

EFFECT OF CRYOGRINDING ON SURFACE PROPERTIES OF WASTE TYRE DERIVED RECLAIMED RUBBER



BACHELOR OF MANUFACTURING ENGINEERING TECHNOLOGY (PROCESS AND TECHNOLOGY) WITH HONOURS

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Bachelor of Manufacturing Engineering Technology (Process and Technology) with Honours

EFFECT OF CRYOGRINDING ON SURFACE PROPERTIES OF WASTE TYRE DERIVED RECLAIMED RUBBER

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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

2022

DECLARATION

I declare that this thesis entitled "Effect Of Cryogrinding On Surface Properties Of Waste Tyre Derived Reclaimed Rubber." is the result of my own research except as cited in the references. The thesis has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.



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 (Process and Technology) with Honours.

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DEDICATION

То

My beloved parents and sister

A strong and emotional support, encouragement for me to not to give up in my completion

of this study.

My supervisor, Professor Madya Ts. Dr. Lau kok Tee

Who patiently guides me and gives me valuable knowledge.

My teammates and friends

Who giving support and accompanied me in the completion of this study.

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ABSTRACT

Rubber is a viscous and elastic elastomer. Natural rubber is an elastomer with excellent elasticity, durability and damping. It's inferior properties can be enhanced by compounding with other polymers or adding additives. However, an increase in waste tyre rubber disposal in landfills has impacted air pollution. In fact, cryogenic grinding is adopted for waste tyre rubber processing. Also, rising demand for natural rubber has led to depletion. Then, waste tyre rubber is introduced as a raw material into natural rubber compound, cutting the final product cost. Prior to grinding, reclaiming technology is employed to process waste tyre rubber. However, reclaimed rubber is non-polar. To clarify, one of the purpose of this study is to investigate the effect of the reclaiming technique to the chemical structure of waste tyre derived reclaimed rubber (WTRR). Following that, pulverization affects the characteristics of reclaimed rubber itself. Hence, the effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size was studied. As reported that WTRR has a weak polar hydroxyl bond (O-H) as determined by Fourier Transform Infrared Spectroscopy (FTIR). Moreover, the intensity of WTRR's peak after cryogenic grinding decreased with the increasing of particle size. On the other hand, Scanning Electron Microscope (SEM) analysis showed a smooth surface of WTRR after cryogenic grinding. Thermogravimetric analysis (TGA) indicated that oxygen molecule in O-H bonding affects the thermal stability of WTRR. To sum, cryoground WTRR may not be feasible alternative in rubber compounding. Properties of cryoground WTRR should be improved further.

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ABSTRAK

Getah ialah elastomer likat dan elastik. Getah asli ialah elastomer dengan keanjalan, ketahanan dan redaman yang baik. Sifat inferiornya boleh dipertingkatkan dengan menggabungkan dengan polimer lain atau menambah bahan tambahan. Walau bagaimanapun, peningkatan dalam pembuangan getah tayar sisa di tapak pelupusan telah memberi kesan kepada pencemaran udara. Malah, pengisaran kriogenik digunakan untuk pemprosesan getah tayar sisa. Selain itu, peningkatan permintaan untuk getah asli telah menyebabkan kehabisan. Kemudian, getah tayar sisa diperkenalkan sebagai bahan mentah ke dalam sebatian getah asli, mengurangkan kos produk akhir. Sebelum mengisar, teknologi tebus guna digunakan untuk memproses getah tayar sisa. Bagaimanapun, getah tebus guna adalah bukan kutub. Untuk memperjelas, salah satu tujuan kajian ini adalah untuk menviasat kesan teknik tebus guna terhadap struktur kimia tavar buangan getah tebus guna (WTRR). Berikutan itu, penumbuk menjejaskan ciri-ciri getah tebus guna itu sendiri. Oleh itu, kesan pengisaran krio ke atas sifat permukaan tayar buangan getah tebus guna (WTRR) dalam saiz zarah yang berbeza telah dikaji. Seperti yang dilaporkan bahawa WTRR mempunyai ikatan hidroksil polar lemah (O-H) seperti yang ditentukan oleh Fourier Transform Infrared Spectroscopy (FTIR). Selain itu, keamatan puncak WTRR selepas pengisaran kriogenik menurun dengan peningkatan saiz zarah. Sebaliknya, analisis Scanning Electron Microscope (SEM) menunjukkan permukaan licin WTRR selepas pengisaran kriogenik. Analisis termogravimetrik (TGA) menunjukkan bahawa molekul oksigen dalam ikatan O-H mempengaruhi kestabilan terma WTRR. Kesimpulannya, tanah krio mungkin bukan alternatif yang boleh dilaksanakan dalam pengkompaunan getah. Sifatsifat cryoground WTRR perlu dipertingkatkan lagi.

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LIST OF SYMBOLS AND ABBREVIATIONS

Ground Tyre Rubber GTR _ Waste Tyre Derived Reclaimed Rubber WTRR Waste Tyre Crumb Rubber WTCR _ Fourier Transform Infrared Spectroscopy FTIR _ SEM Scanning Electron Microscopic -TGA Thermogravimetric analysis -



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CHAPTER 1

INTRODUCTION

1.1 Background

Rubber is a kind of unique material with elasticity and viscosity. Rubber is also referred to as elastomer. Elastomers can be categorized into natural and synthetic elastomers. In the manufacturing process, rubber can be processed into various shapes such as compression molding, extrusion and calendering. It can be compounded to have widely varying properties. In addition, mechanical properties of rubber such as hardness, tensile strength and modulus under set strain are always the primary consideration when designing a rubber formula for specific purpose. Therefore, rubber is widely used in human life. Most common applications are tires, automobile supply industry, constructions, textiles and biomedical applications.

Natural rubber, also known as virgin rubber, mainly has resilience, damping performance and good elasticity. However, it does have some imperfections such as low resistance to organic solvent and low heat resistance. Since then, natural rubber need to be chemically modified so that it can enhance the performance and expand the applications of other rubber materials by blending with other polymers or adding additives. Besides, natural rubber is also vulcanized and used in most applications by forming a three-dimensional network structures, which is said to transmit stress almost constantly among the rubber chains. From that, the requirements of natural rubber are getting higher and more attractive (Mente, Motaung and Hlangothi, 2016). In 2020, the global natural rubber market is valued

to be closed to 40 billion US dollars, and it is predicted by analysis that the rubber market value will be almost 68.5 billion US dollars in 2026 (CNBC, 2021).

It is estimated that more than 1.2 billion new tyres are produced every year in the world and disposed of at the end of the tyre life. Especially in Europe and the United States, 245 million and 270 million waste tyres have been discarded in landfills (Mushunje, Otieno and Ballim, 2018). It is reported that among the billions of waste tyres thrown into the environment around the world, about 1000 million waste tyres were produced by the passenger vehicles (Khed, Mohammed and Nuruddin, 2018). The lack of citizen's environmental awareness makes the problems of dealing with waste rubber tyres more serious. Buried in landfills and illegal garbage dumps are among common, cheapest and easiest method of disposing waste rubber tyre. The poor disposal of rubber tyres lead to the release of toxic chemicals, and mosquitoes breed in stranded water of disposed tyre. Thereafter, this issue has become a likely hazard to human health worldwide or even the living being and decrease quality of environment (Waste Rubber, 2019).

With the vulcanization process, the formation of three-dimensional network makes the rubber a kind of non-biodegradable material, which can resist many external factors. On account of the complex 3D crosslinked structure and the presence of a high number of different additives inside a tyre formulation, there are many processing method are being carried out to address the issue disposal of waste tyre (Fazli and Rodrigue, 2020). For example, waste tyre can be finely shredding using grinding process. Grinding process is the most common method to manage waste rubber tyres. It includes ambient grinding, wet grinding and cryogenic grinding (Mohajerani et al., 2020). This technique produces ground tire rubber (GTR) , which can be used as a substitute material in natural rubber compounds partially or producing more environmentally friendly composite materials. Through this, discarded tires should no longer be regarded as a pollutant and useless wastes, but rather brings a positive influence on the properties performance of rubber.

1.2 Problem Statement

Malaysia is a major producer of natural rubber in Southeast Asia. According to reports, the amount of rubber consumed for natural rubber goods increased by 2% annually in 2018. As a result of the increased demand for natural rubber, there may be a scarcity of raw material in the future. Natural rubber is also pricey in terms of cost. Subsequently, most rubber manufacturers are experimenting with using waste tyre rubber as a raw resource to partially replace and blend with natural rubber (Husna and Azura, 2020).

Thereafter, the introduction of waste tyre rubber able to reduce the cost of the final product (Zedler et al., 2020). The utilization of waste tyre rubber in the rubber compound has potentially manufactured products like floor mats, tread of passenger car, light truck. Therefore, this strategy has allowed sustainable industrial application of tyre recycling technologies. One of the tyre recycling technologies was reclaiming technology, also called as devulcanization. According to the study by Kenawy and Khalil (2021), the authors declared that waste tyre rubber is advocated to be devulcanized before being merged with others rubber compound. However, according to a prior study, pure reclaimed rubber is essentially non-polar (Saeed and Khattab, 2021). A polar group is critical in aiding of interfacial adhesion between two polymers (Kenawy and Khalil, 2021). In order to verify this statement, this study would like to investigate the reclaiming technology to the chemical structure of waste tyre derived reclaimed rubber.

Futhermore, the waste tyre rubber could have a good influence on the properties of rubber compound by adopting appropriate waste rubber processing method. As the waste rubber processing method is one of the key factors affecting the properties of rubber compounds, chemical separation method is employed to remove the contaminants such as metal dust and oil in the waste rubber. It is then grinded to reduce the size of waste rubber dust sheet created by chemical separation, commonly also known as pulverization. A rubber compound containing ground tyre rubber with a smaller particle size will have superior mechanical properties than the one with bigger particles (Hrdlička et al., 2021). Therefore, this research would like to study the effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size.

