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UNIVERSITI TEKNIKAL MALAYSIA MELAKA

CHARACTERIZATION AND MECHANICAL PROPERTIES OF TIN DIOXIDE REINFORCED NATURAL RUBBER FOR ELECTRICAL INSULATORS

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BACHELOR OF MECHANICAL ENGINEERING TECHNOLOGY (AUTOMOTIVE TECHNOLOGY) WITH HONOURS

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MUHAMAD HARIEZ BIN SHAARI

A thesis submitted
in fulfillment of the requirements for the degree of
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2023

DECLARATION

I declare that this report entitled “Characterization and Mechanical Properties of Tin Dioxide Reinforced Natural Rubber For Electrical Insulator” is the result of my study except as cited in the references. The report has not been accepted for any degree and is not currently submitted in the candidature of any other degree.

Signature : Hariez

Name : Muhamad Hariez Bin Shaari

Date : 20 JANUARI 2023

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 Mechanical Engineering Technology (Automotive Technology) with Honours.

Signature : Hairul

Supervisor Name : Mr. Hairul Effendy bin Ab. Maulod

Date : 20 JANUARI 2023

DEDICATION

This report is dedicated to Mr Hairul Effendy bin Ab. Maulod, because none of this would have transpired without his early inspiration, guidance, and excitement. This tribute is especially for my parents. To my father, Shaari Bin Setapa, and mother, Mariah Binti Omar, for their unending love and support, and for teaching me to trust in Allah and value the hard effort. I also dedicate my report to my family, whose unconditional love motivates me to reach a greater goal in finishing this final year project. This devotion is also dedicated to my loving friends who have given me a powerful love shield and have always surrounded me, never allowing any grief to enter my heart.

Thank you.

ABSTRACT

The main objective of this study is on the mechanical and physical properties of Natural Rubber (NR) reinforced Tin Dioxide (SnO_2). Tin Dioxide (SnO_2) is an inorganic-based filler that is used when the melt compounding process is utilised for Natural Rubber. Tin Dioxide was selected for use as a strengthening agent for the mechanical and physical properties of Natural Rubber due to the properties it has that enable it to do so. The primary purpose of this is to determine the composition of Tin Dioxide that is most effective when combined with Natural Rubber. A few tests, including both physical and mechanical ones, will be performed on the compound to identify its properties. In order to investigate the material's different mechanical characteristics, tensile testing will be carried out. The mechanical characteristics of the compound will be determined using a tensile test as well as a Shore hardness test. Under Scanning Electron Microscope, the structure of the compound will be analysed for its morphology. The combination of Natural Rubber compound and Tin Dioxide based on 100 phr of the Natural Rubber and different proportions of tin dioxide (SnO_2) at 0 phr, 0.5 phr, 1 phr,3 phr, and 7 phr was found to be the best compounding formulation for the fabricated compound. All of those rubber-based formulations called for the same amount of curing additives, which were as follows: 2.5 phr of sulphur (s), 5 phr of zinc oxide (Zn0), 2 phr of stearic acid, 1 phr of tetraethyl thiuram disulphate (TMTD), and 1 phr of 6PPD. Based on data collected, 3.0 phr of SnO_2, has the highest tensile strength as well as shore hardness value and also better in morphological compared to other composition.

ABSTRAK

Objektif utama kajian ini adalah mengenai sifat mekanikal dan fizikal Getah Asli (NR) bertetulang Timah Dioksida (SnO_2). Timah Dioksida (SnO_2) ialah pengisi berasaskan bukan organik yang digunakan apabila proses sebatian cair digunakan untuk Getah Asli. Timah Dioksida telah dipilih untuk digunakan sebagai agen pengukuhan bagi sifat mekanikal dan fizikal Getah Asli kerana sifat yang dimilikinya yang membolehkannya berbuat demikian. Tujuan utama ini adalah untuk menentukan komposisi Timah Dioksida yang paling berkesan apabila digabungkan dengan Getah Asli. Beberapa ujian, termasuk ujian fizikal dan mekanikal, akan dilakukan ke atas kompaun untuk mengenal pasti sifatnya. Untuk menyiasat ciri mekanikal bahan yang berbeza, ujian tegangan akan dijalankan. Ciri-ciri mekanikal sebatian akan ditentukan menggunakan ujian tegangan dan juga ujian kekerasan Shore. Di bawah Mikroskop Elektron Pengimbasan, struktur sebatian akan dianalisis untuk morfologinya. Gabungan sebatian Getah Asli dan Timah Dioksida berdasarkan 100 phr Getah Asli dan perkadaran timah dioksida (SnO_2) berbeza pada 0 peratus berat(berat%), 0.5 peratus berat(berat%), 1 peratus berat(berat%) ,3 peratus berat(berat%), dan 7 peratus berat(berat%) didapati sebagai rumusan kompaun terbaik untuk sebatian fabrikasi. Kesemua formulasi berasaskan getah tersebut memerlukan jumlah aditif pengawetan yang sama, iaitu seperti berikut: 2.5 phr sulfur (s), 5 phr zink oksida (ZnO), 2 phr asid stearik, 1 phr tetraethyl thiuram disulphate (TMTD), dan 1 jam daripada 6PPD. Berdasarkan data yang dikumpul, komposisi terbaik untuk Getah Asli/SnO_2 ialah 3 phr. Komposisi dipilih berdasarkan kekuatan tegangan tertinggi serta Kekerasan Shore dan juga lebih baik dari segi morfologi berbanding dengan komposisi lain.

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LIST OF SYMBOLS AND ABBREVIATIONS

NR	-	Natural Rubber
SnO_2	-	Tin Dioxide
Phr	-	Per hundred Rubber
UTM	-	Universal Tensile Machine
%	-	Percent
ºC	-	Degree Celcius
OM	-	Optical Microscope
mm	-	Milimmeters
TMTD	-	Tetraethyl Thiuram Disulphate
CBS	-	Sulfenamide
ZnO	-	Zinc Oxide
TCO	-	Transparent Conducting Oxide
kgf/cm^3	-	kilogrammes force per cubic centimetre

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CHAPTER 1

INTRODUCTION

1.1 Background

Rubber is one of the most used types of products in Malaysia given the fact that its price is relatively low, and it is simple to work with. Rubber is put to use in a wide variety of applications in Malaysia, which contributes to the country's increasing production of rubber goods. Along with proteins, carbohydrates, lipids, and inorganic salts, cis-1,4-poly(isoprene) which is Natural Rubber (NR) may be found in significant levels in the milky sap of the Hevea brasiliensis tree. It is undergoing cleaning prosses at first. Then, through acid coagulation, it is separated and processed into sheets (Bokobza, 2019). Because of its very unique qualities, Natural Rubber simply cannot be substituted with synthetic rubber in some applications, such as the production of electrical wire. The primary emphasis of this proposal is placed on the mechanical properties of Natural Rubber reinforced for use as electrical insulators. To enhance Natural Rubber compounds' physical and mechanical properties, study is being conducted to investigate how different kinds of fillers interact with Natural Rubber mixtures. As a consequence of this, the study is focused on the use of Tin Dioxide as filler in compositions of Natural Rubber.

Tin is a group 14 element with oxidation values of +2 and +4 that has several uses including fuel cells and window coatings. It is also widely used in semiconducting and nanowires. Because of its mechanical, optical, and electrical characteristics, researchers are actively doing research on several oxides of tin. In the periodic table, SnO_2 is one of the metal oxides. SnO_2 is used because of its semiconductivity and strong thermal stability. SnO_2

also have 3.6 eV band gap. As a catalyst in the organic synthesis of various compounds, ferroelectric, gas sensing material along with solid electrolyte and inorganic pigment, SnO_2 has been employed in a number of applications.

This study chooses the combination of Natural Rubber modified with Tin Dioxide as the filler using melt compounding method. Tin Dioxide are being choose because of its properties that able to be to strengthen mechanical and physical properties of Natural Rubber. Besides Tin Dioxide is one of the minerals extracted fillers and also environmentally, low cost and friendly nature. Tin Dioxide is globally used mostly in technological applications such as transparent conductors, and high-efficiency solar cells and has primarily been used in the catalyst.

Electrical insulation technology is strongly reliant on cable technology. This requires the development of innovative insulating materials that have a low dielectric loss and also high electrical strength, including the optimization of stress management methods in cable connections and terminations and the development of efficient heat dissipation systems.

1.2 Problem Statement

Insulating materials for electrical cables made from Natural Rubber have been used since the beginning of the electrical industry. Early manufacturers discovered that, in addition to vulcanization chemicals, the benefit of rubber comes from different compounding components, like mineral fillers as well as process aids. Electrical insulation may get degraded over time as a result of the several stresses that it is subjected to, including those that are electrical and mechanical elements. This shortens the material's life span. Nevertheless, mechanical loads pose the greatest risk to insulators. Airborne contaminants may also pose significant problems since they have the potential to settle on the surface of the insulator, which can result in leakage, dry band arcing, and flashover.(Adupa et al., 2021)

Furthermore, with the price of rubber rising by the day, it is no longer feasible to manufacture a product only from Natural Rubber. In this scenario, the study requires a few components that can be blended with Natural Rubber to make better quality things that meet the consumer's expectations. According to the findings, fillers may be used as an added substance while also improving the properties of Natural Rubber. In improving both mechanical and physical properties, Tin Dioxide can be mixed into the Natural Rubber compound as the fillers.

Tin Dioxide is the mineral extracted that is used as a filler with their characteristic like low cost, good for nature, and because of enhances the properties of the compound. Tin Dioxide as one of the synthesized fillers can be used to improve the application of Natural Rubber through this route has both economic and environmental benefits. This study is about the effect of Tin Dioxide on Natural Rubber through its mechanical properties such as tensile strength and physical properties such as density and microscopy.

1.3 Objective of Study

The main purpose of this study is to identify the mechanical and physical properties of Natural Rubber reinforced with Tin Dioxide. Specifically, the objectives are as follows:

a) To prepare samples of Tin Dioxide with different filler loadings reinforced Natural Rubber for
b) To perform mechanical testing on Tin Dioxide with different filler loadings reinforced Natural Rubber samples.

1.4 Scope of Study

This study is to identify the "Mechanical and Physical Properties of Natural Rubber Reinforced with Tin Dioxide" through the melt compounding method. Moreover, this study focuses on the physical and tensile behaviour of Natural Rubber compound with Tin Dioxide through melt compounding the mixing material using two roll mill mixer followed by various and mechanical testing such as Tensile test, Shore hardness and Scanning Electron Microscope (morphology). The study of this topic can come out with benefits for the surroundings.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter will clarify the details of Natural Rubber and filler used to blend with Natural Rubber (Tin Dioxide). This section contains discoveries obtained from literature reviews research, which derived from the books, journal, and article that has the related topic to this study.

2.2 History of Natural Rubber

The pre-Columbian peoples of South and Central America used rubber for a variety of purposes, including the manufacture of balls, containers, and shoes as well as the waterproofing of textiles. Rubber did not pique the curiosity of early European explorers and settlers when it was first discovered. Before the year 1800, intensive research was conducted on the discovery of rubber solvents and the process of waterproofing fabrics. However, rubber was only used for the production of elastic bands and erasers, and these items were manufactured by cutting pieces of rubber that had been imported from Brazil. Joseph Priestley is credited with being the one who first discovered that rubber might be used as an eraser, which is how the material got its name.

Further study led to the discovery of a method that is both practical and effective for waterproofing textiles, as well as the vulcanization process, which fundamentally altered the way the rubber industry operated. Since then, rubber was demanded increasingly due to the expansion of the electrical industry's need for rubber insulation and the development of the pneumatic tyre.

Natural Rubber for electrical insulation has been used since the beginning of the electrical industry. Vulcanization agents found by Charles Goodyear were not the only compounds used by early manufacturers to improve rubber's performance; they also included fillers and process aids. Rubber for electrical insulation was frequently blended with a product created by heating certain vegetable oils with sulphur in a reaction similar to Goodyear's vulcanization process and probably inspired by the same idea. These components, called variously as factice or pitch, are the origins of the term "oil-based rubber," which is frequently used to describe old rubber compounds. A. G. Day began studies in the early 1860s to improve rubber compounds while simultaneously decreasing their cost. He produced "Kerite" after 3,000 tests, which may have been inspired by the Greek term Keros, which means wax. Up until the 1930s, when the first acceptable synthetics were available, Natural Rubber-based electrical insulation was the only polymeric material utilised as a wire and cable dielectric(Zuidema et al., 2011).

The late 19th century development of electrical insulation compounding technology was prompted by the communication and supply requirements of telegraph, telephone, and electrical networks. Samuel Morse created the telegraph in 1837, and the first (air-insulated) telegraph lines were established in 1844 between Washington, DC, and Baltimore, then between Baltimore and Philadelphia in 1846. Telegraph line insulation was difficult, especially if they were to be buried or placed underwater(Zuidema et al., 2011).

2.3 Natural Rubber

Both natural and synthetic sources may be used in the production of rubber. Natural Rubber is derived from the milky white fluid known as latex, which may be found in a number of different plants. However, the tree Hevea brasiliensis is the only significant commercial source of Natural Rubber (Figure 2.1). After being sheeted, extruded in unique forms, coated as a coating, or moulded, the compounded rubber goes through the vulcanization process.

Figure 2.1 Rubber tree (Hevea *brasiliensis)*

When compared to vulcanised rubber, uncured rubber has a much less number of applications. It is also used to make crepe rubber, which is used in the production of insulating blankets and footwear, in addition to adhesive and friction tapes, and cements. On the other hand, vulcanised rubber may be used in many fields of applications. Because of their resistance to abrasion, gentler types of rubber are useful for the treads of vehicle tyres and conveyor belts, while harder types of rubber are useful for pump housings and pipework used in the handling of abrasive sludge.

Rubber's flexibility allows it to be used in hoses, tyres, and rollers for a wide variety of devices, ranging from domestic clothes wringers to printing presses; rubber's elasticity makes it suitable for various kinds of shock absorbers and for specialised machinery mountings designed to reduce vibration(Ismail et al., 2011). Rubber's ability to resist the passage of gases makes it an excellent material for use in the production of products like air hoses, balloons, balls, and cushions, among other things.

