



**EFFECT OF CRYOGRINDING ON PARTICLE SIZE OF WASTE
TYRE DERIVED RECLAIMED RUBBER**



**BACHELOR OF MANUFACTURING ENGINEERING
TECHNOLOGY WITH HONOURS**

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**Faculty of Mechanical and Manufacturing Engineering
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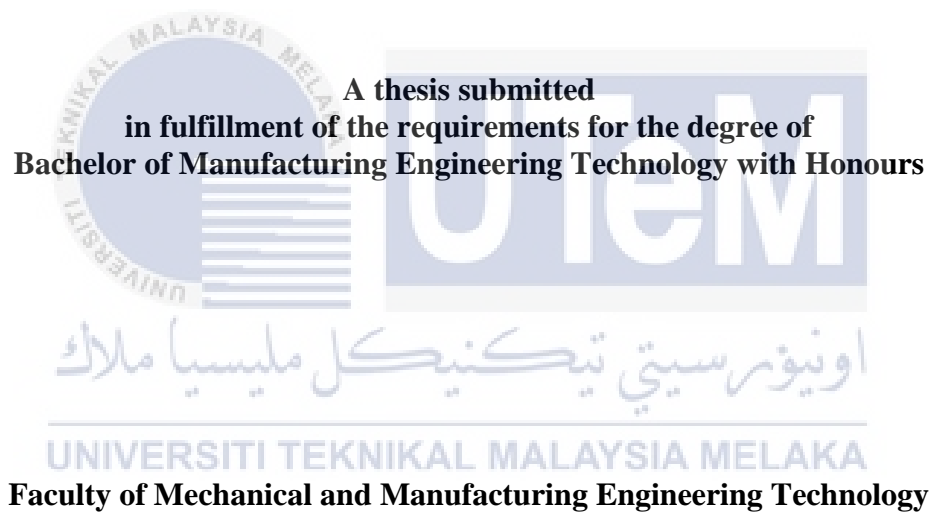
DINESS KUMAR A/L DANABAL

Bachelor of Manufacturing Engineering Technology with Honours

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**EFFECT OF CRYOGRINDING ON PARTICLE SIZE OF WASTE TYRE
DERIVED RECLAIMED RUBBER**

DINESS KUMAR A/L DANABAL



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2022

DECLARATION

I declare that this report entitled “Effect of Cryogrinding On Particle Size Of Waste Tyre Derived Reclaimed Rubber” is the result of my own research except as cited in the references. The choosed title has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

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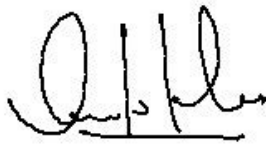
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APPROVAL

I hereby declare that I have checked this thesis and in my opinion, this thesis is adequate in terms of scope and quality for the award of the Bachelor of Manufacturing Engineering Technology with Honours.

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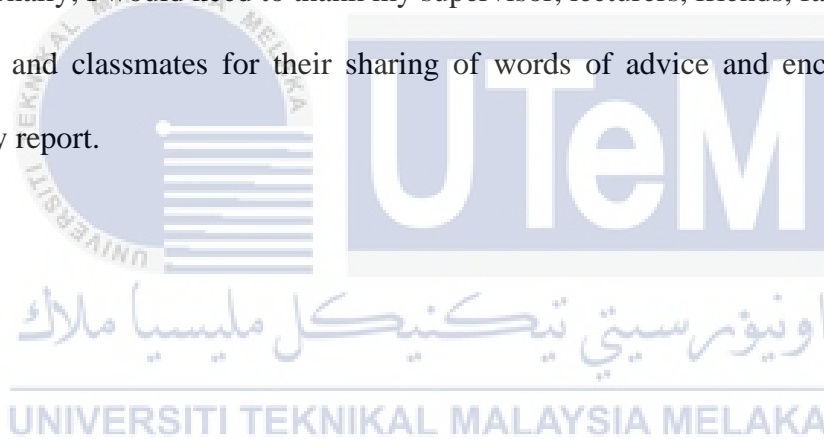
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DEDICATION

This research project study is committed to my beloved parents, who have consistently been a wellspring of motivation and have consistently given good, otherworldly, passionate, and monetary support.

Additionally, I would need to thank my supervisor, lecturers, friends, family members, my mentor, and classmates for their sharing of words of advice and encouragement in finishing my report.



