



**THE EFFECT OF FILLER LOADINGS TO THE
MECHANICAL AND PHYSICAL PROPERTIES OF TIN OXIDE
REINFORCED NATURAL RUBBER NANOCOMPOSITES**

This report is submitted in accordance with requirement of the University Teknikal Malaysia Melaka (UTeM) for Bachelor Degree of Manufacturing Engineering (Hons.)



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Tajuk: **THE EFFECT OF FILLER LOADINGS TO THE MECHANICAL AND PHYSICAL PROPERTIES OF TIN OXIDE REINFORCED NATURAL RUBBER NANOCOMPOSITES**

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APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Bachelor Degree of Manufacturing Engineering (Hons). The member of the supervisory committee is as follow:

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ABSTRAK

Getah asli mempunyai sifat penebat yang tinggi. Bahan penguat boleh ditambah untuk menguatkan lagi komposit getah asli. Penebat komposit getah asli yang baik adalah dengan adanya kehadiran bahan penguat yang sesuai. Tambahan pula, terdapat banyak kajian mengenai nano komposit dan komposit berasaskan getah asli, tetapi terdapat sedikit kajian yang menggabungkan getah asli dan nanopartikel dalam fabrikasi komposit. Oleh itu, penyelidikan ini dijalankan untuk membangunkan nanokomposit DPNR/SnO₂ yang berasaskan DPNR. Tujuan kajian ini adalah untuk membangunkan sistem nanokomposit dengan menggabungkan SnO₂ sebagai penguat bahan asas dan DPNR sebagai resin matriksnya. Nanokomposit dihasilkan dengan menggunakan gabungan teknik campuran leburan dan kemudian diikuti dengan proses mampatan panas untuk meningkatkan sifat komposit yang dihasilkan. Pemilihan komposisi optimum resin matriks DPNR adalah berdasarkan pemuatan SnO₂. Oleh itu, dalam kajian ini, pembebanan berbeza SnO₂ (0 %, 1.0 %, 3.0 %, 5.0 %, 7.0 %) digunakan untuk menyiasat sifat mekanikal, sifat terma, sifat dielektrik dan sifat fizikal. Kemudian, morfologi permukaan sampel yang dipilih dianalisis dengan mengimbas pemerhatian mikroskop elektron (SEM). Pada akhir kajian ini, sifat mekanikal dan dielektrik yang baik telah dibangunkan dan dicadangkan untuk aplikasi pecahan penebat. Di samping itu, analisis mengatakan bahawa nanokomposit DPNR/SnO₂ pada 3.0 wt.% beban pengisi mempunyai sifat penebat yang sangat baik.

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ABSTRACT

Natural rubber has higher insulation properties. Filler can be added to reinforce the natural rubber. Good insulator composites of natural rubber are better with suitable reinforcement filler. Furthermore, there are many researches about nanocomposite and natural rubber based composite, but there is little research that combine the natural rubber and nanoparticles in fabricating the composites. Thus, this research was carried out to develop a DPNR reinforced SnO₂ nanocomposite. The purpose of this study was to develop a nanocomposite composite system by incorporating SnO₂ as the reinforcement nanofiller and DPNR as their matrix resin. The nanocomposite were produced by using a combination of melt-blending technique and then followed by the hot compression process, to enhance the properties of produced composites. The selection of optimum composition of DPNR matrix resin are based on the SnO₂ loading. Therefore, in this study, different loading of SnO₂ (0 %, 0.5%, 1.0 %, 3.0 %, 7.0 %) were used to investigate the mechanical properties, physical properties and dielectric properties. Later, the surface morphology of the selected samples were analysed by scanning electron microscopy (SEM) observation. At the end of this study, good mechanical and dielectric properties were developed and proposed for insulation breakdown application. In addition, it was analysed that DPNR/SnO₂ nanocomposite at 3.0 wt.% of filler loading has better insulation breakdown properties.

