CHARACTERIZATION OF NATURAL RESIN AND SYNTHETIC RESIN IN FIBER COMPOSITE LAMINATE

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DECLARATION

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LIST OF ABBREVIATIONS

TGA	Thermogravimetric Analysis
SEM	Scanning Election Microscopy
EDX	Energy Dispersive X-Ray
ISO	International Organization for Standardization
MEKP	Methyl Ethyl Ketone Peroxide
IC-TAC	International Confederation for Thermal Analysis and Calorimetry

CIRI-CIRI RESIN SEMULAJADI DAN RESIN SINTETIK DALAM LAMINAT KOMPOSIT FIBER

ABSTRAK

Isu alam sekitar dan kemampanan ditambah dengan peningkatan kesedaran telah menggesa para penyelidik untuk memberi tumpuan kepada sumber semula jadi yang boleh menggantikan bahan sintetik. Matlamat kajian ini adalah untuk mencirikan ciri fizikal dan mekanikal bagi tiga bahan berbeza iaitu resin asli dan resin sintetik. Ini akan membentuk rangka kerja untuk aplikasi masa depan untuk membangunkan bahan semula jadi dalam industri pembuatan. Resin semula jadi gajus dan neem, dan resin sintetik poliester dicirikan dengan menjalankan empat ujian berbeza. Analisis Termogravimetrik (TGA), Scanning Electron Microscopy (SEM), Analisis X-Ray Penyebaran Tenaga (EDX) dan teknik nanoindentation digunakan sebagai ujian pencirian fizikal dan mekanikal untuk spesimen. Keputusan menunjukkan bahawa komposit poliester mempunyai kestabilan haba yang paling tinggi kerana ia mempunyai kumpulan fungsian dan elemen struktur yang lebih kuat manakala gajus dan neem mempunyai kematangan haba yang rendah. Struktur molekul komposit poliester adalah kecil dan padat dengan keliangan yang rendah, ini menghasilkan kekerasan yang lebih tinggi antara spesimen. Gajus menunjukkan kekuatan hasil dan kekuatan mampatan yang lebih tinggi kerana keliangan yang lebih rendah daripada resin neem. Komposisi unsur spesimen adalah serupa, ini bermakna resin sintetik dan semula jadi mempamerkan ciri kimia yang sama tetapi kekotoran dalam resin boleh mengubah tindak balas. Keputusan daripada nanoindentation menunjukkan modulus elastik tertinggi dalam resin gajus. Penembusan lapisan komposit poliester membawa kepada kekerasan yang lebih rendah dalam ujian mekanikal.

CHARACTERIZATION OF NATURAL RESIN AND SYNTHETIC RESIN IN FIBER COMPOSITE LAMINATE

ABSTRACT

Environment and sustainability issues coupled with increased awareness have urged researchers to focus on natural sources that can substitute synthetic materials. The goal of this study is to characterize the physical and mechanical characteristics of three different materials of natural resin and synthetic resin. This will form the framework for future application for developing natural materials in the manufacturing industry. Natural resin of cashew and neem, and synthetic resin of polyester is characterized by conducting four different tests. Thermogravimetric analysis (TGA), Scanning Electron Microscopy (SEM), Energy Dispersive X-Ray (EDX) analysis and nanoindentation technique are used as physical and mechanical characterization test for the specimens. The results demonstrated that polyester composite has the highest thermal stability as it has stronger functional groups and structural elements whereas cashew and neem have low thermal maturity. The molecular structure of polyester composite is small and compact with low porosity, this resulted in higher hardness between the specimens. Cashew showed higher yield strength and compressive strength due to lower porosity than neem resin. The element compositions of the specimens are similar, this would mean synthetic and natural resin exhibit the same chemical characteristics but impurity in the resins may alter the reactions. The results from nanoindentation showed highest elastic modulus in cashew resin. The delamination of polyester composite layers lead to lower hardness in the mechanical test.

CHAPTER 1

INTRODUCTION

1.1 Overview of natural resin-natural fiber composite

In current age of industrial revolution, sustainable manufacturing has been an important aspect that is taken into considerations for manufacturing processes. Many large manufacturers are realizing the importance of sustainable manufacturing as the numbers of environmental and sustainability issues are emerging. The goal of researching in sustainable methods in manufacturing process is to minuses wastes and reduces environmental impact for a sustainable future. The environmental issues coupled with increased awareness have urged researchers to focus on natural sources that can substitute several forbidden materials. For example, natural fibre-natural resins have succeeded in attracting global attention toward its suitability to replace syntactic materials (Chandgude & Salunkhe, 2021).

The fiber polymer composites are the trend in the automotive industry as their composition and strength also eclipses their environmental-friendly advantage. The fiber polymer composites are further categorized into two groups that are biodegradable and non/partially biodegradable composites. The biodegradable composites consist of both natural resin such as neem, cashew, amber and natural fibers such as jute, flax, cotton etc(Yan et al., 2014). The automotive industry currently uses partially biodegradable composites. These are usually natural fiber reinforced with synthetic resins which require manufacturing process at higher degrees. In the industry, the composites are formed at very high temperature to weaken the adhesion between the fiber-matrix (Chandgude & Salunkhe, 2021).