ABSTRACT

Significant growth in the automobile industry boosted tire production contributing to the huge stock piles of waste tires. Retreading of worn rubber tires became an example of remanufacturing and waste dust produced from tire retreading is also reusable, but cannot be used directly in rubber processing due to the presence of impurities, hence it must be reclaimed to remove contaminants using chemical method. The problem statement gearing the research is that blend produced from the reaction between WTRR sheet through chemical separation method with SMR sheet will have deteriorated properties because of the chemical process involved and dimension effect of the sheet. Therefore, cryo-pulverization process was introduced to retrieve the sheet into particle sizes. The research objectives are to study the effect of cryogrinding on the particle size of WTRR and to investigate the effect of cryogrinding on the surface microstructure of WTRR particles. For this research, the WTRR pieces were ground cryogenically into powder form followed by particle characterization using sieve with aperture width of 5mm, 4mm, 3.15mm, 2mm and 1mm. Powder characteristics including the particle size distribution and surface area were explored as well as calculated respectively. Subsequently, the surface microstructure of the WTRR particles were observed through an Optical Nikon Microscope and Scanning Electron Microscope to study the physical texture of the cryoground WTRR particles. The results from the study indicates that grinding the cryogenically-prepared WTRR through commercial blender possess a quite large particles with majority the sieved ground fragment falls in the sieve of 5 mm with weight percentage of 60.56 wt % as well as have low total calculated average surface area of 1.50 mm²/g. Also, the surface microstructure of the cryogenically-prepared particles possessed rough texture with an uneven irregular shape after grinding. All findings present confirms that, although particles produced from the commercial blender is large in size yet this technology as the grinding equipment for the cryogenic technique can be a good choice to modify the smooth surface of the cryogenically-prepared WTRR to be rough.

ABSTRAK

Pertumbuhan yang ketara dalam industri automobil telah meningkatkan pengeluaran tayar yang menyumbang kepada longgokan stok tayar sisa yang besar. Pengilangan semula tayar getah yang haus menjadi contoh pengilangan semula dan habuk sisa yang dihasilkan daripadanya juga boleh diguna semula tetapi tidak boleh digunakan secara langsung dalam pemprosesan getah kerana mempunyai kekotoran dan campuran sisa lain, oleh itu ia mesti dituntut semula untuk membuang bahan kekotoran tersebut menggunakan kaedah kimia. Kenyataan masalah yang menjurus kepada penyelidikan adalah bahawa campuran yang dihasilkan daripada tindak balas antara lembaran WTRR melalui kaedah kimia dengan lembaran SMR akan merosot sifat-sifat kerana proses kimia yang terlibat dan juga sebab kesan dimensi lembaran. Oleh itu, proses pengisaran kriogenik diperkenalkan untuk mengisar helaian ke dalam saiz zarah. Objektif penyelidikan adalah untuk mengkaji kesan pengisaran kriogenik pada saiz zarah WTRR dan untuk menyiasat kesan pengisaran kriogenik pada mikrostruktur permukaan zarah WTRR. Untuk penyelidikan ini, kepingan WTRR akan dikisar menggunakan proses kriogenik dalam bentuk serbuk diikuti dengan penapisan zarah menggunakan lebar saiz 5mm, 4mm, 3.15mm, 2mm dan 1mm. Ciri-ciri serbuk termasuk taburan saiz zarah dan kawasan permukaan telah diterokai serta dikira masing-masing. Seterusnya, mikrostruktur permukaan zarah WTRR diperhatikan melalui Mikroskop Nikon Optik dan Mikroskop Elektron Pengimbas untuk mengkaji tekstur fizikal zarah WTRR yang telah dikisar dari proses kriogenik. Hasil kajian menunjukkan bahawa pengisaran WTRR yang disediakan oleh kriogenik melalui pengisar komersial mempunyai zarah yang agak besar dengan majoriti zarah kasar yang disedut jatuh dalam penapis 5 mm dengan peratusan berat 60.56 wt % serta mempunyai kawasan permukaan dikira purata rendah 1.50 mm² / g. Juga, mikrostruktur permukaan zarah-zarah yang disediakan oleh kriogenik menimbulkan tekstur kasar dengan bentuk yang tidak teratur sekata selepas pengisaran. Semua penemuan yang hadir dalam laporan, mengesahkan bahawa, walaupun zarah yang dihasilkan dari pengisar komersial adalah besar dalam saiz namun teknologi ini sebagai peralatan pengisaran untuk teknik kriogenik boleh menjadi pilihan yang baik untuk mengubah suai permukaan licin WTRR yang disediakan oleh kriogenik menjadi kasar.

