Thermal, Static, and Dynamic Mechanical Properties of Bark Cloth (*Ficus brachypoda*) Laminar Epoxy Composites

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Bark cloth is a natural nonwoven fibrous fabric produced in Uganda from three species of trees: *Ficus natalensis*, *Ficus brachypoda,* and *Antiaris toxicaria*." The recently developed bark cloth composites has created a new class of textile composites which can find applications in interior automotive panels. Four bark cloth plies were utilized, the resin was infused using Vacuum Assisted Resin Transfer Molding (VARTM). Scanning Electron Microscopy was used to study the fabric's fiber morphology, surface functional groups were characterized using Fourier Transform Infrared. Overall, the static and the Dynamical mechanical analysis showed that the developed composites had a tensile strength ranging from 22 to 29 MPa whereas the flexural strength ranging from 45 to 100 MPa. The glass transition temperature of the composites was ranging from 53°C to 63°C. POLYM. COMPOS., 00:000–000, 2015. © 2015 Society of Plastics Engineers

INTRODUCTION

!!The global climate change that is partly due to increase in greenhouse gas emissions, has led to the industry and researchers' interest in alternative and sustainable materials that are candidates for cleaner production mechanisms and sustainable development. Wood is an important resource that's readily available, biodegradable, and source of a host of byproducts that are environmentally friendly. Natural fibers after processing can act as reinforcing agents for production of natural fiber reinforced composites.

Petroleum is by far the world's highest source of energy, especially for automobiles. With the fluctuating oil prices coupled with instability from some of the biggest oil producing nations, depending on products whose feedstock is petroleum is to be reduced. Synthetic fibers' performance in the clothing, automotive and aerospace industry is unparalleled; however, there is waste disposal concerns, dangerous toxic fumes, and high energy demands arising from their production. Unlike synthetic fibers, natural fibers are biodegradable [1], exhibit low specific weight [2], sustainable [3, 4] and some have closely comparable mechanical properties to those of synthetics [5]. Natural fibers are superior in terms of environmental friendliness based on indicators such as climate change, acidification and non-renewable energy. However, since most commercial natural fibers undergo retting processes and utilization of fertilizers, optimization of the two has a high impact on the eutrophication indicator and energy consumption.

Research on the commonest natural fibers such as kenaf, flax, jute, sisal, abaca has been done; their respective composites have found applications in automotive components that don't need high mechanical strength [6 − 11].

Bark cloth is a natural non-woven fibrous fabric produced in Uganda from three species of trees: *Ficus natalensis*, *Ficus brachypoda,* and *Antiaris toxicaria*. Rwawiire et al. [12] studied the morphology, thermal, and mechanical characterization of *Ficus natalensis* bark cloth. The fabric was found to be stable below 200°C, treatment with alkaline increased the thermal stability, and it was found to have a similar infrared absorbance spectrum to that of cellulose. The thermo-physiological and comfort properties showed that the fabric has robust comfort properties a reason for its surge in art, fashion, and design articles [13]. The advantages of bark cloth fabric in comparison to other natural fibers are: reasonable thermal stability, no need of fertilizers for growing trees, low energy demands in production, good thermal insulation properties, and environmental friendliness because the tree isn't cut [14].

In this study, an exploratory investigation of the microstructure, thermal, static, and dynamic mechanical properties of bark cloth reinforced epoxy composites is presented.

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FIG. 1. Bark cloth non-woven laminar fabrics. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