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DEDICATION

Only

My beloved father, Mohamad bin Ibrahim

My appreciated mother, Zaimah binti Omar

My adored sister and brother, Azira and Akma

Honourable lecturers and loyal friends

for giving me moral support, money, cooperation, encouragement and also understandings

Thank You So Much & Love You All Forever



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LIST OF ABBREVIATIONS

| | | |
|------------------|---|--|
| ASTM | - | American society for testing and materials |
| EV | - | Vulcanisation |
| DPNR | - | Deproteinized Natural Rubber |
| DS | - | Dielectric Spectroscopy |
| SEM | - | Scanning electron microscope |
| SnO ₂ | - | Tin Oxide |
| SOP | - | Standard operation procedure |
| TGA | - | Thermogravimetric analysis |
| UTM | - | Universal Tensile Testing |
| Min | - | Minute |
| wt | - | weightage |



LIST OF SYMBOLS

| | | |
|--------------------|---|---------------------------|
| cm | - | Centimetre |
| m | - | Metre |
| % | - | Percent |
| g/cm ³ | - | Grams per centimetre cube |
| wt. % | - | Weight percent |
| mm | - | Millimetre |
| MPa | - | Mega Pascal |
| GPa | - | Giga Pascal |
| °C | - | Degree Celsius |
| W/mK | - | Watt per metre per Kelvin |
| K | - | Kelvin |
| nm | - | Nanometre |
| kg.cm ³ | - | Kilogram centimetre cube |
| kg | - | Kilograms |
| mm/min. | - | Millimetre per minute |
| rpm | - | Revolution per minute |
| kN | - | Kilo newton |
| W | - | Sample width |
| °C/min | - | Degree celcius per minute |

CHAPTER 1

INTRODUCTION

1.1 Background

Industries nowadays are very emphasized to apply the natural and mineral resources in production to ensure the materials used is sustainable. This material is able to use for various potential applications. However, the level of dispersion and distribution of fillers in matrices for a polymer composite is important to study because it will affect the resulted structural characteristics, mechanical properties and etc. In this study, the tin oxide will be utilized as filler to reinforce the natural rubber composites. This fabrication will be performed by melt compounding and hot pressing method onto tin oxide producing new material gen for electrical packaging. Therefore, the aim of this study is to investigate the effect of filler loadings on the mechanical and dielectric properties of tin oxide reinforced natural rubber nanocomposites.

Composite is a nature existing materials that combined together by two or more materials to enhance their properties. They have very distinctive properties and the combination of materials never dissolve or mix into each other (Tri-Dung Ngo, 2020). Each material works together but holds its partitioned chemical, physical, and mechanical properties. Therefore, the two constituents are a reinforce and a framework that most preferences of composite materials which their stiffness and high strength, combined with low density, when compared with bulk materials, permitting for a weight decrease within the wrapped up standard (Suhair G.Hussein, 2019). F.C. Campbell (2010) stated that the lighter weight, flexibility to adjust the layup for maximum strength and stiffness, enhanced fatigue life, corrosion resistance, excellent design practice, lower assembly costs due to fewer detail components and fasteners are just a few of the benefits of composites. Thus, it is important to the most common infrastructure, construction and transportation among applications such aerospace, marine goods, sporting goods, and, more recently. Because of their distinct advantages over competing metallic, advanced composites are a diverse and growing industry.

Furthermore, the composites are widely used as a filler and matrix for reinforcement materials. The improvement in physical and structural properties of nanofiller based materials is nanostructures (Monika Supova, 2011). The, the composite materials are classified based on

their content, which includes the base material and the filler material. The two components of composites are a reinforcement of matrix and filler. F.C. Campbell (2010) stated in their previous researches that the strength and stiffness are provided by the reinforcing phase. Most of the time, the reinforcement is harder, stronger, and more rigid than the matrix. Typically, the reinforcement is in the form of a fiber or a particulate which the particulate composites come in a variety of sizes and it is also roughly equal. Thus, they could be spherical, platelets, or any other regular or irregular shape. Particulate composites have a tendency to be weaker and less stiff than continuous fiber composites, but they are typically much less expensive. In addition, particulate reinforced composites typically have less reinforcement up to 40 to 50 percent volume percentage as a result of processing difficulties (F.C. Campbell, 2010).

