

Physical, Mechanical, and Thermal Properties of Wood Flour Reinforced Maleic Anhydride Grafted Unsaturated Polyester (UP) Biocomposites

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Physical, mechanical, and thermal properties of wood flour reinforced ungrafted and maleic anhydride grafted unsaturated polyester biocomposites were investigated. Composites were prepared using a Resin Transfer Moulding technique by varying wood flour loading (10, 15, 20, and 25 wt%) for both ungrafted and maleic anhydride grafted unsaturated polyester. Fourier transform infrared (FTIR) spectroscopic and scanning electron microscopic (SEM) analysis was utilized to study physical properties. Tensile and flexural tests were conducted for mechanical characterization, while thermal properties were evaluated using thermogravimetric analysis (TGA). FTIR and SEM results confirmed the presence of grafting onto the unsaturated polyester. The flexural strength and modulus of the composites were increased up to 20% filler loading, after which those values decreased, whereas, the tensile strength and Young's modulus values increased only up to 15% filler loading. Maleic anhydride grafted composites had better mechanical properties compared to ungrafted composites. According to TGA results, maleic anhydride grafted composites showed enhanced thermal stability at the final decomposition stage.

Keywords: Maleic Anhydride Grafting; Unsaturated Polyester; FTIR; SEM; TGA; Mechanical Properties

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INTRODUCTION

Recently agro-based renewable materials have received significant attention from the relevant industry and research communities due to their low cost, low weight, ecofriendliness, and worldwide environmental awareness (Thwe and Liao 2002; Rana *et al.* 2003; Yang *et al.* 2007; Zimmermann *et al.* 2014). Composites prepared from natural reinforcing fillers are inexpensive and could minimize environmental pollution, as they are biodegradable. They could play a vital role in solving environmental problems that we would otherwise face in the future. Although fully renewable materials are more eco-friendly, they do not possess desired properties for specific applications. Reinforcing synthetic polymers with natural fillers show strong potential as agro-based natural resources; they also present an added advantage of showing desired properties from a more sustainable material that would otherwise cause environmental pollution if thrown away (Rahman *et al.* 2009).

Compared to synthetic fillers, the lignocellulosic fillers such as jute, coir, bamboo, rice husk, abaca, wood flour, *etc.*, are lightweight, easily available, inexpensive, and easily available (Singleton *et al.* 2003; Yang *et al.* 2004; Brahmakumar *et al.* 2005; Hong *et al.* 2014). Malaysia has one of the largest rainforests in the world and produces tons of wood flour as a byproduct. Wood flour was used as reinforcing filler in the present study as it is renewable, lightweight, cheap, easily available, and biodegradable, and it does not leave residue that might harm the environment (Rahman *et al.* 2009). In most cases the addition of wood flour in polymer has resulted in narrower cell size distribution and lower average cell dimension. Wood particles are also believed to act as a nucleating agent that enhances heterogeneous nucleation (Rizvi *et al.* 2008).

Nowadays, unsaturated polyesters are the most widely used cross-linkable polymeric materials for composites, comprising in excess of 80% of all thermoset resins. Increase in the usage of this resin in the plastic market is mainly due to its possibility to be highly filled and easily processed (Penczek *et al.* 2005). Unsaturated polyester differs from saturated polyester in that the acids and glycols used as raw materials carry carbon-carbon double bonds, which provide reactive olefinic positions in the unsaturated polyester alkylid. This reactive unsaturation can later be used in a second step of cross-linking (Kandelbauer *et al.* 2014). The physical and mechanical properties of lignocellulosic filler reinforced polymer largely depend on the type of polymer, the content and properties of the reinforcing filler, and filler-polymer interaction. One of the ways of improving the compatibility between the two components is by polymer grafting (Kim *et al.* 2007; Hwang *et al.* 2012). Maleic anhydride is one of the most widely used reactive compatibilizers as it has good reactivity, low toxicity, and low potential to polymerize with itself under free-radical grafting conditions (Hwang *et al.* 2012). Grafting of polymers with maleic anhydride forms single and oligomeric grafts and forms a bridge between maleic anhydride and polymer chains (Gaylord and Mehta 2003).

The present study investigates the effect of maleic anhydride grafted on unsaturated polyester reinforced with wood flour. The physical, mechanical, and thermal properties of wood flour reinforced ungrafted and maleic anhydride grafted unsaturated polyester biocomposites are described. The effect of wood flour loading on ungrafted and maleic anhydride grafted unsaturated polyester is also reported.

