

### UNIVERSITI TEKNIKAL MALAYSIA MELAKA

## PHYSICAL AND MECHANICAL PROPERTIES OF NATURAL RUBBER REINFORCED CARBON BLACK FOR EFFECT OF TAPIOCA STARCH LOADING

This report submitted in accordance with requirement of the UniversitiTeknikal Malaysia Melaka (UTeM) for the Bachelor's Degree in Manufacturing Engineering Technology (Process and Technology) (Hons.)

by

### MUHAMMAD NASIRUDDIN BIN SHAMSUDDIN

B071310888

910913-01-5163

# FACULTY OF ENGINEERING TECHNOLOGY

2016





## UNIVERSITI TEKNIKAL MALAYSIA MELAKA

### **BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA**

TAJUK: PHYSICAL AND MECHANICAL PROPERTIES OF NATURAL RUBBER REINFORCED CARBON BLACK FOR EFFECT OF TAPIOCA STARCH LOADING

SESI PENGAJIAN: 2016/17 Semester 1

Saya MUHAMMAD NASIRUDDIN BIN SHAMSUDDIN mengaku membenarkan Laporan PSM ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

- 1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
- 2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
- 3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
- 4. \*\*Sila tandakan ( $\checkmark$ )

SULIT	(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia sebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)
TERHAD	(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)
TIDAK TERHAD	
	Disahkan oleh:
Alamat Tetap: No.10 Jalan GU 2/10	Cop Rasmi:
Taman GaringUtama	
48000 Rawang	
Selangor DarulEhsan	
Tarikh:	Tarikh:
	u TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi ekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai

### APPROVAL

This report is submitted to the Faculty of Engineering Technology of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Manufacturing Engineering Technology (Process and Technology) (Hons.). The member of the supervisory is as follow:

.....

HairulEffendy Bin AbMaulod

### ABSTRACT

This study focuses on physical and mechanical properties of natural rubber reinforced carbon black for effect of tapioca starch loading. Tapioca Starch (TS) is used as organic based filler for Natural Rubber (NR) as a primary matrix material by using literature based formulation in search of improvement on their physical and mechanical properties. The main objective of this research is to study the effect of physically modified tapioca starch on the properties of the Natural Rubber compound. Physical modification of the tapioca starch is through thermal treatment by heating the tapioca below its pasting temperature of about  $50^{\circ}C \pm 0.1^{\circ}C$  to improve compatibility with the natural rubber compound. The compound will undergo a few testing to determine both physical and mechanical testing. Tensile testing will be performed to determine various mechanical properties. Tensile test and Shore hardness test will determine physical properties of the compound. Morphology of the compound will be observed under optical microscopy. The optimum compounding formulation of the fabricated compound wasobserved at the combination of natural rubber compound and tapioca starchbased on 100 phr of the natural rubber (NR) SMR-20 and different proportions tapioca starch (TS) at 5, 10, 20, 40 and 60 phr. Curing additives for all those formulations based rubber were 2.5 phr of Sulphur (s), 5 phr of Zinc oxide (Zn0), 2 phr of stearic acid, 1 phr of Tetraethyl ThiuramDisulphate (TMTD) and 1 phr of 6PPD.

### ABSTRAK

Kajian ini memberi tumpuan kepada ciri-ciri fizikal dan mekanikal getah yang diperkukuhkan oleh karbon hitam untuk mengetahui kesan dari kanji ubi kayu. Tepung Ubi (TS) digunakan sebagai pengisi berasaskan organik untuk Getah Asli (NR) sebagai bahan matriks utama dengan menggunakan rumusan berasaskan penyelidikan dalam mencari penambahbaikan pada sifat-sifat fizikal dan mekanikal mereka. Objektif utama kajian ini adalah untuk mengkaji kesan kanji ubi kayu yang diubah suai secara fizikal pada sifat-sifat sebatian Getah Asli. Pengubahsuaian fizikal menggunakan kanji ubi kayu yang melalui rawatan haba dengan memanaskan ubi kayu di pada suhu kira-kira 50 ° C  $\pm$  0.1 ° C untuk meningkatkan keserasian dengan sebatian getah asli. Sebatian yang akan menjalani ujian beberapa untuk menentukan kedua-dua ujian fizikal dan mekanikal. Ujian tegangan akan dilakukan untuk menentukan pelbagai sifat-sifat mekanikal. Ujian tegangan dan ujian kekerasan akan menentukan ciri-ciri fizikal kompaun. Morfologi kompaun itu akan diperhatikan di bawah mikroskop optik. Penggubalan pengkompaunan optimum kompaun direka diperhatikan pada gabungan sebatian getah asli dan kanji ubi kayu berdasarkan 100 phr getah asli (NR) SMR-20 dan perkadaran yang berbeza ubi kanji (TS) pada 5, 10, 20, 40 dan 60 phr. Pengawetan bahan tambahan untuk semua orang-rumusan berasaskan getah telah 2.5 phr daripada Sulphur (s), 5 phr daripada zink oksida (Zn0), 2 phr asid stearik, 1 phr daripada tetraetil ThiuramDisulphate (TMTD) dan 1 phr daripada 6PPD.