A matrix or binder is the continuous phase material which is the base material that binds or holds the filler material in structures, whereas filler material can take the form of sheets, fragments, particles, fiber, or other materials (S.K. Mazumdar, 2002). It can be either polymer, metal, or ceramic. Polymers have low stiffness and strength, metals have intermediate stiffness and strength but high ductility, and ceramics have high stiffness and strength but are brittle. The matrix transmits loads from the matrix to the fiber via shear loading at the interface in polymer and metal matrix composites with a strong bond between the fiber and the matrix. The figure 1.0 below shows the composite structure for three major categories (Dipen Kumar Rajak, 2019).

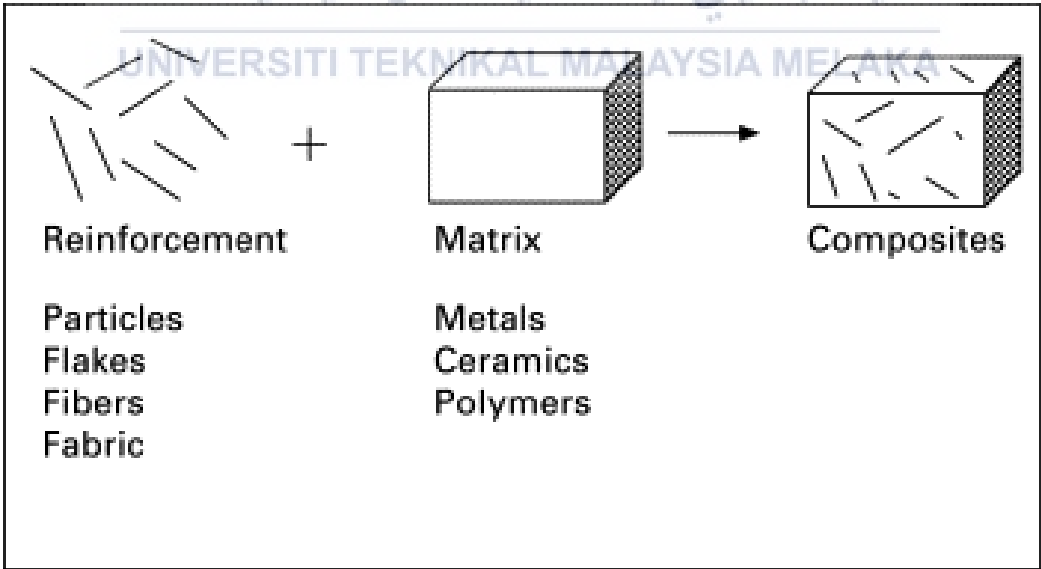


Figure 1.0: Structure of composite

Nanocomposite is a material formed by mixture of materials such as inorganic nanoclusters, fullerenes, clays, metals, organic polymers and also oxide with numerous organic polymers organic and etc. This solid combination of bulk matrix and nano-dimensional phases have different properties because they have different structure and properties. This structure build in a nanometre range 10-100 Nano-meters (nm) or structures composite in one phase having nanoscale morphology. It can be classified on the basis of their dispersed matrix and dispersed phase material (Mousumi Sen, 2019).

Moreover, the uses of nanoparticle-rich materials long predate the understanding of the physical, mechanical and dielectric properties including strength, limitation and thermal stability. In the other hands, the nanocomposite comes from nature such as oxide and differ from conventional composite material which is high surface to volume ratio if the reinforcing phase. The reinforcing material can make up the particles and the area of interface between matrix and reinforcement will be greater. So, the larger the reinforcement surface area, the smaller amount of nanoscale reinforcement. Nanoparticles are widely used for reinforcing rubbers. Based on this and related to this study, the effect on the properties of the nanocomposite can be define by adding nanoparticle such as by added Tin Oxide (SnO_2) as a filler material in order to utilized as functional fillers in natural rubber based nanocomposite to enhance their thermal and dielectric properties (Zhang et al, 2018).