Natural fibre-natural resin are replacements for synthetic materials such as fiberglass as they are toxic, require chemical treatment, and requires high quantity of energy of energy for production process. Traditional manufacturing process of synthetic fibre composites represents a health risk for workers when precautions are not followed. Natural fibre-natural resin composites are one of the solutions for the disadvantages posed from producing synthetic materials as they are ecological composite materials that consists of natural fibres and epoxidized vegetable oil resins (Torres-Arellano et al., 2020a).

1.2 Natural Resin for Fiber-Reinforced Composites

Natural resin in the fiber-reinforced composites have long chain of repeating units. These resins are classified into 2 groups depending on the structure of molecule of resins. The 2 groups are thermoplastic and thermosetting characteristics according to the effect of heat on their properties. Thermoplastics are materials that have elastic properties when heated. They are soft under heating and harden again after cooled. The most common thermoplastics are polypropylene, glass fibres, nylon, etc. Thermosetting materials are formed after undergoing a chemical reaction with hardeners. They are called thermosets where the resin and hardener are mixed to form a strong and hard material.

Generally, thermosets are resins and hardeners mixed to undergo an irreversible process that cannot change back into liquid form. Even after heat treatment and curing process, thermosets do not change in physical characteristics although mechanical properties are affected above the glass transition temperature (Tg) (Croccolo et al., 2015). The resins chosen for this study as the synthetic resins is polyester. Epoxy is also used as a strong material for experimental purposes.

Natural resin used in this study are extracted from the locals in Sri Lanka. Therefore, they are acquired organically and have no uniform shapes and sizes. The climate and environment affect the overall quality and structure of the natural resin. Resin acquired from different plants and plantation may also vary. The difference in structure and quality makes it not suitable for industrial use without pre-processing to obtain a purer raw material. It is necessary to study and test the sample to understand its characteristics for grading its quality under industrial use. As the increasing of emphasis on sustainability manufacturing and biosafety of manufacturing industry, quality management of materials is important for applications for industrial use before human contact and consumption (Srivastava et al., 2016).

As fiber-reinforced composites are increasingly popular in the automotive industry, a uniform quality is required to be supplied to sustain trade, it is necessary to study on the characteristics of variety of natural resins to test the sample according to international standards such as International Organization for Standardization (ISO) specifications (Srivastava et al., 2016).

1.3 Problem Statement

Fiber-reinforced composites are used intensively in automotive industry. Synthetic materials such as fiberglass are toxic, require chemical treatment and high energy for production. Traditional manufacturing of synthetic material composites represents health risks for workers when precautions are not followed. In addition, characterization of natural resins is not standardized to be effectively used in the industry. Extensive tests are needed to study the characterization of variety of natural resins to be a solution and replacement for synthetic materials . The properties of natural resins and synthetic resins must be compared.

1.4 Objectives

There are two main objectives for this study:

i. To characterize the physical properties of natural resin to align with the application of automotive component.

ii. To distinguish the performance of natural resins to the synthetic resin in terms of physical properties.

1.5 Scope of Project

The materials are prepared in advance from supervisor and technicians from the composite lab of School of Aerospace. Fabrication process of natural fiber-synthetic resin composite is conducted in the composite lab for the equipment and apparatus needed for the process. The material used as synthetic resins is polyester, hardened with Methyl Ethyl Ketone Proxide (MEKP). As for the natural resins, neem and cashew as provided to conduct the tests on. The hot and cold mounting for natural resins is conducted as samples preparation for the proceeding tests. The hot mounting process is conducted for synthetic resin in the material lab in School of Mechanical, whereas cold mounting process is conducted for natural resins as the temperature in hot mounting affects the physical characteristics. The physical and mechanical properties determine the compatibility of the materials to be utilized in automotive industry. To characterize the resins, thermogravimetric analysis (TGA), scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and nanoindentation tests are conducted in the labs of School of Mechanical Engineering. By comparing the results from the tests and analysis, the comparison and characterization of synthetic and natural resins can be studied.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction on natural fiber

To work towards sustainable manufacturing, natural fiber reinforced polymer has significant potential towards the automotive industry. Natural fibers provide new energy in the composite matrix to overcome the shortcomings such as high brittleness and high curing temperatures of the composites (Ramdani et al., 2022). Natural fibers are essential in replacing synthetic materials due to their environmental suitability, ease of processing, and low cost. There are several ways of improving the performance of natural fiber in the composite such as applying surface treatment and coupling agents. Alkaline and silicane treatment treatments are the most effective methods on improving performance of natural fiber in its mechanical properties, thermal and water absorption properties through the increasing the fiber interfacial adhesion (Ramdani et al., 2022). The main compositions of natural plant fibers are cellulose, hemicellulose, and lignin. Natural fibers as natural raw materials for insulations are emphasized due to the benefits of sustainability and environmental friendliness. Many natural fibers are renewable resource, and do not lead to depletion of natural resources. Plant cultivation consumes less energy than the production of synthetic materials. The merits of natural fibers are positively in replacing the synthetic materials to overcome the issues where synthetic plastic insulations are encountering (Zhao et al., 2022).

