



# **Mechanical and Thermo-Acoustic Characterization of Barkcloth and Its Polymer Reinforced Composites**

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SUMMARY OF THE THESIS

**Title of the thesis:** MECHANICAL AND THERMO-ACOUSTIC  
CHARACTERIZATION OF BARKCLOTH  
AND ITS POLYMER REINFORCED  
COMPOSITES

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## Abstract

Natural fiber reinforced composites have attracted interest due to their numerous advantages such as biodegradability and comparable mechanical strength. The desire to mitigate climate change due to greenhouse gas emissions has led to the utilization and development of sustainable and environmentally friendly raw materials. Plant fibers and biodegradable resins are explored as the alternative material selection for composite materials apart from their synthetic counterparts which are non-renewable. In this thesis, barkcloth, a naturally occurring non-woven fabric and its reinforced epoxy polymer composites are characterized for possible automotive applications. Since there have been no tangible scientific studies that have been made elsewhere on barkcloth except those published by the author, the thesis gives an in-depth analysis of the mechanical and thermo-acoustic properties of barkcloth and its polymer composites.

The fabric microstructure and morphology were investigated using Scanning Electron Microscopy (SEM) whereas the Chemical constituent analysis and Surface functional group characterization was done using FTIR. To further understand the behavior of the fabric after surface modification, X-Ray Diffraction (XRD) characterization was done on alkaline treated fabrics. Surface modification of the fabric was done using the enzyme, plasma, and alkali treatment. The design of the composites utilized fabrics which were surface modified and used for reinforcement of synthetic and green epoxy polymer resins.

In order to produce composites with required thickness as per the tensile testing standards (ASTM D3033), four barkcloth fabric layers were sufficient for the fabrication of composites. A hierarchal fabric architecture based on the micro-fiber angles was utilized in order to find out the best fabric layer design. Vacuum Assisted Resin Transfer Moulding (VARTM) and hand layup were utilized in the production of the synthetic epoxy and green epoxy composites respectively. The composites produced as the effect of the hierarchal architecture were evaluated and the best set of composite layup was utilized to design green epoxy polymer composites utilizing green epoxy resin. The ply stacking sequence  $90^\circ$ ,  $0^\circ$ ,  $-45^\circ$ ,  $45^\circ$  had the best mechanical properties and, therefore, was the stacking sequence investigated for the production of biocomposites. Static and thermal mechanical analyses were done on the set of composites.

In this investigation, for the first time, the thermo-acoustic properties of barkcloth and its polymer reinforced composites were investigated in order to study the potential of barkcloth as a sound absorption material. Theoretical empirical sound absorption models were employed to predict the behavior of the fabrics and the results were compared with commercially available products in automotive applications.

**Keywords:** Barkcloth, Composites, Thermo-acoustics, Sound Absorption, Modeling

## Anotace

Kompozity vyztužené přírodními vlákny dnes přitahují pozornost díky mnoha svým výhodám – jsou biologicky odbouratelné a mechanicky odolné. Potřeba zmírnit klimatické změny způsobené emisemi skleníkových plynů vede k vývoji udržitelných a environmentálně šetrných surovin. Rostlinná vlákna a biologicky odbouratelné pryskyřice jsou zkoumány jako alternativa pro kompozitní materiály, které jsou na rozdíl od svých syntetických protějšků recyklovatelné.

V této práci je zkoumána netkaná textilie získaná z kůry (barkcloth) stromů z čeledi morušovníkovitých rostoucí přirozenou cestou a tedy bez potřeby tovární výroby, která je použita jako výztuž epoxidových polymerů. Tato textilie se jeví jako vhodná výztuž pro aplikace v automobilovém průmyslu. Protože dosud nebyly publikovány studie, které by se tímto typem materiálu zabývaly, kromě těch, které publikoval autor, je součástí této práce podrobná analýza mechanických a tepelně-akustických vlastností studovaného materiálu a z něj vyrobených polymerových kompozitů.

Mikrostruktura a morfologie materiálu byly zkoumány s využitím rastrovacího elektronového mikroskopu (SEM), analýza chemického složení a charakteru funkčních skupin na povrchu proběhla s využitím FTIR. K hlubšímu porozumění chování materiálu po modifikaci povrchu byla využita rentgenová difrakce (XDR) na alkalicky ošetřeném povrchu. Modifikace povrchu byly provedeny enzymaticky, plazmaticky a vybranými alkalickými činidly. V navržených kompozitech byl použit povrchově upravený materiál, kterým byly vyztuženy jak syntetické tak biodegradabilní pryskyřice.

Testovací vzorky kompozitů byly vyrobeny tak, aby jejich tloušťka odpovídala standardům používaným při zkoušce tahem (ASTM D3033). Pro jejich vyztužení byly použity čtyři vrstvy netkané textilie z kůry. Hierarchická architektura materiálu založená na rozložení mikrovláken byla využita při návrhu orientace jednotlivých vrstev. Při výrobě kompozitů ze syntetické i bio- pryskyřice byly použity technologie VARTM a technologie ručního kladení. Nejprve byl analyzován vliv rozložení jednotlivých vrstev na vlastnosti kompozitu ze syntetické pryskyřice a následně bylo vybráno nejvhodnější rozložení vyztužujících vrstev. Takto rozložené vrstvy byly využity pro přípravu kompozitu s biologicky odbouratelnou pryskyřicí. Jako nejlepší se ukázalo následující kladení vrstev: 90°, 0°, -45°, 45°, které vykazovalo nejlepší mechanické vlastnosti, a proto byly pro další výzkum připravovány biokompozity s takto rozloženou výztuží. Statické a tepelně-mechanické analýzy byly dále provedeny pro tento soubor kompozitů.

V provedeném výzkumu byly poprvé prozkoumány také tepelně-akustické vlastnosti textilie z kůry a z nich vyrobených kompozitů. Cílem studie bylo prozkoumat potenciál textilie z kůry jako zvukově izolačního materiálu, zejména pro interiéry automobilů. Teoreticko-empirické modely zvukové izolace byly užity při predikci chování materiálu a výsledky byly srovnány s komerčně dostupnými produkty v automobilovém průmyslu.

**Klíčová slova:** barkcloth, kompozity, termo-akustika, zvuková izolace, modelování.

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# 1. Introduction

## 1.1. Background

Worldwide, researchers are embroiled in a race for niche products whereby industries can boost production processes as well as putting into consideration sustainability. The quest for structural materials which are environmentally friendly, to mitigate global warming effects is on the agenda of industrialized nations and recommendations are put forward for production of recyclable, biodegradable products or materials with zero emissions. Transition to a more sustainable biobased economy, as a political consequence of the Kyoto protocol on global climate change, includes a shift from petrochemical to renewable sources [1].

The ecological “green” image of cellulosic fibers is the leading argument for innovation and development of products which are biodegradable and can be applied to the automotive industries [2], building and construction [3], geotextiles and agricultural products [4,5]. Plant based fibers like flax, hemp; nettle and kenaf which were previously used for fiber in the western world have attracted renewed interest in textile and industrial composite applications. Natural fibrous materials were used by early civilizations from biblical times; Egyptians reinforced mud bricks with straw. However, the turn of the 20<sup>th</sup> century, new fibers, largely from petrochemical sources and with exceptional strength impeded the use and production of natural fibers.

Ever since the discovery of synthetic fibers, their contribution towards fuel economy in automotive and aerospace industries is unparalleled. The unique structural properties of composites have seen their application in construction beside the traditional automotive and aerospace sectors. Table 1 shows synthetic fibers whose feedstock are fossil fuels with negative effects such as environmental degradation due to the toxicity of the fumes emitted, demanding energy for production and non-biodegradability; whereas natural fibers have advantages such as biodegradability, low cost, non-toxicity, acoustical properties etc. [6].

### 1.1.1. Drivers for Change

The Intergovernmental Panel on Climate Change (IPCC) most recent report recommends cutting of greenhouse gas emissions by 70% and an increase of the use of clean green energy by 2050 respectively. Effective strategies such as utilization of sustainable biodegradable materials instead of synthetic materials can contribute to lowering greenhouse gas emissions thus combating climate change [7].

In reference to European Union (EU) guideline 2000/53/EG [8] issued by the European Commission, 95% of the weight of a vehicle have to be recyclable by 2015. This has led to a surge of utilization of natural fiber reinforced composites among the European automotive industry by designing and production renewable biodegradable composites based on natural fibers [9–17]. Furthermore, the EU Landfill Directive 1999/31/EC whose main goal is to reduce on the quantity of biodegradable municipal waste that ends up in landfill has sparked research in sustainable and biodegradable materials that can be reused [18].

The increasing costs of oil vis-à-vis the diminishing oil reserves world over and environmental concerns has led to the reigniting of the natural fiber spark. Production of chemicals and materials from bio-based feedstock is expected to increase from today’s 5% level to approximately 12% in 2010, 18% in 2020 and 25% in 2030 [19].

The need for a lightness of materials with superb performance characteristics has sparked interest in lightweight composite materials especially in automotive applications (Table 2). With the dwindling petroleum resources, coupled with high prices, fiber from lignocellulosic materials will play a major role

in the transition from synthetic to environmentally friendly, biodegradable green composites whose feedstock is from wood and plants [19,20].

Application of natural fiber reinforced composites in the EU is on the increase with various researchers and industries taking the lead in the application of bio fibers for polymer reinforcement (Figure 1). According to the report on Global Natural Fiber Composites Market 2014-2019: Trends, Forecast and Opportunity Analysis [21], by 2016 the natural fiber composites market is expected to be worth US \$531.2 million with an expected annual growth rate of 11% for the next five years. Currently, natural fibers account to over 14% share of reinforcement materials; however, the share is projected to rise to 28% by 2020 amounting to about 830,000 tons of natural fibers [22].

**Table 1.** Fibrous Materials Comparison [19,23]

	<b>Properties</b>	<b>Unit</b>	<b>Plant Fibers</b>	<b>Glass Fibers</b>	<b>Carbon Fibers</b>
<b>Economy</b>	Annual global production	Tonnes	31,000,00	4,000,000	55,000
	Distribution for FRPs in EU	Tonnes	Moderate (~60,000)	Wide (600,000)	Low (15,000)
	Cost of raw fiber	Euros	Low	High	High
<b>TECHNICAL</b>	Density	gcm <sup>-3</sup>	Low (~1.35-1.55)	High (2.50-2.70)	Average (~1.70-2.20)
	Tensile Modulus	GPa	Moderate (~30-80)	Moderate (70-85)	High (150-500)
	Tensile Strength	GPa	Low (~0.4-1.5)	Moderate (2.0-3.7)	High (1.3-6.3)
	Percentage Elongation	%	Low (~4-3.2)	High (2.5-5.3)	Low (0.3-2.2)
	Tribological resistance		No	Yes	Yes
	Energy requirements	MJ/kg	Low (4-15)	Moderate(30-50)	High (>130)
<b>Ecological</b>	Renewability		Yes	No	No
	Recyclable		Yes	Partially	Partially
	Biodegradable		Yes	No	No
	Dermal and inhalation toxicity during processing		No	Yes	Yes



**Figure 1.** Helmet, Car body and other natural fiber composite products by INVENT GmbH

**Table 2.** Vehicle Manufacturers and use of natural fiber composites [24].

Automotive Manufacturer	Model	Model Applications
Audi	A2, A3, A4 (and Avant), A6, A8.	Roadster, coupe, seat backs, side and back door panels, boot lining, hat rack, spare tyre lining
BMW	3,5,7 Series	Door panels, headliner panel, boot lining, seat backs, noise insulation panels, moulded foot well linings
Citroen	C5	Interior door paneling
Daimler-Chrysler	A,C,E and S-Class; EvoBus (exterior)	Door panels, windshield, dashboard, business table, pillar cover panel
Ford	Mondeo CD 162, Focus	Door panels, B-pillar, boot liner
Lotus	Eco Elise	Body panels, spoiler, seats, interior carpets
Mercedes-Benz	Trucks	Internal engine cover, engine insulation, sun visor, interior insulation, bumper, wheelbox, roof cover
Peugeot	406	Seat backs, parcel shelf
Renault	Clio, Twingo	Rear parcel shelf
Rover	2000 and Others	Insulation, rear storage shelf/panel
Toyota	Brevis, Harrier, Celsior, Raum	Door panels, seat backs, Spare tyre cover
Vauxhall	Corsa, Astra, Vectra, Zafira	Headliner panel, Interior door panels, pillar cover panel, instrument panel
Volkswagen	Golf, Passat, Bora	Door panel, seat back, boot lid finish panel, boot liner
Volvo	C70, V70	Seat padding, natural foams, cargo floor tray



## **2. Purpose and Aim of the Thesis**

### **2.1. Aim and Objectives of the Study**

The purpose of this study was the investigation of the mechanical and thermo-acoustic properties of barkcloth and its polymer reinforced composites for automotive applications.

The specific objectives are investigation of:

1. Morphology, Chemical and Thermo-physiological properties of Barkcloth.
2. Mechanical and Thermal Analysis of Barkcloth Laminar Epoxy Polymer Composites.
3. Thermo-acoustic Properties and Sound Absorption Models of barkcloth.

### **2.2. Research Design**

In reference to the present research and available literature, there are three novel/original concepts found: There has been limited laboratory work done on barkcloth fabrics except for the work which is now documented in the publications arising from this work.

This work is the first to study the morphological and chemical characterization of barkcloth nonwoven fabrics.

The utilization of barkcloth fabrics as a promising sound absorption and thermal insulation material is the first of its kind and the fabric showed exceptional properties.

## **3. Overview of the current state of the problem**

With increasing level of technology and research, new fibers have been developed. The shift from overdependence on synthetic fibers with fossil fuels as raw material sources and the need for reduction of adverse effects of man-made fibers which contribute to the carbon footprint is realized worldwide. The quest for sustainable materials from renewable sources is being fronted as one of the ways of becoming resilient to climate change and to provide for a reduction in emissions. Plant based fibers haven't been fully characterized and their respective processing and pretreatment technologies for the production of industrial composites are still under development.

Barkcloth has been in existence as far back as the 13th century, however, the fabric had not been characterized and there was limited scientific published information on the naturally occurring non woven felt. This report, therefore, presents the findings of the investigation of the microstructural, mechanical and sound absorption properties of barkcloth and its reinforced epoxy polymer composites for possible automotive applications.

## **4. Materials and Methods**

### **4.1. Raw Material Analysis**

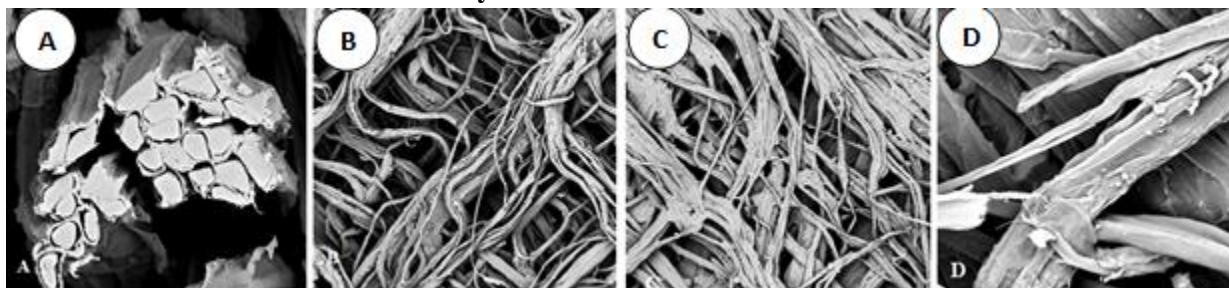
#### **4.1.1. Barkcloth Extraction**

Barkcloth utilized in this study was from two species *Ficus natalensis* and *Antiaris toxicaria*. Both species were obtained from Uganda and were extracted using the method described by Rwawiire and Tomkova, 2014 [25].



**Figure 2.** Extraction of Barkcloth non-woven natural fabric: (a) Scrapping of tree outer layer. (b) Use of local wedged tool to peel off the bark. (c) Peeling of the bark. (d) Covering of the tree stem for environmental sustainability. (e) Pummeling under the shade. (f) Sun drying of the non-woven fabric. [25]

#### 4.1.2. Barkcloth Microstructural Analysis

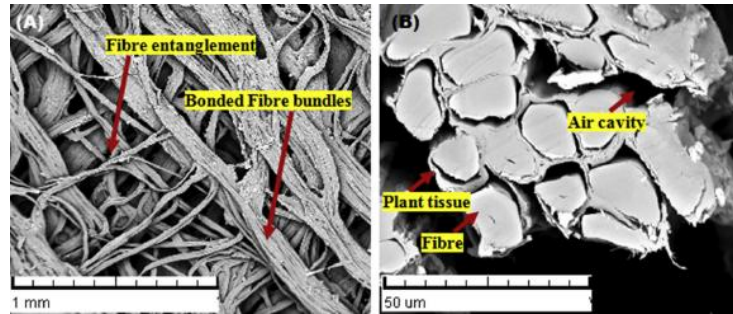


**Figure 3.** SEM morphology of treated barkcloth at magnifications (A) 50X, (B) and (C) 100X and (D) 500X

Figure 2 shows the detailed process of the production of barkcloth. The utilization of Scanning Electron Microscopy (SEM) for fabric morphology is advantageous due to the fact that more fibers in the fabric are in focus and included in the image compared with other methods [26,27].

In order to show a representative image of the fabric, the magnification was optimized by using magnifications of 50, 100, 500, 1000 and 2000 (Figure 3). The fabric morphology is made up of a dense network of naturally bonded microfibers that are oval in shape with diameters 10-20 $\mu$ m. The microfiber bundles appear to be aligned at angles, (Figure 4a). The inter-fiber bond structure gives the strength of the load bearing microfibers and damage is initiated through separation of the individual microfiber bundles through the failure of the inter-fiber bond and thence fracture [28]. The transverse section of the fabric is

characterized by air cavities and microfibers surrounded by plant material, (Figure 4b). The air cavities are responsible for the thermal insulation and sound absorption properties of the fabric [29,30].