Natural Rubber (NR) are commonly used widely in industrial such as in medical, automation, aviation and manufacturing industry. NR is a milky suspension which extracted from latex which their scientific name known as *Hevea Brasiliensis*. It can provide high specific properties because it is a natural polymer that composed of an association of poly (cis-1,4-isoprene), poly (2-methyl-1,3-butadiene) and biological elements (Laurent Vaysse, 2009). To reach strong properties, this rubber through the coagulation process by using the chemical substance such dilute acid. NR alone is not enough good to use in commercial applications because of properties of this rubber is low strength, low elasticity, soft, gummy and have low resistance to shape deformation. Besides, the crosslinking density between polymer chain of NR also low because of the dried process from the raw material. Therefore, this process and vulcanized decreasing the mechanical properties with a high sensitivity to thermal (Liliane Bokobza, 2018). However, the mechanical properties and structure of natural rubber can be improved by adding the fillers material in order to reinforce and improve the polymer chain,

strength, thermal and dielectric properties. Properties of materials are very important to have a sustainable materials of NR nanocomposite and can be used widely in industries.

In addition, based on the statistical reported Malaysia holds ranks of third largest NR production in the world (H.Y. Umar, 2011). Therefore, the quality and performance of deproteinized natural rubber (DPNR) is better than NR. DPNR is one type of natural rubber product that is required for special purposes which is made of latex through a purification process to remove components other than the rubber particles. The DPNR also known as solid rubber that has been reduced to its protein level. Based on previous study, there are many experiments have been carried out in order to produce natural rubber with a low protein content either as solid rubber or as latex (Ade Sholeh Hidayat, 2014). DPNR also conducted to prepare a non-allergy vulcanized rapidly and efficiently. Thus, DPNR act to improve as a rubber product which is necessary to investigate the properties of vulcanized latex and the effect of filler loadings (Warunee Ariyawiriyanan, 2013). This type of rubber also categorized as NR that has the excellent properties and had established as an important raw material allowed to apply in wide industrial applications (Jitladda Tangpakdee Sakdapipanich, 2013).

Tin Oxide (SnO_2) is an inorganic nanocomposite are one of the nanostructured materials semiconducting oxide materials are that attracting a lot of attention due to its particular electrical, physical, chemical, structural, and optical properties. This material had classified as transparent conductive oxides and their properties are highly dependent on stoichiometry, doping and the preparation method. SnO_2 is a low cost material but it has suitable physicochemical properties and have a good reaction to various gases and vapors such as oxygen, hydrogen, carbon monoxide, ethanol, NO_2 , and ammonia at ambient temperature. Generally, a possible working mechanism of tin oxide depend on the resistance changes when it exposed to the molecules of gas. Then, the conductivity of the material will increase when the surface electrons and the oxygen molecules in the air are no longer together when reducing gas contaminates the air. Besides, SnO_2 also known as a non-stoichiometric crystal structure which act to adsorption of oxygen quickly and react rapidly to reduced gases (A.Shavorsky, 2019). However, the SnO_2 commonly faced a few limitations such as sensors poor stability and poor selectivity (D.Dastan, 2017). But, this limitation can be improved by doping or implementing nanostructures. Otherwise, the performance SnO_2 also can be determined by parameters such as sensitivity, selectivity, operating temperatures, and response and recovery

time. While, SnO₂ is a good insulator in its stoichiometric form. However, their properties are highly dependent on stoichiometry, doping and the preparation method.

Dielectric properties known as an important role in electronic packaging applications. This is because of ability to provide an excellent material option particularly due to their dielectric properties when agglomeration occurred on nanocomposite low filler loading (Santanu Singha, 2010). According to the advantages of processing, excellent insulation, lightweight, and good mechanical properties, polymers are widely used in electronic packaging. Hence, polymers got a big demand on electronic devices which provide low dielectric properties, high energy density and have shorter signal delays. However, dielectric properties always have changes in polymer composites (R, Li, 2021). That is why the filler loading added in polymer material in order to enhance the dielectric properties. When compared to unfilled matrix, nanocomposites have lower permittivity (ϵ) or tan delta (δ) values over a wide frequency range. This unexpected finding has been attributed to the interaction of the matrix chains and the nanoparticles loading (Santanu Singha, 2010). So, when the filler added to eliminate the electrostatic motion and not allow the current to flow.

