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DEVELOPMENT AND CHARACTERIZATION OF ROSELLE FIBRE REINFORCED VINYL ESTER COMPOSITES

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DEVELOPMENT AND CHARACTERIZATION OF ROSELLE FIBRE REINFORCED VINYL ESTER COMPOSITES

By

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September 2016

Chairman: Prof. Ir. Mohd Sapuan bin Salit, PhD

Faculty: Engineering

Recently, in line with numerous raising environmental concerns, researchers are now replacing synthetic fibers with natural fibers as the main component in composites. Natural fibers are preferred compared to synthetic fibers due to several imminent advantages, such as biodegradable, light in weight, low in cost, and good mechanical properties. With such attributes, roselle is a plant that has been found to be suitable to be used to produce natural fibers. Hence, this research focused on the capability and the sustainability of roselle fiber as lignocellulosic reinforcement for polymer composites. For that purpose, this research had been comprised of four parts. The first part refers to characterization of roselle fiber as a reinforcement material in terms of its mechanical, thermal, physical, morphology, and chemical properties. In this part, the roselle fiber had been compared to other established natural fibers, such as kenaf and jute. The results obtained indicated that the moisture content of roselle fiber was 10.867%, while water absorption was 286.4714%. On top of that, the thermal gravimetric analysis (TGA) was conducted to determine the thermal stability of roselle fiber at high temperature. The results showed that the initial degradation of roselle fiber started at 225^{0}C and the decomposition of lignocellulosic component was completed at 400 ^{0}C. Furthermore, tensile test was conducted to investigate the mechanical properties of roselle fiber. The tensile strength of roselle fiber had been 130-562 MPa. Thus, on the basis of the identified properties, the roselle fiber has been proven to be one of the good and natural fibers that can be used as a reinforced material for manufacturing of polymer composites for different applications, besides saving the cost required to manage agro waste. Next, the second part of this research looked into the effect of plant age (3, 6, and 9 months) upon physical, morphological, chemical, thermal, and mechanical properties. The highest yields of the properties were used for the next study in this research. From the results retrieved, it had been observed that the diameter of roselle fiber increased as the plant attained maturity. However, in contrast to this, the moisture content and the water absorption of roselle fiber decreased as the plant began to mature. Moreover, the

chemical content of roselle fibers from plants of different ages indicated that as the plant matured, the cellulose content decreased. In addition, the tensile strength of roselle fiber decreased from 3 months old to 9 months old. Meanwhile, the thermal analysis results showed that the effect of thermal decomposition of roselle fiber was almost similar for all plant ages. Nevertheless, the 3-month-old roselle fiber displayed the highest yield in mechanical properties. Thus, 3-month-old roselle fiber was used as reinforcement material for the next study, which included the treatment and the composites analysis. Moving on, the third part of this research investigated the effect of chemical treatment upon roselle fiber. This study examined chemical, physical, thermal, mechanical, and morphological characteristics of roselle fiber-reinforced vinyl ester (RFVE) subjected to different fiber treatments. The roselle fiber was treated with alkalization and a silane coupling agent. The treated roselle fiber significantly enhanced most of the properties of vinyl ester (VE) biocomposites compared to those of untreated biocomposite. The results further revealed that alkalization and silane treatments of the fiber changed its chemical properties. The treated fiber improved the attribute of water repellence of the RFVE compared to that of untreated fiber. In addition, the use of silane coupling agent was determined as the best chemical treatment for optimum water absorption effect. Besides, TGA demonstrated that alkalization-treated fiber had improved thermal stability; however, the opposite result was obtained with the silane-treated fiber. Nonetheless, the morphological examination of both treated and untreated RFVE exhibited good fiber adhesion between the treated fiber and the matrix, and less fiber pull-out from the matrix was observed. This observation, particularly, provides a good indication of the interfacial interlocking between the fiber and the matrix, which improved the tensile properties of the composites. In contrast, the impact results revealed that the treated fiber had decreased impact energy compared to that of untreated fiber. Finally, the last part of this research evaluated the mechanical properties (tensile, flexural, and impact strength) and the thermal properties of RFVE subjected to fiber loading. The composites samples were prepared with two different parameters; with fiber contents of 10%, 20%, 30%, and 40 vt%, as well as without fiber (neat VE). The morphological properties of impact fracture samples were studied by using Scanning electron microspcope (SEM). From the examination, the RFVE composite had been found to increase both the tensile strength and the tensile modulus. In fact, the highest tensile strength and modulus had been observed at 20 vt % of fiber loading. However, a decrement was noted on the impact and the flexural strength with the increase in fiber loading. Nevertheless, the SEM showcased good fiber/matrix adhesion and fiber dispersion at 20% fiber loading, which reflected the good properties of tensile strength. However, the agglomeration of the fiber was seen at higher fiber loading. In addition, the TGA and the DTG curves showed three major degradations of RFVE in terms of the loss of moisture content, as well as the degradation of hemicelluloses and cellulose. Other than that, the thermal analysis showed enhancement in the residual content of the composite materials, thereby improving thermal stability. However, no significant difference was observed for degradation in temperature subjected to fiber loading.