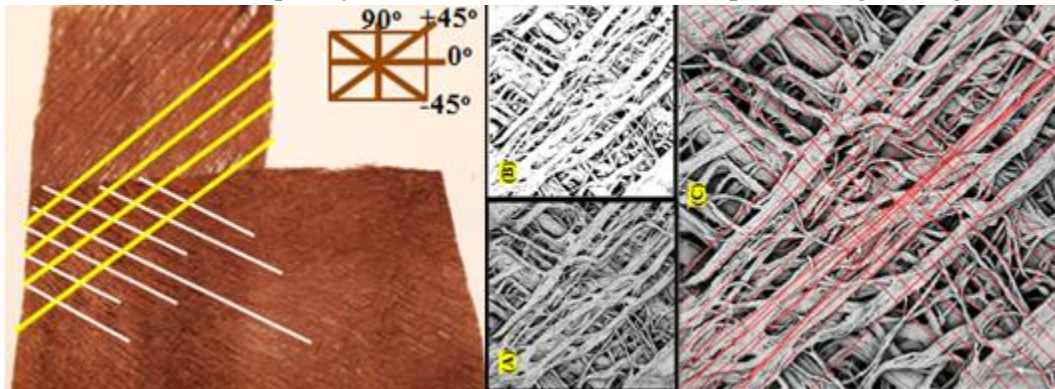


**Figure 4.** Barkcloth morphology

#### 4.1.3. Fiber Orientation Distribution in Barkcloth

Image processing techniques are used in the microstructure investigation of nonwoven fabrics so as to understand fiber orientation distribution and fiber diameter. The Fourier Transform (FT), Hough Transform (HT) and Direct Tracking are the methods used in the estimation of the fiber orientation distribution in nonwoven materials[31]. Unlike other methods, the HT method obtains the fiber distribution in the nonwoven directly and the actual orientation of the straight lines is plotted on the image with minimal computational power.

Using a digital camera image as shown in Figure 5, it's observed that the bonded barkcloth microfiber bundles are nearly aligned at an angle of  $\pm 45^\circ$  to the horizontal. These fiber bundles are held together through fiber entanglement. While using the HT, the image magnification affects the results obtained, elsewhere Ghassemieh [26] showed that a magnification of 30X and 50X produced the best representative image of the nonwoven; consequently, the results were favorable compared to higher magnifications.



**Figure 5.** The barkcloth ply representation: Image Processing: Scanning Electron Microscope image at 50X magnification (A); Binarized image (B); Hough Transform Lines showing Fiber Orientation in the image (C).

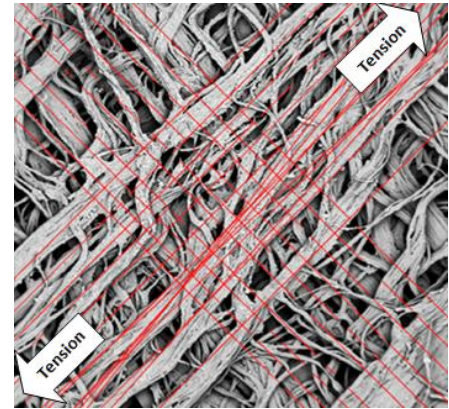
The 50X SEM image magnification was therefore utilized for image processing in matlab (Figure 5A). In order for the HT to function optimally, the image is binarized (Figure 5B); therefore the HT algorithm identifies points in the image which fall on to the straight lines. The lines are thereafter plotted on the original image. Figure 5C shows the orientation of the most microfiber bundles in the barkcloth. The application of the HT further justifies the barkcloth microfiber bundle orientation of  $\pm 45^\circ$ .

#### 4.1.4. Barkcloth Material Properties

**Table 3.** Overview of Barkcloth Material Properties

Property	Unit	Value
<i>Physical and Mechanical Properties</i>		
Areal Weight (Alkali Treated)	g/m <sup>2</sup>	142
Areal Weight (Untreated)	g/m <sup>2</sup>	327
Average Thickness	mm	1.12
<i>Fabric Strength</i>		
Microfiber bundle Direction (Figure 3.15)	N	101.7
Transverse	N	23.5
<i>Chemical Composition</i>		
$\alpha$ -Cellulose	%	68.69
Hemicellulose	%	15.07
Lignin	%	15.24
<i>Thermo-physiological properties</i>		
Thermal conductivity coefficient	W/m.K	0.0357
Thermal absorptivity	Ws <sup>1/2</sup> /m <sup>2</sup> K	0.197
Thermal Resistance	m <sup>2</sup> K/W	81.4
Thermal Diffusivity	m <sup>2</sup> s <sup>-1</sup>	0.034
Peak heat flow density	[Wm <sup>2</sup> ] x10 <sup>-3</sup>	0.234
Relative Water Vapor Permeability	%	66
Evaporation resistance	Pa.m <sup>2</sup>	4.4

The mean fabric thickness was computed as 1.084mm from ten samples of readings at different positions of the fabric. The mean strength of the fabric was 101.7N and 23.5N in the direction of the most microfiber bundle arrangement and transverse section respectively. Since barkcloth microfiber bundles are aligned at angles as shown in Figure 4 and Figure 5, the fabric samples were cut in such a way that the tests are applied in the longitudinal (microfiber bundles direction) and transverse directions (perpendicular) as shown in Figure 6.

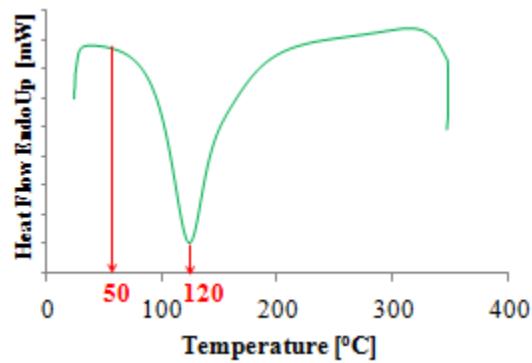


**Figure 6.** Representation of fabric strength tests with respect to microfiber bundle direction

#### 4.1.5. Green Epoxy

Green epoxy CHS-Epoxy G520 is a low molecular weight basic liquid epoxy resin containing no modifiers, certified by International Environmental Product Declaration Consortium (IEC). The green epoxy utilized for the production of biocomposites had not been characterized before; therefore its behavior was investigated in this section.

#### 4.1.5.1. Curing behaviour of the bio-epoxy



**Figure 7.** Green Epoxy Curing Behavior

The curing behaviour curve of the bio-epoxy resin was obtained from DSC and is shown in Figure 7. The selected temperature for the curing process of barkcloth biocomposites was 120°C which is near the temperature peak of the curing curve.

The curve shows only one exothermic peak, which was attributed to the cross-linking reaction between the green epoxy polymer and the hardener. The reaction starts at a temperature of approximately 55°C and ends at about 200°C. The peak temperature of the curve indicates the maximum cross-linking temperature or fastness of the curing reaction that was obtained at 123°C. For optimization of the curing of the resin, the oven temperature was therefore set at 120°C and the samples baked for 45min. The selected temperature of 120°C ensures maximum cross-linking within a short period of time less than an hour. The 45min was chosen based on the fact that from room temperature to the maximum cross-linking temperature obtained from the curing curve, the virgin resin-hardener mixture took 12 min, therefore introduction of the reinforcing fabric means that baking from 30 to 45 min is sufficient for efficient bond formation and cure of the resin. The exothermic peak at 123°C, released heat of 373 J/g which is higher than the petroleum based epoxy resins exothermicity of 200J/g. This implies that the curing of renewable green epoxy is higher than that of petroleum based epoxy resins at room temperature [32,33].

## 4.2. Composite Materials Design Fundamentals

This section is aimed at introducing the steps taken in designing of the Barkcloth Fabric Reinforced Epoxy Laminate Composites (BFRP). The theories and assumptions which dictated the design phase are presented such that the reader is well versed with the topic at hand.

### 4.2.1. Design Criteria

Design of a structure or component is aimed at avoiding failure of the component during its service life. Currently, the design of fiber reinforced plastic composites uses the same design criteria for metals. In this investigation, since the envisaged barkcloth composites were for interior automotive applications, the designed barkcloth laminate composites must sustain the design ultimate load in static testing [34]. Design Load of Interior automotive components should be >25MPa [35].

### 4.2.2. Design Allowable

Design allowable properties of laminate composites are based on testing the laminates or using laminate analysis. The latter is utilized for composites obtained for glass and carbon fiber because the material properties do not vary to a larger degree compared to natural fiber composites. Therefore in this

investigation, the former i.e. static and dynamic mechanical testing was chosen as a route in this investigation.

### 4.3. General Design Fundamentals

Mallick [34] showed three principal steps followed in designing a composite laminate:

1. Selection of composite material properties (Fiber, Resin and Volume fraction).
2. Selection of the optimum fiber orientation in each ply and the overall stacking sequence of the composite.
3. Selection of the number of plies needed in each orientation and this also determines the thickness of the composite.

#### 4.3.1. Material Selection

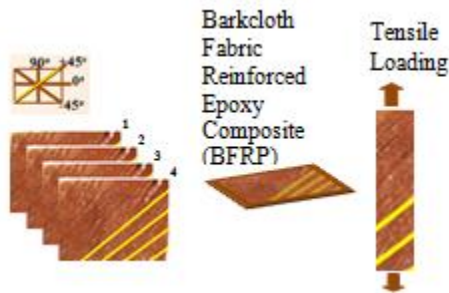
Natural nonwoven barkcloth fabrics were utilized in the study. Untreated and surface modified fabrics were utilized for the production of the composites. It was showed in Chapter 4 that the *Ficus natalensis* barkcloth's microfibers bundles are aligned majorly aligned at an angle of  $\pm 45^\circ$  (Figure 3.13 and 3.14); it's this angle that was used as the reference angle so as to come up with the laminar layering sequence for the barkcloth laminar epoxy composites (BFRP).

#### 4.3.2. Fiber-Matrix interface

In order to tailor the fiber-to-matrix interface, surface modification of the fibers was performed. The fabric's surface was treated with the enzyme and plasma for synthetic epoxy composites whereas alkali treatment was preferred for the green composites.

#### 4.3.3. Types of fibers and the reason for the fabric ply arrangement

##### Layering Sequence I



**Figure 8.** Composite Laminate layering sequence I

Composites	Ply Arrangement [°]
BFRP I	-45, 45, 0, 90
BFRP II	45, -45, 90, 0
BFRP III	0, 90, 45, -45
BFRP IV	90, 0, -45, 45



**Figure 9.** Barkcloth Fabric Reinforced Composite (BFRP) Laminate layering sequence

Figure 8 above shows the stacking sequence utilized for the fabrication of the Untreated, Enzyme and Plasma treated Barkcloth Fabric Reinforced Composites; consequently, the direction of the loading of the composites is herewith shown.

##### Layering Sequence II

A second set with advanced hierarchal architecture as shown (Figure 9) was investigated in order to understand if at all the ply angle arrangement has an effect on the thermo-mechanical properties.

The green epoxy composites (Biocomposites) were fabricated utilizing this layering sequence as well as synthetic epoxy composites with notation BFRP I-IV. Optimization of the fiber properties was achieved through varying the fiber angles through the hierarchical architecture of the barkcloth layers. The layering pattern of the barkcloth fabrics used for the purpose of the study of the effect of layering pattern is shown in Figure 4.11. A proper selection of laminar stacking sequence eliminates the deleterious free edge effects in a laminar and therefore alternations of  $+\theta$  and  $-\theta$  plies should be done so as to achieve positive results. When the stress state in the structure is unknown, a common approach in laminate design is to make it quasi-isotropic and using the layers to determine the total thickness of the laminate [34].

#### **4.3.4. Reinforcement Volume Fraction**

The composites produced with VARTM maintained a 40% fiber volume fraction ( $v_f$ ) whereas the hand lay-up composites reached a volume fraction between 14-18%. The variation of the volume fraction of the composites and laminate thickness is shown in *Appendix B* of the thesis. Information about the volume fractions of composite components were used for prediction of especially the thermal conductivity of designed composites, as shown in *Appendix C* of the thesis.

#### **4.3.5. Number of Layers (plies) and thickness**

As shown above, in this investigation since the overall loading of the envisaged composites in automotive interior panels should have a minimum of 25MPa, a quasi-isotropic laminate design process was utilized (Figure 9). Four barkcloth layers were selected so as to achieve a thickness in the range of 2-4mm which is the range of thickness of most interior automotive components. The green composites (biocomposites) utilized only one layering sequence (BFRP IV) reason being that after the investigation of the effect of the stacking sequence on the mechanical properties, BFRP IV and BFRP II emerged as the best fabric layering sequence obtained.

#### **4.3.6. Types of Matrix**

Two sets of matrices were utilized, synthetic and green epoxy matrices. The synthetic epoxy polymer was utilized with untreated, enzyme and plasma treated composites and also composites investigating the effect of layering/stacking sequence.

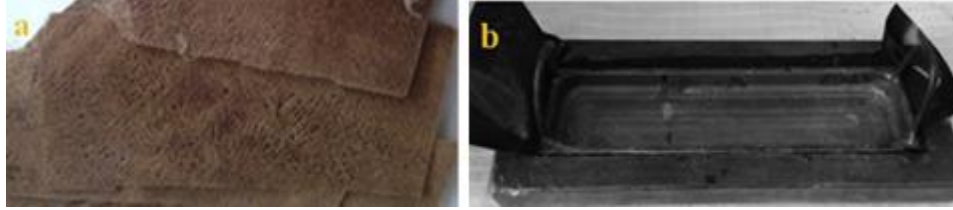
#### **4.3.7. Manufacturing Process**

##### ***Synthetic Epoxy Composite***

Vacuum Assisted Resin Transfer Molding (VARTM) was used to prepare the composites. The resin to hardener ratio was 100:40 as per the manufacturer's specifications. VARTM ensured that the composites produced had a 40% fiber volume fraction. Four barkcloth plies were used for the composite sample preparation for each set of composites. Synthetic epoxy was utilized for the production of Barkcloth Reinforced Plastic Composite (BFRP). After the resin infusion, the composite was left to cure at room temperature for 72 hours.

##### ***Green Epoxy Composite***

The biocomposite specimens were prepared using the hand lay-up method due to the fact that the viscosity of the green epoxy polymer was high. The mould was treated with a mould release agent and thereafter Teflon sheets were applied to aid the fast removal of cured composite specimens.



**Figure 10.** Biocomposites processing: (a) Barkcloth fabrics. (b) Fabricated composite mould.

Alkali treated barkcloth fabrics (Figure 10a) were impregnated with green epoxy resin and placed in the fabricated mould (Figure 10b). The resin to hardener ratio was maintained at 100:32 as per the manufacturer's specifications. Curing of the composites was done using a hot air oven for 45 min. For each set of composites, four barkcloth plies with ply angles  $90^\circ$ ,  $0^\circ$ ,  $45^\circ$ ,  $45^\circ$  were utilized for biopolymer reinforcement.

#### **4.4. Characterization and Measurements**

##### **4.4.1. Morphology**

The surface morphologies of the fabric and composite fracture surfaces were investigated using a TS5130 Vega-Tescan Scanning Electron Microscope with accelerating voltage of 20kV. The samples (fabrics, fractured composites) were sputter coated with gold so as to increase the surface conductivity.

##### **4.4.2. Surface functional groups**

The Nicolet iN10 MX Scanning FTIR Microscope was used for the investigation of the surface functional groups of the barkcloth and epoxy composite samples. The infrared absorbance spectrum of each sample was obtained in the range of  $4000\text{-}700\text{cm}^{-1}$ .

Further analysis using the X'Pert<sup>3</sup> X-ray powder diffractometer (PANalytical, USA) with Cu-K $\alpha$  radiation ( $1.54056\text{\AA}$ ) was used in obtaining diffraction patterns for alkali treated and untreated barkcloth. A Circular cut sample was directly mounted on the sample holder and analyzed from  $8$  to  $70^\circ$  with  $0.017^\circ$  incremental step.

##### **4.4.3. Thermal behaviour**

Thermogravimetric analysis of fiber samples weighing approximately 7-8mg was carried out using a Mettler Toledo TGA/SDTA851<sup>o</sup> under a dynamic nitrogen atmosphere heating from room temperature ( $25^\circ\text{C}$ ) to  $500^\circ\text{C}$  at a heating rate of  $10^\circ\text{C}/\text{min}$ . The Perkin Elmer Differential Scanning Calorimeter DSC6 was used. Samples weighing approximately 10mg were placed in aluminum pans and sealed. The specimens were heated in an inert nitrogen atmosphere from room temperature ( $25^\circ\text{C}$ ) to  $450^\circ\text{C}$  at a heating rate of  $10^\circ\text{C}/\text{min}$ .

##### **4.4.4. Mechanical properties**

The fabric strength was quantified through measurements of samples for the bursting strength of the non-woven felt. Samples measuring 5cm by 15cm were tested using a Larbotech fabric tensile testing machine at room temperature.

Tensile properties of barkcloth reinforced epoxy composite samples with dimensions  $150\times 25\times 2.5\text{mm}$  were characterized in accordance with ASTM D3039. Tensile tests were carried out using a Testometric (M500-25kN) universal mechanical testing machine operating at a crosshead speed of  $4\text{mm}/\text{min}$  until fracture. Four specimens with tabs were tested to obtain average tensile properties of the composite. The



flexural test was conducted as per ASTM D790 using a Tiratest 2300 universal testing apparatus. The samples were tested using three point bending with a recommended speed of testing of 2mm/min. The span length to thickness ratio was 32:1.