### **DEDICATIONS**

To my beloved family, friends and lecturers which have been the source of my inspiration to undergo this project to a success. Thank you for all the support and encouragement from the start until end.



### ACKNOWLEDGMENTS

I would like to express my appreciation to Allah S.W.T. for giving me grace, love and blessing, to help me to pursue my ambition up to this point of my life.

I would like to submit my greatest gratitude to my supervisor, Mr Hairul Effendy Bin Ab Maulod for his sincere guidance and help to complete this project.

I am very indebted to my respectful teaching engineers Mrs.Mazliah Binti Mazlan and Dr.Noraiham Binti Mohamad from the Manufacturing Engineering Department for their sincere help in preparing this project.

I would also like to record my appreciation to my friends at Universiti Teknikal Malaysia Melaka for their encouragement and moral support that make my project more enjoyable.



## TABLE OF CONTENT

APPRO	DVAL	i
ABSTF	RACT	ii
ABSTF	RAK	iii
DEDIC	ATIONS	iv
ACKN	OWLEDGMENTS	v
TABL	E OF CONTENT	vi
LIST C	OF TABLES	X
LIST C	OF FIGURES	xi
LIST C	F ABBREVIATIONS, SYMBOLS AND NOMENCLATURE	xiv
CHAP	TER 1	1
INTRC	DUCTION	1
1.0	Background of Study	1
1.1	Problem Statement	2
1.2	Objectives	2
1.3	Scope	2
CHAP	TER 2	3
INTRC	DUCTION	3
2.0	Introduction	3
2.1.	History of natural rubber	4

2.2.	Natur	al rubber	4
	2.2.1.	Properties of Natural Rubber	6
	2.2.2.	Structure and characteristics of Natural Rubber	8
	2.2.3.	Vulcanization of Rubber	10
	2.2.4.	Sulphur vulcanizing agents	13
	2.2.5.	Non-sulphur vulcanizing agents	14
	2.2.6.	Uncured vs. vulcanized rubber	16
2.3.	Activ	ator (Sulphur Vulcanization)	17
2.4.	Filler	S	18
	2.4.1.	Carbon black	19
	2.4.2.	Non-black fillers	19
2.5.	Plasti	cizers	20
2.6.	Antid	egradants	20
2.7.	Tapio	ca	21
2.8.	Tapio	ca starch	21
	2.8.1.	Starch microstructures	22
CHAP	TER 3		24
3.0	Introc	luction	24
3.1.	Meth	odology	25
3.2.	Mater	rials Preparation	25
	3.2.1.	Preparation of Natural rubber compound	26

	3.2.2.	Preparation of Tapioca Starch	28
3.3.	Samp	le fabrication	28
	3.3.1.	Internal Mixer (Haake Internal Mixer)	31
	3.3.2.	Hot Press Compressing	35
3.4.	Mech	anical Testing	36
	3.4.1.	Tensile Test Machine	36
	3.4.2.	Shore Hardness	39
3.5.	Analy	vsis	40
	3.5.1.	Optical Microscope	40
CHAP	TER 4		42
4.0	Introd	luction	42
4.1.	Tensi	le test analysis	42
	4.1.1.	Tensile test analysis for tensile stress versus	
		tensile strain and load versus elongation	42
4.2.	Shore	hardness test	53
	4.2.1.	Shore hardness test result by between	
		different tapioca starch percent (phr)	53
4.3.	Optic	al Microscope analysis	55
	4.3.1.	Optical Microscope analysis for tensile	
		fractured surface of material	55

### CHAPTER 5

5.0	Conclusion	60
5.1.	Recommendations	60
REFER	ENCE	61



60

## LIST OF TABLES

Table 2.1: A generalized rubber recipe	6
Table 2.2: Raw Natural Rubber versus Vulcanized Natural Rubber	16
<b>Table 3.2:</b> The Natural Rubber with Tapioca Starch Formulation in gram	33
Table 3.3: Durometers of various common materials	39
Table 4.1: Control sample tensile test result	43
Table 4.2: Sample 1 tensile test result	44
Table 4.3: Sample 2 tensile test result	46
Table 4.4: Sample 3 tensile test result	47
Table 4.5: Sample 4 tensile test result	49
Table 4.6: Sample 5 tensile test result	50
Table 4.7: Shore hardness test reading	54

