc oxide with 5 phr from total part of 111

$$\begin{aligned} \text{Weight of Zinc oxide (g)} &= \frac{5}{111} \times 250 \text{ g} \\ &= 11.26 \text{ g} \end{aligned}$$

d) 6-PPD with 1 phr from total part of 111

$$\begin{aligned}\text{Weight of 6 - PPD (g)} &= \frac{1}{111} \times 250 \text{ g} \\ &= 2.25 \text{ g}\end{aligned}$$

e) CBS with 0.5 phr from total part of 111

$$\begin{aligned}\text{Weight of CBS (g)} &= \frac{0.5}{111} \times 250 \text{ g} \\ &= 1.13 \text{ g}\end{aligned}$$

f) Sulphur with 2.5 phr from total part of 111

$$\begin{aligned}\text{Weight of Sulphur (g)} &= \frac{2.5}{111} \times 250 \text{ g} \\ &= 5.63 \text{ g}\end{aligned}$$

g) Oil palm trunk fiber (OPTF) with 0 phr from total part of 111

$$\begin{aligned}\text{Weight of OPTF (g)} &= \frac{0}{111} \times 250 \text{ g} \\ &= 0.00 \text{ g}\end{aligned}$$

3.2.4.2 Calculation Summary of Each Ingredient

Table below shows the weight of each ingredient for all five samples according to the rubber formulation and calculation that has been made in the previous section.

Table 3.3: Weight of each ingredient for all five different of filler content

Ingredient	Weight (grams)				
	1	2	3	4	5
SMR-10	225.23	198.41	177.30	165.56	155.28
Stearic Acid	4.50	3.97	3.55	3.31	3.11
Zinc Oxide	11.26	9.92	8.87	8.28	7.76
6-PPD ^a	2.25	1.98	1.77	1.66	1.55
CBS ^b	1.13	0.99	0.89	0.83	0.78
Sulphur	5.63	4.96	4.43	4.14	3.88
OPTF ^c	0.00	29.76	53.19	66.23	77.64
Total	250.00	250.00	250.00	250.00	250.00

^a N-(1,3-dimethylbutyl)-N'-phenyl-p-phenylene-diamine)

^b N-cyclohexyl-2-benzothiazyl sulphenamide

^c Oil Palm Trunk Fiber

3.3 Mastication

Mastication is a process that was conducted to prepare the rubber compounds that can readily accept the addition of chemicals or additives for rubber processing. The objective of this process is to break the polymer chains so that the viscosity of rubber compounds is reduce as well as the elasticity of the rubber. The slice of rubber that was cut from the bulk rubber with the weight according to the rubber formulation is allowed to go through the two roll mill. The rubber slice then is compressed by two roll mill with particular gap between both rollers at temperature of 70 °C and speed of 50 rpm. Figure 3.8 below shows the method for mastication process for raw natural rubber.



Figure 3.8: Natural rubber in mastication process

The process of mastication is continued with the rubber is allowed to be compressed repeatedly for some period about 15 minutes until it become homogenous compound. The physical outcome of rubber slice is changed from the thickness which is certainly must be less than the initial thickness and the physical appearances seems to become quite homogenous than the initial form. Figure 3.9 below shows the physical appearances of the rubber slice before and after the mastication process



Figure 3.9: Physical appearances of rubber before (left) and after (right) mastication process

The physical and chemical properties of natural rubber might be change after the process like viscosity and hardness. The changes certainly could give some facilitate for next rubber processing that is compounding.

3.4 Compounding

Compounding or mixing is conducted to incorporate all the chemicals and fillers with natural rubber until become homogenous mixture. All chemicals in rubber formulation are incorporated by stages and can be classified into three stages. First stage is addition of fillers with natural rubber, second stage is addition of chemicals to natural rubber that was added with fillers and last stage is addition of vulcanization agent with present of accelerator. The details of each stage are described below with attachment of figure related to the method.

3.4.1 Addition of Fillers

The first stage is started with addition of filler that is oil palm trunk fiber into natural rubber by using two roll mill and the rubber compound is compressed by two metal rollers. Fillers that is in form of powder is added properly to the rubber compound in the present of heat around 70 °C from the metal rollers that rolling towards each other. Figure 3.10 below shows the process of incorporation of fillers with natural rubber.



Figure 3.10: Natural rubber is incorporated with filler

The process is repeated until all fillers that were used are well mixed with natural rubber and become homogenous mixture of solid. The process was take a long time due to the light weight of filler and caused long process of incorporation and the period is increasing as the increasing of filler part. Figure 3.11 below shows the physical appearances of natural rubber before and after being added by fillers.



Figure 3.11: Natural rubber before (left) and after (right) addition of filler

3.4.2 Addition of Chemicals

The second stage is continued by addition or incorporation of chemicals with the rubber compound. The chemicals are incorporated by using spatula on the rubber compound surface and are allowed to mix by compress it between two metal rollers in the present of heat around 70 °C. The chemicals that contain zinc oxide, stearic acid and 6-PPD are incorporated properly so that these additives are well mixed with the rubber compounds. Figure 3.12 below shows the rubber compound is incorporated with chemicals.

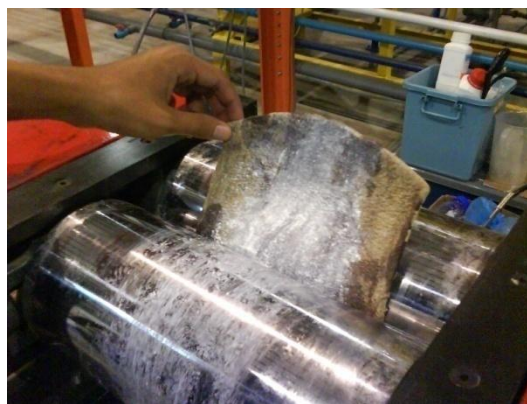


Figure 3.12: Chemicals is being added to rubber compound

During the process, some of the chemicals might stick or paste on metal roller but it will well mixed to the rubber compound after the repeated process about 30 minutes per sample. After the incorporation of chemicals, the rubber compound is allowed to cool down into room temperature at least two hours for preparation of next process. Figure 3.13 below shows the physical appearances of the rubber compound before and after incorporation with chemicals.



Figure 3.13: Natural rubber before (left) and after (right) addition of chemicals

3.4.3 Addition of Vulcanization Agent

The last stage of incorporation of chemicals is addition of vulcanizing agent in the present of accelerator. Both two chemicals are representing by sulphur as vulcanizing agent and N-cyclohexyl-2-benzothiazyl sulphenamide (CBS) as accelerator. The process is started after the rubber compound is allowed to cool down into room temperature for at least two hours. The mixture of sulphur and CBS is added on the rubber surface and was compressed by two roll mill in the present of heat around 70 °C. Figure 3.14 below shows the sulphur and CBS is being incorporated with rubber compound.



Figure 3.14: Sulphur and CBS is being added to rubber compound

After repeated mixing and compressing the rubber compound around 15 minutes, the compound might be well mixed because of the small amount of sulphur and CBS are used and not take a long time. Figure 3.15 below shows the physical appearances of the rubber compound before and after incorporation with sulphur and CBS.



Figure 3.15: Natural rubber before (left) and after (right) addition of vulcanizing agent

3.5 Molding

Molding was conducted after the mixing process in order to prepare the sample for testing part. Hot and cold molding press was used as the compressor in the present of heat around 160 °C for about 25 minutes. Among objectives of this process are to prepare the rubber compound in the form of sheet and to mix the rubber so that it become homogenous mixture. The result of this process then is mold according to the dumbbell shape for testing part. Details procedures of molding process from rubber compound until it become in the form of dumbbell shape is described as below.

3.5.1 Preparation of Sample

Rubber compounds that were mix with fillers and chemicals are cut into several parts for at least two parts. One or two of the part is cut into the small pieces like dice and is accumulated to undergo molding process. Figure 3.16 below shows the step of preparing the sample for molding process.



Figure 3.16: Preparation of rubber compound before molding

3.5.2 Loading of Sample and Shaping

After completing cut all the required part for molding process, the small pieces of sample is placed on the metal plate and was accumulate into the center so that the end product would be without holes. Then another plate is placed on the sample and will close the bottom plate with rubber sample inside it. Figure 3.17 below shows the procedure of loading the sample on molding press.



Figure 3.17: Sample is being prepared before molding process

Then the plate is placed inside the hot and cold molding press and is allowed to melt about 25 minutes at temperature of 160 °C. After 25 minutes the sample is unloaded from the molding press and is allowed to cool down for a while. The sample then separated from the metal plate and was packaged and labeled. Figure 3.18 below shows the physical appearances of rubber compound after being pressed by hot press.



Figure 3.18: Sample is being separated from mold after being pressed by hot press

After being cooled about a few hours, the sheet of sample is cut according to the mold into several parts. The dumbbell shape of sample is packaged and labeled for next procedure that is testing to obtain the mechanical properties of the samples. Figure 3.19 show the sample after being molded into dumbbell shape.



Figure 3.19: Rubber after being molded with dumbbell mold

3.6 Testing

Several types of testing are conducted in order to obtain the required data for analysis of data. All the testing was conducted in UMP Kampus Gambang and details of the procedure of each testing is described as below.

3.6.1 Tensile Test

Tensile test is conducted for measuring the tensile strength of rubber compound for all part of filler content. The testing is started with set up the outcomes result like the parameter of tensile strength, elongation, and time required meeting the break point. Sample with dumbbell shape is loaded and was gripped at the both end of the sample. Then the machine is started and the sample is pulled at the rate of 20 mm/min. after certain elongation the sample was break and deformed and the outcomes data is recorded and analyzed. Figure 3.20 below shows the condition of sample before and after is being pulled by tensile machine.



Figure 3.20: Sample before (left) and after (right) undergo tensile testing

3.6.2 Swelling Test

Swelling test is conducted to calculate the swelling percentage of rubber compound. All the samples of rubber are cut into particular shape and were immersed in kerosene and diesel for 24 hours. Objective of this test is to determine swelling resistance of rubber compound that was filled with fillers. The calculation for swelling percentage can be obtained by using equation below.

$$\text{Swelling percentage (\%)} = \left[\frac{(M_2 - M_1)}{M_1} \right] \times 100 \% \quad (3.2)$$

Where M1 is the initial mass of sample and M2 is the mass of specimen after immersion. Figure 3.21 below shows the swelling test is being conducted at laboratory.



Figure 3.21: Swelling test of rubber compound using kerosene (left) and diesel (diesel)

3.7 Summary of Methodology

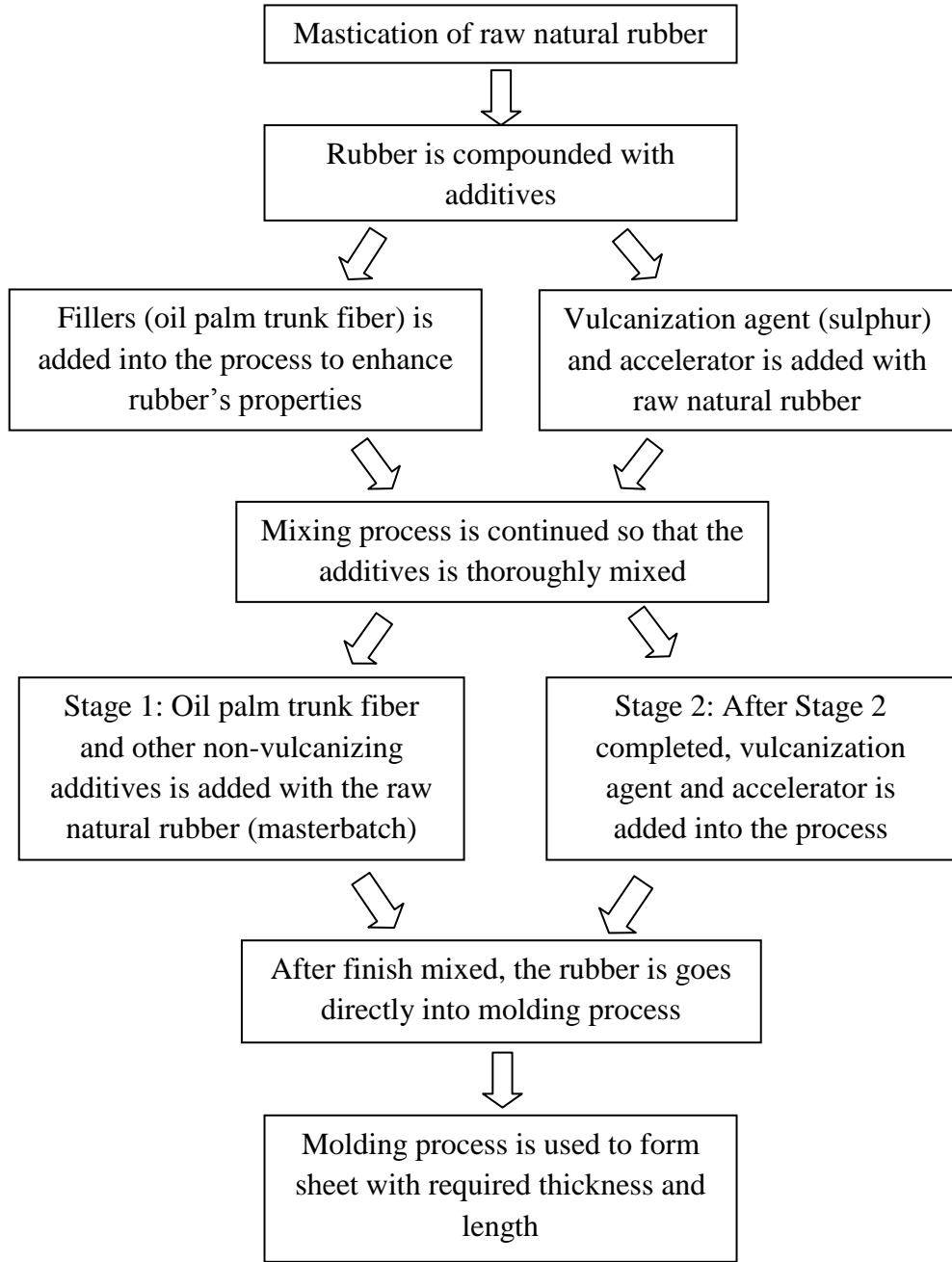


Figure 3.22: Summary of methodology

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This section will review about the results from the tests that were made. The effect of the fillers loading or fillers content on mechanical properties of natural rubber will be analyzed and discussed. The reasons for each result will be investigated and the data of each sample is plotted on the graph for better comparison and discussion. Among the mechanical properties that were included in this section are tensile strength, Young's modulus and stress-strain behaviour. Besides that, the elongation, time at break and mixing time are also will be review and discussed.

4.2 Tensile Test

Tensile test was conducted for five samples of rubber compound with different of filler content. Tensile testing unit is used as the platform and result of each sample is shown at Appendix B. Data that was obtained is analyzed and the calculation has been made to plot graph in order to compare the performance of each sample among the various types of parameters.

4.2.1 Effect of Filler Content on Tensile Strength

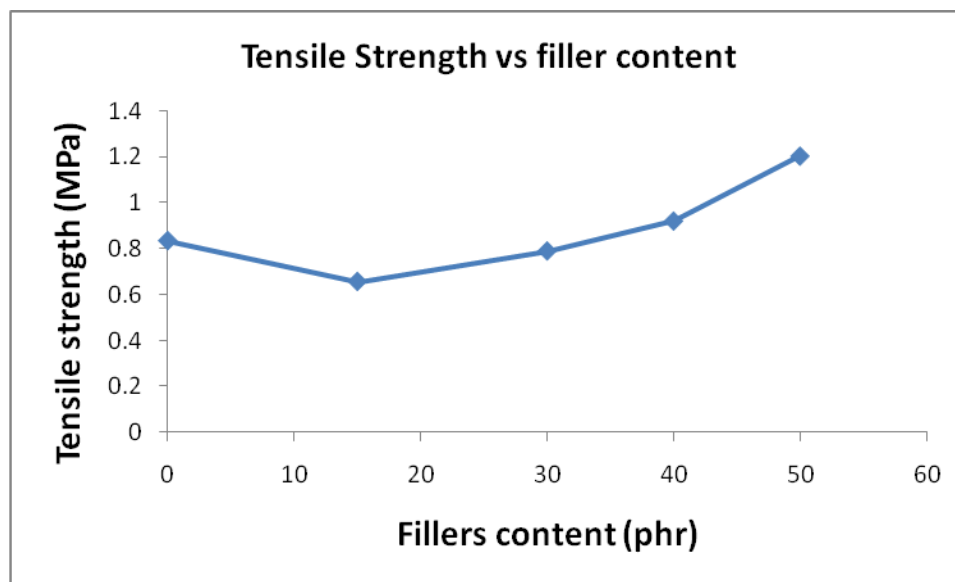


Figure 4.1: Graph of tensile strength against fillers content

The graph above shows the relationship between tensile strength with fillers content from 0 phr to 50 phr. The trend shows that the tensile strength decreases from 0 phr to 15 phr but increase back until 50 phr. The increasing amount of fillers content caused the tensile strength increase because of the good interaction between rubber and fillers. The high tensile strength in 0 phr caused by the high elongation that leading to the high tensile strength. Time for 0 phr of fillers content in rubber compound to break require long time because of the high elasticity of the natural rubber that is not filled with filler.