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Finally, I hope this thesis will benefit the readers.

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TABLE OF CONTENTS

	PAGE
DECLARATION	
APPROVAL	
DEDICATION	
ABSTRACT	i
ABSTRAK	ii
ACKNOWLEDGEMENTS	iii
TABLE OF CONTENTS	iv
LIST OF TABLES	vi
LIST OF FIGURES	viii
LIST OF SYMBOLS AND ABBREVIATIONS	x
LIST OF APPENDICES	xi
CHAPTER 1 INTRODUCTION	1
1.1 Background	1
1.2 Problem Statement	2
1.3 Research Objectives	3
1.4 Scope of Research	3
CHAPTER 2 LITERATURE REVIEW	5
2.1 Introduction	5
2.2 Vulcanisation of rubber	5
2.3 Reclaiming technology	7
2.3.1 Physical processes	7
2.3.2 Chemical process	9
2.4 Pulverization technology of waste rubber	11
2.4.1 Grinding technology	11
2.4.2 Extrusion method	12
2.4.3 Water jet technology	14
2.4.4 Sieving process	16
2.5 Raw materials & Additives	18
2.5.1 Natural rubber (NR)	18
2.5.2 Carbon black	19
2.5.3 Processing oil	20
2.5.4 Liquid nitrogen	22
2.5.5 Sulfur and accelerators	23

2.5.6	Conclusion	26
2.6	Characterization Technique	27
2.6.1	Fourier transform infrared spectroscopy (FTIR)	27
2.6.2	Tensile Stress-Strain	28
2.6.3	Hardness	29
2.6.4	Cure characteristics	30
2.6.5	Scanning Electron Microscopy (SEM)	32
2.7	Sample preparation of rubber product	34
2.7.1	Compounding process	34
2.7.2	Mixing	34
2.7.3	Shaping	35
2.8	Summary	39
CHAPTER 3 METHODOLOGY		44
3.1	Introduction	44
3.2	Raw Materials	44
3.2.1	Waste Tyre Derived Reclaimed Rubber (WTRR)	45
3.2.2	Liquid Nitrogen	46
3.3	Research methodology	46
3.4	Knife slicing of WTRR sheet	48
3.5	Waste rubber pieces pulverization	48
3.6	Sieving and particle characterization	49
3.7	Specific surface area of the characterized particles	49
3.8	Structure microstructure of the cryo-pulverized characterized particles	50
CHAPTER 4 RESULTS AND DISCUSSION		52
4.1	Introduction	52
4.2	Particle size distribution of cryo-pulverized waste rubber	52
4.3	Calculated average surface area of WTRR powder from cryo-pulverization	57
4.4	Surface microstructure of the cryo-pulverized particle	59
4.5	Summary	64
CHAPTER 5 CONCLUSIONS AND RECOMMENDATIONS		66
5.1	Conclusions	66
5.2	Recommendations	67
REFERENCES		68
APPENDICES		76

LIST OF TABLES

TABLE	TITLE	PAGE
Table 2.1	Functions of the three zones of system (Wang et al, 2017).	14
Table 2.2	Specifications of the technically graded natural rubber (Mark, 2013).	19
Table 2.3	Reference price for Standard Malaysian Rubber (Lembaga Getah Malaysia, 2021).	19
Table 2.4	Description of processing oil (Mark, 2013).	21
Table 2.5	Three types of oils used in the rubber sector have different physical characteristics (Mark, 2013).	21
Table 2.6	Guide on choose the right production oil for a variety of commercial elastomers (Mark, 2013).	22
Table 2.7	Types of accelerators used for sulfur vulcanization (Mark, 2013).	25
Table 2.8	Sulfur and accelerator dosages and ratios (Arvind Mafatlal Group, 2021).	25
Table 2.9	Rubber Property produced using Conventional (CV), Semi efficient (SV) and Efficient (EV) system (Akrochem Corporation, 2021).	26
Table 2.10	Mechanical properties of Natural Rubber (NR) and Ground Tire Rubber (GTR) composites (Wang et al, 2017).	33
Table 2.11	Strength and weaknesses of the pulverization technology.	43
Table 3.1	Chemical composition of the reclaimed WTRR (TYREC M-1) (JYRR Sdn Bhd).	45
Table 3.2	Physical properties of the reclaimed waste rubber sheet (TYREC M-1) (JYRR Sdn Bhd).	45