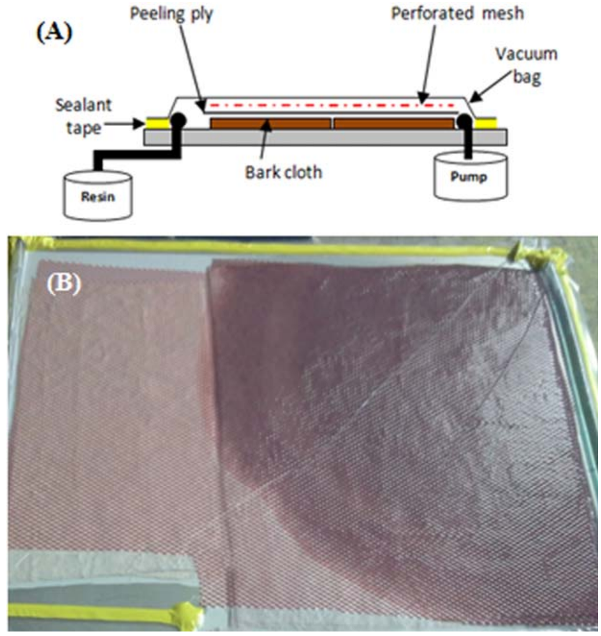

FIG. 2. (A) Scheme of Vacuum Assisted Resin Transfer Molding (VARTM). (B) Resin infusion with VARTM. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Research has been done utilizing thermoplastic polymers for natural fiber composites; however, there is limited research on utilization of thermoset polymer resins for natural fiber reinforced composites [15]. Although bark cloth has been around dating back as far as the 13th century, there is limited scientific study of bark cloth.

The composites were prepared using Vacuum Assisted Resin Transfer Molding (VARTM) a molding process that infuses resin with the assistance of vacuum pressure into a dry fiber preform [16]. The tensile and flexural properties were investigated using universal testing machine; thermal behavior was investigated using Differential Scanning Calorimetry (DSC). The Dynamical mechanical analysis was used to study the response of the composites under temperature, whereas the surface functional groups were investigated using Fourier Transform Infrared spectroscopy.

EXPERIMENTAL

Materials

Bark cloth (Fig. 1) was extracted from the *Ficus brachypoda tree* using hand extraction method described by Rwawiire et al. [12]. Epoxy resin LG285 and hardener HG 285 supplied by GRM systems, s.r.o was used in composite sample specimen fabrication.

Composite Fabrication

The resin to hardener ratio used was 100 : 40 as per the manufacturer's specifications; Vacuum Assisted Resin Transfer Molding (VARTM) was used to prepare the composites (Fig. 2). Four untreated bark cloth plies were used for the composite sample preparation for each set of composites. The Bark cloth Reinforced Epoxy Composite (BREC) stacking sequence is shown in Table 1. The composite was left to cure at room temperature for 72 h. Rwawiire et al. [12] showed that the microfibers of bark cloth are aligned at an angle of 45°; therefore it was taken as the reference angle so as to come up with the above stacking sequence.

Characterization Methods

Morphology. The fabric and fracture surface morphologies were investigated using a Vegas-Tescan scanning electron microscope with accelerating voltage of 20 KV.

Fourier Transform Infrared Spectroscopy (FTIR). Nicolet iN10 MX Scanning FTIR Microscope was used to provide the spectrum of the sample. The infrared absorbance spectrum of each sample was obtained in the range of $4000-700$ cm^{-1}.

Thermal Behavior. The Mettler Toledo TGA/SDTA851^{e} instrument was used to study the thermal gravimetric behavior of the fabric whereas the Perkin Elmer Differential Scanning Calorimeter DSC6 was used for Differential

TABLE 1. Bark cloth reinforced epoxy composites (BREC) stacking sequence.

Composites	Ply arrangement (°)
BREC I	−45, 45, 0, 90
BREC II	45, −45, 90, 0
BREC III	0, 90, 45, −45
BREC IV	90, 0, −45, 45