#### 4.4.5. Dynamic Mechanical Analysis (DMA)

The dynamic mechanical properties were analyzed using a DMA 40XT machine. The samples with dimensions 56x13x2.5mm were tested using three point bending mode at a frequency of 1Hz from room temperature to 150°C at a heating rate of 3°C/min.

#### 4.4.6. Fabric Surface Modification

##### *Alkali*

Barkcloth fabrics were subjected to alkali treatment of 5% NaOH solution. The barkcloth fabrics were soaked in an alkaline solution at room temperature for one hour thereafter thoroughly cleaned using distilled water to remove the alkali together with other impurities and then dried in an oven at 80°C.

##### *Enzymes*

DLG enzyme was used together with BFE to form a mixture. The enzyme solution and fabric weight ratio was all throughout maintained at 1:30. 0.3g of DLG and 0.6g of BFE were added in 900ml of distilled water. 0.2g/l of Texawet DAF which is an anti-foaming agent was added and the mixture was conditioned at 55°C ensuring a neutral pH for 90 minutes. Another bath was prepared using BFE enzyme with the same bath ratio above of 1:30. 0.6g of the enzyme was mixed with the anti foaming agent and the bath maintained at 55°C with for 90 minutes. An alkali was added so as to set the pH of 9. Caution was taken such that both enzymes are not heated with a direct heat source.

##### *Plasma*

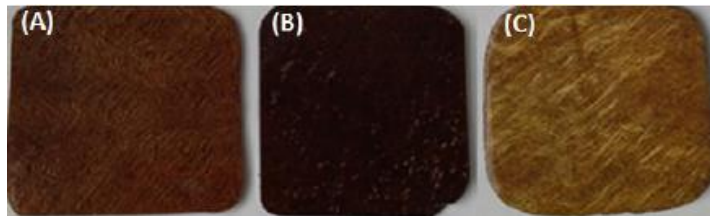
Bark cloth fabrics were treated with Dielectric Barrier Discharge (DBD) plasma using a laboratory device (Universal Plasma Reactor, model FB-460, Class 2.5 from Czech Republic). The sample fabrics were placed in the reactor for the duration of 30s and 60s respectively at power of 150W.

#### 4.4.7. Chemical Composition

The lignin, hallowcellulose, cellulose and hemicelluloses content was obtained by the method described by Bledzki et al., 2008 [36].

#### 4.4.8. Thermo-physiological properties

The thermo-physiological properties were evaluated using ISO 11092 (EN 31092) standard with laboratory room temperature at 24°C and at a relative humidity of 40%.



**Figure 11.** Thermal Conductivity Samples: (A) *Ficus brachypoda*; (B) *Ficus natalensis* and (C) *Antiaris toxicaria*

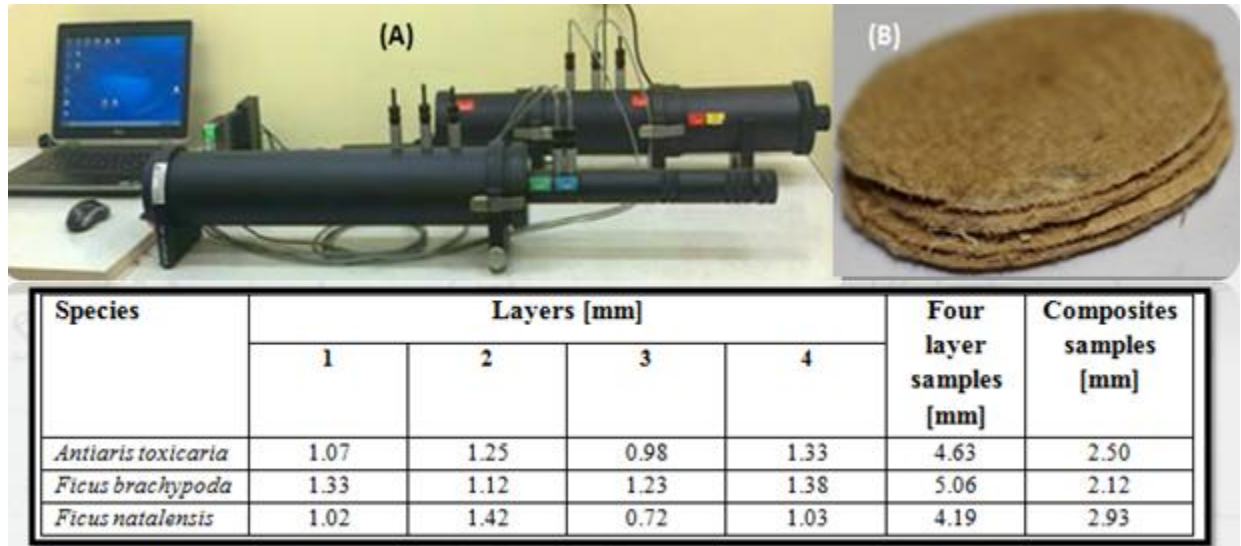
The Alambeta instrument [37] was used to measure the thermal conductivity, thermal diffusivity, thermal absorptivity, thermal resistance, sample thickness and peak heat flow density. Alambeta thermal conductivity measuring device also measures thermal conductivity of laminar composite specimens up to 8mm. The composite samples (Figure 11) were grinded using sandpaper so as to achieve a uniform smooth surface for thermal conductivity tests.

#### 4.4.9. Bio-epoxy resin characterization

The bio-epoxy resin curing behaviour was characterized by thermal analysis using the Perkin Elmer Differential Scanning Calorimeter DSC6. A small drop of resin: hardener (100:32 weight %) weighing approximately 7mg was placed in aluminium crucible and subjected to a heating rate of 10°C/min from 25°C to 400°C.

#### 4.4.10. Acoustic properties

The barkcloth and its composite acoustic properties were investigated using a type 4206 Brüel&Kjær impedance tube according to ISO10534-2 standard using two quarter-inch condenser microphones type 4187 (Figure 5.13).



**Figure 12.** Sound Absorption Measurement procedure: (A) Brüel&Kjær impedance tube set up; (B) Sample of the barkcloth layers utilized.

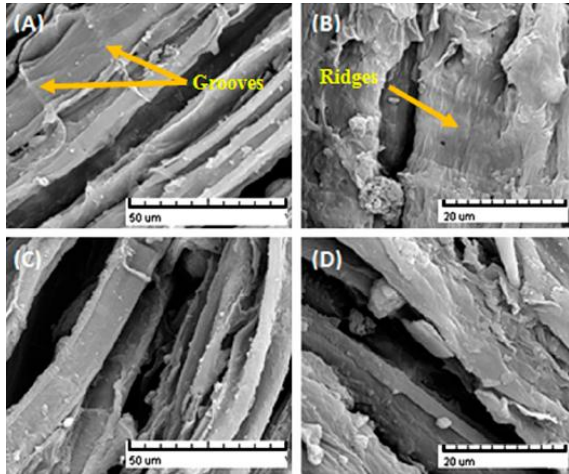
The material samples had a diameter of 29 mm and were studied in the frequency range of 500–6400 Hz. The composite and multi-layer samples were cut to the above diameter according to the standard. Measurements for composites, single layer, double layers, triple layers and finally quadruple layers. Since the fabricated BFRPs specimens had four plies, acoustic behaviour had to be characterized for the respective barkcloth layers and their effects on sound absorption. Table above shows the thickness of the fabric non-woven felts used. The airflow resistivity was measured utilizing the air resistance meter and the value of airflow resistivity was calculated utilizing the equation below:

$$\sigma = \frac{\Delta P}{Ud} \quad (1)$$

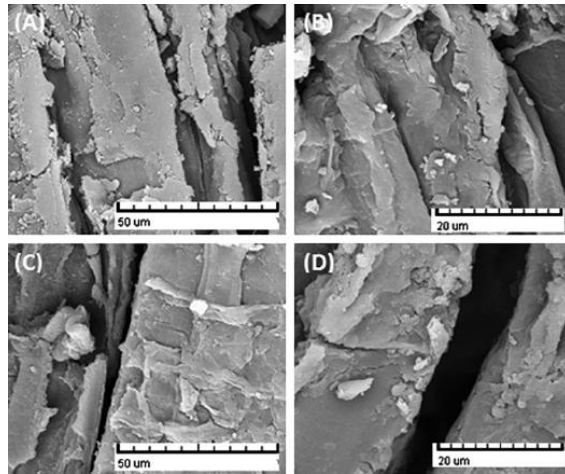
Where  $\Delta P$  is the set pressure difference between the surfaces; U is the air flow velocity and d is the thickness of the sample.

## 5. Summary of the Results achieved

### 6.1. Morphology of Surface Modified Barkcloth



**Figure 13.** Enzyme-treated barkcloth: (A) and (B) BFE enzyme; (C) and (D) DLG enzyme.



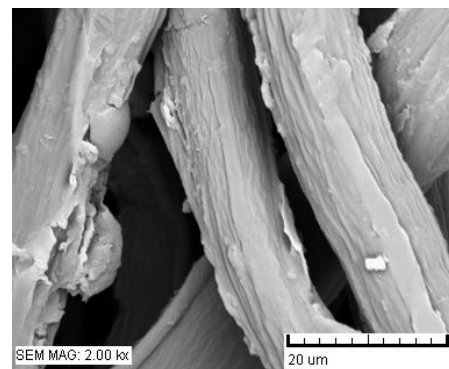
**Figure 14.** Plasma-treated barkcloth: (A) and (B) 30 s; (C) and (D) 60 s.

Treatment of barkcloth with commercial enzyme BFE led to ridges and grooves along the microfibers (Figure 13). DLG enzyme is a textile auxiliary agent, it catalyzes the decomposition of hemicelluloses and partially lignin in the binder layers of bast fibers. The combination of DLG and BFE treatment led to change in mass of the substrates by 31% whereas BFE alone led to 30% change in mass of the substrates. The change in mass of the weighed substrates shows that the enzymes used dissolved a considerable amount of plant material in the form of waxes, pectins, and other impurities. Because the main function of BFE enzyme is to dissolve impurities, it is observed that its performance was better than DLG and the surfaces are fairly cleaner compared to DLG. The loss in weight of the substrates was more with the DLG enzyme, due to the fact that the nature of DLG affects cellulose, unlike BFE.

Plasmas are used to modify lignocellulosic fibers, therefore, it aids in surface activation of the fiber network [38]. Barkcloth fabric is made up of cellulose; therefore, the only reactive functional group is the hydroxyl group. Treatment with atmospheric plasma leads to oxidation of the cellulose.

The morphology of oxygen plasma-treated fabrics is shown in Figure 14. In comparison to untreated fabrics, there was no striking visible difference on the surface. This was due to the fact that the treatment times were too short and the plasma generator device had no option of changing the power rating and the intensity of the plasma. The effect of plasma treated fabrics was observed when the fabrics were utilized in the production of the fiber reinforced composites as explained in the proceeding section on composites.

Alkali treatment is one of the leading most common vegetable fiber surface modification methods. Treatment with alkali aids in the fiber to matrix adhesion and also helps to dissolve the lignin, wax and impurities. Figure 15 shows the morphology of alkali treated fabric. Elementarization of the barkcloth cellulose microfibers measuring 12-14 $\mu$ m was observed. The microfibers appear pronounced and cleaner due to the dissolving of plant impurities.



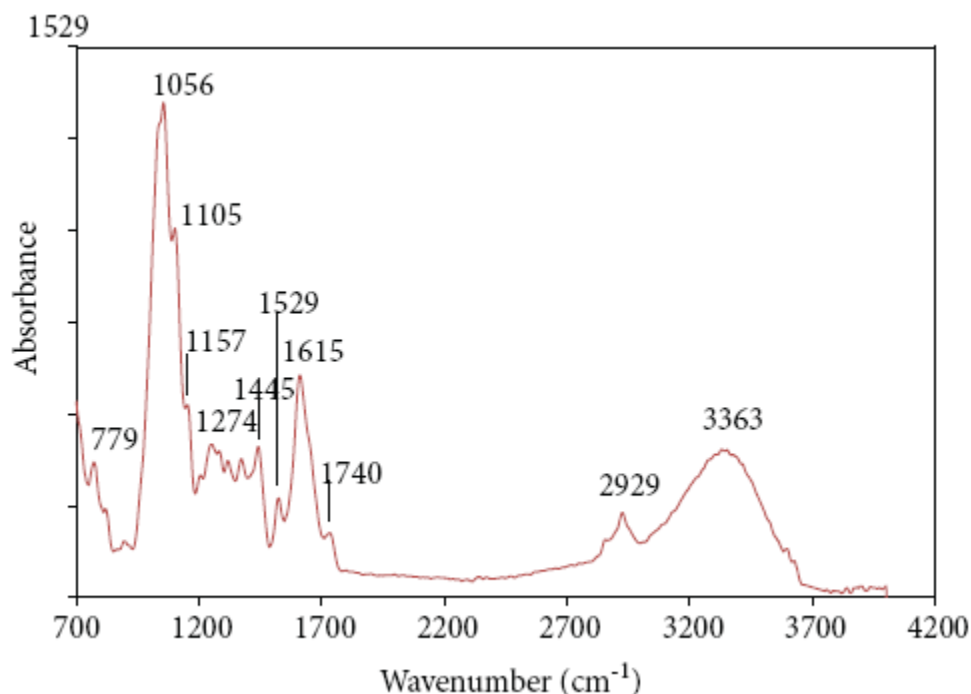
**Figure 15.** Alkali-treated (5wt% NaOH) barkcloth

## 6.2. Surface Functional Groups Of Barkcloth

Functional groups assignments and their respective bonding interactions of barkcloth can be deduced using Fourier Transform Infrared Spectroscopy.

### 6.2.1. Fourier Transform Infra-red (FTIR)

#### *Untreated Barkcloth*



**Figure 16.** FTIR spectra of untreated barkcloth.

There's a variation in the reported bands from one researcher to another, however the difference is not too significant because most natural fibrous materials are made up of celluloses, hemicelluloses and lignin. Figure 16 shows the FTIR spectrum of typical barkcloth. A broad absorption band in the range of 3300 - 3500cm<sup>-1</sup> is due to O-H stretching vibrations of cellulose and hemicelluloses. The band at 2900 - 2940cm<sup>-1</sup> corresponds to CH<sub>2</sub> and CH<sub>3</sub> stretching vibrations [39]. The band at 1740cm<sup>-1</sup> is due to carbonyl groups (C=O) stretching and vibration of acetyl groups of hemicelluloses [39]. After this peak, the sudden leveling off shows that the hemicelluloses are removed from the fiber. The aromatic vibration of the benzene ring in lignin may be at 1615cm<sup>-1</sup>.

The absorption band at 1529cm<sup>-1</sup> was owing to CH<sub>2</sub> bending in lignin whereas the peak at 1445cm<sup>-1</sup> was due to O-H in-plane bending [40]. The peak at 1380cm<sup>-1</sup> was assigned to CH symmetric bending. The band at 1274cm<sup>-1</sup> may correspond to C-O stretching of acetyl group of lignin [39]. The band at 1157cm<sup>-1</sup> may be due to C-O-C asymmetrical stretching in cellulose. The broad peak at 1056 cm<sup>-1</sup> is due to -C-O-C- pyranose ring skeletal vibration [41]. The band at 779 cm<sup>-1</sup> represents glycosidic -C-H deformation, with a ring vibration contribution and -O-H bending which are the characteristics of  $\beta$ -glycosidic linkages between the anhydroglucose units in cellulose [41,42].

The decrease in the absorption band of enzyme treated fabrics is attributed to the lower content of hemicelluloses of the fabric structure which is further confirmed by the band at 1749cm<sup>-1</sup> that is decreased and is due to carbonyl groups (C=O) stretching and vibration of acetyl groups of hemicelluloses. After

this peak, the sudden leveling off shows that the hemicelluloses are removed from the fiber. The aromatic vibration of the benzene ring in lignin may be at  $1615\text{cm}^{-1}$ . The absorption band at  $1529\text{cm}^{-1}$  was owing to  $\text{CH}_2$  bending in lignin.

### 6.2.2. X-Ray Diffraction

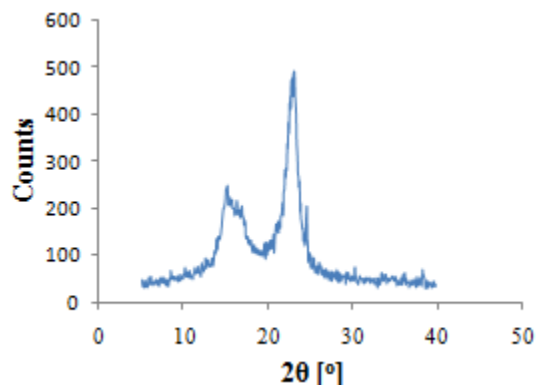
Figure 17 shows the X-ray diffraction pattern of the barkcloth. Untreated and alkali barkcloth exhibits main  $2\theta$  diffraction peaks between  $22.8^\circ$  and  $23.2^\circ$  which correspond (002) crystallographic planes of cellulose I. The peak at  $15.3^\circ$  is due to 001 crystallographic plane of cellulose I.

The Segal crystalline index was calculated using the expression [43]

$$CI = \frac{H_{22.55} - H_{18.5}}{H_{22.55}} \quad (2)$$

Where  $H_{22.55}$  is the height of the XRD peak at  $2\theta = 22.55^\circ$  which is responsible for both amorphous and the crystalline fractions whereas the small peak at  $2\theta = 18.5^\circ$  corresponds to the amorphous fraction.

