Mechanical and Thermal Properties of Roselle Fibre Reinforced Vinyl Ester Composites

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Roselle (*Hibiscus sabdariffa* L.) bast fibre reinforced vinyl ester (VE) was prepared using a hand lay-up method and an internal mixer. The composite samples were prepared under two different parameters: with various fibre contents; and without fibre (neat VE). The mechanical properties (tensile and impact strength) and thermal properties were investigated. The morphological properties of impact fracture samples were studied using a scanning electron microscope (SEM). Roselle fibre reinforced VE (RFVE) composites showed increased tensile strength and tensile modulus. The highest tensile strength and modulus were at 20vt% fibre loading. However, impact strength decreased as the fibre loading increased. SEM showed that there was good fibre/matrix adhesion and fibre dispersion for 20% fibre loading, which was reflected in the good tensile strength properties. However, fibre agglomeration was seen at higher fibre loads. The results from thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) showed three major degradations of the RFVE, which were the loss of moisture content, degradation of hemicelluloses, and degradation of cellulose. The thermal analysis showed enhancements in the residual content of the composite materials, thereby improving the thermal stability. However, there was no major difference seen in the degradation temperature.

Keywords: Roselle fibre; Vinyl ester; Mechanical properties; Thermal properties

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INTRODUCTION

Environmental issues are currently being addressed by many scientists and researchers. It has become common agreement that these efforts are vital to ensure the future survival of mankind. In line with this objective, material engineers have conducted studies to replace current reinforced materials with natural fibres (Aji *et al.* 2009). It is important for these replacement materials to be able to portray desired capabilities similar to their counterparts while introducing other "green" characteristics.

Natural fibres have been utilised for material reinforcement for more than 3000 years (Taj *et al.* 2007). With recent advances in technology, natural fibres have been combined with polymers (Azwa *et al.* 2013). Several types of natural fibres have been used for this purpose, such as kenaf, roselle, jute, sugar palm, oil palm empty fruit bunch, sisal, pineapple leaf, rice husk, kapok, wood, barley oats choir, and abaca (Nguong *et al.* 2013).

Several different reasons have attracted material engineers to the use of natural fibres other than wood for reinforcing polymer composites such as the reduction of timber usage and degradation of unused natural fibres. Other advantages include low cost, good mechanical properties, abundant availability, material renewability, biodegradability, and an abrasive nature for ease of recycling (Joshi et al. 2004). Unused natural fibres can be processed into composite boards, or other forms suitable for various applications, while still preserving the environment.

Natural fibres such as roselle (*Hibiscus sabdariffa*) are found in abundance in nature and are cultivated in Borneo, Guyana, Malaysia, Sri Lanka, Togo, Indonesia, and Tanzania. To date, very limited studies have been done on the application of roselle fibres and their composites (Ramu and Sakthivel 2013). The probable uses of roselle fibres and their composites have been explored to further investigate the fibre's potential for development as a "green" composite (Razali *et al.* 2015). Roselle fibre is a bast type fibre. The difference between roselle fibres and other fibres lies in their composition, *i.e.*, the ratio of cellulose and lignin/hemicellulose and orientation or the spiral angle of the cellulose microfibril (Kalia *et al.* 2011). However, the structure of bast fibre is mostly similar to all of the other types of fibres. Usually, the tensile strength and Young's modulus of fibres increase as the cellulose content increases. The ductility of plant fibres depends on the orientation of the microfibrils compared with the fibre axis. If it is spiral, then the material is ductile. However, if it is parallel, it is rigid, inflexible, and has a high tensile strength (Kalia *et al.* 2011). The surface of a roselle fibre is smooth, but burrs or foreign particles and dirt can be present if no surface treatment is applied. Having a smooth surface is a major disadvantage for natural fibres that adds to undesirable hydrophilic properties. However, a smooth surface may be roughened using a pre-treatment process. When the surface is rough, interlocking between the polymeric materials is present, resulting in improved interfacial bonding. This shows the importance of studying fibre morphology (Chauhan and Kaith 2012a).

For the past few years, there have been many studies conducted on the roselle fibre surface modification and the capability of roselle fibre in the polymer composites application. Singha and Thakur (2008) carried out an evaluation on the roselle fibre reinforced phenol fomaldehyde subjected to the different forms of fibre (particle, short, and long fibre) and fibre loading. In this study they have found that 30% of fibre loading and particle type of fibre gave the optimum properties (Singha and Thakur 2008a,b, 2009). Chaudhan and Kaith (2012) studied a novel of graft copolymer of roselle fibre that focused on the evaluation of the modulus elasticity, modulus of rupture, stress at the limit of proportional, and hardness of the modified roselle reinforced phemol formaldehyde (Chauhan and Kaith 2011, 2012a,b). Besides that, some of the researchers used roselle fibre combined with other natural fibre to form hybrid composites. Their objective was to know the optimum content of fibre loading in order to obtain the optimum results of mechanical properties. From searching the literature, there appears to be a need for detailed studies conducted on thermal behaviour of roselle fibre reinforced polymer material (Nadlene *et al.* 2016).