MATERIALS AND METHODS

Materials

The Unsaturated Polyester (UP) resin used in this study was obtained from Reversol Malaysia with the trade name of “P9509”. For the hardening process, the matrix was mixed with Methyl Ethyl Ketone Peroxide (MEKP) catalyst. Maleic Anhydride (MA) was used as a compatibilizer as received and was a product of Aldrich with a molecular weight of 98.06 g/mol. All chemicals were of analytical grade obtained from local commercial sources. The wood flour used in this research was obtained from timber research institute Sarawak Malaysia. For this experiment, the mesh size of the wood flours was 40 μm . To reduce the moisture content of the wood flour further, it was left in a convection oven at 80 °C for 24 h prior to use.

Grafting of Unsaturated Polyester

The unsaturated polyester resin was grafted using 1 wt% maleic anhydride by mechanical mixing at 70 rpm at room temperature into 1000 mL of unsaturated polyester resin for 1 h until the maleic anhydride was completely dissolved into the matrix. The grafted unsaturated polyester resin was then bottled and stored under ambient temperature before experiment.

Preparation of Resin Transfer Moulded Thermoset Composites

Unsaturated polyester (UP) reinforced with wood flour (WF) composites (UPWFC) and Maleic anhydride (MA) grafted unsaturated polyester (UP) reinforced wood flour composites (MA-g-UPWFC) were fabricated using a Resin Transfer Moulding (RTM) technique. Randomly dispersed wood flour were placed in the first half of the mould (female), while the other half of the mould (male) was clamped over the top. The wood flour was pre-pressed into the mould before the resin injection. After completion of preform process, the mould was closed, clamped and unsaturated polyester resin mixed with 1 wt% of Methyl Ethyl Ketone Peroxide (MEKP) were injected at an optimum pressure of 1 kg/cm². Vacuum was applied simultaneously during the resin impregnation in order to drive the resin through the cavity and reduce the void formation. The hardening of composite was carried out at room temperature for 6 h. The composites were post-cured at 80 °C in a convection oven for 3 h.

Fourier Transform Infrared Analysis (FTIR)

FTIR spectra of untreated composite (UPWFC) and maleic anhydride treated composite (MA-g-UPWFC) were recorded on a Perkin Elmer FTIR RX1 (4000 to 500 cm⁻¹) spectrophotometer (Norwalk, CT) with a 4.0 cm⁻¹ resolution.

Scanning Electron Microscopy (SEM)

The morphology of the composites and interfacial adhesion between the filler and the UP matrix was examined using a scanning electron microscope (JSM-6701F) supplied by JEOL Company Limited, Japan. An acceleration voltage of 10 kV was used to record the SEM images of the composite specimens. The samples were viewed perpendicular to the fractured surfaces. The micrographs were taken at a magnification of 500 and 2000, respectively.

Mechanical Testing

Tensile and flexural tests of the composites were carried out. For each test and type of the composite, ten specimens were tested, and the average values were reported. Tensile tests were conducted according to ASTM D 638-01 (ASTM D 638-01, 2002) using a Universal Testing Machine (Model: MSC-5/500, Agawn Seiki Company Limited, Japan) at a crosshead speed of 5 mm/min. Static flexural tests were carried out according to ASTM 790M-93 (ASTM 790M-93, 1995) using the same Testing Machine mentioned above at the same crosshead speed.

Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) measurements were carried out on 5 to 10 mg of ungrafted and MA grafted composites samples at a heating rate of 10 °C/min in nitrogen atmosphere using TGA analyzer model name TA Instrument SDT Q600. Treated and untreated composites samples were subjected to TGA in high purity nitrogen

atmosphere under a constant flow rate of 5 mL/min. Thermal decomposition of each samples occurred in a programmed temperature range of 20 °C to 800 °C. The continuous weight loss and temperature were recorded and analyzed to determine the following TGA parameters: thermal degradation rate (% weight loss/min), initial degradation temperature, and residual weight (RW) at 800 °C. The activation energy was obtained through Broido method (Rana *et al.* 2003). The equation associated for the calculation of the activation energy (E_a) is,

$$\ln[\ln(1/Y)] = -E_a + \text{Constant} \quad (1)$$

$$\text{where } Y = (W_t - W_\infty)/(W_0 - W_\infty) \quad (2)$$

In these equations T is the temperature in Kelvin, R is the gas constant in $\text{Jmol}^{-1}\text{K}^{-1}$, Y is the fraction of the number of initial molecules not yet decomposed, W_t is the weight at any time t , W_∞ is the weight at infinite time, and W_0 is the initial weight. A plot of $\ln[\ln(1/Y)]$ vs. T gives an excellent approximation to a straight line. The slope is related to the activation energy.