To characterize the physical properties of different types of natural fiber in composite matrix, dynamic mechanical analysis is essential. DMA method is used due to the high accuracy and sensitive detection of storage and loss moduli, damping factor and glass transition temperature of polymeric composites. Usually, the use of fiber in polymer matrix increases the performance of the material. However, hybridization of

reinforcing agents may not always positively influence the DMA properties of the composites. Hence, it is crucial to optimize the formulation of composite to acquire the desired DMA properties of the materials (Haris et al., 2022).

2.2 Natural fiber-natural resin composite

Natural fibre-natural resins attracted global attention to replace synthetic materials for sustainable manufacturing. The primary function of fibres is to carry loads and provide the composite with enough stiffness, this is especially important in the automobile industry where choice of material is vital to improve quality of parts. Synthetic fibres require chemical treatment and high level of energy for production while poses health risks for workers are they are neurotoxic (Kumar et al., 2021). Natural fibres are obtainable from animal, vegetable, or mineral source to convert into nonwoven fabrics and are classified according to their origin. In this research, the natural fibres used are vegetable or cellulose-base class such as cotton, flax and jute. Natural fibres such as jute have affinity for water that causes swelling of fibres, which facilitates dyeing in watery solutions. Unlike synthetic fibres, natural fibres are nom-thermoplastic that do not soften when heat is applied, shows low sensitivity to dry heat, and does not turn brittle at low temperature. They are typically used as reinforcement for thermosetting resins (Torres-Arellano et al., 2020b). The downside of natural fibres is that they are prone to microbial decomposition and decomposes at high humidity and temperature. This however can be treated with chemical modification of fibre substrate and modern developments allow treatment of natural fibres to make them immune to decomposition.

Natural resin is a resin product which normally comes from plant in comparison to synthetic resin. Natural resins are classified as spirit-soluble and oil-soluble, making

it more loses its volatile components by evaporation, leaving a soft residue at readily soluble but becoming insoluble as it ages. However, natural resins are getting replaced by synthetic resins which are thermoplastic resins and thermosetting resins in modern manufacturing industry. This is because synthetic fibers have higher elastic properties, absorbs less moisture, and has higher tolerance to heat, and light compared to natural fibers. The most typical synthetic resins used in the industry is epoxy. The epoxy resins exhibit excellent adhesive properties and high compatibility with carbon fiber but are expensive and toxic to the workers.

2.3 Review on Thermogravimetric Analysis

Thermal analysis (TA) has been studied as a quantitative technique that monitors the physical and chemical properties of a sample with time under different temperature. The International Confederation for Thermal Analysis and Calorimetry (IC-TAC) defined thermogravimetric analysis (TGA) that records the sample mass, time, and temperature (Saadatkhah et al., 2020). The temperature set for the samples included heating, cooling, and isothermal holds to see how the specimens interact under different temperature conditions.

For the results, a graph of sample mass change against temperature or time is plotted. The graph plotted is a TG thermal curve which indicates the graphical representation of the data. At different point of temperature, polymer generally shows sections of thermograms and different behaviour. In the graph, the first and second derivatives of the weight loss profile (DTG and DDTG) identify as inflection points and discriminate phenomena of multicomponent mixtures that react at overlapping temperatures (Saadatkhah et al., 2020). The temperature at which two extrapolated lines intersect is the onset temperature.

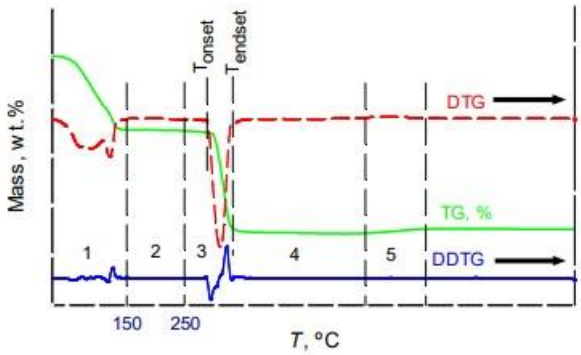


Figure 2.1: Different section of thermogram indicated with onset temperatures (Saadatkhah et al., 2020).

To conduct a TGA analysis, we need to set the temperature program, temperature ramp, hold-time, maximum temperature, and sample size based on the type of material used. ISO standards for polymers are used in the testing TG of natural resins. However, the main limitation of TGA analysis is the limit of sample size, heat, and mass transfer rate. In our sample size of 6-10mg, heating rates within the sample are not rapid enough to ensure isothermal conditions, and poor mass transfer creates radial and axial concentration gradients (Mansa & Zou, 2021). The limitations might cause some random error and uncertainty to the kinetic parameters derived from the analysis.

2.4 Review on Scanning Electron Microscopy

Scanning Electron Microscopy (SEM) is a tool that can be useful when light microscopy is inaccessible for more details and complexity. The analysis is done by applying stream of electrons which are emitted by a thermal source. The lenses used in the microscope compress the shot and direct the electrons on the specimen. The image is displayed on the viewing screen and the brightness and magnification of the image can be adjusted by the user. It is recommended magnification beyond 10,000 should be applied as the capability of SEM is much higher than a standard light microscope. The

details of surface information are all depends on the electron voltage emitted from the thermal source (Mohammed & Abdullah, n.d.). Low accelerating voltages (5kV) are generally used to inspect surface information whereas high accelerating voltage (15-30kV) are used to penetrate underneath the surface and inspect the details on the interior of the samples.