The rubber's resilience to water and its ability to repel from the majority of fluid chemicals has led to its use in rainwear, diving gear, chemical and medical tubing, as well as the liner for storage tanks, processing equipment, and train tank cars. In addition to having an electrical resistance, the rubber may be used in the production of soft rubber items such as insulation and protective footwear, gloves, and blankets. Because of its durability, hard rubber is often used in the manufacturing of goods such as telephone housings, components for radio sets, metres, and several other types of electrical equipment. Rubber's friction, which has a high coefficient on dry surfaces but a low coefficient on wet surfaces, enables it to be used for power-transmission belting as well as for water-lubricated bearings in deep-well pumps in order to create a good seal.

Rubber compounding is the process of adding compounding materials and the quantity to blend to produce the best rubber formulation that can be processed, meets or exceeds related product requirements, and can be cost-friendly. This process involves both the fine art and the scientific study of compounding.

Table 2.1 Generalized rubber Compound

Ingredient	***phr***
Natural Rubber	100
Filler	50
Softener	5
Antioxidant	1
Stearic acid	1
Zinc oxide	5
Accelerator	1
Sulfur	2
Total	*165*

2.3.1 Properties of Natural Rubber

Rubber compound is a large-molecular-weight chemical made up of one or more molecules, such as monomers, linked together in a chain. Rubber compounds are all used in the production of rubber. Vulcanization, also known as curing, is the process of creating chemical linkages between loosely coiled polymeric chains. These bonds provide the material flexibility because the chains may be stretched, and when the stress is released, the crosslinks force the chains to return to their original shape. Rubber that has not been vulcanised may be dissolved in many different types of hydrocarbons, such as benzene, toluene, gasoline, and lubricants.

Rubber is water-resistant, alkali resistant, and acid-resistant. Because of its elasticity, toughness, impermeability, adhesiveness, and electrical resistance, the rubber may be used as an adhesive, coating composition, fibmolding compound, and electrical insulator. Synthetic rubber outperforms Natural Rubber in the following areas: superior ageing and weathering, greater resistance to oil, solvents, oxygen, ozone, and some chemicals, and durability across a larger temperature range. Natural Rubber has the advantage of reduced heat buildup from flexing and better resistance to ripping when heated.(Bormashenko et al., 2009)

Rubber has distinct chemical characteristics. Rubber's stress-strain relationship is frequently modelled as hyperelastic. The rubber strain crystallizes. Natural Rubber is subjected to vulcanization and ozone cracking due to the double bond in each repeating unit. When stretched, they form a line, the length of which is determined by the number. Some of the molecules are interconnected. Aside from that, when the rubber band is stretched too much, the band snaps.

Hardness, tensile strength, tensile modulus, elongation, tear resistance, abrasion resistance, compression set, resilience, and specific gravity are the fundamental physical characteristics of Natural Rubber. When heated, most materials expand, whereas rubber does the reverse. This occurs as a result of the heat causing the molecules to become entangled with one another. Rubber bands with tangled molecules at rest get much more twisted when heated. When the heat is removed, the rubber band returns to its natural form, just as it did before the stretching.

2.3.2 Characteristics and Structure of Natural Rubber

Natural Rubber is an isoprene-based linear polymer of an unsaturated hydrocarbon (2-methyl butadiene). A Natural Rubber polymer chain may have 11,000 to 20,000 isoprene units.

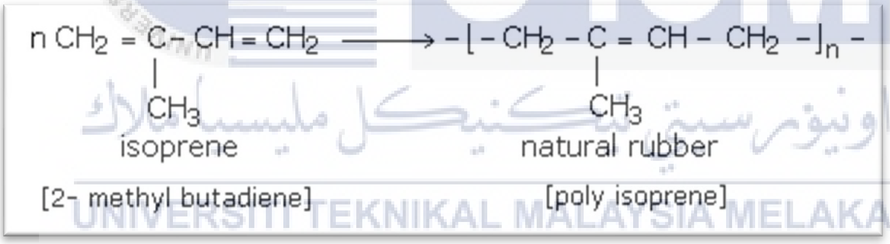


Figure 2.2 Natural Rubber Polymer chain

The elasticity, flexibility, and durability of elastomers are the fundamental features that might make these materials useful in a variety of industrial industries. Aside from these basic characteristics, each rubber has its distinct qualities. The properties can be significantly influenced by the suitable choice of compounding elements as rubber products are heavily reliant on the base elastomer even after the processing and final qualities.

Natural and synthetic are the types of rubber. Natural Rubber (NR) are in the first group. Natural Rubber generally known as cis-1, 4 polyisoprenes and is produced in many parts of the world from the latex of various plants. Nonetheless, the Hevea brasiliensis tree is the most frequently utilized commercial source of NR. Natural Rubber latex is a colloid with a specific gravity between 0.96 and 0.98 and a pH between 6.5 and 7.0. The dispersed phase consists mostly of rubber, with water serving as the distribution medium. Latex includes minor quantities of proteins, resins containing fats, fatty acids, other lipids, sterol and sterol esters, carbohydrates, and mineral materials in addition to rubber and water(Sadhan K. De, 2001).

Natural Rubber is also a polymeric material with high molecular weight and viscoelastic qualities. It is composed of cis1,4-polyisoprene. Isoprene is a diene and the 1,4 addition results in a double bond in each isoprene unit of the polymer. Natural Rubber, according to the equation formula, exhibits all of the reactions of an unsaturated polymer. It produces halogen addition compounds, ozone, hydrogen chloride, and a variety of additional reactants that react with olefins.

Furthermore, Natural Rubber is made of polymer polyisoprene. Natural Rubber often contains a tiny fraction of additional compounds such as proteins, fatty acids, resins, and organic elements. While polyisoprene is synthesize is referred to as "synthetic Natural Rubber." Natural Rubber in its raw state is subject to vulcanization and ozone cracking due to its unique physical and chemical qualities.

Natural Rubber combines strong tensile and tearing strength with excellent fatigue resistance. It has good green strength and tack, which means it can attach to itself and other materials, making it easier to manufacture. One disadvantage of Natural Rubber is its modest resistance to environmental deterioration caused by heat, light, and ozone.

2.3.3 Vulcanization of Rubber

Natural Rubber (NR) was the first to be utilized industrially. Due to its superior tack and strength in the unvulcanized state, which is high tensile strength and fracture development resistance demonstrated by vulcanizates, NR occupies a unique place in rubber technology(Hernández et al., 2015).

Vulcanization is a chemical procedure used to improve the characteristics of natural or synthetic rubber(Ulfah et al., 2015). Vulcanization is a specific elastic curing method that includes high heat and the addition of sulphur or other appropriate curatives. The final rubber has higher tensile strength, is less prone to swelling and denting, and is more flexible across a wider temperature range. It also helps to smooth the material's surface and prevents it from sticking to metal or plastic chemical catalysts.

The technique was discovered in 1839 by Charles Goodyear, a U.S. creator who also observed the important potential of specific additional compounds at the same time(Mesoamericans, 1834). A quickening agent is a substance that causes vulcanization to proceed more quickly or at lower temperatures. This strongly cross-linked polymer contains strong covalent connections with solid strengths across the chains, making it an insoluble and infusible thermosetting polymer. The vulcanization technique is a dynamic response and hence takes a certain period into account.

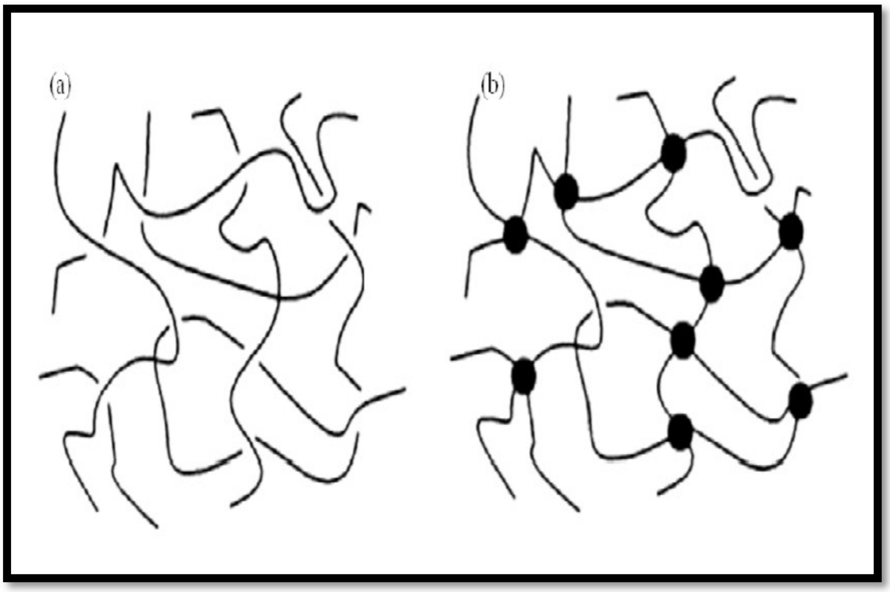


Figure 2.3 (a) raw rubber (b) vulcanized rubber schematic view

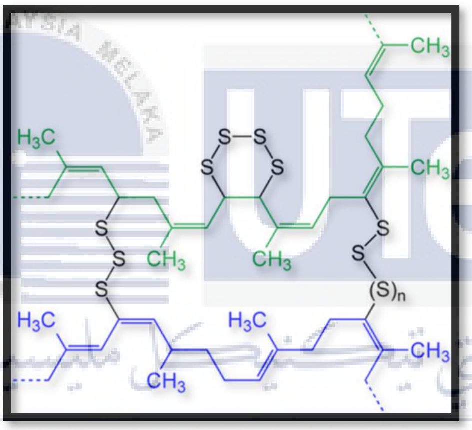


Figure 2.4 Blue and Green lines indicate vulcanization of Natural Rubber with sulphur(J.R. White and S.K. De, Rubber Technologist's Handbook, 2009).

The density of cross-links substantially influences the properties of a vulcanised substance (Figure 2.5). With increasing cross-link density, modulus and hardness rise, but hysteresis decreases. As cross-linking increases, fracture parameters such as rip and tensile strength reach a peak(Mok & Eng, 2018).

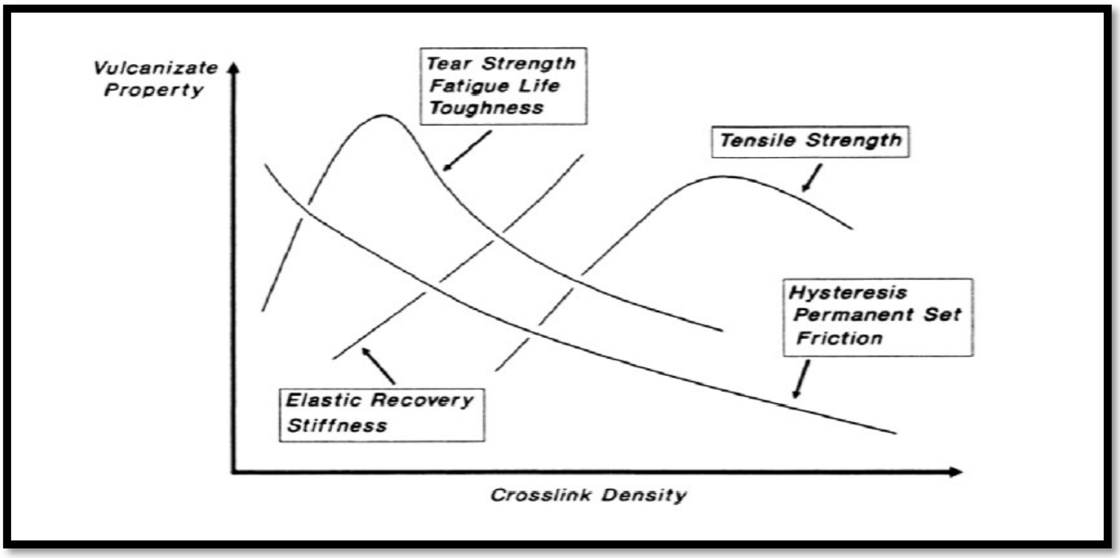


Figure 2.5 Effects of cross-link density vulcanizate properties

2.3.4 Sulphur Vulcanization Agent

In the vulcanization process, two types of sulphur are used: soluble and insoluble. Soluble sulphur has an eight-linking ring molecular structure that is crystalline in form. Sulphur bloom is a phenomenon that occurs occasionally in compounds having high levels of sulphur(Rodgers & Waddell, 2013). Because most compounds migrate when their solubility limit is surpassed, it appears as an off-white powder-like layer on the surface of the uncured compound.

As a result, insoluble sulphur is utilised to prevent the blooming form from occurring. This is actually a crystalline, polymeric form of sulphur that is insoluble in solvents and elastomers. However, beyond 120°C, the insoluble form of sulphur transforms into a soluble form. As a result, the mixing temperature must not exceed 120°C.

The development of polysulfide connections between rubber chains causes sulphur vulcanization (Figure 2.6). However, most of the sulphur is not engaged in cross-links, rather dangling sulphur fragments and cyclic sulphides are formed. As a result, the resulting network is unpredictable and has low ageing resistance.

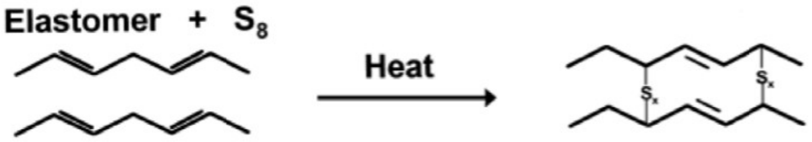


Figure 2.6 Sulphur vulcanization

Accelerators are commonly used to boost the speed and efficiency of sulphur cross-linking. These sulphur donors are used to substitute elemental sulphur in the production of vulcanizates with few sulphur atoms per cross-link (mono- and di-sulphidic linkages). These are organic substances that fall into five categories: guanidines, thiazoles, dithiocarbamates, xanthates, and thiurams.