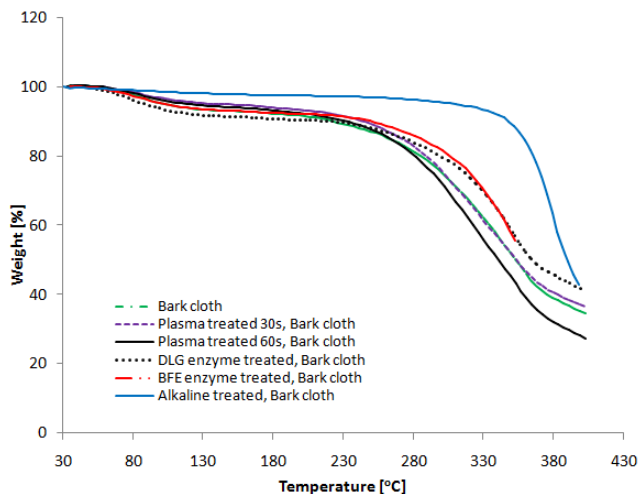
The calculated crystallinity index was 79% higher than sisal (71%), jute (71%), *Sansevieria cylindria* leaf fibers (60%) [44]. A higher value of CI shows that barkcloth crystallites are orderly in nature.



**Figure 17.** X-ray Diffraction of Treated and Untreated Barkcloth

### 6.3. Thermal Behavior Of Barkcloth

One of the drawbacks for natural fibers is their limited thermal stability. Therefore, a study of their thermal behavior is of utmost importance for material engineers. For natural fibers, the thermogravimetric behavior is directly proportional to the chemical constituents of the fibers [45,46].



**Figure 18.** Thermogravimetric behavior of treated barkcloth

Figure 18 shows the thermogram of untreated barkcloth and surface modified fabrics. The first stage from  $25^\circ\text{C} - 100^\circ\text{C}$  is attributed to evaporation of water accounting for about 5-10% loss in weight.

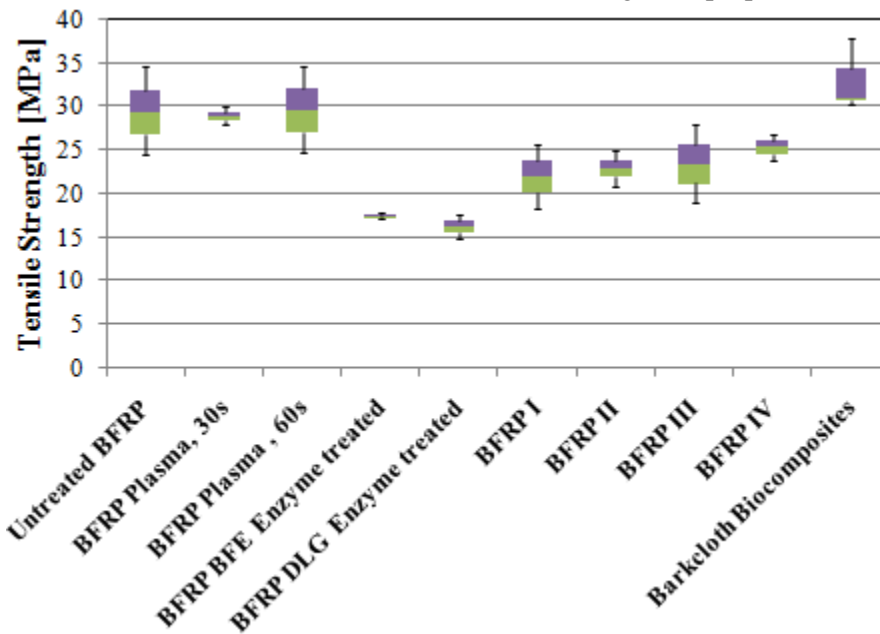
The weight loss is higher in enzyme-treated fabrics than plasma due to the fact that the enzymes degraded the plant material in the form of waxes and impurities however this led to thermal stabilization of the fabrics. Untreated and plasma-treated fabrics were less thermally stable compared to enzyme-treated fabrics. In terms of moisture evaporation, it was observed that plasma treated fabrics had less moisture followed by BFE enzyme-treated fabrics. This is attributed to the fact that plasma made the surface hydrophobic, whereas the BFE enzyme did not degrade the fabric compared to the action by DLG enzyme.

The second stage accounting to about 70% weight loss starts from about 220°C- 370°C with a maximum decomposition temperature corresponding to around 325°C. The temperature range 200°C-315°C corresponds to the cleavage of glycosidic linkages of cellulose which leads to the formation of H<sub>2</sub>O, CO<sub>2</sub>, alkanes and other hydrocarbon derivatives [41]. The last stage of decomposition starting from around 370°C corresponds to 20% loss in weight is due to char or other decomposition reactions [47].

#### 6.4. Mechanical Properties of Barkcloth Laminar Epoxy Composites

##### 6.4.1. Static Mechanical Properties

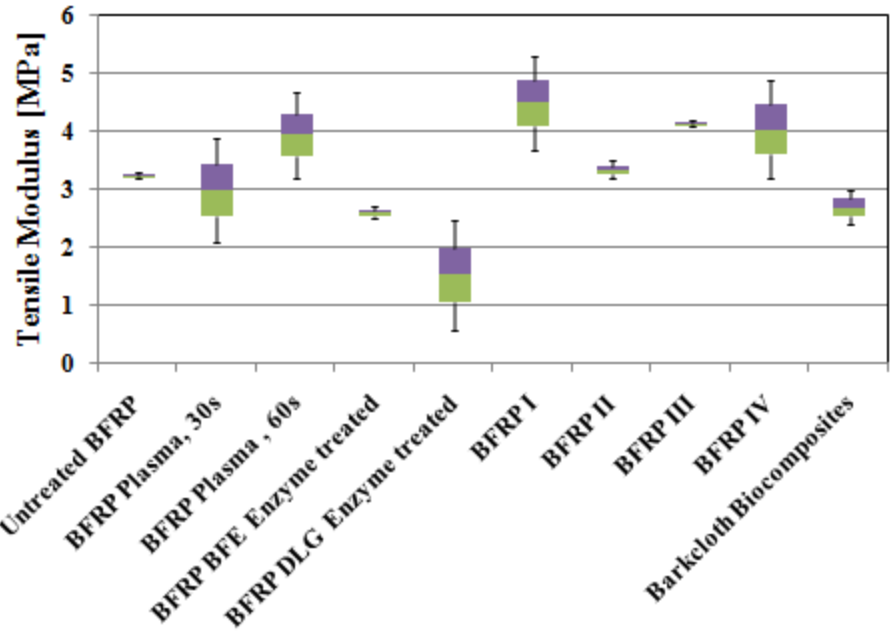
The tensile behavior of the barkcloth composite specimens was partially linear due to the highly anisotropic structure of barkcloth fabrics. The tensile strength and modulus of a composite material is dependent on the matrix, fiber to matrix adhesion and the reinforcing fiber properties. [48].



**Figure 19.** Tensile Strength of Barkcloth Fabric Reinforced Plastic Composites

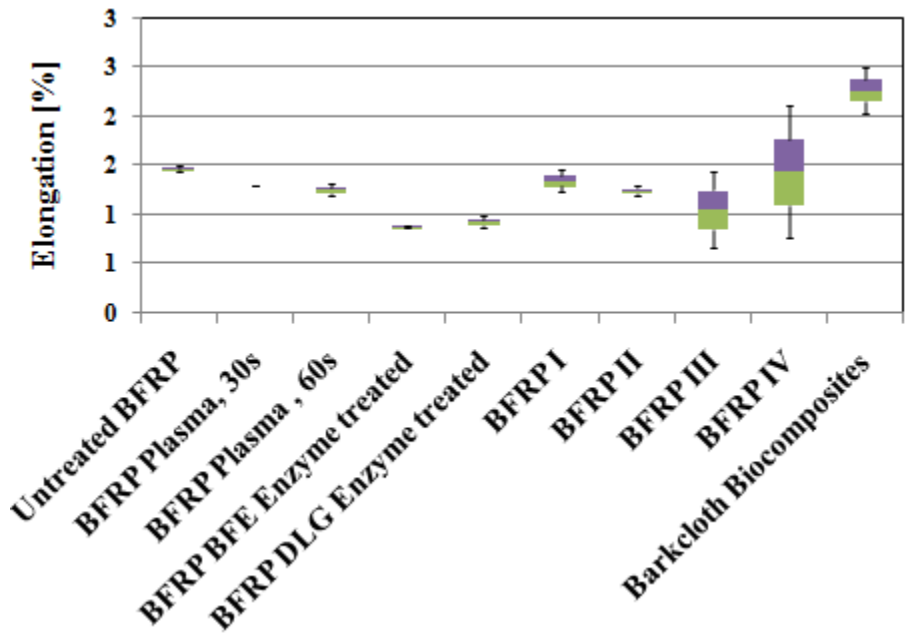
Barkcloth is a naturally occurring non-woven fabric, therefore, the climatic conditions, types of soils; the part from which the bark is extracted and the processing conditions are all variables that affect the strength of the fabric. The Stress - Strain behavior of the tested fabric reinforced composites showed a partially linear behavior. This behavior is due to the high anisotropy of barkcloth.

The tensile strength and modulus of the developed composites is shown in Figure 19 and Figure 20 respectively. The strength and modulus of a composite material is dependent on the reinforcing fiber properties, and the matrix properties [49].



**Figure 20.** Tensile Modulus of Barkcloth Fabric Reinforced Plastic Composites

Treatment with atmospheric plasma for 60s slightly enhanced the strength to an average of approximately 30MPa and average modulus ranging from 3.3GPa to 4.5GPa (Figure 20). High modulus of the plasma treated composites compared to enzyme treated composites is attributed to the fact that plasma opened up the reactive sites of cellulose, therefore, offering effective fiber-to-epoxy polymer bonding. Enzyme treated composites exhibited the lowest strength and modulus due to the fact that the natural bonding that binds the barkcloth microfibers together was dissolved and it led to weaker fabrics that was thereafter transferred to the composites.



**Figure 21.** Percentage Elongation of Barkcloth Fabric Reinforced Plastic Composites

The percentage elongation of enzyme treated composites was the lowest compared to untreated and plasma treated composites (Figure 21); this is attributed to reduced strength of the fabrics which are the load bearing materials.

#### 6.4.1.1. Effect of Fabric layering

It is observed that the stacking sequence BFRP IV had the highest tensile strength, whereas BFRP I had the highest tensile modulus and second lowest percentage of elongation at break. The strength and modulus of a composite material is dependent on the reinforcing fiber properties, fiber-to-matrix adhesion. Because barkcloth fabric has microfibers that are aligned at an angle, it is important to have a stacking sequence that will be beneficial for composite applications. It is observed that BFRP II exhibited the highest average flexural rigidity followed by BFRP IV, BFRP III, and BFRP I (Figure 22). The flexural modulus (Figure 23) shows that composites with stacking sequences II and IV are more rigid than composites with stacking sequences I and II. This rigidity is due to the presence of plies with 45° alignment at the surface.

#### 6.4.1.2. Biocomposites

The composites had ply orientations of 90°, 0°, 45°, 45° due to the fact that in the investigation of the effect of layering pattern of barkcloth composites using a synthetic epoxy polymer, it was shown that the stacking sequence of barkcloth with orientation 90°, 0°, 45°, 45° (BFRP IV) was ideal and had higher and consistent favorable mechanical properties [50].

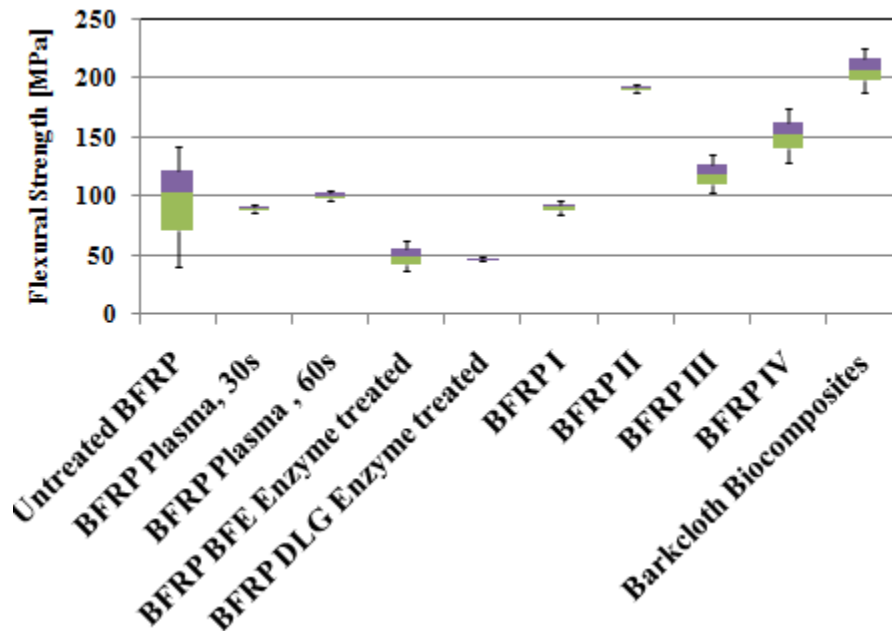
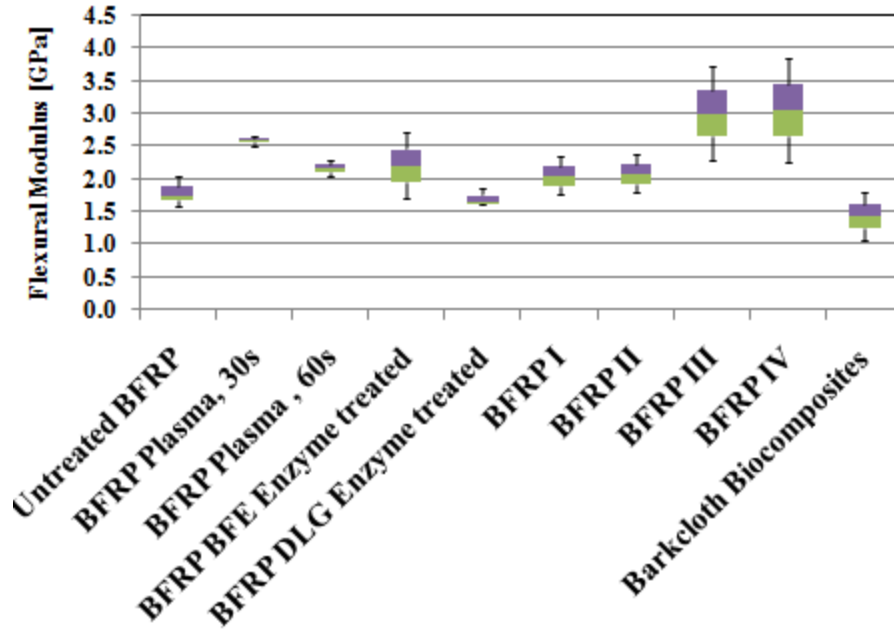


Figure 22. Flexural Strength of Barkcloth Fabric Reinforced Plastic Composites



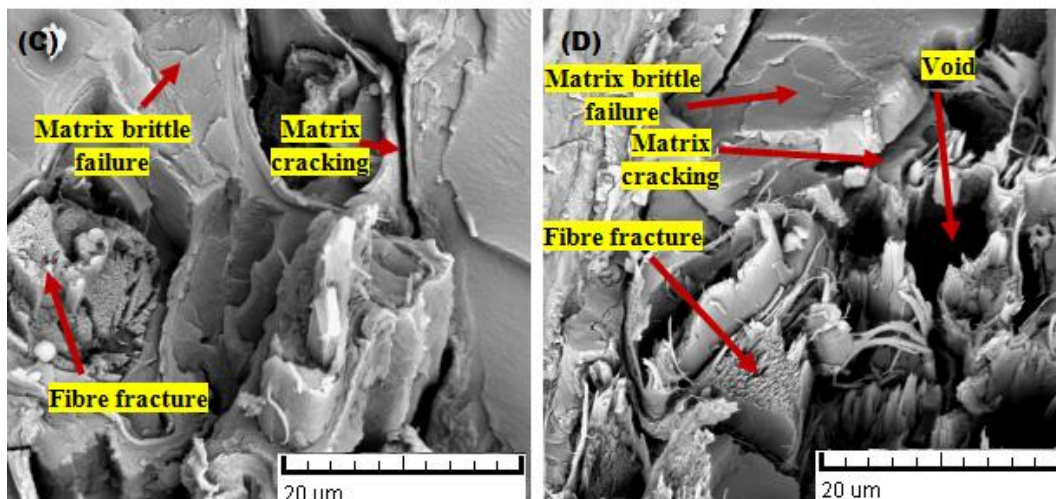


**Figure 23.** Flexural Modulus of Barkcloth Fabric Reinforced Plastic Composites

The developed biocomposites had an average strength of 33MPa higher than the strength obtained using synthetic epoxy. The percentage elongation of the biocomposites was higher than the synthetic composites. This is attributed to the green epoxy polymer properties, however, the variability of the reinforcing material is observed with the low modulus of the biocomposites owed to the treatment with alkali that dissolved impurities.

The average flexural strength of the developed green epoxy biocomposites was 207MPa higher than the untreated and synthetic composites. This was due to the effective fiber to matrix adhesion owed to the alkali treatment. During the three points bending, the upper and lower laminate surfaces are loaded with tension and compression forces respectively, whereas the axisymmetric plane is subjected to shear. Therefore, failure during flexure is achieved when the flexural and shear stress reach a critical value [51].

