# SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING UNIVERSITI SAINS MALAYSIA

# EFFECT OF VARIOUS MATERIALS SELECTION ON PHYSICAL AND MECHANICAL PROPERTIES OF TIRE TREAD

By

# ZAIHAN AZARINA BINTI SALIM

Supervisor : Associate Professor Dr Nadras binti Othman

Dissertation submitted in partial fulfillment of the requirements for the degree of Bachelor of Engineering with Honours (Polymer Engineering)

Universiti Sains Malaysia

**JULY 2017** 

# DECLARATION

I hereby declare that I have conducted and completed the research work and work the dissertation entitle "Effect of Various Materials Selection on Physical and Mechanical Properties of Tire Tread". I also declare that it has not been previously submitted for award of any degree or diploma or other similar title of this for any other examining body or university.

Name of student:	Zaihan Azarina binti Salim	Signature:
Date:	3 July 2017	

Witness by,

Supervisor: Assoc. Prof. Dr Nadras binti Signature: Othman Date: 3 July 2017

#### ACKNOWLEDGEMENTS

First and foremost, I want to thank Allah the Almighty for showing me inner peace and for all blessing.

Second, I would like to express my sincere gratitude to Dr. Nadras Othman, who has been my supervisor since the beginning of my final year project. Her immense knowledge and motivation have been of great value to me. This thesis would not have been possible without her many helpful suggestions, constructive advices and constant encouragement which indeed helped improve on this dissertation.

The opportunity I had with Eversafe Rubber Works Sdn Bhd was a great chance for learning and individual development. Therefore, I am so grateful for having chance to meet so many wonderful people and professionals who guide me throughout this project.

Then, I would like to express my gratitude to Mr Cheah Siang Ti, En Anuar, Production Manager, En Adzharuddin, QA Manager, En Rushdi, all QA and QC department for their help, support and advices during my time at Eversafe Rubber Works Sdn Bhd. I would like to show my gratitude towards all of the Eversafe Rubber staffs for their kindness, guidance, generosity to share their tremendous knowledge, experience and unlimited motivation from the starting of the final year project until the end of the project.

The lab assistances and all my fellow friends are gratefully acknowledged for their enthusiasm in helping and sharing their precious experiences which enabled me to complete this project in time.

Finally, a special word of gratitude and dedication must go to my parents who always inspired me to work hard and to be positive at all times, also to my siblings who all showed belief in me when times were difficult.

iii

# TABLE OF CONTENTS

Cont	tents		Page
DECL	ARATION		ii
ACK	NOWLEDGEMEN	ITS	iii
TABL	E OF CONTENT	S	iv
LIST	OF TABLES		vii
LIST	OF FIGURES		viii
LIST	OF ABBREVIATI	ONS	х
LIST	OF SYMBOLS		xi
ABST	ſRAK		xii
ABST	FRACT		xiii
CHAI	PTER 1 INTROD	UCTION	1
1.1		Background	1
1.2		Problem Statement	3
1.3		Research Objectives	4
1.4		Thesis Outline	5
CHAI	PTER 2 LITERAT		6
2.1		Tire Technology	6
2.2		Green Tire Technology	7
2.3		Rubber Formulation	8
2.4		Raw Materials	9
	2.4.1	Natural Rubber	9
	2.4.2	Styrene-Butadiene Rubber	10
	2.4.3	Polybutadiene Rubber	11
	2.4.4	Fillers	12
		i. Carbon Black	
		ii. Silica	13
	2.4.5	Rubber Processing Oil	14
2.5		Compounding	15
2.6		Equipment	16
2.7		Background of Study on Material Selection in	18
		Tire Tread Application	

# CHAPTER 3 METHODOLOGY

CH/	APTER 3	METHO	DOLOGY	19
3.1			Materials	19
	3.1.1		Natural Rubber (SMR10)	19
	3.1.2		Polybutadiene Rubber (BR)	19
	3.1.3		Styrene-Butadiene Rubber (SBR)	20
	3.1.4		Carbon Black	20
	3.1.5		Silica	20
	3.1.6		Rubber Processing Oil	21
	3.1.7		Coupling Agent	21
	3.1.8		Processing Aid	21
	3.1.9		Zinc Oxide	21
	3.1.10		Stearic Acid	22
	3.1.11		Antioxidant	22
	3.1.12		Accelerator	22
	3.1.13		Sulphur	22
	3.1.14		Retarder	23
3.2			Instruments	23
3.3			Methodology	24
	3.3.1		Preparation of Sample	24
		3.3.1.1	Sample Designation	24
		3.3.1.2	Weighing of the Ingredients	28
		3.3.1.3	Prepared of Masterbatch Sample	32
		3.3.1.4	Prepared of Final Mixing	33
3.4			Rheological Properties Test	33
3.5			Tensile and Tear Test	34
3.6			Hardness Test	35
3.7			Abrasion Test	35
3.8			Specific Gravity	35
3.9			Swelling Test	36
СН	APTER 4	RESUL	TS AND DISCUSSIONS	37
4.1			Comparative Study on Different Grade of	37
			Synthetic Rubber on Physical and Mechanical	
			Properties of Tire Tread	
	4.1.1		Curing Characteristic	37
	4.1.2		Swelling Test	40
	4.1.3		Mechanical Properties	41