Thus far, there are many research mostly studied about Natural Rubber Nanocomposite, Tin Oxide and effect of filler loading. But, this these researches separated into other cases. However, there are still no similar study or research about the effect of SnO₂ filler loadings on the mechanical and dielectric properties of SnO₂ reinforced DPNR. Therefore, there are a new journey to do a research about fabrication of SnO₂ reinforced DPNR nanocomposites. Two major process was conducted to fabricate this material which is by melt compounding and hot pressing for electrical packaging material. Besides, the testing and observation also conduct to support this research by using SEM, UTM, and VNA analyses in order to obtain the experimental findings. Then, the effect of tin fillers loading on the mechanical and dielectric properties of tin-reinforced natural rubber composites were investigated further.

1.2 Problem Statement

Reinforcement of materials is important in helping them to strengthen and able to be used for higher potential variety applications in any industry. In general, the reinforcement can be done by using mineral based or nature based materials which occur single materials in widespread industrial application such as automotive, aerospace and medical industries. The research about reinforcement of DPNR nanocomposite that is actively used in worldwide researches because they possessed many benefits in reinforcement the DPNR with different type of fillers for industrial application, which it can improve their own mechanical properties and dielectric properties including the electrical and thermal conductivities and the barrier properties. Moreover, DPNR nanocomposite also has higher of demands in the industrial applications.

In general, there are many researches about deproteinized natural rubber, nanocomposite and also tin oxide. But this researches comes separately and do not specific focus on the title of SnO₂ reinforce DPNR Nanocomposite as well. Otherwise, there is no previous study about the effect dielectric properties of SnO₂ DPNR composite. Therefore, this study has no similarities research with the other previous researches in order in their filler materials and also effect loading happened. Thus, this research provides a fundamental kick start to explore and understand the properties of SnO₂ and how it works when it filled into DPNR nanocomposite. This research also to study what is the effect of dielectric properties when the filler is loading.

In addition, according to Liliane Bocobza, (2019) there are some challenge in developing this study of DPNR/SnO₂ composite which required strength and good electrical insulator properties for Insulation Breakdown properties. The mechanical response of DPNR depends on several parameters including the processing conditions, the state of filler dispersion, the polymer-filler interactions and the filler morphological aspects. All of these parameters are very important in order to get the advance mechanical properties and dielectric properties. Besides, the other challenge in developing this study is about the properties. Due to the properties of resulted composite may be affected significant factors for filler reinforcing ability are directed by its specific properties. These properties are including the particle shape, size and, surface properties, agglomeration and also degree of interaction between filler and matrix.

Thus, the other challenge to face is about DPNR which is the agglomeration of SnO₂ in DPNR based composite. The same filler can improve the filler in certain properties but it will

deteriorate others. This is because if the contact area between the matrix and filler particles increase, the property will achieve. Therefore, the bonding of matrix and the filler are important because it will represent the reinforce quality. Therefore, the problem is to explore how the interaction between DPNR matrix and SnO₂ filler particles. Compatibility of SnO₂ nanofiller within the DPNR matrix and its processability need to be understood as nanofiller added prone to agglomerate and reduce the performance of nanocomposite. So, the potential of SnO₂ nanofiller to reinforce DPNR rubber for insulation breakdown is something new and further investigation need to be conduct to understand the fundamental behavior of it.

1.3 Objectives

The objectives of the research are as follows:

- (a) To evaluate the cure characteristics of DPNR/SnO₂ nanocomposite at various loading using the cure rheometer.
- (b) To evaluate the mechanical and physical characteristics of DPNR/SnO₂ nanocomposites at various loading using standard mechanical and physical testing.
- (c) To correlate the dielectric and mechanical properties performance with the morphological behaviour of DPNR/SnO₂ nanocomposites by using Scanning Electron Microscopy (SEM).