In general, a delayed start of vulcanization is a desirable property of rubber compounds. This enables forming activities to be completed before curing begins. "Scorch" refers to premature vulcanization. The impacts of the five accelerator groups on the curing process are completely distinct. As a result, they are often used in combination to obtain the necessary scorch resistance and cure rate.

The amount and kind of cross-links are determined by the following factors: sulphur level, accelerator type, sulphur/accelerator ratio, and cure duration. A high sulphur/accelerator ratio and a long cure time enhance the amount of monosulphidic links in the final vulcanised products, enhancing heat stability, set resistance, and reversion resistance. These characteristics result from C-S bonds being more stable than S-S bonds. Compounds with a large number of polysulphidic links, on the other hand, have superior tensile strength and fatigue cracking resistance. This behaviour is dependent on the capacity of S-S bonds to break reversibly.

In sulphur vulcanization for natural rubber, the basic materials are Zinc Oxide, stearic acid, accelerators and sulphur. Zinc Oxide always had been used for utilization in the vulcanization of natural rubber. Zinc oxide and accelerators make up the important fundamental elements of the vulcanization process for natural rubber that uses sulphur. Stearic acid is also one of the elements that important that must be included. The vulcanization of natural rubber has always included the employment of zinc oxide in some capacity. It has been common knowledge for many years that vulcanization of rubber may be achieved by the application of zinc oxide to the crosslinking network. Particles of zinc oxide have the potential to boost the efficiency of the vulcanizing process that uses sulphur in rubber compounds. The use of zinc oxide as an activator in the vulcanization of rubber may boost the system's overall efficiency, whether it be in the vulcanizing or cross-linking processes. In the vulcanization process, it is assumed that stearic acid has the effect of accelerating the action of zinc oxide, which in turn has the effect of accelerating the action of the accelerator and sulphur(Roy et al., 2014).

2.3.5 Uncured vs Vulcanized Rubber

The uncured Natural Rubber has sticky characteristics, deforms rapidly when heated, and is brittle when cold. It is a poor material in this form when a high amount of flexibility is required. The cause for the inelastic deformation of unvulcanized rubber can be found in its chemical composition; rubber is made up of long polymer chains. These chains may move independently of one another, allowing the material to change form rather than having a fixed shape. By employing cross-linking linking, vulcanization stops polymer chains from moving independently. As a result, when stressed, vulcanised rubber deforms, but returns to its original shape when the force is released.

Table 2.2 Raw Natural Rubber versus Vulcanized Natural Rubber

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

There are several processes for vulcanization. After the curative has been applied to the rubber, the most significant method of vulcanization is the employment of high pressure and high temperature. These are compression moulding procedures, in which the rubber product is meant to take on the shape of the mould in order to make a tyre. Other techniques include hot air vulcanization and microwave heated vulcanization (both continuous processes).

2.5 Tin Dioxide (SnO_2)

Tin (IV) oxide, or stannic oxide, is an inorganic chemical having the formula SnO_2. Cassiterite is the mineral form of SnO_2, and it is the primary resource of tin. This tin oxide, also known by several other names, is an essential substance in tin chemistry. It is an amphoteric, colourless, diamagnetic solid. Tin Dioxide (SnO_2) is a 3.6–4.0 eV band gap n-type semiconducting material with a tetragonal rutile structure. Transparent conducting oxide thin film research is important in the realm of semiconducting materials. Nontoxicity, excellent optical transmittance, higher electrical conductivity, good piezoelectric activity, consistency, heat stability, and cheap cost are all important characteristics of SnO_2 nanostructured thin films. SnO_2 is used in a wide range of technologies, including gas sensor devices, transistors, solar cells, and optoelectronic devices(Nascimento et al., 2022). According to a review of the literature, tin oxide thin films may be produced using a variety of chemical and physical processes, including the sol-gel method, chemical bath deposition, spin coating, electron beam evaporation, PLD, sputtering, and spray pyrolysis(Eqbal & Anila, 2021).

2.6 Application of Tin Dioxide

SnO_2 surface and material characteristics (especially impurity-doped SnO_2) should be considered in the context of its three principal uses. These include (i) use as a transparent conducting oxide (TCO), (ii) use as an oxidation catalyst, and (iii) use as a solid-state gas sensing material. The surface of the material is where the "activity" occurs during the latter two applications, hence surface science inquiries are directly relevant. The bulk characteristics are responsible for SnO_2's TCO in the initial application. Many TCO applications, nevertheless, necessitate its interface with a different material. As a result, the surface and interface characteristics of SnO_2 are critical for using SnO_2 in TCO applications(Batzill & Diebold, 2005).

2.6.1 Transparent conductors

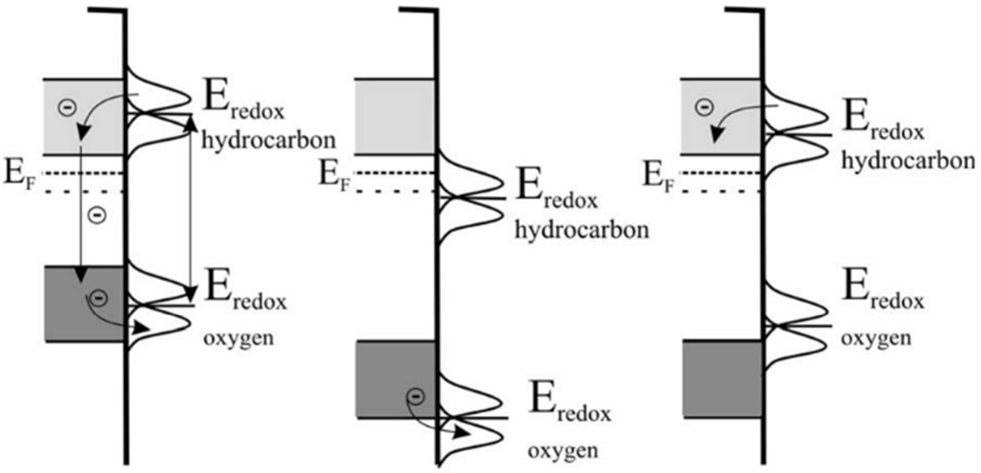
An essential oxide material, SnO_2 has low electrical resistance and good optical transparency in the visible region of the spectrum. Optoelectronic devices such as transparent FETs, LEDs, flat panel displays, and solar cells all use these materials as electrodes because they don't restrict photons from entering or leaving the optical active region, and they also have these characteristics(Batzill & Diebold, 2005). Although transparent to visible light, SnO_2 and other TCOs have a significant reflectance when exposed to infrared light. This is one of the compounds' other distinguishing characteristics. Because of this characteristic, SnO_2 is now the material of choice for usage in applications requiring energy conservation. Windows in architectural structures that have been coated with SnO_2 enable light to pass through while preventing heat from escaping or entering the structure, respectively, depending on the climatic location. Modern architectural windows, often known as "smart windows," depend on transconductance optical elements, or TCOs, to make electrical contact with electrochromic films. These films change colour and transparency in response to an applied voltage that is applied across the film.

2.6.2 Heterogeneous catalysis

Tin oxide, on the other hand, is an oxidation catalyst, unlike the majority of oxides, which serve primarily as a support medium for scattered heterogeneous catalysts. Catalysts based on Sn02 have a high degree of activity in reactions involving CO/O2 and CO/NO. The

Mars–van Krevelen technique is expected to be followed by the oxidation processes, just as it is expected to be followed by the majority of oxide catalysts. The molecules are oxidised by this method by the consumption of the lattice oxygen of the oxide catalyst, which is then re-oxidized through the use of gas-phase oxygen. This is feasible due to the fact that transition and post-transition oxides contain multivalent oxidation states. These states enable the material to react with molecules that have been adsorbed and readily give up lattice oxygen, and the material may then be re-oxidized by oxygen in the gas phase. It has been shown that surfaces containing either Sn4+ or Sn2+ are stable regardless of the oxygen chemical potential. In catalytic oxidation processes, this suggests that a straightforward reduction and also re-oxidation of SnO_2 surfaces may be anticipated to take place. Video provides a powerful way to help you prove your point. When you click Online Video, you can paste in the embed code for the video you want to add.

The inclusion of heteroelements into tin-oxide catalysts has the potential to significantly increase both the activity and selectivity of the catalysts. For example, the inclusion of copper, palladium, chromium, and antimony results in an increase in the amount of carbon monoxide and hydrocarbons that have been completely oxidised. There has been discussion over the use of copper and chromium promoted Tin (IV) Oxide for effective utilisation in three-way automobile emission control(Batzill & Diebold, 2005). According to the findings of these research, the performance of such catalysts for the oxidation of CO and hydrocarbons is comparable to that of a Pt/Al2O3 catalyst. The partial oxidation and ammoxidation of hydrocarbons need the addition of antimony, bismuth, molybdenum, and vanadium to tin oxide. Phosphorus and bismuth are additives that are utilised for oxidative coupling and oxidative dehydrogenation processes, respectively. Harrison provides a synopsis of the processes that are catalysed by both unmodified and modified SnO_2.



Catalytic oxidation of hydrocarbon molecules can proceed

Catalytic oxidation of hydrocarbon molecules cannot proceed, because the molecule is not activated

Catalytic oxidation of hydrocarbon molecules cannot proceed, because the catalyst is not reoxidized

Figure 2.7 Electron transfer across the adsorbate/oxide interface in the course of the heterogeneous catalytic oxidation of hydrocarbon molecules by gas-phase oxygen

The electrons coming from the redox pair are injected into the oxide in the first phase of this oxidation cycle. This is possible in the event that the redox pair of the molecule that has been adsorbed is located above the Fermi level as well as above the bottom of the conduction band. In order to complete the second stage, known as the reoxidation of the catalyst, it is necessary to remove electrons from the oxide in order to activate the oxygen that has been adsorbed.

2.6.3 Solid-state gas sensors

As potential candidates for use as gas sensing materials, one kind of material to look for is one whose characteristics are influenced by the surrounding gas. Changes in electrical conductance as a result of exposure to ambient gases are often measured and analysed. SnO_2 falls within the category of surface sensitive materials, despite the fact that bulk flaws might alter the conductivity of the material. The vast majority of gas sensors are constructed using porous, thick films of SnO_2 that have a high surface to volume ratio. During normal operation, the gas detecting material is heated to a temperature of around 300 degrees Celsius. Although the precise underlying processes that generate a gas response are still the subject of some debate, it is generally accepted that a change in conductivity is caused by the capture of electrons by adsorbed molecules and the band bending that is induced by the presence of charged molecules.

2.7 Properties of Tin Dioxide

Table 2.3 Properties of Tin Dioxide

Electrical Properties	
Dielectric Constant	9.86
Band Gap	3.6 eV
Mechanical Properties	
Density	6.9-7 g/cm^3
Melting Point	1630°C
Boiling Point	1800 - 1900°C
Optical Properties	
Refractive Index	2.006

Tin Dioxide is a type of inorganic compound that may be represented by the chemical formula SnO. The mineral cassiterite is where it is most often found, and its crystals have a tetragonal crystal structure. It is a solid that lacks colour, exhibits amphoteric and diamagnetic properties, and is often considered to be an oxygen-deficient n-type semiconductor. It does not dissolve in water, but it does dissolve in alkaline and acidic environments. Tin Dioxide may be purified when the metal is reduced and then burned in the air to produce the byproduct(Identification, 2017). The computed band gap was in the range of 3.6 eV, and the optical transparency was in the region of 50 percent. The presence of unoxidized tin may be responsible for both the increased value of the band gap and the decreased value of the optical transparency. The Hall apparatus determined that the resistivity of the material was 78ohm/cm, and the mobility of the material was 2.92 cm2/Vs. Because it has optical and electrical properties that are within a tolerable range, Tin Dioxide is an excellent candidate for use in optoelectronic and electronic device applications. Tin Dioxide, which is composed of large structures, has high melting and boiling temperatures. This is because it takes a significant amount of energy to break the strong ionic or covalent interactions that are functioning in three dimensions.

CHAPTER 3

METHODOLOGY

3.1 Introduction

The aim of this study is to come up with the ideal formulation for combining Natural Rubber with Tin Dioxide. This study focuses on the process of preparing materials, conducting mechanical testing, and analysing samples. Figure 3.1 shows the complete processes that were followed in order to complete the study.