Table 4.1	Mass and weight percentage of the characterized cryo-pulverized WTRR particles.	54
Table 4.2	Factors that affects the size of the rubber particles after grinding cryogenically.	56
Table 4.3	Calculated average surface area of the characterized cryo-pulverized WTRR particles.	58
Table 4.4	Microscopic image of pulverized WTRR particle focusing on the surface.	61



LIST OF FIGURES

FIGURE	TITLE	PAGE
Figure 2.1	Addition of sulphur into cross-links (Ramnath, 2017).	6
Figure 2.2	Chemical structure of natural rubber (a) before and after vulcanization (b) (Rowhani et al, 2016).	6
Figure 2.3	Sulfides and mercaptans used in rubber reclaiming (Bockstal et al., 2019).	10
Figure 2.4	Reactions of the cross-link under devulcanization processes involving organic disulfides and mercaptans (Bockstal et al., 2019).	10
Figure 2.5	Diagram of ambient grinding (Mark, 2013).	12
Figure 2.6	Diagram of cryogenic grinding (Mark, 2013).	12
Figure 2.7	Diagram of a single screw extruder for rubber pulverisation (a) and the shape of the screw channel with changeable depth (b) (Mark, 2013).	13
Figure 2.8	Diagram of water jet system and the processing parameters involved to pulverize waste rubber (Wang et al, 2017).	15
Figure 2.9	Examples of mesh screen sizes (Scrap News Tire, 2021).	17
Figure 2.10	Example of sieving process separating the fine particles from the coarse particles (Scrap News Tire, 2021).	17
Figure 2.11	History of vulcanization by sulfur (Mark, 2013).	24
Figure 2.12	Example of total reflectance attenuated the spectra of waste tire rubber and Ground Tire Rubber (GTR) from water jet pulverization in Fourier Transform Infrared Spectroscopy (FTIR) (Wang et al, 2017).	27

Figure 2.13 Tensile stress strain curve for natural rubber (red curve) (Monadjemi et al, 2016).	29
Figure 2.14 Cure meter assembly (ASTM D2084-11).	31
Figure 2.15 Example of types of cure curve (ASTM D2084-11).	31
Figure 2.16 Rotorless oscillating shear rheometer diagram (ASTM D6204-99).	32
Figure 2.17 Scanning electron microscope (SEM) images of tensile fracture surfaces of Natural Rubber (NR) and Ground Tire Rubber (GTR) composites under different conditions: (A) Mix B, (B) Mix C, (C) Mix D, (D) Mix E, and (E) Mix F (Wang et al, 2017).	33
Figure 2.18 Rotors of internal mixer (Amos et al, 2015).	35
Figure 2.19 Schematic diagram of single screw extruders (Crowley, 2007).	36
Figure 2.20 Rubberizing textiles (a), and profiling with a four-roll engraving calendar (Mark, 2013).	37
Figure 2.21 The compression mold containing a rubber blank; before closing the molding (a) and after closing the molding (b) (RPM Industries, 2002).	38
Figure 3.1 Flow chart of methodology.	47
Figure 3.2 Scanning Electron Microscope (Model: ZEISS EVO-18).	51
Figure 4.1 Particle size distribution of cryogenically pulverized waste rubber.	55
Figure 4.2 Calculated surface area of WTRR powder from cryo-pulverization.	58
Figure 4.3 SEM micrographs of the surface microstructure of characterized particles; (a) before grinding, after grinding; (b) > 5mm, (c) 5mm – 4mm, (d) 4mm – 3.15mm, (e) 3.15mm – 2mm, (f) 2mm – 1mm.	63

LIST OF SYMBOLS AND ABBREVIATIONS

D,d	-	Diameter
ASTM	-	American Society For Testing and Materials
NR	-	Natural Rubber
WR	-	Waste Rubber
WTRR	-	Waste Tire derived Reclaimed Rubber
wt%	-	Weight Percentage
rpm	-	Revolution Per Minute
°C	-	Degree Celcius
SEM	-	Scanning Electron Microscope
mm	-	Millimeter
SMR	-	Standard Malaysian Rubber
%	-	Percentage
phr	-	Parts per Hundred Rubber