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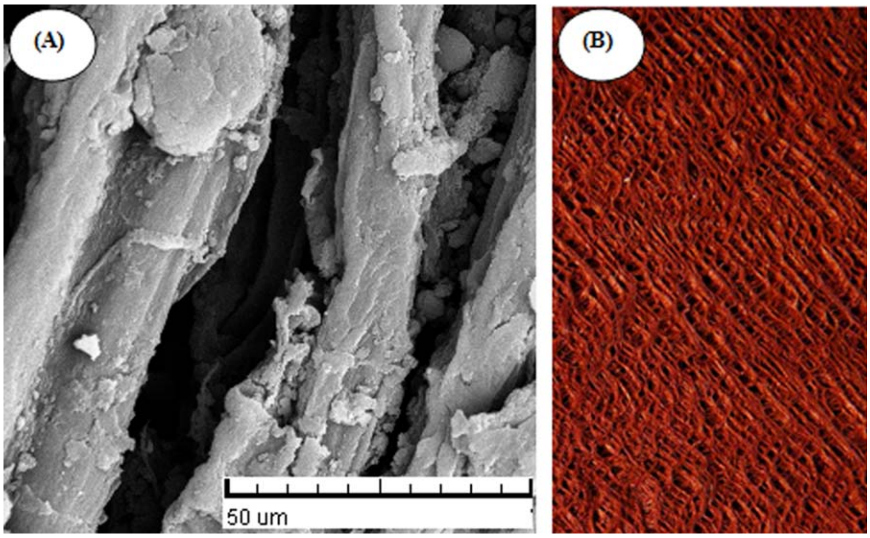

FIG. 3. Morphology of composites (A) SEM microstructure; (B) micro-fiber angle arrangement. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

Scanning Calorimetry (DSC) of composites under air atmosphere.

DSC samples weighing ~10 mg were placed in aluminum pans and sealed. The specimens were heated from 25°C to 400°C at a heating rate of 10°C/min.

Mechanical Properties. Tensile properties of fabric-reinforced composites samples were characterized in accordance with ASTM D3039. Tensile tests were carried out using Testometric (M500-25kN), a universal mechanical testing machine operating at crosshead speed of 4 mm/min. Four specimens were tested to obtain average tensile properties of the composite.

Flexural test was conducted as per ASTM D 790 using a Tiratest 2300 universal testing apparatus. The samples were tested using three point bending with a recommended speed of testing of 2 mm/min. The span length to thickness ratio was 32 : 1.

Dynamic Mechanical Properties (DMA). The DMA was carried out on a DMA 40XT machine. The samples with dimensions 56 × 13 × 2.5 mm^3 were tested using three point bending mode at frequency of 1 Hz from 30°C to 150°C at a heating rate of 3°C/min.

Water Immersion Ageing. Water absorption tests were conducted by immersion of composite specimens in distilled water for 14 days (336 h) to the point of saturation. The water was changed after every 2 days so as to prevent chemical reaction of the degraded components with the composite. Moisture absorption content was calculated by:

$$Humidity\,[\%]=\frac{m_w-m_d}{m_d}x100 \qquad (1)$$

Where m_w is the mass of immersed sample; m_d is the mass of dry sample

RESULTS AND DISCUSSION

Fiber Morphology

The bark cloth fabric is composed of microfibers arranged in a fairly ordered manner bonded with waxes and other impurities (Fig. 3A). The various processing avenues, including grooved hammers are responsible for the micro fiber orientations of the fabric (Fig. 3B).

Surface Functional Groups of Composites

The FTIR spectra of *Ficus brachypoda* bark cloth reinforced epoxy composites is shown in Fig. 4. FTIR analysis helps to analyze the bark cloth fabric's free hydroxyl groups available after cross linking with the epoxy polymer [17]. The peak at 3,353 cm^{-1} is attributed to cellulose-OH. 2,917 cm^{-1} is due to Symmetric and asymmetric stretching vibrations of CH_2 and CH_3 groups of cellulose and hemicellulose. Benzene ring stretching of Lignin is at 1,602 cm^{-1}

Thermal Behavior of Composites

The thermogravimetric (TGA) behavior of bark cloth is shown in Fig. 5 whereas Fig. 6 shows the Differential Scanning Calorimetry (DSC) thermogram.