1.3 Research Objective

The objectives of this research as follows:

- a) To investigate the effect of the reclaiming technology to the chemical structure of waste tyre derived reclaimed rubber (WTRR).
- b) To study the effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size.

1.4 Scope of Research

In general, the scope of research is to investigate the effect of the reclaiming technology to the chemical structure of waste tyre derived reclaimed rubber (WTRR) and to study the effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size. This study's process flow is divided into 5 steps, including raw material preparation, sample preparation and sample characterization.

The raw material used is the pulverized chemical separation (CS)-grade waste rubber, also called waste tyre derived reclaimed rubber (WTRR). The cost price of waste tyre derived reclaimed rubber (WTRR) dust produced by chemical separation method is about RM 3.00/kg.

First of all, WTRR is immersed and cooled in liquid nitrogen approximately -196°C. The embrittled WTRR is further ground by a blender to obtain smaller particles. Then, the pulverized WTRR is mechanically sieved by siever shaker. 6 groups of samples will be prepared for the characterization of the samples. These 6 samples include 1 sample taken from WTRR before cryogrinding and 5 samples taken from different particle size, including 1.0 - 2.0 mm, 2.0 - 3.15mm, 3.15 - 4.0mm, 4.0 - 5.0mm and > 5.0mm.

The types of characterization carried out on the prepared samples are surface properties, morphological properties and thermogravimetric analysis.

The surface properties of WTRR before and after the cryogrinding are studied by Fourier Transform Infrared Spectroscopy (FTIR) in the range of 400 to 4000 cm⁻¹; the morphological characteristics of WTRR are observed by Scanning Electron Microscope (SEM) at an accelerating voltage of 15kv; and its thermal analysis is characterized by Thermogravimetric Analyzer (TGA), with heating rate at 10 cel/min.



CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In today's world, waste rubber can be a significant raw material added to the rubber compounds. By varying its particle size, resulting in different properties of rubber compound. Therefore, this chapter will provide an overview of the previous research on recycling method of waste rubber from waste rubber tyre, so as to effectively reuse them. In order to understand the properties of waste rubber more clearly, this chapter will also be introducing the pulverization and blending technologies, additives and sample preparation as well. The knowledge on characterization properties of natural rubber as well as blending of waste rubber with natural rubber are reviewed as they are mostly similar to the properties of waste rubber.

2.2 Natural Rubber

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Natural rubber (NR) comes from a kind of tree, which is called *Heavea tree*. It is a kind of milk-like liquid extracted from the tree. This milk-like liquid, called latex, is obtain by tapping the inner skin layer of the tree and collecting it in a cup. Natural rubber is the name of polymer 1,4 cis-polyisoprene. Natural rubber has inherent superior properties such as high abrasion temperature, good tensile strength and elastic reistance (Zhao et al., 2019). As a result, it is utilised as one of the components in the production of rubber tyres for passenger cars, trucks and off-road vehicles. The composition of various tyres is listed below (Formela, 2021):

Composition	Passenger car tyre	Truck tyre	Off-road tyre
NR (%)	22	30	47
SBR (%)	23	15	-
Carbon black (%)	28	20	22
Other additives (eg:			
curing agents,	14	10	19
textiles) (%)			
Steel (%)	13	25	12

Table 2.1 Composition of passenger, truck and off-road vehicles (Formela, 2021).

2.2.1 Chemical Properties of natural rubber

In general, natural rubber latex contains different percentages of compounds. Containing 30 - 40 weight percent wt% of rubber, 1.0 - 1.5 wt% of proteins, 1.5 - 3.0 % of resins, 0.7 - 0.9% of minerals, 0.8 - 0.1% of carbohydrates and 55 - 60 wt% of water. Components of natural rubber latex are shown in the following table.

ble 2.2 Composition of natural rubber latex (Dinsmore, 20				
يسيا ملات	Constituent	Content, %	اويو	
	Rubber	30-40	ΔΚΔ	
UNIVERSITI	Proteins	1.0 - 1.5		
	Resins	1.5 - 3.0		
	Minerals	0.7 - 0.9		
	Carbohydrates	0.8 - 0.1		
	Water	55 - 60		

Table 2.2 Composition of natural rubber latex (Dinsmore, 2007).

The natural rubber latex is further processed into coagulum by diluting it with formic acid or acetic acid or by aiding of electrophoreseis. The coagulum is formed by separating the raw natural rubber from the water. In common, coagulum is used in natural rubber grades which have less stringent requirements with respect to its great degree of contamination. Conventionally, a Technically specified natural rubber (TSR) are divided into different grades in accordance with the technical specifications stated by the International Standards Organisation (ISO). According to the TSR quality, coagulum can be used in different derived form include collecting cups (cup lump), latex coagulated by adding acid or rubber sheets. In 1965, Malaysia introduced the Standard Malaysia Rubber (SMR). The grading of SMR is based on dirt content, and the general types of SMR that is available in the market include (Service, n.d.):

Table 2.3 The quality standard of different general types of SMR grades natural rubber (Service, n.d.).

t contont			8
i content	content	matter	
(max %)	(max %)	(max %)	
0.60	0.60	0.80	Pale green
AYSIA			
0.75	0.60	0.80	Brown
E			
1.00	0.60	0.80	red
•			
(6) (max %) 0.60 0.75 1.00	6) (max %) (max %) 0.60 0.60 0.75 0.60 1.00 0.60	6) (max %) (max %) 0.60 0.60 0.80 0.75 0.60 0.80 1.00 0.60 0.80

2.2.2 Physical Properties of natural rubber

Natural Rubber is a very versatile material, which has been used in various industrial sectors for many years, and has great market acceptability. This is due to its outstanding physical properties. All physical properties are affected by its flexible, loosely connected and long polymer chains. Generally, the density is ranging from 920 to 930 kg/m^3. Below glass transition temperature of about -70 °C, natural rubber can still give its elastic behavior due to the reattachment between chains. The stress-strain performance of natural rubber is the best of all the elastomeric polymers. The ultimate elongation of natural rubber is up to 700%. However, its functional groups are easy to react with oxygen and ozone, which reduces the aging resistance. In addition, its oil resistance is poor, and it will swell and disintergrate after absorbing the oil (Sisanth et al., 2017).

2.2.3 Structure of natural rubber

As a kind of polymer material, the structure of natural rubber can be divided into two categories: macrostructure and microstructure. Macrostructure refers to the molecular weight distribution of each polymer molecules. On the other hand, microstructure indicates the way distribution of single monomer units along the chain and their geometric distribution (Miller, 2018).

In terms of macrostructure, the molecular weight distribution of natural rubber is wide, ranging from 8.0×10^5 g/mol to 1.2×10^6 g/mol, which is significant for the good processability of rubber products (Yusof, Baratha Nesan and Mohd Rasdi, 2020).

When considering the microstructure of natural rubber, it is composed of loosely connected long isoprene polymer chains. These long isoprene polymer chains are called polyisoprenes, also called a diene polymer. The term polymer comes from Greek, prefix "poly" denote many, and suffix "mer" refer to components. The numbers in the isomer name refer to specific carbon atom in each unit that are connected with adjacent units. Therefore, referring to the name of natural rubber, 1,4 cis-polyisoprene, carbon atoms 1 and 4 are connected to form a chain. Cis means that the 1 and 4 carbon atoms are connected to the same side of the carbon-carbon double bond (Miller, 2018).



Figure 2.1 Microstructure of natural rubber (Sisanth et al., 2017).

2.3 Vulcanization of Natural Rubber

Vulcanization was discovered by Charles Goodyear in 1839. Vulcanization of rubber, also known as curing is a chemical process in which elastomer materials are heated together with an agent, basically sulfur, to form cross-linked molecular network. Figure 2.2 shows a schematic molecular structure of elastomeric materials while Figure 2.3 shows a schematic molecular structure of vulcanized rubber. The yellow molecules represent the sulfur atoms crossed chains (Rowhani and Rainey, 2016).



Figure 2.2 Schematic molecular structure of natural rubber (Rowhani and Rainey, 2016).



Figure 2.3 Schematic molecular structure of vulcanized natural rubber (Rowhani and Rainey, 2016).

The vulcanization process can be divided into three phases include induction, curing

and overcuring. Induction phase, also known as the scorching time refers to the period during

which the rubber can work at the processing temperature before curing. The rubber and others compounding ingredients are in preparation for the cross-linking reaction and avoiding premature curing (Setyadewi, Indrajati and Darmawan, 2019). The second phase is curing, this phase where the formation of crosslinked network between rubber chains. At this period, the properties of rubber are relative to its best performance, which indicates that rubber has matured. The last phase is overcuring, which is also called as reversion. In this phase, there will be an undesirable-phenomena, that is the mechanical and physical properties of rubber will be degraded. This occurs when continuing entering the curing phase which is beyond the time needed to develop an optimum cure (Vulcanization process of rubber, n.d.).

In ideal vulcanization process, all the properties in the rubber compound will formed with the end of process. As a result of vulcanization process, the properties of rubber, such as tensile strength, elasticity and hardness can be improved. This is because when external force is applied, sulfur crosslinking can prevent the polymer chains from sliding easily (Monroe, 2019).

2.4 Reclaiming Technology

Reclaiming is a process, in which the waste rubber or vulcanized waste is transformed, using mechanical and thermal energy and chemicals to enter a state where it can be reprocessed, re-crosslinked and reshaped. It will break the crosslinking sites, resulting in waste rubber with relatively low molecular weight. The waste rubber that has undergone this process and the rubber obtained at the end of this process are called reclaimed rubber (Mente, Motaung and Hlangothi, 2016).