#### 6.4.2. Failure of Composites

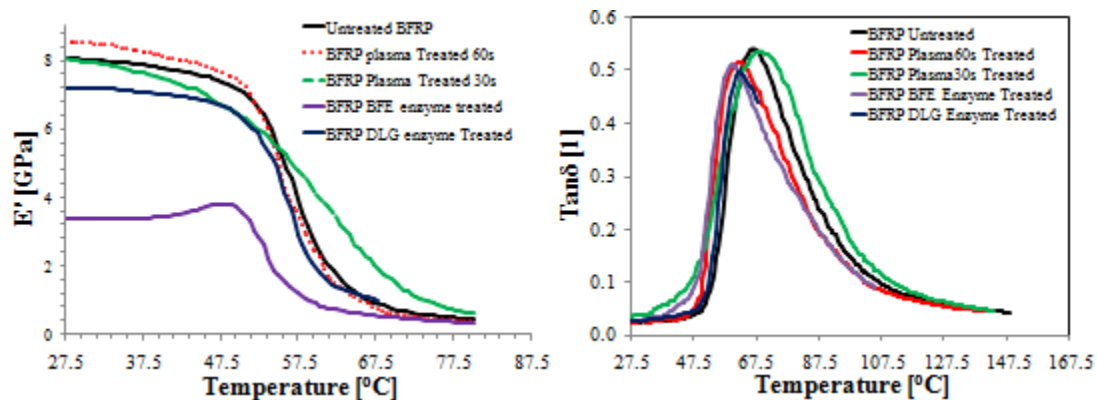


**Figure 24.** Morphology of barkcloth fabric and biocomposites

Failure by tensile was through matrix failure and the disintegration of the non-woven structure through the tensile forces. The entangled microfiber web of the fabric has natural bonds holding the microfibers together. Generally, BFRP composites experienced three modes of failure: the brittle failure of the epoxy polymer matrix; matrix cracking and fiber fracture (Figure 24). Damage of the non-woven structure is triggered by the inter-fiber bond structure; re-arrangement of the fiber network and reloading and finally fiber fracture [52].

### 6.4.3. Dynamic Mechanical Properties

In order to assess the performance of structural applications, the dynamic mechanical properties help in material evaluation so as to understand the viscoelastic behavior of the material against temperature, time and frequency. Three parameters storage modulus ( $E'$ ), loss modulus ( $E''$ ) and damping factor ( $\tan \delta$ ) were obtained over the temperature range from 28°C to 80°C for synthetic epoxy composites and 25°C to 250°C for bioepoxy composites as shown in Figures 25-27. The storage modulus indicates the viscoelastic rigidity of the composites and is proportional to the energy stored after every deformation cycle. A weak material has a low storage modulus, whereas a strong material exhibits a higher storage modulus. As the composite approaches the glass transition temperature, there's a sudden decrease in the storage modulus attributed to the free molecular movement of the polymer chains. A low mechanical damping factor indicates closer packing of the composites and elasticity of the material and a higher damping factor indicates a weaker material under loading, which could be due to weak fiber-to-matrix adhesion

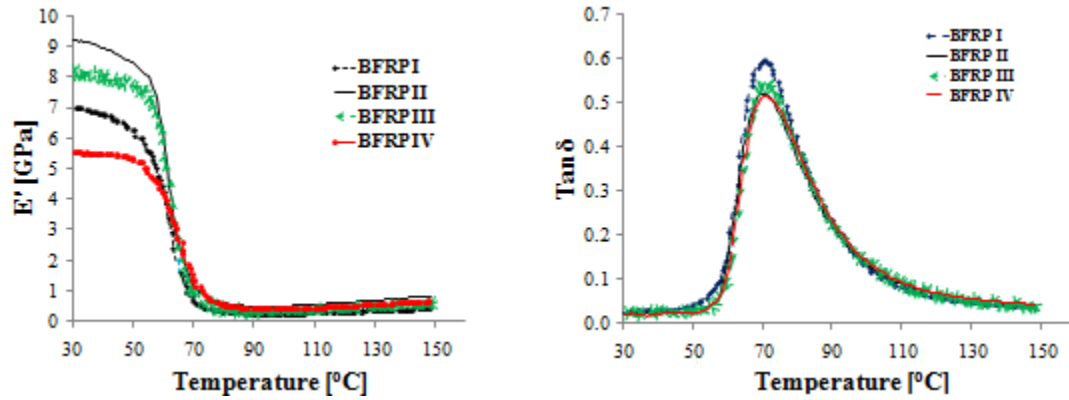


**Figure 25.** DMA of enzyme and plasma treated composites

The increased strength observed with the plasma treated composites is attributed to the strength of the plasma treated fabrics. Plasma treatment positively aided the effective cross-linking between the fabric and epoxy polymer. The enzyme treated composites; especially BFE had low storage modulus that is attributed to the enzymes that partially dissolved the microfiber natural binders leaving a very light non-woven fleece with reduced strength.

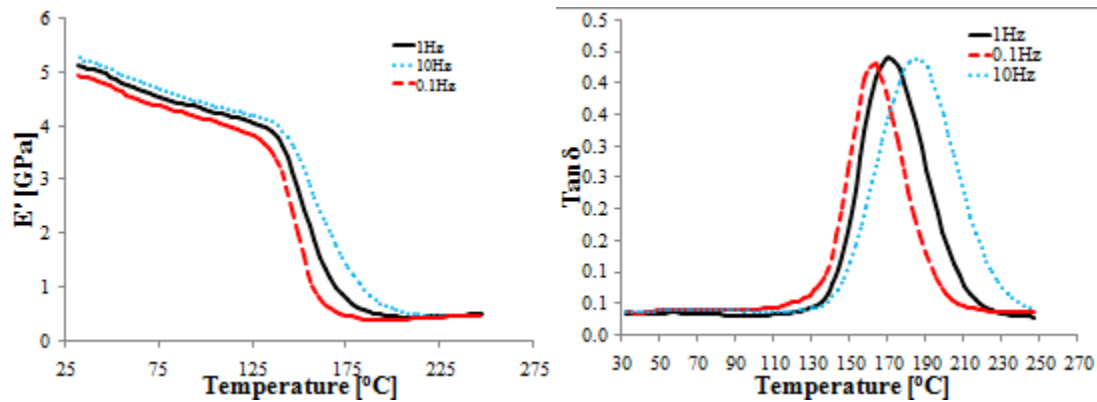
Plasma treated composites for 60s and enzyme treated composites had the best fiber-to-matrix adhesion as can be observed by the lower  $\tan \delta$  (Figure 25). The glass transition temperature is obtained at the level at which the damping factor and loss modulus attain maximum damping values [53]. The glass transition obtained using  $\tan \delta$  is usually higher; therefore, a more conservative glass transition temperature

obtained by the loss modulus is usually taken into consideration. The glass transition temperature of the enzyme and plasma treated composites was between 52.9°C to 59.4°C.



**Figure 26.** Investigation of the effect of layering on the DMA of layered composites.

Figure 26 shows the dynamic mechanical behavior of composites with advanced hierarchical architecture. An increase in the temperature led to the fall in the storage modulus in the temperature range 50° to 70°C, which is the glass transition temperature range as observed from the damping factor curve. This fall in the storage modulus is attributed to the mobility of the macromolecular polymer chains, which distorts the initial close packing [54]. The composites from the highest storage modulus to the lowest were those with stacking sequences II, III, I, and IV, which is confirmed by the flexural modulus obtained from the flexural tests in Table III.



**Figure 27.** DMA of biocomposites with layered architecture.

Figure 27 shows the variation of the storage modulus with the temperature at three scan frequencies of 0.1, 1 and 10 Hz. The addition of reinforcement to the epoxy polymer greatly enhanced the dynamic mechanical properties increasing the storage modulus from 2.6 GPa of virgin resin to 5.1 GPa of biocomposites at 30°C. The high value of storage modulus of biocomposites is attributed to the reinforcement. Under loading, the polymer chains move about and are re-arranged, with the addition of the barkcloth, the mobility of the polymer chains is greatly reduced. A sudden fall of the modulus of the

composites was observed at 130°C which is marked by a sharp decrease in the storage modulus until to around 450MPa at 225°C. As the composite approaches the glass transition temperature, there's a sudden decrease in the storage modulus attributed to the free molecular movement of the polymer chains. Polymer viscoelastic behaviour is a function of time, frequency and temperature. A frequency scan showed that the storage modulus increases with increase in the frequency. So the modulus at 10 Hz (Short time) is higher than the modulus at 0.1Hz (long time). The variation of  $\tan\delta$  against temperature, (Figure 27) aids in obtaining the glass transition temperature. It's observed that the  $T_g$  obtained by the damping factor curve was 163°C, 170°C and 185°C for 0.1 Hz, 1 Hz and 10 Hz respectively. The  $T_g$  increases to a higher temperature as the analysis frequency increases [55]. Beyond the glass transition temperature, the biocomposite transitions from glass to rubbery state due to the high mobility of the polymer molecular chains.

## 6.5. Thermal Behaviour of Barkcloth Laminar Epoxy Composites

### 6.5.1. Enzyme and Plasma Treated Fabric Composites

The composites thermal behavior as characterized by DSC is shown in Figure 28. Incorporation of barkcloth into the epoxy polymer has an effect on the crystallization behavior of semicrystalline synthetic epoxy polymer. The barkcloth reinforced epoxy laminar composites experienced endothermic and exothermic phase transformation. The first endothermic peak at around 55–61°C corresponds to the glass transition temperature ( $T_g$ ).

Table 6.5. DSC of Enzyme and Plasma Treated Composites

Composites	$T_g$ [°C]	$T_c$ [°C]	$T_m$ [°C]
Untreated	59	141	220
Virgin epoxy	56	133	200
BFE Composites	55	140	210
DLG Composites	56	137	206
Plasma 30s Composites	61	146	213
Plasma 60s Composites	61	150	215

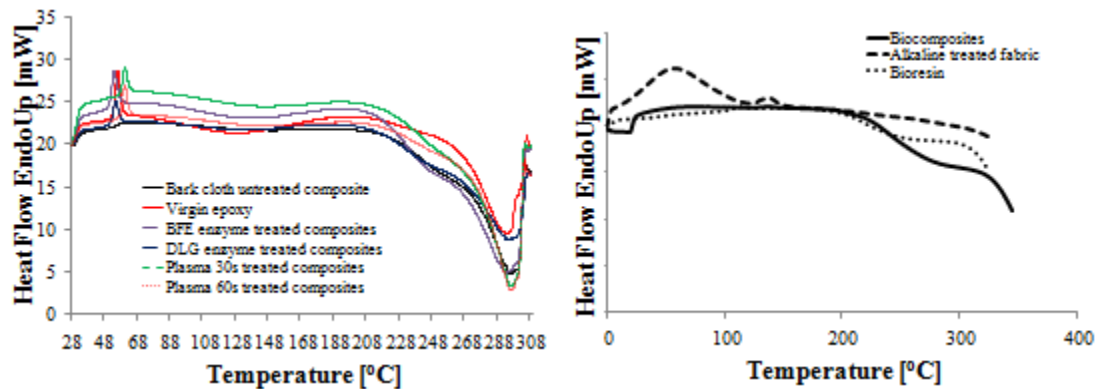


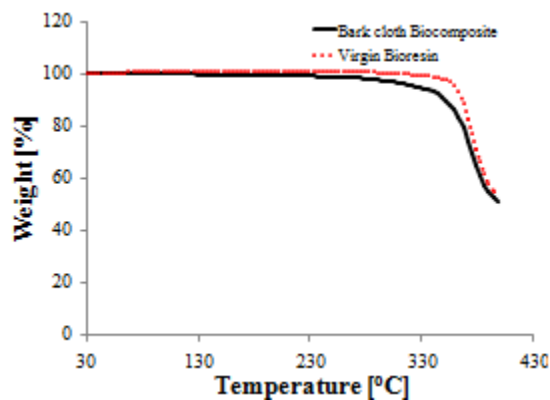
Figure 28. DSC of (A) enzyme and plasma treated composites; (B) green composites

The exothermic peak at around 133-150°C represents the cold crystallization temperature ( $T_c$ ) of the epoxy polymer chains. As the temperature is increased, a second endothermic peak is observed at around 200-220°C. This peak signifies the melting temperature ( $T_m$ ) of Epoxy polymer. Table 6.5 shows the effect of surface modification of barkcloth on the  $T_g$ ,  $T_c$  and  $T_m$ . Addition of reinforcement generally

increased the glass transition temperature, crystallization temperature as well as the melting temperature of the composites because addition of reinforcement limits the mobility of the polymer chains and therefore, a positive effect on the glass transition temperature.

#### 4.6.2. Thermal behavior of Biocomposites

Thermal degradation of natural fiber components is dictated by the supramolecular structure of the cellulosic materials [56]. The composites and fabric behavior as characterized by DSC (Figure 29), shows an endothermic peak starting from 20°C to 120°C centered at around 52°C. This peak is characterized by the removal of adsorbed moisture from the fabric. Studies with NMR have shown that moisture is concentrated in the amorphous or non-crystalline regions of cellulose [57]. Therefore, the endotherm at 52°C corresponds to the amorphous component of cellulose in barkcloth. The peak at 140°C is attributed to the decomposition of paracrystalline molecules of pectin and hemicelluloses in the barkcloth [46]. The leveled behavior of biocomposite confirms that the selected curing temperature of 120°C for 45 min was sufficient for cure.



**Figure 29.** TGA of green composites

Thermal stability of the polymer and reinforcing materials is an important parameter because manufacturing of composites in most cases requires curing; therefore, the degradation behavior of the reinforcing fibers helps in selecting the processing temperature and also the working temperature of the developed composite materials. Thermal stabilization of alkali treated fabrics led to stable biocomposites which had a higher temperature of degradation than synthetic based composites. The onset of degradation was observed at 270°C whereas cellulose decomposition occurred at 350°C, (Figure 29).

## 6.6. Overview Of Barkcloth Fabric Reinforced Composite Material Properties

Table 6.6. Summary of the Barkcloth Composite Materials Properties

Laminar Composites/ Property	Ultimate Tensile Strength [MPa]	Tensile Modulus [GPa]	Flexure [MPa]	Flexural Modulus [GPa]	Elongation [%]	Onset of Degradation [°C]	Tan $\delta$ @1Hz [-]	Glass Transition Temp [°C]
Untreated	25-31	3.2-3.3	40-142	1.6-2.1	1.5	266	0.54	67.2
Plasma Treated 30s	28-30	2.1-3.9	87-94	2.5-2.7	1.31	262	0.54	69.7
Plasma Treated 60s	25-35	3.2-4.7	97-105	2.0-2.3	1.2-1.3	262	0.51	63.1
Enzyme Treated (DLG)	15-18	0.58 -2.5	37-49	1.6-1.9	0.9	285	0.50	62.3
Enzyme Treated (BFE)	17-18	2.5-2.7	49-62	2.2-2.7	0.9	285	0.51	59.7
BFRP I*	18-26	3.7-5.3	85-87	1.8-2.3	1.2-1.5	-	0.60	70.4
BFRP II**	21-25	3.2-3.5	189-195	1.8-2.4	1.3	-	0.52	70.4
BFRP III***	19-28	4.1-4.2	103-136	2.3-3.7	0.6-1.5	-	0.54	70.4
BFRP IV****	24-27	3.2-4.9	130-175	2.3-3.5	0.8-2.1	-	0.52	70.4
Green Composites	30-38	2.4-3	189-227	1.1-1.8	2.1-2.5	345	0.44	170

\*BFRPI layering sequence

\*\*BFRPII layering sequence

\*\*\*BFRPIII layering sequence

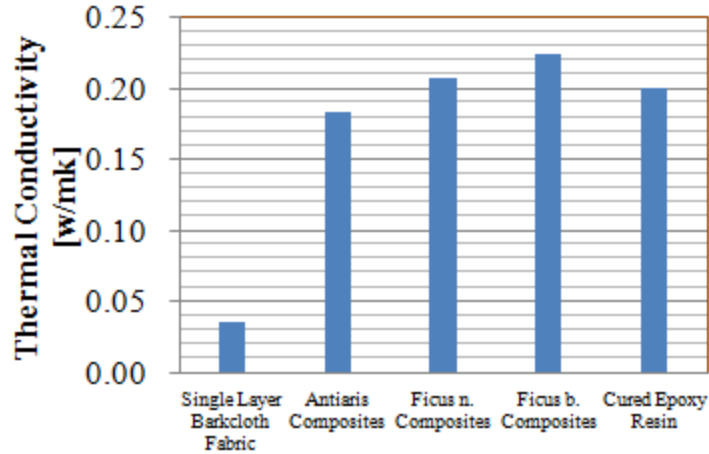
\*\*\*\*BFRPIV layering sequence

## 6.7. Thermal and Acoustic Properties

The amount of heat transmitted through a unit area of the material was measured as the thermal conductivity coefficient ( $k$ ). There is dependence between the thermal conductivity of a material and its sound absorption. When a sound wave strikes a porous fiber network like barkcloth; the sound waves cause vibration in the fiber network. The vibration causes minute heat buildup in the fibers due to friction. Therefore a good absorbing material absorbs the thermal energy of the sound waves and less heat is generated. The case is somewhat different with solid composite materials. The compaction of the barkcloth nonwoven felt results in reduced porosity, therefore increasing flow resistivity and reduced vibration of the fiber network therefore a reduced sound absorption coefficient and higher thermal conductivity. The combination of several nonwoven fabric layers allows the realization of different absorption degrees in one composite structure, which can then absorb sound in wide range of frequencies. High values of thickness and fabric density facilitate sound insulation. Microstructure parameters such as fiber orientation, tortuosity, pore structure, influence the sound absorption efficiency [58].

### 6.7.1. Thermal insulation behavior of BFRPs

The ficus species had a higher thermal conductivity among the measured specimens whereas Antiaris had the lowest thermal conductivity (Figure 30). The high thermal conductivity coefficient is attributed to the epoxy polymer used whose thermal conductivity is approximately 0.2W/mK. A lower value of  $k$  is characterized as a better thermal insulation material due to the fact that it helps in resisting outside heat transmitted through the fibrous network.