4.2.2 Effect of Filler Content on Elongation

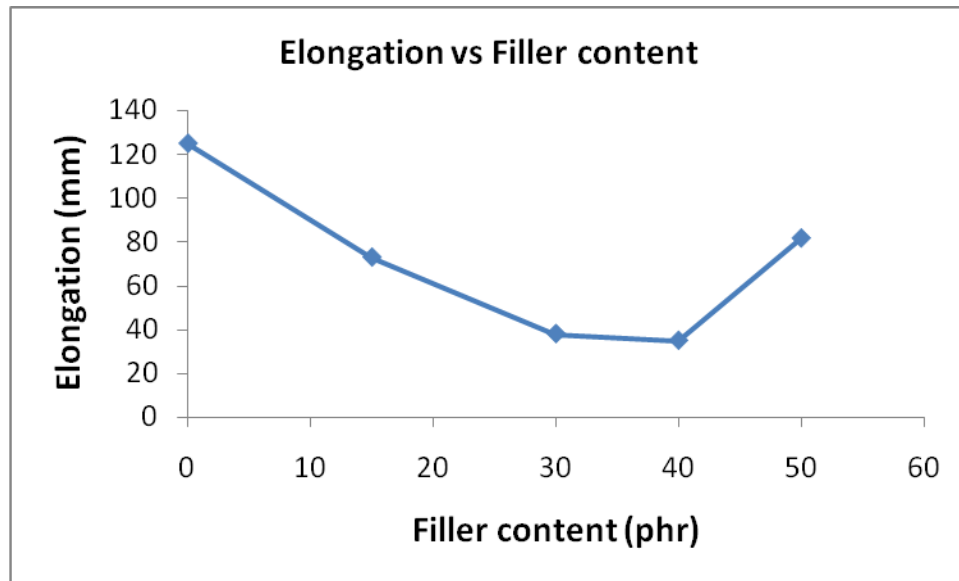


Figure 4.2: Graph of elongation against fillers content

Figure above shows that the elongation of rubber compound is decreased as the increasing of fillers content. The incorporation of fillers has made the rubber compound less elastic from 0 phr to 40 phr because of the increasing interactions between the filler and rubber. Then it caused the bonding inside the rubber compound is reinforced and improved. Ismail *et. al.*, (1999) reported that the increasing amount of natural fillers in rubber composite, caused the stiffness and brittleness of the composite increased regularly with related decrease in the elongation at break. The 50 phr of fillers content shows the high elongation due to the poor interaction between fillers and rubber and might be caused by incomplete mixing during the mixing process.

4.2.3 Effect of Filler Content on Time at Break Point

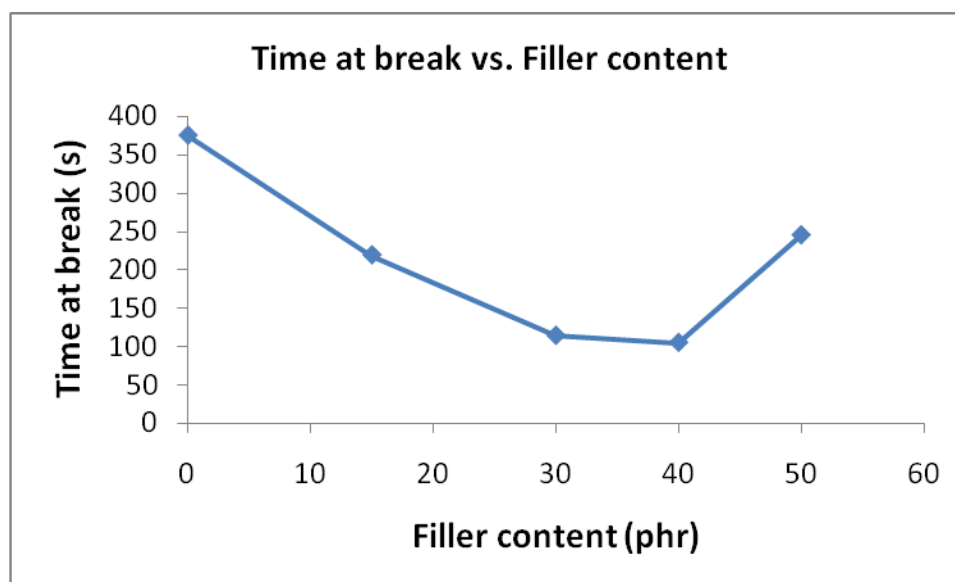


Figure 4.3: Graph of time at break against fillers content

Graph above shows the relationship between the times at break with filler content in rubber compounds. The graph shows the decreasing trends from 0 phr to 40 phr and increase back from 40 phr to 50 phr. The decreasing trends from 0 phr to 40 phr caused by the high elasticity of rubber compounds that leading to the low elongation. Then the time is reduced because of the break point of sample become faster as the increasing amount of fillers.

4.2.4 Effect of Filler Content on Young Modulus

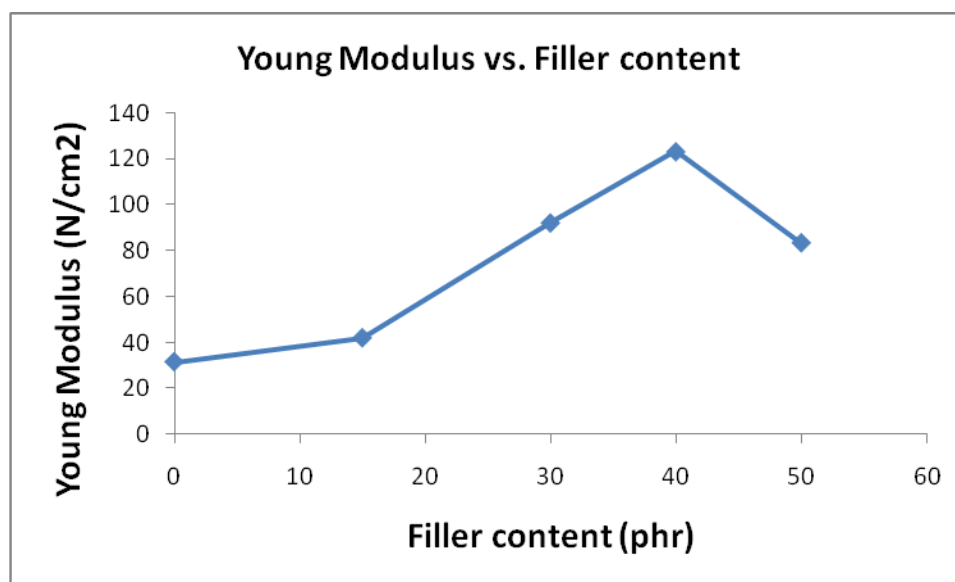


Figure 4.4: Graph of Young modulus against fillers content

Graph above shows the relationship between Young's modulus and the fillers content in rubber compound. The graph shows that the Young's modulus is increased as the increasing amount of fillers from 0 phr to 40 phr. This is due to the high tensile strength and low elongation of samples which indicates that the bonding of rubber compounds is reinforced by addition of fillers. The incorporation of fillers with natural rubber caused the bonding inside the rubber compounds is improved and the strength of the bonding is increased as the increasing amount of fillers. The 50 phr shows the decrease from the 40 phr because of the high elongation and high tensile strength that yield low Young's modulus. This might be caused by the incomplete mixing between rubber and fillers during the mixing process and contribute to the low Young's modulus.

4.2.5 Effect of Filler Content on Time at Break Point

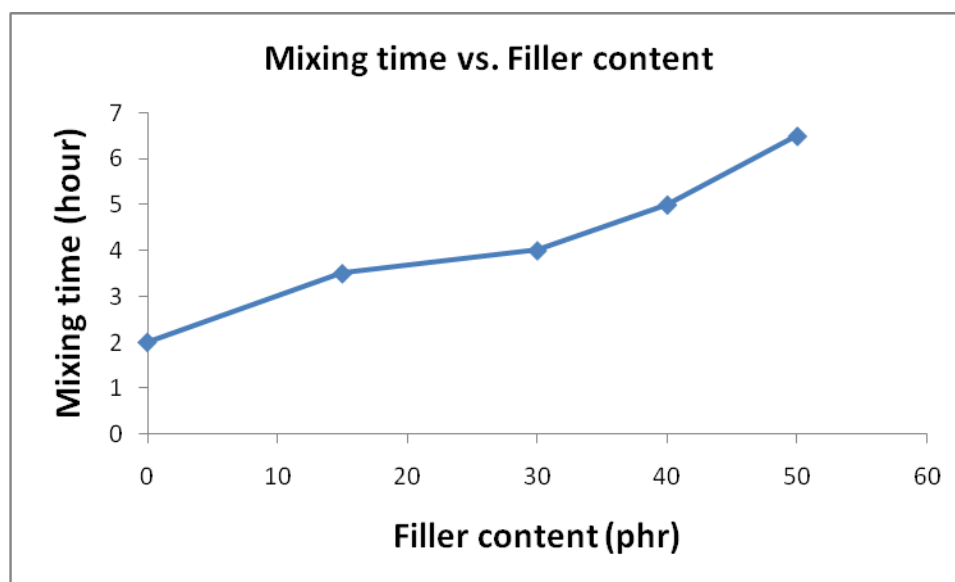


Figure 4.5: Graph of mixing time against fillers content

Graph above shows the relationship between the mixing time and fillers content in rubber compound. It can be described that the mixing time is increased as the increasing amount fillers content in rubber compound. The mixing process require a long time for higher fillers content because the amount of fillers is increased as the increasing amount of fillers but amount of natural rubber is decreased. Hence the mixing is become longer by increasing amount of fillers and the 50 phr is the longest and the hardest because there a highest amount of fillers but lowest amount of natural rubber.

4.3 Behaviour of Graph Stress against Strain

The graph of stress against strain for all five samples is plotted as in figures below and the stress and strain is determined by using the equations 4.1 and 4.2. The calculation for each point is shown in Appendix B and from the graph stress against strain, the Young modulus is determined by determined the best line of each graph and the gradient is equal to the Young's modulus. Young's modulus is described as the ratio of stress over strain and represent as the modulus of elasticity of the material and can be determined by using equation 4.3 (Callister *et al.*, 2008).

$$\text{Stress} = \frac{\text{Force (N)}}{\text{Area (cm}^2\text{)}} \quad (4.1)$$

$$\text{Strain} = \frac{\text{Change in length, } \Delta l \text{ (cm)}}{\text{Initial length } l_0 \text{ (cm)}} \quad (4.2)$$

$$\text{Young's Modulus} = \frac{\text{Stress}}{\text{Strain}} \quad (4.3)$$

4.3.1 Sample of 0 phr of filler content

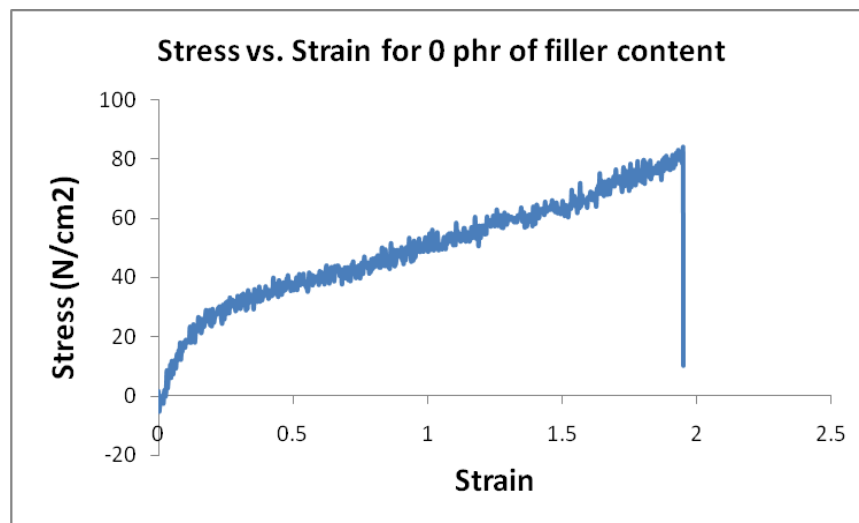


Figure 4.6: Graph of stress against strain for 0 phr of fillers content

The graph above shows the relationship between stress with strain for 0 phr of filler content. From the graph, the sample experience high strain before it reached the break point and lead to the high stress because more forces is needed to breakdown the samples. The gradient of this graph is equal to the Young's modulus that is 31.555 N/cm^2 . This value is the lowest among the five samples and caused by the high strain or long elongation that was experienced by rubber compound. High strain also requires long time to meet the break point and contribute to the high stress or high forces are needed in order to reach the break point.

4.3.2 Sample of 15 phr of filler content

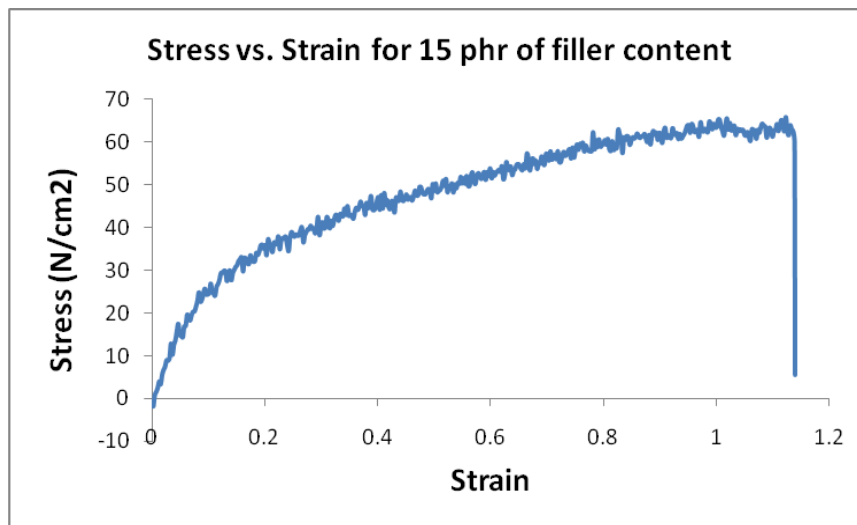


Figure 4.7: Graph of stress against strain for 15 phr of fillers content

The graph above shows the relationship between stress and strain for 15 phr of fillers content. The strain of the sample is decreased almost to the 1.10 compared to the sample with 0 phr that is exceeding 1.5. The gradient or Young's modulus of this graph is 41.977 N/cm^2 which is higher than 0 phr and caused by the low strain and the ratio of stress over strain is increased. It means that sample of 15 phr need more force to reach the break point and the time to reach the break point become faster as compared to the 0

phr. Lower strain means that the elongation of the samples is decreased because of the high elasticity due to the good interaction between rubber and fillers. The mixing time also increased because of the addition amount of fillers and reduction amount of natural rubber.

4.3.3 Sample of 30 phr of filler content

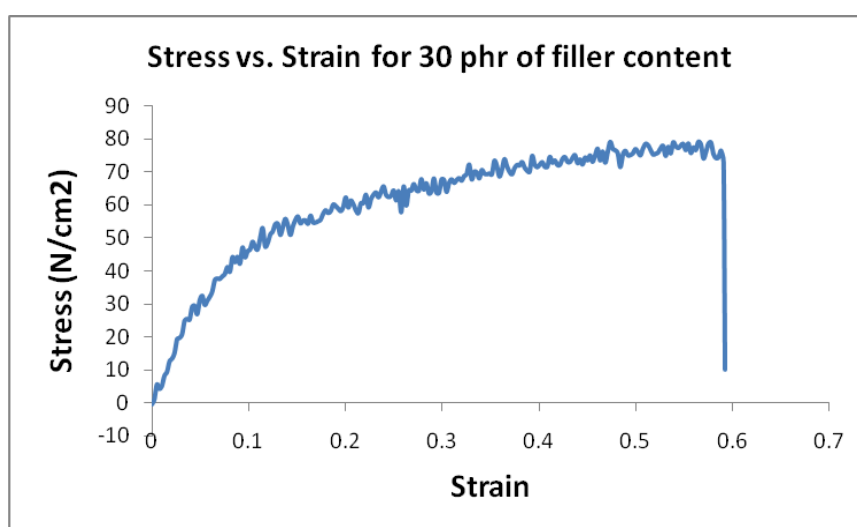


Figure 4.8: Graph of stress against strain for 30 phr of fillers content

Graph above shows the relationship between the stress and strain for 30 phr of fillers content in rubber compound. The stress that was experienced by the sample is around 80 N/cm^2 before it reached the break point and break down. The strain is reduced into around 0.6 and is lower compared to the 0 and 15 phr. This might be caused to the good interaction between rubber and fillers that leading to the high elasticity and more force is needed to reach the break point. The Young's modulus or gradient of this graph is 92.17 N/cm^2 and in the middle range among the five samples. Mixing time also in the middle range due to the 30 phr is in the middle range of fillers content. The time to reach the break point is faster than the 15 phr because of the lower strain experienced by the sample.

4.3.4 Sample of 40 phr of filler content

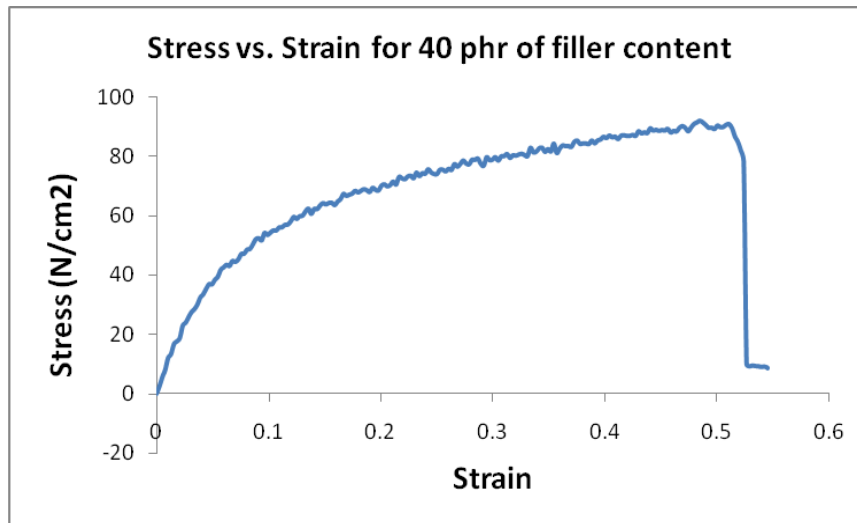


Figure 4.9: Graph of stress against strain for 40 phr of fillers content

Graph above shows the relationship between the stress and strain for 40 phr of fillers content in rubber compound. The graph shows that the sample experiences the stress around 90 N/cm² before it reach the break point. The sample also experiences the break point after being stretched about half of its initial length or it's reached the strain about 0.5 before break down. It shows that the sample has high tensile strength and need high force to reach the break point. But in order to reach the break point, high force is needed and caused the sample break down faster than other first three samples. The low strain means that the sample has the high elasticity and the Young's modulus or gradient of the graph is 123.3 N/cm² which is the highest among the five samples. This is due to the low elongation and high tensile strength and high force is needed to reach the break point.