During the process of reclaiming, the three-dimensional network is split at the sulfurto-sulfur bonds and some sulfur-to-carbon bonds. Then, the crosslinking bond is scission, so called devulcanization alters the vulcanized rubber back to its original form. In the ideal reclaiming process, the crosslinking bonds (C-S bonds) should be selectively broken, while the main chain (C-C bonds) backbone generated in the vulcanization process should be kept intact. The higher the selectively in reclaiming process, the better the mechanical properties of the material (Markl and Lackner, 2020). Moreover, in a research by Kenawy and Khalil, (2021), the authors revealed that rubber chains breakdown occured predominantly on the surface, with a thin layer of breakdown occuring inside the matrix.

In order to obtain a better rubber compounding formula, reclaimed rubber can be regarded as a potential candidate of rubber compounding ingredient. There are several advantages to using reclaimed rubber in rubber compounding, so it deserves the special consideration of all rubber technologists. Reclaim-added compounds are much economic. It is studied that reclaimed rubber has excellent bonding properties, and gives rubber products a wider range of uses. In addition, it can be easily processed in mixing mills, extruders, fractioning and calendaring. Hence, a reclaim-added compounds has a good processability. (Phadke, Bhowmick and De, 1986).

According to this study, due to the unbroken cross-linked structure of the ground waste tyre rubber and the weak interfacial adhesion between the ground waste tyre rubber and matrix rubber, the direct addition of ground waste tyre rubber to natural rubber will lead to poor performance (Shi et al., 2013). Therefore, the compatibility of blending ground waste tyre rubber into natural rubber can be improved through reclaiming process. The good compatibility of reclaimed rubber and natural rubber should be attained, in order to improve product quality and reduce production cost (Mente, Motaung and Hlangothi, 2016).

2.5 Pulverization Technology

Pulverization is a well-established technique undergone before the actual application of recycling waste tyre rubber. Pulverizing using ambient grinding, cryogenic grinding and ultra-high pressure water jet pulverization is a recycling method, which is used to reduce the particle size and/or surface area of waste tyre rubber (Wang et al., 2017).

Ambient grinding technology is considered as a mechanical grinding of rubber tyres, which does not need to control the processing temperature intentionally. During ambient grinding process, waste tyre rubber will be crushed into smaller size by passing through a shredder. The crushed waste tyres are sent to a granulator, during which the steel and fibers are removed, and the crushed waste tyres are broken into smaller pieces not larger than 50mm. Finer rubber particles can be obtained through further grinding in a secondary granulator and high-speed rotating grinder. Rubber products produced by this technology may be exposed to more heat due to friction between particles during processing, which can change the properties of material (Mohajerani et al., 2020). This process might face difficulty of screen blockage. This is due to the elasticity of rubber, which will soften and stick together, and become massive substances (Junghare et al., 2017).

Junghare et al. (2017) explained that cryogenic grinding technology is the process of cooling materials and crushing them to small sized particles. In the process of cryogenic grinding, the rubber sheet is cooled below its glass transition temperature with the presence of liquid nitrogen. During this cryogenic cooling, the material will behave in a brittle behavior. Then, the embrittled rubber sheet is immediately grounded by a hammer mill. The liquid nitrogen plays a vital role in providing the refrigeration needed to retain the extremely low temperature by absorbing the heat generated during grinding operation. As a matter of fact, the volatile substances are condensed and can significantly increase the protection from rubber oxidation due to inert milling atmosphere and elimination of caking product within the mill. Hence the properties of waste rubber dust can be maintained. By cryogenic size reduction, grounded waste rubber proficiently contributing good flowability when substituted into rubber compound because it has a smoother and finer mesh size. However,

cryogenic grinding is more expensive due to added cost of cooling agent, liquid nitrogen (Mohajerani et al., 2020).

Wang et al. (2017) studied the pulverization of waste tire rubber by means of ultrahigh pressure. Ultra-high pressure water jet pulverization basically uses the high compressive shear stress produced by water jet and strong erosion to pulverize waste tire rubber. As a result, the pulverized product is fine, has a rough surface area and strong surface activity. These surface characteristics of pulverized waste tire rubber are favourable to be added into natural rubber matrix, thereby improving good mechanical properties. Meanwhile, it can be carried out at normal temperature without causing the thermal degradation of waste tire rubber. This is due to the existence of coolant, which is the continuous flow of water. According to Wang et al. (2017)'s research, the continuous impact from water flow will cause defects or hole on the surface of waste tire rubber. This lead to errosion effect, resulting in main chain degradation during pulverization.

2.6 Sieving

Sieving is the most common process of measuring particle size. This particle size **UNVERSITITEKNIKAL MALAYSIA MELAKA** measurement, also known as sieve analysis can be conducted in conformance of ASTM D5644 standard. The particle size distribution of waste rubber is critical as it may influence the processing and properties of end-use rubber product. During the sieving process, a certain amount of waste rubber sample is subjected to a mechanical force, which shakes and tapping through a specified number of sieve mesh over a specified time. Sieving is accomplished by the aid of a sieve container with a sieve hole or porous bottom through (Scrap Tire News, n.d.).

The smaller sieves are usually numbered based on the number of openings per inch. Then, the opening between the wires of a sieve screen is used to measure the size of crumb rubber during the sieving process. The finer the screen or mesh, the more openings there are per linear inch. For instance, 30 mesh means 30 holes or openings per linear inch. Therefore, when the number of openings increase, it indicates that the small-sized crumb rubber must pass through the sieve screen (Scrap Tire News, n.d.).



Figure 2.4 Diagram (a) and (b) show the different opening size of sieze (Scrap Tire News, n.d.).



Figure 2.5 The schematic diagram shows the sieving process (Scrap Tire News, n.d.).

2.7 Sample Preparation

Sample preparation is done with the purpose of making the rubber compound into a suitable shape, so that it can be shaped according to standard and then characterized. Hence, a combination of various processing stages is applied. Inevitably, compounding, calendering and molding are considered to be the processes of producing sample.

2.7.1 Rubber Compounding

Compounding process involve measuring and mixing chemicals such as curing agents, processing oils and carbon black into the raw rubber to obtain desired properties. The basic compounding ingredients are in fixed proportions in what are termed formulations. The purpose is to analyze the expected performance of the rubber compound and select the ingredients appropriately. Then, the finished rubber compound can be further processed into rubber products by molding, calendering or extrusion (Health and safety executive, no date).

Moreover, compounding can be performed either by internal mixer or two-roll mill, which will change the characteristics of the rubber. Although these operations are almost always carried out in an internal mixer today, compounding has been carried out in two-roll mill since mid-19th century. The open mill consists of two metal rolls which are jacketed for temperature control. These rollers rotate toward each other at regular intervals, and usually at different speeds. This provides a high shear mixing forces. On the other hand, Banbury Mixers which was first established by Fernley H.Banbury in 1916, are the most commonly used internal mixers today. They consist of two rotor blades turning toward each other in an enclosed metal cavity. The cavity is fed from a loading chute, rubber is added through the charging chute, and fillers and compound chemicals are added in the subsequent steps. Mixing shear and subsequent mixing time are determined by the shape, size and speed of the rotor (Sisanth et al., 2017).

2.7.2 Calendering

Calendering is a mechanical process in which a rolling process is carried out by a two roll rubber milling machine in order to press the rubber compound into sheet form. In this process, hot rubber compound is pressed into textiles (cloth, fabric), and the compound passes through two horizontally opposed stainless steel rollers, which rotate towards each other at different speeds an in opposite direction (The Art of Owning and Operating Rubber Calenders, 2016). The applied force of the rollers are normal to the direction of the rubber compound flow. Moreover, the speed difference between the rollers is called friction ratio, which allows the shearing action. Continuous rubber sheets with a thickness 3-4 mm are produced by this method.



Figure 2.6 General structure of a two-roll mill (Ghaleb, Jaafar and Rashid, 2019).

2.7.3 Compression Molding

Compression molding is a process in which the rubber compound is molded into a blank, which is then placed in the mold cavity. In order to ensure that the mold is properly filled, a certain overcharge of between 5-10% is needed. A simple stamping or cutting process directly produces blanks from rubber sheets, or with the help of rubber compounds being extruded, plunger or screw pass through an extruder die. And then the obtained strip is cut into appropriate lengths. The supply of heat depends on the heat transfer rate from the mold to the rubber. Due to the poor thermal conductivity and slow heating of rubber, the curing time is relatively long. Typical curing times are from 3 minutes for thin goods to

several hours for products with thick walls. In the latter case, it is more effective to pre-heat the rubber. This can be done in a hot air oven or microwave oven, with a maximum temperature of about 100 degree. When heated to above 100 degree, the danger of scorching becomes too great (TRANPAK, n.d.).

Compression molding is used, because this method is very simple, and only relatively simple presses and dies are needed. This is a suitable method for running for a short time. Suitable for products with large surface area or spread (TRANPAK, n.d.).



Figure 2.7 General configuration of compression molding (Graf, Consulting and Process, 2018).

2.8 Additives

Generally, rubber is usually compounded with additives. The optimum rubber compound's properties cannot be achieved only by cross-linking rubber molecules, but other additives must be added like reinforcing fillers, processing oil and vulcanizing agent. By mixing, a specific formulation of compounded rubber is designed to meet a specific application in terms of performance, cost and processability (Sisanth et al., 2017).

2.8.1 Semi-EV as a vulcanization system

Under normal circumstances, if only sulfur is used, the process of vulcanizing rubber is slow and ineffective. To overcome this limitation, there are three different sulfur vulcanization systems that rubber can be cured with, including conventional (CV), semiefficiency (semi-EV) and efficiency (EV) with an accelerator-to-sulfur ratio of 0.1-0.6, 0.7-2.5 and 2.5-12, respectively. The ratio of accelerator-to-sulfur can be varied to improve the
processing and different end used properties of rubber such as mechanical properties, thermal stability, curing properties and aging properties (Larpkasemsuk et al., 2019).