A composite is highly influenced by its matrix. It can be customised for a particular use by selecting the appropriate polymer. For example, use of vinyl ester (VE) may improve a composite's stiffness, dimensional stability, chemical resistance, and strength. In addition, vinyl ester costs less than epoxy resins (Aprilia *et al.* 2014).

VE possesses mechanical properties relatively similar to those of epoxy resins, particularly in terms of hydrolytic stability. It is not difficult to handle this polymer at room temperature, so it offers greater control over cure rate and reaction conditions compared with epoxy resins.

However, VE resin is brittle, and reinforcement of the fillers is necessary to improve performance and minimise the cost of the resin (Ku *et al.* 2011). Particulate fillers are normally mixed with VE during shape casting of the product to reinforce the resin.

In this study, roselle fibre was chosen as the filler material for VE. Roselle fibres were pre-treated before being blended with the matrix to form the composites. The characteristics of roselle fibre reinforced VE containing various percentages of roselle fibre were compared with neat VE. The effects of roselle fibre on the mechanical and thermal properties as well as morphology of composites were investigated.

EXPERIMENTAL

Materials

Fibre samples

The VE used in this study was obtained from the Polymer Technology Pte. Ltd. (Singapore). The density, heat distortion temperature (HDT), viscosity, and glass transition temperature of VE were 1.6 g/cc, 120 °C, 400 cps, and 104.44 to 143.33 °C, respectively. Methyl ethyl ketone peroxide (MEKP) was used as a hardener.

Roselle plants were collected from Selangor, Malaysia. Roselle fibres were isolated using a water retting process over 14 days. The retted stems of the roselle plants were washed in running water, and the fibres were removed manually. The fibres were then cleaned and dried in sunlight for four days. The fibres were then immersed in 6% NaOH solution for three hours in a basin at room temperature. After that, the fibres were immersed in silane solutions for 24 h.

After chemical treatment, the fibres were thoroughly washed with running water and dried in an oven at 104 °C for 48 h to eliminate moisture. Finally, the fibres were ground and segregated using a sieving machine (100 to 425 µm) to create the composite samples. The water retting process, chemical treatment, and particle form fibre preparation are illustrated in Fig. 1.

Composite samples

A wet hand lay-up process was used for sample preparation of roselle fibre reinforced vinyl ester (RFVE) composites. A rectangular mould constructed from an aluminium sheet was used for the composite samples. The samples were prepared by mixing 5, 10, 20, and 30 wt% of roselle fibres.

The roselle fibres were heated in an oven at 104 °C to eliminate moisture. First, the roselle fibres were gradually added to the VE composite and stirred using a mechanical stirrer at 100 to 250 rpm until the mixture was uniformly distributed. Then, 2.5 wt% MEKP as a hardener was added to the composite mixture for curing (Aprilia *et al.* 2014).

Finally, the mixture of roselle fibre and VE resin was poured into the aluminium mould and cured for 24 h at room temperature, as shown in Fig. 2. The samples were cut from the cured composites into certain dimensions, according to the ASTM, for the tensile, and impact tests.

Fig. 1. Fibre preparation of roselle fibre; a) roselle plant; b) stems were cut and immersed in water during the water retting process; c) after 14 days, the fibre was ready to be extracted; d) fibre extraction; e) roselle fibres were dried under sunlight; f) alkalisation of roselle fibres; g) silane treatment; h) fibres ready to grind; i) roselle fibres in particle form (100 to 425 µm)

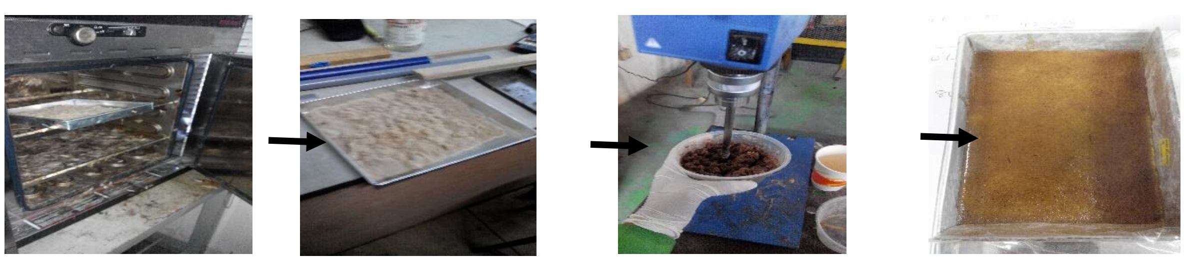

Fig. 2. Preparation of roselle fibre reinforced vinyl ester composites; a) fibre heated in the oven; b) fibre preparation; c) mix of resin and fibre; d) mixed composites poured in the mould

Methods

Thermogravimetric analysis

Thermal characterisation of the treated and untreated roselle fibres was conducted using a Q-series thermal analysis machine from TA Instruments, Malaysia. ASTM D3850 (2006) was used in this analysis. Thermogravimetric analysis (TGA) measures the weight changes in a material as a function of temperature (or time) under a controlled atmosphere. It is important to determine the degradation of natural fibres at a high temperature before they are used in a polymer composite. Approximately 4.8 mg of roselle fibres was placed in the chamber. The analysis was performed in a nitrogen atmosphere at a temperature range between 50 and 600 °C, and with a heating rate of 10 °C/min.