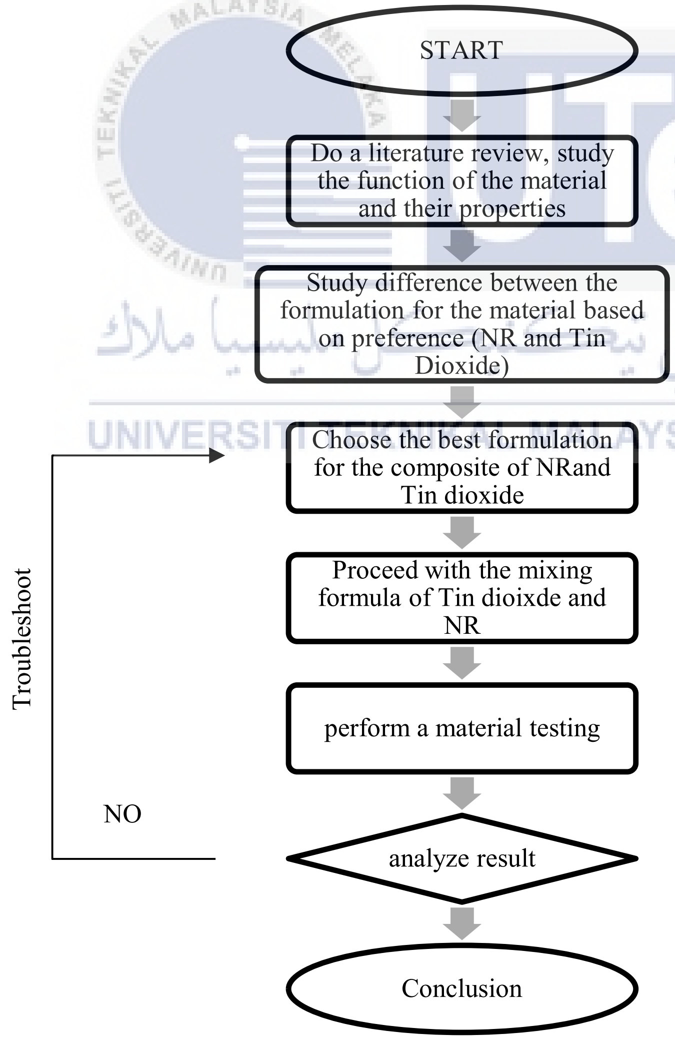
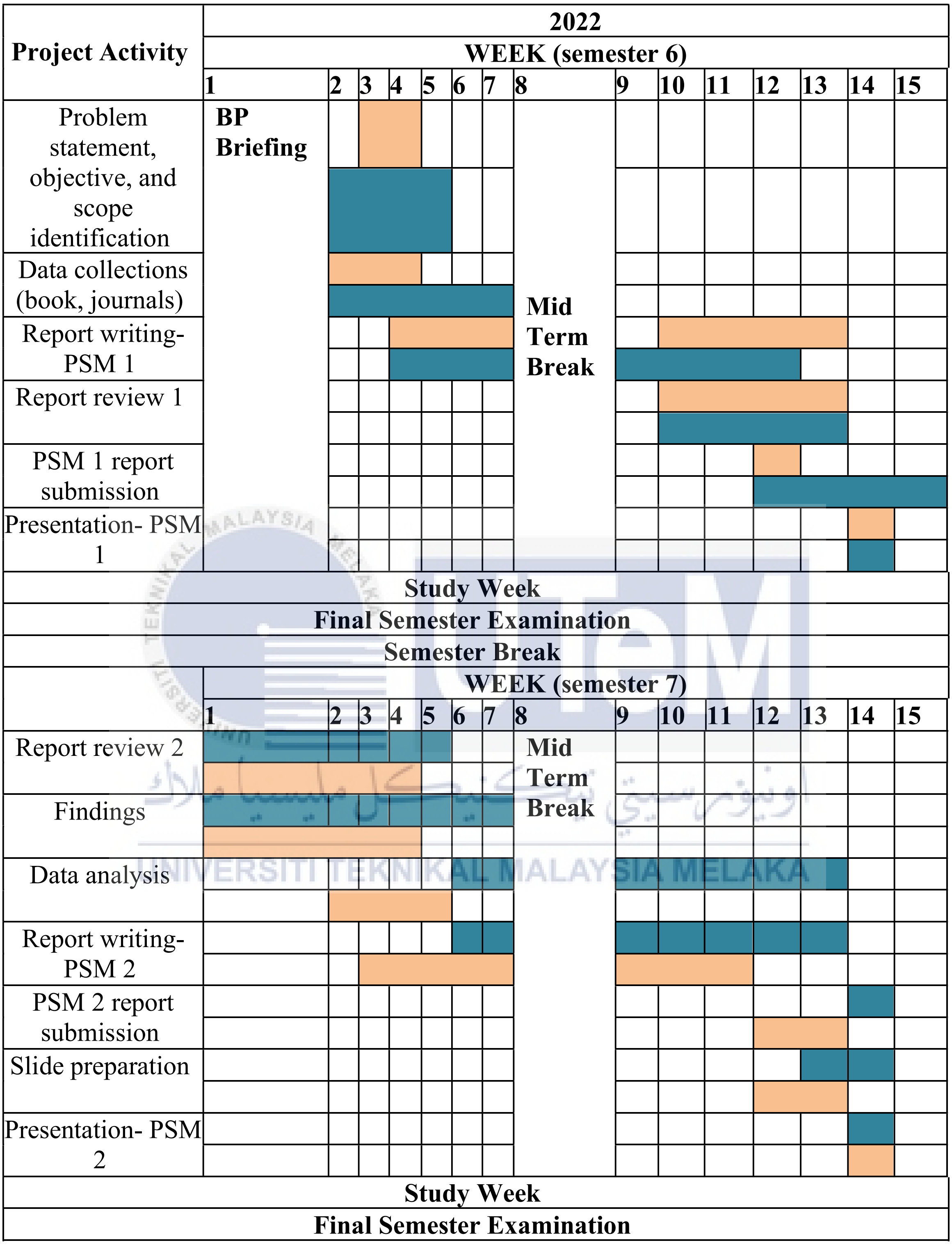


Figure 3.1 Flow chart of methodology process

3.1.1 Gantt Chart



Project Activity	**2022**														
	WEEK (semester 6)														
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
Problem statement, objective, and scope identification	**BP Briefing**		🟧	🟧				**Mid Term Break**							
		🟦	🟦	🟦	🟦										
Data collections (book, journals)		🟧	🟧	🟧											
		🟦	🟦	🟦	🟦	🟦	🟦								
Report writing- PSM 1				🟧	🟧	🟧	🟧			🟧	🟧	🟧	🟧		
				🟦	🟦	🟦	🟦		🟦	🟦	🟦	🟦			
Report review 1										🟧	🟧	🟧	🟧		
										🟦	🟦	🟦	🟦		
PSM 1 report submission												🟧			
												🟦	🟦	🟦	🟦
Presentation- PSM 1														🟧	
														🟦	
Study Week															
Final Semester Examination															
Semester Break															
	WEEK (semester 7)														
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**	**15**
Report review 2	🟦	🟦	🟦	🟦	🟦			**Mid Term Break**							
	🟧	🟧	🟧	🟧											
Findings	🟦	🟦	🟦	🟦	🟦	🟦	🟦								
	🟧	🟧	🟧	🟧											
Data analysis						🟦	🟦		🟦	🟦	🟦	🟦	🟦		
		🟧	🟧	🟧	🟧										
Report writing- PSM 2						🟦	🟦		🟦	🟦	🟦	🟦	🟦		
			🟧	🟧	🟧	🟧	🟧		🟧	🟧	🟧				
PSM 2 report submission														🟦	
												🟧	🟧		
Slide preparation													🟦	🟦	
												🟧	🟧		
Presentation- PSM 2														🟦	
														🟧	
Study Week															
Final Semester Examination															

🟧 PLAN

🟦 ACTUAL

3.2 Methodology

This chapter will provide an in-depth explanation of the methods that were used throughout the whole the study activities, including the raw materials, experimental procedure, properties characterization, and analysis. Natural Rubber and Tin Dioxide (SnO_2), both of which are organic and inorganic in nature, served as the study's primary sources of raw materials. After that, the Natural Rubber was measured, and then it was compounded with the use of equipment called a two roll mill mixer. The findings of the filler study were analysed, and recommendations will be made on the ideal formulation of Natural Rubber and Tin Dioxide (SnO_2). For the purpose of sample preparation prior to carrying out various tests, a hot compression moulding machine was used to apply pressure to a Natural Rubber compound. Following that, an optical microscope analysis of the sample will take place.

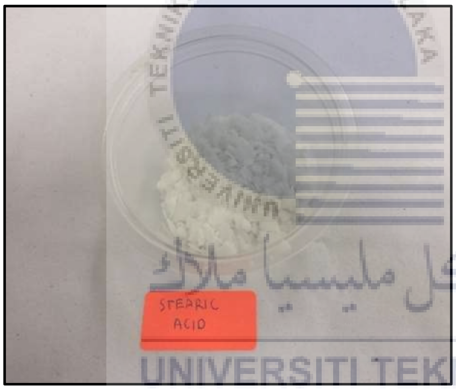
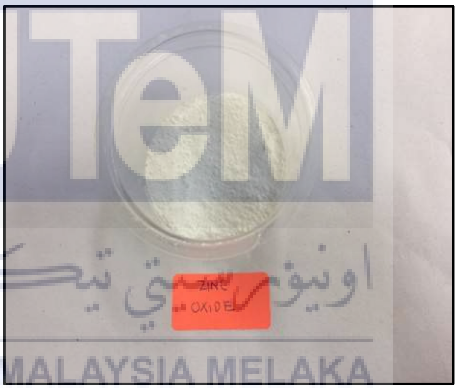
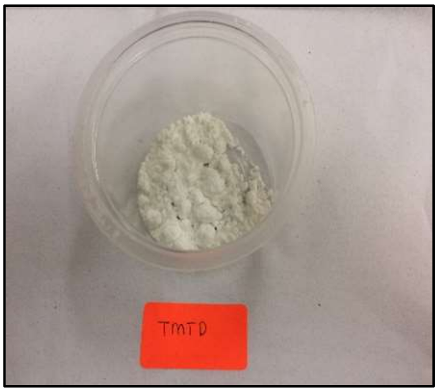
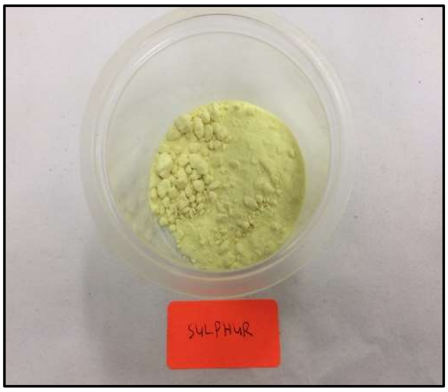
The tensile testing was carried out in its entirety with the purpose of identifying the ideal formula. All of the procedures and tests in the methodology are based on a study that examined the effects of Tin Dioxide reinforced Natural Rubber on the physical and mechanical properties.

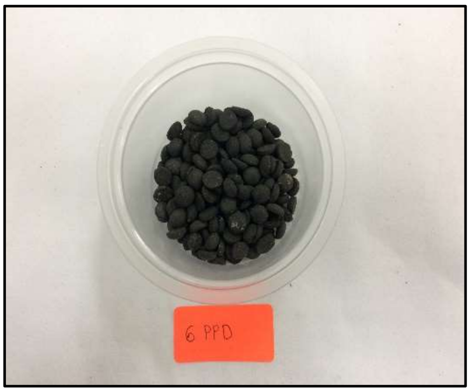
3.3 Material Preparation

Natural Rubber and Tin Dioxide, which served as a filler, were the materials that were used for this study. In order to aid the completion of the final year project, the Universiti Teknikal Malaysia Melaka (UTeM) provided Natural Rubber as well as Tin Dioxide. In the literature review chapter, the details of each of the materials are being explained.

3.3.1 Natural Rubber compound

Table 3.1 Materials for Natural Rubber compound

Natural Rubber 	Tin Dioxide 
Stearic acid  	Zinc Oxide 
TMTD  	Sulfur 

6PPD  	CBS 

Compound that was made using 100 phr of Natural Rubber and various amounts of Tin Dioxide (SnO_2) at 0 phr, 0.5 phr, 1 phr, 3 phr and 7 phr of the total weight. All of those rubber-based formulations called for the same amount of curing additives, which were as follows: 2.5 phr of sulphur (s), 5 phr of zinc oxide (Zn0), 2 phr of stearic acid, 1 phr of tetraethyl thiuram disulphate (TMTD), and 1 phr of 6PPD.(Mazliah et al., 2019)

3.4 Sample Fabrication

Compounding of Natural Rubber and Tin Dioxide (SnO_2) in the internal mixer was the first step in the sample fabrication process. This was immediately followed by hot compression moulding. During the process of compounding, two requirements need to be taken into consideration, and those are the phr of SnO_2 loading as well as the overall mixing proportion.

The rotor speed was also adjusted to 60 revolutions per minute (rpm) while the temperature was set to 60 degrees Celsius(°C). Because Natural Rubber has to be melted in order to be readily mixed with other materials, it is the first substance that is blended during the process of compounding using an internal mixer.

3.4.1 Mixing Flow Process

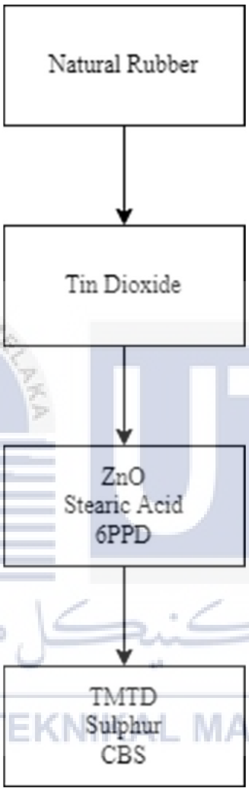


Figure 3.2 Mixing process

Figure 3.2 shows the process for mixing Natural Rubber and Tin Dioxide into a single substance. Natural Rubber and Tin Dioxide were combined in a Two Roll Mill mixer for the purpose of mixing the material compound. To begin, the mixer is only used to mix Natural Rubber during the first two minutes of the compounding process. The next step is to mix the Natural Rubber with Tin Dioxide. Following that, zinc oxide (ZnO) and 6PPD were mixed to be blended with stearic acid and the other ingredients. In the last step of the compounding process, in addition to the other components, TMTD, sulphur, and CBS are added to the mix. This brings the total number of materials to a complete process.

3.4.2 Two Roll Mill Mixer

The mixing process is conducted at Rubber Leisure Products Sdn Bhd in Serkam. These samples are mixed using two roll mill mixer that is provided by the Rubber Leisure Products Sdn Bhd as Haake Rheomix OS at the FKP UTeM not able to use. This company has been given the formulation needed for the mixing process and produce the samples.