A typical sulfur curing system include zinc oxide, stearic acid, accelerator and sulfur. Based on the typical curing system formulation, activator is added to activate the action of accelerators. It can improve the vulcanization rate by reducing the curing time. Generally, zinc oxide is used in the vulcanization system together with stearic acid (fatty acid). In addition, curing agents such as sulfur is used to crosslink rubber matrix, and these crosslinks come from different bonds, such as carbon-carbon, carbon-sulfur and sulfur-sulfur bonds. At the same time, acclerator plays a vital role in improving the rate of vulcanization of rubber compound (Sisanth et al., 2017).

F	Ingredients	The amount, phr	1
E		(Parts per hundred parts	
°33.		of rubber)	
1	Zinc Oxide	3.0 to 10.0	
shi			. 1
للالك	Stearic acid	1.0 to 4.0	أوي
LINIV	Accelerator	0.5 to 4.0	KΔ
OTHE			
	Sulfur	0.5 to 3.0	

Table 2.4 Table shows a typical curing system formulation (Nocil, 2017).

With the reference to Semi-EV vulcanization system, the amount of sulfur is basically within the range of 1.0 - 1.7 phr, and the amount of accelerator is about 1.2 - 2.4 phr. The common ingredients in a Semi-EV curing system are N-Cyclohexyl-2benzothiazole sulfenamide (CBS), Tetramethylthiuram Disulfide (TMTD), Di(morpholin-4yl) disulphide (DTDM) and sulfur. Generally, secondary accelerator such as TMTD is used to activate primary accelerator, which is CBS. The Primary Accelerators are used with the amount 0.5 - 1.5 phr in most rubber compounds, while the Secondary Accelerator is generally between 10 - 40% of the primary accelerator (Nocil, 2017).

Ingredients	The amount, phr (Parts per hundred parts of rubber)			
CBS	1.5	0.6		
TMTD	0.5	-		
DTDM	-	0.6		
Sulfur	1.5	1.5		

Table 2.5 Table shows typical Semi-EV curing system formulation for natural rubber(Nocil, 2017).

Kinasih and Fathurrohman (2017) state that natural rubber with semi-EV vulcanization system showed the highest crosslink density, mainly disulfide and polysulfide crosslinking, which influences its mechanical properties. The highest crosslinking density will increase hardness and tensile strength, and slightly decrease elongation at break and compression set. In addition, due to the formation of cross-linking, the semi-EV curing system shows a shorter optimum curing time values than other curing systems. The low value of optimum curing time is beneficial to improve productivity. Increased vulcanization rates accordingly lead to increase productivity, decrease energy requirements in curing operations and increase cost savings in the curing of rubber articles.

2.8.2 Carbon Black

Carbon black is produced from the combustion of fossil fuels. Generally, it is processed into different particle size, aggregate size, shape, porosity and surface chemical properties. The size of carbon black particles ranges from 10 nm to about 500 nm. These particles do not exist as individual particles, but fuse together to form aggregates. This threedimensional arrangement represents the "structure" of the carbon black grade. Carbon black is one of the most common reinforcing filler. It is an active filler with a functional group, which plays a role in enhancing the bonding between molecules forming rubber products (Farida et al., 2019). Hence, the addition of carbon black will affect the properties of vulcanized rubber and enhance functionality of this compound. Basically, it is added in the process of rubber mixing, which will change the stress-strain behavior of vulcanized rubber. The shape of the stress-strain curve of vulcanized rubber reflects the more elastic network structure. In addition, the morphology and mechanical properties of natural rubber are improved when carbon black is compounded. Based on study, carbon black is effective in reinforcing natural rubber, because carbon black can enhance the properties (hardness, 300% modulus, tensile strength and tear strength) of natural rubber (Mente, Motaung and Hlangothi, 2016).

2.8.3 Processing oil

In order to reduce the viscosity of the rubber mixture, obtain better processability, increase flexibility at low temperature and reduce cost, various types of processing oils can be added to rubber compound. Since the 1950s, the production of oil-extended rubber by using petroleum-based extender oils, such as aromatic oil, napthenic oil and paraffin oil, has been industralized in the United States (Nasruddin and Susanto, 2018). J. Li and Isayev, (2018) stated that the addition of processing oils will also influence the consecutive processes such as molding, sheeting, calendering as well. Carbon-type composition of petroleum-based processing oils can be determined according to ASTM D2140 standard. Different petroleum processing oils have different compatibility with rubber. Therefore, it is necessary to use appropriate processing oil in a pecific formulation. Several analytical methods have been developed to characterize the solubility parameters of oil. Too much oil or incorrect compatibility often leads to blooming of oil and provide poor mechanical

propertoes and mold fouling. Aromatic oil (also known as mineral oil), such as distillate aromatic hydrocarbon extracts (DAE), is the most commonly used processing oil in modern tire manufacturing because of its compatibility with tire rubber, such as natural rubber.

2.9 Characterization properties of natural rubber

Generally, from the perspective of material science, characterization refers to the fundamental analysis and measurement of the structure and properties of materials. The characterization of material is vital to be carried out in order to increase in the level of understanding the material behavior and microstructure. Therefore, specific materials can be introduced to various applications.

In our thesis research, the characterization of reclaimed rubber was studied. However, the study regarding the reclaimed rubber characterization was not comprehensive. As a result, the characterization of natural rubber was considered as P et al. (2016) declared that the properties of reclaimed rubber were mostly identical to those natural rubber, but with a lower molecular weight and mechanical properties.

The common characteristics that can be characterized are its mechanical properties **UNIVERSITI TEKNIKAL MALAYSIA MELAKA** such as tensile properties and hardness, curing characteristics, surface properties, morphological properties and as well as its thermal properties.

2.9.1 Characterization of tensile properties of natural rubber with tensile test

Tensile properties show the behavior of material under tensile load. A tensile test is a basic mechanical test, which is used to measure the tensile properties of a material or parts. In fact, tensile tests are a destructive method. Through destructive methods, the samples will be damaged and cannot be used again.

In tensile testing, a dumbbell-shaped specimen which is conforming to an International testing standard, ASTM D412, is deformed by applying a uniaxial-forces. One end of the specimen is fixed in a static clamp, and the other end of the specimen is stretched

at a constant speed. Generally, the test is carried out until the specimen is broken (Thackeray, n.d.).

Through tensile testing, a load versus elongation curve is obtained, then it will be converted into and engineering-stress versus engineering-strain curve. In order to determine the material properties of specimen, the value of stress, σ is obtained by dividing the force measurement, F applied by the machine in axial direction by its cross-sectional area, A₀, which is measured before to running the experiment. Mathematically, it is expressed in equation (2.1). To obtain strain, ε measurement, equation (2.2) is used. In this equation, L is the instantaneous length of the specimen and L₀ is the initial length (Mechanical Properties of Materials, n.d.).

$$\sigma = \frac{F}{A_0}$$
(2.1)
$$\varepsilon = \frac{L - L_0}{L_0}$$
(2.2)

Then, these two values are drawn and displayed on an XY graph called as the curve of engineeing stress versus engineering strain.



Figure 2.8 Stress-strain curve of natural rubber (Monadjemi, McMahan and Cornish, 2016).

Based on Figure 2.8, the line graph indicated with NR is the green strength of a uncured raw rubber. Green strength is the strength of unvulcanized rubber From the engineering stress versus engineering strains curves, 5 important characteristics include (i) ultimate tensile strength, (ii) yield strength, (iii) modulus of elasticity, (iv) percent reduction in an area at fracture and (v) percent elongation at fracture can be determined (Mechanical Properties of Materials, n.d.).

- i. Ultimate tensile strength is the maximum stress that a material can withstand while being stretched or pulled before breaking.
- ii. Yield strength is the maximum stress that can be applied along its axis before the material undergo plastic deformation. Yield strength is corresponding to yield point. Below the yield point, a material will deform elastically and will still return to its original shape when the applied stress is removed.
- iii. Modulus of elasticity is the measurement of a material's stiffnes where can be obtained from the slope of the stress-strain graph. The steeper the slope, the more stiffness that is exhibited by a material, the less deformation of the material occurs under loading. The relationship between modulus of elasticity, E, the stress, σ and the strain, ε can be expressed according to Hooke's law by using the Equation (2.3).

$$\sigma = E\varepsilon \tag{2.3}$$

- iv. Percent of reduction in an area of fracture is the measurement of the deformation required to produce fracture and the contribution from the necking process.
- v. Elongation is the percent of change in length from the sample's original gage length prior to the force being applied to its ultimate length at an applied force.

In a word, the results of a tensile test should provide data which is helpful to characterize mechanical properties of parts or material. Tensile tests will classify the sample's ductility or brittleness and their elastic and plastic behavior. Additionally, tensile testing can determine the applicability and long-term performance of a component or material.

2.9.2 Characterization of hardness of natural rubber by using Shore A durometer

Hardness is defined as a measure of the indentation resistance of a material's surface. The durometer is an instrument that measures the penetration of a stress-loaded metal sphere into the rubber. Hardness measurements in rubber are expressed in Shore A or Shore D units according to ASTM D2240 test procedures. Shore A is for "soft" rubber, while the Shore D is for "hard" rubber. Generally, Shore A type is the most commonly used method. Due to the viscoelasticity of rubber, once the metal ball penetrates into the sample to the maximum extent, the durometer reading will reach the maximum value and then decrease in the next 5 to 15 seconds. For the measurement of reading, the hardness is measured on a scale from 0 to 100. A high number means a large hardness. In addition, due to the elasticity of rubber, the hardness reading may change over time, so the indentation time is sometimes reported together with the hardness values (Sisanth et al., 2017).