RESULTS AND DISCUSSION

FTIR analysis

The FTIR spectra of maleic anhydride (MA), unsaturated polyester (UP), and maleic anhydride grafted unsaturated polyester (MA-g-UP) are shown in Figs. 1(a), (b), and (c), respectively.

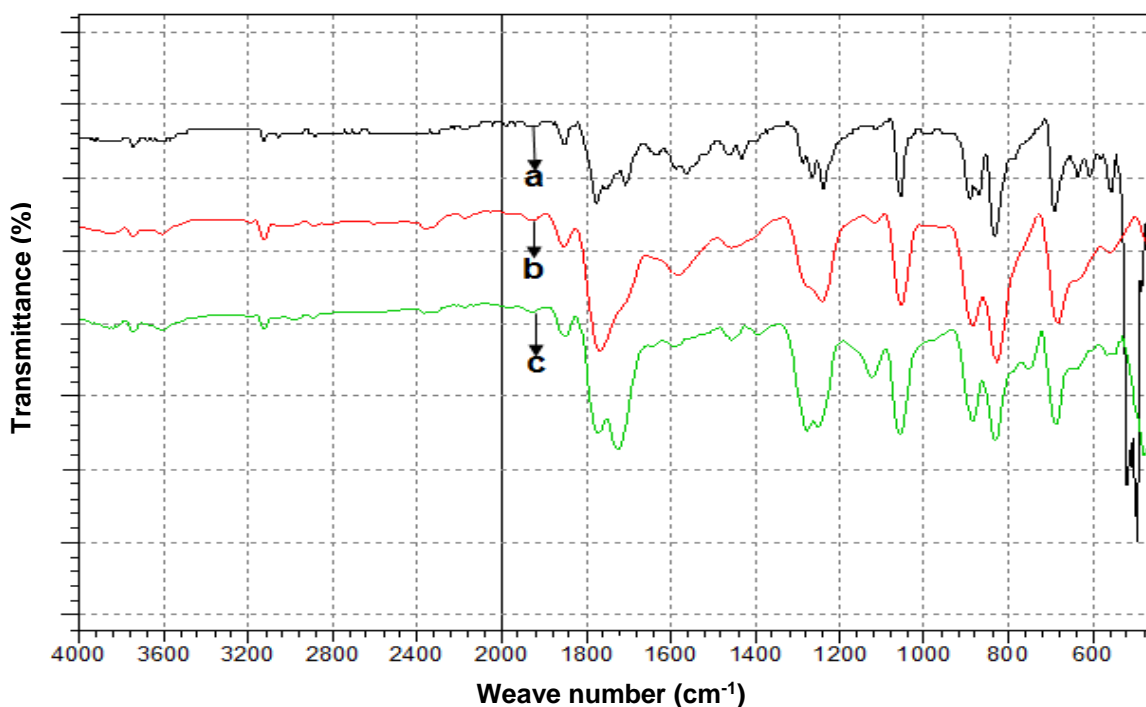


Fig. 1. FTIR spectra of (a) maleic anhydride, (b) unsaturated polyester (UP) and (c) maleic anhydride grafted unsaturated polyester (MA-g-UP)

The presence of grafting onto the unsaturated polyester was confirmed through FTIR analysis. Figure 1(a) shows the C=O stretching vibration at 1774 cm^{-1} , whereas the C=C double bond in UP shows stretching vibration at 1778 cm^{-1} (Figure 1(b)). Due to the grafting of MA with UP, the band intensity was shifted to 1713 cm^{-1} , which represents the carbonyl group attached with maleic anhydride. It is concluded from Fig. 1(c) that maleic anhydride was grafted onto the unsaturated polyester.

Scanning Electron Microscopy (SEM)

The scanning electron micrographs (SEM) for unsaturated polyester (UP) and maleic anhydride grafted unsaturated polyester (MA-g-UP) are shown in Figs 2(a) and 2(b), respectively. Dramatic changes were observed in MA-g-UP as compared to UP. A number of cavities and uneven surface was clearly in UP from Fig. 2(a). This is due to trapped, small air bubbles that led to the formation of cavities and uneven surface upon the completion of hardening process. The surface of MA-g-UP was smooth with no cavities (Fig. 2(b)). The smooth surface of MA-g-UP was due to the good penetration of maleic anhydride to bond with the monomer of unsaturated polyester. The grafted chains of maleic anhydride were formed on the polymer surface.