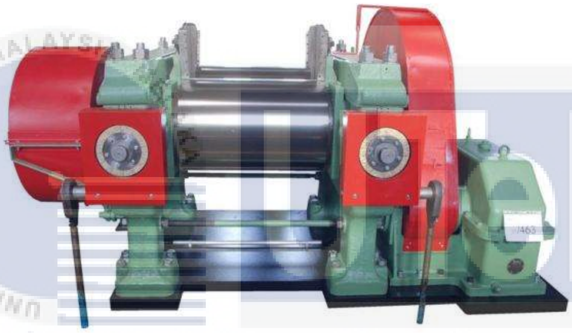


Figure 3.3Two Roll Mill Mixer

Table 3.2 Formulation of Natural Rubber compound

SAMPLE	1	2	3	4	5
Natural Rubber	100	100	100	100	100
Tin Dioxide	0	0.5	1	3	7
ZnO	5	5	5	5	5
Stearic Acid	2	2	2	2	2
6PPD	2	2	2	2	2
TMTD	0.3	0.3	0.3	0.3	0.3
CBS	1	1	1	1	1
Sulphur	1.5	1.5	1.5	1.5	1.5

The formulas were broken down into five (5) primary compositions of per hundred rubber units (PHR)so that Natural Rubber and Tin Dioxide could be compounded. After that, the analytical balance was employed to accurately weigh each and every one of the materials that were used. Calculations were performed in order to determine the formulation's per hundred rubber percentage (PHR %).

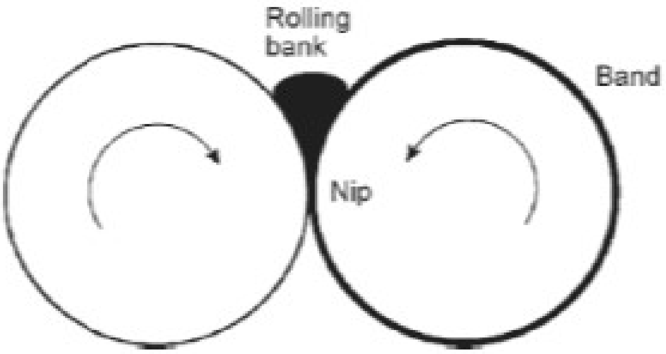


Figure 3.4 Two-roll mill schematic

In the process of blending, the crude rubbers are either masticated or broken down into smaller pieces so that an even and smooth band may be produced around the front side of the roll. After the raw rubber has been worked until it is pliable and soft, the additional materials are added.

The mixing behaviour consists of a band that does not sag and stays in full exposure to the mill roll, an effective rolling bank that has little to no stagnation, and consistent fixing throughout the working (front) revolving roll during loading.

3.4.3 Hot Press Compressing

Figure 3.5 Hot Press Compressing machine

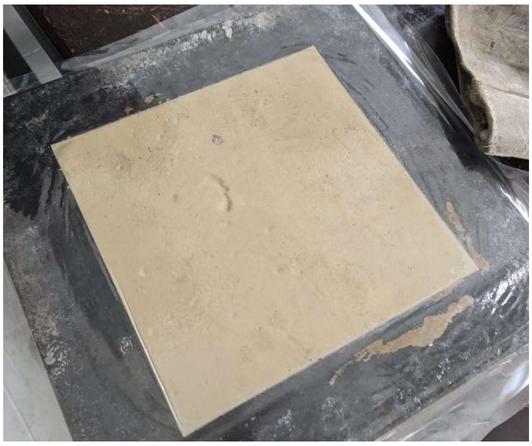


Figure 3.6 Sample after being compressed

As can be seen in Figure 3.4, the mixture of Natural Rubber and Tin Dioxide is compressed with the assistance of a hot press compression moulding machine operating at a temperature of 180 degrees Celsius (℃) for a duration of six minutes under a load of 110 kilogrammes force per cubic centimetre(kgf/cm^3).

In order to produce a sample, this method involves pressing a mixture of Natural Rubber and Tin Dioxide into a mould that has been heated beforehand, with the resulting material having a thickness of three millimetres(mm). In the context of this investigation, the substance under investigation will be subjected to evaluation through not one but two distinct types of testing: a tensile test as well a shore hardness test.

3.4.4 Cutting Process

Figure 3.7 Laser Cutting Machine for the cutting process

Figure 3.8 Material with Dumbbell shaped after went through the cutting process.

In order to assure that the specimen was precisely clamped when the tensile test was performed, the specimen that is to be tested for tensile strength must be shaped in accordance with the ASTM standard for rubber, which is known as ASTM D412. Before moving on to the mechanical testing process, the method of cutting was used to create the Dumbbell shape. The procedure of cutting was carried out with the assistance of the laser cutting machine that can be found in FTK UTeM. The laser cutting machine was selected because it is capable of cutting through a workpiece with a thickness of 3 millimetres(mm) without requiring any additional abrasive materials; instead, the operation is carried out only with the help CO_2 laser.

3.5 Mechanical Testing

These studies make use of composite materials that are mostly composed of Natural Rubber and Tin Dioxide as their basis. The testing is to investigate and analyse the behaviour of the mixture of Natural Rubber and Tin Dioxide.

3.5.1 Tensile Test

The tensile test is essential because it is the method that can be used to determine the mechanical properties and tensile behaviour of the Natural Rubber compound that mixes with Tin Dioxide. The modulus of elasticity is part of the tensile behaviour, and it is one of the methods that can be used to calculate the load-carrying ability as well as the amount of sample deformation that occurs before fracture. As part of this study, a tensile test was performed in accordance with ASTM D412 Type C by making use of the Universal Testing Machine (UTM) from INSTRON machine type 5960 Dual Column Testing Systems. The results of this test are shown in Figure 3.9**.** The tests were carried out at temperatures ranging from 25 to 20 degrees Celsius (℃) with a humidity of fifty percent (50%).

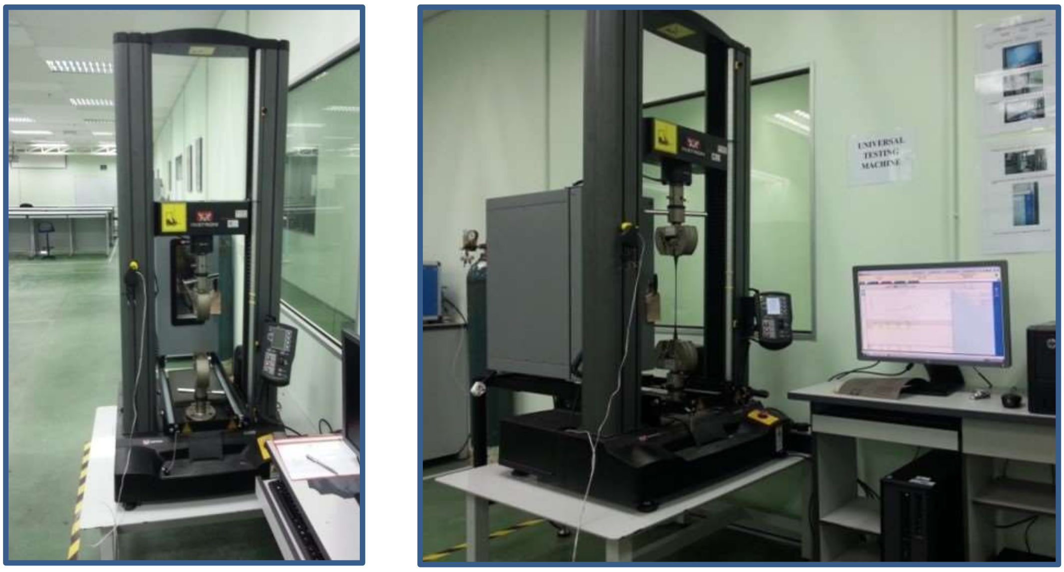


Figure 3.9 Tensile Test machine

The tensile test is one of the most basic tests in engineering, and it offers vital information about the material and the properties that are linked with it. Its properties may be used in the planning and analysis of design structures, as well as in the design of novel materials that are more ideally suited for a particular application.

The samples had a reduced gauge section and a cross-section that was cylindrical in shape. The smaller gauge section ensured that the maximum tensions occurred inside the gauge, rather than close to the grips of the load frame. This helped to avoid strain and fracture of the specimen close to or in the grips of the load frame.

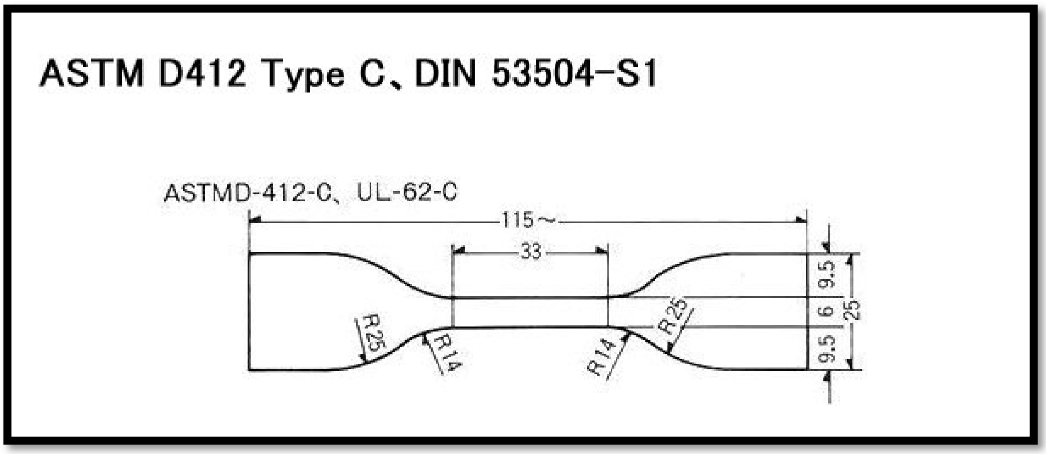


Figure 3.10 Specification of Dumbbell shaped sample

The accompanying figure 3.10 provides a representation of the standard sample dimension specification that applies to this test. In order to ensure that our results are reliable, we will take the average of the results from all five samples that we examine. Utilizing the Solidwork software, the dimension of the compound will be crafted in its entirety. The dimensions of the sample shaped like a dumbbell are shown in Figure 3.10. These dimensions adhere to the ASTM standards for the rubber material.

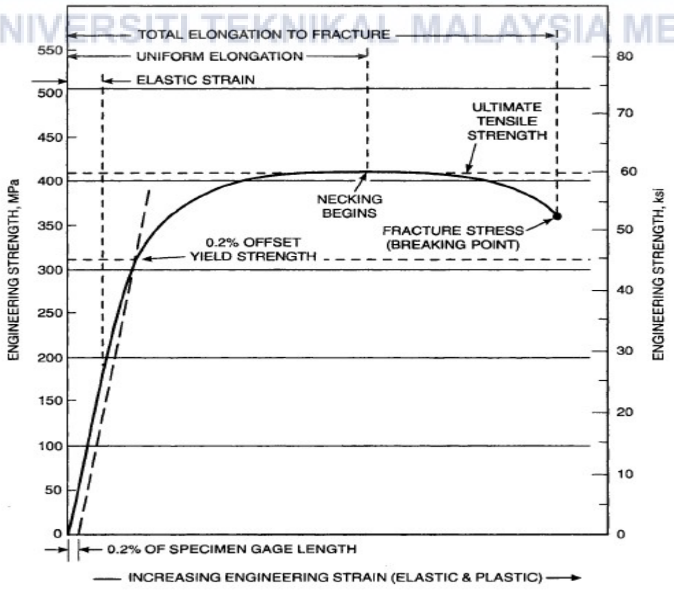


Figure 3.11 Stress and Strain diagram

The data were used to determine the elastic modulus, yield strength, and ultimate tensile strength of each material. After that, the results were plotted on a stress-strain diagram so that the sample could be compared. This test was designed to gather information about the materials so that their properties could be determined. The aim of this test was to collect information about the materials. The data that were utilised for this study report was processed according to the laboratory professor's instructions in order to get the correct and consistent results.

As a consequence, a curve will be produced that shows how it responded to the forces that were applied. The point of failure attracts a great deal of attention and is referred to as the Ultimate Strength or UTS in Figure 3.11.

3.5.2 Shore Hardness

The durometer hardness test is most often used in order to qualitatively evaluate and compare the behaviour of elastomeric and elastomeric-like materials from a mechanical behaviour. The elastomer industry makes extensive use of durometer hardness tests, which evaluate the material's resistance to indentation. These measurements are utilised for quality control as well as for the rapid and easy assessment of the material's mechanical properties. The elasticity of the material is the primary factor that determines the hardness rating of the material. The elastic modulus of elastomeric materials is linked to the durometer hardness of the material. The durometer is a predefined test force applied to an indenter that is either spherical or conical in form. The indenter is then pressed into the material while it is subjected to the test force for the specified amount of time.(Qi et al., 2003)

Figure 3.12 Shore-A

Table 3.3 Durometers of several materials

Material	Durometer	Scale
Bicycle gel seat	15–30	OO
Chewing gum	20	OO
Sorbothane	30–70	OO
Rubber band	25	A
Door seal	55	A
Automotive tire tread	70	A
Soft wheels of roller skates and skateboard	78	A
Hydraulic O-ring	70–90	A
Hard wheels of roller skates and skateboard	98	A

Ebonite rubber	100	A
Solid truck tires	50	D
Hard hat (typically HDPE)	75	D

The dial gauge uses the resulting indentation to determine the value of the material's hardness. The A scale loads used in the tests range from 822 gf to 4550 gf (D scale). For the purpose of measuring the shore hardness in this experiment, an indenter of type A was used (a hardened steel rod with a diameter ranging from 1.1 to 1.4 millimetres(mm) and a truncated 35° cone with a diameter of 0.79 millimetres(mm)).

3.6 Analysis

In this experiment, Scanning electron microscopy (SEM) was used during the morphological examination so that the agglomeration and dispersion of the sample could be analysed.

3.6.1 Fourier Transform Infrared Spectroscopy (FTIR)

Functional group availability at the backbone of Natural Rubber polymer and its related chemical composition were determined through Fourier Transform Infrared Spectroscopy (FTIR). Analysis of the spectra obtained from a Jasco FTIR spectrometer, as shown in Figure 3.8, confirmed the presence of a functional group and revealed structural differences between pure Natural Rubber and Natural Rubber/SnO2 nanocomposites. This FT-IR spectroscopy analysis was carried out at a temperature of 25°C, and spectra were collected at a resolution of 4.0 cm-1, over a wave number range of 400 cm-1 - 4000 cm-1, with a scanning speed of 2 mms-1 and an aperture size of 7.1 mm. Attenuated total reflectance (ATR) was used for this analysis.