4.3.5 Sample of 50 phr of filler content

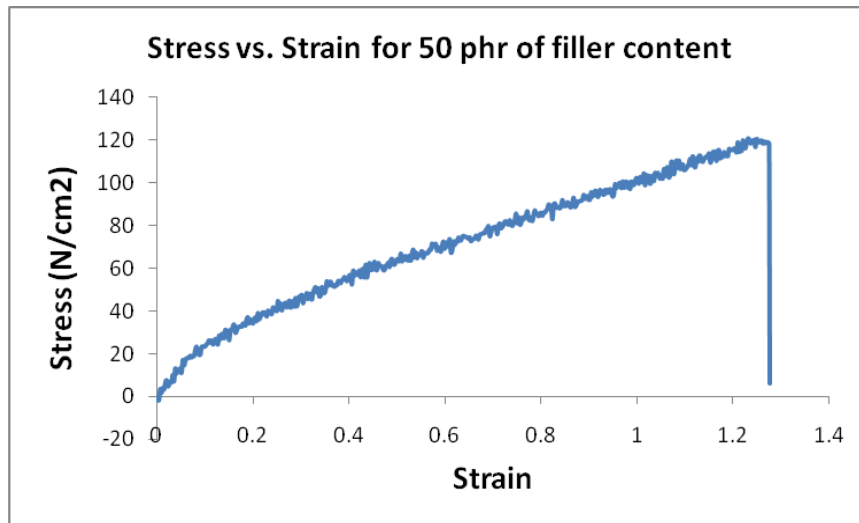


Figure 4.10: Graph of stress against strain for 50 phr of fillers content

The graph above shows the relationship between the stress and strain for 50 phr of filler content in rubber compound. The graph shows that the sample experiences high stress before it reach the break point but at the break point, it stretched about 1.2 of its initial length or strain before it reached the break point is around 1.2. It means that the Young's modulus is low compared to the 40 and 30 phr that is 83.531 N/cm². The sample also is being stretched longer than 15, 30 and 40 phr because of the high elasticity of the rubber compound that leading to the high tensile strength in order to reach the break point. The low Young's modulus is caused by the low ratio of stress over strain due to the high elongation. This might be caused by the incomplete mixing between the rubber and fillers during the mixing process and has made the poor bonding inside the rubber compound.

4.4 Swelling Test

Swelling test is conducted to determine the swelling resistance of rubber compound. Generally, among the solvent that is used are benzene and toluene but for this study diesel and kerosene are used and the testing was conducted for 24 hours. The initial weight of all samples is weighed and recorded and the samples were immersed and are allowed to swell for 24 hours. Then the final weight of rubber samples is determined and the percentage of swelling is calculated using equation 3.2. Details calculation of swelling percentage of each sample for both solvent is shown at Appendix C.

4.4.1 Effect of filler content on Swelling in Kerosene

For swelling test using kerosene as the solvent, the initial and final weight of rubber compound is shown as in table 4.1 below. The change in weight is determined by calculate the different between initial and final weight of rubber compound. Then percentage of swelling resistance is calculated by dividing the different in weight (ΔM) with initial weight (M_1) and times by 100 %.

Table 4.1: Calculation of swelling percentage in kerosene

Part	Initial weight, M_1 (g)	Final weight, M_2 (g)	Change in weight, ΔM (g)	$\Delta M/M_1$	Swelling percentage (%)
0	0.30	2.37	2.07	6.90	690.00
15	0.78	3.24	2.46	3.15	315.38
30	0.70	3.90	3.20	4.57	457.14
40	0.45	1.41	0.96	2.13	213.33
50	0.59	3.47	2.88	4.88	488.14

The swelling percentage of rubber compound in kerosene for 0 phr to 50 phr of filler content is plotted as in figure 4.11. The pattern of the graph is increase and decrease alternately between filler content.

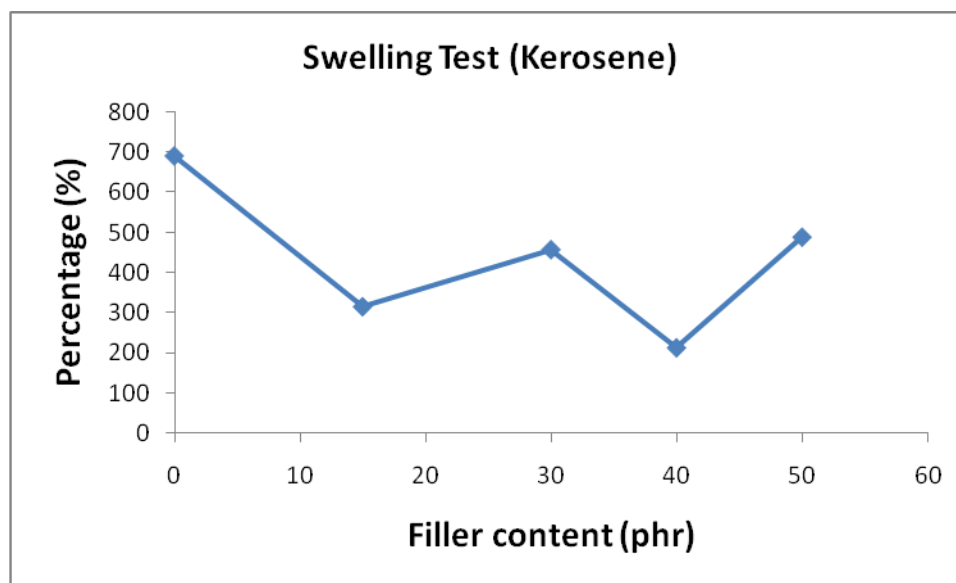


Figure 4.11: Swelling percentage of rubber compound in kerosene

The swelling percentage is decreased as the increasing amount of fillers during 0 phr to 15 phr and 30 phr to 40 phr but increase back during 15 phr to 30 phr and 40 phr to 50 phr. The swelling percentage or swelling resistance basically should decrease as the increasing amount of fillers because of decreasing amount of natural rubber and contribute better swelling resistance (Ismail *et al.*, 2002). For swelling resistance between 0 phr 15 phr shows the normal behaviour of theoretical of swelling resistance but it is found that 30 phr and 50 phr exhibits abnormal swelling resistance. It might be caused by incomplete mixing part between rubber and filler and caused the rubber component expose to the kerosene and experienced interaction between both substances.

4.4.2 Effect of filler content on Swelling in Diesel

For swelling test using diesel as the solvent, the initial and final weight of rubber compound is shown as in table 4.2 below. Same as in kerosene before, the swelling resistance is determined by the change in weight is determined by calculate the different between initial and final weight of rubber compound. Then percentage of swelling resistance is calculated by dividing the different in weight (ΔM) with initial weight (M_1) and times by 100 %.

Table 4.2: Calculation of swelling percentage in diesel

Part	Initial weight, M_1 (g)	Final weight, M_2 (g)	Change in weight, ΔM (g)	$\Delta M/M_1$	Swelling percentage (%)
0	0.4	1.52	1.12	2.80	280.00
15	0.85	2.05	1.2	1.41	141.18
30	0.65	2.07	1.42	2.18	218.46
40	0.46	1.02	0.56	1.22	121.74
50	0.71	1.86	1.15	1.62	161.97

The swelling percentage of rubber compound in diesel for 0 phr to 50 phr of filler content is plotted as in figure 4.12. The pattern of the graph is increased and decreased alternately between filler content.

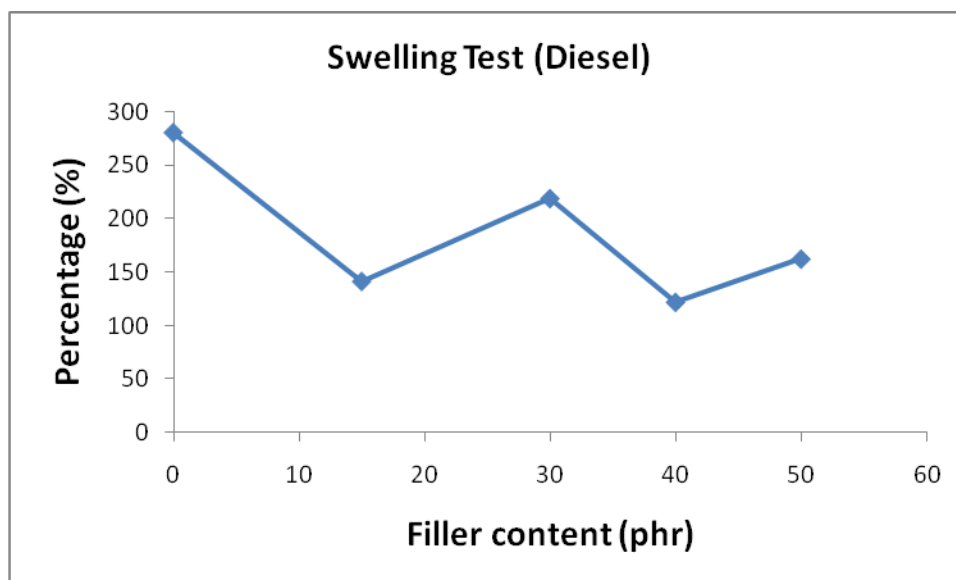


Figure 4.12: Swelling percentage of rubber compound in diesel

Graph above shows the relationship between swelling percentages with filler content that varied from 0 phr to 50 phr. The graph indicated that the swelling percentage or swelling resistance is decreased from 0 phr to 15 phr and 30 phr to 40 phr but increased back from 15 phr to 30 phr and 40 phr to 50 phr. Its shows that the graph is increased and decreased alternately due to the poor swelling resistance in rubber compound that filled with 30 phr and 50 phr. This might be caused by incomplete mixing between the fillers particle and rubber component that contributed to the poor swelling resistance. For the rubber compounds that filled with 0, 15 and 40 phr shows the decreasing trends that caused by increasing amount of fillers and made the samples become stiffer as well less penetrable by the solvent (Teh *et al.*, 2004). The increasing amount of fillers in rubber compounds has made it become the bonding agent that binds the filler fibers with natural rubber so that the swelling is highly stopped and functioned as barrier in the compound (Jacob *et al.*, 2004).

4.4.3 Comparison of Swelling Test between Both Solvent

Both graph of swelling in kerosene and diesel is plotted together in one graph and the comparison between both graphs is shown in figure 4.13 below.

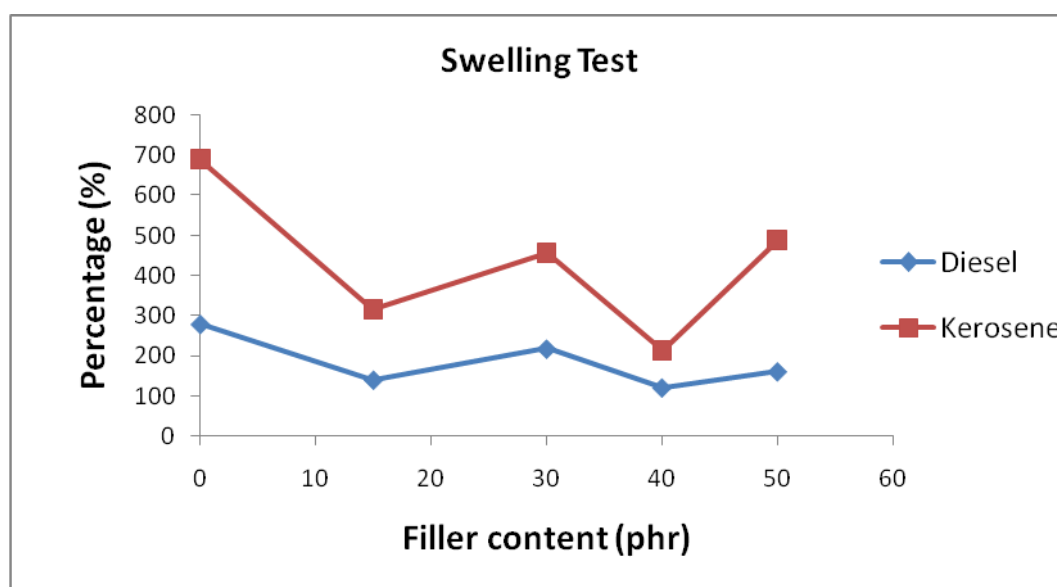


Figure 4.13: Comparison of swelling percentage between two solvent

From the graph, the swelling resistance in kerosene is higher than swelling resistance in diesel for all five samples. This is due to the smaller chemical structure of kerosene compared to the diesel which is kerosene is compilation of hydrocarbon chains that are made up of between 12 to 15 carbon atoms and diesel has a rigid molecular structure consisting of 16 carbon atoms and 34 hydrogen atoms. Besides that, kerosene has less density than diesel and both factors contribute to the high swelling percentage of kerosene. But both graphs show the similarity of trend which is swelling resistance for both graphs decreasing and increasing alternately between samples.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The effect of oil palm trunk fiber on natural rubber has been carried out by incorporation of Standard Malaysian Rubber 10 (SMR-10) with fillers and others rubber compounding. In order to measure the performance of the rubber compound, two tests has been conducted that is tensile test and swelling test. Then the performance of the rubber compound with different filler content used has been investigated and analyzed. From the results that were obtained, the increasing amount of fillers content used caused the tensile strength and Young's modulus increased until optimum 40 phr.

The increasing part of fillers content in rubber compound made the compound become more elastic and caused the elongation of samples reduce as the increasing part of fillers content. Hence, more forces are needed in order to stretch the samples until it reach the break point and the time to reach the break point also will be reduced as increasing amount of fillers content. This condition indicates that the incorporation of fillers made the tensile strength of compound increased. Mixing time also is increased as the increasing amount of fillers because the incorporation of fillers with natural rubber become harder because amount of fillers is increased but amount of natural rubber is

reduced. So there are some difficulties during incorporation of fillers with natural rubber.

Swelling test also shows the increasing amount of fillers has made the swelling percentage or swelling resistance is reduced because of the good barrier that was made by fillers. The increasing amount of fillers content made the better barrier in order to stop the penetration of solvent towards natural rubber. The swelling percentage also shows the higher value when is used in less dense solvent. The swelling resistance inside kerosene shows higher value than diesel due to the smaller chemical structure of kerosene.

5.2 Recommendation

There are some suggestions on improving or enhancing the quality of this research. The most significant part is mixing process because masterbatching part is conducted by using two roll mill. In order to improve the quality of the rubber compound, the masterbatching should be conducted by using internal mixer so that the rubber ingredients will incorporate well and become homogeneous mixture. Besides that, extrusion process should be included after masterbatching in order to mix the rubber compound until become homogenous. Another suggestion is calendering process might be included in order to obtain uniform thickness of rubber compound and could facilitate while cut the sample into dumbbell shape.

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APPENDIX A

Manual calculation of weight for each ingredient for all five samples is shown below.
The total weight of each sample is 250 grams.

First sample with 0 phr of filler loading with total part of 111

- a) Natural rubber (NR) with 100 phr from total part of 111

$$\begin{aligned}\text{Weight of NR (g)} &= \frac{100}{111} \times 250 \text{ g} \\ &= 225.23 \text{ g}\end{aligned}$$

- b) Stearic acid with 2 phr from total part of 111

$$\begin{aligned}\text{Weight of Stearic acid (g)} &= \frac{2}{111} \times 250 \text{ g} \\ &= 4.50 \text{ g}\end{aligned}$$

- c) Zinc oxide with 5 phr from total part of 111

$$\begin{aligned}\text{Weight of Zinc oxide (g)} &= \frac{5}{111} \times 250 \text{ g} \\ &= 11.26 \text{ g}\end{aligned}$$

- d) 6-PPD with 1 phr from total part of 111

$$\begin{aligned}\text{Weight of 6 - PPD (g)} &= \frac{1}{111} \times 250 \text{ g} \\ &= 2.25 \text{ g}\end{aligned}$$

e) CBS with 0.5 phr from total part of 111

$$\begin{aligned}\text{Weight of CBS (g)} &= \frac{0.5}{111} \times 250 \text{ g} \\ &= 1.13 \text{ g}\end{aligned}$$

f) Sulphur with 2.5 phr from total part of 111

$$\begin{aligned}\text{Weight of Sulphur (g)} &= \frac{2.5}{111} \times 250 \text{ g} \\ &= 5.63 \text{ g}\end{aligned}$$

g) Oil palm trunk fiber (OPTF) with 0 phr from total part of 111

$$\begin{aligned}\text{Weight of OPTF (g)} &= \frac{0}{111} \times 250 \text{ g} \\ &= 0.00 \text{ g}\end{aligned}$$

Second sample with 15 phr of filler loading with total part of 126

a) Natural rubber (NR) with 100 phr from total part of 126

$$\begin{aligned}\text{Weight of NR (g)} &= \frac{100}{126} \times 250 \text{ g} \\ &= 198.41 \text{ g}\end{aligned}$$

b) Stearic acid with 2 phr from total part of 126

$$\begin{aligned}\text{Weight of Stearic acid (g)} &= \frac{2}{126} \times 250 \text{ g} \\ &= 3.97 \text{ g}\end{aligned}$$

c) Zinc oxide with 5 phr from total part of 126

$$\begin{aligned}\text{Weight of Zinc oxide (g)} &= \frac{5}{126} \times 250 \text{ g} \\ &= 9.92 \text{ g}\end{aligned}$$

d) 6-PPD with 1 phr from total part of 126

$$\begin{aligned}\text{Weight of 6 - PPD (g)} &= \frac{1}{126} \times 250 \text{ g} \\ &= 1.98 \text{ g}\end{aligned}$$

e) CBS with 0.5 phr from total part of 126

$$\begin{aligned}\text{Weight of CBS (g)} &= \frac{0.5}{126} \times 250 \text{ g} \\ &= 0.99 \text{ g}\end{aligned}$$

f) Sulphur with 2.5 phr from total part of 126

$$\begin{aligned}\text{Weight of Sulphur (g)} &= \frac{2.5}{126} \times 250 \text{ g} \\ &= 4.96 \text{ g}\end{aligned}$$

g) Oil palm trunk fiber (OPTF) with 15 phr from total part of 126

$$\begin{aligned}\text{Weight of OPTF (g)} &= \frac{15}{126} \times 250 \text{ g} \\ &= 29.76 \text{ g}\end{aligned}$$

Third sample with 30 phr of filler loading with total part of 141

- a) Natural rubber (NR) with 100 phr from total part of 141

$$\begin{aligned}\text{Weight of NR (g)} &= \frac{100}{141} \times 250 \text{ g} \\ &= 177.30 \text{ g}\end{aligned}$$