Monteiro et al. [18] showed that natural fibers exhibit three stages of thermal stability behavior. The first stage from 30 to 100°C is attributed to the removal of water accounting for about 5% loss in weight. The second stage accounting to about 60% weight loss starts from about 220 − 370°C with a maximum decomposition temperature corresponding to around 325°C. The temperature range 215 − 305°C corresponds to the cleavage of glycosidic linkages of cellulose, which leads to formation of H_2O,

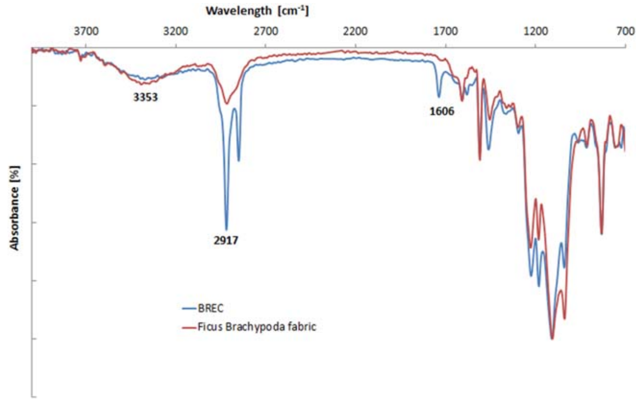

FIG. 4. Surface functional groups of composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

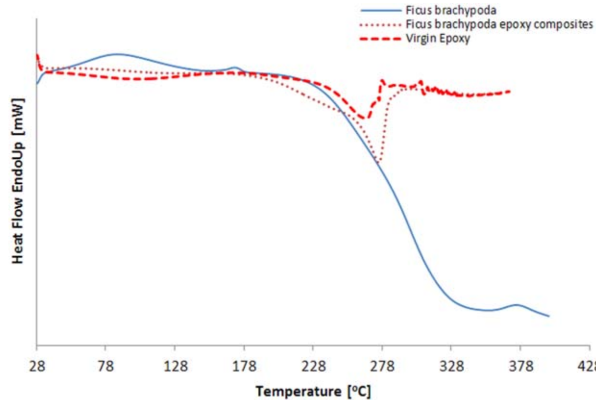

FIG. 6. DSC Behavior of composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

CO_2, alkanes, and other hydrocarbon derivatives. The last stage of decomposition starting from around 370°C corresponds to 35% loss in weight is due to char or other decomposition reactions.

The first peak at 85°C of DSC thermogram of fabric (Fig. 6) corresponds to water removal whereas a small peak at 172°C with heat 43.5 J/g is due to hemicellulose decomposition. Onset at 232°C is due to decomposition of cellulose, which is in agreement with the weight loss as can be observed from the TGA thermogram in Fig. 5. The last peak at temperature 376°C is attributed to the decomposition of lignin. The bark cloth reinforced epoxy composites onset of degradation is observed at 200°C. The DSC thermogram shows virgin epoxy stable until around 220°C. The slightly lower onset of degradation is attributed to the decomposition of the cellulose in the fabric microstructure.

Mechanical Properties of Composites

Tensile. Table 2 shows the tensile properties of the composites. It's observed that stacking sequence BREC III had the highest tensile strength and modulus. It's observed that the composites have almost the same per-

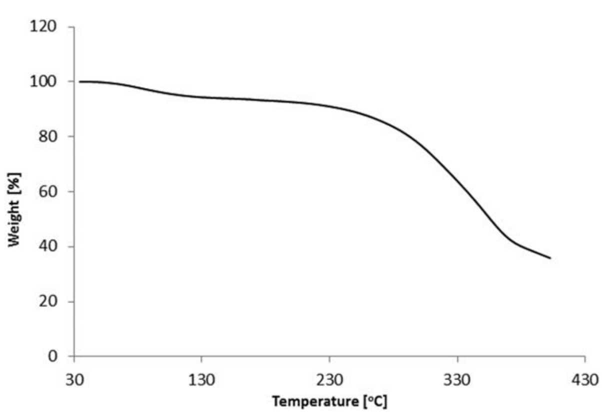

FIG. 5. TGA behavior of barkcloth fabric.

centage of elongation except BREC IV composites that had the least percentage of elongation. Because bark cloth fabric has microfibers that are aligned at an angle, it's therefore important to have a stacking sequence that will be beneficial for composite applications.