2.9.3 Characterization of cure characteristics of natural rubber by using rheometer

Measurement of torque is obtained both in test method according to standard ASTM D6204 and ASTM D2084. In order to characterize the viscoelastic behavior of an unvulcanized rubber, the viscosity is determined through the measurement of torque on a verticle shaft that rotates a spindle in accordance with ASTM D6204. Meanwhile, standard ASTM D2084 is introduced to characterize the curing characteristic of a rubber after undergoing vulcanization. Torque values, such as maximum and minimum torque, are obtained during the process of curing behavior characterization. In fact, the increase of torque value indicates the increase of rubber viscosity and curing behavior, as because the crosslink density of rubber increases (ASTM D2084, 2016).

Evaluation of curing characteristics is a method used for observing the vulcanization reaction rate of rubber where the characterization can used to verify the rubber product meets specifications for production. The curing characteristics are scorching time (ts_2), optimum cure time (t_{90}), maximum torque (M_H), minimum torque (M_L) and torque difference ($M_H - M_L$).

Referring to the difference time period taken during the vulcanization, scorch time is measured before the mixture begins to vulcanizate. When the rubber compound is vulcanized at a certain temperature, optimum cure time is taken. This is the period in which the best curing is adopted and result in best physical properties. Meanwhile, maximum torque and minimum torque values are obtained where indications of cross-linking density of fully vulcanized rubber by the maximum torque whereas the processability of rubber indicated by minimum torque. When the minimum torque value increases, it means that the material's procesability decreases. For the torque difference, the greater value obtained, meaning that the crosslink density is high (Khimi and Pickering, 2014).

Rheometer is the most widely used instrument in characterizing the curing characteristics of rubber mixture. The influence of the additives on curing characteristics is very important in rubber compounding. There are several studies observing that addition of fillers leads to an increase in minimum and maximum torque and a decrease in scorch time and cure time with increasing filler loading. Increase in torque indicates strong filler-matrix interactions and therefore stronger composites (Mente, Motaung and Hlangothi, 2016).



Figure 2.9 Figure shows the type of cure curve. (a) is the curve to equilibrium torque, (b) is the cure to a maximum torque with reversion and (c) is the curve to no equilibrium in maximum torque (ASTM D2084, 2016).

2.9.4 Characterization of surface properties of natural rubber using Fourier Transform Infrared Spectroscopy (FTIR)

In general, Fourier Transform Infrared Spectroscopy (FT-IR) is an analytical method that scans test materials and observes chemical characteristics using infrared light. Furthermore, the method is based on the discovery of functional groups inside molecules that vibrate when exposed to certain wavelengths of light. For this study, to better understand particle surface chemical structure and analyze how pulverization affect the cross link and structure, FTIR technique was introduced (Wang et al., 2017).

In an FTIR graph, there are two distinct modes: absorbance and transmittance. **UNVERSITITEKNIKAL MALAYSIA MELAKA** Absorbance is a measurement of how much incident light is absorbed when it goes through a substance. The transmittance, on the other hand, measures how much light is transferred. Both graphs are depicted in Figure 2.10 and Figure 2.11. In fact, transmittance is commonly utilised in the graph of the infrared spectrum (Wade Jr, 2006).

The shape of the absorption band on the FTIR graph can be used to classify the interpretation of the intensity of a material's functional group. The absorption band is frequently referred to as a peak. The majority of the y-axis is covered by a strong band. A medium band occupies almost half of the axis, while a weak band occupies roughly one-third or less of the y-axis (Wade Jr, 2006). Futhermore, the intensity of an absorption band

is affected by the polarity of the bond; the bond with a greater polarity will exhibit a more intense absorption band. The intensity is also affected by the number of bonds engaged in the absorption; the absorption band with the most bonds involved has a higher intensity(Liu, 2021).

Figure 2.13 shows the example of the total reflectance attenuated the spectra of waste tyre rubber and Ground Tyre Rubber (GTR) from water jet pulverization in Fourier Transform Infrared Spectroscopy (FT-IR).



Figure 2.12 Classification of absorption band (Wade Jr, 2006).



Figure 2.13 Example of total reflectance attenuated the spectra of waste tyre rubber and Ground Tyre Rubber (GTR) from water jet pulverization in Fourier Transform Infrared



2.9.5 Characterization of morphology of natural rubber using Scanning Electron Microscopic (SEM)

Scanning Electron Microscopy (SEM) characterization on rubber is carried out to study the morphology of a material by probing and mapping the surface structure and subsurface structure of a material (Mente, Motaung and Hlangothi, 2016). This technique can use a focused beam of high-energy electrons to generate a variety of signals at the surface of sample structure in a seried of length scales.

Referring to the earlier study by Mangili et al. (2014) and Araujo-Morera et al. (2021), the researchers employed the aid of Scanning Electron Microscopy to analyse the surface morphology of Ground Tyre Rubber (GTR) that underwent low temperature grinding, also termed cryogenic grinding. From the SEM micrograph presented in Figure 2.11 and Figure 2.12, revealing that the surface of cryo-ground GTR seems smooth. And, this smooth surface was inferring to the surface fracture caused by brittle failure mechanism (Araujo-Morera et al., 2021).

Besides, this characterization allows the compatibility and dispersion of fillers in compounds to be determined. This may be done by observing the distribution of fracture surface or distribution of particulates throughout the matrix and it allows for prediction of the material's behavior under mechanical loading. Generally, the fracture surface morphology of compound predicts mechanical, thermal and viscoelasticity (Mente, Motaung and Hlangothi, 2016). Zhao et al. (2019) reported that the fracture surfaces of reclaimed rubber and natural rubber blends are decreasing accordingly with the addition amount of reclaimed ruber, and the surface are smoother as shown in Figure 2.16. This is due to the better compatibility of reclaimed rubber and natural rubber and natural rubber.



Figure 2.14 SEM micrograph of cryo-ground GTR (Mangili et al., 2014).



Figure 2.15 SEM micrograph of cryo-ground GTR (Araujo-Morera et al., 2021).



Figure 2.16 SEM micrographs reclaimed rubber and natural rubber: (a) 30/70 (b) 50/50 (c) 70/30 blend (Zhao et al., 2019).

2.9.6 Characterization of thermal properties of natural rubber using Thermogravimetric analysis (TGA)_MALAYSIA MELAKA

Thermogravimetric Analysis (TGA) is the most extensively used method for analyzing polymer decomposition and thermal stability (Mente, Motaung and Hlangothi, 2016).

As the sample specimen is submitted to a controlled temperature programme in a controlled atmosphere, the mass of the material is monitored as a function of temperature or time. The weight of the sample increases or decreases when it is heated. Water loss, solvent loss, plasticizer loss, oxidation, degradation and weight percent ash can all be evaluated via TGA analysis (Thermogravimetric and Family, 1960).

Following that. The TGA thermal curve will be interpreted from the left side, as the curve itself is shown from left to right. The decreasing TGA thermal curve suggests that there was a weight loss. Futhermore, the peak of the first derivative denotes the location on the weight loss curve with the largest rate of change. This is referred to as the inflection point. In addition, the solid lines in the thermal curve are the weight loss curves, while the dotted lines are their corresponding derivatives (Thermogravimetric and Family, 1960). Note that the term DTG is used to define the derivative of weight loss in TGA (Cantrell et al., 2010).

The following are some previous studies that used Thermogravimetric analysis in their research. Hrdlička et al. (2021) and Ayyer et al. (2012) use the above-mentioned analysis to identify the element components in cryoground rubber powders and ambient ground tyre rubber, as well as its thermal degradation by weight loss (%). The TGA thermal curves are depicted below.



Figure 2.17 TGA thermal curve of ambient ground tyre rubber (Hrdlička et al., 2021).

Figure 2.18 TGA thermal curve of cryoground rubber powder (Ayyer, Rosenmayer and Papp, 2012).

2.10 Summary or Research Gap

Disposal of waste tyre rubber is increasing drastically in annual, reclaiming of waste tyre rubber should be effectively carried out. Previous researcher have looked into certain reclaiming technologies, as shown below:

No.	Literature Title	Strength	Weakness	Devulcanized Rubber	Reference
1.	Thermal devulcanization/ Heated Pan Process	 Allows lower temperatures Shorter reclaiming times Reclaimed product has superior mechanical properties 			Rajan et al. (2006)
		Contraction of the second	Unable to control crosslink breakage selection		Saputra et al. (2021)
2	Digester Process	Allin		Sheet Form	Shomnath Chakravarty, (2014)
3	Thermo- mechanical	کل ملیسیا ملاک	• Leads to devulcanisation and the formation of shorter polymer chains by the rearrangement of the polysulfide cross-links and the degradation of carbon-carbon bonds	اونيۇم	Bockstal et al. (2019)
	devulcanisation	UNIVERSITI TEKNI	KAL MALAYSIA ME	Particle size (0.8 -4 mm)	Formela, (2021)
		• More selective sulfur bond scission at temperature 80°C and 100°C	-	-	Seghar et al. (2019)

Table 2.6 Summary of reclaiming technology from previous researchers findings

With regards to reclaiming technologies, thermo-mechanical and microwave devulcanization process are candidates for industrial application as both of the processes are highly productive (Seghar et al., 2019; Simon et al., 2020). An ideal reclaimed rubber is that it has higher selectively of sulfur bond breaking. Seghar et al. (2019) studied that thermomechanically devulcanisation process able to produce a more selective sulfur bond splitting at a lower temperature, 100 °C. Based on Horikx's analysis from Simon et al. (2020), showed that the main chain of the thermomechanically devulcanized waste tyre rubber suffered less severe degradation than that of the microwave-devulcanized waste tyre rubber. When comes to mechanical properties, tensile strength values of rubber compounds containing thermomechemically devulcanized waste tyre rubber. However, Bockstal et al. (2019) stated that microwave devulcanized waste tyre rubber. However, Bockstal et al. (2019) stated that microwave devulcanisation process can replace thermo mechanical devulcanisation, as it provides a potential selective treatment based on the lower dissociation energy of polysulfide cross linking and avoids the degradation of the main chain.

