Processability of Calcined Tin Dioxide Nanoparticles DPNR (Deproteinized Natural Rubber) through Melt Compounding Method using Two Roll Mill

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BACHELOR OF MECHANICAL AND MANUFACTURING ENGINEERING TECHNOLOGY WITH HONOURS

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Faculty of Mechanical and Manufacturing Engineering Technology

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

I declare that this Choose an item. entitled " Processability of Calcined Tin Dioxide Nanoparticles DPNR (Deproteinized Natural Rubber) through Melt Compounding Method using Two Roll Mill '' is the result of my own research except as cited in the references. The Choose an item. has not been accepted for any degree and is not concurrently submitted in candidature of any other degree.

Signature : Haafiz

Name : Muhammad Haafiz Bin Rahim.

Date : 06 JUNE 2022

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 and Manufacturing Engineering Technology with Honours.

Signature : 

Supervisor Name : Sir Hairul Effendy bin Ab. Maulod

Date : 15.01.2023

DEDICATION

This report is dedicated to my beloved family in particular, for their endless love, support and encouragement. To my supervisor Sir Hairul Effendy Bin Ab Maulod who has guided me along the way to finish this project. Thank you for all your support, and give me strength until this project is finished.

ABSTRACT

Nanotechnology has become the most promising research area because of its wide-ranging applications in all scientific fields. Due to its intriguing properties, calcined tin oxide has gotten a lot of attention lately, thanks in part to the nanometer-scale synthesis that has made it even better. Many physical and chemical methods are now being used to make nanoparticles of calcined tin oxide. There are less expensive alternatives, but the synthesis involves a variety of toxic chemicals and is time-consuming and expensive. The development of a cost-effective and environmentally friendly process for its production has been spurred on by concerns about human health and the environment. In this study, deproteinized natural rubber (DPNR) were used to combine with calcined tin oxide nanoparticles (SnO2) as the fillers. This project will undergo melt compounding method and hot pressing process to produce the combination of deproteinized natural rubber (DPNR) and calcined tin dioxide nanoparticles (SnO2). Thus, this review summarises the methods used for the synthesis of tin oxide nanoparticles with deproteinized natural rubber (DPNR) and the impact on their properties. Research on the parameters and characterization methods is also discussed in this paper, along with some of the new findings that have come to light in the process.

ABSTRAK

Nanoteknologi telah menjadi bidang penyelidikan yang paling menguntungkan kerana aplikasinya yang meluas dalam semua bidang saintifik. Disebabkan sifatnya yang menarik, timah dioksida telah mendapat banyak perhatian akhir-akhir ini, sebahagiannya berkat sintesis skala nanometer yang menjadikannya lebih baik. Banyak kaedah fizikal dan kimia kini digunakan untuk membuat zarah nano timah dioksida. Terdapat alternatif yang lebih murah, tetapi sintesisnya melibatkan pelbagai bahan kimia toksik dan memakan masa serta mahal. Pembangunan proses yang kos efektif dan mesra alam untuk pengeluarannya telah didorong oleh kebimbangan mengenai kesihatan manusia dan alam sekitar. Dalam kajian ini, getah asli ternyahprotein (DPNR) digunakan untuk bergabung dengan nanozarah timah dioksida (SnO2) sebagai pengisi. Projek ini akan melalui kaedah sebatian cair dan proses penekan panas untuk menghasilkan gabungan getah asli ternyahprotein (DPNR) dan nanozarah timah dioksida (SnO2). Oleh itu, ulasan ini meringkaskan kaedah yang digunakan untuk sintesis nanozarah timah dioksida dengan getah asli ternyahprotein (DPNR) dan kesan ke atas sifatnya. Penyelidikan tentang parameter dan kaedah pencirian juga dibincangkan dalam kertas ini, serta beberapa penemuan baharu yang telah didedahkan dalam proses tersebut.

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My utmost appreciation goes to my main supervisor, Mr Hairul Effendy bin Ab Maulud, for all his support, advice and inspiration. His constant patience for guiding and providing priceless insights will forever be remembered.

I would also like to thank my beloved parents for their endless support, love and prayers. Finally, thank you to all the individual(s) who had provided me the assistance, support and inspiration to embark on my study.

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LIST OF SYMBOLS AND ABBREVIATIONS

UTeM	-	Universiti Teknikal Malaysia Melaka
DPNR	-	Deproteinized Natural Rubber
SnO_2	-	Tin Dioxide
kg	-	Kilogram
phr	-	Per Hundred Rubber
ZnO	-	Zinc Oxide
CBS	-	Sulfenamide
TMTD	-	Tetraethyl Thiuram Disulphate
°C	-	Degree Celsius
ASTM	-	American Society for Testing and Materials
°	-	Degree

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CHAPTER 1

INTRODUCTION

1.1 Background

SnO2 is the chemistry formula for Tin dioxide. The chemical compound stannic oxide is another name for tin dioxide. One tin atom and two oxygen atoms make up tin dioxide, an inorganic molecule. Tin (IV) oxide, often known as stannic oxide, is another name for SnO2. Tin oxide has the formula SnO2 as its chemical formula. The oxidation state of the tin in this compound is +4. As a result, tin (IV) oxide is the designation given to it. Tin (IV) oxide is another mineral that can be found in the Earth's crust. In its mineral form, cassiterite is an oxide of tin (IV). One of the most important tin ores on the planet is found in this location. Tin (IV) oxide compounds, whether ionic or covalent, are known by the chemical name SnO2. Basically, tin dioxide is refined by metal reduction and burning in the presence of air[1]

A solid with a melting point of 2966 degrees Fahrenheit, tin (IV) oxide is colourless and appears to be inorganic. It weighs 6.95 g/cm^3. It is not water-soluble. The rutile structure is crystallised by using Tin(IV) oxide. Tin atoms and oxygen atoms have six and three coordinates, respectively. SnO2 is typically thought of as an n-type semiconductor since it lacks oxygen in its structure. Tin dioxide is more thermally stable than SnO in terms of its chemical characteristics. It is an amphoteric acid that reacts with both acids and bases.

There is some uses of tin dioxide which is ceramic glasses are made using tin oxides.There are a number of dyes that employ tin oxide as a pigment. Next,surfaces are polished with tin oxides. By using tin dioxide, detecting gas is also possible with its help. As a catalyst, tin oxides are utilised in numerous chemical reactions, such as photosynthesis. As a lighting agent, it's a popular choice[2]

Deproteinized Natural Rubber (DPNR) is a natural rubber that has had the protein and ash removed. This rubber is produced in a controlled environment. It has a higher percentage of rubber hydrocarbons than regular natural rubber grades, which have a lower percentage [3] . Removal of non-rubber components enhances the value of the rubber in particular applications by providing unique properties. The characteristics of DPNR is it has a low protein level, which makes it unique. It has a very low levels of dirt and ash where the volatile matter content is low. It is also light-colored and no detectable amount of nitrosamines[4].

DPNR is offered in two main grades. First DPNR CV, it is between 60 and 70 Mooney units of viscosity have been stabilised. Secondly, DPNR S, the disadvantages is there is no viscosity stabilisation between 70 and 80 Mooney units, the initial viscosity is found. The DPNR S has a substantially lower storage hardening than the regular natural rubber because of the education on non-rubber content [5]

1.2 Problem Statement

Before this, calcined tin dioxide (SnO2) is produced basically by evaporation process where the Silicon (Si) are used as the substrate. The problem that occur at the end of this process is, it is difficult to produce tin dioxide due to its high melting point which is by 2966 degree Fahrenheit. A changes happened on the mechanical properties at the base material (tin dioxide) after the process.[6] As a result, this study basically is now trying to

develop new opportunity to change the available properties of Deproteinized Natural Rubber (DPNR) to a better version. In other hand, by studying this project it is able to expand the area of application that is suitable based on the new parameters or properties.

A product can no longer be made solely from Natural Rubber due to the skyrocketing cost of the raw material. To meet the needs of the consumer, the study needs a few components that can be mixed with Natural Rubber to produce better quality products. Fillers can be added to Deproteinized Natural Rubber (DPNR) to enhance its properties, according to the findings, mechanical and physical properties can be improved by using fillers like Calcined Tin Dioxide Nanoparticles that is mainly used in this study to produce a better properties.

1.3 Research Objective

The objectives are as follows:

a) To produce Deproteinized Natural Rubber (DPNR) with different percentage content of Calcined Tin Dioxide Nanoparticles (SnO2) using melt compounding method.

b) To investigate the impact for different volume of Calcined Tin Dioxide Nanoparticles (SnO2) in the formation of Deproteinized Natural Rubber (DPNR) in terms of time, temperature and torque.