## LIST OF FIGURES

Figure 2.1: Rubber tree (Hevea brasiliensis)	4
Figure 2.2 Polymer chain of natural rubber	8
<b>Figure 2.3:</b> Schematic of (a) raw rubber and (b) vulcanized rubber (Ma al. 2013)	ark et 11
<b>Figure 2.4:</b> Picture of two line (blue and green) of natural rubber vulcanization with sulphur.(J.R. White and S.K. De, 2001.)	after 12
Figure 2.5: Effects of cross- link density vulcanizate properties(Mark 2013)	et al. 12
Figure 2.6: Sulphur vulcanization	13
Figure 2.7: Peroxidic vulcanization	15
Table 2.2: Raw Natural Rubber versus Vulcanized Natural Rubber	16
Figure 2.8: Carbon black	19
<b>Figure 2.9:</b> Chemical structures and schematic representation of (a) arr starch and (b) amylopectin starch	nylase 23
Figure 3.1: Flow chart of methodology process	24
Figure 3.2: Material for compound	27
Figure 3.3: Mixing flow process	29
Figure 3.4: Cutting Process using Water jet Machine	30
Figure 3.6: HAAKE Rheomix OS	32
Figure 3.7: Schematic of a two-roll mill.	34

<b>Figure 3.8:</b> (a) Hot Press Compressing Machine and (b) Sample of mate after hot press	erial 35
Figure 3.9: Standard specification of sample dimension	37
Figure 3.10: Tensile testing machine	37
Figure 3.11: Stress/Strain Diagram(O'Brien 1989)	38
Figure 3.12: Optical Microscope	40
Figure 3.13: Example of microphotograph of tapioca starch (TS)	41
Figure 4.1: (a) Graph for Control sample tensile stress verses tensile st	rain
and(b) Graph for Control sample load verses extension	44
Figure 4.2: (a) Graph for Sample 1 tensile stress verses tensile strain and	d(b)
Graph for Sample 1 load verses extension	45
<b>Figure 4.3:</b> (a) Graph for Sample 2 tensile stress verses tensile strain and Graph for Sample 2 load verses extension	d(b) 47
Figure 4.4: (a) Graph for Sample 3 tensile stress verses tensile strain and	d(b)
Graph for Sample 3 load verses extension	48
Figure 4.5: (a) Graph for Sample 4 tensile stress verses tensile strain and	d(b)
Graph for Sample 4 load verses extension	50
Figure 4.6: (a) Graph for Sample 5 tensile stress verses tensile strain and	d(b)
Graph for Sample 5 load verses extension	51
Figure 4.7: Tensile strength by sample mean	52
Figure 4.8: Tensile extension by sample mean	52
Figure 4.9: Graph for shore hardness	54
Figure 4.10: Control sample (500x)	56
<b>Figure 4.11:</b> Sample 1 (500x)	56

xii

<b>Figure 4.12:</b> Sample 2 (500x)	5	7
<b>Figure 4.14:</b> Sample 4 (500x)	5	8



# LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

A12O3	-	Aluminium Oxide
С	-	Carbon
СВ	-	Carbon Black
СН	-	Carbon
DMA	-	Dynamic Mechanical Analysis
F	-	Fahrenheit
IA	-	Iodine affinities
NR	-	Natural Rubber
OM	-	Optical Microscope
PHR	-	Per Hundred Rubber
RM	-	Ringgit Malaysia
SR	-	Synthetic Rubber
TS	-	Tapioca Starch
U.S	-	United State
UTM	-	Universal Testing Machine
%	-	Percentage
°C	-	Degree Celcius
σ	-	Stress
E	-	Strain

## CHAPTER 1 INTRODUCTION

This chapter will explain the overview of the study and the purpose of this study. The chapter includes the background of the study, problem statement, objectives that is expected to be achieved and the scope of the study that is going to be conducted.

#### 1.0 Background of Study

Rubber is one of the most common types of material used in Malaysia due to their affordable price and ease of use. In Malaysia, the use of rubber in so many applications results in growing volume of rubber products. This proposal is about physical and mechanical properties of natural rubber reinforced carbon black for effect of tapioca starch loading. The effects of different types of fillers on Natural Rubber compounds are been studied, in search of improvements on their physical and mechanical properties. Thus, this study is aimed at the potential use of tapioca starch in Natural Rubber compound.