REF	ERENCI	ES		80
5.2			Recommendation for Future Work	79
5.1			Conclusion	78
CHA	PTER 5	CONCLU	USION AND FUTURE STUDIES	78
		4.3.4.2	Abrasion Resistance	76
		4.3.4.1	Hardness	74
	4.3.4		Physical Properties	74
		4.3.3.4	Tear Resistance	72
		4.3.3.3	Modulus	70
		4.3.3.2	Elongation at Break	68
		4.3.3.1	Tensile Strength	66
	4.3.3		Mechanical Properties	66
	4.3.2		Swelling Test	64
	4.3.1		Curing Characteristic	62
			Tread	
			Physical and Mechanical Properties of the Tire	
4.3			Effect of Various Aromatic Processing Oil on	62
		4.2.4.2	Abrasion Resistance	61
		4.2.4.1	Hardness	59
	4.2.4		Physical Properties	59
		4.2.3.4	Tear Resistance	58
		4.2.3.3	Modulus	55
		4.2.3.2	Elongation at Break	54
		4.2.3.1	Tensile Strength	52
	4.2.3		Mechanical Properties	52
	4.2.2		Swelling Test	50
	4.2.1		Curing Characteristic	48
			Mechanical Properties of the Tire Tread	-
4.2			Effect of Various Carbon Black on Physical and	48
			Abrasion Resistance	47
		4.1.4.1	Hardness	46
	4.1.4		Physical Properties	46
			Tear Resistance	45
			Modulus	44
		4.1.3.2	Elongation at Break	43
		4.1.3.1	Tensile Strength	41

# LIST OF TABLES

		Page
Table 2.1	Sample Rubber Recipe	8
Table 3.1	List of instruments used during the project	23
Table 3.2	Designation for different grade of synthetic rubber of tire	24
	tread	
Table 3.3	Designation for different ratio of carbon black loading	26
Table 3.4	Designation for different ratio of aromatic processing oil	27
Table 3.5(i)	Formulation for different ratio of synthetic rubber of a rubber	29
	compound	
Table 3.5(ii)	Formulation for different concentration of carbon black of a	30
	rubber compound	
Table 3.5(iii)	Formulation for different concentration of rubber processing	31
	oil of a rubber	
Table 4.1	Cure characteristic on different grade of synthetic rubber of	38
	tire tread	
Table 4.2	Cure characteristic on different ratio of carbon black loading	49
Table 4.3	Cure characteristic on different ratio of aromatic processing	62
	oil	

# LIST OF FIGURES

# Page

Figure 2.1	Configuration of Natural Rubber (NR)	9
Figure 2.2	Configuration of Styrene-Butadiene Rubber (SBR)	10
Figure 2.3	Configuration of Polybutadiene Rubber (BR)	11
Figure 2.4	Mixing by using two roll mill	16
Figure 2.5	Mixing by using internal mixer	17
Figure 4.1	The effect of different grade of synthetic rubber on swelling	40
	percentage of tire tread	
Figure 4.2	The effect of different grade of synthetic rubber on the tensile	41
	strength of tire tread	
Figure 4.3	The effect of different grade of synthetic rubber on the	43
	elongation break of tire tread	
Figure 4.4	The effect of different grade of synthetic rubber on the modulus	44
	of tire tread	
Figure 4.5	The effect of different grade of synthetic rubber on the tear	45
	resistance of tire tread	
Figure 4.6	The effect of different grade of synthetic rubber on the	46
	hardness of tire tread	
Figure 4.7	The effect of different grade of synthetic rubber on the abrasion	48
	resistance index of tire tread	
Figure 4.8	The effect of carbon black loading on the swelling percentage	50
	of tire tread	
Figure 4.9	The effect of carbon black loading on the tensile strength of tire	52
	tread	
Figure 4.10	The effect of carbon black loading on the elongation break of	54
	tire tread	

Figure 4.11	The effect of carbon black loading on the modulus 100% of tire	56
	tread	
Figure 4.12	The effect of carbon black loading on the modulus 300% of tire	57
	tread	
Figure 4.13	The effect of carbon black loading on the tear resistance of tire	58
	tread	
Figure 4.14	The effect of carbon black loading on the hardness of tire tread	60
Figure 4.15	The effect of carbon black loading on the abrasion resistance	61
	index of tire tread	
Figure 4.16	Swelling percentage of tire tread at various aromatic	64
	processing oil contents	
Figure 4.17	Tensile strength of tire tread at various aromatic processing oil	66
	content	
Figure 4.18	Elongation at break of tire tread at various aromatic processing	68
	oil contents	
Figure 4.19	Modulus 100% of tire tread at various aromatic processing oil	70
	contents	
Figure 4.20	Modulus 300% of tire tread at various aromatic processing oil	71
	contents	
Figure 4.21	Tear resistance of tire tread at various aromatic processing oil	72
	content	
Figure 4.22	Hardness of tire tread at various aromatic processing oil	74
	contents	
Figure 4.23	Abrasion resistance index of tire tread at various aromatic	76
	processing oil contents	

# LIST OF ABBREVIATIONS

NR	Natural rubber
SBR	Styrene butadiene rubber
S-SBR	Solution styrene butadiene rubber
BR	Polybutadiene rubber
СВ	Carbon black
phr	Parts per hundred rubber
SMR	Standard Malaysian Rubber
TMQ	2,2,4-trimethyl-1,2-dihydroquinoline polymer
6PPD	N-(1,3-dimethylbutyl)-N'-phenyl-P-phenylenediamine
TBBS	N- tert-butyl-benzothiazole sulphonamide
UV	Ultraviolet
ZnO	Zinc oxide
PVI	N-(cyclohexylthio) phthalimide
IRHD	International Rubber Hardness Degree
ODR	Oscillating Disk Rheometer