Mechanical testing

To evaluate the effects of fibre surface treatment on its mechanical properties, tensile and impact tests were performed. The goal was to analyse the interfacial bonding between fibre and matrix from failure samples. The tensile test is a simple method that reveals the mechanical properties of a composite material. Several significant mechanical properties can be obtained from tensile tests, such as Young's modulus, tensile stress, maximum elongation, tensile strain, and yield stress. The samples were cut into 150 x 15 x 3 mm strips using a band saw. The tensile properties of the RFVE composites were determined using a universal testing machine (model Instron 5556) according to ASTM D5083 (2010). The gauge length of the samples was 100 mm, and the cross-head speed was 1 mm/min, with a 5-kN load cell. Five samples were prepared to reflect each group of the different fibre treatments.

The Izod impact test was performed according to ASTM D256 (2010) at room temperature. The impact test was performed using a digital Instron Ceast 9050 pendulum impact tester. The samples were cut into 64 x 12.7 x 3 mm strips for testing. At least five samples were examined in each test. The impact strength was calculated by dividing the impact energy by the cross section area of the specimen, as shown in Eq. 1.

$$\text{Impact strength} = \text{Impact energy (J)} / \text{area (mm}^2\text{)} \qquad (1)$$

Morphological analysis

Morphological studies were performed in detail on the fractured surface of impact test samples using a Hitachi S-3400N (Japan) scanning electron microscope (SEM), at an accelerating voltage of 15 kV. The samples were coated with gold to provide electrical conductivity, which did not significantly affect the resolution, allowing for high-quality results.

RESULTS AND DISCUSSION

Tensile Properties

According to previous reports, tensile properties depend on several factors: material properties, method of composite preparation, sample condition, speed of testing, void content, and volume percent of reinforcement.

The effects of fibre loading from 0 to 40% on the tensile properties of VE composites are presented in Fig. 3. From the results obtained, it can be observed that the load increased gradually to the maximum value, and then it suddenly decreased, implying that a brittle fracture occurred in the material. It can be seen that both the tensile strength and tensile modulus of the composite samples indicated the same trends, which increased up to 20% roselle fibre. However, further increases in fibre loading (20% to 40%) showed a reduction in results for tensile strength and tensile modulus. El-Sheikeil *et al.* (2014) also observed that tensile strength decreased after 20% kenaf fibre loading in polyurethane composites. The presence of 10% fibre loading increased tensile strength by approximately 51% (26.61 MPa) compared with the neat vinyl ester (17.52 MPa). The optimum value was at 20% fibre loading, which increased the tensile strength of the roselle fibre reinforced VE by approximately 136% (41.50 MPa). The lowest tensile strength was at 40% fibre loading. The total reduction at 40% loading was 57% (7.43 MPa) compared to neat VE.