- b) Stearic acid with 2 phr from total part of 141

$$\begin{aligned}\text{Weight of Stearic acid (g)} &= \frac{2}{141} \times 250 \text{ g} \\ &= 3.55 \text{ g}\end{aligned}$$

- c) Zinc oxide with 5 phr from total part of 141

$$\begin{aligned}\text{Weight of Zinc oxide (g)} &= \frac{5}{141} \times 250 \text{ g} \\ &= 8.87 \text{ g}\end{aligned}$$

- d) 6-PPD with 1 phr from total part of 141

$$\begin{aligned}\text{Weight of 6 – PPD (g)} &= \frac{1}{141} \times 250 \text{ g} \\ &= 1.77 \text{ g}\end{aligned}$$

- e) CBS with 0.5 phr from total part of 141

$$\begin{aligned}\text{Weight of CBS (g)} &= \frac{0.5}{141} \times 250 \text{ g} \\ &= 0.89 \text{ g}\end{aligned}$$

- f) Sulphur with 2.5 phr from total part of 141

$$\text{Weight of Sulphur (g)} = \frac{2.5}{141} \times 250 \text{ g}$$

$$= 4.43 \text{ g}$$

g) Oil palm trunk fiber (OPTF) with 30 phr from total part of 141

$$\text{Weight of OPTF (g)} = \frac{30}{141} \times 250 \text{ g}$$

$$= 53.19 \text{ g}$$

Fourth sample with 40 phr of filler loading with total part of 151

a) Natural rubber (NR) with 100 phr from total part of 151

$$\text{Weight of NR (g)} = \frac{100}{151} \times 250 \text{ g}$$

$$= 165.56 \text{ g}$$

b) Stearic acid with 2 phr from total part of 151

$$\text{Weight of Stearic acid (g)} = \frac{2}{151} \times 250 \text{ g}$$

$$= 3.31 \text{ g}$$

c) Zinc oxide with 5 phr from total part of 151

$$\text{Weight of Zinc oxide (g)} = \frac{5}{151} \times 250 \text{ g}$$

$$= 8.28 \text{ g}$$

d) 6-PPD with 1 phr from total part of 151

$$\text{Weight of 6 - PPD (g)} = \frac{1}{151} \times 250 \text{ g}$$

$$= 1.66 \text{ g}$$

e) CBS with 0.5 phr from total part of 151

$$\begin{aligned}\text{Weight of CBS (g)} &= \frac{0.5}{151} \times 250 \text{ g} \\ &= 0.83 \text{ g}\end{aligned}$$

f) Sulphur with 2.5 phr from total part of 151

$$\begin{aligned}\text{Weight of Sulphur (g)} &= \frac{2.5}{151} \times 250 \text{ g} \\ &= 4.14 \text{ g}\end{aligned}$$

g) Oil palm trunk fiber (OPTF) with 40 phr from total part of 151

$$\begin{aligned}\text{Weight of OPTF (g)} &= \frac{40}{151} \times 250 \text{ g} \\ &= 66.23 \text{ g}\end{aligned}$$

Fifth sample with 50 phr of filler loading

a) Natural rubber (NR) with 100 phr from total part of 161

$$\begin{aligned}\text{Weight of NR (g)} &= \frac{100}{161} \times 250 \text{ g} \\ &= 155.28 \text{ g}\end{aligned}$$

b) Stearic acid with 2 phr from total part of 161

$$\begin{aligned}\text{Weight of Stearic acid (g)} &= \frac{2}{161} \times 250 \text{ g} \\ &= 3.11 \text{ g}\end{aligned}$$

c) Zinc oxide with 5 phr from total part of 161

$$\begin{aligned}\text{Weight of Zinc oxide (g)} &= \frac{5}{161} \times 250 \text{ g} \\ &= 7.76 \text{ g}\end{aligned}$$

d) 6-PPD with 1 phr from total part of 161

$$\begin{aligned}\text{Weight of 6 - PPD (g)} &= \frac{1}{161} \times 250 \text{ g} \\ &= 1.55 \text{ g}\end{aligned}$$

e) CBS with 0.5 phr from total part of 161

$$\begin{aligned}\text{Weight of CBS (g)} &= \frac{0.5}{161} \times 250 \text{ g} \\ &= 0.78 \text{ g}\end{aligned}$$

f) Sulphur with 2.5 phr from total part of 161

$$\begin{aligned}\text{Weight of Sulphur (g)} &= \frac{2.5}{161} \times 250 \text{ g} \\ &= 3.88 \text{ g}\end{aligned}$$

g) Oil palm trunk fiber (OPTF) with 50 phr from total part of 161

$$\begin{aligned}\text{Weight of OPTF (g)} &= \frac{50}{161} \times 250 \text{ g} \\ &= 77.64 \text{ g}\end{aligned}$$

APPENDIX B

Results of effect of filler content on natural rubber by using tensile test are listed as below:

- a) Tensile strength
- b) Elongation
- c) Time at break
- d) Young modulus
- e) Mixing time

Table B.1: Results of tensile test

Part	Tensile strength (MPa)	Elongation (mm)	Time at break (s)	Young Modulus (N/cm ²)	Mixing time (hour)
0	0.8349	124.9059	374.718	31.555	2.0
15	0.6565	72.8954	218.686	41.977	3.5
30	0.7900	37.9326	113.798	92.170	4.0
40	0.9207	34.8599	104.500	123.300	5.0
50	1.2047	81.5947	244.784	83.531	6.5

Sample of 0 phr of filler content

Area = 0.08272 cm²

Length = 6.4 cm

Table B.2: Stress against strain for 0 phr of fillers content

Time (s)	Load (N)	Elongation (cm)	Stress (N/cm ²)	Strain
0	0.140377	0	1.697015232	0
7	0.441641	0.233325	5.338984526	0.036457
14	1.006505	0.466681	12.16761364	0.0729189
21	1.498873	0.699993	18.11983801	0.1093739

28	1.873077	0.933319	22.64358075	0.1458311
35	2.192362	1.166675	26.50340909	0.182293
42	2.292235	1.4	27.71077128	0.21875
49	2.632328	1.633325	31.822147	0.255207
56	2.460418	1.866669	29.74393133	0.291667
63	2.69664	2.1	32.59961315	0.328125
70	2.82343	2.333331	34.13237427	0.364583
77	2.825116	2.566669	34.15275629	0.401042
84	2.918654	2.799993	35.28353482	0.4374989
91	2.994656	3.033337	36.20232108	0.4739589
98	3.078998	3.266668	37.2219294	0.5104169
105	3.192773	3.499987	38.59735251	0.546873
112	3.271806	3.733331	39.55278046	0.583333
119	3.333538	3.966675	40.29905706	0.619793
126	3.619253	4.199988	43.75305851	0.6562481
133	3.584777	4.433325	43.33627901	0.692707
140	3.568856	4.666669	43.14381044	0.729167
147	3.888385	4.9	47.00658849	0.765625
154	3.596323	5.133325	43.47585832	0.802082
161	3.928447	5.366675	47.490897	0.838543
168	4.070544	5.599994	49.20870406	0.8749991
175	3.680783	5.833331	44.49689313	0.911458
182	3.785515	6.0666687	45.76299565	0.947917
189	4.024533	6.2999937	48.65247824	0.984374
196	4.262868	6.5333375	51.53370406	1.020834
203	4.184195	6.7666687	50.58262814	1.057292
210	4.316914	6.9999937	52.1870648	1.093749
217	4.347716	7.2333375	52.5594294	1.130209
224	4.588527	7.4666749	55.47058752	1.166668
231	4.711981	7.6999936	56.96301983	1.203124
238	4.913203	7.933331	59.39558752	1.239583
245	4.693975	8.166669	56.74534574	1.276042
252	4.99667	8.399994	60.40461799	1.3124991
259	5.248037	8.633325	63.44338733	1.348957
266	5.02669	8.866675	60.76752901	1.385418
273	5.218431	9.1	63.08548114	1.421875
280	5.319793	9.333325	64.31084381	1.458332
287	5.076739	9.566668	61.37257012	1.4947919
294	5.354321	9.799993	64.72825193	1.5312489
301	5.938128	10.03333	71.78588008	1.5677078
308	5.603793	10.266668	67.74411267	1.6041669

315	6.210709	10.499993	75.08110493	1.6406239
322	5.637226	10.733331	68.14828337	1.677083
329	6.122155	10.966668	74.01057785	1.7135419
336	5.723604	11.199993	69.19250484	1.7499989
343	6.014836	11.433333	72.71320116	1.7864578
350	6.452065	11.666674	77.99885155	1.8229178
357	6.499844	11.899993	78.57645068	1.8593739
364	6.457091	12.133318	78.05961074	1.8958309
371	6.860881	12.366674	82.94101789	1.9322928

Sample of 15 phr of filler content

Area = 0.144288 cm²

Length = 6.4 cm

Table B.3: Stress against strain for 15 phr of fillers content

Time (s)	Load (N)	Extension (cm)	Stress (N/cm ²)	Strain
0	-0.1305861	0	0	-0.90504
4	0.9490183	0.133338	0.020834063	6.57725
8	1.94591	0.266675	0.041667969	13.48629
12	2.809807	0.399993	0.062498906	19.4736
16	3.56656	0.533337	0.083333906	24.71834
20	3.860846	0.666681	0.104168906	26.75791
24	4.221441	0.8	0.125	29.25705
28	4.328216	0.933344	0.145835	29.99706
32	4.693418	1.066682	0.166669063	32.52812
36	4.825025	1.2	0.1875	33.44024
40	5.179203	1.333338	0.208334063	35.8949
44	5.371735	1.466675	0.229167969	37.22926
48	5.486512	1.6	0.25	38.02473
52	5.490471	1.733344	0.270835	38.05217
56	5.697672	1.866682	0.291669063	39.48819
60	5.964646	2	0.3125	41.33848
64	6.211873	2.133338	0.333334063	43.0519
68	6.15633	2.266675	0.354167969	42.66696
72	6.467088	2.399988	0.374998125	44.82069
76	6.399224	2.533337	0.395833906	44.35035
80	6.675989	2.666675	0.416667969	46.2685

84	6.655908	2.8	0.4375	46.12932
88	6.77235	2.933344	0.458335	46.93634
92	6.91067	3.066675	0.479167969	47.89497
96	6.990625	3.199994	0.499999063	48.44911
100	7.387717	3.333331	0.520832969	51.20119
104	7.261249	3.466675	0.541667969	50.32469
108	7.150363	3.599994	0.562499063	49.55619
112	7.387375	3.733338	0.583334063	51.19882
116	7.62875	3.866681	0.604168906	52.87169
120	7.529343	4.000006	0.625000938	52.18274
124	7.864524	4.133338	0.645834063	54.50574
128	8.08332	4.266682	0.666669063	56.02212
132	7.908198	4.399994	0.687499063	54.80842
136	8.037833	4.533331	0.708332969	55.70687
140	8.302345	4.666681	0.729168906	57.54009
144	8.347892	4.8	0.75	57.85576
148	8.411727	4.933344	0.770835	58.29817
152	8.724563	5.066675	0.791667969	60.46631
156	8.333733	5.2	0.8125	57.75763
160	8.264172	5.333338	0.833334063	57.27553
164	8.751602	5.466675	0.854167969	60.65371
168	8.72292	5.6	0.875	60.45492
172	8.70742	5.733344	0.895835	60.3475
176	8.81236	5.866681	0.916668906	61.07479
180	8.749301	6.0000002	0.937500031	60.63776
184	9.155331	6.1333377	0.958334016	63.45178
188	9.079739	6.2666753	0.979168016	62.92789
192	9.126755	6.3999877	0.999998078	63.25374
196	9.049659	6.5333315	1.020833047	62.71942
200	9.064227	6.6666815	1.041668984	62.82038
204	9.067325	6.8000003	1.062500047	62.84185
208	9.180049	6.93333775	1.083334023	63.62309
212	9.035023	7.06668149	1.104168983	62.61798
216	9.034742	7.199994	1.124999063	62.61603

Sample of 30 phr of filler content

Area = 0.098412 cm²

Length = 6.4 cm

Table B.4: Stress against strain for 30 phr of fillers content

Time (s)	Load (N)	Elongation (cm)	Stress (N/cm ²)	Strain
0	-0.03899	0	-0.39617	0
2	0.513766	0.066663	5.220557	0.010416
4	1.321448	0.133332	13.42771	0.020833
6	2.046018	0.2	20.79033	0.03125
8	2.86524	0.266663	29.11474	0.041666
10	3.193801	0.333337	32.45337	0.052084
12	3.324105	0.4	33.77744	0.0625
14	3.771512	0.466656	38.3237	0.072915
16	4.341557	0.533338	44.11613	0.083334
18	4.629076	0.600006	47.03772	0.093751
20	4.799368	0.666656	48.76812	0.104165
22	5.204697	0.733331	52.88681	0.114583
24	5.10611	0.8	51.88503	0.125
26	5.236846	0.866663	53.21349	0.135416
28	5.25957	0.933338	53.4444	0.145834
30	5.430946	1.000001	55.18581	0.15625
32	5.378832	1.066656	54.65626	0.166665
34	5.630923	1.133331	57.21785	0.177083
36	5.903264	1.2	59.98521	0.1875
38	5.833623	1.266656	59.27756	0.197915
40	5.914982	1.333332	60.10428	0.208333
42	5.945887	1.4	60.41831	0.21875
44	6.194156	1.466656	62.94106	0.229165
46	6.444378	1.533344	65.48366	0.239585
48	6.322406	1.600006	64.24426	0.250001
50	6.440946	1.666669	65.44879	0.260417
52	6.514368	1.733338	66.19485	0.270834
54	6.353929	1.800006	64.56458	0.281251
56	6.679579	1.866663	67.87362	0.291666
58	6.634655	1.933338	67.41713	0.302084
60	6.552175	1.999988	66.57902	0.312498
62	6.752088	2.066662	68.61041	0.322916

64	6.875351	2.133332	69.86293	0.333333
66	6.826531	2.199994	69.36686	0.343749
68	7.205298	2.266663	73.21564	0.354166
70	7.255733	2.333332	73.72813	0.364583
72	6.989887	2.4	71.02678	0.375
74	7.008412	2.466663	71.21501	0.385416
76	7.091596	2.533331	72.06028	0.395833
78	7.029631	2.600007	71.43063	0.406251
80	7.215978	2.666663	73.32417	0.416666
82	7.318497	2.733338	74.3659	0.427084
84	7.375135	2.8	74.94142	0.4375
86	7.304932	2.866662	74.22806	0.447916
88	7.367642	2.933331	74.86528	0.458333
90	7.15069	3.000006	72.66075	0.468751
92	7.521734	3.066657	76.43107	0.479165
94	7.504088	3.133325	76.25176	0.489582
96	7.554393	3.199994	76.76293	0.499999
98	7.708315	3.266663	78.32698	0.510416
100	7.415467	3.333331	75.35125	0.520833
102	7.354166	3.4	74.72835	0.53125
104	7.640983	3.466656	77.6428	0.541665
106	7.524021	3.533325	76.4543	0.552082
108	7.528411	3.600006	76.49891	0.562501
110	7.465979	3.666663	75.86452	0.572916
112	7.287712	3.733338	74.05308	0.583334

Sample of 40 phr of filler content

Area = 0.20976 cm²

Length = 6.4 cm

Table B.4: Stress against strain for 40 phr of fillers content

Time (s)	Load (N)	Elongation (cm)	Stress (N/cm ²)	Strain
0	-0.013786	0	-0.065723017	0
2	2.527077	0.066668	12.04746854	0.0104169
4	3.944302	0.133325	18.80388063	0.020832
6	5.779432	0.2	27.55259344	0.03125
8	7.033237	0.266663	33.52992468	0.0416661

10	8.056575	0.333324	38.40853833	0.0520819
12	9.121337	0.400006	43.48463482	0.0625009
14	9.5027	0.466668	45.30272693	0.0729169
16	10.2483	0.533331	48.85726545	0.0833333
18	10.84338	0.600012	51.69422197	0.0937519
20	11.55745	0.666674	55.09844584	0.1041678
22	11.95596	0.733325	56.99828375	0.114582
24	12.37592	0.8	59.00038139	0.125
26	13.09717	0.866663	62.43883486	0.1354161
28	13.32054	0.933325	63.50371854	0.145832
30	13.51606	1	64.43583143	0.15625
32	14.13794	1.066669	67.40055301	0.166667
34	14.35475	1.133325	68.43416285	0.177082
36	14.39647	1.199994	68.63305683	0.1874991
38	14.38342	1.266662	68.57084287	0.1979159
40	14.76254	1.333331	70.3782418	0.208333
42	15.24173	1.4	72.66270976	0.21875
44	15.24567	1.466675	72.68149314	0.229168
46	15.56929	1.533331	74.22430397	0.239583
48	15.54111	1.600006	74.08995995	0.2500009
50	15.91584	1.666675	75.87643021	0.260418
52	16.26414	1.733331	77.53689931	0.270833
54	16.55671	1.8	78.93168383	0.28125
56	16.12894	1.866669	76.89235317	0.291667
58	16.74024	1.933325	79.806663616	0.302082
60	16.96448	2	80.87566743	0.3125
62	16.92548	2.066662	80.68974066	0.3229159
64	17.40368	2.133325	82.96948894	0.333332
66	17.40925	2.199994	82.9960431	0.3437491
68	17.6959	2.266669	84.36260488	0.354167
70	17.52055	2.333325	83.5266495	0.364582
72	17.91297	2.400006	85.39745423	0.3750009
74	17.67135	2.466675	84.24556636	0.385418
76	17.99329	2.533325	85.78036804	0.395832
78	18.14636	2.600006	86.51010679	0.4062509
80	18.29022	2.666675	87.19593822	0.416668
82	18.23984	2.733325	86.95575896	0.427082
84	18.44099	2.8	87.91471205	0.4375
86	18.59325	2.866662	88.64058924	0.4479159
88	18.48403	2.933331	88.11989893	0.458333
90	18.94208	3	90.30358505	0.46875

92	19.09702	3.066662	91.04223875	0.4791659
94	19.01831	3.133325	90.66700038	0.489582
96	18.98055	3.199999	90.48698513	0.4999998
98	19.07942	3.266669	90.95833333	0.510417
100	17.21343	3.333331	82.0625	0.520833
102	1.99681	3.400006	9.519498474	0.5312509
104	1.922345	3.466669	9.164497521	0.541667