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No.	Pulverization Technology	Strength	Weakness	Particle Size	Surface characteristic	Reference
		Able to produce fine rubber powder with a homogenous size	-	-	-	Bockstal et al. (2019)
1	Cryogenic grinding	STAT WALAYSIA W	Reduces interfacial adhesion with rubber matrix due to low surface activity	Less than 0.075 mm	 Smooth surface Average surface area (less than 0.12 m³/g) 	Wang et al. (2017)
		 Prevent rubber from thermal degradation Capable of retaining the original structure and properties of rubber 	Costly liquid nitrogen	a		Mohajerani et al. (2020)
		Paranin	Polymer chain degradation caused by heat generation during grinding			Bockstal et al. (2019)
2	Ambient grinding	مليسيا ملاك	Soften rubber will stick together and cause screen blockage	سيتي تيھ	اويتوم	Junghare et al. (2017)
				0.5 – 1.5 mm	Average surface area (less than 0.153 m ³ /g)	Wang et al. (2017)
		Strong surface activity		ALAT SIA M	Rough surface	Mohajerani et al. (2020)
3	Ultra-high pressure	 Strong surface activity 	-	Average size of 0.27	Rough and spongy	Wang et al.
	water jet	• Prevent thermal degradation		mm	surface	(2017)
	pulverization	• Sulfur bond (S-C and S-S			• Average surface $(0, 152, m^{3/2})$	
		group) are partially cleaved			area $(0.155 \text{ m}^3/\text{g})$	

Table 2.7 Summary of pulverization technology from previous researchers findings

According to earlier sub-topic 2.5 pulverization technologies, we have already mentioned the different kinds of pulverization being applied to the recycling of WTRR. Based on Li et al. (2004), the researcher reporterd that the properties of natural rubber compound will deteriorate with the compounding of bigger ground rubber powder. Hence, suitable pulverization technology is critical for producing fine grind. Referring to Table 2.7, Wang et al. (2017) pointed out that cryogenic grinding can produce WTRR with size less than 0.25 mm, and the surface is smooth. These fine rubbers are thoroughly in homogenous size (Bockstal et al., 2019). Inversely, the average particle size produced by ultra-high pressure water jet pulverization is 0.27 mm, while ambient ground WTRR has a particle size of 0.5-1.5 mm. An issue worthy of attention is the surface activity of WTRR pulverized by ALAYSI different pulverization technology. Although cryoground WTRR has smooth surface, its surface activity is low (average surface area is less than $0.12 \text{ m}^3/\text{g}$), where might reduce the interfacial adhesion with other rubber matrix. Therefore, there is still a need for additional surface treatment, like grinding under ambient conditions, to roughen the surface. Meanwhile, WTRR from ultra-high water jet pulverization and ambient grinding has rough surface which promotes strong surface activity (Wang et al., 2017; Mohajerani et al., 2020). However, the average surface area of WTRR from ultra-high pressure water jet pulverization is nearly larger than that from ambient grinding.

Moreover, Mohajerani et al. (2020) revealed that cryogenic grinding is capable of preventing rubber from thermal degradation and results in retaining the original structure and properties of rubber itself. This is due to the existence of refrigerant liquid nitrogen, which embrittle the WTRR under its glass transition temperature and break it. However, the high consumption of refrigerant is high in price. Furthermore, the WTRR from ambient grinding will face polymer degradation due to heat generation (Bockstal et al., 2019). Therefore, the softened elastic rubber will stick together, resulting in blockage of screen of the machine (Junghare et al., 2017). Wang et al. (2017) studied that ultra-high water jet pulverization have partially cleaved the sulfur bond such as S-C and S-S group of WTRR, which led to the improvement of mechanical properties. Besides, thermal degradation of WTRR can be avoided, because the continuous water flow acts as a coolant during the pulverization process.

In a nutshell, we will opt for cryogenic grinding as our experimentation of pulverize WTRR. As the raw material of our raw material is sheeted WTRR, cryogenic grinding might be the most consistent and effective way to pulverize WTRR sheet into large number of fine particles. It simply immersing the WTRR sheet in liquid nitrogen bath, and pulverize it all below its glass transition temperature. Unlike cryogenic grinding, ultra-high water jet pulverization takes time to pulverize bundle of WTRR sheet, because this pulverization is directly carried out on the surface of WTRR sheet under the strong erosion of high shear stress and high pressure water jet. Besides, this technique is somewhat complicated, because various processing parameters such as impacting angle, transverse velocity and pump pressure need to be considered. In addition, cryogenic grinding tends to retain the properties of WTRR, but ambient grinding will degrade the WTRR due to heat generated during pulverization.

In the manner of characterization of surface properties of WTRR, Kenawy and Khalil, (2021) said that the surface of ground tyre rubber (GTR) can constitute polar groups such as carbonyl, peroxy and hydroxy groups after devulcanization. The interaction between GTR particles and polar polymers improves easier owing to the existence of such polar groups in GTR particles. FTIR analysis was believed to be an indirect depiction of a material's mechanical properties. Commonly, this analysis acquired from the microstructure of a material, with the microstructure and properties of the material being represented equally (Gunasekaran, Natarajan and Kala, 2007). Based on Table 2.8 below shows different

functional group with respective wavenumber range (cm⁻¹), assignment as well as intensity that was taken from previous studies by Nandiyanto et al. (2019), Merck KGaA, Darmstadt (2021) and Mohrig, J. R.; Hammond, C. N.; Schatz (2006).

Table 2.8 FTIR peak assignments (Nandiyanto, Oktiani and Ragadhita, 2019) (Merck KGaA, Darmstadt, 2021) (Mohrig, J. R.; Hammond, C. N.; Schatz, 2006).

Wavenumber ran (cm ⁻¹)	nge Functional group	Assignment	Intensity
2990 - 2850	Alkanes	C-H stretching	Medium to Strong
2900 - 2800	Aldehydes	C-H stretching	Strong
2250 - 2100	Alkynes	C≡C stretching	Medium to Weak
2000 - 1900	Allene	C=C=C stretching	Strong
2000 - 1500	Imino	C=N	-
1740 - 1720	Aldehydes	C=O	Strong
1625 - 1440	Aromatic	C=C stretching	Medium to Weak
1440 -1395	Compounds Carboxylic acid	O-H bending	medium
1000 - 1400	Alkyl & Aryl Halides	C-F stretch	-
995 - 685	Alkenes	=C-H bending	strong
705 – 570	Disulfides	S-C stretching	- اونىۋ
500 - 470	Polysulfides	S-S stretching	<u></u>

Table 2.9 Examples of infrared spectrum peak position identified in previous study.

Wavenumber (cm ⁻¹)	Assignment	Motorial	Previous study	
Peak position	Assignment	Material		
2920 C-H stretching		Ground Tyre Rubber (GTR) and Devulcanized GTR	(Mangili et al., 2014)	

However, until now the published research on the characterization surface properties of WTRR itself is not comprehensive. Therefore, the following discussion will be related some to the interfacial adhesion between reclaimed rubber in virgin rubber.

CHAPTER 3

METHODOLOGY

3.1 Introduction

In general, this chapter describes all the methodological approaches for studying this research problem. A good methodological flow is very significance for guiding our research to be more manageable, smooth and effective. Methodological aspects include raw material preparation, sample preparation and characterization of sample. In Figure 3.1 below shows the methodology flow chart.





Figure 3.1 Methodology Flow Chart. Note: *Only FTIR analysis was revised

3.2 Raw materials

In this study, waste tyre derived reclaimed rubber (WTRR) are used as raw material. The supplier of waste tyre derived reclaimed rubber (WTRR) is produced by Jeng Yuan Reclaimed Rubber Sdn Bhd.

3.2.1 Waste tyre derived reclaimed rubber (WTRR)

Chemical separation was performed on waste tyre derived reclaimed rubber (WTRR) dust produced from truck tyre material. The chemically separated waste rubber is graded as TYREC M-1. It gives a premium and super-fine grade. General analysis content of TYREC M-1 is shown in Table 3.2.







Figure 3.2 TYREC M-1 grade waste tyre derived reclaimed rubber (WTRR) (Jeng Yuan Reclaimed Rubber, 2013).

3.3 Sample preparation of waste tyre derived reclaimed rubber (WTRR)

3.3.1 WTRR size reduction

Before the waste tyre derived reclaimed rubber (WTRR) was pulverized cryogenically, the WTRR block was cut into certain small pieces with a knife and scissors.

3.3.2 Cryogenic grinding

This sample preparation process was followed by pulverizing at a low temperature of about -196 °C. First, the liquid nitrogen was poured into the blender. Next, the WTRR was immersed in a liquid nitrogen bath, and then the embrittled WTRR was immediately pulverized for about 2 minutes with a blender (Model: 1.5 litre Faber, power 300-327W) to produce fine particles.





Cryoground WTRR

WTRR was ground at low temperature



3.3.3 Sieving

Using a test sieve (Model: Endecotts), the WTRR was separated into five size fractions after cryogenic pulverization process. This sieving process was carried out mechanically, with sizes ranging from 1.0 - 2.0 mm, 2.0 - 3.15mm, 3.15 - 4.0 mm, 4.0 - 5.0 mm and > 5.0 mm.