Figure 3.13 Fourier Transform Infrared Spectroscopy (FTIR)

Through the use of FTIR, we are able to investigate and define the sample to determine how Natural Rubber and Tin Dioxide interact with one another.

3.6.2 X-ray Diffraction

X-ray diffraction, known as XRD, is a method that may be used in the field of materials science to determine the atomic and molecular structure of a substance. This is accomplished by irradiating a sample of the material with incoming X-rays and then measuring the intensities and scattering angles of the X-rays that are dispersed by the material. The sample is then used to determine the properties of the material. The intensity of the scattered X-rays is displayed as a function of the scattering angle, and the structure of the material may be deduced from the study of the position, angle, and the intensities of scattered intensity peaks. In addition to being able to measure the typical locations of the atoms in the crystal, it is also possible to get information on how the real structure varies from the ideal one. These variations might be the result of internal stress or flaws, for example.

Figure 3.14 XRD

In this study, X-ray diffraction analyses were carried out in order to determine the crystalline structure of Natural Rubber that had been mixed with Tin Dioxide and the influence that this crystallinity had on Natural Rubber that had been reinforced with Tin Dioxide. For each of the various compositions of Tin Dioxide, X-ray patterns of samples that were either cross-linked or not cross-linked were obtained.(Abraham et al., 2012)

3.6.3 Electron Scanning Microscope

Figure 3.15 Electron Scanning Microscope

An electron scanning microscope is a form of electron microscopy that scans the surface with a focussed beam by creating sampling images. Within the sample, electrons are able to interact with atoms, which results in the generation of a variety of signals that offer information on the surface roughness, sample composition, crystalline structure, and substrate orientation of the specimen. images are collected across a given sample surface area and a 2-dimensional picture is formed that indicates spatial disparities.

3.6.4 Density

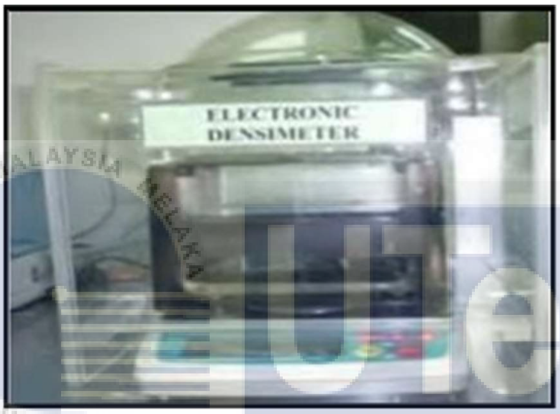


Figure 3.16 Electronic Densimeter (MD-300S)

The Natural Rubber composite containing Tin Dioxide (SnO_2) was analysed using an Electronic Densimeter model MD-300S, which was utilised to conduct the density test. The procedure was performed for a total of three times with each five different formulations. After taking the reading, we recorded the overall average of the density measurement result for each formulation.

3.7 Expected Outcome

It was hypothesised that increasing the amount of filler in the compound would lead to an improvement in the tensile strength of the material. However, earlier studies on Natural Rubber reinforced with various fillers show that the Natural Rubber tensile strength decreases as more quantity fillers are added, which is the opposite of the effect that was anticipated. These findings contradict each other. According to the findings of the earlier studies, the separation between the fillers particle and the Natural Rubber causes the mechanical properties of the compound to quickly deteriorate. This separation is caused by the high amount of fillers content. Because of the separation, the compounds were able to fracture quite quickly. In addition, the incorporation of the additive does not have a direct impact on the strength of the materials, but it does have a significant impact on the young's modulus that was recorded. It was hypothesised that the influence of the cross-linking conditions between the matrix and its additive was the main factor of these findings(Mohamed et al., 2017).

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

This chapter provides a concise summary of the results analysis and related discussions on all of the experimental data findings that were gathered from a variety of tests and observations. The primary objective of this study is to investigate how the properties of Natural Rubber/SnO_2 composites are affected by varying amounts of SnO_2 in terms of weight percentage. Therefore, the parameters that were used in this study are the weight percentages of the fillers, which were varied as follows: 0 phr, 0.5 phr, 1 phr, 3 phr and 7 phr respectively. These weight percentages were used. Consequently, the tensile test is performed in order to investigate the Natural Rubber/SnO_2 composites loaded with varying weight percentages of SnO_2 addition in order to determine their respective mechanical properties. After that, tests such as density, hardness, XRD, and FTIR were carried out in order to determine the physical properties of the nanocomposites that had been produced. In conclusion, the morphology of the failure surface of each Natural Rubber reinforced tin oxide composite was examined using SEM.

4.2 Cure characteristics of Natural Rubber/ SnO_2 Composites

In Table 4.1, how different fillers loading affect the Natural Rubber curing process is shown. Curing characteristics at 150 ºC for SnO_2 filled Natural Rubber compounds are provided, including minimum torque (ML), maximum torque (MH), scorch time (Ts_2), and 90% cure time (T_{90}). Torque-time relationships were calculated for all of the rubber compounds in Table 4.1 to analyse their vulcanization properties. The results showed that

3.0 phr filler loading produced the highest minimum torque in comparison to any other filler loading (ML). Torque increases because of the 3.0 phr filler loading sample's increased strength and stiffness. This is because of the limitations on the molecules' ability to move around in the sample. Still, as filler loading increased, the maximum torque (MH) amount dropped. This happened because the interaction between the Natural Rubber and SnO_2 was reduced at higher filler loading(Nor & Othman, 2016).

The scorch time also reveals the maximum time a rubber compound can be worked at a given temperature before it begins to cure. Clearly, the scorch time was greatly impacted by the addition of filler. The already reduced scorch time of Natural Rubber compounds has been further reduced as filler loadings have increased. At 3.0 phr filler loading, the value of scorch time (Ts_2) increased significantly. The presence of sulphur in the accelerator lengthened the scorch time, which led to this result. One possible explanation is that the Natural Rubber's higher polarity content interacts with the Natural Rubber/SnO_2 content to reduce the vulcanization rate. The rubber's processing ability, measured by the amount of heat history it can withstand before changing to a crosslinking condition, improves in proportion to the square of the scorch time. Suphurate agent improved nanocomposite crosslinking.

In this way, the cure rate index (CRI) is a metric for determining cure rate by comparing the latter to the former (Ts_2). The CRI discovered that curing efficiency could be greatly improved by increasing the SnO_2 filler loading. Due to SnO_2's role as an auxiliary accelerator, the rubber molecules used in the cross-linking reaction had a greater number of reactive sites. The crosslink density in the vulcanised Natural Rubber was affected by the filler loading. Due to the high concentration of sulphur on Natural Rubber chains and the role that SnO_2 played as an additional accelerator during curing, this phenomenon was explained. Because of this connection, the crosslink density increased. This means that the crosslink density and stiffness of the vulcanised rubber are related to the torque that can be generated. The increased torque may indicate that incorporating the filler into the Natural Rubber matrix improved the rubber composites' viscosity and modulus. When the torque is increased, the curing time decreases because of the higher cure rate index. That's why the resulting connection between them is so sturdy: it's a crosslinking. Possible explanation: the Natural Rubber matrix shows that SnO_2 bonding is advantageous. This was first observed during mastication, suggesting higher processing efficiency thanks to the deproteinized rubber's lower viscosity(Ngamsurat et al., 2011).

Table 4.1 Curing properties all compounds.

Filler Loading (phr)	T_2 (min.sec)	T_{90} (min.sec)	ML (dNm)	MH (dNm)	ΔTorque (dNm)	CRI (min^{-1})
Control (average)	0.85	1.63	0.46	6.93	6.47	128.21
0.5 (average)	0.79	1.40	0.52	6.38	5.86	163.93
1.0 (average)	0.81	1.48	0.33	7.79	7.46	149.25
3.0 (average)	0.85	1.51	0.57	6.51	5.94	151.52
7.0 (average)	0.79	1.43	0.53	6.56	6.03	156.25

4.3 Mechanical and physical characteristic of Natural Rubber filled with SnO_2 composites.

Based on the results of the tensile testing, x-ray diffraction (XRD), Fourier transform infrared (FTIR), density, and hardness testing, the results of the mechanical and physical characteristics were discussed.

4.3.1 Tensile test analysis

The relationship between the matrix and the filler in the reinforcement is examined through tensile testing. Mechanical strength properties of Natural Rubber/SnO_2 composites can then be determined through tensile testing. The ultimate tensile strength, tensile modulus, and elongation at break percentage are the three (3) primary responses considered in tensile property analysis. The results of tensile tests on composites made of Natural Rubber and tin oxide showed how well the matrix and filler were bonded to one another.

The ultimate tensile strength of a Natural Rubber/SnO_2 is the maximum force it can withstand relative to its initial cross-sectional area for varying amounts of SnO_2 is added. To improve with increasing SnO_2 addition weight percentages, this method was put into action. As shown in Figure 4.1, the value begins to drop dramatically after reaching a peak at 3.00 phr due to the addition of SnO_2. When supplementing the reinforcement with 3 phr SnO_2, ultimate tensile strength increases by the most. Compared to a control sample without SnO_2 addition, which had a strength of about 22.09 MPa, the sample with SnO_2 added had an ultimate strength of about 32.20 MPa. Intermolecular bonding between the filler particles and the resin particles is responsible for the enhanced strength. In addition, the formed variational composites were much less strength as SnO_2 concentrations were increased.

The tensile strength drastically decreases to 30.63 Mpa when using a filler loading of 7.0 phr. This is because an insufficient dispersion of nano reinforcements occurred as the

Natural Rubber/SnO2 matrix concentration increased. This also showed that agglomeration and aggregation of nanoparticles were inevitable, even at low nanoparticle concentrations, and had an immediate effect on the mechanical properties of nanocomposite systems, as evidenced by a reversal in the trend of tensile strength as nanoparticle concentration was increased. The formation of the nanomatrix structure has been hypothesised to be responsible for the dramatic increase in tensile strength; a smaller increase could result from insufficient nanofiller concentration or improper nanomatrix formation. This means that the optimal tensile strength was achieved with a filler loading of 3.0 phr in Natural Rubber composite. High tensile strength was achieved, which was attributed to the effective dispersion of Natural Rubber and SnO_2 nanofiller. Thus, the strongest possible crosslinking connection and the most refined state of plasticizers were advocated for. The nanoscale nature and high element filler loading of nanocomposite significantly enhanced their mechanical properties. Natural Rubber/ SnO_2 composite stress versus strain graph (Figure 4.2). The results statements were all supported by the stress vs. strain graph, which showed a linear increase.

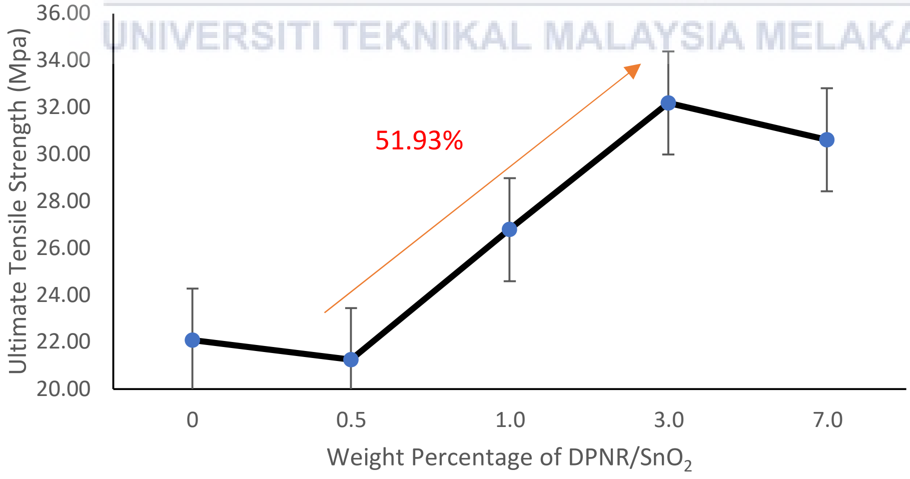


Figure 4.1 The influence of SnO2 addition on the ultimate tensile strength of Natural Rubber composites systems at varying weight percentages

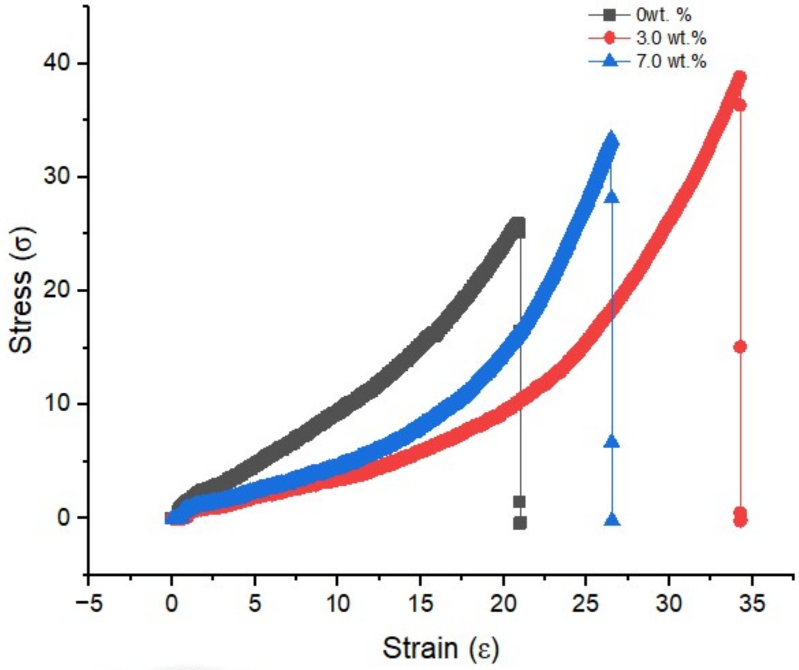


Figure 4.2 Natural Rubber/SnO2 composites' reaction to stress versus strain

Stiffness is proportional to the Young's modulus of an engineering material, and vice versa. A higher Young's modulus indicates a stiffer sample, which may lead to greater ultimate strength. As a result, there is a connection between the sample's strength and the Young's modulus. The Young modulus results for Natural Rubber composites with SnO_2 nanofiller are shown in Figure 4.3. Figure 4.3 is a graph depicting the results of incorporating varying amounts of SnO_2 into the composites used in fabrication. The 0 phr of SnO_2 resulted in a young modulus of about 0.90 GPa. Young's modulus increases from 0.90 GPa at 0 phr SnO_2 filler loading to 1.11 GPa at 3.00 phr SnO_2. This lower value for the young modulus at 7.0 phr can be attributed to the unique properties of rubber, which include its high elasticity, low viscosity, and high deformation tolerance. After being bent, the rubber quickly and firmly returns to its original shape. It is internally dampened despite being very sturdy. Rubber can be moulded into various forms and formulated into a wide variety of properties. It is possible to modify the load displacement curve by adjusting its parameters. Otherwise, agglomeration associated with the increased concentration of SnO_2 could be to blame for the diminished properties.