# LIST OF SYMBOLS

°C	Degree celcius
o	Degree
%	Percentage
S	Second
T <sub>s</sub>	Tear strength in kiloNewtons per metre of thickness
F	Force
d	Thickness
mm	Milimeter
MPa	Mega Pascal
dN.m	Deci Newton meter
t <sub>s1</sub>	Scorch time
t <sub>90</sub>	Cure time
ML	Minimum torque
M <sub>H</sub>	Maximum torque
Min	Minute
lb-in	Pound per inch

# KESAN PEMILIHAN PELBAGAI BAHAN KE ATAS CIRI CIRI FIZIKAL DAN MEKANIKAL BUNGA TAYAR

## ABSTRAK

Dalam kajian ini, objektif utama adalah untuk mengenalpasti gred getah sintetik yang disyorkan, variasi kepekatan karbon hitam dan kandungan minyak pemprosesan aromatik pada sifat-sifat fizikal dan mekanikal untuk penggunaan bunga tayar. Pertama sekali, ciri-ciri pematangan diukur menggunakan Disk reometer (ODR) yang berayun. la digunakan untuk mengukur ciri-ciri pematangan sebatian getah yang tidak matang dan matang pada suhu yang tertentu mengikut ASTM D-2084. Sifat-sifat mekanikal seperti kekuatan tegangan, modulus tensil, pemanjangan pada takat putus cabikah dan rintangan telah diukur menggunakan mesin Go Tech. Ciri-ciri fizikal seperti kekerasan diukur menggunakan penguji kekerasan, Croydon Wallace dan Teclock. Indeks rintangan lelasan diukur menggunakan penguji lelasan, GT-7012-D. Dalam projek ini, terdapat enam gred yang berbeza yang digunakan, phr karbon hitam telah ditetapkan pada 28 phr, 33 phr, 35 phr dan 38 phr dan kandungan minyak pemprosesan aromatik telah ditetapkan pada 13 phr, 17 phr, 20 phr dan 22 phr. Keputusan mendedahkan bahawa gred getah yang berbeza menunjukkan perbezaaan di antara sifat-sifat fizikal dan mekanikal manakala penambahan kandungan karbon hitam dan kandungan minyak pemprosesan aromatik yang lebih tinggi membawa kepada kenaikan kekuatan tegangan, modulus, rintangan cabikah, kekerasan dan indeks rintangan lelasan manakala pemanjangan pada takat putus dikurangkan pada beban pengisi dan kandungan minyak yang lebih fungsi. Sifat-sifat ini telah bertambah baik kerana interaksi pengisi yang lebih baik dengan getah dan meningkatkan penyebaran pengisi dalam sebatian getah.

xii

# EFFECT OF VARIOUS MATERIALS SELECTION ON PHYSICAL AND MECHANICAL PROPERTIES OF TIRE TREAD

#### ABSTRACT

In this study, the main objectives was to identify recommended grade of synthetic rubber, variations of carbon black concentration and aromatic processing oil content on physical and mechanical properties for tire tread application. First of all, the curing characteristics was measured using the Oscillating Disk Rheometer (ODR). It is used to measure the complete curing characteristic of the rubber compounds from an uncured compound to a cured compound at a specific temperature according to ASTM D-2084. The mechanical properties such as tensile strength, tensile modulus, elongation at break and tear resistance were measured through Go Tech Testing machine. The physical properties such as hardness was measured using hardness tester, Croydon Wallace hardness and Teclock. The abrasion resistance index was measured using abrasion tester, GT-7012-D. In this project, there were six different grades of synthetic rubber that were used, the phr or carbon black loading was set at 28 phr, 33 phr, 35 phr and 38 phr and the aromatic processing oil content was set at 13 phr, 17 phr, 20 phr and 22 phr respectively. The results revealed that different grade of synthetic rubber shows different physical and mechanical properties while the incorporation of higher carbon black loading and aromatic processing oil content lead to the increment of tensile strength, modulus, tear resistance, hardness and abrasion resistance index while the elongation at break was reduced at higher filler loadings and oil content. These properties were improved due to a better filler interaction with rubber and enhanced the dispersion of the filler inside the rubber compound.

# **CHAPTER 1**

#### INTRODUCTION

#### 1.1 Background

In tire production, the function of the tire is to provide interface between the vehicle and the road surface. Historically, tires began in Great Britain during 19<sup>th</sup> century as an upgrade from the solid rubber tires (Lindemuth, 2006). Tires are an important component in automobile technology. The functions of tires can be viewed as a support for the load, acted as a spring, conveying driving and driving forces and facilitated steering of the vehicle. This functions are important in order to provide mutual relationship between the vehicle and the road surface (Ishikawa, 2011).

For tire tread production, the tread is specially formulated to provide grip for driving, braking and cornering. It also must provide a balance between wear, traction and rolling resistance. Besides that, the tread design also plays an important role on a variety of road surfaces. The design must provide uniform wear, passage for water out of the footprint and minimize the pattern noise (Lindemuth, 2006).

There are two major ingredients in a rubber compound are the rubber itself and the filler. In tire industry, natural rubber (NR), styrene butadiene rubber (SBR) and polybutadiene rubber (BR) are the most used elastomeric blends. A material that has ability to return to its original shape after being stretched is entitled to be called rubber. Natural rubber (NR) also known as polyisoprene is extracted from Hevea braziliensis tree (Ciesielski, 1999). NR has high mechanical strength and low mechanical hysteresis. It also has its drawback such as poor resistance to ozone, high temperature, weathering, acids and bases. BR and SBR have the kindness of good resistance to crack propagation. These properties make the rubbers mentioned highly attractive to develop blends in order to improve the performance of tires (School,

2001). Besides that, SBR must be reinforced with filler in order to provide the mechanical strength such as tensile strength and tear resistance (School, 2001).