Abstrak tesis dikemukakan kepada senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk ijazah Doktor Falsafah

PEMBANGUNAN DAN PENCIRIAN KOMPOSIT VINIL ESTER DIPERKUAT GENTIAN ROSEL

Oleh

NADLENE BINTI RAZALI

September 2016

Pengerusi: Prof. Ir. Mohd Sapuan bin Salit, PhD

Fakulti: Kejuruteraan

Kebelakangan ini, sejajar dengan pelbagai isu alam sekitar, penyelidik sedang menggantikan gentian sintetik dengan gentian semulajadi sebagai komponen utama dalam komposit. Gentian semulajadi akan diutamakan berbanding dengan gentian sintetik kerana terdapat beberapa kelebihan, seperti biodegradasi, ringan, murah , dan sifat-sifat mekanikal yang baik. Dengan sifat-sifat tersebut, rosel adalah sejenis tumbuhan yang sesuai digunakan untuk menghasilkan gentian semulajadi. Oleh yang demikian, kajian ini tertumpu kepada keupayaan dan kelestarian gentian rosel sebagai bahan lignosellulosik bagi pengukuhan polimer. Bagi tujuan itu, kajian ini telah terdiri daripada empat bahagian. Bahagian pertama merujuk kepada pencirian gentian rosel sebagai sebagai bahan pengukuhan dari segi mekanikal, termal, fizikal, morfologi, dan komposisi kimia. Dalam bahagian ini, sifat gentian rosel dibandingkan dengan gentian semulajadi yang telah dikomersialkan seperti kenaf dan Jut. Keputusan yang diperolehi menunjukkan bahawa kandungan kelembapan gentian rosel adalah 10.867%, manakala penyerapan air adalah 286.4714%. Selain itu, analisis termal gravimetrik (TGA) telah dijalankan bagi menentukan kestabilan termal rosel serat pada suhu yang tinggi. Hasil kajian menunjukkan bahawa degradasi awal gentian rosel bermula pada 225^0C dan penguraian komponen lignosellulosik pada 400 ^{0}C. Selain itu, ujian tegangan dijalankan untuk mengkaji sifat mekanikal gentian rosel. Kekuatan tegangan gentian rosel adalah 130-562 MPa. Oleh itu, berdasarkan keputusan yang diperolehi, gentian rosel mempunyai potensial untuk digunakan sebagai bahan pengukuhan untuk pembuatan polimer komposit bagi sesuatu aplikasi yang berlainan, di samping menjimatkan kos yang diperlukan untuk menguruskan sisa pertanian. Seterusnya, bahagian kedua kajian ini mengkaji mengenai kesan umur gentian (3, 6, dan 9 bulan) keatas sifat fizikal, morfologi , kimia, termal , dan mekanikal. Keputusan analisi yang optimum akan digunakan untuk kajian seterusnya dalam projek kajian ini. Daripada keputusan yang diperolehi, diameter gentian rosel adalah berkadar terus dengan kematangan gentian rosel. Walau bagaimanapun, berbeza dengan, kandungan kelembapan dan penyerapan