Sample of 50 phr of filler content

Area = 0.1088 cm²

Length = 6.4 cm

Table B.6: Stress against strain for 50 phr of fillers content

Time (s)	Load (N)	Elongation (cm)	Stress (N/cm ²)	Strain
0	0.002015	0	0.018517316	0
4	0.853593	0.133331	7.845525735	0.020833
8	1.196232	0.266668	10.99477941	0.041667
12	1.971829	0.399993	18.12342831	0.062499
16	2.552408	0.533325	23.45963235	0.083332
20	2.680499	0.666662	24.63693934	0.104166
24	2.919279	0.799987	26.83160846	0.124998
28	3.330827	0.933324	30.61421875	0.145832
32	3.367323	1.066662	30.94965993	0.166666
36	3.938905	1.199993	36.20317096	0.187499
40	3.857168	1.333337	35.45191176	0.208334
44	4.090168	1.466668	37.59345588	0.229167
48	4.361664	1.599987	40.08882353	0.249998
52	4.549421	1.733331	41.81453125	0.270833
56	4.603727	1.866662	42.31366728	0.291666
60	5.271307	1.999987	48.44951287	0.312498
64	5.313124	2.133331	48.83386029	0.333333
68	5.873272	2.266662	53.98227941	0.354166
72	5.67348	2.399993	52.14595588	0.374999
76	5.875414	2.533337	54.00196691	0.395834
80	6.320302	2.666668	58.09101103	0.416667
84	6.06927	2.799981	55.78373162	0.437497
88	6.436167	2.933324	59.15594669	0.458332

92	6.642283	3.066656	61.05039522	0.479165
96	6.992057	3.199993	64.26522978	0.499999
100	7.015176	3.333331	64.47772059	0.520833
104	7.044589	3.466668	64.74806066	0.541667
108	7.284508	3.599993	66.95319853	0.562499
112	7.421607	3.733318	68.21329963	0.583331
116	7.846938	3.866656	72.12259191	0.604165
120	7.869157	3.999981	72.32681066	0.624997
124	8.087388	4.133331	74.33261029	0.645833
128	8.130381	4.266662	74.72776654	0.666666
132	8.539279	4.399993	78.48602022	0.687499
136	8.264854	4.533337	75.96373162	0.708334
140	8.812698	4.666662	80.9990625	0.729166
144	8.804552	4.799987	80.92419118	0.749998
148	9.422771	4.933331	86.6063511	0.770833
152	9.288157	5.066662	85.36909007	0.791666
156	9.537016	5.199987	87.65639706	0.812498
160	9.608796	5.333337	88.31613971	0.833334
164	9.876295	5.466668	90.77477022	0.854167
168	9.905265	5.599993	91.0410386	0.874999
172	10.20007	5.733325	93.75064338	0.895832
176	10.1643	5.866662	93.421875	0.916666
180	10.51495	5.999987	96.64476103	0.937498
184	10.76912	6.1333245	98.98088235	0.958332
188	10.62537	6.2666745	97.65965074	0.979168
192	10.99771	6.3999933	101.0818934	0.999999
196	11.01247	6.5333308	101.2175551	1.020833
200	11.37519	6.666662	104.5513787	1.041666
204	11.6476	6.799987	107.0551471	1.062498
208	11.63182	6.93331825	106.9101103	1.083331
212	11.71179	7.06665573	107.6451287	1.104165
216	11.92312	7.199987	109.5875	1.124998
220	12.16255	7.3333369	111.7881434	1.145834
224	12.50144	7.4666619	114.9029412	1.166666
228	12.2139	7.5999869	112.2601103	1.187498
232	12.91913	7.7333307	118.7420037	1.208333
236	13.1079	7.8666557	120.4770221	1.229165
240	12.95099	7.999981	119.0348346	1.249997
244	12.92042	8.133331	118.7538603	1.270833

APPENDIX C

Table C.1: Initial and final weight of samples in kerosene and diesel

Part	Kerosene		Diesel	
	Initial weight	Final weight	Initial weight	Final weight
0	0.3	2.37	0.4	1.52
15	0.78	3.24	0.85	2.05
30	0.7	3.9	0.65	2.07
40	0.45	1.41	0.46	1.02
50	0.59	3.47	0.71	1.86

Detail calculations of swelling percentage of rubber compounds in kerosene.

a) 0 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{2.37 - 0.30}{0.30} \times 100 \% \\ &= 690.00 \%\end{aligned}$$

b) 15 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{3.24 - 0.78}{0.78} \times 100 \% \\ &= 315.38 \%\end{aligned}$$

c) 30 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{3.90 - 0.70}{0.70} \times 100 \% \\ &= 457.14 \%\end{aligned}$$

d) 40 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{1.41 - 0.45}{0.45} \times 100 \% \\ &= 213.33 \%\end{aligned}$$

e) 50 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{3.47 - 0.59}{0.59} \times 100 \% \\ &= 488.14 \%\end{aligned}$$

Detail calculations of swelling percentage of rubber compounds in diesel.

a) 0 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{1.52 - 0.40}{0.40} \times 100 \% \\ &= 280.00 \%\end{aligned}$$

b) 15 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{2.05 - 0.85}{0.85} \times 100 \% \\ &= 141.18 \%\end{aligned}$$

c) 30 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{2.07 - 0.65}{0.65} \times 100 \% \\ &= 218.46 \%\end{aligned}$$

d) 40 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{1.02 - 0.46}{0.46} \times 100 \% \\ &= 121.74 \%\end{aligned}$$

e) 50 phr of filler content

$$\begin{aligned}\text{Swelling percentage (\%)} &= \frac{1.86 - 0.71}{0.71} \times 100 \% \\ &= 161.97 \%\end{aligned}$$

The Effect of Oil Palm Trunk Fiber on Characterization of Natural Rubber

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Abstract

The strength of a material to stress depends on the chemical bonds found in the chemical structure of the material itself. This thesis deals with the effect of different filler loading on the natural rubber vulcanizates so that the dynamic mechanical properties of vulcanized natural rubber can be obtained. The objective of this thesis is to study the effect of different filler loading or amount of filler loaded on natural rubber that is Standard Malaysian Rubber 10 (SMR-10). For this study, natural filler that is oil palm trunk fiber which is varies from 0 to 50 phr have been used as an additive to strengthen the chemical bonds of natural rubber as well as other chemicals that were added to natural rubber. Rubber compounds filled with natural fillers and other chemical additives is prepared using a two roll mill and the characteristics of the mechanical properties are studied and analyzed to determine the effect on the strength of natural rubber when natural filler is added as filler. The functions for each material that is stated above might be vary based on their physical and chemical properties data but all fillers generally increase the tensile strength and modulus of vulcanized natural rubber into particular level.

Abstrak

Kekuatan sesuatu bahan terhadap tekanan bergantung kepada ikatan kimia yang terdapat didalam struktur kimia bahan itu sendiri. Tesis ini berkaitan dengan kesan perbezaan jumlah kemasukan bahan pengisi semula jadi pada getah asli yang divulkanasikan supaya sifat-sifat kekuatan dan sifat mekanik dinamik dari getah alam yang divulkanasikan boleh diperolehi. Tujuan tesis ini adalah untuk mempelajari kesan perbezaan jumlah kemasukan bahan pengisi semula jadi ke atas getah asli iaitu Standard Malaysian Rubber 10 (SMR-10). Untuk kajian ini, bahan penambah asli iaitu fiber batang kelapa sawit telah digunakan sebagai bahan penambah untuk menguatkan ikatan kimia pada getah asli serta bahan kimia lain yang turut ditambah bersama ke atas getah asli. Sebatian getah yang dicampur dengan bahan pengisi asli serta bahan penambah lain disediakan menggunakan 'two roll mill' dan ciri-ciri terhadap sifat-sifat mekanikal dikaji serta dianalisa untuk melihat kesan terhadap kekuatan getah asli apabila bahan pengisi semula jadi ditambah sebagai bahan pengisi. Fungsi untuk setiap material yang dinyatakan di atas mungkin berbeza-beza dari segi sifat-sifat fizik dan kimianya tetapi semua bahan pengisi ini secara umumnya berfungsi meningkatkan modulus dan kekuatan tensile getah asli yang divulkanasikan ke suatu tahap tertentu.

Keywords: oil palm trunk fiber, natural rubber, natural filler, mechanical properties.

1.0 INTRODUCTION

Natural rubber is a chemical compound that produced from latex. Usually latex is collected from the tapped rubber tree and also can be produced by synthetically. Both the natural and synthetic rubber is being used widely in many applications and products. But there is a different between natural rubber and synthetic rubber especially their strength or its elasticity. Natural rubber is known to exhibit numerous outstanding properties such as good oil resistance, and low gas permeability (Kaushik et al., 2010). It is also more elastic than the synthetic rubber and because of its special properties made it preferred raw material for manufacturers that producing rubber products.

Rubber tree in Malaysia is brought from Brazil by Sir Henry Wickham in 1876. He gathered thousand of rubber tree seeds and these seeds were shipped to Sri Lanka, Indonesia,

Singapore, and Malaysia. Then Malaya (Malaysia) was later to become the biggest producer of rubber at that time. New plantations areas also have been started in Thailand, Philippines, Africa, and Europe to support insufficient raw material in Malaysia and Indonesia. Today, Malaysia, Indonesia and Thailand are among the biggest rubber producers and produces almost 80% of world's natural rubber.

Filler is a compound that is used to enhance the quality of rubber products. Generally, filler has no fixed classification but it can be categorized into two types according their colour that are black filler and white filler. In order to improve the strength of natural rubber, filler is added while processing the natural rubber and the mechanism took part in this process is fillers could modify the properties of natural rubber (Chuayjuljit *et al.*, 2002). Most

popular reinforcing filler is carbon black which is widely used in tyre manufacturing industry.

Carbon black is material that produced from incomplete combustion of gaseous or liquid hydrocarbon in the form of colloidal particles. Usually, its physical appearance is in form of black powder and widely used in rubber and plastic industry. It is also available in various sizes and specifications. In tyre manufacturing industry, carbon black is used as reinforcing agent which is it enhance the strength and improve the properties of natural rubber.

Besides being as reinforcing agent, carbon black also can conduct heat away from tyre and make it become long-lasting. Smaller size of the carbon black used in rubber processing will cause bigger surface area of the filler. This will contribute great and high degree of reinforcement towards natural rubber. (Peng, 2007).

Beside the widely used carbon black fillers, there is another type of filler that is non-black filler. Non-black filler or white filler is can be differentiate by its colour and label. Most popular white fillers that are used in rubber processing are calcium carbonate, precipitated silica and clay. Like carbon black, white fillers also can give different effect based on their particle size. The smaller size of the white filler and then more big surface area of the white filler which is the bigger surface area give greatest degree of reinforcement towards natural rubber.

Non-black fillers like natural fillers such as rice husk, empty fruit bunch and oil palm trunk fiber together with clay and asbestos is being used as replacement for black filler to increase the production as well diversify of rubber product. The addition of these reinforcing agents also functioning as additives that will be enhance the strength, increase hardness and reduce the cost of compound (Peng, 2007). Another example of non-black filler are organoclay and alumina nanoparticles.

Natural fillers generally can be described as reinforcing agent that was produced from natural resources like rice husk, empty fruit bunch and oil palm trunk fiber. Basically, the function of these natural fillers might be same as chemical fillers that are to enhance the mechanical properties of rubber compound so that it reaches certain level of improvement. There is a question about how natural fillers that was produced from the plantation waste can be used as fillers in rubber processing. In recent years, a lot of study has been done to investigate the effectiveness of natural fillers on

characterization of natural rubber. The contribution towards enhancing or improving the mechanical properties might be become significant and the main target, hence natural fillers is used in research. The advantages of using the natural filler instead of chemical fillers like carbon black is it is not harmful to human health compared to the carbon black for example. The most significant part is the cost might be lower than chemical filler but can give quite same level of reinforcement of rubber compounds.

2.0 METHODOLOGY

2.1 Equipment

Two Roll Mill

Two roll mill in FKKSA's laboratory was used in rubber processing for research of effect of oil palm trunk fiber on characterization of natural rubber. This equipment consists of two metal rollers and a heater so that the metal roller can be heat up to 100 °C.

Hot & Cold Molding Press

Hot and cold molding press in FKKSA's laboratory is used to prepare the rubber compound in the form of homogenous sheet so that the sheet can be cut into dumbbell shape for testing procedures.

Tensile Testing Unit

Tensile testing was conducted by using the tensile testing unit at FKP's laboratory at UMP Kampus Gambang. This machine can be loaded until maximum of 50 kN and can be used for various types of material such as metal, nanocomposite, polymer and others materials.

2.2 Preparation of Raw Material

The natural rubber that is Standard Malaysian Rubber 10 (SMR-10) and oil palm trunk fiber are obtained from the supplier in a bulk package. In order to prepare the raw material that need small amount that is 250 grams per sample, it is necessary to cut the natural rubber into small amount or in the form of slice with the thickness of around 1 centimeter. The oil palm trunk fiber also needs to be sieve and the ashes are collected while the fiber waste is eliminated. Table 1 below is the shows the rubber formulation for this research.

Table 1: Rubber formulation

Ingredient	Formulation (phr)				
	1	2	3	4	5
SMR-10	100	100	100	100	100
Stearic Acid	2	2	2	2	2
Zinc Oxide	5	5	5	5	5
6-PPD ^a	1	1	1	1	1
CBS ^b	0.5	0.5	0.5	0.5	0.5
Sulphur	2.5	2.5	2.5	2.5	2.5
OPTF ^c	0	15	30	40	50
Total	111	126	141	151	161

^aN-(1,3-dimethylbutyl)-N'-phenyl-p-phenylene-diamine)

^bN-cyclohexyl-2-benzothiazyl sulphenamide

^cOil Palm Trunk Fiber

Natural Rubber

Natural rubber is obtained in bulk package and to prepare into small amount, it is necessary to cut the rubber into slice using a small saw. The compound it is in form of high viscosity and has high elasticity hence it is hard to cut the rubber into smaller size. Then the sample of rubber is weighed according to the rubber formulation and was keep as well labeled. After that, the rubber slice will undergo mastication to prepare rubber compound that well homogeneous and low viscosity.

Filler

Oil palm trunk fiber is in the form of coagulation of waste palm oil fiber. In order to obtain the powder from the fiber, it is necessary to sieve the filler using siever and was sieved manually using size of 400 µm. the filler has a light weight and for every session of sieving, only produce around 20% from the coagulate fiber. Hence the sieving process was taking a long time in order to yield around 226.82 grams of fillers for all samples.

The sieving process started by taking some amount of coagulates oil palm trunk fiber into siever and was shacked so that the powder will fall down with the uniform size. The process is repeated for large amount of filler in order to obtain total amount of filler for each sample.

Chemicals

Chemicals are used as additives for the rubber processing and five chemicals were used and can be separate into two groups. First is the activator and antidegradant group and second is vulcanizing group that represent stage 1 and stage 2.

Table 2: Summary of chemical in rubber compounding

Stage 1	Stage 2
Zinc oxide	Sulphur
Stearic acid	CBS
6-PPD	

All chemicals are weighed according to the rubber formulation and were packed according to the stages. Zinc oxide, stearic acid and 6-PPD are packed together and were shacked so that it well mixed in a mixture. The rubber processing is conducted by addition of chemicals by stages. Each of the stage is added to the rubber compound and for each interval of the stages will take some period in order to ensure the rubber compound is well mixed with the chemicals.

Weight Calculation of Each Ingredient

The rubber formulation in table 3.1 shows the part per hundred rubber (phr) for each ingredient. In order to proceed to the rubber processing, it is necessary to calculate the weight of each compound based on the phr as listed in the rubber formulation. The equation that can be used to calculate the weight is shown below.

$$\text{Weight of ingredient (g)} = \frac{P_i}{P_t} \times 250 \text{ g} \quad (2.1)$$

Where P_i is equal to number part of ingredients (phr) and P_t is equal to total parts of a compound (phr).

2.3 Mastication

Mastication is a process that was conducted to prepare the rubber compounds that can readily accept the addition of chemicals or additives for rubber processing. The objective of this process is to break the polymer chains so that the viscosity of rubber compounds is reduce as well as the elasticity of the rubber. The slice of rubber that was cut from the bulk rubber with the weight according to the rubber formulation is allowed to go through the two roll mill. The rubber slice then is compressed by two roll mill with particular gap between both rollers at temperature of 70 °C and speed of 50 rpm.

2.4 Compounding

Compounding or mixing is conducted to incorporate all the chemicals and fillers with natural rubber until become homogenous mixture. All chemicals in rubber formulation are

incorporated by stages and can be classified into three stages. First stage is addition of fillers with natural rubber, second stage is addition of chemicals to natural rubber that was added with fillers and last stage is addition of vulcanization agent with present of accelerator. The details of each stage are described below.

Addition of Fillers

The first stage is started with addition of filler that is oil palm trunk fiber into natural rubber by using two roll mill and the rubber compound is compressed by two metal rollers. Fillers that is in form of powder is added properly to the rubber compound in the present of heat around 70 °C from the metal rollers that rolling towards each other.

The process is repeated until all fillers that were used are well mixed with natural rubber and become homogenous mixture of solid.

Addition of Chemicals

The second stage is continued by addition or incorporation of chemicals with the rubber compound. The chemicals are incorporated by using spatula on the rubber compound surface and are allowed to mix by compress it between two metal rollers in the present of heat around 70 °C. The chemicals that contain zinc oxide, stearic acid and 6-PPD are incorporated properly so that these additives are well mixed with the rubber compounds.

Addition of Vulcanization Agent

The last stage of incorporation of chemicals is addition of vulcanizing agent in the present of accelerator. Both two chemicals are representing by sulphur as vulcanizing agent and N-cyclohexyl-2-benzothiazyl sulphenamide (CBS) as accelerator. The process is started after the rubber compound is allowed to cool down into room temperature for at least two hours. The mixture of sulphur and CBS is added on the rubber surface and was compressed by two roll mill in the present of heat around 70 °C.

2.5 Molding

Preparation of Sample

Rubber compounds that were mix with fillers and chemicals are cut into several parts for at least two parts. One or two of the part is cut into the small pieces like dice and is accumulated to undergo molding process.