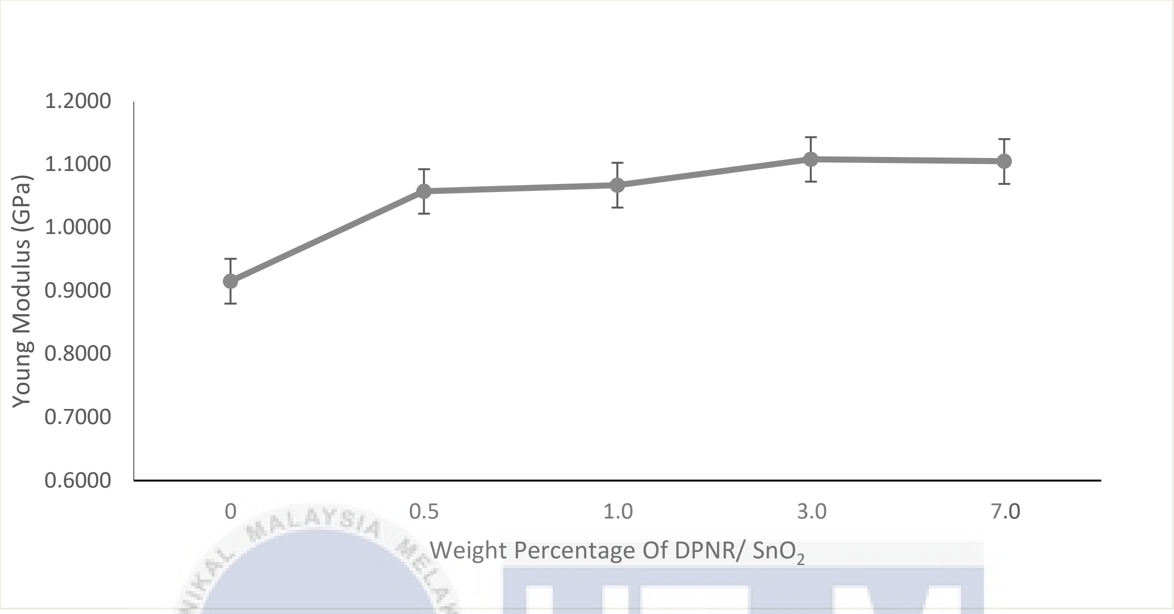


Figure 4.3 Effect on the Young's modulus of Natural Rubber composites by varying the percentage of SnO2 added

Elongation at break percentages for Natural Rubber composites are shown for a range of SnO_2 addition weight percentages in Fig. 4.4. Rigidity and stiffness of the vulcanizate are reflected in its cross-linking, which is directly proportional to the percentage of elongation break. As SnO_2 is present, sulphur undergoes vulcanization, which is initiated by the formation of active accelerator complexes that react with sulphur to produce an active sulfurating agent. The active sulfurating agents reacted with Natural Rubber to form a rubber-bound intermediate. So, the accelerator particles were initially fastened to the Natural Rubber chains, restricting the therapeutic's movement toward the Natural Rubber. Data for percentage elongation at break shows that SnO_2 addition loadings of up to 1.0 phr improved elongation characteristics.

The percentage of elongation at break for samples made with increasing amounts of SnO_2 added by weight is displayed in Figure 4.4. One possible explanation for this is that the Natural Rubber matrix and the filler reinforcement have a high-quality interfacial interaction. This demonstrated that the Natural Rubber matrix and SnO_2 filler nanocomposite were well dispersed and interacted with one another(Abdullah et al., 2018). After the SnO_2 loading reaches 3.0 phr, the percentage of elongation at break unexpectedly tends to decrease. The term "enhancement" of rigidity in nanocomposite was used to describe the phenomenon. The combination of a high tensile strength and a low percent elongation is indicative of a high-quality material. Accordingly, the higher the filler loading, the stiffer and brittle the sample. This is because the elongation limits of the Natural Rubber/SnO_2 composites had already been reached prior to the addition of more SnO_2.

Thus, SnO_2 became significantly agglomerated, which later disrupted the proper interaction between a Natural Rubber and SnO_2. Consequently, the Natural Rubber matrix had been cured too quickly. Therefore, the external surface of the sample had been inadequately cured, while the interior area was partially uncured and displayed any porosity and agglomeration in the surface structure. This makes it hard for the testing device to accurately determine the elongation properties of the sample because the sample tends to break there. As a result of the first compound's enhanced curative dispersion, the vulcanizate's tensile strength and elongation at break are both significantly increased.

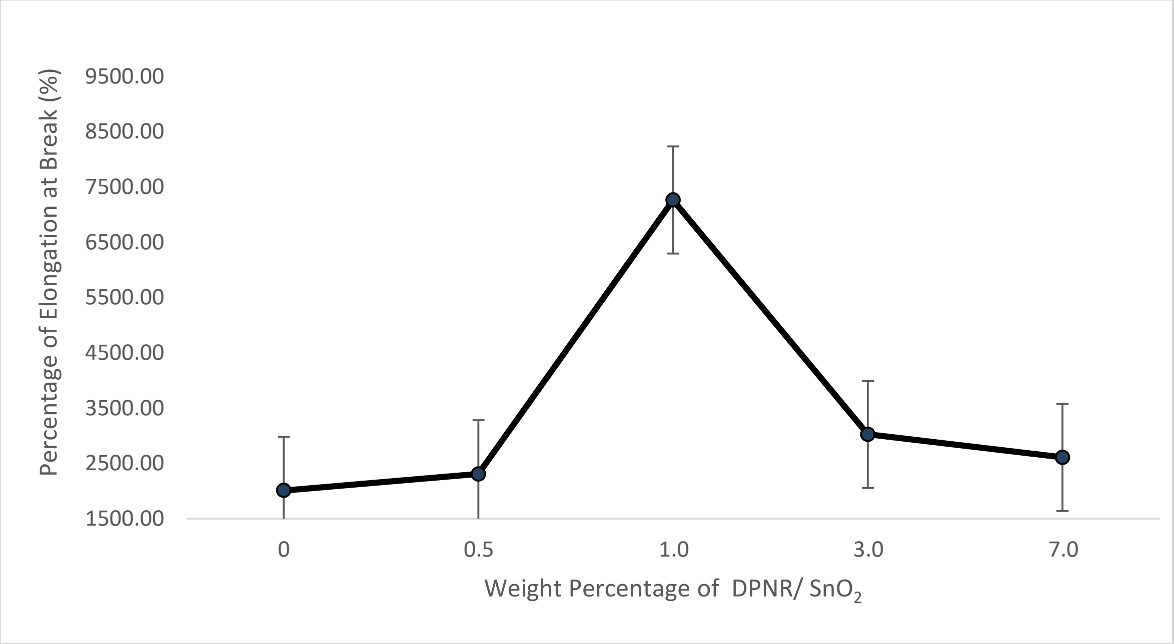


Figure 4.4 Different SnO_2 loading had different effects on Natural Rubber composites' elongation at break.

4.3.2 Shore Hardness Analysis by Shore-A method

Surface hardness was measured using the Shore-A scale as part of the mechanical characterization. As the filler degrades, the composite's hardness characteristics change. It's all in the filler treatment when it comes to the mechanical characteristics(Latinwo et al., 2010). Figure 4.5 provides a visual representation of these surface hardness measurements. From 0 phr to 3 phr filler loading, the results showed a progressive increase in hardness. Following that, it also decreases from 3.0 phr to 7.0 phr. An increase in the percentage of SnO_2 in the composite's weight increase the surface hardness, and other compositional changes also affect the hardness. With a 3.0 phr filler loading, the hardness value increase by about 5.89%. As the rubber's elasticity decreases due to the addition of filler particles are to be expected(Ugbaja et al., 2016).

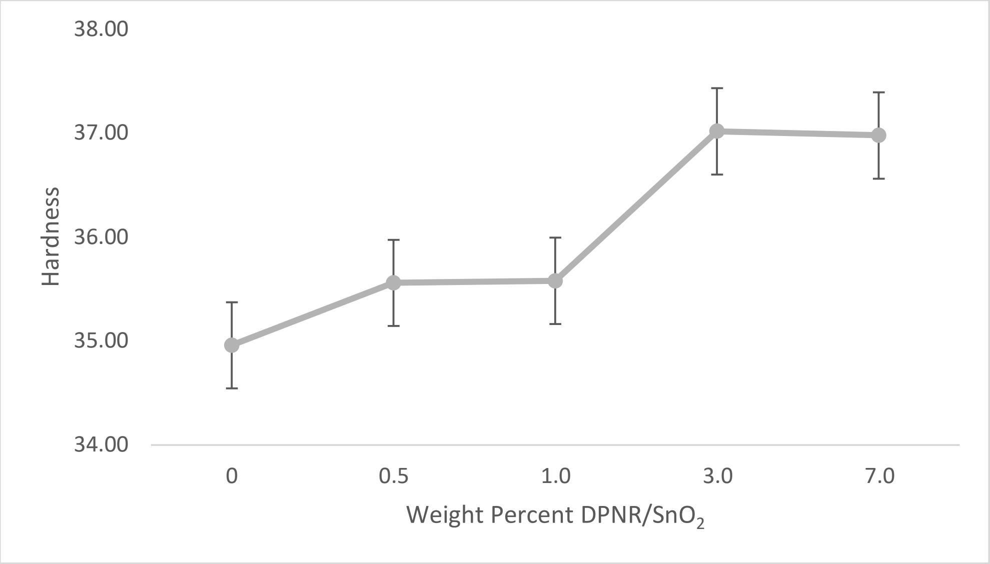


Figure 4.5 Reinforcing SnO2 filler loadings increases the Natural Rubber's hardness.

4.3.3 Fourier Transform Infrared (FTIR) analysis

Figure 4.6 shows FTIR spectra collected at a scanning speed of 2 mm^{-1}s and a wavenumber range of 500-4000 cm^{-1} that were used to investigate the chemical structures of Natural Rubber containing SnO_2 filler at varying loadings. When filler loading was set to 0 phr, 3.0 phr, and 7 phr, a broad peak appeared between 2000 and 2500 cm^{-1}. The peaks at 1580, 2000, and 2300, cm^{-1} originated from SnO_2 present in the Natural Rubber matrix. The rising peaks were all connected to the addition of nanofiller components, specifically SnO_2 filler loadings in Natural Rubber. According to the data, the peak indicates that the Natural Rubber matrix and the SnO_2 filler loading of the nanocomposite do interact physically or through chemical bonding. An activator-enhanced diffusion process and/or changes in polymer structure brought on by the activating agent were likely responsible for the observed enhancement in band intensity or shift to lower wavenumber values in the matrix(Kowalczuk & Pitucha, 2019).

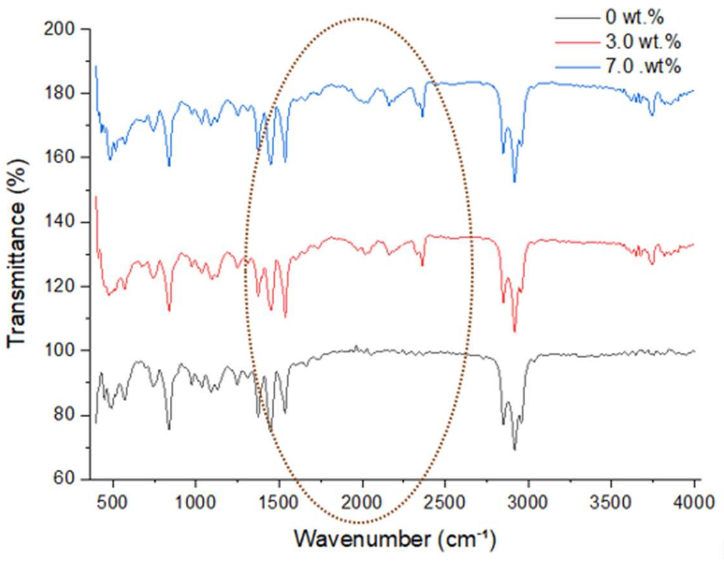


Figure 4.6 FTIR spectra of Natural Rubber filled with SnO_2.

4.3.4 X-Ray Diffraction analysis of Natural Rubber/SnO_2 structure

The presence or absence of a chemical reaction between the components was determined by XRD analyses (Irez et al., 2017). X-ray diffraction (XRD) diffractograms of crystalline polymers have distinct peaks, while those of amorphous polymers have progressively broader peaks. The crystallin sharp peak of intensity was seen in the XRD pattern of Natural Rubber doped with 0 phr, 3.0 phr, and 7.00 phr SnO_2 filler loading. Crystallinity variations in composites reveal themselves as peaks in XRD patterns. X-ray diffraction (XRD) patterns of nanocomposites of Natural Rubber and SnO_2 at different loadings are shown in Figure 4.7. This peak proved that crystalline structure could be found in Natural Rubber. The formation of the composite's crystalline structure was also affected by factors such as temperature, pH, and nanofiller concentration. After being doped with 0 phr, 3.0 phr, and 7.00 phr SnO_2, the peak intensity of Natural Rubber broadened, indicating that the crystalline structure of the Natural Rubber/SnO_2 had improved. In the presence of SnO_2 nanocomposite, polymerization of aniline results in an increased XRD peak, indicating that the composite structure provides strong support for a significant connection between the

Natural Rubber main chain and the SnO_2 nanocomposite. When SnO_2 is added, XRD peaks become more pronounced. Prior to reduction, filler ions are adsorbed at these binding sites, covering the entire rubber particle's surface. It is well-known that the strength of adsorption depends on the types of functional groups, and that stronger adsorption increases system stability and leads to the formation of smaller nanoparticles. Nanoparticles formed from the adsorption of ions were allowed to have their initial chemical composition and distribution be affected by the phase interface.