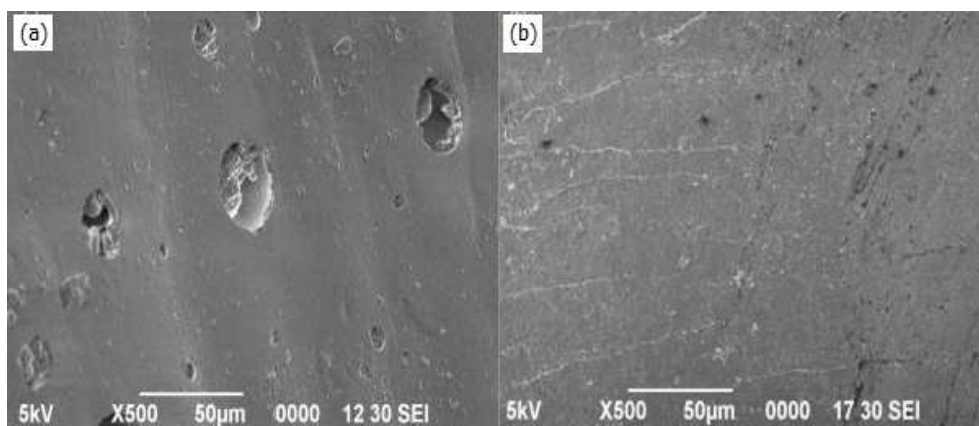


Fig. 2. SEM micrographs of (a) UPWFC and (b) MA-g-UPWFC

The fracture surfaces morphology of the UPWFC and MA-g-UPWFC composites are shown in Figs. 3(a) and 3(b), respectively.

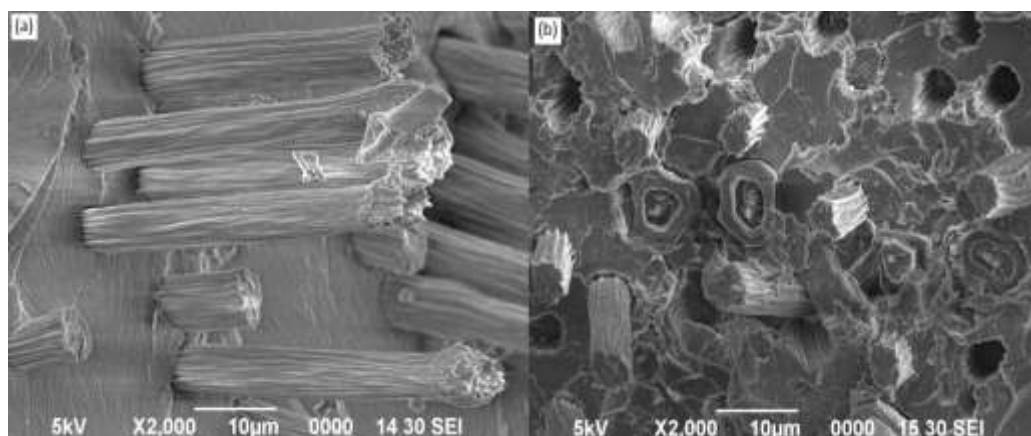


Fig. 3. SEM micrographs of flexural fracture surfaces of (a) UPWFC and (b) MA-g-UPWFC

It can be observed that the fillers were well dispersed in the unsaturated polyester matrix. A few voids were found on the fracture surface, as the filler was trapped by the unsaturated polyester matrix as a result of filler pull-outs. The wood flour filler formed a network inside the composite. This bridging effect prevented crack propagation and enabled effective stress transfer between the matrix and the filler. It can be noticed in Fig. 3(a) that there were many filler pull-outs, whereas Fig. 3(b) shows broken fillers in-line with the fracture surface. It reveals that the maleic anhydride treatment enhanced the interfacial adhesion between the unsaturated polyester matrix and wood flour.