Loading of Sample and Shaping

After completing cut all the required part for molding process, the small pieces of sample is placed on the metal plate and was accumulate into the center so that the end product would be without holes. Then another plate is placed on the sample and will close the bottom plate with rubber sample inside it.

Then the plate is placed inside the hot and cold molding press and is allowed to melt about 25 minutes at temperature of 160 °C. After 25 minutes the sample is unloaded from the molding press and is allowed to cool down for a while. The sample then separated from the metal plate and was packaged and cut into dumbbell shape.

2.6 Testing

Tensile Test

Tensile test is conducted for measuring the tensile strength of rubber compound for all part of filler content. The testing is started with set up the outcomes result like the parameter of tensile strength, elongation, and time required meeting the break point.

Swelling Test

Swelling test is conducted to calculate the swelling percentage of rubber compound. All the samples of rubber are cut into particular shape and were immersed in kerosene and diesel for 24 hours. Objective of this test is to determine swelling resistance of rubber compound that was filled with fillers. The calculation for swelling percentage can be obtained by using equation below.

$$\text{Swelling percentage (\%)} = \left[\frac{(M_2 - M_1)}{M_1} \right] \times 100 \quad (2.2)$$

Where M_1 is the initial mass of sample and M_2 is the mass of specimen after immersion.

3.0 RESULTS AND DISCUSSION

3.1 Introduction

This section will review about the results from the tests that were made. The effect of the fillers loading or fillers content on mechanical properties of natural rubber will be analyzed and discussed.

3.2 Tensile Test

Tensile test was conducted for five samples of rubber compound with different of

filler content. Tensile testing unit is used as the platform and result of each sample is discussed as below.

Effect of Filler Content on Tensile Strength

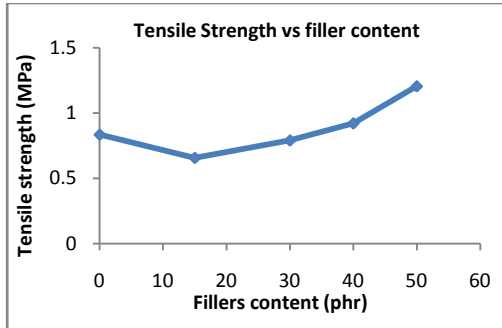


Figure3.1: Graph of tensile strength against fillers content

The graph above shows the relationship between tensile strength with fillers content from 0 phr to 50 phr. The trend shows that the tensile strength decreases from 0 phr to 15 phr but increase back until 50 phr. The increasing amount of fillers content caused the tensile strength increase because of the good interaction between rubber and fillers. The high tensile strength in 0 phr caused by the high elongation that leading to the high tensile strength. Time for 0 phr of fillers content in rubber compound to break require long time because of the high elasticity of the natural rubber that is not filled with filler.

Effect of Filler Content on Elongation

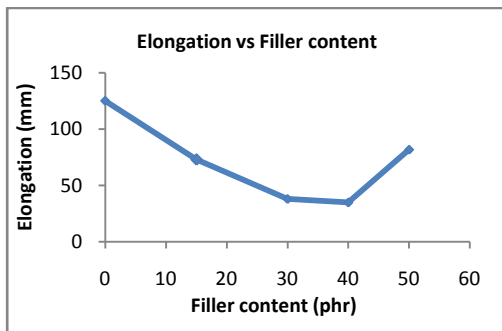


Figure 3.2: Graph of elongation against fillers content

Figure above shows that the elongation of rubber compound is decreased as the increasing of fillers content. The incorporation of fillers has made the rubber compound less elastic

from 0 phr to 40 phr because of the increasing interactions between the filler and rubber. Then it caused the bonding inside the rubber compound is reinforced and improved. The 50 phr of fillers content shows the high elongation due to the poor interaction between fillers and rubber and might be caused by incomplete mixing during the mixing process.

Effect of Filler Content on Time at Break Point

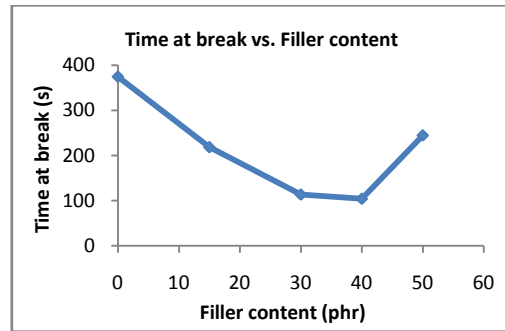


Figure 3.3: Graph of time at break against fillers content

Graph above shows the relationship between the times at break with filler content in rubber compounds. The graph shows the decreasing trends from 0 phr to 40 phr and increase back from 40 phr to 50 phr. The decreasing trends from 0 phr to 40 phr caused by the high elasticity of rubber compounds that leading to the low elongation. Then the time is reduced because of the break point of sample become faster as the increasing amount of fillers.

Effect of Filler Content on Young's Modulus

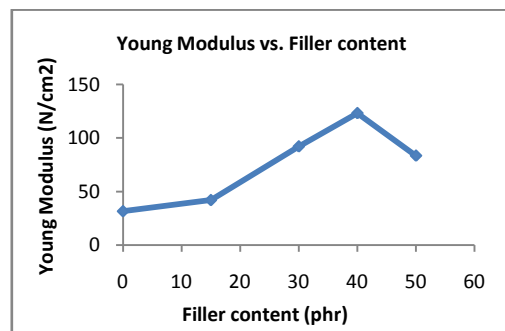


Figure 3.4: Graph of Young's modulus against fillers content

Graph above shows the relationship between Young's modulus and the fillers content

in rubber compound. The graph shows that the Young's modulus is increased as the increasing amount of fillers from 0 phr to 40 phr. This is due to the high tensile strength and low elongation of samples which indicates that the bonding of rubber compounds is reinforced by addition of fillers. The incorporation of fillers with natural rubber caused the bonding inside the rubber compounds is improved and the strength of the bonding is increased as the increasing amount of fillers. The 50 phr shows the decrease from the 40 phr because of the high elongation and high tensile strength that yield low Young's modulus. This might be caused by the incomplete mixing between rubber and fillers during the mixing process and contribute to the low Young's modulus.

Effect of Filler Content on Mixing Time

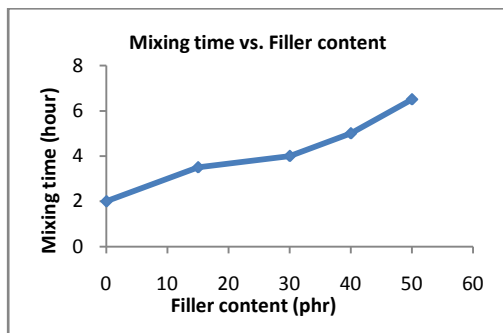


Figure 3.5: Graph of mixing time against fillers content

Graph above shows the relationship between the mixing time and fillers content in rubber compound. It can be described that the mixing time is increased as the increasing amount fillers content in rubber compound. The mixing process require a long time for higher fillers content because the amount of fillers is increased as the increasing amount of fillers but amount of natural rubber is decreased. Hence the mixing is become longer by increasing amount of fillers and the 50 phr is the longest and the hardest because there a highest amount of fillers but lowest amount of natural rubber.

3.3 Behaviour of Graph Stress against Strain

The graph of stress against strain for all five samples is plotted as in figures below and the stress and strain is determined by using the equations 3.1 and 3.2. The calculation for each point is shown in Appendix B and from the

graph stress against strain, the Young modulus is determined by determined the best line of each graph and the gradient is equal to the Young's modulus. Young's modulus is described as the ratio of stress over strain and represent as the modulus of elasticity of the material and can be determined by using equation 3.3 (Callister *et al.*, 2008).

$$\text{Stress} = \frac{\text{Force (N)}}{\text{Area (cm}^2\text{)}} \quad (3.1)$$

$$\text{Strain} = \frac{\text{Change in length, } \Delta l \text{ (cm)}}{\text{Initial length } l_0 \text{ (cm)}} \quad (3.2)$$

$$\text{Young's Modulus} = \frac{\text{Stress}}{\text{Strain}} \quad (3.3)$$

Sample of 0 phr of filler content

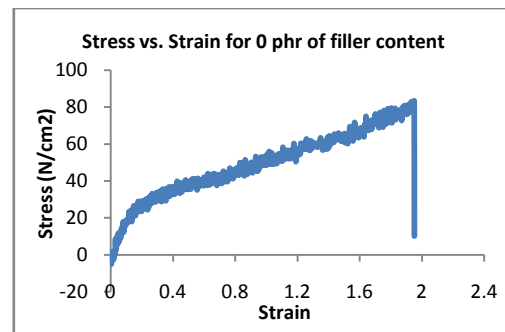


Figure 3.6: Graph of stress against strain for 0 phr of fillers content

The graph above shows the relationship between stress with strain for 0 phr of fillers content. From the graph, the sample experience high strain before it reached the break point and lead to the high stress because more forces is needed to breakdown the samples. The gradient of this graph is equal to the Young's modulus that is 31.555 N/cm². This value is the lowest among the five samples and caused by the high strain or long elongation that was experienced by rubber compound. High strain also requires long time to meet the break point and contribute to the high stress or high forces are needed in order to reach the break point.

Sample of 15 phr of filler content

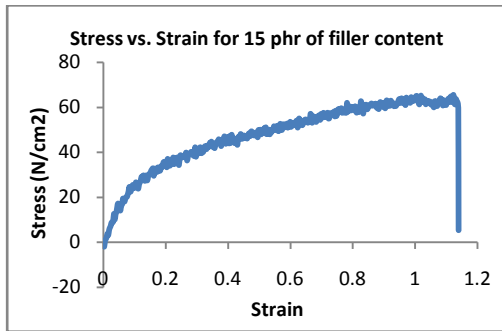


Figure 3.7: Graph of stress against strain for 15 phr of fillers content

The graph above shows the relationship between stress and strain for 15 phr of fillers content. The strain of the sample is decreased almost to the 1.10 compared to the sample with 0 phr that is exceeding 1.5. The gradient or Young's modulus of this graph is 41.977 N/cm² which is higher than 0 phr and caused by the low strain and the ratio of stress over strain is increased. It means that sample of 15 phr need more force to reach the break point and the time to reach the break point become faster as compared to the 0 phr. Lower strain means that the elongation of the samples is decreased because of the high elasticity due to the good interaction between rubber and fillers. The mixing time also increased because of the addition amount of fillers and reduction amount of natural rubber.

Sample of 30 phr of filler content

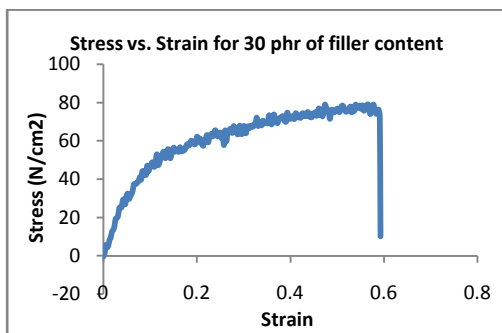


Figure 3.8: Graph of stress against strain for 30 phr of fillers content

Graph above shows the relationship between the stress and strain for 30 phr of fillers content in rubber compound. The stress that was

experienced by the sample is around 80 N/cm² before it reached the break point and break down. The strain is reduced into around 0.6 and is lower compared to the 0 and 15 phr. This might be caused to the good interaction between rubber and fillers that leading to the high elasticity and more force is needed to reach the break point. The Young's modulus or gradient of this graph is 92.17 N/cm² and in the middle range among the five samples. Mixing time also in the middle range due to the 30 phr is in the middle range of fillers content. The time to reach the break point is faster than the 15 phr because of the lower strain experienced by the sample.

Sample of 40 phr of filler content

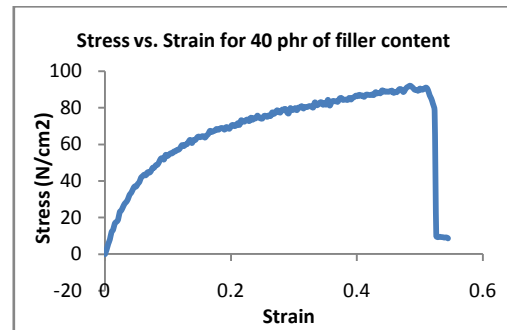


Figure 3.9: Graph of stress against strain for 40 phr of fillers content

Graph above shows the relationship between the stress and strain for 40 phr of fillers content in rubber compound. The graph shows that the sample experiences the stress around 90 N/cm² before it reach the break point. The sample also experiences the break point after being stretched about half of its initial length or it's reached the strain about 0.5 before break down. It shows that the sample has high tensile strength and need high force to reach the break point. But in order to reach the break point, high force is needed and caused the sample break down faster than other first three samples. The low strain means that the sample has the high elasticity and the Young's modulus or gradient of the graph is 123.3 N/cm² which is the highest among the five samples. This is due to the low elongation and high tensile strength and high force is needed to reach the break point.

Sample of 50 phr of filler content

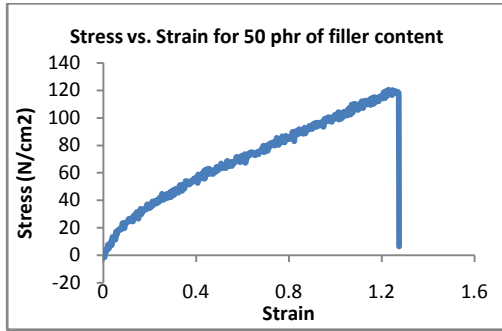


Figure 3.10: Graph of stress against strain for 50 phr of fillers content

The graph above shows the relationship between the stress and strain for 50 phr of filler content in rubber compound. The graph shows that the sample experiences high stress before it reach the break point but at the break point, it stretched about 1.2 of its initial length or strain before it reached the break point is around 1.2. It means that the Young’s modulus is low compared to the 40 and 30 phr that is 83.531 N/cm². The sample also is being stretched longer than 15, 30 and 40 phr because of the high elasticity of the rubber compound that leading to the high tensile strength in order to reach the break point. The low Young’s modulus is caused by the low ratio of stress over strain due to the high elongation. This might be caused by the incomplete mixing between the rubber and fillers during the mixing process and has made the poor bonding inside the rubber compound.

3.4 Swelling Test

Swelling test is conducted to determine the swelling resistance of rubber compound. Generally, among the solvent that is used are benzene and toluene but for this study diesel and kerosene are used and the testing was conducted for 24 hours. The initial weight of all samples is weighed and recorded and the samples were immersed and are allowed to swell for 24 hours. Then the final weight of rubber samples is determined and the percentage of swelling is calculated using equation 2.2. Details calculation of swelling percentage of each sample for both solvent is shown at Appendix C.

Effect of Filler Content on Swelling in Kerosene

For swelling test using kerosene as the solvent, the initial and final weight of rubber compound is shown as in table 3.1 below.

Table 3.1: Calculation of swelling percentage in kerosene

Part	Initial weight, M ₁ (g)	Final weight, M ₂ (g)	Change in weight, ΔM (g)	ΔM/M ₁	Swelling percentage (%)
0	0.30	2.37	2.07	6.90	690.00
15	0.78	3.24	2.46	3.15	315.38
30	0.70	3.90	3.20	4.57	457.14
40	0.45	1.41	0.96	2.13	213.33
50	0.59	3.47	2.88	4.88	488.14

The swelling percentage of rubber compound in kerosene for 0 phr to 50 phr of filler content is plotted as in figure 3.11.

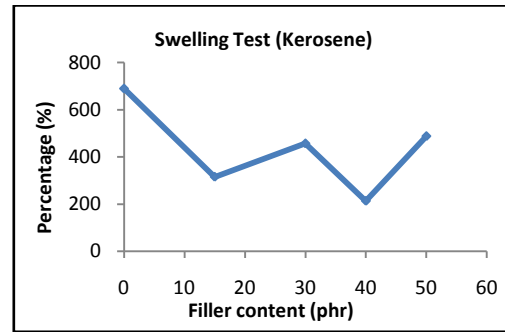


Figure 3.11: Swelling percentage of rubber compound in kerosene

The swelling percentage is decreased as the increasing amount of fillers during 0 phr to 15 phr and 30 phr to 40 phr but increase back during 15 phr to 30 phr and 40 phr to 50 phr. The swelling percentage or swelling resistance basically should decrease as the increasing amount of fillers because of decreasing amount of natural rubber and contribute better swelling resistance (H. Ismail *et al.*, 2002). For swelling resistance between 0 phr 15 phr shows the normal behaviour of theoretical of swelling resistance but it is found that 30 phr and 50 phr exhibits abnormal swelling resistance. It might be caused by incomplete mixing part between rubber and filler and caused the rubber component expose to the kerosene and experienced interaction between both substances.

Effect of Filler Content on Swelling in Diesel

For swelling test using diesel as the solvent, the initial and final weight of rubber compound is shown as in table 3.2 below.

Table 3.2: Calculation of swelling percentage in diesel

Part	Initial weight, M_1 (g)	Final weight, M_2 (g)	Change in weight, ΔM (g)	$\Delta M/M_1$	Swelling percentage (%)
0	0.4	1.52	1.12	2.80	280.00
15	0.85	2.05	1.2	1.41	141.18
30	0.65	2.07	1.42	2.18	218.46
40	0.46	1.02	0.56	1.22	121.74
50	0.71	1.86	1.15	1.62	161.97

The swelling percentage of rubber compound in diesel for 0 phr to 50 phr of filler content is plotted as in figure 3.12.

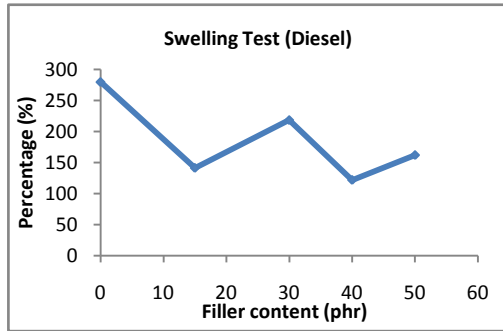


Figure 3.12: Swelling percentage of rubber compound in diesel

Graph above shows the relationship between swelling percentages with filler content that varied from 0 phr to 50 phr. The graph indicated that the swelling percentage or swelling resistance is decreased from 0 phr to 15 phr and 30 phr to 40 phr but increased back from 15 phr to 30 phr and 40 phr to 50 phr. Its shows that the graph is increased and decreased alternately due to the poor swelling resistance in rubber compound that filled with 30 phr and 50 phr. This might be caused by incomplete mixing between the fillers particle and rubber component that contributed to the poor swelling resistance. For the rubber compounds that filled with 0, 15 and 40 phr shows the decreasing trends that caused by increasing amount of fillers. The increasing amount of fillers in rubber compounds has made it become the bonding agent that binds the filler fibers with natural rubber so that the swelling is highly stopped and

functioned as barrier in the compound (Jacob *et al.*, 2004).