Carbon black and silica are the most common reinforcing filler that is used in rubber compounds (Kaewsakul et al., 2011). They offer some advantages that contributes to the processibility or utility of the product. Addition of fillers increased the mechanical strength, hardness and stiffness of the cured product. Carbon black is basically elemental carbon in the form of fine amorphous particles (Ciullo et al., 1999).

Addition of carbon black in a rubber compound will improve the mechanical properties. Addition of precipitated of silica in the rubber compound results in low hysteresis and heat build-up. Lower hysteresis is translated into lower fuel consumption. Usually, the addition of precipitated silica is accompanied with the addition of carbon black (Ciesielski, 1999).

Vulcanization is an important process that caused the rubber molecules to bond together. This process is discovered by Goodyear in 1939. The reaction caused to adjoining molecules to bond together and form a network. Any stress to any part of the network is transmitted to the entire network. This demonstrates rubber elasticity (Ishikawa, 2011). Sulphur is the most used crosslinking agent and it reacts chemically with the raw rubber which results in more dimensionally stable and less heat-sensitive product (Ciesielski, 1999).

#### 1.2 **Problem Statement**

Tire industry is moving forward to improve the mechanical and physical properties, production cost and the product safety. This can be achieved with the improvement of wear resistance, rolling resistance and skid resistance. In recent study conducted by Hartomy et al, (2015), the performance of the tire increased due to incorporation of fillers and type of rubber inside the rubber compounding.

From this experiment, there are some problems that occur during the production of the tire tread. First, the high cost of the existing synthetic rubber. As the crude oil process continue to decline, the demand for synthetic rubber which is made of a crude oil is very high which keeps production rates high as well.

Besides that, the lower tear strength of the tire tread is also one of the problems occur during the production. If the tread has low tear strength, it is easy to tear off. By introducing high filler loading, the physical and mechanical properties would be increased. For example, by using N220, it will increase the wear resistance tires, high resistance to tensile, tolerable tear resistance. N220 is recommended for use in the manufacturing of conveyor belts and other rubber products includes in the tread rubber of tires for passenger cars and trucks, which require increased durability.

Lastly, the compound is too hard as the carbon black loading increased. The tire must be capable of transmitting strong longitudinal and lateral forces (during braking, accelerating and cornering) in order to assure optimal and reliable road-holding quality. It must be able to do all of this even when the road provides little traction in wet or slippery conditions or when the road is covered with snow or ice.

Therefore, it would be necessary to study the recommended grade of synthetic rubber, physical and mechanical properties of the tire tread with the variations in carbon black concentration and aromatic processing oil.

# 1.3 Research Objective

- i. To identify recommended grade of synthetic rubber with good physical and mechanical properties for tire tread application.
- ii. To investigate the physical and mechanical properties of the tire tread with the variations of carbon black concentration.
- iii. To investigate the physical and mechanical properties of the tire tread with the variations of aromatic processing oil concentration.

## 1.4 Thesis Outline

This thesis covered all chapters that consists of :

Chapter 1 : Background, problem statement, research objective and thesis outline that involved in this research.

Chapter 2 : Consists of literature review of the project, extensive review of the project, several materials used during compounding and testing that is conducted to test the sample.

Chapter 3 : The details of the raw materials that are used in this project, experimental procedure, machine handling procedure and testing that involved during generating experimental data for this research.

Chapter 4 : Data and results obtained for this research are tabulated as well as the discussion on the mechanical and rheological properties in details.

Chapter 5 : Summary and conclusion on this research work as well as suggestions for further research in future.

# **CHAPTER 2**

## LITERATURE REVIEW

### 2.1. Tire Technology

In the middle of 19<sup>th</sup> century, the pneumatic tire was originally invented by a Scottish inventor, Robert William Thomson. Tires are an important component of an automobile and this technology has showed dramatically growth during the World War 2 which also played an essential role in military supplies. During the War, the tire manufacturing has significantly damaged but it is rapidly recovered by a dramatic growth of motorisation (Ishikawa, 2011).

There are three stages of the growing post-War motorisation. First stage was when new materials such as nylon and synthetic rubber were developed for the tire. Second stage was in the modification of tire construction where the bias tire was changed to radial tire completely. Next, advance in the development of the steel radial tire was incorporated during the third stage. The adhesion between the rubber and steel cords and also preventing rubber fatigue during tire's working life also achieved in this stage (Ishikawa, 2011). Now, radial tires has dominated the passenger tire market.

#### 2.2. Green Tire Technology

Nowadays, the tire industry is moving forward in the development of green tires for the next generation. Thus, a further step is implemented to reduce the environmental impact because the current tire technology is primarily petroleum based and the availability of the fossil fuels sources is limited (Martin et al., 2015).

According to the study, instead of using the carbon black in tire tread compounds, silane-treated silica was introduced as a reinforcing filler in the compounds when the green tire's concept is introduced. As a result, tire rolling resistance and wet traction were improved. As the rolling resistance is lower, this will improve the fuel efficiency because of the less friction between the tire and the road surface also, the energy is less wasted (Job, 2014).

However, one of the weakness of this technology is the difference in polarity between silica and rubber because it is difficult to process them. The silica surface is acidic with a number of silanol groups. A strong hydrogen bond is formed and the reaction between the polar materials such as accelerators resulting in the adsorption of curatives by silica. Besides that, the strong hydrogen bonds between the silica particles also caused poor dispersion in rubber compounds as the silica particles themselves form aggregates and agglomerates. This difficulties can be overcome by using silane coupling agents (Kaewsakul et al., 2011).