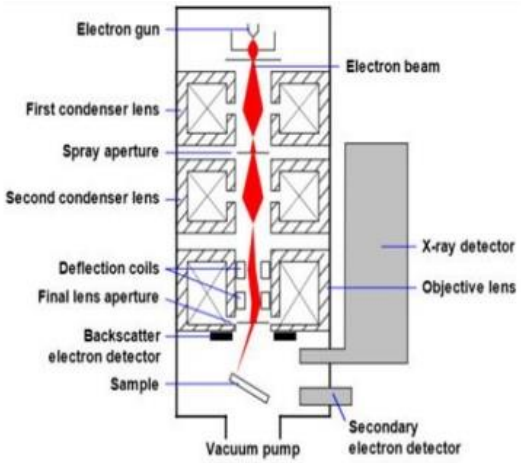


Figure 2.2: Schematic of SEM (Mohammed & Abdullah, n.d.).

While SEM is a great tool to inspect the surface of specimen in this study, the image processed by SEM might not have high accuracy due to the collisions by the scattering and defocussing of the electron beam, which causes error and uncertainty of the position of election beam on the specimen. However, SEM lessens dehydration on the surface of specimens due to the elevated gas pressure in the chamber. This also eliminate the needs of coating on the surface which may cause ionization of gas molecule (Joy & Joyt, 1996). This ensures concrete imaging to be acquired. The images from SEM instrument are essential for qualitative and quantitative analysis of the topography of the specimen.

2.5 Review on Energy Dispersive X-Ray Analysis

Energy Dispersive X-Ray (EDX) analysis is generally used to characterize the elemental composition of specimens and indicate its chemical analysis. EDX analysis and SEM usually run at the same time as each analysis requires shooting beams of high energy elections onto the surface of specimen (Markowicz, 2011). The process of shooting electron beams onto specimen is classified into two categories which are the Bremsstrahlung X-rays and characteristics X-ray (Thambiratnam et al., 2020). Bremsstrahlung X-rays are also known as continuous X-rays. The continuous ray is generated for the interaction between electron and nuclei of the specimen so that it manifests itself as the background spectrum which the characteristics X-rays spectrum is superimposed (Thambiratnam et al., 2020).

The energy between the lower energy state and higher energy state are generated from the characteristics X-rays. The differences between the energy state indicates the characteristics of the specimens. The characteristics X-ray has capital Roman letters (K, L, M) to identify the shell containing the inner vacancy in the X-ray spectrum and uses Greek letters (α, β) and numbers to indicate the elements in the specimens which lines in the X-ray spectrum. In short, EDX analysis presents both the Bremsstrahlung and characteristics X-rays as a graph of X-ray energy against the intensity (Thambiratnam et al., 2020). The characteristics X-rays represents the qualitative analysis and reveals the elements consists in the specimen whereas Bremsstrahlung X-rays reveals its elements' concentration in its specimen.

2.6 Review on Nanoindentation Technique

Indentation tests are common for testing mechanical properties of materials, and it is continuously used as quality control test in many industries. The advancement in

instrumentation has developed with high spatial resolution made it feasible to characterize the physical and chemical composition of specimens in nano scale. Nanoindentation is a technique that is effective in evaluating the physical and mechanical properties of material structure. The technique is suitable for testing the mechanical behaviour of specimen such as stiffness, strength, and toughness. However, this can be derived into more detailed quantitatively such as calculating the plastic deformation, yield strength, stress, strain, etc. Hardness obtained from the technique has been found to depend on strain rate as it is calculated from the graph of the loading curves (Gautham & Sasmal, 2019).

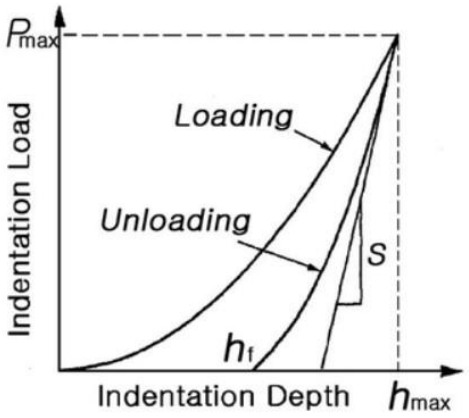


Figure 2.3: Typical example of load-displacement curve from nanoindentation (Ramamurty & Jang, 2014).

Due to the capability of testing small volume of specimen, nanoindentation technique is suitable on characterization of materials as limited specimens can be obtained. However, the testing parameters must also be chosen carefully to limit the uncertainty in the extracted data. Maximum load (P_{max}), maximum displacement (h_{max}) and feed rate are adjusted accordingly for the technique (Ramamurty & Jang, 2014).