1.4 Scope of Research

Through the melt compounding method, this study will investigate the "Processability of Deproteinized Natural Rubber Reinforced (DPNR) with Calcined Tin Dioxide Nanoparticle." It also examines the physical properties of Deproteinized Natural Rubber (DPNR) compounded with Calcined Tin Dioxide Nanoparticle (SnO_2) through the use of a hot press machine and mechanical tests, such as tensile testing. This study also will analyse the specimen using under optical microscope. The study of this subject may have a positive impact on the surrounding area while improvise the properties.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

An important n-type semiconductor oxide, tin oxide (SnO2) has a band gap of approximately 3.6–4.0 eV. These properties include high electrical conductance, high transparency to visible light, and strong interactions between certain toxic gas molecules and its surfaces . Transparent electrodes, liquid crystal displays, solar cells, and lithium-ion batteries are just a few of the many potential uses for SnO2 due to its unique properties. For semiconductor gas sensors, SnO2 is an important material. Nanostructures of SnO2 in various forms such as nanofibers, nanowires, and nanoparticles have been widely studied in recent years in an effort to expand their applications in the scientific community[6]

2.2 Deproteinized Natural Rubber (DPNR)

Latex from Hevea brasiliensis, the plant that produces natural rubber (NR), contains 30–40% rubber by weight dispersed as rubber latex particles in water, along with a few minor nonrubber components. There is cis-1,4-polyisoprene encased by proteins and lipids on the outside of freshly formed rubber particle latex particles. Natural rubber latex particles are thought to have a protein layer that is only found on the particle's surface. Type I immunoglobulin E, which is found in high concentrations on the surface of NR particles in the latex state, has been implicated in the development of latex allergy. Deproteinized NR latex, particularly for medical devices and hypoallergenic products, has

seen an increase in demand due to the potential protein allergy risk in NR products for sensitised users[7].

A number of different chemical and enzyme methods for deproteinizing NR have been described. By enzymatic degradation, proteins on the rubber particle's surface were effectively removed in the latex stage. As a result, the nitrogen content of NR was significantly reduced to less than 0.02%, or about 5% of the starting material. Aside from the lengthy incubation time required for the enzyme process, the remaining proteins, peptides or amino acids may cause intraoperative anaphylactic reactions in hypersensitive patients of allergy. Within one hour of treatment with urea and saponification, the total nitrogen content was reduced to less than 0.02 wt percent[8]

2.2.1 Production Process of Deproteinized Natural Rubber (DPNR)

Polypeptide-N-Resin (DPNR) is made by hydrolyzing the latex protein in field latex, which is then washed away during processing. In a stainless steel conical bottom reaction tank, ammonia, a non-ionic surfactant, proteinase, and hydroxylamine Neutral Sulphate (HNS) are added to bulked field latex and allowed to react for 72 hours. A steam column coagulator designed specifically for this purpose is used to coagulate the reacted latex after it has been neutralised with diluted formic acid and the enzymatic hydrolysis reaction is complete. [9] The coagula that is formed are then continuously processed through a series of crepers before being shredded into small crumbs in a shredder to finish. Before entering the dryers, the crumbs are pumped through a static screen to remove any foreign matter. A hot air dryer is used to dry the wet crumbs for about six to eight hours at about 85oC. It is after cooling that the SMR standards are met for the packaging of the baked goods[10]

2.2.2 Properties of Deproteinized Natural Rubber (DPNR)

Deproteinized Natural Rubber (DPNR) is characterized as a material that is extremely low concentration of protein. It is because during the process from natural rubber, it already remove the protein that have in the natural rubber. There is also very little grit and ash content. Deproteinized natural rubber (DPNR) also have a low level of volatile matter with a palish colour. There is also no detectable amount of nitrosamines. The antigen content is also extremely low[11]

2.2.3 Grades of Deproteinized Natural Rubber

There are two levels of DPNR training available. In the first DPNR CV, the viscosity has been stabilised between 60 and 70 Mooney units. DPNR S, on the other hand, suffers from a lack of viscosity stabilisation between 70 and 80 Mooney units. The DPNR S has a significantly lower hardening rate than regular natural rubber because of the increased awareness of non-rubber content [11]

2.3 Tin Dioxide Nanoparticles

The wide range of applications of tin oxide semiconductors, such as gas sensors, transistors, electrodes, liquid crystal displays, catalysts, photovoltaic devices, photo sensors, antistatic coating has led to an increase in recent research. It is one of the most important materials because it has a high degree of transparency in the visible spectrum, strong physical and chemical interactions with adsorbent species, a low operating temperature, and strong thermal stability in air up to 5000 °C. Two types of tin oxides are possible because the metal exists in two oxidation states +2 and +4, leading to the formation of both stannous oxide (SnO) and stannic oxide (Sn02). Sn02 is the more stable of the two oxides [12]

Sol Gel, Microwave technique, Solvo-thermal, Hydro thermal, Sonochemical, Mechanochemical, Co-precipitation and other methods have been used to synthesise Sn02 nanoparticles, which have been used in various applications. Co-precipitation has been used to produce Sn02 nanoparticles that are extremely pure and crystallised. It's easy, cheap, and doesn't call for high temperatures or pressures to use this Co-precipitation method Controlling particle size and shape can be done by changing the pH of the medium, increasing the amount of a specific precursor, or precipitating reagents. Filtration and repeated washing of the precipitate removes any remaining impurities [6]

2.3.1 Properties of Tin Oxide Nanoparticles

XRD, or X-ray diffraction, can be used to determine the structural properties of any material. XRD is used to determine the structure of Tin oxide nanoparticles in the angle 2? range of 20° to 70°. These Sn02 nanoparticles have a crystalline structure and are only 36 nm in diameter.

Properties: Tin Nanoparticles

Molar Mass: 150.71 g/mol

Melting point: 1630 °C

Boiling point: 1900 °C

Density: 6.95 g/cm^3

Analyzing the morphology of Sn02 Nanoparticles is done using an SEM. SEM is a scanning electron microscope. SEM reveals a spherical morphology with a foam-like

structure. Laser absorption measurements were performed on nanoparticles of SnO2. Between 300 and 800 nm, the optical absorptivity coefficient has been calculated. In this case, the absorption edge is found at a lower frequency. The quantum confinement of the nanoparticles may be responsible for the widening of the absorption spectrum. In optical devices, Sn02 nano parts are promising because of their excellent crystallinity and strong blue emission. Sn02 nanoparticles have an absorbance peak at 315 nm, according to their UV absorption spectrum.

The dielectric studies demonstrate the influence of temperature and frequency on the conductivity phenomenon in nanostructured materials. It is possible to gain useful insight into the electrical properties of the grain boundaries by making use of the dielectric behaviour. The electronic, ionic, dipolar, and space charge polarizations are the primary contributors to the dielectric properties of materials. Space charge polarization also plays a role. The electronic polarization, which can be found in the optical frequency range, is the most essential controlling factor when it comes to the bulk form of polycrystalline materials[2].

Polarization of space charge arises from molecules possessing a persistent dipole moment that, when subjected to an electric field, is capable of undergoing a shift in its orientation. The fundamental electrical properties of Sn02 nanoparticles are referred to as the dielectric parameters. These dielectric parameters include the dielectric constant and the dielectric loss. The electric processes that take place in Sn02 Nanoparticles can be revealed by the measurement of the dielectric constant and loss as a function of varying frequencies and temperatures [1]

2.3.2 Method Preparation of Tin Oxide Nanoparticles

Nanoparticles with precise dimensions, shapes, structures, and chemical compositions are vital for the development of new technologies in the field of nanoscience. In order to synthesise tin oxide nanoparticles, (SnO2NPs) a variety of chemical, physical, and biological methods are employed.

Physical Method

Physicochemical processes such as spray pyrolysis, chemical vapour deposition, high-energy ball milling, and hydrothermal can also be used to make SnO2NPs. It has been reported that spray pyrolysis can be used to produce SnO2NPs and F-doped (F- SnO2NPs) films. F and SnO2 were synthesised using ammonium fluoride and tin chloride as precursors and hosts, respectively. A 1:4 methanol/deionized water mixture was used to dissolve the precursors and the host. 1 h was spent using a magnetic stirrer to keep the mixture moving. Spraying the resulting solution onto the heated glass substrate was the final step. Sprat interval and time were kept constant at 7 seconds and 0.6 millilitres per minute (mL/min), respectively. The surface morphology of F- SnO2NPs was studied using FESEM.