#### 2.3. Rubber Formulation

Compounding means a number of materials such as elastomer and additives are combined together to produce a product. The basic function of rubber technology is the designation of a rubber compound formulation (Ciullo et al., 1999).

Dick (2001) stated that a rubber compound formulation is usually referred as a recipe. This recipe is based on raw rubbers, a reinforcing filler, a plasticizing oil, antidegradant package and a curative system. Each of this ingredients are expressed in "parts per hundred rubber" or "phr". In a given recipe, the total parts of different rubbers is always defined as 100 while other non-rubber ingredients are "ratioed" against the 100 parts of rubber hydrocarbons.

The variety in different type of recipes are used in the rubber industry to meet a set of goal properties. Rubber companies tend to keep their formulation within themselves because it can cost a great deal to develop a good performance rubber compound and this secrecy adds to the variety of formulation and competition between the rubber companies (Dick, 2001).

No	Ingredients	Phr	Compound Function
1.	SMR 20	70.0	Natural rubber
2.	SBR	30.0	Synthetic rubber
З.	Carbon Black	55.0	Reinforcing agent
4.	Naphthenic oil	5.0	Processing oil
5.	Zinc Oxide	4.0	Activator
6.	Stearic Acid	2.0	Activator
7.	TMQ	4.0	Anti-oxidant
8.	6PPD	0.3	Anti-oxidant
9.	TBBS	1.3	Accelerator
10.	Sulphur	1.50	Vulcanizing agent

Table 2.1 : Sample Rubber Recipe (Dick, 2001)

#### 2.4. Raw materials

## 2.4.1. Natural Rubber

Natural rubber (NR) is extracted in the form of latex from Hevea tree. When it is stretched, NR tends to crystallize spontaneously due to its high structural regularity at low temperature. The strain-induced crystallization results in higher tensile strength and resistance to cutting, tearing and abrasion (Ciullo et al., 1999). Stretching a piece of natural rubber at room temperature turns the amorphous rubber into a semi-crystalline material. These crystallites are highly oriented along the tensile direction and, acting like filler particles or crosslinks, tremendously elevate the tensile strength. The tire industry takes advantage of these outstanding mechanical properties, using compounds high in natural rubber for truck tires to extend the tire lifetime. Due to the local reinforcement of strain-induced crystallites at the highly strained crack tip, the crack growth resistance in natural rubber is orders of magnitude higher than in conventional synthetic non-crystallizing rubbers. Figure 2.1 shows the configuration of natural rubber (NR)

$$\begin{array}{c} \mathsf{CH}_3 \\ | \\ \mathsf{CH}_2 - \mathsf{C} = \mathsf{CH} - \mathsf{CH}_2 \end{array} \right) \\$$

Figure 2.1 : Configuration of Natural Rubber (NR)

It has good dynamic mechanical properties which is a good properties to be used in tires, rubber springs and engine mounts. Addition of carbon black provides good resistance to ultra violet (UV), antiozonants and waxes and ozone resistance (Ciesielski,1999).

Uncured NR has uniquely building tack. In tire manufacturing, the separate pieces are uniquely held together by building tack and during cure, they are pressed and bond together into a single piece (Ciullo et al., 1999).

#### 2.4.2. Styrene-Butadiene Rubber

Styrene-butadiene rubber (SBR) is widely produced by solution and emulsion polymerization. For emulsion polymerization, it depends on the initiating system either to carry out the process in hot or cold process. Usually, both random and block SBR can be produce with solution polymerization that contain the same amount of styrene. Meanwhile, SBR that is produced with emulsion polymerization contains about 23% styrene that is randomly dispersed with butadiene in the polymer chains (Ciullo et al., 1999). Figure 2.2 shows the configuration of styrene-butadiene rubber (SBR).

$$(C_{6}H_{5})$$

$$(CH_{2}-CH=CH-CH_{2})_{5}-(CH-CH_{2})$$

Figure 2.2 : Configuration of Styrene-Butadiene Rubber (SBR)

SBR is highly used in the application of passenger car tires particularly in tread compounds for superior traction and treadwear. Typically, SBR produced a better abrasion, crack initiation and heat resistance compound compared to natural rubber. Arayapranee, (2012) state that polymerization at a lower temperature became possible, giving less branches and gels. "Cold" SBR generally has a higher average molecular weight and narrower molecular weight distribution. Since higher molecular weight can make cold SBR more difficult to process, it is commonly offered in oil-extended form. The random copolymers offer narrower molecular weight distribution, low chain branching, and lighter color than emulsion SBR. They are comparable in tensile, modulus, and elongation, but offer lower heat build-up, better flex, and higher resilience.

The processing is similar to natural rubber in the procedure and type of additive used but the extrusion with SBR are smoother because the shape is maintained better than natural rubber (Ciullo et al., 1999).

#### 2.4.3. Polybutadiene Rubber

Polybutadiene (BR) rubber is produced from a monomer of butadiene. BR are currently come with four variations: high cis content polymers which are produced by with the presence of Ziegler Natta catalysts, low or medium cis content polymers which are produced with alkyl lithium catalyst, vinyl BR which is also produced with alkyl lithium catalyst but in a presence of polar additive and emulsion BR which is produced in water using free radical initiators (Day, 2001). Figure 2.3 shows the configuration of polybutadiene rubber (BR).

 $(CH_2-CH=CH-CH_2)$ 

Figure 2.3 : Configuration of Polybutadiene Rubber (BR)

Traditionally, BR is difficult to process but this difficulty is not apparent when BR is blended with NR which is non polar elastomer. BR offers high resilience which will result in low heat build-up under continuous dynamic deformation, good abrasion resistance when it is blends with other rubbers (Ciesielski, 1999).