Flexure. The flexural properties (Table 3) show that the composites have considerable flexural rigidity. BREC I composites exhibited high flexural strength, followed in order by BREC II, BREC IV, and lastly BREC III. A high modulus of elasticity of BREC IV composites is confirmed by the low percentage of elongation due to their stiffness.

Dynamic Mechanical Properties

Figure 7 shows the elastic behavior ($E^{'}$) known as storage modulus of BREC under dynamic mechanical conditions. The Storage modulus indicates the viscoelastic rigidity of the composites and is proportional to the energy stored after every deformation cycle. The storage modulus falls sharply from around $50-70$°C which is the glass transition temperature range. The composites from the highest storage modulus to the lowest were those with stacking Sequence IV, II, I, III that is confirmed by the flexural modulus obtained from the flexural tests in Table 3 above.

The loss modulus (E') indicates the energy loss of the composites. Figures 8 and 9 show the loss modulus behavior and the mechanical damping factor of the

TABLE 2. Tensile properties.

Composites	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)	Coefficient of variation (%)
BREC I	25	3.9	1.00	17.3
BREC II	22	2.6	1.00	10.5
BREC III	29	4.8	1.04	23.9
BREC IV	25	3.5	0.56	10.2

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TABLE 3. Flexural properties.

Composites	Flexural strength (MPa)	Flexural modulus (GPa)	Coefficient of variation (%)
BREC I	100	2.8	28.6
BREC II	77	3.8	12.2
BREC III	45	1.8	33.6
BREC IV	70	4.1	3.8

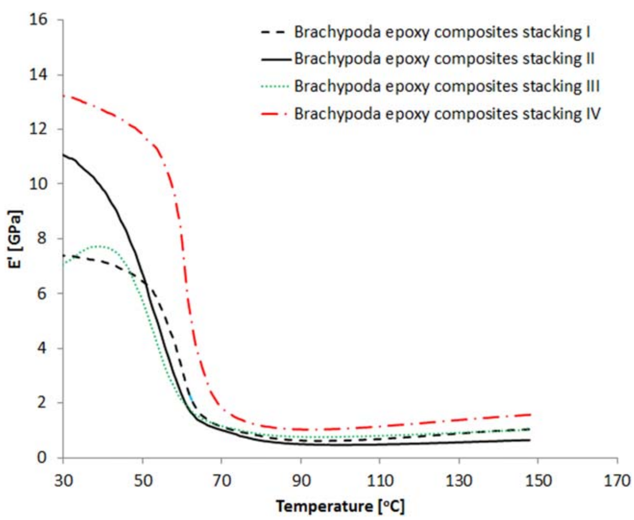

FIG. 7. Storage modulus behavior against temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

composites. It's observed that the glass transition temperature obtained using the loss modulus curve is lower than that obtained from the mechanical damping factor (Table 4).

The composites glass transition temperature is between 53 and 62°C.

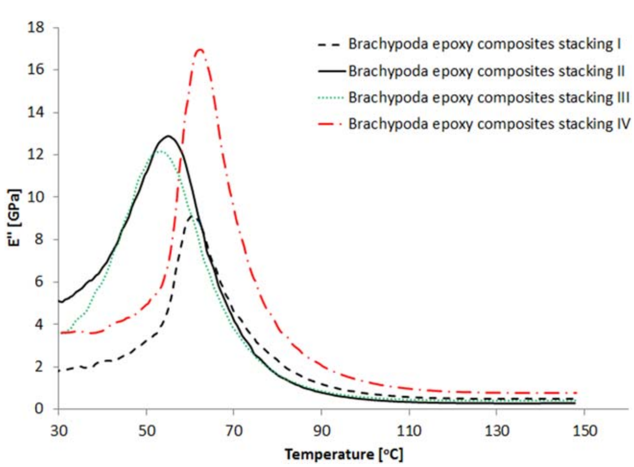

FIG. 8. Loss modulus behavior against temperature. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