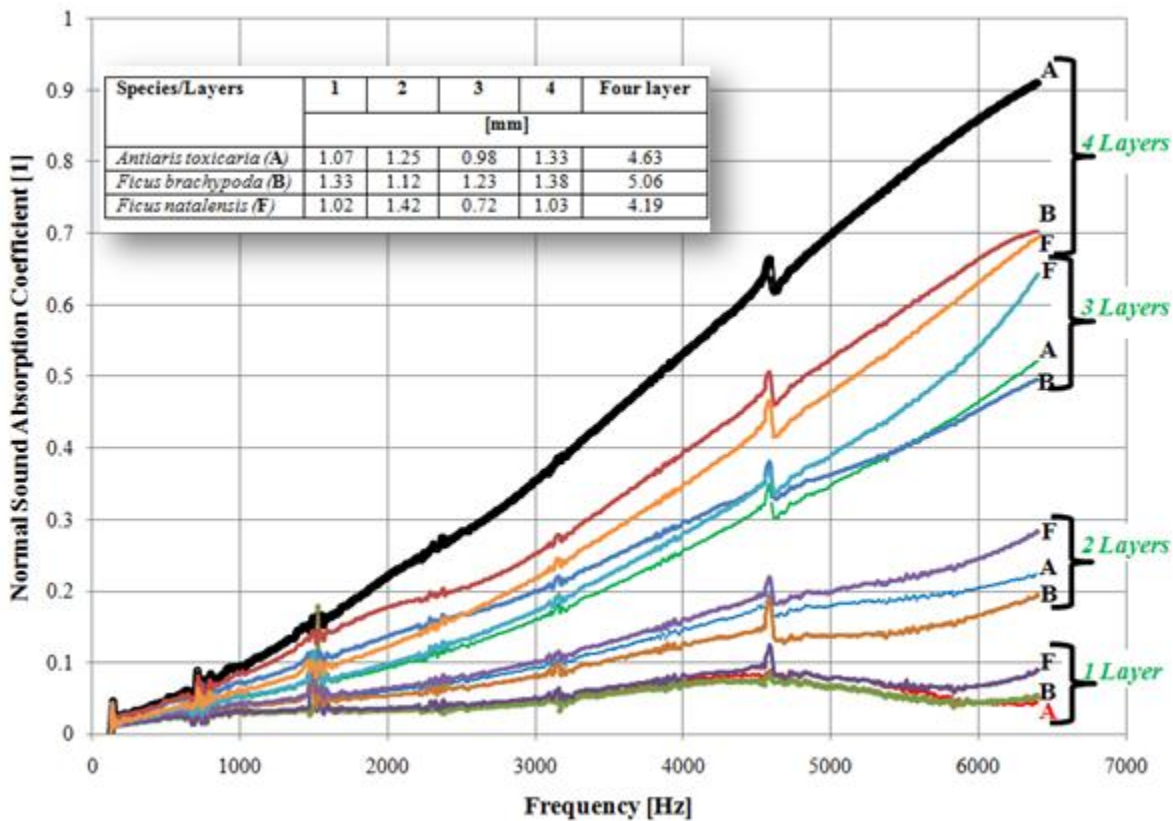
### Materials

**Figure 30.** Thermal conductivity of composites: *Antiaris* Composites (ABRP); *Ficus natalensis* Composites (NBRP); *Ficus Brachypoda* Composites (BFRP) (Measurements were based one sample)

## 6.7.2. Acoustic Properties

### 6.7.2.1. Barkcloth Fabrics

The acoustic properties of layered fabrics of the three species are shown in Figure 31. The sound absorption properties depend on the thickness since the thickness of one layer was an average of 1.14mm, it's observed that the sound absorption performance of the barkcloth fabrics generally increases with the increase of frequency.



**Figure 31.** Sound Absorption Behavior of Barkcloth

The one layer barkcloth fabrics between the frequency range of 1000-3200Hz have an average sound absorption coefficient of 0.05 whereas beyond 5000Hz the fabric's sound absorption is tending to 0.1 and the properties being favorable for *Antiaris* and *F. natalensis* barkcloth.

The effect of layer thickness was investigated and Figure 6.38 shows the sound absorption properties barkcloth fabrics with one, two, three and four layers. The sound absorption properties increase with an increase in sample thickness. All the fabrics studied had a gradual increase of sound absorption coefficient. *Ficus natalensis*' four layer fabrics had a sound absorption coefficient of 0.7 at 6400Hz.

The two layer samples showed a sound absorption coefficient of 0.1 at 3000Hz; 0.15 at 4000Hz; 0.2 at 5000Hz and 0.25 at 6000Hz. The addition of another layer almost doubled the sound absorption performance of the fabrics as can be seen in the behavior of the samples with three layers.

*Ficus brachypoda* fabrics showed the same behavior as *Ficus natalensis* fabrics. It's observed that the three layer fabrics of *f.natalensis* were better than for *f. brachypoda* as can be seen from the graphs. *F. brachypoda* had a sound absorption coefficient of 0.71 at 6400Hz.

The sound absorption performance of *Antiaris toxicaria* fabrics at 6400Hz was overall best having a sound absorption coefficient of 0.92 compared to the average of 0.7 obtained by the *Ficus* barkcloth species. Krucinska et al. [59] showed that cotton/PLA composites of 5.8mm thickness with microfibers had a sound absorption coefficient of 0.93 at 6400Hz. The barkcloth fabrics showed an irregular dependence of sound absorption coefficient of like other nonwovens over a wide frequency range, this behavior was also confirmed elsewhere [60].

The comparable excellent acoustic properties of barkcloth fabrics at high frequencies is attributed to the fiber entanglement of the fabrics and porosity. *Antiaris* barkcloth with four layers showed better sound absorption properties compared to other types of barkcloth. The increase of the thickness of the fabrics will definitely increase the sound absorption coefficients.

#### **6.7.2.2. Fabric Reinforced Composites**

The sound absorption properties were investigated further whereby the four layers of barkcloth were utilized in the production of composites. Two surfaces were investigated in order to understand whether the perturbations on the composite surface have an effect on the sound absorption properties. The molded composites' sound absorption properties of smooth surfaces are shown in Figure 6.38.

The *ficus* barkcloth composites have a sound absorption coefficient below 0.1 for the frequency of upto 3700Hz and thereafter, *f. brachypoda* sound absorption properties increased with increase in frequency reaching its peak of 0.35 at 6400Hz. *Antiaris* BFRC sound absorption increased for frequency of 5000Hz and then decreased, showing the same trend with *F. natalensis* BFRC.

#### **Effect of Composite Surface Roughness**

The effect of surface perturbations on the sound absorption properties showed a marked increase in the sound absorption properties with rough surface. It's observed that the perturbations increased the sound absorption properties of *Antiaris* BFRC having an average sound absorption coefficient of 0.15 between frequency ranges of 2500-5000Hz.

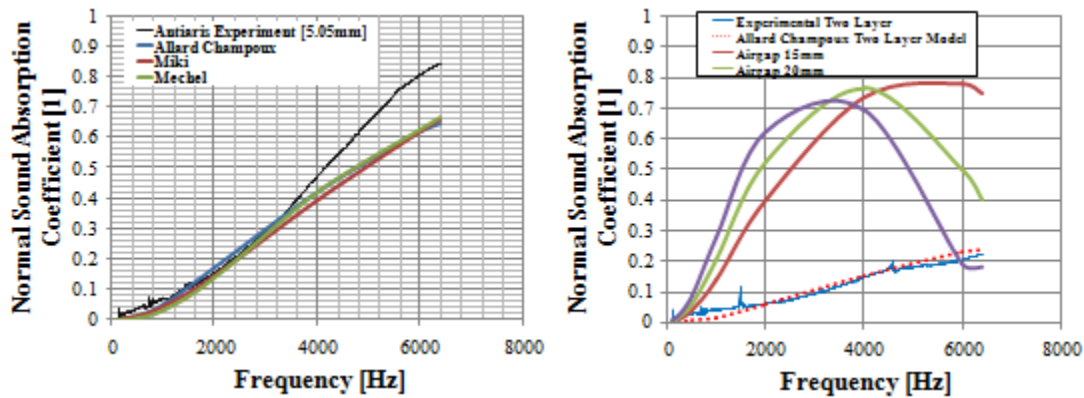
The low performance of the composites with sound absorption is due to the effective packing of the method used for the production of the composites. VARTM is an efficient method of production of composites with fewer voids and with smooth, even packing which showed insignificant sound absorption properties compared to the fabrics which were used to reinforce the epoxy resin.



When a sound wave strikes a porous fiber network like barkcloth; the sound waves cause vibration in the fiber network. The vibration causes minute heat buildup in the fibers due to friction. Therefore, a good absorbing material absorbs the thermal energy of the sound waves and less heat is generated. However, in composites, the compaction of the barkcloth nonwoven felt results in reduced porosity, therefore increasing flow resistivity and reduced vibration of the fiber network therefore a reduced sound absorption coefficient and higher thermal conductivity[50].

### 6.7.3. Modeling of Acoustic Properties

The ability to predict a material behavior using models offers a fast time-saving economical design of structures without prototype production and the rigorous experimental series needed to refine a material. Four other empirical models (Figure 32) were employed with four layers AT fabrics so as to compare the behavior of the predicted models, it was observed that the models are in agreement with experimental data up to the frequency of 3500Hz and thereafter the models' under predicted the sound absorption behavior. The underprediction of the models could be due to the fact that barkcloth is a highly anisotropic material with not uniform fiber distribution network that rendered the underprediction at higher frequencies.



**Figure 32.** (A) Sound Absorption Models of Antiaris toxicaria 4-layer fabrics; (B) Prediction Model of behavior of fabrics with incorporation of an air gap in between.

#### 6.7.3.1. Effect of Air gap on the Acoustic Properties

Since barkcloth is a new material and with prospects of sound absorption applications, another material design parameter was implemented in the model whereby an air gap was incorporated between the two material layers. The Allard – Champoux model was utilized for prediction of the behavior of two layers AT fabrics. It's observed that the model is in good agreement with the experimental data.

Incorporation of an air gap between the two AT fabrics was observed to have positive effects on the sound absorption properties. The larger the distance between the airgap, the higher the absorption at lower frequencies and reduction in the absorption at higher frequencies (Figure 32B).

In the long run, the introduction of a small air gap between the layers would gradually increase the sound absorption of two layers AT fabrics reaching a sound absorption coefficient of 0.78 at frequencies of above 4000Hz.

## **6. Evaluation of Results and new findings**

### **6.1. Morphology And Thermo-Physiological Properties**

The fabric morphology is made up of a dense network of micro-fibers that are naturally bonded and aligned at angles. The inter-fiber bond gives the strength of the load bearing microfibers and damage is initiated through separation of the individual fibers through the failure of the inter-fiber bond and thence fracture. The transverse section of the fabric is characterized by air cavities and microfibers surrounded by plant material.

Barkcloth is majorly composed of Cellulose (69%); based on the Crystallinity Index, barkcloth has highly ordered crystallites (79%) higher than jute (71%) and sisal (71%). Treatment with alkaline solution aided the fiber to matrix adhesion and also helped to dissolve the lignin, wax and other plant impurities.

Enzyme treatment eroded the strength of the fabric, whereas treatment with plasma had a slight effect on the reinforced laminar composites.

The thermal conductivity of barkcloth is comparable to cotton rendering the barkcloth from *F. natalensis*, a comfortable fabric. The lower value of thermal absorptivity of barkcloth, compared to the value of cotton, shows that the fabric has a warm feeling when in contact with the skin. Barkcloth had a higher water vapor permeability compared to cotton and other fabrics meaning its clothing comfort properties are reasonable. In terms of clothing comfort, the fabric fulfills all the requirements for thermal clothing comfort.

### **6.2. Thermal Behavior**

The biocomposites exhibited a high glass transition temperature in the range of 163-185°C depending on the frequency. The thermal analysis illustrated that bark cloth biocomposites are stable until 290°C, a crucial intrinsic temperature that is important if other serial production techniques such as compression moulding are to be used with thermoplastic resins. Synthetic epoxy composites had a low glass transition temperature ranging from 60-70°C.

### **6.3. Mechanical Properties**

For the first time, biodegradable barkcloth reinforced green epoxy biocomposites were developed for the possible application in interior automotive panels. Production of barkcloth composites through the hierarchal architecture of the plies yields varying mechanical properties. Comparative evaluation of the effect layering pattern showed that the ply stacking sequence 90°, 0°, -45°, 45° had one of the best mechanical properties and, therefore, was chosen as the stacking sequence for the investigation of barkcloth reinforced green epoxy biocomposites. The static mechanical properties show that that alkaline treated biocomposites had an average tensile strength 33MPa and modulus of approximately 4GPa. The flexural strength of the composites was 207MPa. The biocomposites exhibited glass transition temperature in the range of 163°C to 185°C depending on the frequency. The developed biocomposites with an average strength of 33MPa higher was higher than the 25MPa threshold strength needed for car instrument or dashboard panels, make barkcloth reinforced green epoxy composites an alternative material for interior automotive panels.

The dynamical mechanical properties showed that the optimum temperature range of application of biocomposites was up to 130°C. Beyond this temperature, the composites enter into a rubbery state and the performance is diminished.

## 6.4. Thermal and Acoustic Properties

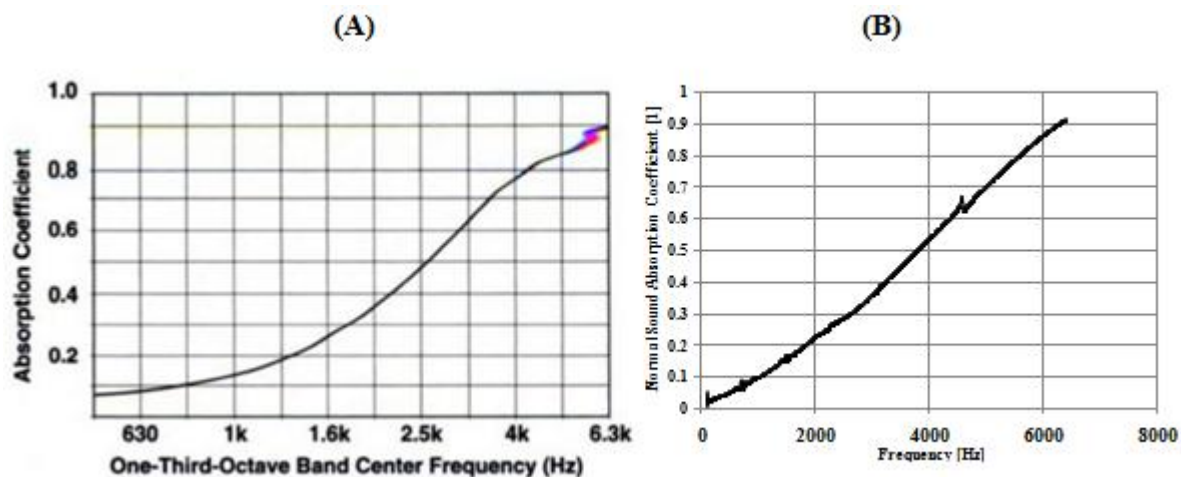
In this investigation, for the first time, barkcloth was presented as a potential sound absorption material. The results show that barkcloth nonwoven fabric had good sound absorption properties and can be used as an alternative replacement for the synthetic commercial fibers which are widely used in the industry. The investigated sound absorption properties showed that *Antiaris toxicaria* barkcloth had higher sound absorption properties at higher frequencies. Increasing the barkcloth fiber layers showed a positive trend towards sound absorption coefficient, therefore giving a prediction of multi-layer products of antiaris barkcloth with potential to provide positive results even at low frequency ranges. Production of composites showed that there's a decrease in the sound absorption properties that's due to the decrease in the porosity and thickness due to the compression of the fabrics under pressure, therefore reducing the vibration of the fibers since they are bonded in the matrix that increases stiffness and thereafter decreasing the overall acoustic properties of the barkcloth reinforced composites. Empirical sound absorption models nearly predicted the sound absorption behavior of barkcloth fabrics, but due to the scope of work, there's need for further modification of existing models and further testing for a perfect model. Nevertheless, the Allard-Champoux model is preferred for predicting the barkcloth sound absorption properties.

## 6.5. Applications And Future Work

### 6.5.1. Automotive Applications

BFRP can be applied in automotive instrument panels. Whereas the layered fabrics can find applications in car headliners.

Headliners are materials installed on the ceilings inside of vehicles and are intended for the purpose of occupant protection through thermal insulation and sound absorption. A good headliner should be able to keep outside heat out of the vehicle and also preserve interior heat for the best comfort of the occupants. Typical car headliners have 200-220g/m<sup>2</sup> [61]. Barkcloth has low thermal conductivity and yet has high sound absorption properties; therefore, its application in car headliners is a novel concept that will serve the triple purpose of decoration, thermal insulation through restriction of heat migration and sound absorption through reduction of noise inside the vehicle.



**Figure 33.** Comparison of Acoustical insulation of commercial Thinsulate™ from 3M Company (A); Barkcloth (*Antiaris toxicaria*) non woven fabric (B)

Thinsulate™ brand acoustic insulation used in vehicles made by the 3M company is shown in Figure 33(A). Sound absorbing properties of Thinsulate acoustical insulation material AU1220, were measured according to ASTM E1050, Dual Microphone Impedance Tube Method measuring Normal Incidence Sound [61]. Barkcloth offers a sustainable green alternative with superior sound insulation properties at higher frequencies which are unpleasant to the human ear.