LIST OF APPENDICES

APPENDIX	TITLE	PAGE
APPENDIX A	Space Diagonal Calculation Of Particles Sized Greater Than 5mm	74
APPENDIX B	Surface Area Calculation Of The Characterized Particles	76
APPENDIX C	Gantt Chart Of FYP 1 & 2	78



CHAPTER 1

INTRODUCTION

1.1 Background

In the last few years, the significant growth of the automobile industry and the step up in the use of cars have tremendously boost the tire production. This resulted in massive waste tire stockpiles (Fazli et al, 2020). Malaysia's yearly waste tire production has been steadily increasing. Furthermore, scrap or waste tires in Malaysia are projected to be 8.2 million, or 57,391 tones, however around 60 percent of total accounts of waste tires are thrown in unknown ways (Kumar, 2006). Due to their large volume, discarded tires take up more space than other types of trash. Furthermore, because scrap tires are unlikely to be decompose, burying them shortens the life of the burial and landfill locations or facilities (Nuzaimah et al., 2018). Additionally, waste tires which is exposed might accumulate water that may breed bacteria, molds, insects or mice which might bring harmful diseases (Adhikari et al, 2018).

In recent years, retreading of worn rubber tires became a very particular example of remanufacturing since it is highly environment friendly. Besides, adding new tread to a used tires brings both environmental and economic benefit (Qiang et al, 2020; Fazli et al, 2020). Although there are many advantages of retreading the worn tires, on the other hand still there are some disadvantages too. One of the major concerns of retreading worn tires is the waste dust produced from this process. Even though, waste dust from tires is reusable and cheap comparing synthetic material in the processing of rubber, but efforts taken to reuse is still not being encouraged widely in the country. Moreover, tire dust produced from retreading cannot be reused directly since it contains impurities. Hence, in order to remove the

contaminants such as oil, metal dust and as such from the tire dust, chemical separation method was introduced as the technique to reclaim (Bockstal et al., 2019; Sun et al., 2007).

1.2 Problem Statement

Over the last few decades, WTRR became an alternative raw material for tire manufacturing over synthetic rubber in terms of its reasonable price and availability (Mente et al., 2016). Although waste dust produced from tire retreading is reusable, but it cannot be directly used in rubber processing since the dust contains impurities such as oil and metal dust. So to recover, separate and remove the contaminants from the dust, the chemical separation method was introduced as the reclaiming technique popularly in the rubber processing factories (Bockstal et al., 2019; Lapkovskis et al., 2020).

Chemical separation is a reclaiming process where the dust reacts with the chemical to produce the waste dust in the form of a solid sheet which is called as Waste Tyre Derived Reclaimed Rubber, WTRR (Bockstal et al., 2019; Fukumori et al, 2004). However, due to the presence of chemical and dimension effects, this WTRR sheet cannot be incorporated straightaway with the SMR sheet since it would deteriorate the mechanical characteristics of the produced blend (Adhikari et al, 2018; Bockstal et al., 2019). Consequently, the WTRR sheet needs to be reduced into particle sizes prior to any practical application of mixing with SMR.

Therefore, to reduce the chemically reclaimed WTRR sheet into particle sizes, pulverization process was employed before the pragmatic implementation of blending the WTRR powder with the SMR sheet. For this research, the WTRR sheet will be pulverized cryogenically to study the effect of cryogrinding on the particle size of WTRR using a commercial blender.

1.3 Research Objectives

The objectives of the research are as following:

- 1) To study the effect of cryogrinding on the particle size of WTRR.
- 2) To investigate the effect of cryogrinding on the surface microstructure of WTRR particles.