Flexural Properties

The results of the flexural strength and flexural modulus for grafted and ungrafted unsaturated polyester reinforced with wood flour composites are shown in Figs. 4 and 5, respectively. The flexural strength and flexural modulus increased with increase in the wood flour loading up to 20% (Mir *et al.* 2013; Sidika *et al.* 2014; Ali *et al.* 2015). The increase in flexural strength and modulus of composites indicates that the wood flour restricted the mobility of the unsaturated polyester matrix when load was forced onto the composite. The movement of the polymer molecules was more difficult in the presence of the wood flour under stress. This is apparent also in the scanning electron micrographs presented earlier. A 25% wood flour loading for both UPWFC and MA-g-UPWFC decreased both flexural strength and modulus. This is mainly attributed to the poor compatibility between filler and unsaturated polyester, which formed weak interfacial regions. The weak interfacial regions result in the reduction efficiency of stress transfer from the matrix to the reinforcement component (Ayrilmis *et al.* 2013). Again the incorporation of the maleic anhydride in the unsaturated polyester significantly increased the flexural strength and flexural modulus as seen in Figs. 4 and 5, respectively.

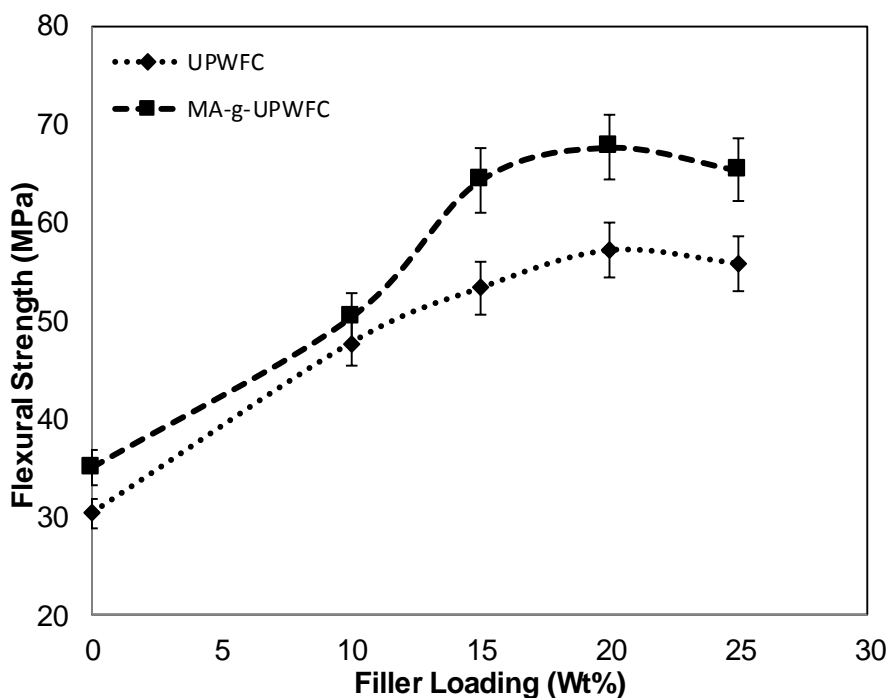


Fig. 4. Variation of flexural strength of wood filler reinforced unsaturated polyester biocomposite at different filler loading