3.4 Sample Characterization

6 samples including before cryo-grinding and after cryo-grinding in 5 different particle size fractions were prepared and characterized. The samples characterization consisted of surface properties, morphological properties and thermogravimetric analysis.





Figure 3.5 6 samples prepared included before cryo-grinding and after cryo-grinding in 5 different size fractions

3.4.1 Surface Properties

The changes in the functional groups of WTRR produced by the reclaiming process and the WTRR that undergone cryogenic grinding were studied by infrared spectroscopy (Model: FT/IR – 6100 type A) from 400 to 4000 cm⁻¹ with a resolution of 8 cm⁻¹ in the ATR mode. Sample WTRR for before cryogenic grinding was in cube form and samples cryoground WTRR were in particle size of 1.0 - 2.0 mm, 2.0 - 3.15 mm, 3.15 - 4.0 mm, 4.0– 5.0 mm and > 5.0mm respectively. Also, to obtain a good signal-to-noise ratio, 25 spectral scans were run on each sample to produce the FTIR spectrum.



3.4.2 Morphological Properties

The morphology of the sample was detected by Scanning Electron Microscope (SEM) (Model: Zeiss 18 Research, Germany) at an accelerating voltage of 15kV. Then, a thin layer of gold palladium was sputter coated on the surface of each sample to provide a conductive layer. Magnification of 100x was used to obtain the SEM images.



Figure 3.7 Scanning Electron Microscope



Figure 3.8 Gold sputtering machine

3.4.3 Thermogravimetric analysis (TGA)

The thermogravimetric analysis (TGA) of WTRR was characterized by using Thermogravimetric Analyzer (Model: Perkin Elmer, TGA 8000). The TGA and DTG curves were recorded from 30 °C to 800 °C at heating rate of 10 cel /min. A WTRR sample weighing 11.348 g was prepared.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

In this chapter, the effect of the reclaiming technique to the chemical structure of waste tyre derived reclaimed rubber (WTRR) and the effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size are analyzed and discussed. Results and discussion were stated below.

4.2 Fourier Transform Infrared Spectroscopy (FTIR) Analysis

A Fourier Transform Infrared Spectroscopy (FTIR) was used to analyze the effect of the reclaiming technology to the chemical structure of waste tyre derived reclaimed rubber (WTRR) and the effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size. Based on Figure 4.1 and Figure 4.2 shown below, the FTIR graph shows the plot of intensity of signal (transmittance) versus wavenumber with range from 3000 cm⁻¹ to 400 cm⁻¹. The following infrared spectrum peak assignment stated in 4.2.1 and 4.2.2 were referring to the range wavenumber (cm⁻¹) stated in Table 2.8, and the supported evidence by previous study was taken from Table 2.9.

4.2.1 Effect of the reclaiming technique to the chemical structure of waste tyre derived reclaimed rubber (WTRR)

Based on the plotted FTIR graph displayed in Figure 4.1, both plot of intensity of signal were obtained from waste tyre rubber (crumb rubber) and another from devulcanized waste tyre rubber (reclaimed rubber), wherein the terms WTCR and WTRR were used respectively in the following writing.



Figure 4.1 Fourier Transform Infrared Spectroscopy (FTIR) of waste tyre rubber (crumb rubber) and devulcanized waste tyre ruber (reclaimed rubber). Man Note: * appeared in the unknown peak.

WTRR sample	KNIKAL MALAYSI Crumb rubber	A MELAKA Reclaimed rubber
Wavenumber (cm ⁻¹)		
2900 - 2800	2841	2846.4
(C-H stretching)		
2000 - 1900	1996	1996.4
(C=C=C stretching)		
2000 - 1500	1535	-
(C=N)		
1440 -1395	1415	1427.1
(O-H bending)		
1000 - 1400	1003	-
(C-F stretching)		
995 - 685	-	991.2
(C-H deflection)		
705 - 570	719.3	-
(S-C stretching)		
500 - 470	422.3	437.8
(S-S stretching)		

Table 4.1 An overview of the FTIR peaks shown in the figure above.

6

In particular, C-H bonding, O-H bonding as well as S-C and S-S bonding were discussed in this subtopic. In accordance to Table 4.1, characteristic peaks with wavenumbers between 2900 cm⁻¹ and 2800 cm⁻¹ were found in both WTCR and WTRR. The observed peaks values were 2841 cm⁻¹ and 2846.4 cm⁻¹ respectively which correspond to C-H deformation in rubber backbone. The C-H bond stretching was more distinct in WTRR. This was evidenced by the research of Mangili et al. (2014). The author reported that both of the peaks in ground tyre rubber (GTR) and devulcanized GTR were identical at peak value of 2920 cm⁻¹. The term ground tyre rubber (GTR) used in the previous study has the same meaning as WTCR used in our study. This stretching C-H bond could be an alkyl group bonded to the carbonyl group of an aldehyde (March, n.d.). The alkyl stretch of the C-H bond vibration was attributed to a wavenumber of 2853 cm⁻¹ in a study by Nuzaimah et al. (2020), but this was a different study.

Meanwhile, discussing about the presence of polar bonding after reclaiming, there is no other peaks at the range of 1740 cm⁻¹ to 1720 cm⁻¹ was found, informing there is no carbonyl groups, C=O. The carbonyl groups were categorised as an unsaturated highly reactive functional group (Coates, 2006). Although C=O polar bonding was not exist, but there is a O-H bending hydroxy group found in WTRR. The presence of hydroxyl group, which is one of the components of carboxylic acid group, was discovered. After the reclamation process, the intensity of polar bonding, O-H bending was observed to be reduced. WTRR's observed peak was less narrower and pointed than WTCR's, indicating that the decreasing of polarity.

Moreover, the existence of stretching S-S bond can be found in WTCR and WTRR. Referring to the peak value at 422.3 cm⁻¹ and 437.8 cm⁻¹, the infrared band shape at 422.3 cm⁻¹ was more thinner and pointed than that at 437.8 cm⁻¹. This was indicating that the 422.3 cm⁻¹ peak has a higher intensity as might because of high composition of sulfur-sulfur bond. Inversely, 437.8 peak has low intensity because of the devulcanization effect wherein most of the sulfur-sulfur bond was selectively scission

4.2.2 Effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size

Referring to Figure 4.2, a total of 6 different samples were scanned to obtain the plotted FTIR graph. The first sample was taken WTRR before undergone cryogrinding and the following 5 samples were from 5 fractions included 1.0 - 2.0 mm, 2.0 - 3.15 mm, 3.15 - 4.0 mm, 4.0 - 5.0 mm and >5.0 mm respectively.



Figure 4.2 Fourier Transform Infrared Spectroscopy (FTIR) of waste tyre derived reclaimed rubber (WTRR) before and after cryogenic grinding. Note: * appeared in the unknown peak

WTRR sample	Before	1.0-2.0	2.0-3.15	3.15-4.0	4.0-5.0	> 5.0
	cryoground	(mm)	(mm)	(mm)	(mm)	(mm)
Wavenumber (cm ⁻¹)						
2990 - 2850	-	2913.9	2913.9	-	-	-
(C-H stretching)						
2900 - 2800	2846.4	2846.4	2846.4	2844.5	2846.4	2844.5
(C-H stretching)						
2250 - 2100	-	2115.5	2115.5	2115.5	2115.5	2113.6
(C≡C stretching)						
2000 - 1900	-	1992.1	1992.1	-	-	-
(C=C=C stretching)						
1440 -1395	1427.1	1429.8	1429.8	-	-	-
(O-H bending)						
995 - 685	991.2	991.2	991.2	-	-	-
(C-H deflection)						
705 - 570	-	-	657.6	667.2	651.8	649.9
(S-C stretching)						
500 – 470 MALA	437.8	-	451.3	439.7	455.1	406.9
(S-S stretching)	100					

Table 4.2 A summary of FTIR peaks shown in the figure above.

Referring to Table 4.1, the discussion of absorption band will be mainly focused on the discussion regarding the trend changes in intensity of absorption band among difference WTRR particle size. Noticed that, C-H bonding, O-H bonding as well as S-C and S-S bonding in WTRR were already discussed in 4.2.1. Generally, there little difference in the intensity of WTRR band before and after cryogenic grinding.

When comparing cryoground WTRR with different particle sizes, more functional groups were found in WTRR with smaller particle size. This could be owing to WTRR's smaller particle size having a larger specific surface area. Futhermore, the intensity of infrared spectrum dropped as the particle size of WTRR increased. The FTIR graph displayed that as particle size of WTRR increased, the peaks become less visible. Coates, (2006) stated that there are some reason the individual functional groups that may fall outside the quoted. This is due to the influences of other functional groups within a molecule and environmental effects such as physical and chemical interactions on the molecule. For instance, the spectra that indicating the presence of S-S stretching. It has been stated in the
Table 2.8 that the polysulfides functional group, S-S stretching can be found within the range of 500 cm^{-1} to 470 cm^{-1} .

4.3 Scanning Electron Microscopic (SEM) Analysis

4.3.1 Morphological properties

Scanning Electron Microscopic Analysis (SEM) was utilised in this section to support the second objective stated in 1.3.



Figure 4.3 SEM micrograph of WTRR before cryogrinding.



Figure 4.4 SEM micrograph of WTRR with particle size in the range of 1.0 to 2.0mm.



Figure 4.5 SEM micrograph of WTRR with particle size in the range 2.0 to 3.15 mm.



Figure 4.6 SEM micrograph of WTRR with particle size in the range 3.15 to 4.0 mm.



Figure 4.7 SEM micrograph of WTRR with particle size in the range 4.0 to 5.0 mm.



Figure 4.8 SEM micrograph of WTRR with particle size in the range > 5.0 mm.