Zhang et al. used chemical vapour deposition on carbon cloth at 750 °C to produce novel SnO2NPs as nanosheets. Thermal chemical vapour deposition was used to create SnO2NPs in a horizontal quartz tube furnace with an inner diameter of 60 mm and a length of 100 cm. This carbon cloth portion in the porcelain boat was positioned downstream of the tube for safety reasons. A quartz tube was heated to 60°C for 130 minutes and kept there. After that, the furnace was cooled down to normal operating temperatures. SEM was used to analyse the SnO2NPs' morphologies. Several nanosheets

were used to assemble the flower-like structure of SnO2 NPs. SnO2NPs structures are flower-like because the surface energy of the freshly created nucleus is minimised, allowing it to "land" on a generated sheet and grow to another layer.

The form and structure of nanoparticles are usually thought to be greatly influenced by ball milling. Ball-milling of several nano oxides was carried out by Begin-Colin et al., who found that the milling media had a significant impact on the transition process of the various oxides. It was discovered that the size of the SnO2NPs was lowered when steel balls were used during the preparation process. Ball milled Fe and SnO2 powders were used to create new fibres, according to Butt and colleagues. Catalyst-assisted chemical vapour deposition with Ar gas for 180 minutes at 1100 C produced the result on a Si substrate. SnO2NPs nanofibers can be synthesised using a vapour-liquid-solid method based on the growth process. Nanoparticles generated via the vapour-liquid-solid process are dense and uniform in growth. Samples from the tetragonal rutile phase of SnO2NPs nanofibers were determined by XRD analysis. The diffraction peaks (77–0447) were indexed using the standard code. SEM images show that SnO2NPs have an average diameter of 95–100 nm. SnO2NPs accumulate consistently and densely on top of the substrate. Several experts agree that this morphology has never been observed before. Gas diffusion is slowed as particle size shrinks[13].

Nanostructures are great candidates for sensing devices because of their inherent limitations. Compared to nanowires and thin films, this nanomaterial is projected to be more effective. At ambient temperature, Guo et al. reported the synthesis of mesoporous SnO2NPs by a hydrolysis technique involving cetyltrimethylammonium bromide (CTAB) as the structural-directing agent and ammonia (NH3) as the alkali source. SnO2NPs containing CTAB and calcined at 300 C showed organised mesoporous frameworks with

an average particle size of about 4.2 nm in the XRD spectra. The results of the gas-sensing experiment show that at a temperature of roughly 300 °C, the gas is detected. For example, the SnO2NPs sensor has a faster reaction rate to ethanol than other gases, such as CO, H2, NH3, Hexane, and Methanol at varied operating temperatures between 160 to 350 degrees Celsius. The sensor's response to ethanol is significantly higher than the sensor's response to CO, H2, NH3, hexane, and methanol at temperatures above 300°C, and the sensor's response to ethanol is much higher than the sensor's response to the other measured gases in the concentration range of 0.05 vol% to 0.3 vol%. This indicates that the ethanol sensor has a high level of selectivity. Using the sensor at temperatures ranging from 160 to 350 C and with ethanol concentrations between 0.05 vol% and 0.3%, the mesoporous SnO2NPs sensor may be used to detect small amounts of ethanol in the environment [14]

Chemical Method

To synthesise colloidal SnO2NPs, organic and inorganic reducing agents are among the most efficient ways due to their simplicity and equipment requirements. Because chemical procedures are easy and affordable to undertake, they have an advantage over other methods. In the synthesis of, the solvents are crucial. SnO2NPs. In this section, various chemical approaches for synthesising SnO2NPs will be discussed. Ammonia was used to synthesise SnO2NPs, Co-precipitation of precipitators, Triton X-100, and tin chloride the way SnCl4 solution was mixed with Triton X solution and ethanol until a clear solution was achieved after 30 minutes of stirring acquired. Drops of NH4OH solution were added in the following manner: Stirring to maintain an ideal pH of 8.0. An ultrasonic transducer will be used after that the homogenous solution was achieved by applying a wave to the mixture 30 minutes The size of SnO2NPs will increase as the PH value rises

from 6 to 9. The precipitate was collected using a filtration process by washing it with ethanol and distilled water three times. Results of XRD analysis the tetragonal structure was clearly visible in each peak the SnO2NPs data results from SEM showed that the SnO2NPs had a structure smaller than 100 nm in diameter. SnO2NPs were synthesised quickly using a similar approach by Moghadam et al. and a simple co-precipitation technique. Ammonia was utilised as a fuel. Bis ethylene served as a capping agent rather than a precipitating agent[13].

Tetrabutylammonium bromide (TBAB) and tetrapropylammonium bromide (TPAB) were used in an organic medium to synthesise SnO2NPs utilising an electrochemical reduction technique. An ACN/THF mixture with a ratio of 1:4 was developed by adjusting the molecular concentration and current density of the ligand. Over the course of two hours, an inert nitrogen environment is used to conduct the reduction process. The sacrificial anode, commercially available in tin metal sheet and platinum (Pt), is used as a cathode in an electrolysis cell. Stabilizer concentration, solvent polarity current density, and distance between electrodes are all used to alter the size of SnO2NPs in this experiment. UV-visible spectra showed that as the current density increases, the absorption edge widens, the SnO2NPs' diameters drop, and the energy gap band widens. These generated nano-sized SnO2NPs were confirmed by a scanning electron microscope (SEM). The SnO2NPs were generated by hydrolysis technique of tin isopropoxide in the presence of p-toluenesulfonic acid and acetylacetone[15].

Magnetic stirring in acidic aqueous was used to ensure that the early hydrolysis ratio was 10 and the acidity ratios ranged from 0 to 0.6 during the hydrolysis process. Systems were matured in an oven (60°C) for around 24 hours after being stirred for 2 hours at room temperature. Surface complexation atoms are anticipated to provide SnO2NPs

with anti-aggregation protection via acetylacetonate ligands. Gas sensing films based on SnO2NPs and their derivatives are presently being investigated for usage. Precursor solution pH (6–12) was varied and heated at various temperatures to synthesis SnO2NPs using the sol-gel method. SnO2NPs at 400 °C have a spherical form with a minor agglomeration. An increase in band gap value of roughly 4.5 eV is seen at pH 8 for the SnO2NPs. When the annealing rate was increased to 600 and 800 degrees Celsius, the SnO2NPs orthorhombic system produced mixed phases. It has been found that different annealing temperatures and pH solutions have altered the band gap's value [10].

In SnO2NPs, the absorption peak at 278 nm is modified from the value of 337 nm due to the effect of quantum confinement. Ohmic contact is studied in detail in the I–V study. SnO2NPs have a 1.6 M resistance value, which suggests that they could be useful as a sensing material. SnO2NPs and SnO2/polyaniline (SnO2NPs/PANI) nanocomposite were synthesised through an in-situ polymerization method, according to Shukla et al. Precursors included SnCl2 and NaNO3-KNO3.

The methanol/aniline combination was used to extract the sulphide ions from the SnO2. A clear solution was formed after 30 minutes of stirring the mixture on a magnetic stirrer at room temperature. Synergy between the surface properties of composite materials could be a useful strategy for developing more tunable sensing platforms, as demonstrated by isothermal TG results a single gaseous sensing model and surface energy molecules. Humidity- resistant nanocomposite, SnO2 improved linearity, sensitivity, and response time for electro- chemical sensing[14]

Biosynthesis of Tin Oxide Nanoparticles (SnO2NP's)

It is now possible to synthesise new materials using green chemistry techniques that do not harm the environment and are safe for humans and animals. SnO2NPs can be prepared using a simple procedure. Washing and drying plants prior to experimentation is the first step, followed by collecting and separating the sections that will be used in the experimentation. It is possible to boil the plants in purified water in certain circumstances. The plants can be crushed and dispersed. In order to obtain a pure plant extract, the solution can be drained to remove any fibre or suspension. SnO2NPs can be made by adding varying amounts of the precursor to plant extracts, or by calcining the extracts at high temperatures (600–800 C) to form SnO2NPs. SnO2NPs have been synthesised from a variety of plant species with great success. It is not uncommon to see the use of plant parts such as seeds, ripe fruits and twigs and peels. In terms of size/diameter, the synthesised SnO2NPs ranged from 2 to 47 nm, and the majority of them were round. antifungals and antibiotics as well as photocatalysts, sensors, antioxidants, and antimicrobials. Boiled extract was combined with SnCl2 and heated for six hours at 60 degrees Celsius to produce SnO2NPs [16].