In passenger car tires, a large volume of BR is blends with SBR or NR to improve hysteresis, abrasion resistance and cut growth resistance of tire treads. It depends on what type of properties that are important to the particular compound. For an excellent abrasion resistance and low rolling resistance, high-cis and medium-cis BR is used but the wet traction is not improved. The wet traction is improved with low rolling resistance in high-vinyl BR but the abrasion resistance is poor. Medium-vinyl BR shows a balance in wet traction with good abrasion resistance and low rolling resistance (Ciullo et al., 1999).

#### 2.4.4. Fillers

#### i. Carbon black

Today, most of the rubber fillers used contribute to the processability of a rubber product. The properties of a filler will impart to a rubber compound and the characteristics to determine the properties of a filler are particle size, surface area, structure and surface activity (Ciullo et al., 1999).

Particle size refers to the size of filler particle. It can be measured with the use of an electron microscope. (Laube et al., 2001). If it exceeds the polymer interchain distance, the area of a localized stress is introduced. This process will further contribute to chain rupture on flexing or stretching (Ciullo et al., 1999).

The amount of carbon black surface available to interact with elastomer refers to the surface area. To form an aggregate, surface area is inversely proportional to the size of carbon black particle for the non-porous carbon black. The structure refers to the number of particles that are fused together to form an aggregate. If the shape is complex, a greater void volume will be created. Meanwhile, surface activity refers to the strength of the filler's surface interaction with the polymer. It also plays a role in modulus, hysteresis and abrasion resistance (Laube et al., 2001).

Most of rubber factory's operation, they used carbon black into the rubber compounds. The used of carbon black will impart the performance characteristics of the final product whether in tire component or in industrial rubber product (Laube et al., 2001).

#### ii. Silica

Precipitated silica is an amorphous silica produced by precipitation from aqueous solution. In tire tread compounds, silica is used because of its properties which shows low heat build-up and low hysteresis. Addition of silica usually associated with the addition of carbon black (Ciesielski,1999).

Nowadays, silicas have been used in tires in order to improve damage resistance in on and off truck treads applications. Silica is highly polar and the silica surface contains adsorbed water. The cure rate is tend to retard because of the hydroxyl groups in silica surface is highly acidic. Therefore, it has high tendency to react with zinc oxide activator (Ciullo et al., 1999).

The physical properties of silica has further increased by using organosilane coupling agents. The bifunctional organosilane reacts with silica with the presence of alkoxy group of the silane in order to form covalent bond structure. The performance of conventional carbon black sidewall compounds is improved with the addition of reinforcing precipitated silica (Waddel et al., 2001).

In recent study shows that moisture on the silica enhances the reaction between the silica and coupling agent. Therefore, the interparticle interactions also will be reduced (Schaal et al., 1999).

#### 2.4.5. Rubber processing oil

During compounding, processing oil is added primarily to serve as a plasticizing function. They also can reduce the compound cost if high amount of oil is used. In addition, the processing oil also promotes mold flow and release, reduce the viscosity of the elastomer, improve milling and extrusion and facilitate incorporation of fillers (Ciullo et al., 1999).

As the filler loading is increased, the addition of aromatic processing oil offer advantages such as wet grip enhancement to tread compounds. There are paraffinic, naphthenic or aromatic types but in tire treads, the use of distilled aromatic extract is attractive due to the similarity in glass transition temperature to SBR polymer (White, 1998).

The addition of oil to rubber compounds primarily to reduce the viscosity, stress/strain resistance and the hardness of the end product. Besides that, the addition of oil also will impart the mixing process as the wetting and lubricity characteristics is improved. However, the hardness and modulus is getting lower if too much process oil is added into the compound. Poor abrasion resistance and the fouling of mould in injection molding also the disadvantages if too much oil is added (Laube et al., 2001).

The properties such as viscosity, hardness, modulus and elongation tend to be affected by the usage of carbon black and processing oil. To maintain the properties, processing oil and carbon black are frequently increased or decreased simultaneously (Laube et al., 2001).

#### 2.5. Compounding

Ciullo et al and Ciesielski (1999) stated that there are two simple operations that are involved during the manufacturing of rubber product which are compounding and curing. The first step in rubber compounding is mastication process. Mastication is described as mechanical breakdown of the polymer chain and this is crucial to make it receptive to the additives and provide a uniform mixture.

Initially, NR has higher viscosity and time taken to achieve uniformity can be longer. Therefore, NR need to be masticated in order to control the viscosity and properties of the rubber compound. Historically, the polymer breakdown takes place on open roll mills. The two metal rolls turned to each other at fixed separation and at different speeds to provide better shear mixing forces. Nowadays, this step is performed in an internal mixer which the mixing shear and mixing time is determined by rotor shape, size and speed (Ciullo et al., 1999).

Remilling process is needed for most of the compound with higher loading of carbon black. This step is to prepare the masterbatch compound for the nest step if it shows poor dispersion (Ciullo et al., 1999).

Masterbatch process is a process to incorporate the compounding ingredients into the rubber, to obtain homogenous blending and develop proper final viscosity. This process excluded the curing system. Without curing system, masterbatch process can extensively mixed without causing premature vulcanization. The incorporation of the curing system completes the rubber compounding. During this process, mixing rate and time is important to ensure the homogeneity and to avoid premature curing. This process is normally done in an internal mixer like Banbury (Ciullo et al., 1999).