#### **1.1 Problem Statement**

There is several type of rubber mixture but the new materials are being developed. The research is study the current formulation of tapioca starch to improve the types of filler to mix it with Natural Rubber compounds. In order to get the best formulation on the mixing of Natural Rubber compound (NR) with Tapioca Starch (TS) as a mixing material, some research studies on the physical and mechanical properties of tapioca starch and natural rubber. The best formulation on the mixing of Natural Rubber compound (TS) will be interpreted in the industrial applications.

#### 1.2 Objectives

The general objective of this project is to create a new formula. The objective of the project is to:-

- i. To study the current formulation of Natural Rubber (NR) compoundtapioca starch (TS).
- ii. To prepare compound from natural rubber (NR) compound and tapioca starch(TS).
- iii. To study the effect of the physicalandtensile behaviour of Natural Rubber (NR) compound with Tapioca Starch (TS).

#### 1.3 Scope

This research is to study isfocusingonthephysical and tensile behaviour of Natural Rubber (NR) compound with Tapioca Starch (TS) through meltcompoundingthe mixing material using Haake internal mixer followed by various and mechanical testing such as Tensile test, Shore hardness and Optical Microscope

## CHAPTER 2 INTRODUCTION

### **2.0 Introduction**

This chapter explains about all findings obtained from many literature reviews, which may come from the internet, journals, article and books about the topic related to this study. This section includes findings about the overview of nature rubber, properties of natural rubber, structure and characteristics of natural rubber, sulphur vulcanizing agents, non-sulphur vulcanizing agents, Uncured vs. vulcanized rubber, activator(sulphur vulcanization) and fillers (carbon black).

In the rubber material usage, there are many environment problems arise. With the increase of usage, the rubber product has increased in developed and less developed countries. One of these problems is a dispose of the rubber. The rubber such as tyre disposed in landfills. The rubbers are left there and take years to be decayed. Some of rubber are burn and it make is worse to environment.

Since rubber materials do not decompose easily, disposal of waste rubber is a serious environmental concern. Widespread studies about biodegradation of rubbers have been carried out in order to overcome this environmental problems associated with rubber waste.(Ali Shah et al. 2013)



#### 2.1. History of natural rubber

Rubber is use for balls, containers, and shoes and for waterproofing fabrics by Pre-Columbian peoples of South and Central America. From the early found, rubber did not attract the interest of Europeans. Deep research in finding of rubber solvents and in waterproofing fabrics was done before 1800, but rubber only used for elastic bands and erasers, and these were made by cutting the rubber to make a pieces imported from Brazil. Joseph Priestley is credited with the discovery of the use of rubber as an eraser, thus the name *rubber*.

There is further research, found a practical process for waterproofing fabrics, and in discovered vulcanization process, which revolutionized the rubber industry. After that, the demand for rubber insulation by the electrical industry and the invention of the pneumatic tire extended the demand for rubber.

#### 2.2. Natural rubber

Rubber is produced from natural or synthetic sources. Nature rubber is obtained from the milky white fluid (latex) found in various plants but the only important commercial source of natural rubber is the tree *Hevea brasiliensis*(Figure 2.2). The compounded rubber is sheeted, extruded in special shapes, applied as coating or molded, then vulcanized.



Figure 2.1: Rubber tree (*Hevea brasiliensis*)

Compared to vulcanized rubber, uncured rubber has relatively few uses. It is used for cements; for adhesive, insulating, and friction tapes; and for crepe rubber used in insulating blankets and footwear. Vulcanized rubber, on the other hand, has numerous applications. Resistance to abrasion makes softer kinds of rubber valuable for the treads of vehicle tires and conveyor belts, and makes hard rubber valuable for pump housings and piping used in the handling of abrasive sludge.

The flexibility of rubber is often used in hoses, tires, and rollers for a wide variety of devices ranging from domestic clothes wringers to printing presses; its elasticity makes it suitable for various kinds of shock absorbers and for specialized machinery mountings designed to reduce vibration. Being relatively impermeable to gases, rubber is useful in the manufacture of articles such as air hoses, balloons, balls, and cushions.

The rubber also resistance to water and repel from most of fluid chemicals has led to its use in rainwear, diving gear, and chemical and medicinal tubing, and as a lining for storage tanks, processing equipment, and railroad tank cars. The rubber also electrical resistance, it be use to make a soft rubber goods as insulation and for protective gloves, shoes, and blankets. For the hard rubber is used for articles such as telephone housings, parts for radio sets, meters, and other electrical instruments. The friction of rubber, which is high coefficient on dry surfaces and low coefficient on wet surfaces, make it to the use for power-transmission belting and for waterlubricated bearings in deep-well pumps for a well seal.(Morawetz 2000)

Rubber compounding is the fine art and science of selecting various compounding ingredients and the quantity to blended produce an useful rubber formulation that can be processed, meets or exceeds the customer's last product requirements, and can be competitively priced.