Comparison of Swelling Test between Both Solvent

Both graph of swelling in kerosene and diesel is plotted together in one graph and the comparison between both graphs is shown in figure 3.13 below.

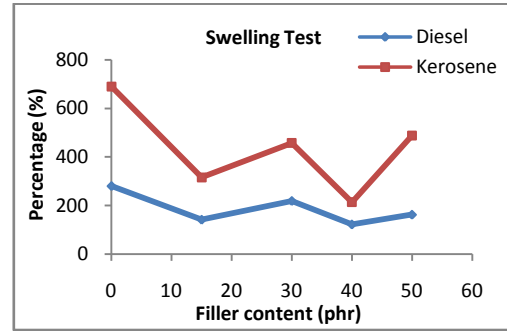


Figure 3.13: Comparison of swelling percentage between two solvent

From the graph, the swelling resistance in kerosene is higher than swelling resistance in diesel for all five samples. This is due to the smaller chemical structure of kerosene compared to the diesel which is kerosene is compilation of hydrocarbon chains that are made up of between 12 to 15 carbon atoms and diesel has a rigid molecular structure consisting of 16 carbon atoms and 34 hydrogen atoms. Besides that, kerosene has less density than diesel and both factors contribute to the high swelling percentage of kerosene.

4.0 CONCLUSION

From the results that were obtained, the increasing amount of fillers content used caused the tensile strength and Young's modulus increased until optimum 40 phr.

The increasing part of fillers content in rubber compound made the compound become more elastic and caused the elongation of samples reduce as the increasing part of fillers content. Mixing time also is increased as the increasing amount of fillers because the incorporation of fillers with natural rubber become harder because amount of fillers is increased but amount of natural rubber is reduced.

Swelling test also shows the increasing amount of fillers has made the swelling percentage or swelling resistance is reduced

because of the good barrier that was made by fillers. The increasing amount of fillers content made the better barrier in order to stop the penetration of solvent towards natural rubber. The swelling percentage also shows the higher value when is used in less dense solvent.

ACKNOWLEDGEMENT

Special thanks to Universiti Malaysia Pahang (UMP) to provide laboratories facilitator and to all technical staffs. Sincere appreciation is extended to Mr. Mohd Bijarimi Bin Mat Piah for constant guidance and supervision for this research.

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The Effect of Oil Palm Trunk Fiber on Characterization of Natural Rubber

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Abstract

The strength of a material to stress depends on the chemical bonds found in the chemical structure of the material itself. This thesis deals with the effect of different filler loading on the natural rubber vulcanizates so that the dynamic mechanical properties of vulcanized natural rubber can be obtained. The objective of this thesis is to study the effect of different filler loading or amount of filler loaded on natural rubber that is Standard Malaysian Rubber 10 (SMR-10). For this study, natural filler that is oil palm trunk fiber which varies from 0 to 50 phr have been used as an additive to strengthen the chemical bonds of natural rubber as well as other chemicals that were added to natural rubber. Rubber compounds filled with natural fillers and other chemical additives is prepared using a two roll mill and the characteristics of the mechanical properties are studied and analyzed to determine the effect on the strength of natural rubber when natural filler is added as filler. The functions for each material that is stated above might vary based on their physical and chemical properties data but all fillers generally increase the tensile strength and modulus of vulcanized natural rubber into particular level.

Abstrak

Kekuatan sesuatu bahan terhadap tekanan bergantung kepada ikatan kimia yang terdapat didalam struktur kimia bahan itu sendiri. Tesis ini berkaitan dengan kesan perbezaan jumlah kemasukan bahan pengisi semula jadi pada getah asli yang divulkanasikan supaya sifat-sifat kekuatan dan sifat mekanik dinamik dari getah alam yang divulkanasikan boleh diperolehi. Tujuan tesis ini adalah untuk mempelajari kesan perbezaan jumlah kemasukan bahan pengisi semula jadi ke atas getah asli iaitu Standard Malaysian Rubber 10 (SMR-10). Untuk kajian ini, bahan penambah asli iaitu fiber batang kelapa sawit telah digunakan sebagai bahan penambah untuk menguatkan ikatan kimia pada getah asli serta bahan kimia lain yang turut ditambah bersama ke atas getah asli. Sebatian getah yang dicampur dengan bahan pengisi asli serta bahan penambah lain disediakan menggunakan 'two roll mill' dan ciri-ciri terhadap sifat-sifat mekanikal dikaji serta dianalisa untuk melihat kesan terhadap kekuatan getah asli apabila bahan pengisi semula jadi ditambah sebagai bahan pengisi. Fungsi untuk setiap material yang dinyatakan di atas mungkin berbeza-beza dari segi sifat-sifat fizik dan kimianya tetapi semua bahan pengisi ini secara umumnya berfungsi meningkatkan modulus dan kekuatan tensile getah asli yang divulkanasikan ke suatu tahap tertentu.

Keywords: oil palm trunk fiber, natural rubber, natural filler, mechanical properties.

1.0 INTRODUCTION

Natural rubber is a chemical compound that produced from latex. Usually latex is collected from the tapped rubber tree and also can be produced by synthetically. Both the natural and synthetic rubber is being used widely in many applications and products. But there is a different between natural rubber and synthetic rubber especially their strength or its elasticity. Natural rubber is known to exhibit numerous outstanding properties such as good oil resistance, and low gas permeability (Kaushik et al., 2010). It is also more elastic than the synthetic rubber and because of its special properties made it preferred raw material for manufacturers that producing rubber products.

Rubber tree in Malaysia is brought from Brazil by Sir Henry Wickham in 1876. He gathered thousand of rubber tree seeds and these seeds were shipped to Sri Lanka, Indonesia,

Singapore, and Malaysia. Then Malaya (Malaysia) was later to become the biggest producer of rubber at that time. New plantations areas also have been started in Thailand, Philippines, Africa, and Europe to support insufficient raw material in Malaysia and Indonesia. Today, Malaysia, Indonesia and Thailand are among the biggest rubber producers and produces almost 80% of world's natural rubber.

Filler is a compound that is used to enhance the quality of rubber products. Generally, filler has no fixed classification but it can be categorized into two types according their colour that are black filler and white filler. In order to improve the strength of natural rubber, filler is added while processing the natural rubber and the mechanism took part in this process is fillers could modify the properties of natural rubber (Chuayjuljit *et al.*, 2002). Most

popular reinforcing filler is carbon black which is widely used in tyre manufacturing industry.

Carbon black is material that produced from incomplete combustion of gaseous or liquid hydrocarbon in the form of colloidal particles. Usually, its physical appearance is in form of black powder and widely used in rubber and plastic industry. It is also available in various sizes and specifications. In tyre manufacturing industry, carbon black is used as reinforcing agent which is it enhance the strength and improve the properties of natural rubber.

Besides being as reinforcing agent, carbon black also can conduct heat away from tyre and make it become long-lasting. Smaller size of the carbon black used in rubber processing will cause bigger surface area of the filler. This will contribute great and high degree of reinforcement towards natural rubber. (Peng, 2007).

Beside the widely used carbon black fillers, there is another type of filler that is non-black filler. Non-black filler or white filler is can be differentiate by its colour and label. Most popular white fillers that are used in rubber processing are calcium carbonate, precipitated silica and clay. Like carbon black, white fillers also can give different effect based on their particle size. The smaller size of the white filler and then more big surface area of the white filler which is the bigger surface area give greatest degree of reinforcement towards natural rubber.

Non-black fillers like natural fillers such as rice husk, empty fruit bunch and oil palm trunk fiber together with clay and asbestos is being used as replacement for black filler to increase the production as well diversify of rubber product. The addition of these reinforcing agents also functioning as additives that will be enhance the strength, increase hardness and reduce the cost of compound (Peng, 2007). Another example of non-black filler are organoclay and alumina nanoparticles.

Natural fillers generally can be described as reinforcing agent that was produced from natural resources like rice husk, empty fruit bunch and oil palm trunk fiber. Basically, the function of these natural fillers might be same as chemical fillers that are to enhance the mechanical properties of rubber compound so that it reaches certain level of improvement. There is a question about how natural fillers that was produced from the plantation waste can be used as fillers in rubber processing. In recent years, a lot of study has been done to investigate the effectiveness of natural fillers on

characterization of natural rubber. The contribution towards enhancing or improving the mechanical properties might be become significant and the main target, hence natural fillers is used in research. The advantages of using the natural filler instead of chemical fillers like carbon black is it is not harmful to human health compared to the carbon black for example. The most significant part is the cost might be lower than chemical filler but can give quite same level of reinforcement of rubber compounds.

2.0 METHODOLOGY

2.1 Equipment

Two Roll Mill

Two roll mill in FKKSAs laboratory was used in rubber processing for research of effect of oil palm trunk fiber on characterization of natural rubber. This equipment consists of two metal rollers and a heater so that the metal roller can be heat up to 100 °C.

Hot & Cold Molding Press

Hot and cold molding press in FKKSAs laboratory is used to prepare the rubber compound in the form of homogenous sheet so that the sheet can be cut into dumbbell shape for testing procedures.

Tensile Testing Unit

Tensile testing was conducted by using the tensile testing unit at FKP's laboratory at UMP Kampus Gambang. This machine can be loaded until maximum of 50 kN and can be used for various types of material such as metal, nanocomposite, polymer and others materials.

2.2 Preparation of Raw Material

The natural rubber that is Standard Malaysian Rubber 10 (SMR-10) and oil palm trunk fiber are obtained from the supplier in a bulk package. In order to prepare the raw material that need small amount that is 250 grams per sample, it is necessary to cut the natural rubber into small amount or in the form of slice with the thickness of around 1 centimeter. The oil palm trunk fiber also needs to be sieve and the ashes are collected while the fiber waste is eliminated. Table 1 below is the shows the rubber formulation for this research.

Table 1: Rubber formulation

Ingredient	Formulation (phr)				
	1	2	3	4	5
SMR-10	100	100	100	100	100
Stearic Acid	2	2	2	2	2
Zinc Oxide	5	5	5	5	5
6-PPD ^a	1	1	1	1	1
CBS ^b	0.5	0.5	0.5	0.5	0.5
Sulphur	2.5	2.5	2.5	2.5	2.5
OPTF ^c	0	15	30	40	50
Total	111	126	141	151	161

^aN-(1,3-dimethylbutyl)-N'-phenyl-p-phenylene-diamine)

^bN-cyclohexyl-2-benzothiazyl sulphenamide

^cOil Palm Trunk Fiber

Natural Rubber

Natural rubber is obtained in bulk package and to prepare into small amount, it is necessary to cut the rubber into slice using a small saw. The compound it is in form of high viscosity and has high elasticity hence it is hard to cut the rubber into smaller size. Then the sample of rubber is weighed according to the rubber formulation and was keep as well labeled. After that, the rubber slice will undergo mastication to prepare rubber compound that well homogeneous and low viscosity.

Filler

Oil palm trunk fiber is in the form of coagulation of waste palm oil fiber. In order to obtain the powder from the fiber, it is necessary to sieve the filler using siever and was sieved manually using size of 400 µm. the filler has a light weight and for every session of sieving, only produce around 20% from the coagulate fiber. Hence the sieving process was taking a long time in order to yield around 226.82 grams of fillers for all samples.

The sieving process started by taking some amount of coagulates oil palm trunk fiber into siever and was shacked so that the powder will fall down with the uniform size. The process is repeated for large amount of filler in order to obtain total amount of filler for each sample.

Chemicals

Chemicals are used as additives for the rubber processing and five chemicals were used and can be separate into two groups. First is the activator and antidegradant group and second is vulcanizing group that represent stage 1 and stage 2.

Table 2: Summary of chemical in rubber compounding

Stage 1	Stage 2
Zinc oxide	Sulphur
Stearic acid	CBS
6-PPD	

All chemicals are weighed according to the rubber formulation and were packed according to the stages. Zinc oxide, stearic acid and 6-PPD are packed together and were shacked so that it well mixed in a mixture. The rubber processing is conducted by addition of chemicals by stages. Each of the stage is added to the rubber compound and for each interval of the stages will take some period in order to ensure the rubber compound is well mixed with the chemicals.

Weight Calculation of Each Ingredient

The rubber formulation in table 3.1 shows the part per hundred rubber (phr) for each ingredient. In order to proceed to the rubber processing, it is necessary to calculate the weight of each compound based on the phr as listed in the rubber formulation. The equation that can be used to calculate the weight is shown below.

$$\text{Weight of ingredient (g)} = \frac{P_i}{P_t} \times 250 \text{ g} \quad (2.1)$$

Where P_i is equal to number part of ingredients (phr) and P_t is equal to total parts of a compound (phr).

2.3 Mastication

Mastication is a process that was conducted to prepare the rubber compounds that can readily accept the addition of chemicals or additives for rubber processing. The objective of this process is to break the polymer chains so that the viscosity of rubber compounds is reduce as well as the elasticity of the rubber. The slice of rubber that was cut from the bulk rubber with the weight according to the rubber formulation is allowed to go through the two roll mill. The rubber slice then is compressed by two roll mill with particular gap between both rollers at temperature of 70 °C and speed of 50 rpm.

2.4 Compounding

Compounding or mixing is conducted to incorporate all the chemicals and fillers with natural rubber until become homogenous mixture. All chemicals in rubber formulation are

incorporated by stages and can be classified into three stages. First stage is addition of fillers with natural rubber, second stage is addition of chemicals to natural rubber that was added with fillers and last stage is addition of vulcanization agent with present of accelerator. The details of each stage are described below.

Addition of Fillers

The first stage is started with addition of filler that is oil palm trunk fiber into natural rubber by using two roll mill and the rubber compound is compressed by two metal rollers. Fillers that is in form of powder is added properly to the rubber compound in the present of heat around 70 °C from the metal rollers that rolling towards each other.

The process is repeated until all fillers that were used are well mixed with natural rubber and become homogenous mixture of solid.

Addition of Chemicals

The second stage is continued by addition or incorporation of chemicals with the rubber compound. The chemicals are incorporated by using spatula on the rubber compound surface and are allowed to mix by compress it between two metal rollers in the present of heat around 70 °C. The chemicals that contain zinc oxide, stearic acid and 6-PPD are incorporated properly so that these additives are well mixed with the rubber compounds.

Addition of Vulcanization Agent

The last stage of incorporation of chemicals is addition of vulcanizing agent in the present of accelerator. Both two chemicals are representing by sulphur as vulcanizing agent and N-cyclohexyl-2-benzothiazyl sulphenamide (CBS) as accelerator. The process is started after the rubber compound is allowed to cool down into room temperature for at least two hours. The mixture of sulphur and CBS is added on the rubber surface and was compressed by two roll mill in the present of heat around 70 °C.

2.5 Molding

Preparation of Sample

Rubber compounds that were mix with fillers and chemicals are cut into several parts for at least two parts. One or two of the part is cut into the small pieces like dice and is accumulated to undergo molding process.

Loading of Sample and Shaping

After completing cut all the required part for molding process, the small pieces of sample is placed on the metal plate and was accumulate into the center so that the end product would be without holes. Then another plate is placed on the sample and will close the bottom plate with rubber sample inside it.

Then the plate is placed inside the hot and cold molding press and is allowed to melt about 25 minutes at temperature of 160 °C. After 25 minutes the sample is unloaded from the molding press and is allowed to cool down for a while. The sample then separated from the metal plate and was packaged and cut into dumbbell shape.

2.6 Testing

Tensile Test

Tensile test is conducted for measuring the tensile strength of rubber compound for all part of filler content. The testing is started with set up the outcomes result like the parameter of tensile strength, elongation, and time required meeting the break point.

Swelling Test

Swelling test is conducted to calculate the swelling percentage of rubber compound. All the samples of rubber are cut into particular shape and were immersed in kerosene and diesel for 24 hours. Objective of this test is to determine swelling resistance of rubber compound that was filled with fillers. The calculation for swelling percentage can be obtained by using equation below.

$$\text{Swelling percentage (\%)} = \left[\frac{(M_2 - M_1)}{M_1} \right] \times 100 \quad (2.2)$$

Where M_1 is the initial mass of sample and M_2 is the mass of specimen after immersion.

3.0 RESULTS AND DISCUSSION

3.1 Introduction

This section will review about the results from the tests that were made. The effect of the fillers loading or fillers content on mechanical properties of natural rubber will be analyzed and discussed.

3.2 Tensile Test

Tensile test was conducted for five samples of rubber compound with different of

filler content. Tensile testing unit is used as the platform and result of each sample is discussed as below.

Effect of Filler Content on Tensile Strength

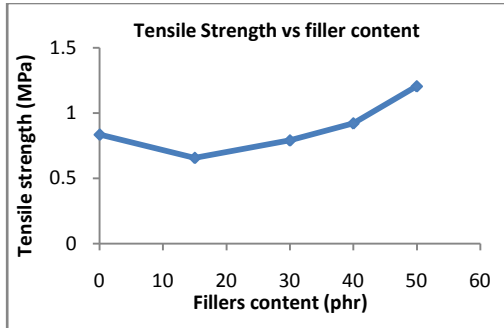


Figure3.1: Graph of tensile strength against fillers content

The graph above shows the relationship between tensile strength with fillers content from 0 phr to 50 phr. The trend shows that the tensile strength decreases from 0 phr to 15 phr but increase back until 50 phr. The increasing amount of fillers content caused the tensile strength increase because of the good interaction between rubber and fillers. The high tensile strength in 0 phr caused by the high elongation that leading to the high tensile strength. Time for 0 phr of fillers content in rubber compound to break require long time because of the high elasticity of the natural rubber that is not filled with filler.