CHAPTER 3

METHODOLOGY

3.1 Introduction

This chapter represents the overall methodology used in this study. For the first section of the project, the experimental plan for the initial study is set to fabricate a composite panel of natural fiber-natural resin as a reference of material for comparison with other materials. Next, the process of preparing of specimens to for testing purposes are conducted. These includes the finishing process, grinding and polishing of material, and mounting process for tests such as scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDX) and nanoindentation processes. For the thermogravimetric analysis testing (TGA), the samples are prepared using different method as the sample size for the test requires to be in smaller scale. The performance evaluation and comparison of each material are determined by characterization of physical and mechanical properties. For physical properties, the surface, molecular structure, and composition of elements of each material are compared. Whereas for the mechanical properties, the measurements that involved is the hardness and behaviour of heating under temperature. The flow chart in the figure illustrates the overall framework study that is involved in this project.

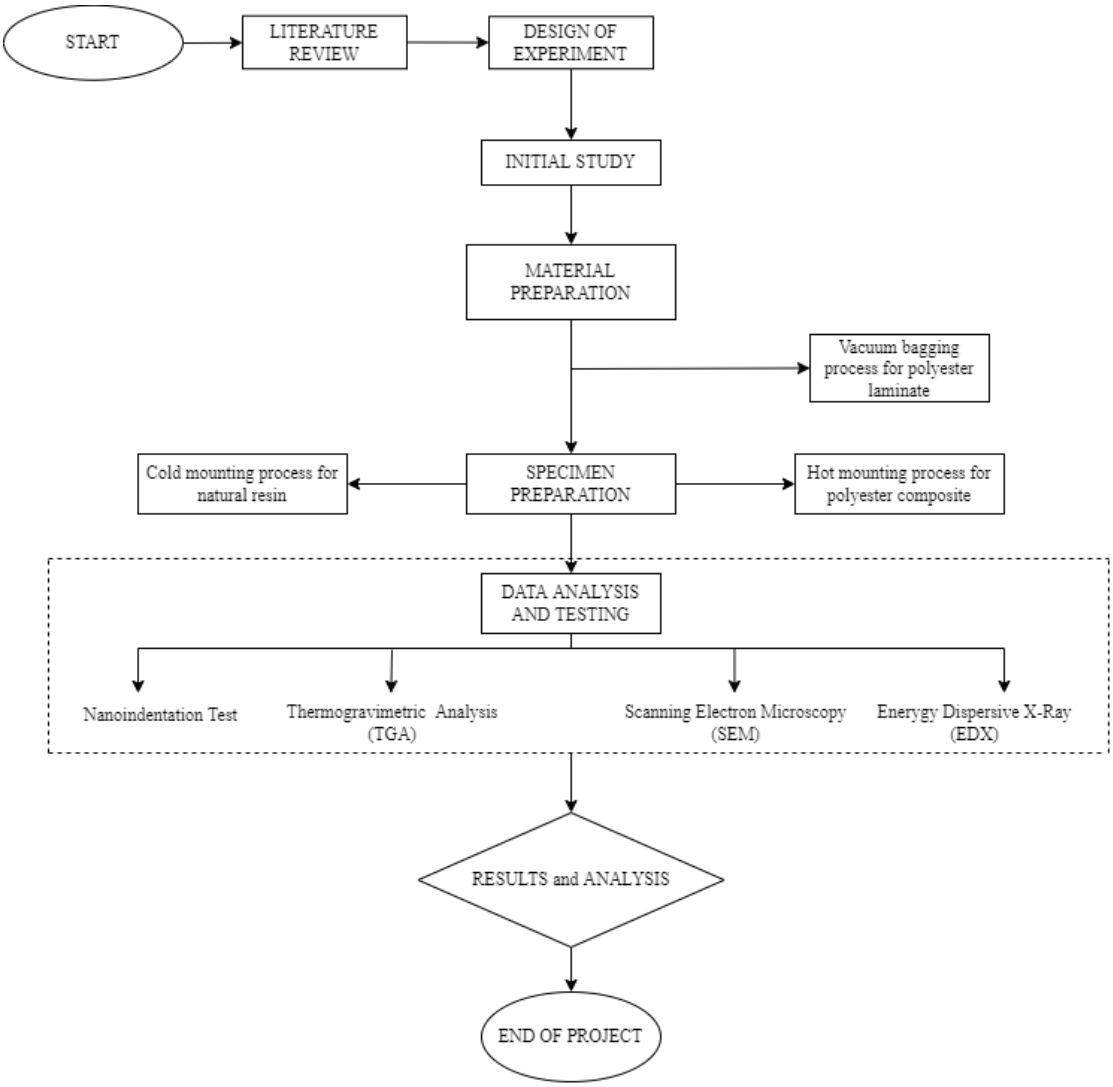
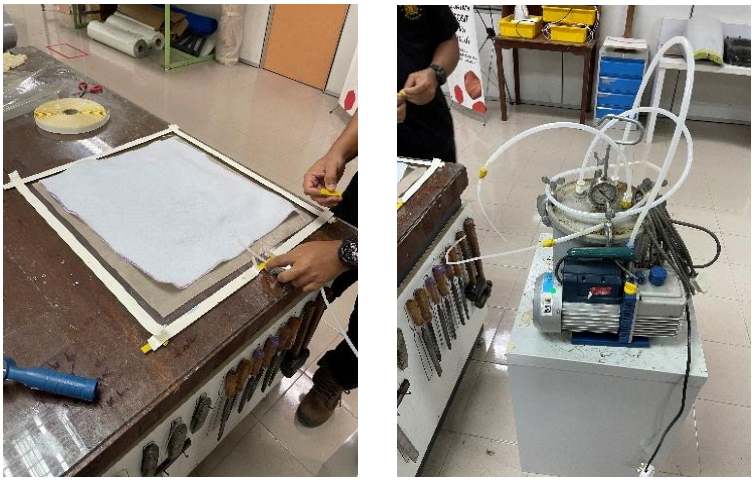