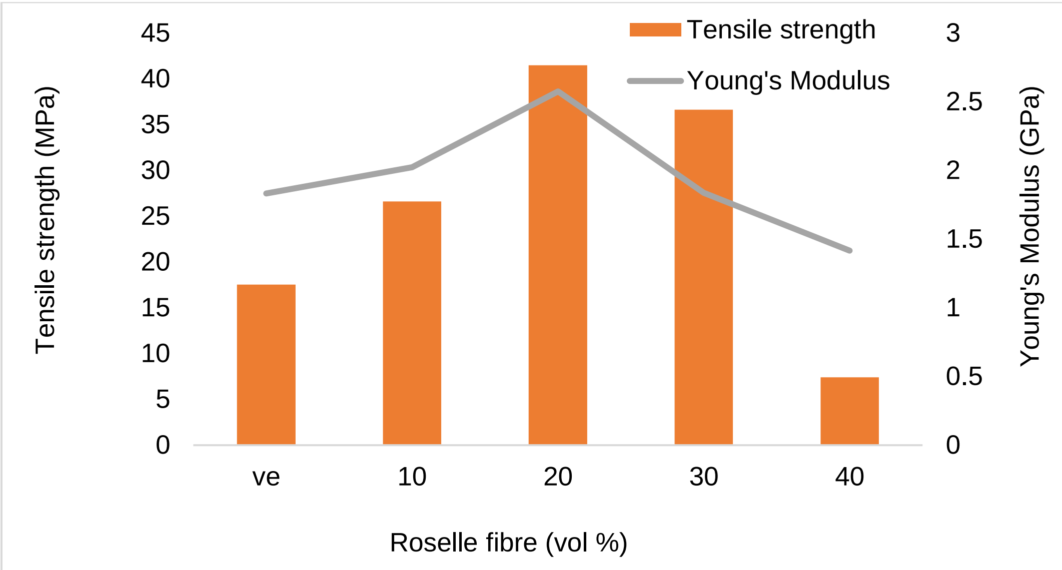

Fig. 3. Tensile properties of roselle fibre reinforced vinyl ester

As expected, the tensile strength and modulus increased with the introduction of roselle fibres. This phenomenon occurred because fibres acted as a load carrier in the matrix. Good tensile strength depends more on the effective and uniform stress distribution between the fibres and the polymer. Fibre also acts as a reinforcing material that can stop crack propagation. Fibre addition can improve the mechanical properties of the composites. Increases in the tensile strength and modulus indicate that the fibre has higher tensile properties than the neat polymer. The optimum tensile strength and modulus from 20% fibre loading was due to the good interfacial bonding between fibre/matrix and uniform dispersion of the fibre in the matrix. It can be seen in Fig. 7(c) that the homogeneity of the distribution was more uniform when compared with the other fibre loadings. The stress applied was transferred effectively because of the good interfacial bonding and lower void content of the samples. Furthermore, the mechanical interlocking fibre/matrix was good enough at 20% fibre loading to transfer load from the matrix to the fibre, allowing the reinforcing effect of the cellulose fibre to predominate. However, the tensile strength and modulus dramatically decreased beyond 20% fibre loading. The decrease in tensile strength and modulus at higher fibre loading was due to the increase in the particle concentration, which led to fibre agglomeration. Agglomeration of fibres can make the composites brittle. Aprillia *et al.* (2014) found that fibre agglomeration reduced the tensile strength of composites because of the low compatibility of the fibre in the matrix. The low compatibility showed that the capability of stress transfer from the matrix was relatively poor (Aprilia *et al.* 2014).

The lowest tensile strength and modulus was at 40% fibre loading. This was due to the weak interfacial bonding between fibre/matrix, which can be clearly seen in Fig. 7(e). The resin was insufficient to cover up all the fibre in the composites. At the beginning of the tensile test, the matrix underwent a low force that transferred easily along the matrix/fibre interface. Shearing forces were developed at the interface because the deformation ability of the matrix was higher than the fibre's. When a higher load was applied, the higher shearing forces formed simultaneously (Yan *et al.* 2013). Higher fibre loading developed more fibre to fibre contact, compared with the fibre/matrix. The composites failed at a small load, which indicated a low tensile strength due to the weak interfacial bonding between fibre and matrix.

The effect of fibre loading considerably influenced the tensile properties of the RFVE. Tensile properties of composites are closely related to the fibre content, fibre properties (Nadlene *et al.* 2015), matrix properties, interfacial bonding between fibre and matrix, and the uniformity of the fibre dispersion in the matrix. In this study, roselle fibres successfully improved tensile properties (strength and modulus) up to a 20% fibre load, compared with the neat VE.

Impact Properties

A material's ability to absorb and dissipate energy under shock loading or impact is best represented by the impact property (Santhosh *et al.* 2014). The composite impact energy level depends on several parameters, such as the nature of the constituents, construction and geometry of the composites, fibre arrangement, fibre/matrix adhesion, and test conditions. For impact loading, the matrix fracture, fibre/matrix de-bonding, fibre breakage, and fibre pull-out are important modes of failure in fibre composites. De-bonding may occur if the applied load exceeds the fibre/matrix interfacial bond. This load is normally transferred by shear force to the fibres. The frictional force along the interface may transfer the stress to the de-bonded fibre. Fibres may break if the fibre stress level exceeds its strength. Ultimately, energy is dissipated when the broken fibres are pulled out of the matrix. The amount of energy is determined by the difference in potential energy before and after the test. The impact strength can be calculated by dividing the recorded impact energy by the cross-section area of the sample (Yahaya *et al.* 2014).

Figure 4 illustrates the impact properties of RFVE subjected to increasing fibre loads (10%, 20%, 30%, and 40%). According to the results obtained, the presence of roselle fibre in the VE matrix reduced the impact strength of the composites. The neat VE showed the highest impact strength (5.438 kJ/m^2). A 10% loading gave a reduction of 14%, compared with the neat VE. However, the impact properties increased from 10% to 20% fibre loading, with 20% providing the optimum result. It is worth noting that the impact properties of 20% fibre loading were only slightly different than neat VE. The reduction in impact strength was only 2.39%. The impact strength of RFVE decreased from 20% to 40% fibre loading. Overall, incorporating roselle fibre loads, from 10% to 40%, into a VE matrix minimally modified the impact properties of VE composites, which agrees with previous literature on the subject. Impact strength indicates the total energy absorbed by the composite material before initiating the crack or failure of the material, and the energy absorption is influenced by the fibre loading and fibre spacing in the composites. Thus, decreased impact strength with increased fibre loading is related to the reduction in the effective stress transfer between the fibre and matrix at a high fibre content, and also the decreased ability of the composites to absorb energy during composite failure. The utilisation of short fibres tends to reduce the elongation at break, thus reducing impact absorbing energy (Manshor *et al.* 2014).