Effect of Filler Content on Elongation

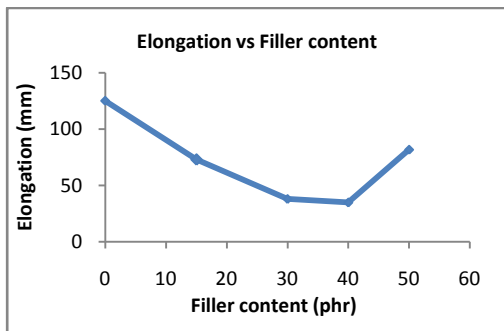


Figure 3.2: Graph of elongation against fillers content

Figure above shows that the elongation of rubber compound is decreased as the increasing of fillers content. The incorporation of fillers has made the rubber compound less elastic

from 0 phr to 40 phr because of the increasing interactions between the filler and rubber. Then it caused the bonding inside the rubber compound is reinforced and improved. The 50 phr of fillers content shows the high elongation due to the poor interaction between fillers and rubber and might be caused by incomplete mixing during the mixing process.

Effect of Filler Content on Time at Break Point

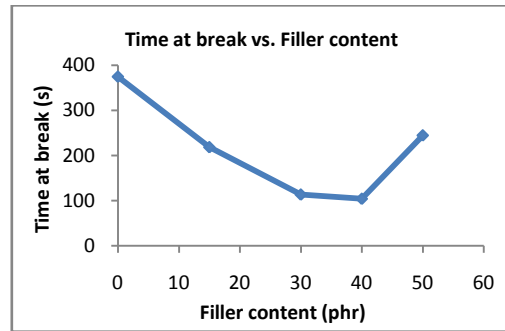


Figure 3.3: Graph of time at break against fillers content

Graph above shows the relationship between the times at break with filler content in rubber compounds. The graph shows the decreasing trends from 0 phr to 40 phr and increase back from 40 phr to 50 phr. The decreasing trends from 0 phr to 40 phr caused by the high elasticity of rubber compounds that leading to the low elongation. Then the time is reduced because of the break point of sample become faster as the increasing amount of fillers.

Effect of Filler Content on Young's Modulus

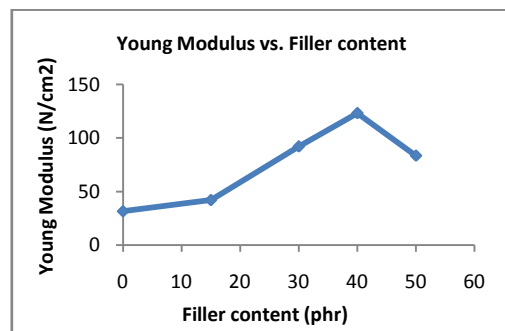


Figure 3.4: Graph of Young's modulus against fillers content

Graph above shows the relationship between Young's modulus and the fillers content

in rubber compound. The graph shows that the Young's modulus is increased as the increasing amount of fillers from 0 phr to 40 phr. This is due to the high tensile strength and low elongation of samples which indicates that the bonding of rubber compounds is reinforced by addition of fillers. The incorporation of fillers with natural rubber caused the bonding inside the rubber compounds is improved and the strength of the bonding is increased as the increasing amount of fillers. The 50 phr shows the decrease from the 40 phr because of the high elongation and high tensile strength that yield low Young's modulus. This might be caused by the incomplete mixing between rubber and fillers during the mixing process and contribute to the low Young's modulus.

Effect of Filler Content on Mixing Time

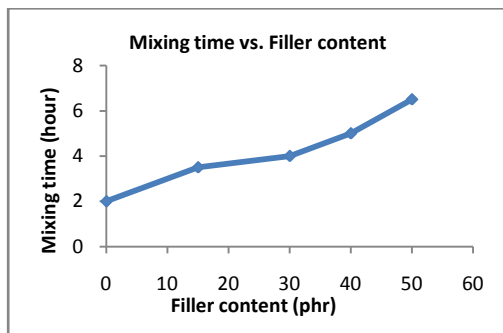


Figure 3.5: Graph of mixing time against fillers content

Graph above shows the relationship between the mixing time and fillers content in rubber compound. It can be described that the mixing time is increased as the increasing amount fillers content in rubber compound. The mixing process require a long time for higher fillers content because the amount of fillers is increased as the increasing amount of fillers but amount of natural rubber is decreased. Hence the mixing is become longer by increasing amount of fillers and the 50 phr is the longest and the hardest because there a highest amount of fillers but lowest amount of natural rubber.

3.3 Behaviour of Graph Stress against Strain

The graph of stress against strain for all five samples is plotted as in figures below and the stress and strain is determined by using the equations 3.1 and 3.2. The calculation for each point is shown in Appendix B and from the

graph stress against strain, the Young modulus is determined by determined the best line of each graph and the gradient is equal to the Young's modulus. Young's modulus is described as the ratio of stress over strain and represent as the modulus of elasticity of the material and can be determined by using equation 3.3 (Callister *et al.*, 2008).

$$\text{Stress} = \frac{\text{Force (N)}}{\text{Area (cm}^2\text{)}} \quad (3.1)$$

$$\text{Strain} = \frac{\text{Change in length, } \Delta l \text{ (cm)}}{\text{Initial length } l_0 \text{ (cm)}} \quad (3.2)$$

$$\text{Young's Modulus} = \frac{\text{Stress}}{\text{Strain}} \quad (3.3)$$

Sample of 0 phr of filler content

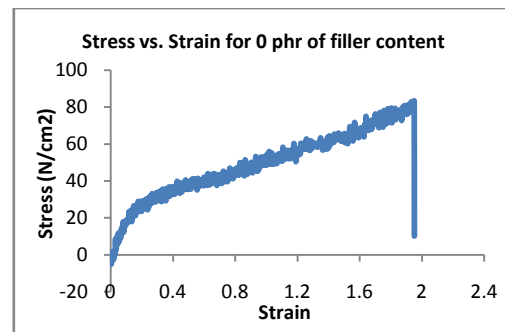


Figure 3.6: Graph of stress against strain for 0 phr of fillers content

The graph above shows the relationship between stress with strain for 0 phr of fillers content. From the graph, the sample experience high strain before it reached the break point and lead to the high stress because more forces is needed to breakdown the samples. The gradient of this graph is equal to the Young's modulus that is 31.555 N/cm². This value is the lowest among the five samples and caused by the high strain or long elongation that was experienced by rubber compound. High strain also requires long time to meet the break point and contribute to the high stress or high forces are needed in order to reach the break point.

Sample of 15 phr of filler content

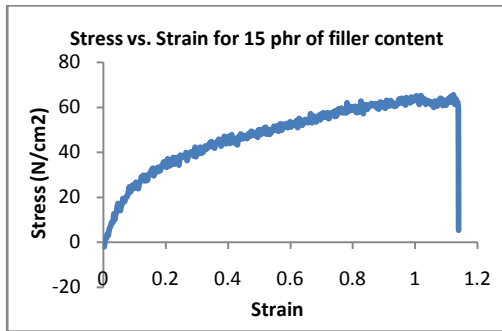


Figure 3.7: Graph of stress against strain for 15 phr of fillers content

The graph above shows the relationship between stress and strain for 15 phr of fillers content. The strain of the sample is decreased almost to the 1.10 compared to the sample with 0 phr that is exceeding 1.5. The gradient or Young's modulus of this graph is 41.977 N/cm² which is higher than 0 phr and caused by the low strain and the ratio of stress over strain is increased. It means that sample of 15 phr need more force to reach the break point and the time to reach the break point become faster as compared to the 0 phr. Lower strain means that the elongation of the samples is decreased because of the high elasticity due to the good interaction between rubber and fillers. The mixing time also increased because of the addition amount of fillers and reduction amount of natural rubber.

Sample of 30 phr of filler content

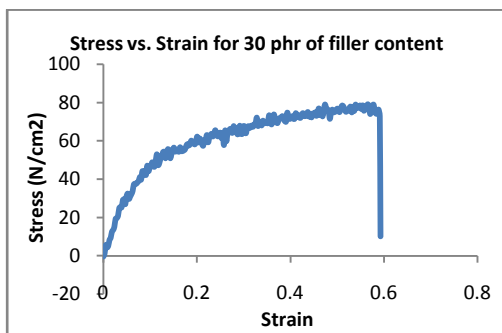


Figure 3.8: Graph of stress against strain for 30 phr of fillers content

Graph above shows the relationship between the stress and strain for 30 phr of fillers content in rubber compound. The stress that was

experienced by the sample is around 80 N/cm² before it reached the break point and break down. The strain is reduced into around 0.6 and is lower compared to the 0 and 15 phr. This might be caused to the good interaction between rubber and fillers that leading to the high elasticity and more force is needed to reach the break point. The Young's modulus or gradient of this graph is 92.17 N/cm² and in the middle range among the five samples. Mixing time also in the middle range due to the 30 phr is in the middle range of fillers content. The time to reach the break point is faster than the 15 phr because of the lower strain experienced by the sample.

Sample of 40 phr of filler content

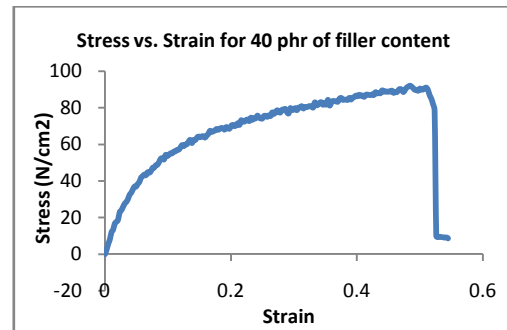


Figure 3.9: Graph of stress against strain for 40 phr of fillers content

Graph above shows the relationship between the stress and strain for 40 phr of fillers content in rubber compound. The graph shows that the sample experiences the stress around 90 N/cm² before it reach the break point. The sample also experiences the break point after being stretched about half of its initial length or it's reached the strain about 0.5 before break down. It shows that the sample has high tensile strength and need high force to reach the break point. But in order to reach the break point, high force is needed and caused the sample break down faster than other first three samples. The low strain means that the sample has the high elasticity and the Young's modulus or gradient of the graph is 123.3 N/cm² which is the highest among the five samples. This is due to the low elongation and high tensile strength and high force is needed to reach the break point.

Sample of 50 phr of filler content

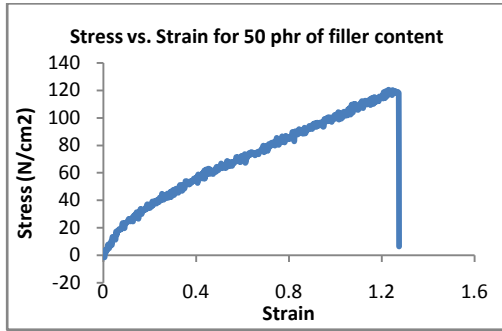


Figure 3.10: Graph of stress against strain for 50 phr of fillers content

The graph above shows the relationship between the stress and strain for 50 phr of filler content in rubber compound. The graph shows that the sample experiences high stress before it reach the break point but at the break point, it stretched about 1.2 of its initial length or strain before it reached the break point is around 1.2. It means that the Young’s modulus is low compared to the 40 and 30 phr that is 83.531 N/cm². The sample also is being stretched longer than 15, 30 and 40 phr because of the high elasticity of the rubber compound that leading to the high tensile strength in order to reach the break point. The low Young’s modulus is caused by the low ratio of stress over strain due to the high elongation. This might be caused by the incomplete mixing between the rubber and fillers during the mixing process and has made the poor bonding inside the rubber compound.

3.4 Swelling Test

Swelling test is conducted to determine the swelling resistance of rubber compound. Generally, among the solvent that is used are benzene and toluene but for this study diesel and kerosene are used and the testing was conducted for 24 hours. The initial weight of all samples is weighed and recorded and the samples were immersed and are allowed to swell for 24 hours. Then the final weight of rubber samples is determined and the percentage of swelling is calculated using equation 2.2. Details calculation of swelling percentage of each sample for both solvent is shown at Appendix C.

Effect of Filler Content on Swelling in Kerosene

For swelling test using kerosene as the solvent, the initial and final weight of rubber compound is shown as in table 3.1 below.

Table 3.1: Calculation of swelling percentage in kerosene

Part	Initial weight, M ₁ (g)	Final weight, M ₂ (g)	Change in weight, ΔM (g)	ΔM/M ₁	Swelling percentage (%)
0	0.30	2.37	2.07	6.90	690.00
15	0.78	3.24	2.46	3.15	315.38
30	0.70	3.90	3.20	4.57	457.14
40	0.45	1.41	0.96	2.13	213.33
50	0.59	3.47	2.88	4.88	488.14

The swelling percentage of rubber compound in kerosene for 0 phr to 50 phr of filler content is plotted as in figure 3.11.

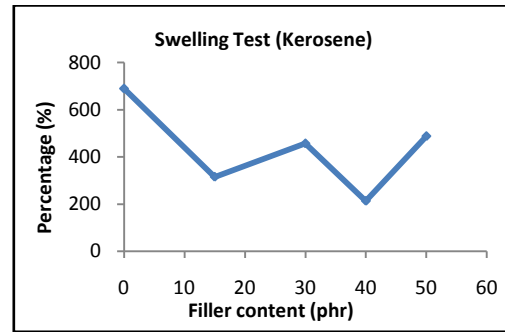


Figure 3.11: Swelling percentage of rubber compound in kerosene

The swelling percentage is decreased as the increasing amount of fillers during 0 phr to 15 phr and 30 phr to 40 phr but increase back during 15 phr to 30 phr and 40 phr to 50 phr. The swelling percentage or swelling resistance basically should decrease as the increasing amount of fillers because of decreasing amount of natural rubber and contribute better swelling resistance (H. Ismail *et al.*, 2002). For swelling resistance between 0 phr 15 phr shows the normal behaviour of theoretical of swelling resistance but it is found that 30 phr and 50 phr exhibits abnormal swelling resistance. It might be caused by incomplete mixing part between rubber and filler and caused the rubber component expose to the kerosene and experienced interaction between both substances.

Effect of Filler Content on Swelling in Diesel

For swelling test using diesel as the solvent, the initial and final weight of rubber compound is shown as in table 3.2 below.

Table 3.2: Calculation of swelling percentage in diesel

Part	Initial weight, M_1 (g)	Final weight, M_2 (g)	Change in weight, ΔM (g)	$\Delta M/M_1$	Swelling percentage (%)
0	0.4	1.52	1.12	2.80	280.00
15	0.85	2.05	1.2	1.41	141.18
30	0.65	2.07	1.42	2.18	218.46
40	0.46	1.02	0.56	1.22	121.74
50	0.71	1.86	1.15	1.62	161.97

The swelling percentage of rubber compound in diesel for 0 phr to 50 phr of filler content is plotted as in figure 3.12.

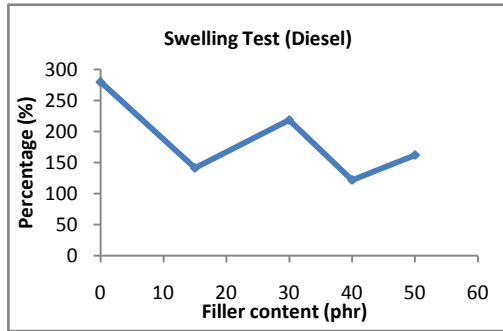


Figure 3.12: Swelling percentage of rubber compound in diesel

Graph above shows the relationship between swelling percentages with filler content that varied from 0 phr to 50 phr. The graph indicated that the swelling percentage or swelling resistance is decreased from 0 phr to 15 phr and 30 phr to 40 phr but increased back from 15 phr to 30 phr and 40 phr to 50 phr. Its shows that the graph is increased and decreased alternately due to the poor swelling resistance in rubber compound that filled with 30 phr and 50 phr. This might be caused by incomplete mixing between the fillers particle and rubber component that contributed to the poor swelling resistance. For the rubber compounds that filled with 0, 15 and 40 phr shows the decreasing trends that caused by increasing amount of fillers. The increasing amount of fillers in rubber compounds has made it become the bonding agent that binds the filler fibers with natural rubber so that the swelling is highly stopped and

functioned as barrier in the compound (Jacob *et al.*, 2004).

Comparison of Swelling Test between Both Solvent

Both graph of swelling in kerosene and diesel is plotted together in one graph and the comparison between both graphs is shown in figure 3.13 below.

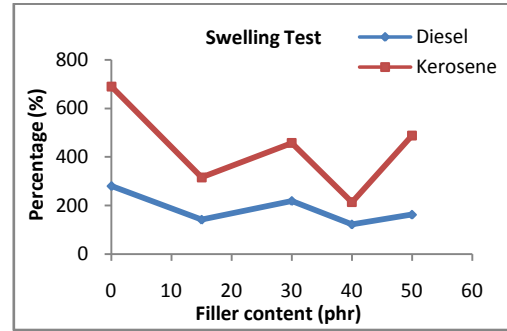


Figure 3.13: Comparison of swelling percentage between two solvent

From the graph, the swelling resistance in kerosene is higher than swelling resistance in diesel for all five samples. This is due to the smaller chemical structure of kerosene compared to the diesel which is kerosene is compilation of hydrocarbon chains that are made up of between 12 to 15 carbon atoms and diesel has a rigid molecular structure consisting of 16 carbon atoms and 34 hydrogen atoms. Besides that, kerosene has less density than diesel and both factors contribute to the high swelling percentage of kerosene.

4.0 CONCLUSION

From the results that were obtained, the increasing amount of fillers content used caused the tensile strength and Young's modulus increased until optimum 40 phr.

The increasing part of fillers content in rubber compound made the compound become more elastic and caused the elongation of samples reduce as the increasing part of fillers content. Mixing time also is increased as the increasing amount of fillers because the incorporation of fillers with natural rubber become harder because amount of fillers is increased but amount of natural rubber is reduced.

Swelling test also shows the increasing amount of fillers has made the swelling percentage or swelling resistance is reduced

because of the good barrier that was made by fillers. The increasing amount of fillers content made the better barrier in order to stop the penetration of solvent towards natural rubber. The swelling percentage also shows the higher value when is used in less dense solvent.

ACKNOWLEDGEMENT

Special thanks to Universiti Malaysia Pahang (UMP) to provide laboratories facilitator and to all technical staffs. Sincere appreciation is extended to Mr. Mohd Bijarimi Bin Mat Piah for constant guidance and supervision for this research.

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