Figure 3.1: Methodology of research study.

3.2 Material Preparation

3.2.1 Material and equipment preparation for polyester laminate

To prepare for vacuum bagging process for polyester laminate, the resin is prepared beforehand and kept away in sealed container at room temperature. The baseline materials for the vacuum bagging process are flax fiber, polyester resin, methyl kethyl ketone peroxide (MEKP). Whereas the apparatus used are bagging films, sealant tape, suction connecting tube, and vacuum pump. The weighing scale is calibrated before each weighing process of materials to have an accurate reading. An oven is also used to reduce the moisture from the flax fiber used in the fabrication process. The oven is set at 160°C over 24 hours for drying.



(a) (b)

Figure 3.2: The yellow sealant tape, bagging films (a) and vacuum pump (b).

3.2.2 Material preparation for natural resins

The natural resins chosen for this study is neem and cashew resins. Neem as a resin is an exudate that are tapped from the trunk by wounding the bark of the tree. The high protein material is widely used in the South Asia as a “Neem glue”. The resin from this project is provided by supervisor which are imported directly from Sri Lanka. To prepare neem resin for various testing, the material is sealed in a bag until the time of

testing. This is to prevent contamination from the air that may distort the characteristics of the resin. It is the same case for cashew resin as it is also an imported material directly from Sri Lanka. Cashew resin is widely used in Asia as a coating for car brakes. The resin itself exhibit characteristics of moldability, adhesiveness, and non-flammability makes it a good replacement for synthetic resins.

Neem resins are crystallized structure and are acquired with small sized samples. This makes the preparation for it much simple than cashew resins. To prepare cashew thoroughly, the resins is left to dry in room temperature to remove excess moisture on the resins. Then, the surface of the cashew must be removed when preparing the specimens for testing either by sawing or grinding to remove the surface as it may be contaminated from the surroundings.

Figure 3.3: Material preparation for neem (left) and cashew (right) resins.

3.3 Vacuum Bagging Process for Polyester Laminate

The fabrication process of the laminate preparation consisted of bagging and curing process. The vacuum bagging process is conducted in the composite lab in School of Aerospace. The fiber is first prepared by cutting out the dimensions needed, which is 300mm x 300mm at 2mm thickness with 5 sheets of fibers. The fiber sheet is weighted at 148g. Before proceeding with the fabrication process, the fiber sheets are cured in an oven to remove moisture in the molecular structure.

To fabricate a composite panel, the fiber to resin ratio should be 1:2. Therefore, the polyester resin added to the composite is weighted at 296g to fit the ratio of bagging process. To harden the polyester resin to have necessary adhesive effect on the fiber, 6g of methyl ethyl ketone peroxide (MEKP) is mixed with the resin. The weight of hardener used is calculated to be 2% of the total weight of the composite.

After the vacuum bagging process, the composite is weighted at 296g after compression. A table is constructed to show the process flow of the fabrication process of polyester laminate.

Table 3.1: The fabrication process of polyester composite panel.

Procedure / Step	Image	Description
1		The fiber sheet is weighted then cured in an oven.
2		Polyester resin is then weighted at 296g before adding the hardener.
3		Vacuum pump set up before the fabrication process.
4		Yellow sealant tape, bagging films, and suction connecting tube are set up for bagging.
5		After the bagging process, the panel is weighted at 393.15g

3.4 Specimen preparation

To proceed the materials to various testing, the resins and composite laminate must be prepared thoroughly. The EDX, SEM and nanoindentation testing requires the specimen to have smooth and shiny surfaces that can be mounted properly on the machine. Therefore, after preparing the necessary samples, the natural resins and polyester laminate are separated into two types of mounting process. The polyester laminate is prepared using hot mounting process, whereas the neem and cashew resins are prepared through cold mounting process. This is because the natural resins cannot withstand high temperature of hot mounting process without changing its characteristics and molecular structure as they are considered thermoplastic resins. Whereas polyester is considered a thermoset resin and does not change its form when heated.

To prepare the hot mounting process of polyester laminate, the specimen is prepared in the material lab in School of Mechanical. The mounting process is done by using the specimen mounting press machine. The polyester laminate is placed in the cylinder together with bakelite. Temperature of 150°C and pressure of 20kN are set during the embedding of the specimen. The embedding process around 20 minutes with the inclusion of 14 minutes of heating and 6 minutes of cooling, before the specimen can be acquired.