#### 2.6. Mixing Equipment

Mixing operations is one of the important stage which all the chemicals are blend together with the rubber. This process can be accomplished by using mills or internal mixer. The objectives of mixing operation are to obtain a uniform dispersion of fillers and viscosity of a rubber compound (Hacker, 2001).



Slower roll Faster rolt

#### Figure 2.4 : Mixing by using two roll mill (Murakami et al., 1985)

In early 1820s, mixing of a rubber compound took place on two roll mills or in single motor machine. Primarily, the mill is used to prepare coloured, sticky or hard compound in the production. Production volume, size of the batch and how to operate the mills affected the batch. The mill operator need to control the sensitive compound and minimize the size of the compound. In modern tire plant, the mill has been placed with internal mixer to sheeting and cooling compounds for further stage (Hacker, 2001).

Maintain sufficient viscosity is the key to ensure an adequate shearing action in mixing in order to distribute the ingredients into the raw rubber. Fernley H. Banbury improved the internal mixer by designing the Banbury mixer in 1916. It has displaced the mill by producing less dust from powdery materials, used less space and less operator sensitive (Ciesielski,1999).



Figure 2.5 : Mixing by using internal mixer

This internal mixer has two type of mixing action, first is Tangential rotor type and next is Intermeshing rotor type. For tangential rotor type, the addition of ram increased the effectiveness of the mixer dramatically. The material is transferred around the mixing chamber from one rotor to other rotor to allow a good dispersion. The Banbury was developed further to promote the cooling of machine by adding a hinged of hopper door and a sight rod attached to the weight at the bottom of the ram. The machine also replaced the sliding gate with the drop door (Hacker, 2001).

The intermeshing rotor type approached the mixing difficulty by transfer the material around the chamber by mill rolls. The material is transferred on each rotor along its length and in the opposite direction from other rotor. Initially, the mixing takes place in the nip between the two rotors (Hacker, 2001).

#### 2.7. The effect of material selection in tire tread production

Composition of the tire tread compound determined the tire properties which is important in wet traction, treadwear and rolling resistance. These properties is difficult to be improved at the same time. Usually, variations in the grade or composition of carbon black improved one or two properties (Schaal et al., 1999).

There are three properties that determine the overall performance and efficiency of tire tread. This properties are good rolling resistance, wet skid resistance and excellent abrasion resistance. Type of polymer, composition and type of filler, processing aids and type of vulcanization system affects these properties in order to reach requirement in the manufacturing (Rathi et al., 2015).

It shows that if the compounds are too soft, this will results in poor wet traction. If the hardness of the compounds is too hard, there is a tendency for that compound to wear and reduced in dry traction. In order to maintain the compound hardness, it is essentially to remove or add one part of carbon black and oil at the same time. The addition of oil is required for higher surface area of carbon black and vice versa (Laube et al., 2001).

Processing oil plays an important element in the formulation of tire tread application. A good compatibility with natural and synthetic rubber is provided with the addition of processing oil in the compounding. Besides that, a better wet skid resistance is achieved with the incorporation of oil. By adding processing oil in the compound, it will reduce the viscosity and act as energy saving because the shear rate is reduced, increases the uniformity of the mixing and filler loading and hence improve the final properties. The final compound improved the cost to performance ratio with the use of process oil (Rathi et al., 2015).

# **CHAPTER 3**

## METHODOLOGY

This chapter provides information on the materials used as well as the description of sample preparations and the testing techniques used throughout the project.

#### 3.1 Materials

#### 3.1.1 Natural Rubber (SMR 10)

Natural rubber (NR) that was used in this project is Standard Malaysian Rubber with grade 10 (SMR 10). This natural rubber was purchased from Lee Rubber Co. which is located in Muar, Johor. The grade of the natural rubber determined the dirt content of the rubber. The lower the number, the lower the dirt content of natural rubber. The grade determines 0.054% of dirt content, 0.25% of nitrogen and 0.50% of ash content. During deformation, NR holds its strength and due to its hysteretic properties, NR has higher resilience (School, 2001).

#### 3.1.2 Polybutadiene Rubber (BR)

It is manufactured by Zeon Corporation, Tokyo, Japan. Polybutadiene (BR) is either produced by an emulsion process or a solution process. It uses lower proportions in the blend such as with natural rubber (NR) and styrene butadiene rubber (SBR) to improve the low temperature flexibility (School, 2001).

BR vulcanizates able to blend with other rubbers as it offers high resilience, low heat build-up and good abrasion resistance (Ciesielski, 1999). The volatile matter is 0.15% and the cis 1,4 content is 98.8%.

#### 3.1.3 S-SBR

Styrene butadiene rubber (SBR) is widely produced either by emulsion or solution polymerization. In tire industry, solution SBR is extensively used because the material offers improvement in both abrasion and traction as well as in hysteresis properties. (School, 2001)

SBR must be reinforced with fillers such as carbon black in order to obtain a good mechanical strength as well as to increase the hardness properties.

#### 3.1.4 Carbon Black

Carbon black was supplied by Cabot Corporation (Malaysia) which is located in Negeri Sembilan. It is a reinforcing filler that is categorized in intermediate super abrasion furnace (ISAF). Particle size and structure determine the reinforcing properties of the carbon black. The size of the N220 is between 24nm to 33nm. It is used to enhance the physical properties such as hardness, tensile strength, modulus, tear strength and abrasion resistance of a compound.

#### 3.1.5 Silica

Silica or silicon dioxide is added to the rubber compound in order to improve the cured physical properties, reduce cost or to impart certain process properties. It also has high abrasion strength and increased with increasing surface area. Precipitated silica is made by precipitation from aqueous solution.