Drying for 8 hours at 75 °C followed the centrifugation of precipitation. Different temperatures were used to treat the dry resultant. Scientists have successfully created SnO2NPs with a sphere shape between 3 and 6 nm in small scales. As the temperature increased, the XRD peaks of SnO2NPs became narrower and more pronounced, suggesting that the crystallinity of SnO2NPs increased as well. Despite this, the authors found that as the temperature rose, so did the particle size, as demonstrated by their TEM study. Absorption spectra have been extrapolated to measure the bandgap. The bandgap decreases with temperature, starting at 4.3 eV and ending at 3.90 eV. Elango and Roopan synthesised

SnO2NPs from Cymphoma betacea methanolic extract. The prepared SnO2NPs have a spherical morphology ranging from 20 to 40 nm.

TEM.50 nm is the smallest possible size. It was revealed by the SEM image that the SnO2NPs clustered with sharp peaks. In a different study, chemical compound that exists in Camellia sinensis serves as an example of flavonol derivatives in the preparation of SnO2NPs, capping agent and stabiliser As a result of their efforts, Spiral NPs were created using TEM analysis to verify their spherical shape particle size ranges from 5 to 30 nm. In addition, the peaks of the XRD analysis have been detected, indicating the emergence of microscopic matter. Rutile-like SnO2NPs were synthesised using this method and found to be tetragonal. SnO2NPs were synthesised using Calotropis gigantean leaf extract, according to Bhosale et al. For six hours at 60 degrees Celsius, the leaf extract was mixed with SnCl2. At 300 degrees Celsius, the nanoparticles were annealed. According to the Debye–Scherer equation, SnO2NPs have a crystalline average size of 35 nm. SnO2NP agglomerations appeared irregular on the TEM image. The agglomeration, according to the authors, was primarily caused by the production of small NPs[13].

The XRD analysis, on the other hand, proved that rutile crystallisation had occurred. Researchers have also used arginine, glycine, and glucose as biocompatible capping and reducing agents to synthesise SnO2NPs. Green and simple are two terms used to describe these approaches. To further aid in NP size and shape control, biomolecules were used as capping agents and reducers[17]

2.3.3 Applications of Tin Oxide Nanoparticles

The wide range of applications of tin oxide semiconductors, such as gas sensors, transistors, electrodes, liquid crystal displays, catalysts, photovoltaic devices, photo sensors, antistatic coating has led to an increase in recent research. It is one of the most important materials because it has a high degree of transparency in the visible spectrum, strong physical and chemical interactions with adsorbent species, a low operating temperature, and strong thermal stability in air up to 5000 °C. Two types of tin oxides are possible because the metal exists in two oxidation states +2 and +4, leading to the formation of both stannous oxide (SnO) and stannic oxide (Sn02). Sn02 is the more stable of the two oxides.

Sol Gel, Microwave technique, Solvo-thermal, Hydro thermal, Sonochemical, Mechanochemical, Co-precipitation and other methods have been used to synthesise Sn02 nanoparticles, which have been used in various applications. Co-precipitation has been used to produce Sn02 nanoparticles that are extremely pure and crystallised. It's easy, cheap, and doesn't call for high temperatures or pressures to use this Co-precipitation method Controlling particle size and shape can be done by changing the pH of the medium, increasing the amount of a specific precursor, or precipitating reagents. Filtration and repeated washing of the precipitate removes any remaining impurities.[18]

CHAPTER 3

METHODOLOGY

3.1 Introduction

This section presents the methodology of this review. Part three contains the project planning, process flow and Gantt chart. With a specific end goal to accomplish the targets that have been resolved, the project should follow the rules to guarantee that the struggles poured into completing this project do not stray from the targets. In this section, we will examine about project planning and the testing procedure. A through method examination were directed on the research of the processing of calcined tin dioxide nanoparticles reinforced DPNR (Deproteinized Natural Rubber) through melt compounding method. Figure 3.1 shows the complete processes that were followed in order to complete the study.

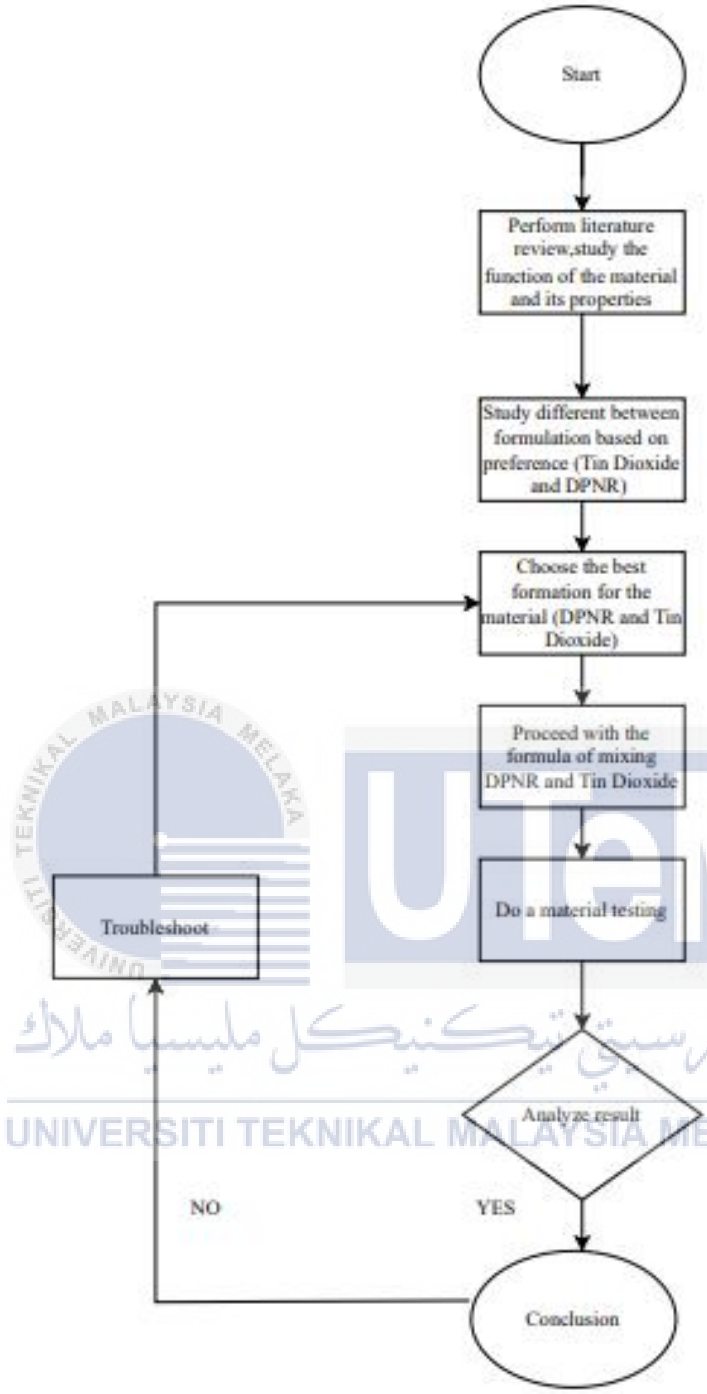


Figure 3.1 : Flow Chart of Methodology Process

3.2 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	PSM Briefi -ng								M I D T E R M B R E A K						
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															

Table 3.1 Activities and tasks perform during PSM 1 and PSM 2.

Planning Actual

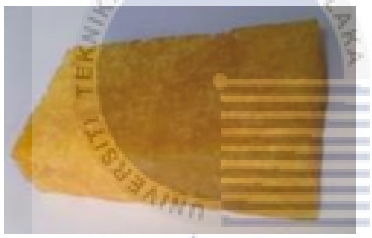
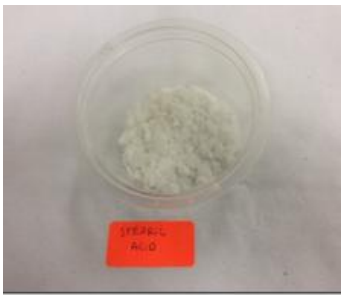
Project Activity	2022														
	Week (Semester 7)														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Report review 2									M I D T E R M B R E A K						
Findings															
Data Analysis															
Report writing PSM 2															
PSM 2 report submission															
Slide preparation															
Presentation – PSM 2															
Final Semester Examination															

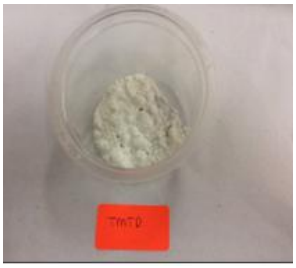
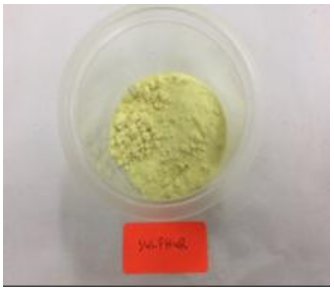
3.3 Material preparation

Deproteinized Natural Rubber (DPNR) and tin dioxide (SnO2) is the main material used in this study. In order for the complete final year project, Universiti Teknikal Malaysia Melaka (UteM) have fully provided the materials. All of the materials details have already been explained in Chapter 2, Literature Review

3.3.1 Preparation of Deproteinized Natural Rubber (DPNR) compound

Table 3.2 Materials for Deproteinized Natural Rubber (DPNR) compound.