Figure 3.4: The mounting press and prepared specimen.

For the cold mounting process of neem and cashew resins, the specimens are prepared in the composite lab in School of Aerospace. Due to the thermoplastic characteristics of natural resins, the same preparation process cannot be used for neem and cashew resins. In this process, suitable amount of epoxy and hardener is prepared for the polymerization process to form a block for the natural resins. PVC pipe is used to give shape to the mounting procedure, so all specimens have equal dimension. The neem and cashew are embedded in the epoxy respectively and left for 24 hours until the specimen is fully dried under room temperature.

Figure 3.5: Cold mounting for neem and cashew resins.

3.5 Evaluation of characteristics of specimens

In this research study, the physical properties of polyester laminate are characterized and compared with the natural resins. The details of physical properties and characteristics of each specimen are tested. The thermogravimetric analysis (TGA) is used to determine the thermal stability and the reactions at different temperatures, Energy Dispersive X-Ray (EDX) analysis is used to check the elements composition in each resin, Scanning Electron Microscopy (SEM) is used to compare the structure of surface of each specimen whereas nanoindentation test is used to calculate the hardness of the surface of specimens. Table 3.2 is constructed to show each testing, machine used and the parameter settings for the specimens.

Table 3.2: The physical and mechanical testing of specimens.

Physical/Mechanical Tests	Machines	Parameter Settings
Thermogravimetric Analysis (TGA)	 Vecstar Naber Therm NII F-24389	Sample weight: 5-6mg Initial temperature: 30°C Heating rate: 30°C - 850°C at 20°C/min

Scanning Electron Microscopy (SEM)	 SEM-Hitachi S-3400N/EDAX	Magnification: x50 – x600 depending on image.
Energy Dispersive X-Ray (EDX) analysis	 SEM-Hitachi S-3400N/EDAX	None
Nanoindentation test	 Nano Indentor – Micro Material Nano Test	Maximum load: 20mN Initial load: 0.01mN Loading rate: 1.0mN/s Unloading rate: 1.0mN/s Dwell time: 20s

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Introduction

This chapter summarizes the results of study for both the initial and extended analysis about the characteristics of synthetic resin and natural resins. The mechanical and physical properties of each material is compared between each other in four different tests. The results are classified into four categories as there are four different tests conducted. The physical characteristics of resins are analysed using thermogravimetric analysis to determine the weight and loss of weight under different heating temperatures. The structure of surface of the specimens are compared in SEM imaging. The surface structure is described and compared between each specimen. The element compositions of the resins are also broken down using EDX analysis to analyse the behaviour of each material. Lastly, the hardness of each material is analysed and discussed with the result of nanoindentation hardness test for the specimens.

4.2 Thermogravimetric Analysis (TGA)

The TGA results of the samples are illustrated in the figure. The solid lines indicate the weight loss of the sample. From the figure, the lines can be divided into several sections based on the temperature sequence. The dotted lines are the derivative weight loss (DTG) of the curve where different peaks can be observed. The shape of the curve can provide insights of the characteristics of the chemical structure of samples.

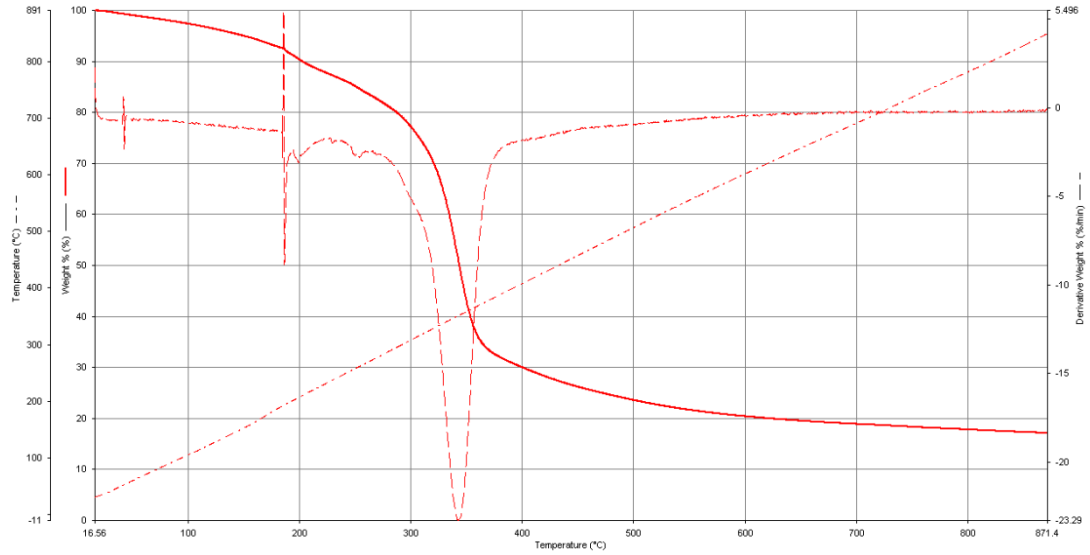


Figure 4.1: TGA graph of cashew resin.