For the fibre reinforced polymer composites, fibre is the main factor that affects the impact resistance of the composites, as fibres act as a medium for stress transfer and as a response to the formation of a crack in the matrix (Sreenivasan *et al.* 2011). The impact strength results that exhibited the capability of roselle fibre to bear the stress transferred from the matrix declined as fibre loading increased in the composites. In this study, the lowest impact strength was at high fibre loading because of the difficulty in wetting of the roselle fibres by resin at high fibre loading, as seen in Fig. 7(e). It is clear in Fig. 7(e) that the fibre was not fully covered by the resin. At high fibre loading, fibre agglomeration may occur, creating more fibre to fibre contact instead of fibre and matrix interaction. Consequently, when there is more fibre to fibre contact, the effective stress transfer from the matrix to fibre is harder to achieve (Rojo *et al.* 2015). The impact at low fibre loading (10%) was slightly lower than that at 20% fibre loading because of the poor fibre population caused by low load transfer capacity among the fibres. As a result, stress accumulated at certain points in the matrix, which eventually failed in the composites. In addition, some defects were present in the form of air bubbles that were incorporated into the composites by the fibres (Fig. 7(b)), which modified the purity of the material and could also have acted as points of stress. The optimum result was found at 20% fibre loading. This might be due to the uniform dispersion of fibre and good adhesion between the fibre/matrix, as seen in Fig. 7(c). The homogeneity of the mixture between the fibre/matrix and uniform fibre dispersion also played an important role in improving the impact properties of the composites.

In conclusion, the main factors that influenced the decreasing impact strength in the RFVE were weak interfacial bonding between fibre/matrix and fibre content in the composites. Poor adhesion, and agglomeration of the fibres weakened the impact properties of the composites because they stopped the stress transfer mechanism from matrix to fibre. Another potential cause that may have reduced the impact strength was stress concentration around fibre ends or areas of poor fibre adhesion (Manshor *et al.* 2014). This is in agreement with Yang *et al.* (2004), who studied the change in impact strength with filler content. Micro-spaces between the filler and matrix may induce poor interfacial bonding, thus causing micro-cracks when impact occurs (Figs. 7(a–d)), consequently inducing easy crack propagation.

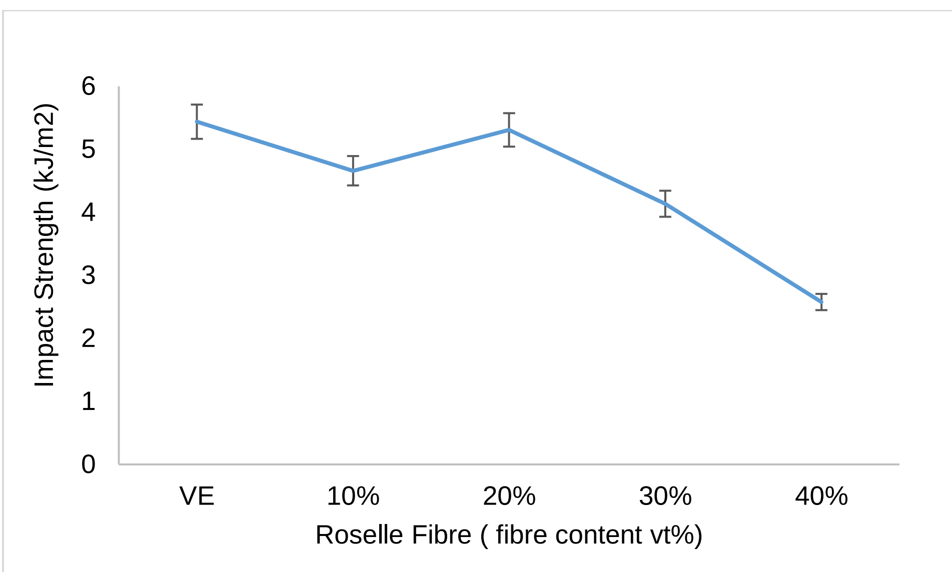

Fig. 4. Impact properties of roselle fibre reinforced vinyl ester

Thermal Analysis

Thermogravimetric analysis is an important tool for analysing thermal behaviour and stability by determining the mass change of the composites when exposed to a high temperature as a function of time (Van de Velde and Baetens 2001). Figures 5 and 6 illustrate the TG and DTG of RFVE subjected to various fibre loadings. The details of degradation temperature can be found in Table 1. Based on the data in Figs. 5 and 6, as well as Table 1, the transition temperature range, transition peak temperature, mass loss, and residual char at 700 °C for RFVE composites were calculated using TGA testing at a heating rate of 10 °C/min.

From the obtained results, it can be seen that the 10% fibre loading of the RFVE samples gave the highest first degradation temperature, at 368.41^{0}C followed by 20 and 40% fibre loading, which are at 367.83 and 365.5 respectively. According to Fig. 5, the neat VE had only one phase of degradation during the heating process. The main range for the degradation temperature was 224.5 to 500 °C, and the peak degradation temperature was at 440.17 °C with a char residue of 5.117%. It can be concluded that the neat VE resin had a very low moisture content because there was no peak in the VE DTG curves.