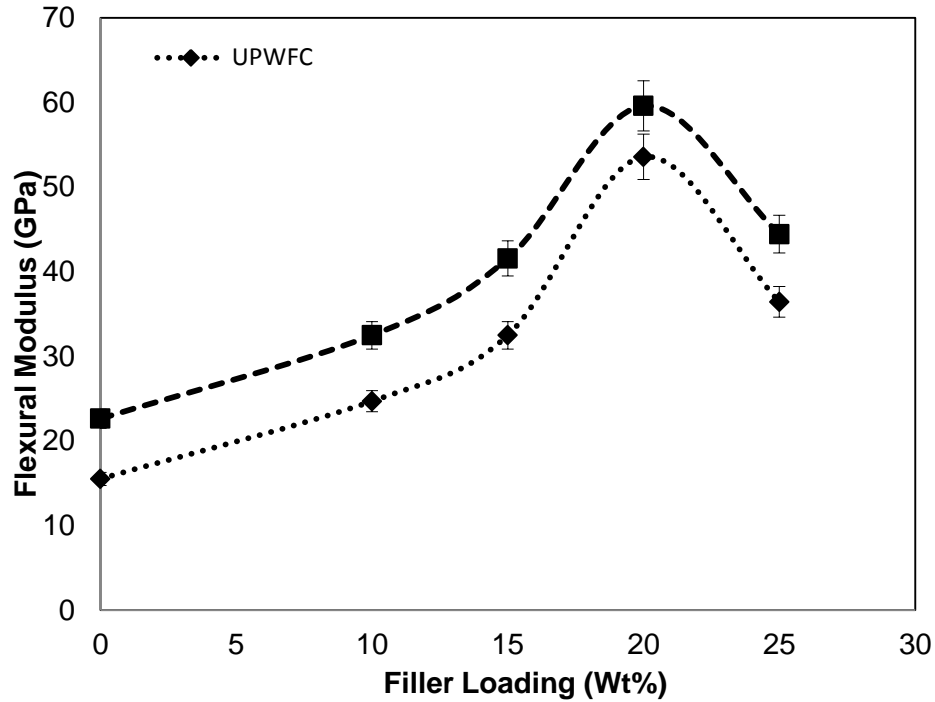


Fig. 5. Comparative flexural modulus of wood filler reinforced unsaturated polyester biocomposite at different filler loading

Tensile Properties

The tensile strength and Young's modulus of UPWFC and MA-g-UPWFC are shown in Figs. 6 and 7, respectively.

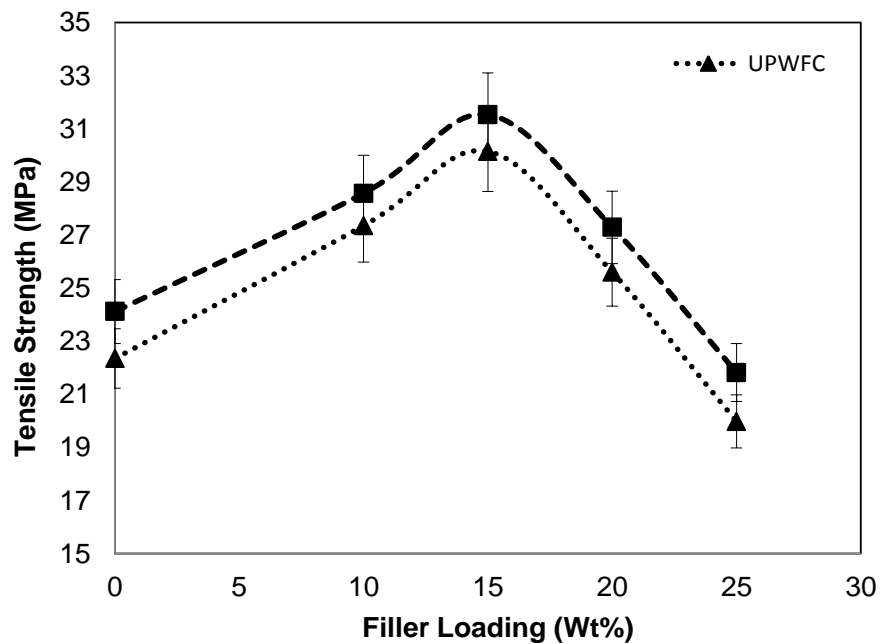


Fig. 6. Variation of tensile strength of wood filler reinforced unsaturated polyester biocomposite at different filler loading

The tensile strength and tensile modulus increased both UPWFC and MA-g-UPWFC with increase in filler loading up to 15%. However, the maleic anhydride grafted UP composites had higher tensile strength and Young's modulus compared to those of the ungrafted one. The result of the tensile properties revealed that the presence of the filler reduced the ductility of the unsaturated polyester composite and increased its modulus (Mohanty *et al.* 2005). The results also show that any substantial increment over filler loading of 15 wt% decreased the tensile strength and tensile modulus. The decrement of tensile results over the filler loading of 15 wt% is contributed mainly by the poor adhesion between fillers and matrix that are seen in the SEM micrograph presented in Fig. 2.