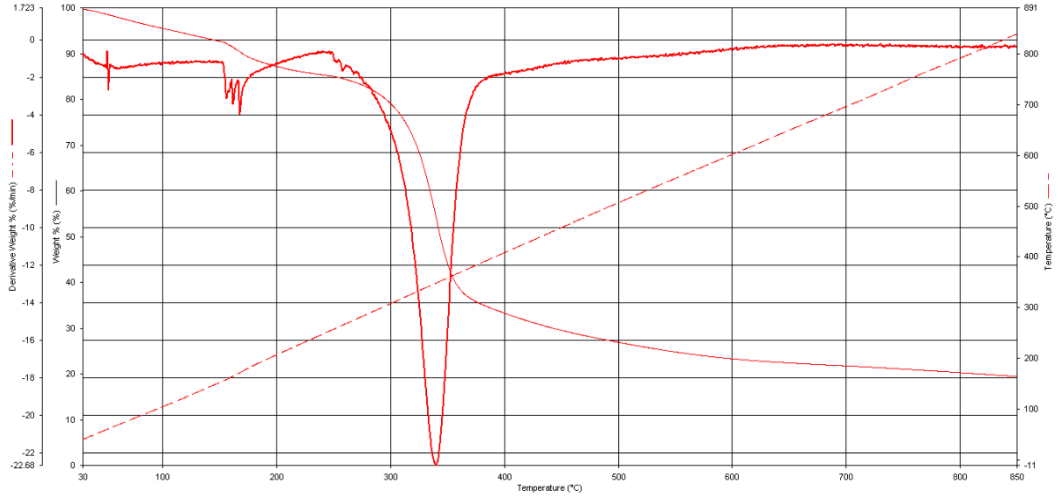


Figure 4.2: TGA graph of neem resin

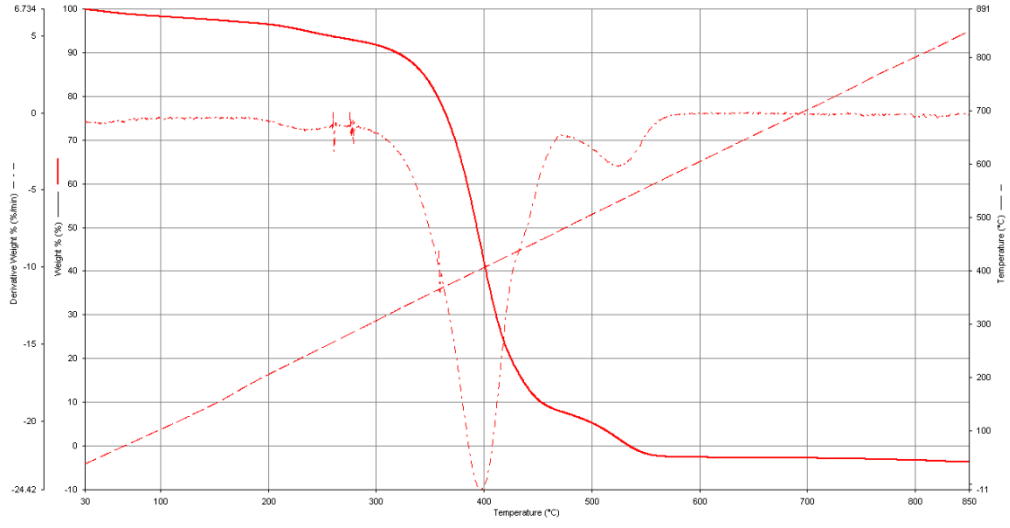


Figure 4.3: TGA graph of polyester composite laminate.

The experimental TGA results are shown in the figures. The figures illustrate the weight loss and derivative weight loss of each specimen at a heating rate of 20°C/min. All the samples were tested at the same temperature range of 30°C to 850°C. For all the specimens above, the weight loss of samples increases as the heating temperature increases. From the figures, the curve can be separated into three major weight loss stages. Neem resin showed the earliest sign of weight loss at 250°C whereas cashew and polyester composite showed slight weight loss at before 300°C. The weight loss might be due to moisture, water, gases, and impurities leave the sample when heating. Further interpretation of this weight loss might be due to physical changes of samples such as softening of molecular arrangement before the degradation under higher temperatures. The principal weight loss of samples started at heating temperature of 300°C to 400°C. The polyester composite showed bigger interval of weight loss at 300°C to 500°C. As the heating temperature increases at 500°C, the weight loss of samples is decreased and ultimately attained constant rate (Sangregorio et al., 2021).

Based on the figures, cashew resins showed the highest weight loss at the early stages of heating below 200°C. This indicates the higher trapped content of water, gases, and low molecular weight volatile components in the sample (Mansa & Zou, 2021). Notable, neem resins showed an irregular weight loss at early heating stage due to impurities in the sample. The differences between the synthetic and natural resins in the material weight loss in early stage is due to thermal maturity of the samples. As thermal maturity of material increases, the low molecular weight compounds are less in the samples. This shows that the synthetic resins showed the highest thermal maturity compared to other natural resins.

In addition, the main break of the curve in neem resins occurred at lower temperature compared to cashew and polyester composite. The neem resins showed