air gentian rosel menurun dengan kematangan gentian. Selain itu, kandungan kimia gentian rosel adalah berbeza beza mengikut umur gentian rosel semakin meningkat umur gentian kandungan selulosa adalah menurun. Di samping itu, kekuatan tegangan gentian rosel adalah menurun daripada 3 bulan sehingga berusia 9 bulan. Sementara itu, keputusan analisis termal menunjukkan bahawa kesan termal terhadap degradasi gentian rosel adalah hampir sama untuk semua peringkat umur gentian. Namun begitu, gentian rosel berusia 3-bulan menunjukkan keputusan yang optimum dalam sifat mekanikal. Oleh itu, gentian rosel berusia 3 bulan digunakan sebagai bahan tetulang untuk kajian seterusnya, termasuk rawatan kimia dan analisis komposit. Bahagian ketiga kajian ini ialah mengkaji kesan rawatan kimia terhadap gentian rosel. Kajian ini mengkaji sifat kimia, fizikal, termal, mekanikal dan morfologi daripada gentian rosel diperkuat vinil ester (RFVE) yang tertakluk kepada rawatan gentian yang berbeza. Gentian rosel adalah dirawat dengan rawatan alkali dan rawatan agen gandingan silan. Gentian rosel dirawat menunjukkan peningkatan terhadap sifat-sifat penting untuk komposit berbanding dengan gentian rosel yang tidak dirawat. Keputusan seterusnya menunjukkan bahawa rawatan alkali dan silan mengubah sifat-sifat kimia gentian rosel. Gentian dirawat memperbaiki penyerapan air komposit RFVE berbanding dengan gentian yang tidak dirawat. Di samping itu, penggunaan agen gandingan silan adalah rawatan kimia yang terbaik untuk kesan penyerapan air yang optimum. Di samping itu, TGA menunjukkan bahawa gentian dirawat alkali mempunyai kestabilan termal yang baik; walau bagaimanapun, bertentangan dengan keputusan telah diperolehi dengan gentian dirawat silan. Walau bagaimanapun, pemeriksaan morfologi bagi dirawat dan tidak dirawat RFVE memperlihatkan daya lekatan yang baik antara gentian dan matrik, dan kurang gentian ditarik keluar dari matrik diperhatikan. Pemerhatian ini, menunjukkan bahawa kurang ruang antara gentian dan matrik dan gentian dan matrik saing berpaut antara satu sama lain dan ini boleh memperbaiki sifat-sifat tegangan yang komposit. Sebaliknya terhadap keputusan kekuatan impak yang diperolehi dimana kekuatan impak menurun bagi gentian yang dirawat berbanding dengan gentian tidak dirawat. Bahagian akhir kajian ini adalah mengkaji sifat mekanikal (tegangan, lenturan, dan kesan kekuatan) dan sifat termal RFVE tertakluk kepada kandungan gentian. Sampel komposit telah disediakan dengan dua parameter yang berbeza; dengan kandungan gentian 10%, 20%, 30% dan 40 vt %, serta tanpa gentian (Neat VE). Sifat-sifat morfologi kesan patah sampel telah dikaji dengan menggunakan Elektron imbasan mikroskop (SEM). Keputusan komposit RFVE telah meningkatkan kekuatan tegangan dan modulus tegangan. Malah, kekuatan tegangan dan modulus yang tertinggi adalah pada 20% vt kandungan gentian. Walau bagaimanapun, penurunan diperhatikan pada kesan dan kekuatan lentur dengan pertambahan kandungan gentian. Walau bagaimanapun, SEM ini mempamerkan gentian/matrix daya lekatan yang baik dan taburan gentian pada kandungan gentian 20%, dan ini menunjukkan sifat-sifat baik kekuatan tegangan. Walau bagaimanapun, penumpuan gentian dapat dilihat pada kadungan komposit yang mempunyai kandungan gentian yang tinggi. Di samping itu, TGA dan lengkungan DTG menunjukkan tiga degradasi utama RFVE dari segi kehilangan kandungan lembapan, serta penguraian hemisellulos dan selulosa. Selain itu, hasil analisis termal menunjukkan peningkatan dalam kandungan sisa bahan-bahan komposit, seterusnya meningkatkan lagi kestabilan termal. Walau bagaimanapun, tiada perbezaan yang signifikan dapat dilihat bagi penurunan suhu tertakluk kepada kandungan gentian dalam sampel komposit.

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I certify that a Thesis Examination Committee has met on 27 Sept. 2017 to conduct the final examination of **Nadlene binti Razali** on her thesis entitled **Development and Characterization of Roselle fibre Reinforced Vynil Ester** in accordance with the Universities and University Colleges Act 1971 and the Constitution of the Universiti Putra Malaysia [P.U.(A) 106] 15 March 1998. The Committee recommends that the student be awarded Degree of Doctor Philosophy.