Generally, the TGA curves showed that there were three main degradation phases present during the RFVE heating process. The final thermal degradation characteristic of reinforced fibre is highly influenced by the individual thermal degradation properties of the matrix and fibre (Ray *et al.* 2004). The first degradation was governed by the moisture that evaporated in the 30 to 110 °C range (Nadlene *et al.* 2015). The percentage of moisture loss increased with increases in the fibre loading. This phenomenon occurred because of the natural characteristics of the natural fibre, which can easily absorb the moisture around it; thus, an increase in fibre loading will ensure more moisture is absorbed. The second and third phases of RFVE occurred between 200 and 380 °C, and 375 and 525 °C, respectively. The degradation of the second and third phases was believed to have derived from the decomposition of hemicellulose and cellulose components in the natural fibre (Ishak *et al.* 2011).

In this study, 10% roselle fibre in VE composites showed the highest degradation temperature compared to other composites, as higher fibre loadings decreased the thermal stability of the composites. However, the difference between the peak degradation temperatures was not great, as the difference was less than 5%.

Table 1. Thermal Properties of Roselle Fibre Reinforced Vinyl Ester

Composites	Weight loss (%) in temperature range of 30 to 110 °C	Transition temperature T_1	Peak temperature	Transition temperature T_2	Peak temperature	Char residue
VE	0.3344	-	-	224.5-500	440.17	5.117
VE + 10% fibre	1.0672	200 - 375	368.41	375 - 525	431.25	4.0696
VE + 20% fibre	1.1916	200 - 380	367.83	380 - 525	428.33	5.392
VE + 30% fibre	1.3466	200 - 378	357.59	380 - 525	427.833	5.7043
VE + 40% fibre	1.6014	200 - 380	365.5	380 - 525	428.84	6.5713

Table 1 shows the amount of residual char produced at the end of the heating process in the TGA analysis.