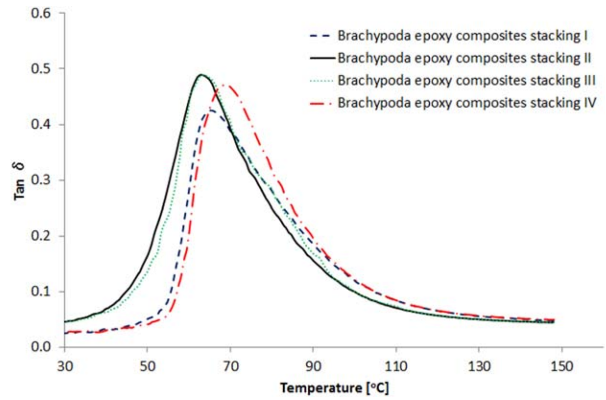

FIG. 9. Mechanical damping factor of composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE 4. Dynamical mechanical properties of composites.

Composites	Storage modulus at 30°C (GPa)	Glass transition temperature (°C)	Tan δ glass transistion (°C)	Tan δ
BREC I	7.4	60.5	64.6	0.42
BREC II	11.1	55.0	63.5	0.49
BREC III	7.0	53.3	63.5	0.49
BREC IV	13.3	62.5	69.0	0.47

Water Immersion Tests

The water absorption as a function of time is shown in Fig. 10. Water uptake in natural fiber reinforced composites is dependent on the disintegration of the matrix-fiber interface through matrix cracking and debonding leading to water penetrating through the pores. The developed composites rapidly absorbed water in the initial stage and finally reach saturation. Bark cloth being majorly composed of cellulose, the presence of OH groups led to more water absorbance of the fabrics.

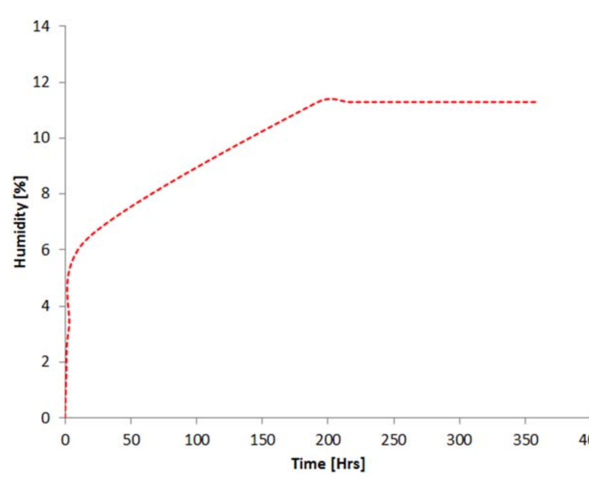

FIG. 10. Water absorption behavior of composites. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

The rate of water uptake is typical of Fickian diffusion, which explains that rapid water absorption takes place at the beginning of exposure of matter to water [19, 20]; thereafter an equilibrium point is reached. Since the composites were produced using VARTM, water uptake was through the porosities in the fiber−matrix interface. Bark cloth being plant fiber, the presence of free cellulose hydroxyl groups leads to water uptake.

CONCLUSIONS

For the first time, the mechanical properties of bark cloth reinforced epoxy composites has been presented. The static mechanical properties show that the composites had the highest tensile strength of 29 MPa and modulus of ∼5 GPa. The BREC III stacking sequence is ideal for a higher tensile strength, whereas BREC IV is preferred for flexural rigidity because of the higher modulus which is supported by the dynamic mechanical properties.

The composites exhibited glass transition temperature 53 − 63°C. According to Sapuan and Abdalla [21] during their investigation on material selection of natural fiber reinforced dashboard composites for automobiles, a tensile strength of at least 25 MPa is required. In comparison to the engineered bark cloth composites in this investigation, it's evident that bark cloth is a promising material for automotive car dashboard, door claddings, and instrument panels.

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