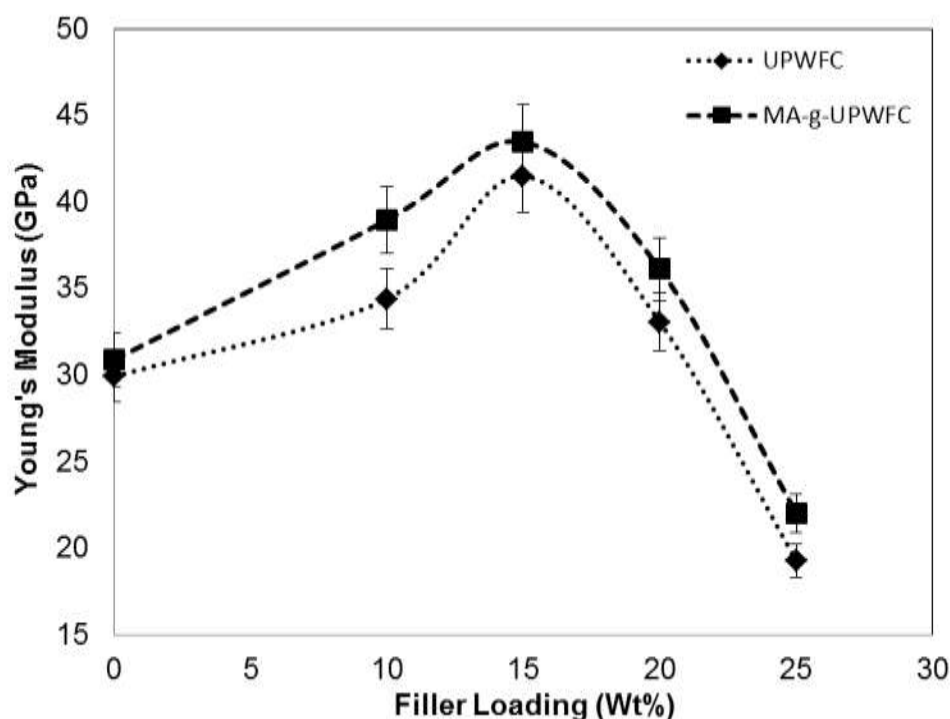


Fig. 7. Comparative Young's modulus of wood filler reinforced unsaturated polyester biocomposite at different filler loading

TGA Analysis

TGA was employed to evaluate the thermal stability and decomposition process of both of the composites. The corresponding TG curve and detailed results are presented in Fig. 8 and Table 1, respectively. There was no difference in the thermal stability in the useful range before 400 °C when the wood flour percentage was increased. Maleic anhydride grafted composites showed enhanced thermal stability at the final decomposition stage, which was below 400 °C. This can be observed through the results obtained from Broido method of calculating the decomposition temperature and activation energy (Table 1) (Hernandez *et al.* 2007). Untreated composite with 10 wt% of wood flour had an initial decomposition temperature of 350 °C, whereas the decomposition temperature of 25 wt% wood flowers composite was 368 °C. According to the finding, the higher filler loaded composites the data represented in Fig. 8 indicate a high mass load when the higher loads were used due to the interfacial bonding and adhesion between filler and matrix reflected in the SEM micrographs in Fig. 3.