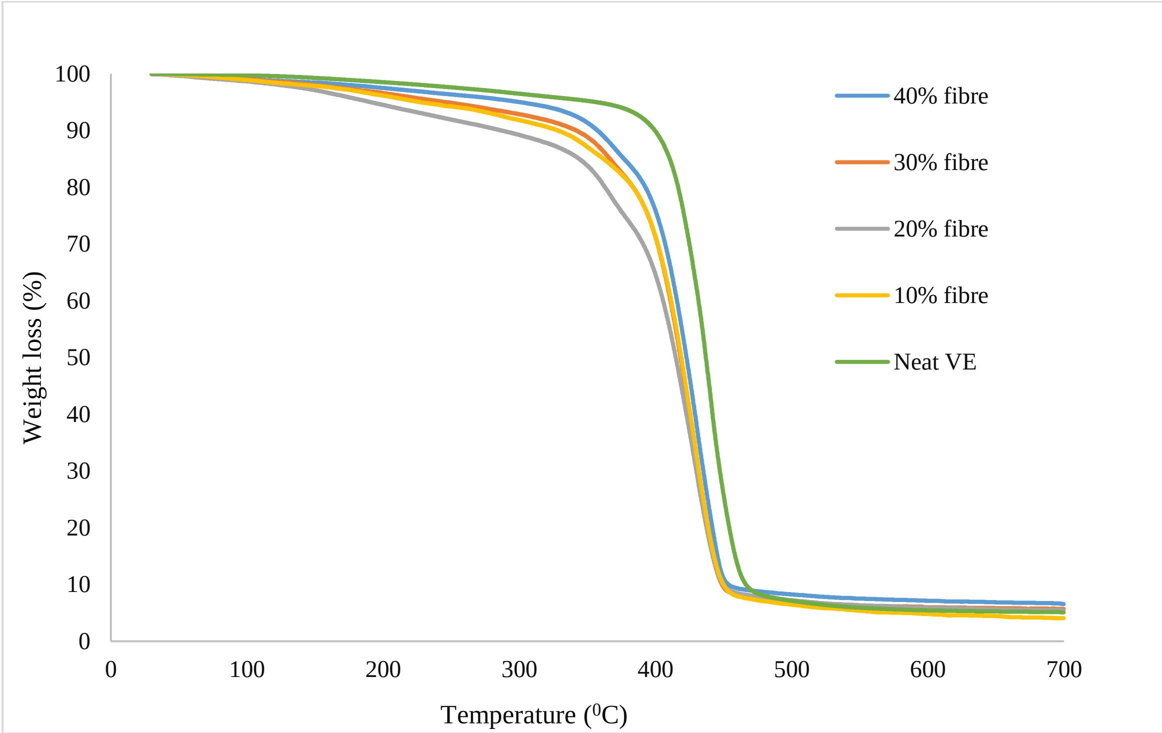

Fig. 5. TGA of roselle fibre reinforced vinyl ester subjected to fibre loading

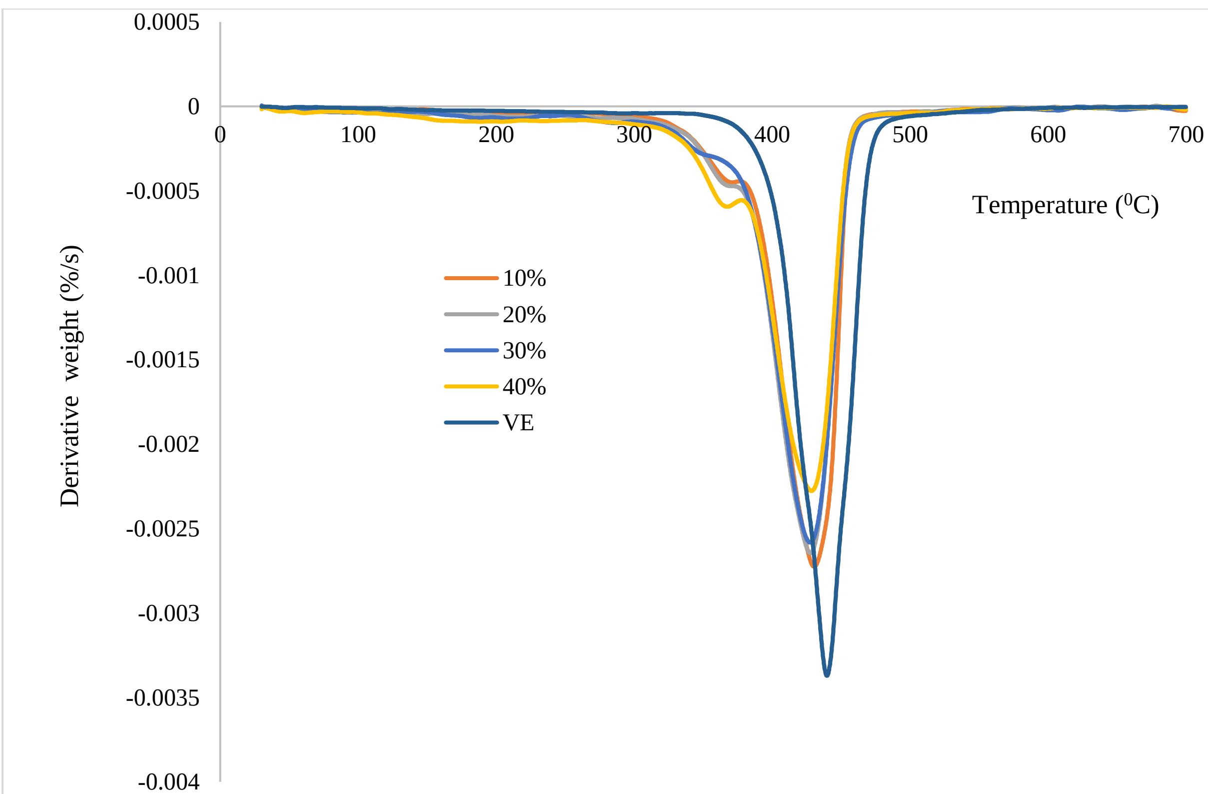

Fig. 6. DTG curve of roselle fibre reinforced vinyl ester subjected to fibre loading