#### *Gear Lever and Steering Wheel Fabric Cover*



**Figure 34.** Barkcloth fabric and reinforced composite automotive applications.  
(Image courtesy of Barktex)

#### **6.7. Future Work**

This work has endeavored to introduce barkcloth to the scientific community, a material with a lot of potential and yet minimal research had been done. Due to the scope of the task, the following is recommended for follow-up work.

1. Extraction of barkcloth nanofibrils for reinforcement of films.
2. Coatings of barkcloth for applied applications such as electromagnetic shielding.
3. Investigation of the Fatigue behavior of Barkcloth Reinforced Composites.
4. Investigation of Durability, Abrasion resistance and Weathering.

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## 8. List of Papers published by the author

### 8.1. Publications in journals

Note: *Impact factors are based on the 2016 Journal Citation Reports by Thomson Reuters*

1. Rwawiire, S., Tomkova, B., Militky, J., Jabbar, A., & Kale, B. M. (2015). Development of a biocomposite based on green epoxy polymer and natural cellulose fabric (barkcloth) for automotive instrument panel applications. *Composites Part B: Engineering*, 81, 149-157. **(Impact Factor: 3.850)**.
2. Rwawiire, S., Tomkova, B., Militky, J., Wiener, J., Kasedde, A., Jabbar, A., & Kale, B. M. (2016). Short-Term Creep of Barkcloth Reinforced Laminar Epoxy Composites. *In press Composites Part B: Engineering*. **(Impact Factor: 3.850)**.
3. Rwawiire, S., & Tomkova, B. (2015). Thermal, static, and dynamic mechanical properties of barkcloth (ficus brachypoda) laminar epoxy composites. *Polymer Composites* **(Impact Factor: 2.004)**.
4. Rwawiire, S., & Tomkova, B., Militky J., Kale, B. M., Prucha, P. (2015). Effect of layering pattern on the mechanical properties of barkcloth (Ficus natalensis) epoxy composites, *International Journal of Polymer Analysis and Characterization*, 20 (2), 160-171. **(Impact Factor: 1.515)**.
5. Rwawiire, S., & Tomkova, B. (2015). Static and Dynamic mechanical properties of barkcloth epoxy laminar composites, *Journal of Natural Fibers*, 13 (2), 137 - 145 **(Impact Factor: 0.582)**.
6. Rwawiire, S., Tomkova, B., Gliscinska, E., Krucinska, I., Michalak, M., Militky, J., & Jabbar, A. (2015). Investigation Of Sound Absorption Properties Of Bark Cloth Nonwoven Fabric And Composites. *Autex Research Journal*, 15(3), 173-180. **(Impact Factor: 0.460)**.
7. Rwawiire, S., Tomkova, B., Weiner, J., & Militky, J. (2015). Effect of enzyme and plasma treatments of barkcloth from Ficus natalensis: morphology and thermal behavior. *The Journal of The Textile Institute*, 107 (5), 663-671. **(Impact Factor: 0.94)**.
8. Rwawiire, S., & Tomkova, B. (2014). Thermo-physiological and comfort properties of Ugandan barkcloth from Ficus natalensis. *The Journal of The Textile Institute*, 105(6), 648-653 **(Impact Factor: 0.94)**
9. Rwawiire, S., G. W. Luggya and B. Tomkova. 2013. Morphology, Thermal, and Mechanical Characterization of Barkcloth from Ficus natalensis. *ISRN Textiles*, 2013
10. Rwawiire, S., Tomkova, B., Militky, J., Hes, L., Bandu, M. Empirical Modeling of Sound Absorption Properties of Natural Nonwoven Fabric (Antiaris toxicaria Barkcloth). *Materials Science Forum*, 866, 201-20.

### 8.2. Contribution in Conference proceedings

1. Rwawiire, S., & Tomkova, B. 2014. Comparative evaluation of the thermal conductivity of barkcloth epoxy composites. *In: Proceedings of the Fiber Society Conference, Liberec, 21-23 May 2014* (Scopus indexed)
2. Rwawiire, S., & Tomkova, B. 2014. Comparative evaluation of the dynamic mechanical properties of barkcloth epoxy laminar composites. *In: Proceedings of the Workshop for PhD students of the faculty of textile engineering and faculty of mechanical engineering, pp 112-116, ISBN 978-80-7494-100-9*
3. Rwawiire, S., & Tomkova, B. 2015. Barkcloth (Ficus natalensis) reinforced epoxy composites: Effect of enzyme and plasma treatments on morphology, thermal, static and dynamic mechanical properties. *In: 5th International Conference on Innovative Natural Fiber Composites for Industrial Applications, ISBN 978-88-9092-400-2, Rome, 15-16 October 2015*
4. Rwawiire, S., Tomkova, B., Militky, J., Hes, L., Bandu, M. Empirical Modeling of Sound Absorption Properties of Natural Nonwoven Fabric (Antiaris toxicaria Barkcloth). *In: Proceedings of First International Conference on Civil Engineering and Materials Science, 201-205, ISBN 978-3-03835-748-3, Singapore, 1-3 May 2016 [BEST ORAL PAPER AWARD]*



### 8.3. Citations as at 17<sup>th</sup> August 2016.

RWAWIIRE, S., Tomkova, B., Militky, J., Jabbar, A., & Kale, B. M. (2015). Development of a biocomposite based on green epoxy polymer and natural cellulose fabric (barkcloth) for automotive instrument panel applications. *Composites Part B: Engineering*, 81, 149-157.

1. Sanjay, M. R., Arpitha, G. R., Naik, L. L., Gopalakrishna, K., & Yogesha, B. (2016). Applications of Natural Fibers and Its Composites: An Overview. *Natural Resources*, 7(03), 108.
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4. Kumar, R. A Review On Epoxy And Polyester Based Polymer Concrete And Exploration Of Polyfurfuryl Alcohol As Polymer Concrete.
5. Huang, K., Rammohan, A. V., Kureemun, U., Teo, W. S., & Lee, H. P. (2016). Shock wave impact behavior of flax fiber reinforced polymer composites. *Composites Part B: Engineering*, 102, 78-85.
6. Baheti, V., Maqsood, H. S., Wiener, J., & Militky, J. (2016). Reinforcement of ozone pre-treated and enzyme hydrolyzed longer jute micro crystals in poly lactic acid composite films. *Composites Part B: Engineering*, 95, 9-17.

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1. 孙进兴,陈斌,&刘培生.(2016). 轻质多孔陶瓷的制备及吸声性能研究. *无机材料学报*, 31(8), 860-864.

RWAWIIRE, S., & Tomkova, B. (2015). Thermal, static, and dynamic mechanical properties of barkcloth (ficus brachypoda) laminar epoxy composites. *Polymer Composites* (Impact Factor: 2.004).

1. Liu, P., Lam, A., Fan, Z., Tran, T. Q., & Duong, H. M. (2015). Advanced multifunctional properties of aligned carbon nanotube-epoxy thin film composites. *Materials & Design*, 87, 600-605.
2. Hong Wang, Li Yuan, Guozheng Liang, Aijuan Gu, Tough and thermally resistant cyanate ester resin with significantly reduced curing temperature and low dielectric loss based on developing an efficient graphene oxide/Mn ion metal-organic framework hybrid, *RSC Adv.*, 2016, 6, 4, 3290

# Curriculum Vitae



Personal information	
First name / Surname	<b>Samson RWAWIIRE</b>
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Religion	Seventh-Day Adventist
Status and Gender	Married; Male Wife: Etro Lisa Mirembe Kids: Elioenai Sharai Rwahwire
<b>Work experience</b>	
Dates	1/2015 to-date
Occupation or position held	<b>SENIOR LECTURER</b> , Busitema University, Faculty of Engineering, Department of Textile & Ginning Engineering (UGANDA)
Dates	10/2011 to 12/2014
Occupation or position held	<b>LECTURER</b> , Busitema University, Faculty of Engineering, Department of Textile & Ginning Engineering (UGANDA)
Dates	06/2010 to 10/2011
Occupation or position held	<b>ASSISTANT LECTURER</b> , Busitema University, Faculty of Engineering, Department of Textile & Ginning Engineering (UGANDA)
Dates	11/2009 to 06/2010
Occupation or position held	<b>AIRCRAFT ENGINEER</b>
Name and address of employer	Eagle Air Ltd., 11 Portal Avenue, P.O Box 7392, Kampala – Uganda
Dates	8/2008 to 11/2009
Occupation or position held	<b>RESEARCH ASSISTANT</b>
Name and address of employer	Czech Technical University in Prague, Department of Strength and Elasticity of Materials
Dates	30/06/2007 - 30/09/2007
Occupation or position held	<b>COMPOSITE LAMINATOR</b>
Name and address of employer	LA Composite s.r.o., Beranových 65, Prague 9, 199 02, Czech Republic
Type of business or sector	Development and production of composite and sandwich structures
Dates	07/2002 - 10/2002
Occupation or position held	<b>LABORATORY ASSISTANT</b>
Type of business or sector	Government Chemist and Analytical Laboratory- Uganda Ministry of Internal Affairs

<b>Education and training</b>	
<b>Dates</b>	<b>10/2012 – 10/2016</b>
<b>Title/ Qualification</b>	<b>Ph.D. (Material Engineering)</b>
Name and type of organisation	Technical University of Liberec, (TU Liberec) Faculty of Textile Engineering Department of Material Engineering
Research field/ Thesis topic	<i>Mechanical and Thermo-acoustic Characterization of Barkcloth and Its Polymer Reinforced Composites</i>
<b>Dates</b>	<b>09/2003 - 08/2008</b>
<b>Title of qualification awarded</b>	<b>MASTER IN MECHANICAL ENGINEERING (BSc. and MSc. integrated)</b>
Principal subjects / occupational skills covered	Mechanical and Aerospace Engineering
<b>PROFESSIONAL UNIVERSITY CERTIFICATES</b>	<ol style="list-style-type: none"> <li><b>FORENSIC SCIENCE</b> – Nanyang Technological University, Singapore</li> <li><b>SMART TEXTILES</b> – University of Ghent, Belgium</li> <li><b>CIVIL AVIATION PRODUCTS UNDER THE REQUIREMENTS OF EUROPEAN AVIATION SAFETY AGENCY, EASA PART 21</b> – Brno University of Technology, Czech Republic.</li> <li><b>DESIGN, ANALYSIS AND MECHANICS OF COMPOSITE STRUCTURES</b> – Czech Technical University, Czech Republic</li> </ol>
<b>AWARDS &amp; ACHIEVEMENTS</b>	<ol style="list-style-type: none"> <li>Uganda-Czech governments Scholarship</li> <li>Deans Merit Scholarship for excellent results in 5th year of integrated Master Studies</li> <li>First Prize Poster Award at 2008 PEGASUS-AIAA Student Conference (Award by Airbus).</li> <li>AAU Small Grants for Theses and Dissertations.</li> <li>Best Paper Presentation Award, ICEMS, 2016, Singapore.</li> </ol>
<b>JOURNAL REVIEWER</b>	<ol style="list-style-type: none"> <li><b>Textile Research Journal</b> (2016 Impact Factor 1.299)</li> </ol>

## **Brief description of the current expertise, research and scientific activities**

### **Studies**

#### **Doctoral Studies**

Full-time student at the Faculty of Textile Engineering, Department of Material Engineering  
Specialization: Material Engineering

#### **List of Exams**

[1]. Heat and Mass Transfer in Porous Media  
[2]. Structure and Properties of Fibers  
[3]. Textile Metrology  
[4]. Mathematical Statistics and Data Analysis  
[5]. Experimental Technique of the Textile

#### **State Doctoral Examination**

Passed

#### **Research Projects**

- Participant in SGS Project
- Contributed book chapter for the Department's Material Science book.

## Record of the state doctoral exam



### ZÁPIS O VYKONÁNÍ STÁTNÍ DOKTORSKÉ ZKOUŠKY (SDZ)

*Jméno a příjmení doktoranda:* **Ing. Samson Rwawiire**  
*Datum narození:* **30. 1. 1982**  
*Doktorský studijní program:* **Textilní inženýrství**  
*Studijní obor:* **Textile Technics and Material Engineering**  
*Termín konání SDZ:* **18. 11. 2015**

prospěl

~~neprospěl~~

*Komise pro SDZ:*

*Podpis*

Předseda:	prof. Ing. Jiří Militký, CSc.	
Místopředseda:	prof. RNDr. Oldřich Jirsák, CSc.	
Členové:	prof. Ing. Jakub Wiener, Ph.D.	
	prof. Ing. Michal Šejnoha, Ph.D., DSc.	
	doc. Ing. Dora Kroisová, Ph.D.	
	Ing. Ondřej Novák, Ph.D.	

V Liberci dne 18. 11. 2015

*O průběhu SDZ je veden protokol.*



## **Recommendation of the supervisor**

## Opponents' reviews **Assessment of PhD Thesis**

<b>Aspirant:</b>	<b>Ing. Samson Rwawiire</b>
<b>Thesis title:</b>	<b>Mechanical and Thermo-Acoustic Characterization of Barkcloth and Its Polymer Reinforced Composites</b>
<b>Specialization:</b>	<b>Textile Technics and Materials Engineering</b>
<b>Supervisor:</b>	<b>Ing. Blanka Tomková, Ph.D.</b>
<b>Reviewer:</b>	<b>doc. Ing. Antonín Potěšil, CSc.</b>

<b>Topicality of the thesis</b>					
Comment:					
The topic of presented dissertation is undoubtedly up-to-date and it respects innovative current and future trends, which prefer development and manufacturing of new products using biodegradable, environmental friendly materials. These materials do not ravage irreplaceable natural resources in all areas of industrial applications.					
excellent <sup>1</sup>	above standard	<b>X</b>	standard	substandard	weak

<sup>1</sup> Mark selected with a cross

<b>Meet the objectives of the thesis</b>					
Comment:					
Although presented study mostly combines form of the search, the description and explanatory, when describing basic approaches to the preparation and testing of bio-composites made from various natural fibers and materials, especially from the tree bark, after studying the work as a whole state it is possible to say, that defined aims and objectives of PhD student were fulfilled.					
excellent	above standard	standard	<b>X</b>	substandard	weak

<b>Methods and solutions</b>					
Comment:					
Analyses of prepared bio-composite structures were performed by standard test methods, procedures and measuring equipment (SEM, FTIR, DSC, XRD, DMA, thermal, acoustic and mechanical tests etc.). It is not obvious from presented work, whether student suggested or developed a new method or his own innovative experimental procedure.					
excellent	above standard	standard	<b>X</b>	substandard	weak



### Results of the Thesis - specific benefits of the student

Comments:

The main contribution of the work is comparison of the properties of several types of bio-composites and their structure, both chemical and mechanical, and thermo-acoustic. Valuable information are particularly material data of bio-composites filled with fibrous reinforcement of tree bark. However, it is appropriate to say that work misses deeper theoretical fundamentals of the mechanics of composite materials, and moreover the reported results of experimental measurements do not always match otherwise usual statistical data processing.

excellent    above standard    standard    substandard **X**    weak

### Significance for practice and for the development of the scientific branch

Comments:

The work is a good starting point for further research and development in the use of bio-composites in various industrial applications. I recommend to focus future work deeper into the theoretical background of physical properties of composite materials both with respect to subsequent processing technology in the manufacture of products, and with respect to their industrial application in a specific sense.

excellent    above standard **X**    standard    substandard    weak

### Formal layout of the Thesis and its language level

Comments:

Submitted Thesis has a logical structure, the text is clear, Czech mutation of the abstract has a minor editing errors, which do not spoil the overall impression of the work.

excellent    above standard    standard **X**    substandard    weak

### Comments and questions

1. The properties of prepared bio-composites are measured only in room and in elevated temperatures. The application in automotive and aviation industry requires knowledge of properties in the range from -60 to 90°C including coefficient of linear thermal expansion (CLTE). Did you run the experiments in subzero temperatures?
2. Properties of polymer matrix composites depend not only on temperature but also on load speed, e.g. impact, vibrations etc. What kind of experimental method would you suggest for such measurements?
3. What method was used to set volume fraction of fibrous reinforcement and of matrix in prepared bio-composites? Explain the relation between mass and volume fraction of reinforcement and matrix in composite structure.
4. For any predictive CAE simulations it is necessary to describe composites as anisotropic, respectively orthotropic material continuum. What relations are in such cases used to describe relationship between stress and strain? Give examples.





**Final evaluation of the Thesis**

Based on the above review I recommend submitted thesis for defense in front of the scientific committee for the defense of doctoral thesis.

<b>I recommend after a successful defense of the dissertation grant Ph.D.<sup>2</sup></b>	<b>yes</b>	<b>#0</b>
---	------------	-----------

<sup>2</sup> Delete where applicable

*Place and Date:* In Liberec 20.06.2016

*Signature:* Antonín Potěšil



## Oponentní posudek

Doktorské disertační práce Ing. Samsona Rwawiire

Mechanical and Thermo-Acoustic Characterisation of Barkcloth and its Polymer Reinforced Composites

V Liberci dne 12. 7. 2016.

Vypracovala: prof. Ing. Bohdana Marvalová, CSc.

---

1) K posouzení předložená disertační práce Ing. Samsona Rwawiire byla vypracována na Fakultě textilní TU v Liberci ve studijním oboru Textile Technics and Materials Engineering. Práce je zaměřena na experimentální prozkoumání chování přírodního textilního materiálu získávaného z kůry afrických stromů nazývaného barkcloth, na stanovení jeho základních vlastností a na nastin možností jeho technického využití zejména jako výztuže kompozitů. Práce má 106 stran textu + 17 stran úvodních včetně výčtu 15 uveřejněných článků disertanta. Práce je rozdělena do osmi kapitol a má 173 položek v seznamu referencí. Práce je psána dobrou angličtinou.