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CHAPTER 1

INTRODUCTION

1.1 Overview

Recently, there are a lot of crucial environmental issues such as industrial materials that are harmful to our health, global warming, and disposal of plastic products in our world. One of the common issues is protecting the environment using non-disposable material. Many researchers nowadays have considered producing green material such as natural fibre composites to replace synthetic fibre composites in industrial and everyday activities. Natural fibre is a biodegradable product, thus is not harmful to community health compared to synthetic fibre. Furthermore, it does not cause skin irritation and is safer to handle compared to glass fibre composites. Natural fibre also has a lot of advantages compared to conventional synthetic fibre. The advantages that can be highlighted are that they have high specific properties (strength/modulus to weight ratio) due to its low density and low costs. According to previous research, its low density character makes the composite product light weight which in return, contributes to fuel efficiency in transporting the product or when they are used in vehicle components. Natural fibre is a relatively low cost product because it can be found abundantly, and some of the fibre is sourced from agricultural waste. This is the main benefit to the industry as they can rely on the sustainable material supply and reduce product costs. There are a lot of bio-composite products that have been commercialized from plant based fibres. Some examples of composites based on natural fibres include kenaf, jute, sugar palm fibres and banana pseudo stems.

The roselle fibre is a natural fibre which has good potential to be used in bio-composite engineering. Roselle fibres are found abundantly in Malaysia, Indonesia, Africa and Sudan. The fibre is sourced from bast fibre which has good mechanical and thermal properties. However, there is still a lack of detailed study done on the roselle fibre. Most research uses the fibre as a hybrid reinforcement material in bio-composites. The properties of the roselle fibre are relatively good compared to other established natural fibres such as kenaf and jute.

Although there are a lot of detailed studies carried out on the usage of natural fibre as reinforced material for composites, they are still not perfect. Bio-composites still have limited applications, especially in outdoor applications. The main concern of natural fibre is its hygroscopic characteristic. Moisture content (MC) and water absorption (WA) of natural fibre are some of the important criteria which need to be considered in choosing natural fibre as reinforcement material. This is because MC and WA affects dimensional stability, electrical resistivity, tensile strength, porosity, and swelling behavior of the natural fibre in composite materials. From other published literature, it was found that low MC and less WA in natural fibre is the most desirable criteria for polymer composites as it can help overcome the problem mentioned above (Jawaid & Abdul Khalil, 2011). This is probably due to the absence of ability of fibre to retain water within composites, thus promoting the degradation of the composites (Rowell et al. 2000). Chemical content contributes a major effect towards the moisture content of natural fibre. This is due to the presence of hydroxyl bonding or OH group in the cellulose structure. There are three major chemical components in fibre which are lignin, hemicelluloses, and cellulose in cell walls which affect the ability to attract and hold water molecules. Lignin has the lowest affinity for water and hemicellulose the highest. The affinity of cellulose for water is between these two. It is important to understand the sorption capacities of the three major chemical components relative to the sorption capacity of the fibre. Besides that, another critical problem in using natural fibre is its hydrophilic behavior which contrasts with resin, which is hydrophobic. This phenomenon creates an adhesion problem between the fibre and the matrix. In order to overcome this crucial problem, chemical modification of natural fibre needs to be done.

This study discusses the roselle fibre's characteristics in terms of its mechanical, chemical, thermal, physical and morphological properties compared to other established bast fibres. Besides that, the optimum mechanical, thermal, and physical properties of different plant ages will also be discussed thoroughly. The roselle fibre used was treated with alkalization and a silane treatment. This treatment processes are hopefully useful in treating the roselle fibre to produce roselle fibre composites with better mechanical, thermal and physical properties.

1.2 Problem Statement

Currently, studies on new materials focus on enhancing their mechanical and physical properties while maintaining their 'green' attributes. This includes studies involving composites which dwell with two discreet materials in a symbiotic manner by taking advantage of each other's properties to create new material with better attributes in general.

Composite materials are widely used in the aircraft and automotive industries. From the viewpoint of resource energy conservation, demands such as lightweight conversion, high strength characteristics and improvement in material design have increased in the manufacturing and industry sector.