It was supplied by Behn Meyer Chemicals. The moisture content is 8% while the particle hardness is 45%.

#### 3.1.6 Processing Oil

It was supplied by GT-Gulf (M) Sdn. Bhd which is located in Petaling Jaya, Selangor. The density of this rubber processing oil is 0.8877g/cm<sup>3</sup> with water content 0.04%. The pour point is below -6°C.

### 3.1.7 Coupling Agent

Coupling agent plays an important role in the surface morphology. It affects the interaction of the silica with other chemicals in a rubber compound.

It was supplied by Behn Meyer Chemicals (M) Sdn Bhd from Subang Jaya. Its appearance is light yellow liquid. The sulphur content is above 22% while the chlorine content is below 0.6%. The flash point is above 100°C. Specific name of coupling agent is not mentioned in this thesis due to the secrecy of the company.

#### 3.1.8 Processing Aid

Processing aid is the ingredients that are added to a rubber compound to facilitate the processing operations or to ease the operations. It was supplied by Performance Additives which is located in Subang Jaya, Selangor.

The shelf life of this processing aid is two years. The ash content is 11.5% while the zinc content is 14%. The melting point is 98°C. Specific name of processing aid is not mentioned in this thesis due to the secrecy of the company.

#### 3.1.9 Zinc Oxide

Zinc oxide (ZnO) acted as an activator in a sulphur vulcanization system. It was supplied by Approfit Zinc Oxide Manufacturing, Senawang, Negeri Sembilan as a white powder which has ZnO content of 99.69%. The moisture content in this Zinc oxide powder is 0.1297%. Besides that, the shelf life is three years if it is tightly closed and properly stored in a dry cool place.

#### 3.1.10 Stearic Acid

Stearic acid reacts with zinc oxide to form zinc stearate. For this project, this stearic acid was supplied by Behn Meyer (M) Sdn Bhd from Subang Jaya. It is in a form of white to yellow powder which contained 6.0 maximum of iodine value.

#### 3.1.11 Antioxidant

6PPD which its chemical name is N-(1,3-dimethylbutyl)-N'-phenyl-Pphenylenediamine is used to prevent the rubber compound from aging. This type of antioxidant was supplied by Jiangsu Sinorgchem Technology, China. It is grey-brown particles in appearance. The melting temperature is more than 45°C and its crystallisation point is above 46°C.

Besides that, TMQ which is also an antioxidant was supplied by Luxchem Trading in the form of grains and in an amber coloured. The ash content is 0.02%. Besides that, the softening point is at 94.70°C.

#### 3.1.12 Accelerator

TBBS which its chemical name is N- tert-butyl-benzothiazole sulphonamide, an accelerator that is added to the compounding ingredients in a small amount with a curing agent to speed up the rate of vulcanization. It was supplied by Behn Meyer (M) Sdn Bhd from Subang Jaya. It is in greyish yellow with melting temperature of 98°C. Without the presence of accelerators, the vulcanization of sulphur requires several hours.

#### 3.1.13 Sulphur

Sulphur is the most used vulcanizing agents. Vulcanizing agents are ingredients that is needed during the compounding so that it forms crosslinking between the polymer chains which will results in more dimensionally stable and less heat sensitive product. It was supplied by Kong Long Huat Chemicals.

The shelf life is one year with a condition to keep in cool and dry place. Moreover, it is suitable for vulcanizing rubber because it has low ash content (0.02%), low acidity (0.005%), moisture (0.05%) and sufficient fineness for adequate dispersion and reaction (Ciesielski,1999).

#### 3.1.14 Retarder

PVI or N-(cyclohexylthio) phthalimide is added into the compounding in order to reduce the scorchiness. Besides that, it will increase the processing safety without affecting cure rate. It is supplied by Behn Meyer Chemicals (M) Sdn Bhd, Subang Jaya. The appearance is white or in a yellow crystalline and the ash content is 0.1%.

## 3.2 Instruments

The instruments used in this project are listed as shown in Table 3.1

Test/Process	Instruments	Model
Mixing	Lab internal mixer	CL-BM16
	Two roll mill	
Rheological	Mooney Viscometer	• MV2000
Measurement	Moving die rheometer	• MDR2000
		Mosanto R100
Vulcanization	Hot press	QLB-35X2
Weighing	Weighing scale	Tanita KD-200
Dumbell Cutting	Dumbell Cutting Machine	-
Machine		
Specific Gravity	Electronic Densimeter	MD300S
Tensile and Tear Test	Tensile and Tear Testing	Go Tech Testing Machine
	Machine	

Table 3.1 : List of instruments used during the project

Abrasion Test	Abrasion tester	GT-7012-D
Hardness Test	Hardness tester	Croydon Wallace
		Hardness
		Teclock durometer
Carbon Black	Carbon Black dispersion tester	UD-3500
Dispersion		

## 3.3 Methodology

In this project, there were three series of study being investigated. For first series, the study was focused on the mechanical and physical properties of the recommended grade of synthetic rubber for tire tread application.

Next, second series was to investigate the physical and mechanical properties of the tire tread with the variations of carbon black concentration. Later, third series was to investigate the physical and mechanical properties of the tire tread with the variations of aromatic processing oil.

## 3.3.1 Preparation of sample

## 3.3.1.1 Sample designation

Table 3.2 shows the designation table for different grade of synthetic rubber of tire tread rubber that was used throughout the thesis.

Code	Designation for different grade of synthetic rubber of tire tread
N60/S1S10	Refer as ;
	N60 = 60  phr of  NR
	S1 = Grade 1 which was supplied from Singapore
	S10 = 10 phr of SBR