The heart of the rubber compounding is the formula usually referred in the industry as a formula. A generalized rubber formulation is given in Table 2. 1. Rubber formulations are almost never be published by manufactures.(Morawetz 2000)

Ingredient	phr
Crude rubber	100
Filler	50
Softener	5
Antioxidant	1
Stearic acid	1
Zinc oxide	5
Accelerator	1
Sulphur	2
Total	165

 Table 2.1:A generalized rubber recipe

#### 2.2.1. Properties of Natural Rubber

All rubber materials are polymers, which are high molecular weight compounds consisting of long chains of one or more types of molecules, such as monomers. Vulcanization (or curing) produces chemical links between the loosely coiled polymeric chains; elasticity occurs because the chains can be stretched and the crosslinks cause them to spring back when the stress is released. Unvulcanized rubber is soluble in a number of hydrocarbons, including benzene, toluene, gasoline, and lubricating oils.

Rubber is water repellent and resistant to alkalies and weak with acids. Rubber's elasticity, toughness, impermeability, adhesiveness, and electrical resistance make it useful as an adhesive, a coating composition, a fiber, a molding compound, and an electrical insulator. In general, synthetic rubber has the following advantages over natural rubber: better aging and weathering, more resistance to oil, solvents, oxygen, ozone, and certain chemicals, and resilience over a wider temperature range. The advantages of natural rubber are less build up of heat from flexing and greater resistance to tearing when hot.(Bormashenko et al. 2009)

Rubber has unique and chemical properties. Rubber's stress-strain and it is often modelled as hyperelastic. Rubber strain are crystallizes. There is double bond in each of the repeated unit, natural rubber is susceptible to vulcanisation and sensitive to ozone cracking.

The two main solvents for rubber are turpentine and naphtha (petroleum). The solvents are use because rubber does not dissolve easily, the material is finely divided by shredding prior to its immersion.(Hernndez et al. 2015)

An ammonia solution can be used to prevent the coagulation of raw latex while it is being transported from its collection site.Crude rubber is a tough and an elastic solid. It becomes soft and sticky as the temperature rises. Its specific gravity is 0.915.

The most important property of natural rubber is its elasticity. When stretched, it expands and attains its original state, when released. This is due to its coil-like structure. The molecules straighten out when stretched and when released, they coil up again. Therefore applying a stress can easily deform rubber. Note that when this stress is removed, it retains its original shape.

Raw natural rubber has elasticity over a narrow range of temperature from 10 to 60 degrees centigrade made of raw natural rubber don't work well in hot weather.Raw natural rubber also has low tensile strength and abrasion resistant.It absorbs large quantities of water. it is insoluble in water, alcohol, acetone, dilute acids and alkalis but soluble in ether, carbon disulphide, carbon tetrachloride, petrol and turpentine.Pure rubber is a transparent, amorphous solid, which on stretching or prolonged cooling becomes crystalline.

#### 2.2.2. Structure and characteristics of Natural Rubber

Natural rubber is a linear polymer of an unsaturated hydrocarbon called isoprene (2-methyl butadiene). There may be as many as 11,000 to 20,000 isoprene units in a polymer chain of natural rubber as shown in Figure 2.2.

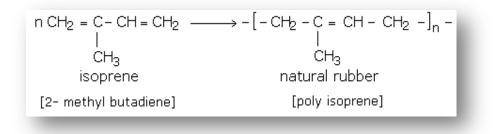


Figure 2.2 Polymer chain of natural rubber

The main characteristics of elastomers, which can make these materials indispensable several industrial sectors, are their elasticity, flexibility, and durability. Beyond these common features, each rubber has it is own unique properties. Even though the processing and last properties of rubber articles are highly dependent on base elastomer, the properties can be extensively altered by appropriate choice of compounding ingredients.

Rubbers can be divided in two types: natural and synthetic. Natural rubber (NR) belongs to the first group, it is known also as cis-1, 4 polyisoprene and is created from the latex of variety of plants in many regions of the world. Even so the most widely used commercial source of NR is the *Hevea brasiliensis* tree. Natural rubber latex is a colloid with a specific gravity of 0.96 to 0.98 and a pH in the range of 6.5 to 7.0. The dispersed phase is mainly rubber and the distribution medium is water. In addition to rubber and water, latex contains small amounts of proteins, resins including fats, fatty acids, other lipids, sterol and sterol esters, carbohydrates and mineral matter. (Sadhan K. De 2001)