1.4 Scope of Research

The scope of this research will be focused on the effect of cryogrinding on the particle size of WTRR. The raw material that is used for this research is Waste Tyre Derived Reclaimed Rubber (WTRR) sheet produced from chemical separation and liquid nitrogen as a freezing agent to make the WTRR pieces brittle. The process flow of this study was splitted into four stages which are the knife slicing of the WTRR sheet into smaller pieces, cryo-pulverization process of WTRR, particle characterization process and surface microstructure study of the WTRR particles. The WTRR sheet was reduced into smaller pieces using a knife before immersing into liquid nitrogen to ease the grinding process. Pulverization process is the breaking down of WTRR pieces into powder form which is introduced before any practical application such as sieving. Since the properties of WTRR is elastic, liquid nitrogen was used to freeze the rubber which will be then ground using a commercial blender to obtain fine-sized particles with rough surface (Adhikari et al, 2018). Powder characteristics, including particle size distribution and surface area will be analyzed and studied here.

Besides, the scope of the study is also being focused on the effect of the cryogrinding on the surface microstructure of WTRR particles. For this study, the surface microstructure of the characterized WTRR particles was observed through the Microscope Nikon 100 and Scanning Electron Microscope (Model: ZEISS EVO-18) to discover the physical shape and

to explore the surface texture morphology of the cryoground WTRR powders respectively. Each samples of the characterized WTRR powder was randomly chosen from the range > 5 mm, 4 mm to 5 mm, 3.15 mm to 4 mm, 2 mm to 3.15 mm, 1 mm to 2 mm and < 1 mm and was sputter coated to dismiss the presence of electrostatic charge to obtain good SEM textural image before placing the WTRR particles under the Scanning Electron Microscope instrument.



CHAPTER 2

LITERATURE REVIEW

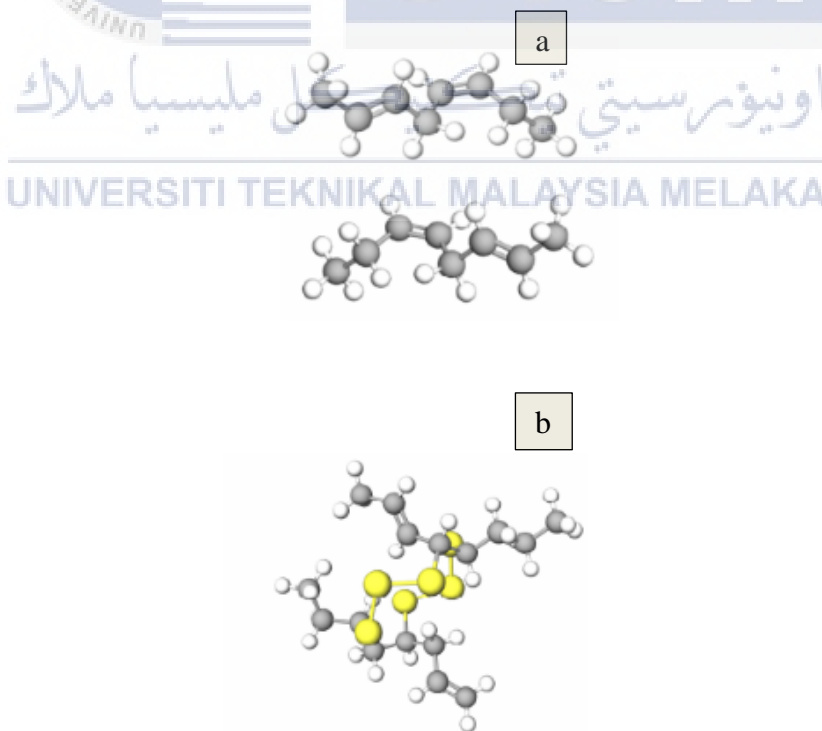
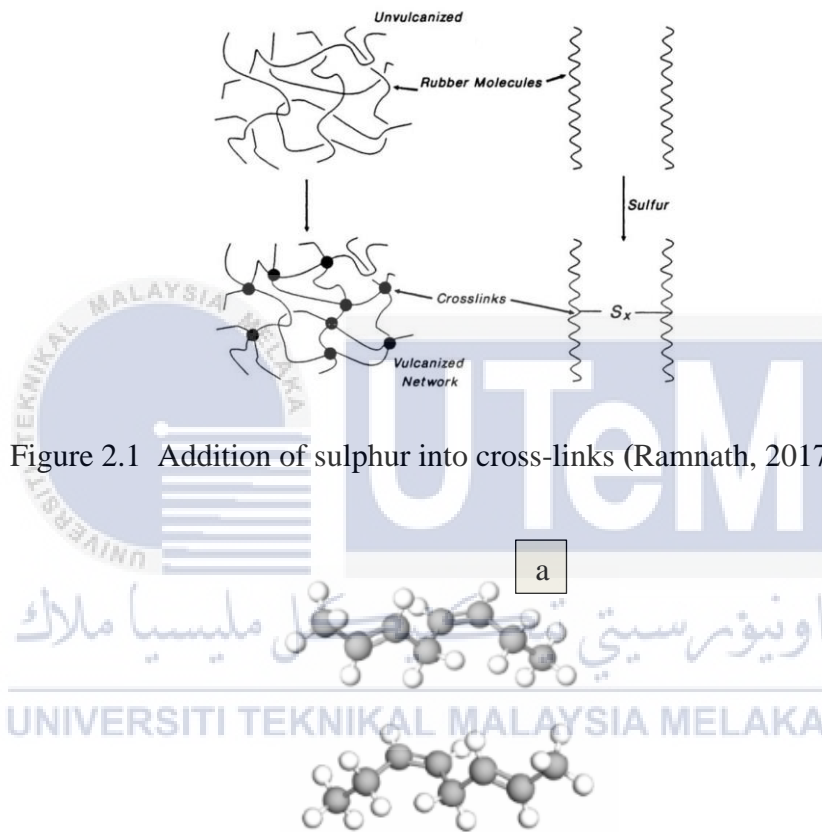
2.1 Introduction