Nowadays, increasing numbers of researchers are focusing on natural fibres as a reforcing agent in polymers. Natural fibres are more environmental friendly compared to glass and carbon fibres which are relatively more expensive and non-biodegradable. In other words, glass and carbon fibres contribute to adverse environmental effects.
Natural fibres have attracted many researchers to explore their potential to replace current reinforced materials such as glass and carbon fibre. Among the interesting characteristics of natural fibres are its relatively low cost, low density, environmental-friendly, bio-degradable material, acceptable specific strength properties, and ease of seperation. Although there are many advantages of using natural fibre, it is not flawless. Previous research have found that its strength is lower compared to glass and carbon fibre. Therefore, re-strengthening is required for natural fibres before it can be used as

composites. In order to increase the strength of natural fibre composites, a detailed understanding of the fibre used is important.

One of the natural fibres that can be found in tropical areas is the roselle fibre. They are commonly used as an infusion and to produce bast fibre. There are various uses of roselle. The fruit is commonly used in medical applications (Tori Hudson, 2011; Mungole and Chaturvedi, 2011) and in the food industry (Wilson, 2009), while the fibre is used as textile (Managooli, 2009). In Malaysia, after a year or the first harvest season (after 3 months), the roselle plant will be cut, and it will become waste. This is because the quality of the roselle fruit is not good after a year or after the first harvest. In order to use this plant efficiently, the fibre can be used as reinforcement material for polymer composites. Besides that, it can increase the planters income by utilizing the roselle stem waste to the reinforced fibre. It is important to understand the physical, thermal, mechanical, and chemical properties of roselle fibre before it is used in industrial applications such as in automotive and construction engineering.

The roselle fibre is one of the natural fibres that have attracted researchers to explore its capability as a reinforcement material in composites. Researchers have reported work on modifications of the roselle fibre in order to improve the fibre/matrix interfacial bonding for the fabrication of polymer composites for different applications (Kaith and Chauhan, 2008; Chauhan and Kaith, 2011, 2012a, b; Ramu and Sakthivel, 2013). A few researchers have also reviewed papers covering the chemical and mechanical properties of roselle fibres in polymer composites (Thiruchitrambalam *et al*. 2010; Chauhan & Kaith, 2012b). From the literature review done, it was found that published research on detailed studies of the roselle fibre in terms of its mechanical, chemical, thermal, physical and morphology analyses are very limited (Ramu & Sakthivel, 2013).

Although roselle fibres have some advantages over synthetic fibres especially in terms of material costs, they present certain disadvantages regarding its fibre matrix interface. Crucial issues in using natural fibres include poor adhesion between fibre and matrix as well as high water absorption and high moisture content which leads to fibre instability that can cause micro-cracking of the composite and degradation of the mechanical properties. The performance and quality of the composite product highly depend on the fibre matrix adhesion because it influences the stress transfer from the matrix to the fibre. Researchers have reported modifications of the roselle fibre to improve the fibre/matrix interfacial bonding for fabrication of polymer composites for different applications. In addition, review papers covering the chemical and mechanical properties of roselle fibres in polymer composites have been published (Nadlene et al. 2016).

Alkalization is a common surface treatment for natural fibres. Many studies have examined the effect of transformed fibres into a more hydrophobic nature by alkali treatment. The alkali treatment also reduces the fibre's diameter, thus increasing the aspect ratio. The increase in aspect ratio roughens the surface which further improves the mechanical properties of the fibre, resulting in better bonding between the fibre and matrix. After treatment, the roselle fibre clearly develops a rough surface and the impurities are removed, leading to an improvement of the mechanical properties of the composites. Physically, the presence of a rough surface provides a mechanical locking between the fibre surface and the matrix, which may enhance their interfacial bonding. An additional effective chemical treatment of natural fibres involves the immersion in a silane coupling agent. Many studies were performed using this method, and an extensive review was performed by Xie et al. (Xie et al., 2010; Dittenber & GangaRao, 2012). Before immersion into this chemical, pre-treatment with NaOH is required to enable a more effective reaction with the chemical. This treatment improves the interface and bonding between the fibre/matrix surfaces. Generally, there have 4 steps in the chemical reaction between the silane coupling agent and natural fibres which are hydrolysis, self-condensation, hydrogen bonding, and finally surface grafting (Xie et al., 2010).