Deproteinized Natural Rubber (DPNR) 	Tin Dioxide 
Stearic Acid 	Zinc Oxide 

TMTD 	Sulphur 
6PPD 	CBS 

Compound that was made using by 100 phr of Deproteinized Natural Rubber (DPNR) with a various amount of Tin Dioxide (SnO2) at 0 w %, 0.5 w %, 1 w %, and 3 w % of the total weight. All of these formulated rubber based is called for the same amount of curing additives, which were as follows 1.5 of sulphur (s) , 5 phr of zinc oxide (ZnO), 2 phr of stearic acid, 0.3 phr of tetraethyl thiuram disulphate (TMTD), and 2 phr of 6PPD.

3.4 Sample Fabrication

The sample of fabrication proceeds by compounding deproteinized natural rubber (DPNR) and tin dioxide (SnO2) in the internal mixer. Weight percentages (wt %) of loading and the overall mixing proportion is two parameters that need to be considered during the compounding process.

3.4.1 Mixing flow process

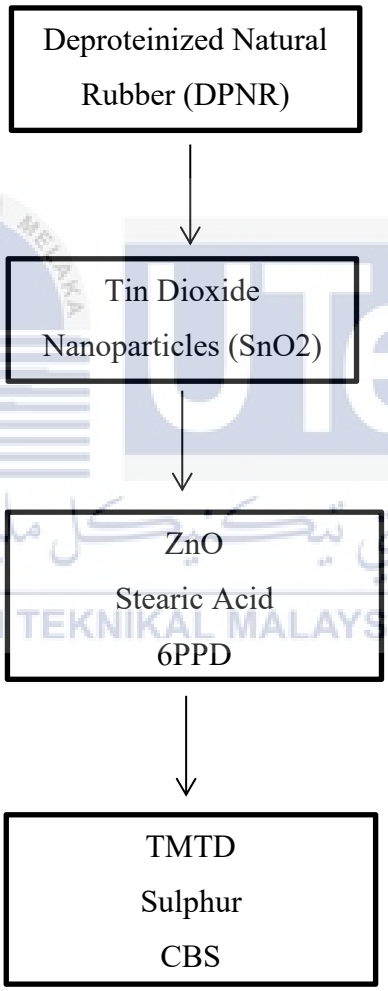


Figure 3.2 Mixing process

Figure 3.2 shows the process for mixing Deproteinized Natural Rubber (DPNR) and Tin Dioxide into a single substance. Deproteinized Natural Rubber (DPNR) and Tin Dioxide were combined in a HAAKE Rheomix OS for the purpose of mixing the material compound. To begin, the internal 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, 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 Machine

Deproteinized natural rubber (DPNR) were mixed with Tin dioxide (SnO2) by using the two roll mill. During the first two minutes, the mixer will only used to mix the Deproteinized Natural Rubber (DPNR). Next, tin dioxide will mix together with the deproteinized natural rubber (DPNR). After mix it, zinc oxide (ZnO), stearic acid, and 6PPD will be added into those mixture. For the last process, TMTD, sulphur, and CBS were added to the mixtures and the process compounding is done.[19]

Figure 3.3 Two Roll Mill Machine

Table 3.3 Formulation of Deproteinized Natural Rubber (DPNR) compound

SAMPLE	1	2	3	4	5
DPNR	100	100	100	100	100
Tin Dioxide	0	0.5	1	3	7
Zn0	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

In order for deproteinized natural rubber (DPNR) and tin dioxide can be mixed together, the formula was broken down into five primary compositions of per hundred rubber units (PHR). The material was then precisely weighed using an analytical balance.

3.4.3 Hot Press Compressing

After deproteinized natural rubber (DPNR), tin oxide (SnO2) , and all ingredients undergoing a compounding process using a Haake internal mixer at specific parameters, it will then be hot-pressed using a hot press machine. The sheets of deproteinized natural rubber will be obtained. It is then carefully poured into the mould cavity of the hot press machine using deproteinized natural rubber (DPNR) melt compounding. To create vulcanised deproteinized natural rubber (DPNR) sheets, a hot press compression vulcanization process was used.The pre-vulcanized compound will then be compressed in order to create a vulcanised sample sheet. The compound of desire was weighed and then put into a mould[20].

Figure 3.4 Hot press machine

Table 3.4 Parameter of Hot Press Compression Process

Parameter	Value
Mould	10cm X 10cm X 0.2cm = 20cm³
Preheat Temperature	150 °C
Preheat Sample Time	1 Minutes
Compress Pressure	140 kg/cm²
Compress Time	5 Minutes
Cooling Time	1 Minutes

3.4.4 Cutting Process

The shape was created by cutting the material. With the help of FTK UTeM's onsite laser cutting machine, the process of cutting was completed. There is no need for additional abrasive materials when using the laser cutting machine because it is able to cut through a 3 millimetre (mm) thick workpiece without the use of any additional abrasive materials.

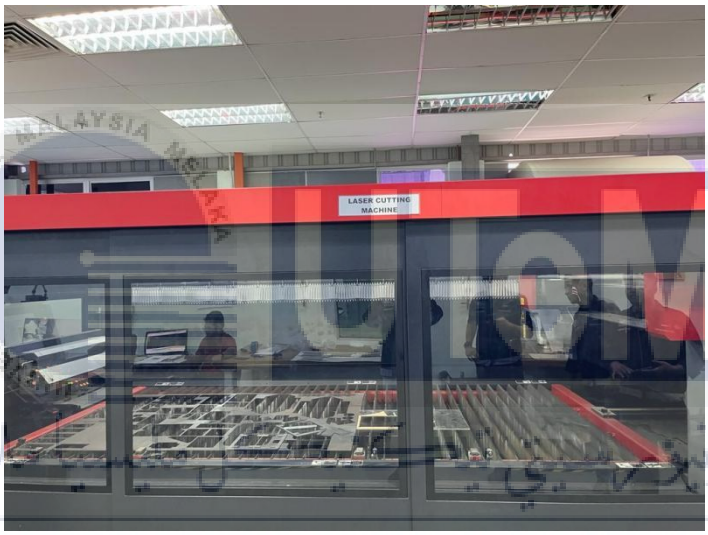


Figure 3.5 Laser cutting machine for cutting process

3.5 Analysis

In this experiment, there is just one analysis used in this study which is used to observe the microstructures of the deproteinized natural rubber (DPNR) with tin dioxide.

3.5.1 Density Test

A density test was carried out in order to determine the apparent density of the material. The testing apparatus that was measured in accordance with ASTM D792 can be seen in Figure 3.6. It was determined that five samples of each different filler loading

should be used, and the average value was given. Consequently, the density could also be determined through computation, as shown in the following Equation, in which m(g) represents a mass and v(m3) represents a volume.

Density, $\rho(g/m^3) = (m/v)$

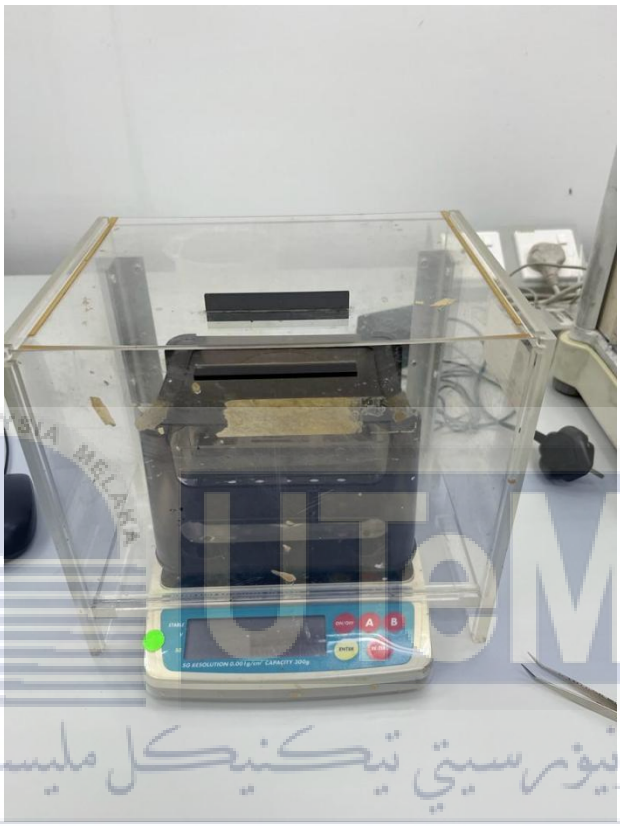


Figure 3.6 Electro Densimeter MD- 300S Machine

3.5.2 Fourier Transform Infrared Spectroscopy (FTIR)

For the identification of functional groups and the characterization of covalent bonds, FTIR is an effective analytical technique. In this study, FTIR is used to reveal the presence of functional groups in the deproteinized natural rubber after been mixed with calcined tin dioxide. The spectrum of the sample was obtained using an IR spectrometer (Nicolet 6700 AEM), as seen in Figure 3.7. Jigsaw was used to acquire the samples. The samples measure 10mm (L) x 10mm (W) x 3mm (T).