The primary goal of this chapter is to review relevant literature, scholarly articles, and other related sources that discuss rubber vulcanisation, rubber reclaiming technologies, raw materials required for the study, sample preparation of the rubber product, and required characterization techniques. There are seven sections in this chapter. The introduction to this chapter is presented in Section 2.1, while Section 2.2 discusses the rubber vulcanization process. Besides, Section 2.3 and 2.4 summarizes about the reclaiming technology of waste rubber and pulverization techniques involved to breakdown rubber particles into smaller size respectively. Raw materials is considered to be a basic and essential substance needed to mix, fabricate and produce rubber products, Section 2.5 discusses and describes about the raw material used for this study. Meanwhile, Section 2.6 and 2.7 of this chapter focused mainly on the characterization techniques involved and also about the sample preparations of rubber products respectively. The sections presented for this chapter provides a better clarity and knowledge in depth relevant to the research topic.

2.2 Vulcanisation of rubber

Vulcanization of rubber is a process of improving the strength and elasticity which was invented by Charles Goodyear in 1839. In other terms, vulcanisation is a chemical process that involves the addition of sulphur or other chemicals to transform polymers into

more durable materials. The addition of sulphur modifies the polymer by forming cross-links in between individual polymer chains (Ramnath, 2017). Figure 2.1 illustrates the addition of sulphur into the cross-links while Figure 2.2 presents the schematic molecule structure of natural and vulcanised rubber clearly. The yellow molecules in Figure 2.2 represents the cross chain which was formed by the sulphur atoms (Rowhani et al, 2016).



2.3 Reclaiming technology

Rubber waste reclaiming has become a technique in which recycled rubber materials, such as waste tyre rubber or any other type of vulcanised rubber waste, are converted into a condition that may be integrated, reprocessed, and vulcanised utilising mechanical and chemical methods using various reclaiming agents. Reclaimed rubber have been widely used in the compounding of tire to lower the cost, improve processing and fatigue life (Dick., 2020; Mark, 2013). Furthermore, utilization of the reclaimed rubber in producing the rubber products, saves as well as minimizes the need of virgin rubber, carbon black and other softeners (Lapkovskis et al., 2020). The principle of this technology is devulcanization. Since waste rubber is very hard to recycle due to the presence of bonding of the three dimensional crosslink chain by sulfur atoms formed during vulcanization, this technology was introduced to break the bonding in order to reclaim the rubber (Lapkovskis et al., 2020; Mark, 2013). Physical and chemical reclaiming technologies are the two types of reclaiming renown technology in this industry.



2.3.1 Physical processes

Physical reclamation is a technique that involves breaking the three-dimensional (3D) structure of crosslinked rubber using various external energy sources. Due to network breaking, waste rubbers are reduced to low molecular weight fragments, which are then blended with virgin rubber for new uses. Physical reclaiming process can be split into many subprocess, this includes the mechanochemical method and mechanical method (Bockstal et al., 2019).