In this study, the properties of roselle fibres that were uniquely treated and reinforced were investigated. The novelty of using silane coupling agent treatment and vinyl ester composite reinforcement were explored in order to contribute to the existing knowledge on roselle fibres application in the field of natural fibre composites. In addition to that, a pioneer study on the optimum roselle plant age for fibre to be extracted for the said purpose was conducted in the spirit of new knowledge.

1.3 Research Objectives

The objectives of this research are stated as below:

1. To characterize the physical, chemical, mechanical, morphological and thermal properties of roselle fibre.
2. To evaluate the effects of the roselle plant's age to the physical, chemical, mechanical, morphological and thermal properties of its fibre.
3. To investigate the effect of treated roselle fibres on the mechanical, physical and thermal properties of vinyl ester composites.
4. To determine the behaviour of roselle fibres reinforced with vinyl ester composites in terms of its mechanical and thermal properties.

1.4 Scope of Study

The aim of this research is to accomplish a greater understanding of the roselle fibre's properties in terms of its mechanical, physical, and chemical properties, and morphology. The roselle fibre were treated with different chemical treatments in order to evaluate the effect of treatment to the properties. Finally the fibre was blended with vynil ester resin to form composite samples. The methodology in this research is experimental investigation. The research was divided into four different phases.

The first phase of the research is fibre characterization. The roselle plant was collected from Mersing, Johor and Merlimau, Melaka. The plant was collected randomly. The roselle fibre was extracted from the roselle plant using the water retting method. The extracted roselle fibre was then used as a research sample. This study does not cover the details on the plant and the biological effects of the plant in terms of location and species.

This study focuses on the characterization of physical (density, dimension, water absorption and moisture content), thermal (Thermogravimetric analysis), mechanical (tensile strength properties), chemical compositions (NDF and ADF) and morphology properties (SEM analysis). The results were then compared to other established bast fibres such as kenaf and jute to evaluate the potential of the roselle fibre as a reinforcement material in polymer composites.

The second phase of this research is to investigate the effect of plant age on the mechanical (tensile properties), chemical composition (NDF and NDT), thermal (Thermogravimetric analysis), physical (dimension, density, water absorption and moisture content), and morphology (SEM analysis) characteristics of the roselle fibre. Three different fibre ages which are 3, 6, 9 months were chosen for this study. The results of these three different ages were compared and the properties of the sample were observed experimentally. The type of fibre which gives the optimum value in mechanical, thermal, physical properties was selected for the next phase of this research.

The third phase focuses on the effect of chemical treatments on the roselle fibre. This is the critical part of the research where the properties were discussed and observed thoroughly. The chemical treatments were studied to investigate which treatment contributes to the biggest improvements in the mechanical, thermal, and physical properties of the roselle fibre reinforced with vynil ester. The parameters used in this phase are different concentrations of NAOH and different types of fibre treatments which are alkalization and silane coupling agent. The parameter studies were conducted to identify the influences of treatment to the properties of the roselle fibre. The roselle fibre underwent two testing in order to check the properties after treatments which are thermal, physical, mechanical and chemical properties. FTIR were conducted for chemical composition analysis. The results of untreated and treated samples were compared. After the fibre was studied upon, a minimum 5 vt% of fibre loading was prepared for the composite samples in order to evaluate the surface interaction between fibre and matrix on the mechanical properties and water absorption. The fibre matrix interaction was studied using Scaning Electron Microscopy (SEM). Moreover, physical properties' tests such as water absorption were conducted. Tensile and impact test for composite samples were carried out to investigate the effect of chemical fibre treatment. The highest yield in mechanical, physical and thermal properties will be used for the next phase.

RFVE (roselle fibre reinforced vinyl ester) was prepared using four different fibre loadings which are 10%, 20%, 30%, and 40%. The fabrication of composite samples was prepared using the hand lay-up method in room temperature. The mix of fibre and resin were blended together using a mechanical stirrer. The composite samples were tested for their mechanical properties and thermal degradation. Tensile, flexural and impact test were conducted to investigate the mechanical properties of RFVE. The results of neat VE and RFVE were compared to obtain the optimum results in mechanical properties. A Thermogravimetric analysis was conducted to investigate the thermal degradation of VE and RFVE. SEM analysis was carried out to analyze the fracture behavior of the composites.

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