Figure 3.7 Fourier Transform Infrared Spectroscopy (FTIR) Machine

3.5.3 Scanning Electron Microscope

A morphological or microstructure surface will be analysed using SEM analysis. Figure 3.8 depicts a Zeiss EVO-50 SEM machine that could be used to carry out the analysis. The cross-sectional area of the samples should be clean prior to observation. SEM images would be taken at magnifications ranging from 100x to 1500x for all samples[23].

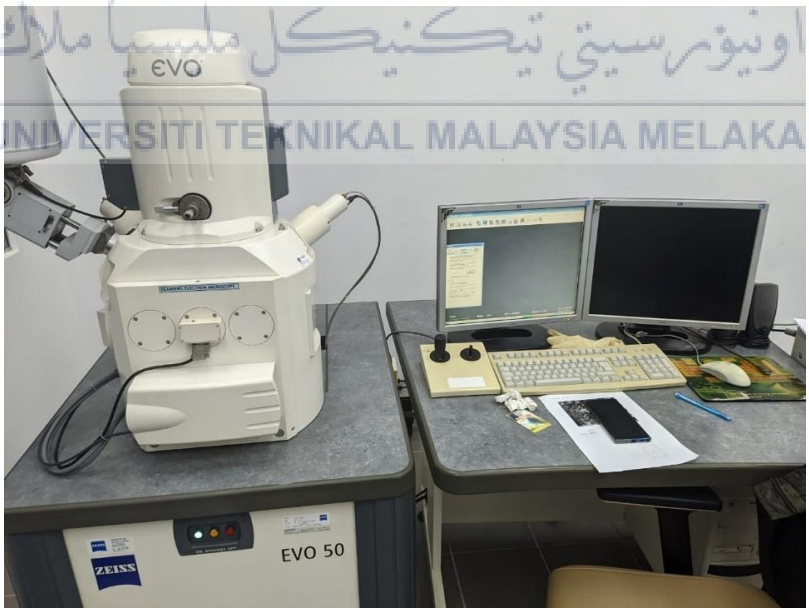


Figure 3.8 Scanning Electron Microscopy (SEM) Machine

3.6 Expected Outcome

This chapter discusses a method for processing calcined tin dioxide nanoparticles with deproteinized natural rubber (DPNR) that has been proposed as a new, efficient, and all-encompassing approach. Furthermore, the ease with which it can be utilised and manipulated on a large-scale distribution network. An effective and integrated working strategy, followed by a thorough testing procedure and data collection process, is expected to complete this project's methodology in its entirety. Hoping for a further understanding while studying this project with analyzing the material properties and optimizing the parameter as much as possible.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

The material has undergone mechanical testing and analysis as was discussed in Chapter 3. Different experiments performed during the course of the study period are shown and discussed in this chapter. Many findings were uncovered through this investigation and are discussed below.

4.2 Cure characteristics of DPNR and Calcined SnO2

In Table 4.1, you can see how different fillers affect the DPNR curing process. Curing characteristics at 150 degrees Celsius for SnO2 filled DPNR compounds are provided, including minimum torque (ML), maximum torque (MH), scorch time (Ts2), and 90% cure time (T90). Table 4.1 displays the results of a study that compared the vulcanization properties of various rubber compounds by measuring the torque-time relationship. Results showed that 7.0 wt.% filler loading produced the highest minimum torque in comparison to any other filler loading (ML). To increase torque, try using a filler loading of 7% by weight, which results in a sample with better strength and stiffness. This happens because there is a limit to how freely molecules can move inside the sample.

Besides, the scorch time reveals how long a rubber compound may be operated at a certain temperature before curing. It is apparent that the inclusion of filler greatly lowered the scorch time. When filler loading increased, that shorter scorch time of the DPNR

compounds have been slowed. 7.0 wt% filler loading value of scorch time (Ts2) increased. It might because of the accelerator contained sulphur so that the scorch time was lengthened. Greater polarity content in DPNR interact with the DPNR and calcined SnO2 content, where it results slows the vulcanization process.

The CRI discovered that curing efficiency may be greatly improved by increasing the SnO2 filler loading. As an additional accelerator, SnO2 allowed for a greater concentration of reactive sites on the rubber molecules used in the cross-linking procedure. The cross link density of vulcanized DPNR was affected by the filler loading. As there is a lot of sulphur on the DPNR chains, this was linked to the fact that SnO2 acted as an additional accelerator during the curing of the DPNR compounds. Because of this connection, the cross link density increased. So, the cross link density and stiffness of the vulcanized rubber are connected to the torque. An increase in torque indicates that the rubber composites' viscosity and modulus have improved as a result of filler being incorporated into the DPNR matrix. The higher the torque, the shorter the curing time will be due to the higher cure rate index .

Table 4.1 Curing Properties all compounds

Type(phr)	T2 (Min.Sec.)	T90 (Min.Sec.)	ML (dNm)	MH (dNm)	Δ Torque (dNm)	CRI(min^{-1})
Control(1)	1.48	2.28	3.83	21.32	17.49	150.00
Control (2)	1.49	2.29	3.70	21.40	17.70	150.00
Control Avg	**1.49**	**2.29**	**3.77**	**21.36**	**17.60**	**150.00**
0.5 (1)	1.54	2.35	3.77	21.51	17.74	146.34
0.5 (2)	1.54	2.33	4.53	22.39	17.86	153.85
0.5 Avg	**1.54**	**2.34**	**4.15**	**21.95**	**17.80**	**150.09**
1.0 (1)	1.50	2.30	3.56	21.55	17.99	150.00
1.0 (2)	1.50	2.33	3.76	22.32	18.56	139.53
1.0 Avg	**1.50**	**2.32**	**3.66**	**21.94**	**18.28**	**144.77**
3.0 (1)	1.47	2.27	3.71	22.02	18.31	150.00
3.0 (2)	1.46	2.24	4.01	22.10	18.09	157.89
3.0 Avg	**1.47**	**2.26**	**3.86**	**22.06**	**18.20**	**153.95**

7.0 (1)	1.51	2.34	4.15	23.38	19.23	153.95
7.0 (2)	1.47	2.28	4.15	22.99	18.85	146.34
7.0 Avg	**1.49**	**2.31**	**4.15**	**23.19**	**19.04**	**142.94**

4.3 Mechanical and physical characteristics of DPNR filled with Calcined SnO_2

Mechanical and physical characteristics been analyze based on the Density and Fourier Transform Infrared Spectroscopy (FTIR).

4.3.1 Density analysis of DPNR Filled with Calcined SnO_2

The density of the DPNR/SnO2 nanocomposite is shown in Figure 4.1, and it varies depending on the filler loading. The addition of filler loading led to an increase in density, as evidenced by the results, which showed that the pattern had grown. In all of the theoretical aspects, increasing the amount of SnO2 addition would result in a lower density due to the fact that the nature of the filler loading is hollow. Despite this, the data obtained suggested a growing amount of variation. This is because SnO2 is a filler that has poor dispersion characteristics which it make the problem more worse by the excessive viscosity of the DPNR/ Calcined SnO2 , which may have been the result of a malfunction in the technology associated with the sensitivity of the weighing balance machine. The excessive number of equipment that are susceptible to vibration from their surroundings, which results in inaccurate experimental data. Following that, DPNR/SnO2 with 7.00 wt.% filler loading addition has the highest voids content due of the excessive viscosity of the SnO2 that was filled in DPNR. As a consequence of this, the nanofiller was not distributed very

well throughout the matrix, which led to the generation of samples with significant voids restricted to their outermost layers.