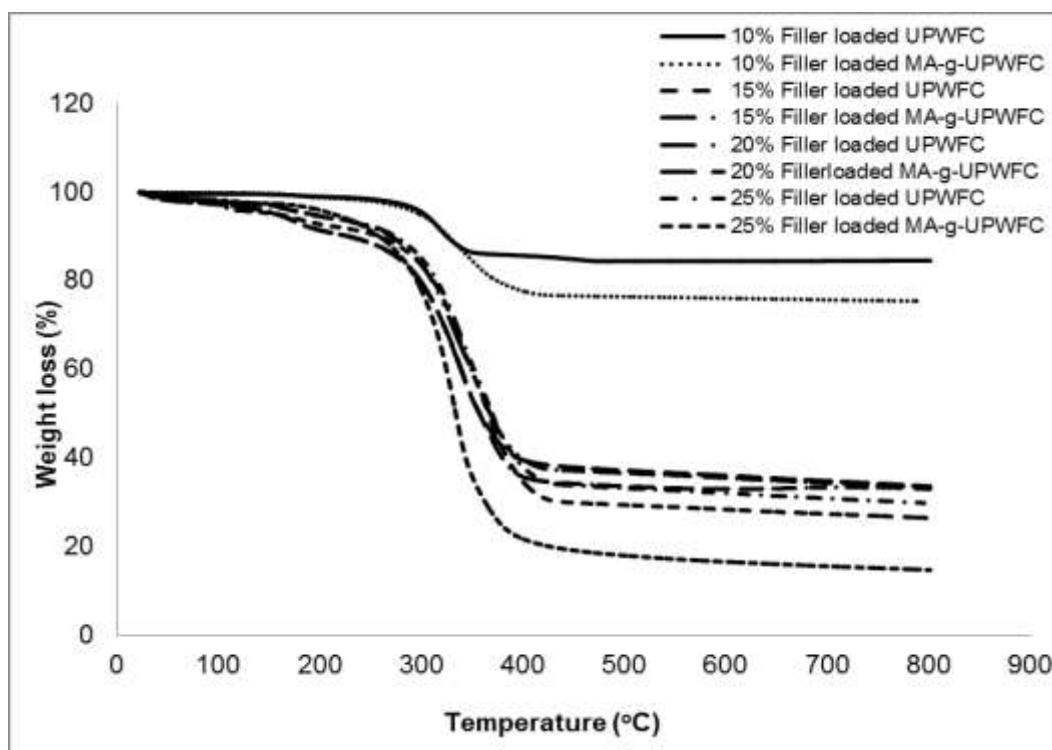


Fig. 8. TGA thermographs of UPWFC and MA-g-UPWFC

Table 1. Thermal Characteristics of UPWFC and MA-g-UPWFC

Wood flour Content (wt %)	Sample	T_i ($^{\circ}\text{C}$) ^a	T_m ($^{\circ}\text{C}$) ^b	T_f ($^{\circ}\text{C}$) ^c	W_{Ti} (%) ^d	W_{Tm} (%) ^e	W_{Tf} (%) ^f	Activation Energy, E_a ($\text{J}/^{\circ}\text{K}$)
10	UPWFC	350.16	396.62	445.92	78.94	39.49	13.49	18.36
	MA-g-UPWFC	352.65	399.90	447.56	83.59	41.78	16.02	32.53
15	UPWFC	355.33	398.62	450.21	70.50	43.44	12.04	22.84
	MA-g-UPWFC	356	399.88	451.56	77.57	46.63	20.30	38.62
20	UPWFC	361	400.53	454.11	65.67	34.39	13.99	23.23
	MA-g-UPWFC	363.92	401.06	455.92	68.25	42.71	23.16	46.67
25	UPWFC	368.77	400.77	458.67	57.94	34.87	13.51	26.66
	MA-g-UPWFC	370.48	401.90	459.26	59.65	41.58	20.47	47.87

^a Temperature corresponding to the beginning of mass loss

^b Temperature corresponding to the maximum rate of mass loss

^c Temperature corresponding to the end of mass loss

^d Mass remaining at temperature corresponding to the beginning of mass loss

^e Mass remaining at temperature corresponding to the maximum rate of mass loss

^f Mass remaining at temperature corresponding to the end of mass loss

For natural fillers, the decomposition profiles are generally characterized by three-step degradation (Cheng *et al.* 2009). The first step is attributed to the evaporation of moisture or low volatile impurities (*e.g.* trapped solvent residues) from 100 to 150 °C and 100 to 180 °C for untreated composites (UPWFC) and treated composites (MA-g-UPWFC), respectively. From the percentage of weight loss, it can be assumed that there were few chains with these weak bonds, particularly head-to-head bonds, and the unsaturated end groups in polymeric chain that were incorporated during the starting and finishing reactions (Hernandez *et al.* 2007). The second step corresponds to the degradation of wood flour, which started at 200 °C and 230 °C for untreated composites (UPWFC) and treated composites (MA-g-UPWFC), respectively. The final step, which corresponds to the degradation of the wood flour and the breaking of chemical composition to produce the radical or molecular products, started at approximately 350 °C for both composites, which is coherent with other research findings (Kaczmarek *et al.* 2008; Cheng *et al.* 2009). The incorporation of maleic anhydride into unsaturated polyester led to improved thermal behavior with higher activation energy and higher decomposition temperature as compared to the untreated composites. Analysis of the activation energy can assist in reaching the conclusion regarding the thermal stability of the composites (Rahman *et al.* 2011). Larger activation energy implies greater stability.

CONCLUSIONS

1. Maleic anhydride grafted and ungrafted unsaturated polyester composites reinforced with wood flour were prepared using a Resin Transfer Moulding technique. Physical, mechanical, and thermal properties of those biocomposites were later characterized.
2. FTIR confirmed the presence of grafting onto the unsaturated polyester.
3. SEM micrographs showed good penetration and bonding of maleic anhydride with the monomer of unsaturated polyester.
4. The mechanical properties increased with filler loading, while the maleic anhydride grafted biocomposites had better mechanical properties compared to the ungrafted ones.
5. TGA indicated that maleic anhydride grafted composites showed enhanced thermal stability at the final decomposition stage that is above 400 °C.

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