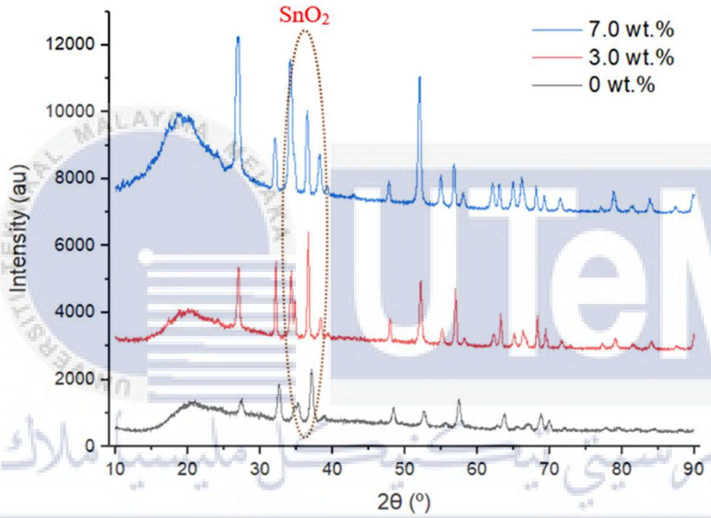


Figure 4.7 X-ray diffraction (XRD) patterns for Doped Natural Rubber with 0, 3, and 7 phr of SnO_2

4.3.5 Fracture surface morphology analysis of Natural Rubber/SnO_2 composites using Scanning Electron Microscopy (SEM)

At a magnification of 200X, SEM is used to analyse the Natural Rubber and SnO_2 filler loading's morphology. Particles, among other morphologies, can manifest as Natural Rubber during doping. Figures 4.8 and 4.9 show that the Natural Rubber can take on an agglomerated or globular image. The particle morphology of Natural Rubber was significantly changed during the production of Natural Rubber doped with SnO_2. Figure 4.9

shows that compared to the composite at 3.0 phr of filler loading, the composite at 7.0 phr of filler loading exhibited a higher level of voids and particle agglomeration. Certain voids, indicated by the red arrows in the images, were observed on the fracture surfaces of the Natural Rubber/SnO_2 test specimens as a result of the reinforcements pulling away. Higher concentrations of SnO_2, around 7.0 phr, appear to contribute to the brittle behaviour. However, the importance of the gaps can only be appreciated at the higher magnification. Recent research suggests that plastic void formation and nanoparticle debonding are key mechanisms for toughening nanoparticle/Natural Rubber composites. Figure 4.8 and Figure 4.9 show that the rubber particles were not present in the voids because they had debonded from the matrix as a result of the plastic deformation of the matrix. The inability of the Natural Rubber matrix and SnO_2 nanofiller to interact and foster good crosslinking is the root cause of the nanocomposite's failure. As a result, the presence of voids and agglomeration brought about by the non-homogenous dispersion of SnO_2 filler loading in the Natural Rubber matrix has a negative impact on the material's mechanical and physical properties.

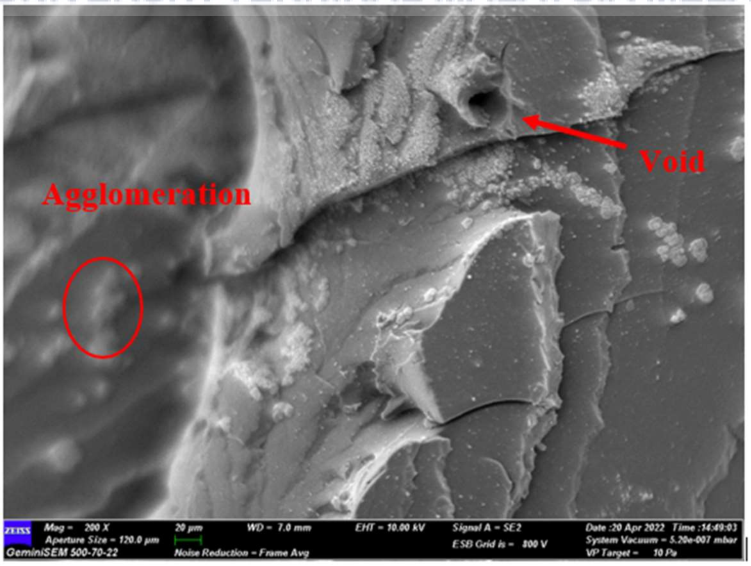


Figure 4.8 Scanning Electron Microscope Micrograph of Particle 3.0 phr of Natural Rubber/SnO_2. (Mag. 200 X)

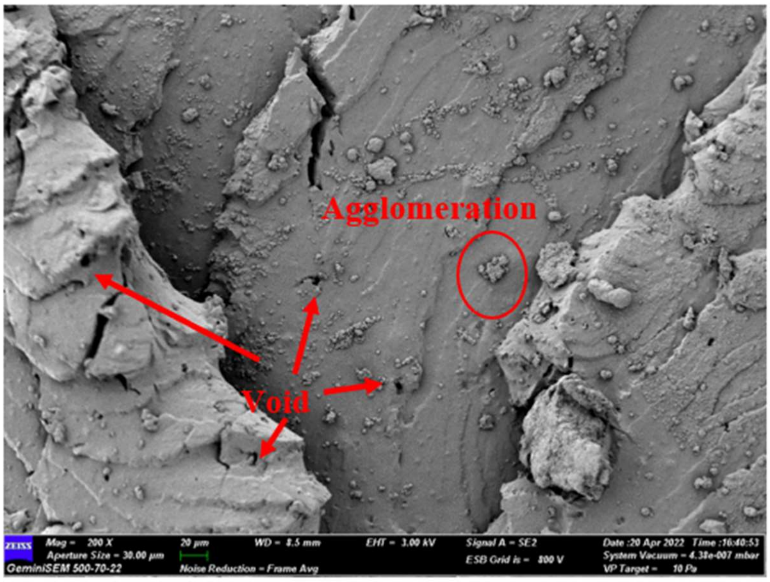


Figure 4.9 Scanning Electron Microscope Micrograph of Particle 7.0 phr of Natural Rubber/SnO_2. (Mag. 200 X)

4.3.6 Density analysis of Natural Rubber Filled with SnO2 composite.

Natural Rubber/SnO_2 composite density versus filler loading, shown in Fig. 4.10. As expected, the density results demonstrated an decraese in the pattern as a result of the increased filler loading. Theoretically, the density would decrease due to the filler loading if SnO_2 was added in greater quantities. However, the collected data at 7.00 phr showed increasing variation. This is due to SnO_2's ineffective dispersion as a filler. The problem was made worse by the Natural Rubber/SnO_2 nanocomposite's high viscosity, which may have been caused by a malfunction in the instrument's sensitivity. Due to the abundance of machines that are easily perturbed, it is common for inaccurate experimental results to be obtained. Next, based on the Scanning Electron Microscope Micrograph of Particle, the Natural Rubber/SnO_2 blend with 7.00 phr filler loading addition has the higher voids content compared to 3.00 phr. Higher void content in the Natural Rubber/SnO_2 blend with 7.00 phr filler loading resulting the increasing of its density.

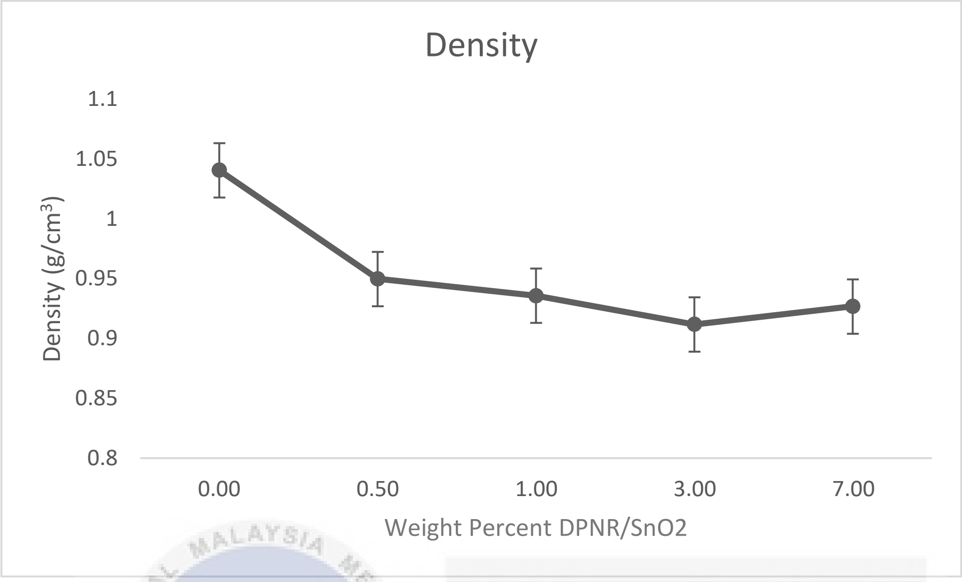


Figure 4.10 SnO_2 filler loadings effect on the density of Natural Rubber.

4.4 Summary

To be conclude that the properties of the resulting Natural Rubber composites are enhanced by the addition of SnO_2 nanofiller based on all of the experimental data. But the SnO_2 dispersion was discovered to be an important factor that needs to be controlled. The effective dispersion of SnO_2 nanofiller improved the properties of the rubber nanocomposite by strengthening the interfacial bonding between the matrix and filler. Since under ideal conditions matrix and filler loadings interact and crosslink favorably, this is the case. Agglomeration of SnO_2 due to the higher surface energy of the nanofiller, on the other hand, tends to degrade the composite properties.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

With the melt compounding and hot compress method, SnO_2 nanofiller reinforced Natural Rubber composites were successfully manufactured. The production of nanocomposites rubber with improved insulating properties led this study.

The first objective of this study is to carry out the preparation of the samples of Natural Rubber/SnO_2 with a range of different filler loadings. In this study, the raw materials that were utilised were a Natural Rubber matrix and a SnO_2 nanofiller. There are five samples that were produced with different loadings of SnO_2 nanofiller additions. These loadings are approximately 0 phr, 0.50 phr, 1.0 phr, 3.0 phr, and 7.0 phr of SnO_2 additions, respectively. Those sample were successfully mix by using two roll mill mixer and cured during hot press process. Those samples also successfully prepared with cutting process using Laser Cutting Machine to assure that the specimen was precisely clamped when the tensile test was performed.

In this study, the second objective was to conduct mechanical testing on Natural Rubber/SnO_2 composites with varying filler loadings. The mechanical testing on the samples conducted using INSTRON machine type 5960 Dual Column Testing Systems for tensile test and for shore hardness test, the samples were tested using Shore-A scale. The results from tensile test such as Ultimate Tensile Strength, Stress and Strain and Young Modulus are successfully collected. For the Shore hardness test, the data collected is the surface hardness of Natural Rubber when reinforced with different SnO_2 filler loadings.

For the last objective in this study is to optimize formulation and result of mechanical testing on Tin Dioxide reinforced Natural Rubber was successfully achieved based on all testing results. Sample with 3.00 phr of SnO_2 additions has highest ultimate tensile strength among other samples, 32.20 MPa. Young's modulus measured at 1.11 GPa have increase compared to the other sample. In addition, the FTIR peaks at 3.0 phr SnO_2, demonstrating that the Natural Rubber matrix and the SnO_2 filler loading of the nanocomposite are interacting physically and chemically. At a filler loading of 3.0 phr of SnO_2, hardness was increase. This occurred because as the rubber's elasticity decreases due to the addition of filler. In the presence of SnO_2 nanocomposite, polymerization of aniline results in an increased XRD peak, indicating that the composite structure provides strong support for a significant connection between the Natural Rubber main chain and the SnO_2 nanocomposite.

5.2 Recommendations

This study could benefit from a few recommendations for further improvement. The following are the suggestions:

i. The Natural Rubber control sample should be run first in the mix blending process to prevent any attached SnO_2 powders, as this will have an impact on the results of the other tests.

ii. More study is needed to determine the optimal compression moulding time for ensuring that SnO_2 filler loading is evenly distributed throughout the Natural Rubber matrix during vulcanization.

iii. It is advised to use the strong grip to hold the rubber during tensile testing to prevent the specimen from slipping away and affecting the measurement of tensile testing result, which would lead to poorer results.

iv. In order to get a reliable density trend, it's best to repeat the tests in the same conditions (room temperature, good atmosphere, and a consistent-sized sample). So, the density can be analysed more accurately.

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Tuan

PENGKELASAN TESIS SEBAGAI TERHAD BAGI TESIS PROJEK SARJANA MUDA

Dengan segala hormatnya merujuk kepada perkara di atas.

2. Dengan ini, dimaklumkan permohonan pengkelasan tesis yang dilampirkan sebagai TERHAD untuk tempoh **LIMA** tahun dari tarikh surat ini. Butiran lanjut laporan PSM tersebut adalah seperti berikut:

Nama pelajar: MUHAMAD HARIEZ BIN SHAARI
Tajuk Tesis: CHARACTERIZATION OF MECHANICAL PROPERTIES FOR TIN DIOXIDE REINFORCED NATURAL RUBBER FOR ELECTRICAL INSULATORS

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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Saya yang menjalankan amanah,

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