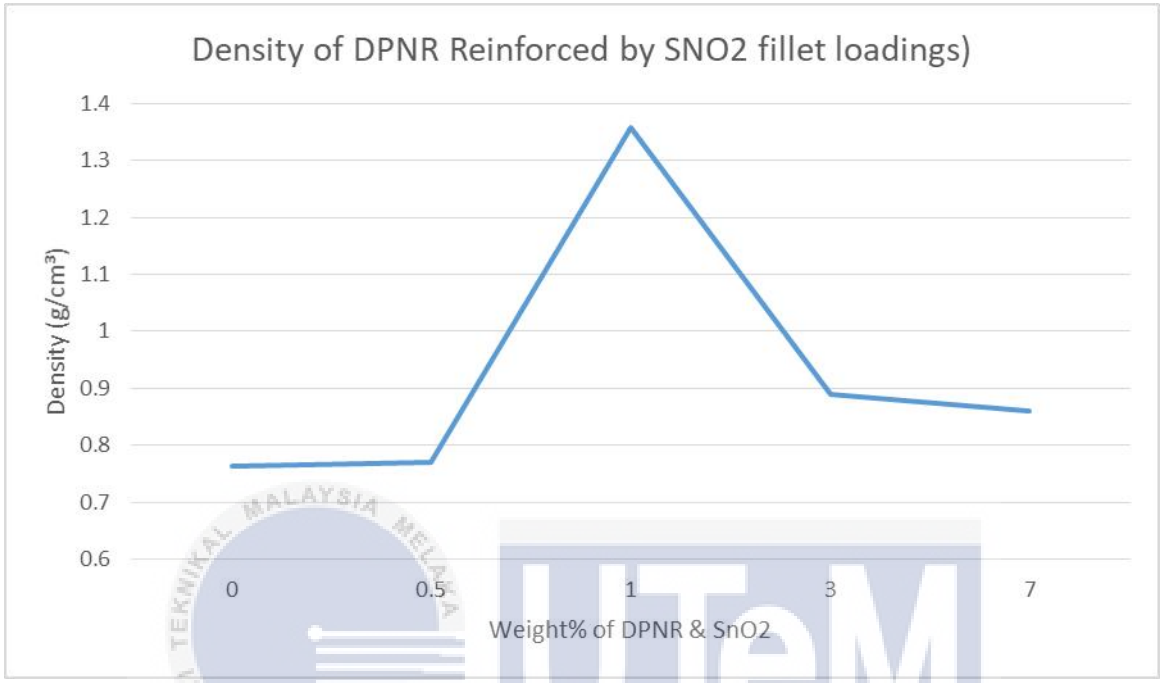


Figure 4.1 : Density of DPNR reinforced by SnO_2 filler loading

4.3.2 Fourier Transform Infrared (FTIR) analysis

By using FTIR analysis, the chemical structure of Calcined SnO2 with DPNR with various filler loading was been analyzed with the wave number range between 400 - 40000 $cm^{-1.}$ with the scanning speed of $2mm^{-1}s$. The results of this research are depicted in figure 4.2 and 4.3. The filler loadings of 1 weight percent(%) and 3 weight percent(%) were corresponding to the broad peak in the wavenumber range of 2000-2500 cm^{-1}. The peaks at 2000, and 2300 c^{m-1} can be attributed to the SnO2 that filled the matrix of DPNR. As a result, every ascending peak was connected to nanofiller groups like SnO2 filler loadings in DPNR. According to the findings, the peak demonstrates that there is neither a physical interaction nor an interaction involving chemical bonding between the DPNR matrix and the SnO2 filler loading in the nanocomposite. An increase in band intensity or a shift to

lower wavenumber values was most likely incorporated into the matrix by an activator-enhanced diffusion process and/or by the changes in polymer structure caused by the activating agent that were observed. Alternatively, the increase in band intensity was most likely incorporated into the matrix by an activator-enhanced diffusion process.

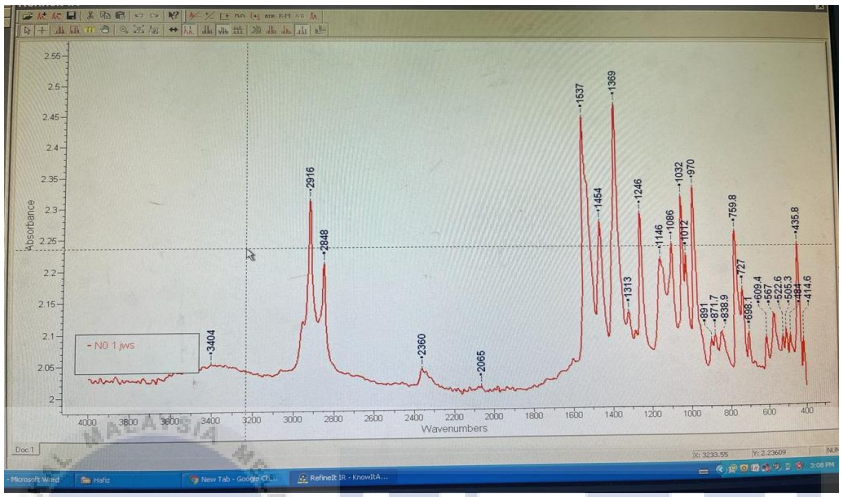


Figure 4.2 : FTIR Spectra of DPNR reinforced by SnO_2 filler loading (1wt%)

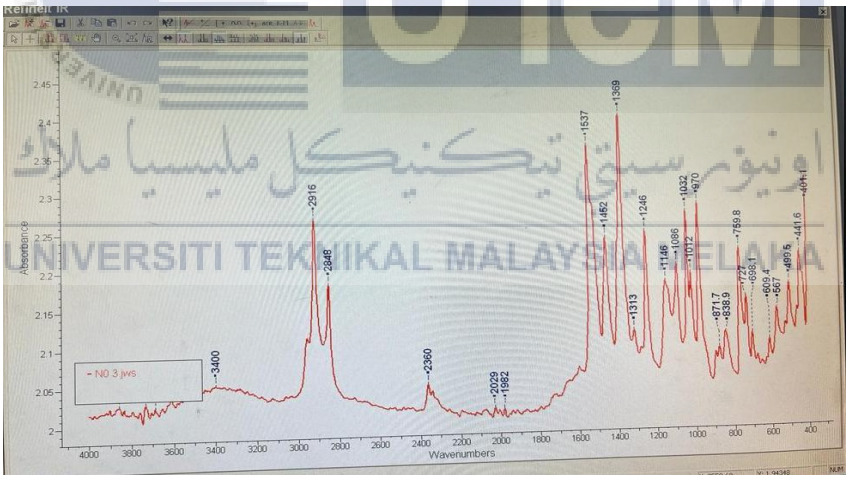


Figure 4.3 : FTIR Spectra of DPNR reinforced by SnO_2 filler loading (3wt%)

4.3.3 Scanning Electron Microscope analysis

The DPNR and SnO2 filler loading morphology is analyzed using SEM at a magnification of 200 X. Among other particle morphologies of DPNR, it can emerge during doping. Figures 4.3 and 4.4 show that the DPNR can take on a globular or agglomerated appearance. The particle shape of DPNR was significantly changed during the manufacture of DPNR doped with SnO2. Figure 4.4 shows that compared to the composite with 1.0 wt.% of filler loading, the composite with 3.0 wt.% of filler loading exhibited a higher level of voids and particle agglomeration. Certain voids, shown by the red arrows in the images, were observed on the fracture surfaces of the DPNR/SnO2 test specimens as a result of the reinforcements pushing away. Increases in SnO2 content, namely above 3.0 wt.%, appear to be responsible for the brittle behaviour. Recent research suggests that plastic void generation and nanoparticle debonding are key mechanisms for toughening nanoparticle/DPNR composites. Figures 4.3 and 4.4 show that the rubber particles were not located in the voids because they had debonded from the matrix during plastic deformation and fallen out of the spaces. The inability of the DPNR matrix and SnO2 nanofiller to interact and foster excellent crosslinking is the root reason of the nanocomposite's failure. Thus, the existence of voids and agglomeration brought about by the non-homogenous dispersion of SnO2 filler loading in the DPNR matrix has a detrimental influence on the material's mechanical and physical properties.