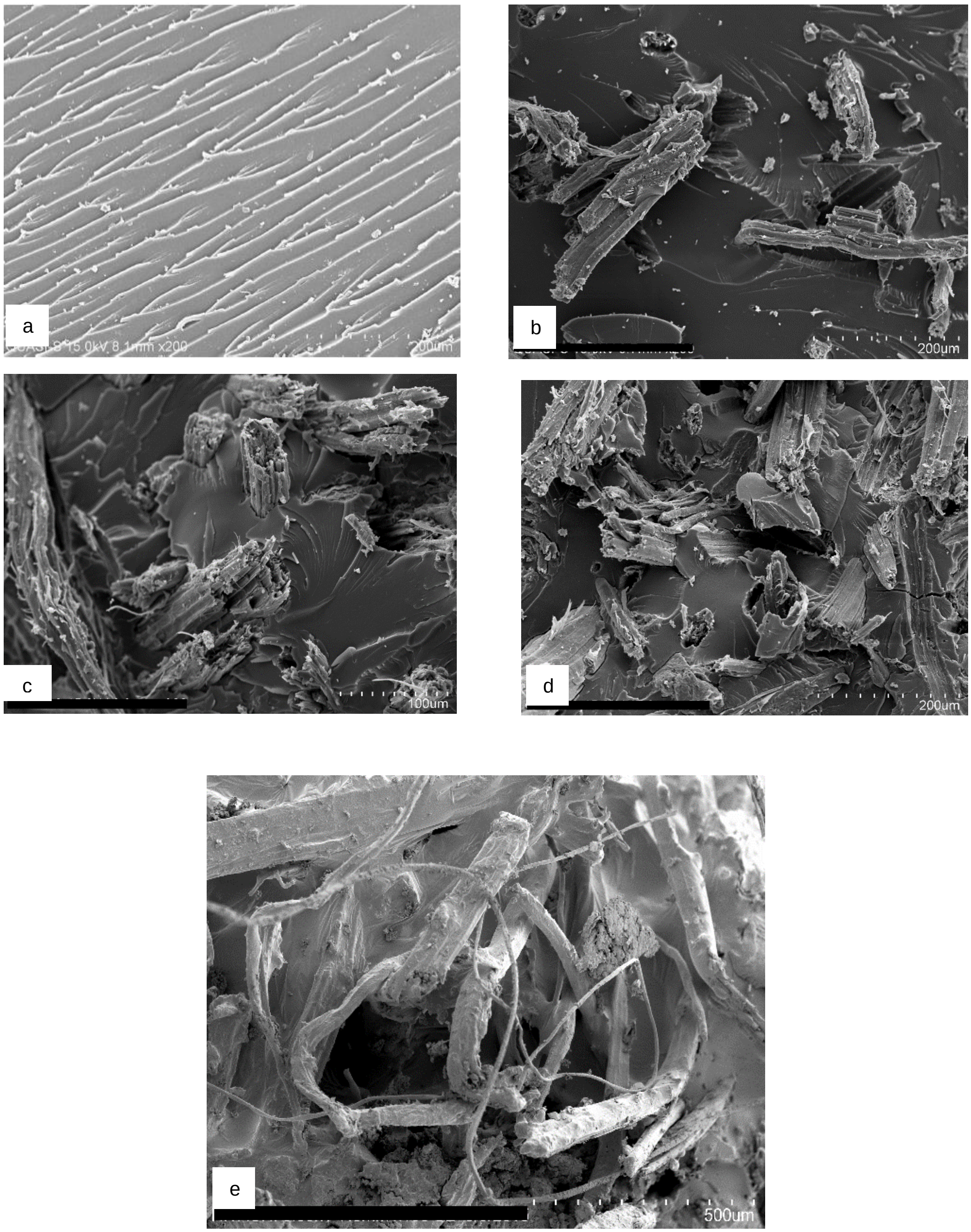

Fig. 7. Micrographs of fracture surfaces of roselle fibre reinforced VE

It is worth noting that char residue is a carbonaceous material that cannot be further dissociated into smaller volatile fragments. Char appears at the highest temperatures of the

TGA analysis. Char residue was obtained when roselle fibre reinforced VE cellulose decomposed at high temperatures. 10% of fibre loading of RFVE showed the lowest char residue followed by neat VE. On the other hand, increasing the roselle fibre content in the VE also increased the residue content. The degradation of neat VE and roselle fibre influenced the formation of char residue in the roselle fibre reinforced VE during the heating process.

The cell wall of the natural fibres experience pyrolysis at high temperatures and form char layers, which protect the fibre from thermal degradation (Nguong *et al.* 2013). As such, an increase in the roselle fibre content in the VE composites increased the formation of char residue in this study. The increase in residue weight is evidence for an effective mixture of higher filler content in the matrix material, which ultimately improves the thermal stability (Aprilia *et al.* 2014).

Morphology

Figure 7 is a micrograph of the fracture surface of the RFVE. The micrographs were taken from the impact test fracture samples to analyse the effect of roselle fibre, the adhesion of roselle fibre, and the matrix interphase in the composites. There was a clear difference between the samples of neat VE and the 10%, 20%, 30%, and 40% fibre loaded samples of RFVE. In addition, the fracture morphology of the neat VE looks smoother (brittle surface) when compared with the roselle fibre composites. The increase in roselle fibre loading caused the accumulation of roselle fibres in VE, thus increasing the strength of the composite samples up to 20%. From observation, Fig. 7(c) shows a better dispersion of fibres and demonstrates the homogeneity between the fibre and matrix of the composites (Aprilia *et al.* 2014). The uniform distribution of fibre in the matrix can improve the strength of the composites, which can be seen where the optimum tensile and impact properties were at 20% fibre loading. Additional fibres (beyond 20% fibre loading) added to the matrix decreased the mechanical properties. This phenomenon reflects the lower filler to matrix bonding in the composites. This can be seen in Fig. 7(e), where the 40% fibre loading samples experienced wetting problems and the tendency for fibres to agglomerate was high. As the amount of filler content increases in composites, stress concentration points may develop in the matrix. Both of these effects are results of agglomeration, which produces discontinuity in the matrix (El-Shekeil *et al.* 2014).

CONCLUSIONS

1. The roselle fibre vinyl ester composites (RFVE) showed improvements in tensile strength and modulus compared with neat VE. The optimum fibre loading was at 20vt% fibre content. However, the impact properties of roselle fibre composites decreased with increased fibre loading because of the ineffectiveness of stress transfer from the matrix to the fibre when an impact load is applied.
2. The RFVE composites showed an increase in thermal stability after the addition of roselle fibre. The degradation temperatures of RFVE composites decreased with an increase in the fibre loading.
3. Morphological study showed uniform distribution of fibre at 20vt% fibre loading, and a ductile fracture surface at 20% and 30% fibre loading. However, at higher fibre loads,

the fibres tended to agglomerate and accumulate on the fracture surface in properties of roselle fibre reinforced vinyl ester composites.

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