According to the SEM micrograph analysis, the surface of WTRR before cryogenic grinding appeared to be rougher as compared to WTRR that undergone cryogenic grinding as shown in Figure 4.3. Similarly, this can be evidenced by Ayyer et al. (2012) showing that the surface appearance shows irregular and rough.

From the Figure 4.4 to Figure 4.7 showed the smooth surface appearance of waste tyre derived reclaimed rubber (WTRR) after underwent cryogenic grinding. Similar result obtained from two previous studies as mentioned previously in 2.9.5. Owing to the nitrogen steam may have slowed the rate at which the WTRR sample degraded and oxidised, preventing a rise in temperature (Mangili et al., 2014). Stated in a previous study (Araujo-Morera et al., 2021), the smoother fracture surface generated in the WTRR samples were indicating the brittle failure mechanism during cryogenic grinding. It was proven with the obervation of some lines or ridges on the WTRR sample that radiate from the origin of the cracks in fan-shaped pattern and river pattern as shown in Figure 4.4, Figure 4.5 and Figure 4.7. In addition, this might be the tear fracture caused by the blade of blender. This is due to

the low embrittlement of WTRR during cryogenic grinding will still have its authentic properties, which is ductility.

Additionally, sharp edges were found in WTRR after cryogenic grinding. This was evident by the study of (Hrdlička et al., 2021). And, more sharp edges were found in WTRR with smaller particle size. As stated by Fazli and Rodrigue, (2021), the smaller the particle size, the higher the specific surface area. As a result, WTRR has lower the interfacial adhesion between rubber compounds as it has lower surface activity. This can be concluded from the summary in Table 2.7. Nonetheless, a previous study by Fazli and Rodrigue (2021) mentioned that the reducing WTRR particle size enhance interfacial interactions via bonding (mechanical and physical) and co-crosslinking possibilities during mixing leading to better tensile properties.

With smooth surface has less voids, while after grinding the void spots cannot be seen (Ayyer, Rosenmayer and Papp, 2012). From this current finding, occurence of voids, it would later be resulting in the crack propagation in the WTRR samples (Fazli and Rodrigue, 2021)

4.4 Thermogravimetric Analysis (TGA) MALAYSIA MELAKA

4.4.1 Thermal properties

Under this section, the analysis of thermal properties by Thermogravimetric Analysis (TGA) is used to support the result in 4.2.1. Saeed & Khattab (2021) mentioned in their study that there is a relationship between the functional group and thermal stability of rubber material. Futhermore, Król-Morkisz & Pielichowska (2018) stated that the thermal stability of polymer is heavily influenced by its chemical structure, degree of crystallinity, and molecular weight. Aromatic structures in the polymer backbone, as well as cross-linking activities, are well recognised to improve polymer heat stability. Additionally, polymers with

double bonds or oxygen-containing structures in the main chain are less resistant to high temperatures. The thermogravimetric graph below showed the weight loss (%) as a function of temperature (°C).



Figure 4.9 Thermogravimetric (TGA) graph analysis of WTRR before cryogenic grinding.

Figure 4.8 shows the TGA results generated on waste tyre derived reclaimed rubber (WTRR). The plot showed the overall curve with a decreasing trend corresponding to the increasing of the temperature. Firstly, the analysis of TGA curve showed an amount of 7.33% drop which was attributed to the emission of the volatile content. This can be evidenced by Ubaidillah et al. (2019) mentioned that the onset temperature of the volatile compounds in reclaimed waste tyre rubber was greater than 200 °C. In the region from temperature at 200 °C to 500 °C, a large decrease of 51.173% weight loss was recorded, in which WTRR undergoes decomposition of polymer content. Similarly reported by a previous study that the second transformation occured involving the decomposition of rubber content that was releasing hydrocarbon compounds at temperature around 379 °C and has been stopped at a

temperature that is higher than 450 °C (Ubaidillah et al., 2019). The hydrocarbon compounds might be the O-H bonding as stated in 4.2.1. In addition, Then, a drop of 27.762% of carbon black content in the WTRR in between 500 °C and 600 °C. And the others stated weight loss is 13.732%.

4.5 Summary

To summarise the findings, a weak polar hydroxyl bond (O-H) was discovered in waste tyre derived reclaimed rubber (WTRR). Because of the scission devulcanization effect, the intensity of S-S stretching bond decreased after reclaiming process. Following that, the intensity of the absorption band in WTRR was nearly identical before and after cryogenic grinding. The intensity of WTRR in smaller particle size appeared to be higher than that of WTRR in larger particle size based on the FTIR graph of WTRR before and after cryogenic grinding. According to scanning electron microscopic (SEM) analysis, the WTRR has a smooth surface acquired through cryogenic grinding. There are fewer voids on a smooth surface, whereas void spots cannot be seen after grinding. Because of the presence of oxygen molecules in the O-H bonding, the degradation of the thermal characteristics will occur in WTRR.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

It is evident that there are numerous methods for processing and recycling waste tyre rubber in order to handle the growing amount of waste tyre rubber in recent years. This study advocated cryogenic grinding as a reduction waste tyre derived reclaimed rubber (WTRR) particle size process. As such, both of the stated objectives are achievable.

Throughout this study, the FTIR analysis was utilized to show the presence of different bonding of the samples. In accordance to the first objective which is the effect of reclaiming technology to the chemical structure of waste tyre derived reclaimed rubber (WTRR), we discovered out that polar bonding exists reclaimed rubber. As a result, it is evident that polar bonding exists in pure reclaimed rubber. Inversely, the presence of oxygen molecules in the polar bonding deteriorate the thermal stability of WTRR.

According to the second objective of this research, we studied the effect of cryogrinding on surface properties of waste tyre derived reclaimed rubber (WTRR) in different particle size and discovered that the cryogenic grinding had a significant impact on WTRR's surface roughness, but not on the functional group of WTRR. The scanning electron microscope (SEM) used to examine this.

To be concluded, cryoground waste tyre derived reclaimed rubber (WTRR) may not be viable alternative in the compounding with other rubber compounds. Additional surface property enhancements should be implemented to boost surface activity and polar bonding activation.

5.2 Recommendations

As perspective, listed following are some future improvements in order to obtain a more accurate results:

- i) Further particle size measurement for waste tyre derived reclaimed rubber (WTRR) sample after cryogenic grinding at the particle size range of greater than 5 mm.
- ii) Study the details of reclaiming process include the rubber chemicals (additives, curing system, processing oil, carbon black) and process parameters used.
- iii) Include surface characterization of WTRR sample with aid of X-ray photoelectron spectroscopy (XPS) analysis wherein the surface chemistry can be analyzed.
- iv) Study the chemical reaction between the chemical structure of WTRR and rubber chemicals during reclaiming and cryogenic grinding process

5.3 Project Potential

This findings of this study may be useful to researchers as a reference for UNIVERSITI TEKNIKAL MALAYSIA MELAKA characterization of waste tyre derived reclaimed rubber (WTRR) in terms of surface properties.

Waste tyre derived reclaimed rubber (WTRR) could be substitued or replaced as a raw resource in the natural rubber compounds for sustainability. After that, this WTRR containing rubber product might be used for low-end products.

Meanwehile, using waste tyre derived reclaimed rubber (WTRR) is more ecofriendly and prevents soil contamination caused by the extended degradation of waste tyre rubber.

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APPENDICES

No.	Task ALAYSIA	Week														
	a strain	1. A.	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	PSM briefing	Y														
2	PSM meeting with supervisor															
3	Logbook Preparation & Submission															
4	Chapter 1 (Background of study, Problem Statement, Objective, Scope)				-						A					
5	Chapter 2 (Literature Review)															
6	Chapter 3 (Methodology)		4		.: 6	-	э.í	1			24					
7	Preliminary Data	6						5	2.8	V.	7	2				
8	Proposal Draft Submission	_	Z 1 11	1.7.4				101			A 1.					
9	Proposal Report Submission	IEI	N	KA		ЛА	LA	131	AN	IEL	.AP	A				
10	Presentation Evaluation															

APPENDIX A Gantt Chart for FYP 1

Plan
Midterm Break

APPENDIX B Gantt Chart for FYP 2

No.	Task	Week											Study Break	Final Exam				
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15		
1.	Weekly E-logbook																	
2.	Revision of Chapter 1, 2, 3	AY	514															
3.	Chapter 4 & 5			1														
5.	Sample Preparation				2													
6.	Sample Characterization				5													
7.	Four pages summary				-										1			
8.	Report Draft Submission																	
9.	Submission of Combined Logbook (W1-12)								/			2	A	A				
10.	Submission of Four pages summary	1			-													
11.	Submission of Report & Video	4	a.	6	4		2	. 6		2	: 2	-	u	~	aus			
13.	Online Q&A Session	10	-		5						. (5.		/	1.00			
14.	Presentation Evaluation											4.H						
15.	Submission of Thesis (Library Copy)	RS	TI	TE	ΕK	NI	KA		MA	1.1.7	YY:	SLA	M	EL	AK.	A		

Plan
Midterm Break



APPENDIX C The first result analysis of waste tyre rubber (crumb rubber) and devulcanized waste tyre rubber (reclaimed rubber) using Fourier Transform Infrared Spectroscopy (FTIR)

APPENDIX D The first result analysis of waste tyre rubber derived reclaimed rubber (WTRR) before and after cryogenic grinding using Fourier Transform Infrared Spectroscopy (FTIR)



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TAJUK: EFFECT OF CRYOGRINDING ON SURFACE PROPERTIES OF WASTE TYRE DERIVED RECLAIMED RUBBER

SESI PENGAJIAN: 2021/22 Semester 1

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3. Hal ini adalah kerana IANYA MERUPAKAN PROJEK YANG DITAJA OLEH SYARIKAT LUAR DAN HASIL KAJIANNYA ADALAH SULIT.

Sekian, terima kasih.

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