Disertant uvádí cíl práce v podkapitole 1.3. jako :

*The overall aim of this study was the investigation of the mechanical and thermo-acoustic properties of barkcloth and its polymer reinforced composites for automotive applications. The specific objectives are investigation of:*

- 1. Morphology, Chemical and Thermo-physiological properties of Barkcloth.*
- 2. Mechanical and Thermal Analysis of Barkcloth Laminar Epoxy Polymer Composites.*
- 3. Thermo-acoustic Properties and Sound Absorption Models of barkcloth.*

### 2) Aktuálnost tématu a adekvátnost cílů práce

Výzkum vlastností a možností využití nových materiálů označovaných jako zelené kompozity je velmi aktuální téma. Disertant se zaměřil na přírodní materiál, který je v jeho vlasti dostupný a jehož možnosti se rozhodl prozkoumat a prezentovat. Naplnění velmi rozsáhlého cíle práce, který si disertant stanovil, vyžadovalo velký objem časově náročné experimentální práce. Vedlo to však v některých i klíčových otázkách k tomu, že experimenty nejsou jasně popsány (použité metody, materiál a počet vzorků apod.) a výsledky experimentů nejsou vždy dostatečně přehledně zpracovány, vyhodnoceny a analyzovány (většinou jsou prezentovány jen grafické záznamy měření se stručným popisem). Přesto je třeba ocenit velké nasazení, se kterým se disertant tohoto náročného a rozsáhlého úkolu zhostil.

### 3) Vyjádření k obsahu práce, postupu řešení, použitým metodám a výsledkům práce

Práce je rozdělena na osm kapitol.

- První kapitola obsahuje úvod a cíle práce.
- Ve druhé kapitole disertant uvádí rešerši vlastností přírodních vláken, jejich složení a přehled jejich chemických a termo-mechanických vlastností a jejich využití v zelených kompozitech. Uvádí zde rovněž přehled jednoduchých modelů mechanického chování jednosměrných kompozitů a přibližného výpočtu jejich tepelné vodivosti. Část této kapitoly je věnována přehledu problému stanovení akustických vlastností přírodních materiálů a je zde uveden přehled empirických modelů používaných ke stanovení koeficientu zvukové pohltivosti vláknitých a porézních materiálů.

- Třetí kapitola je věnována popisu získávání přírodní netkané textilie barkcloth, experimentálnímu stanovení její mikrostruktury (př. směr vláken) ze snímků SEM metodou zpracování obrazu. Disertant zde v tabulce 3.2 uvádí celou řadu hodnot fyzikálních veličin pro barkcloth, které pravděpodobně sám naměřil. Je zde rovněž zkoumáno chování zelené pryskyřice G520, později použité k výrobě kompozitu vyztuženého vrstvami barkcloth.
- Čtvrtá kapitola je věnována procesu zhotovení kompozitních vzorků s různým uspořádáním vrstev barkcloth a dvěma typy epoxipryskyřic a uvádí se zde i zpracování barkcloth pomocí plasmu, enzymů a alkálií.
- Pátá kapitola je nazvaná Experimental a je v ní uveden přehled použitých přístrojů a metod, pomocí kterých disertant zkoumal morfologii, složení, mechanické vlastnosti, tepelné a akustické chování barkcloth a kompozitu s barkcloth výztuží. Je zde výčet některých výsledků.
- Šestá kapitola je nazvána Results and Discussion. Jsou zde srovnána měření morfologie, X-difrakce a FTIR na vzorcích bez chemického zpracování a chemicky zpracovaných, měření termální stability gravimetrickou metodou, dále výsledky tahových zkoušek a DMA kompozitních vzorků. Chování za zvýšených teplot a degradace je zkoumána pomocí DSC. Jsou zde výsledky akustických měření.
- V sedmé kapitole autor shrnuje poznatky v předchozích částech a v osmé kapitole je výhled do budoucna. Následují reference a 4 strany příloh s drobnými doplňky.

Z tohoto přehledu je zřejmé, že disertant pojal svou práci velmi široce a snažil se vyřešit velké množství otázek a získat co nejvíce údajů o předmětu svého zkoumání. Jeho záměr se naštěstí setkal s velmi dobrým a moderním vybavením Katedry materiálového inženýrství FT TUL, které mu umožnilo provést velkou řadu potřebných speciálních experimentů a shromáždit velké množství dat. Bohužel jej to vedlo k poněkud povrchnějšímu přístupu k jejich interpretaci a neumožnilo to vytěžit do hloubky příležitosti, které by se mu otevřely, kdyby téma své práce zúžil - včetně příležitosti získat hlubší vhled do důležitých problémů termomechaniky kompozitů.

#### 4) Vyjádření k formální úpravě práce.

Zásadní chybou, která není pouze formální, je roztržení stěžejní experimentální části práce, do dvou kapitol (5 a 6). První z nich – Kap. 5 - obsahuje spíše beletristický výčet jednotlivých použitých experimentálních metod bez podrobnějších údajů o průběhu experimentů, velikosti a počtu vzorků typu jejich materiálu a způsobu odběru vzorků z nehomogenního přírodního materiálu. V Kap. 6 pak jsou naskládány výsledky měření mnohdy bez podrobnějších komentářů.

Příklad: v Kap. 6 na str. 84 je *Table 6.7 Airflow resistivity of samples*, není však jasné, jakých vzorků a disertant výsledky v tabulce nikde nekomentuje. Po delším zkoumání nalezneme na konci Kap. 5 na str. 53 zmínku o tom, že bylo provedeno měření odporu proti proudění vzduchu, opět však není uvedeno na jakých vzorcích a jakého materiálu.

Podobně na str. 48 v Kap. 5 autor uvádí, že prováděl zkoušky pevnosti protržením (*bursting strength*), ale o jejich výsledku není nikde zmínka.

Práce má spíše průměrnou formální úroveň. Rozdělení jednotlivých kapitol na přemíru podkapitol je nefunkční a ztěžuje čtenáři orientaci (kupř. podkapitola 2.3.3. *Mechanical Properties* obsahuje pouze jeden a půl řádku textu). Použití víceúrovňové číslování obrázků je nevhodné a matoucí a jeho výsledkem je například to, že po obrázku 2.13 následuje obrázek 2.3. "Flowchart" na str. 6 zaujme svou barevností, ale má minimální informační hodnotu a patří spíše do sféry marketingu.

Rovnice, obrázky i text obsahují některé nepřesnosti, které nelze přehlédnout:

- str. XVI: *Volume fraction of the composite, Angular Frequency [Rad], Temperature between human skin and the textile, Bending Stiffness [N]* (použita na str. 31)
- str. 23: vztah (8) není korektní
- str. 24: odvolání na Figure 2.31, která neexistuje, nejspíš má být (Figure 2.41), obdobně na str. 25 dole má být (Figure 2.43) a na str. 26 (Figure 2.42)
- str. 30: ve vztahu (10) je navíc závorka
- str. 39: autor uvádí "*barkcloth thermal absorptivity of 81.4 Ws<sup>1/2</sup>/m<sup>2</sup>K*", ale na str. 37 v Table 3.2 autor uvádí hodnotu 0,197 Ws<sup>1/2</sup>/m<sup>2</sup>K
- str. 44: (Figure 4.1) neexistuje
- str. 46: nesprávné odvolání na čísla obrázků (téměř pravidlem)
- str. 53: odvolání na Table 2.1, která sice existuje (str. 12), ale obsahuje něco naprosto jiného
- str. 80: nesprávné číslo obrázku – místo 6.38 má být 6.37
- str. 83: nesprávné číslo obrázku – místo 6.41 má být 6.40
- str. 93: reference [1] „No Title“.

## 5) Dotazy

1. V podkapitole 4.2.3. *Types of fibers and the reason for the fabric ply arrangement* uvádí disertant na str. 44 čtyři různé sekvence vrstvení výztuže ze 4 vrstev BFRP I-IV (-45/45/0/90), (45/-45/90/0), (0/90/45/-45) a (90/0/-45/45), které použil pro výrobu kompozitní vzorků. Disertant uvádí, že tímto vrstvením docílil kvazi-izotropního chování laminátu. Mohl by disertant vysvětlit, z jakých zásad při návrhu „*stacking sequence*“ vycházel?
2. Na základě jakých předpokladů a dat stanovil disertant předpovědi koeficientu zvukové pohltivosti v grafech na následujících obrázcích, mohl by tyto závislosti vysvětlit podrobněji?  
*Figure 6.41. Sound Absorption Models of Antiaris toxicaria 4-layer fabric*  
*Figure 6.42. Prediction Model of behavior of fabrics with incorporation of an air gap in between.*

## 6) Závěrečné hodnocení.

Disertant ve své práci uvádí řadu původních výsledků experimentálních zkoušek a široce zmapoval vlastnosti a možnosti uplatnění vybraného přírodního textilního materiálu. Disertant uvádí 15 zveřejněných publikací, jichž je prvním autorem. Publikace se týkají tématu disertační práce a řada z nich je uveřejněna v renomovaných zahraničních časopisech. Výsledky jeho výzkumu tedy byly průběžně publikovány. Domnívám se, že předložená disertační práce Ing. Samsona Rwawiire splňuje kritéria daná v zákoně č. 111/1998 Sb. Par. 47, odst. (4). Prokázal v ní schopnost samostatné a systematické vědecké práce. Doporučuji práci k obhajobě. Doporučuji, aby v případě uspokojivých odpovědí na dotazy oponentů a úspěšné obhajoby byl Ing. Samsonu Rwawiire přiznán titul Ph.D.



prof. Ing. Bohdana Marvalová, CSc.

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*Our Ref.: MIE/TX-2.22/LYM/lym*

*Date: 02-08-2016*

**Ing. Jana Drašarová**

Dean, Faculty of Textile Engineering

Technical University of Liberec

**Check Republic**

Dear Ing. Drašarová

**Comments on the Doctoral (PhD) Thesis Submitted by Ing, Samson Rwawiire**

The Doctoral Thesis mentioned above was received on 23<sup>rd</sup> June, 2016 through email from **Ing. Jana Drašarová**, Dean, Faculty of Textile Engineering, Technical University of Liberec Check Republic. The thesis has been submitted to me for review and the following arrangement is how the comments have been organised.

The review has been organised in five parts as follows: Theoretical Background, Objectives, Research Methodology, Results and Discussions, Conclusions and General Presentation.

**Theoretical Background Section**

The candidate has provided most of the necessary literature to back up the experimental and research findings including the basis for the formulation of the objectives. About 173 references have been cited in the entire thesis of which 141 have been dedicated to the Literature Section. The literature review is quite comprehensive and legible. However, the following are some of the specific comments which might help to strengthen the quality of the thesis. More specific comments appear in the text (Thesis), which will be submitted to the candidate during the defence session.

The way the terms 'green' epoxy (page 7 and 40) have been implied should be scientifically justified because epoxy is a thermoset polymer thus cannot qualify to be a green material. Furthermore, it does not melt rather it decomposes like all thermoset polymers.

On the first page of the thesis the reader is made to harbour the notion that biodegradation is always an advantage (page 8), which is definitely misleading.

It is worth mentioning that the candidate has done an excellent work particularly involving backcloth which is a renewable resource abundantly available in Uganda. However, I don't think studies on 'sound proof' is of any practical value in automotives, with the exception perhaps of the racing cars (Formula 1) sector.

The author is advised to include discussions about the fundamental role of the hydroxyl groups in the manufacture of polymer matrix composites.

Table 2.2 page 14 could be enriched by adding a column for the microfibril angle ( $\theta$ ) of the respective plant fibres. There is a strong correlation between the micro-fibril angle and the Young's Modulus (stiffness) of the plant fibres. The fibre stiffness is influenced by the spiral angle of the crystalline fibrils as well as the concentration of non-crystalline materials.

Section 2.1.2.1 could add more discussions about the variation on the tensile strength and Stiffness of the leaf and bast fibres.

Glass fibre is not a synthetic material. Please revisit references (page 17, 3<sup>rd</sup> paragraph from top of page) to confirm this claim.

The candidate should state whether the type of polyester used in this study was a thermoset or thermoplastic one. My understanding is that it was the unsaturated polyester which is a thermoset polymer widely used for composite manufacture and state its advantage compared with other thermoset resins such as phenolics and/or expoxies.

It is hard to agree that the Isotactic Polypropylene (PP) melts at 176 °C !!! Provide proof of reference to support such high temperature. Has Ref. 66 indicated under what circumstances the PP used melted at 176 °C!

Fig. 3.11 B, C and D, 3.12 A, 3.14 A, B and C, and 3.15 shows SEM spectrograph of the longitudinal view of the Backcloth fibre bundles with different magnification. The same applies to Fig. 6.1, Fig 6.11 and Fig. 6.12 page 54, 55 and 56 respectively while Fig. 3.11 A and 3.12 B also shows SEM spectrograph with differing magnifications. The candidate should note that only one magnification should be used throughout the thesis/text for comparison purposes.

Please show 1<sup>st</sup> and 2<sup>nd</sup> run of the DSC to avoid the shadowing of impurities and small molecular weight components which might be available in the mixture. Also, it is important to provide the thermograms of the neat resin and composite on the same scale for comparison purposes

Page 45 and 46 the composites cannot be compared because the hardener ratio used is different besides, they were produced using different manufacturing techniques

### **Objectives**

The Specific objectives are measurable and they have been adequately addressed

### **Research Methodology Section**

It is interesting to note that the candidate has used quite a number of analytical testing techniques which arguably should provide complementing results. This indeed is expected to strengthen the discussion and conclusion of the work done!

The candidate should provide reasons why a concentration of 5% NaOH (caustic soda) was used to treat the Backcloth (page 49). Provide reference to support that the average human skin temperature is 32 °C.

### **Results and Discussion Section**

It should be noted that the alkali used in this study does not only clean the surface of the plant fibres, it also modifies the fine structure. Hence, changes on some characteristics

such as one mechanical properties of the backcloth should be expected following caustic soda treatment.

The IR spectra in this section should be reorganised in such way that the wavenumber ( $\text{cm}^{-1}$ ) increases from left to right. Also please note that there should be clear distinction between the Absorbance and Transmittance spectra!!! Also make sure that the labelling of the axes of the IR spectra is consistent throughout the thesis.

The candidate should have concentrated discussion only on the spectra which are of interest to the major reactions in this study such as the peak for hydroxyl groups, the peak for the carbonyl group or any other and if necessary one or two peaks in the figure print region

On the thermograms the inclusion of the DTG spectra would have helped to elucidate which temperature is responsible for the different degradation parameters. Furthermore, the heating range should go up to around  $800\text{ }^{\circ}\text{C}$  because that would capture the formation of the tar materials, which could help to differentiate between the thermal behaviour of polymeric and cellulosic substances.

Efforts should have been directed to study the mechanical properties of the single fibre strands. This could have been achieved by unravelling the fibre strands from the backcloth fabric mesh.

The candidate should have appreciated the contribution of the microfibrils by studying the influence of the microfibril angle to the mechanical properties of the fibre strands and use that to postulate the mechanical properties of the composites.

The value for the flexural strength of the so called 'green' epoxy biocomposites is almost 7 times the value of its tensile strength!! This needs convincing explanation.

Fig. 6.26 shows the SEM spectrograph of the Backcloth-Epoxy composite. What is referred to as matrix cracking is actually the fibre-matrix debonding, indicating poor interface hence would require explanation what caused that to happen.



The candidate should consult the textile books to understand the meaning of the term fleece. It should not be loosely explained as this will mislead first time students of textiles and materials science.

The discussion in section 6.5.1 has to be revisited because basic principles of thermal behaviour of polymers have not been properly grasped/understood, Epoxy is a thermoset polymer therefore it cannot exhibit melting temperature. What the candidate might have observed is the decomposition temperature.

The candidate is advised to show evidence how the onset degradation temperature is determined on the thermograms

Could Backcloth exhibit both isotropic and anisotropic characteristics typical of woody materials? If yes or no explain under what circumstances either of the two would be observed.

### **Conclusion Section**

The conclusion that cotton exhibit higher values of thermal absorptivity need to be verified experimentally otherwise a reference to support the claim should be provided.

Because epoxy is a thermoset polymer it, therefore, cannot impart biodegradation on the composite-based epoxy polymer. The statement in section 7.3, first paragraph, should be revisited.

The stiffness of 4GPa for the composite is too low for a reinforced polymer because it is nearly that of the polymer itself. Discuss what happened to the backcloth cell wall!!

The thesis has not shown any work on the thermal conductivity of cotton hence it should not be part of the conclusion.

A concluding remark on the reason to the use of several analytical tools some of which complement each other should have been provided. For instance, the stiffness obtained when the material is tested under tension is confirmed or complemented by the DMTA method

### **General Presentation of the Thesis**

The overall impression resolved from this thesis is that it has been expertly prepared beside the fact that advanced analytical methods have been adequately presented. It is quite impressive that the PhD has produced 14 Journal Papers and 5 conference papers. However, the candidate should remove the following articles from the list of references: 26, 127, 130, 159, these articles were published during the time of the PhD studentship and they are therefore under examination. Please include them on the list of original publications on page roman x. There chronological arrangement of the references in the text should be similar to the list at end of the thesis. For instance reference 141 does not appear in the text please check and rectify.

### **Concluding Comment:**

The candidate was well prepared for the study carried out and this is reflected on the number of references cited and the good command of the analytical techniques used and more importantly he has posed a challenge to oncoming fellow researchers evidenced by the number of journal publication alongside the production of the Thesis.

I therefore strongly recommend that the candidate **PASS** subject to addressing the minor correction stated in this submission and those indicated in the text of the Thesis, which I humbly submit together with this report.

This Report has been submitted by:



Prof. LY Mwaikambo  
**Associate Professor in Textile and Materials Science**