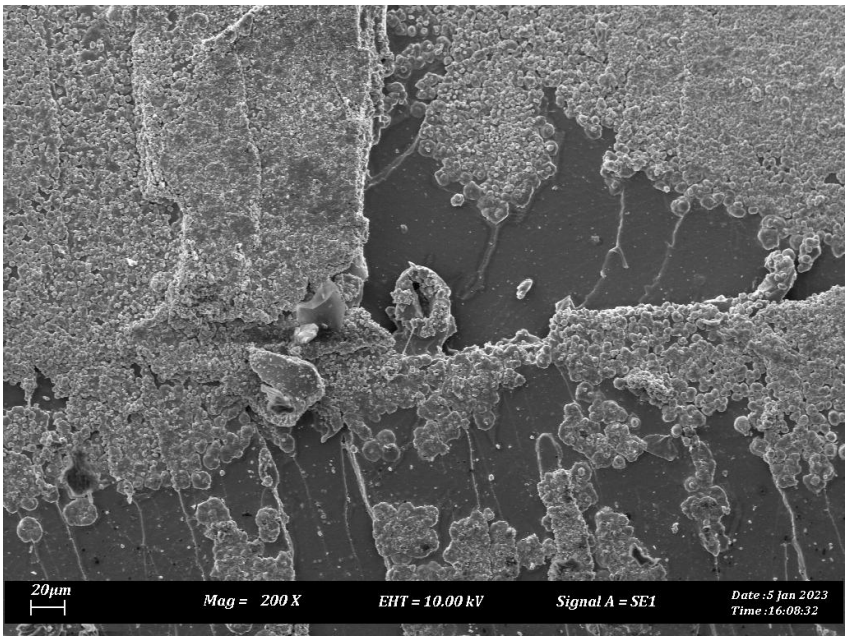


Figure 4.4 : SEM images of Calcined SnO2 filled with DPNR with 1wt% of filler with 200x magnification

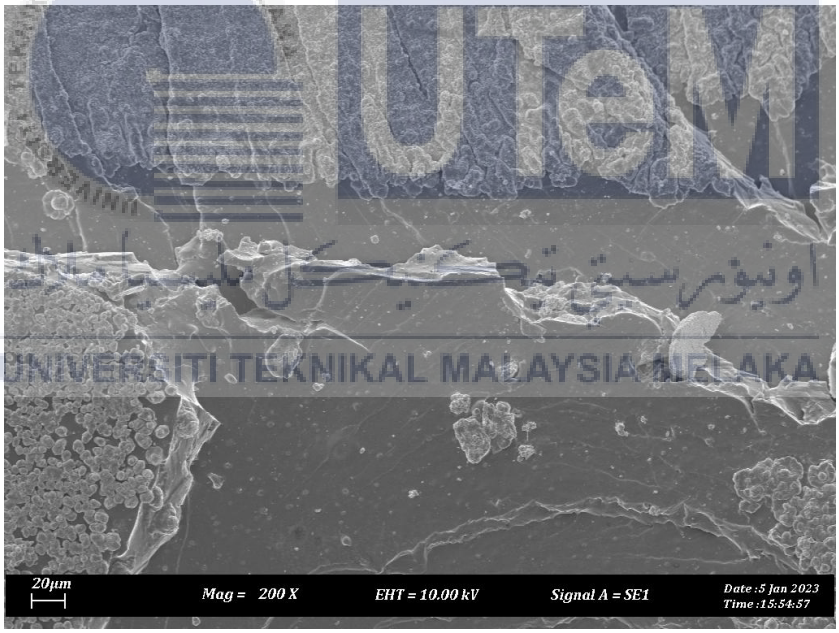


Figure 4.5 : SEM images of Calcined SnO2 filled with DPNR with 3wt% of filler with 200x magnification

4.4 Summary

On the basis of all the experimental data, it can be concluded that the properties of the DPNR composites tend to be enhanced by the addition of SnO2 nanofiller. SnO2 dispersion was discovered to be a crucial component that needed to be controlled. Effective SnO2 nanofiller dispersion produced the highest interfacial bonding between the matrix and filler, improving the rubber nanocomposite's properties. This is a result of the filler and matrix loadings having good interaction and crosslinking. However, SnO2 agglomeration caused by the nanofiller's higher surface energy tends to degrade the nanocomposite's properties.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

In conclusion, the fundamental purpose of this study is to explore the impact of varying volumes of calcined tin dioxide nanoparticles (SnO2) in the formulation of deproteinized natural rubber (DPNR) with regard to the passage of time, the temperature, and the torque. The melt compounding process with a two roll mill was utilized in the production of these SnO2 nanofiller composites with DPNR. There are five samples total, each of which was created using a unique combination of loadings for the SnO2 nanofiller. These loading correspond to about 0 weight percent, 0.50 weight percent, 1.0 weight percent, 3.0 weight percent, and 7.0 weight percent of SnO2 additions, respectively. The two roll mill mixer was used to successfully combine those samples, and the curing procedure was carried out utilizing a hot press. These samples were likewise made with the cutting procedure utilizing a laser cutting machine effectively.

In the discussion section, the physical and mechanical parameters were investigated and evaluated. These included the curing characteristics of the composites, density analysis, Fourier Transform Infrared Spectroscopy (FTIR), and Scanning Electron Microscope (SEM) test. It is essential to investigate all of those tests in order to improve the qualities of Calcined Tin Oxide Nanoparticles (SnO2) with Deproteinized Natural Rubber (DPNR) in terms of physical and mechanical parameters (DPNR).

5.2 Recommendations

In order to advance the quality of this research in the future, there are a few recommendations that should be made. The following are some recommendations:

i. In the process of mix blending, the DPNR control sample ought to be run first to prevent any hooked SnO2 powders from affecting the results of the other tests.

ii. It is necessary to do additional research in order to get the optimum amount of time for compression moulding in order to guarantee that the SnO2 filler loading will be distributed uniformly throughout the DPNR matrix throughout the vulcanization process.

iii. It is best to do the tests a second time under the identical circumstances in order to obtain a valid density trend (good atmosphere,room temperature and a consistent-sample sized). Therefore, a more precise analysis of the density is possible.

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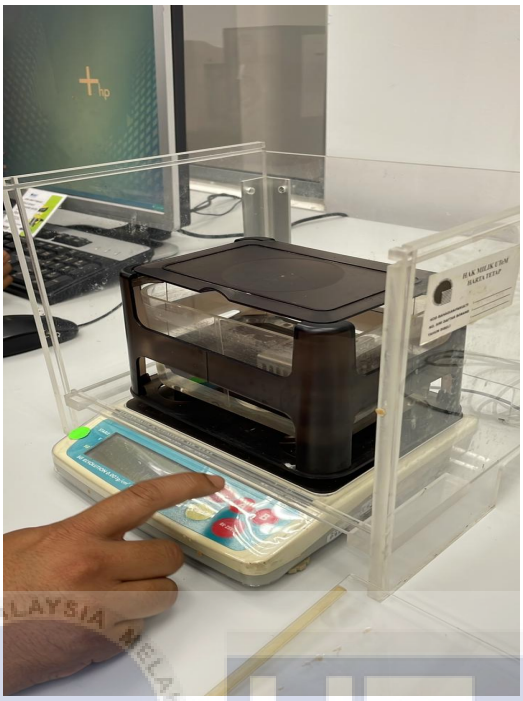
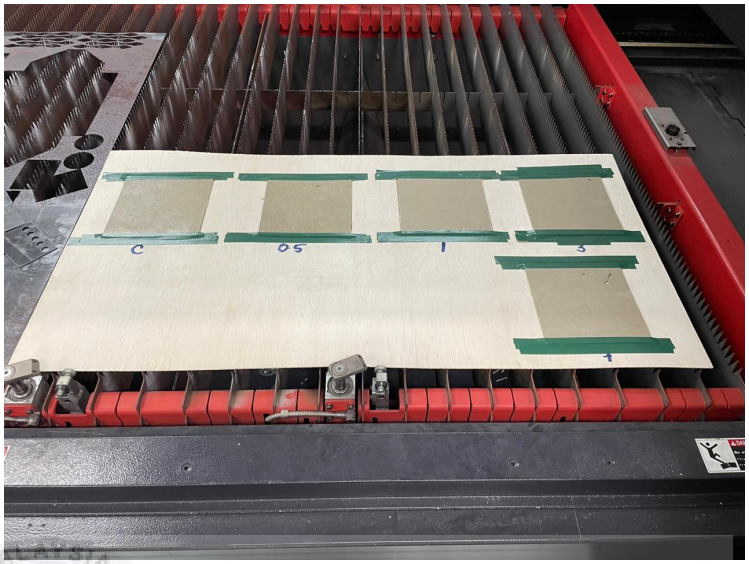
Appendix B **Specimen been tested for density value**



Appendix C **Specimen been shredded into small pieces before undergo hot press compression process**

Appendix D **Specimen before undergo cutting process by using Laser Cutting Process**



Appendix E **Specimen undergo FTIR analysis**