

Characterization of Selected Natural Fibres As Reinforcement Materials for Polymer Matrix Composites

I. C. Ezema^{(1)*}, A.R.R. Menon⁽²⁾, S.O Edelugo⁽³⁾, V.B. Manilal⁽²⁾, M. Brahmakumar⁽²⁾, A.D Omah⁽¹⁾

¹ Department of Metallurgical & Materials Engineering, University of Nigeria, Nsukka

² National Institute for Interdisciplinary Science and Technology, (NIIST) CSIR Trivandrum, India

³ Department of Mechanical Engineering, University of Nigeria, Nsukka

ABSTRACT

In this paper the stress-strain behavior of banana stem (trunk), banana bunch, sisals and *Ulera lobeta* (*ebari*) fibres were studied at micro level of a single fibre strand of diameter between 0.1-0.3 mm. The study showed that treated *ebari* fibre has the best ultimate tensile strength of 116.8 MPa and elastic modulus of 39.56 GPa, whereas that of the treated banana bunch fibre was the lowest with ultimate tensile strength of 45.5 MPa and elastic modulus of 1.26 GPa. The water absorption test carried out on these natural fibre mats bonded with raw natural rubber latex indicated higher absorption capacity for the (NaOH/Na₂SO₃) surface treated banana bunch fibre than the untreated, while other fibre also treated had a reduction in their water uptake. The Thermo-Gravimetric Analysis (TGA) and Differential Thermal Analysis (DTA) showed a lower % weight loss at higher degradation temperatures of the fibres after surface treatment. The texture surface of the fibres was characterized using Scanning Electron Microscope (SEM).

Keyword: Natural Fibres, Mechanical properties, TGA/DTA, Electron microscopy, Water absorption.

*Corresponding Author: e-mail: ikeezema@gmail.com

Introduction

Polymeric composites are being used extensively in various applications from domestic household items to industrial automobile and aerospace applications due to their high strength to low weight ratios. Recently, extensive research work has been carried out on the use of natural fibres as replacement for synthetic fibres in polymer reinforced composites in some applications mainly because of their biodegradability advantages [1-8]

Natural fibres are available in abundance in many parts of the world, mainly as agro-wastes and

ornamental plants. These include banana stem, banana bunch, sisals, jute, coir, pineapple leaves, *ebari*, kenaf, bamboo, okra, bagasse. The properties of these fibres differ slightly and depend on the part of the plant from where they are obtained, the local climatic conditions and the nature of retting processes [9]. Measurement of the tensile strength of a single strand of the natural fibres, thus, becomes crucial for the comparison between different kinds of it for the prediction of the mechanical properties, if it is to be used for composite manufacture.

It is reported that Single Fibre Tensile Strength Test (SFTST) is a widely applied method for measurement of the tensile properties of fibre [10].. This method provides acceptable strength and modulus for synthetic fibres, but it fails to provide accurate result with low standard deviation for natural fibres. In SFTST, the fibre diameter/cross-section area is assumed to be perfectly round which is true for synthetic fibres but not true for natural fibres because natural fibres always exist in bundles of tiny fibres which results in an irregular shape depending on the number of these tiny elementary fibres. For simplicity, however, this paper assumes that the natural fibre are round and free from inherent artificial and natural flaw and has studied the

single fibre tensile properties of banana stem, banana bunch, *ebari* and sisals fibres using an average result of more than 10 samples in each category.

Material and Methods

Materials

The Environmental/Process Division of NIIST, Trivandrum, India, supplied the banana stem fibre (BSF) and banana bunch fibre (BBF). The sisals and *ebari* fibres were harvested, processed by retting in a flowing stream in Ngbakwu, Ozi Edem, Nsukka, Nigeria. NaOH crystals by Excel (AR) and Na_2SO_3 by Merck, Mumbai India were used. Natural rubber latex and fevicol adhesives were obtained from Polymer Science Lab. NIIST, Trivandrum, India.



Methods

Fibre surface treatments

Four natural fibres from banana stem ((BSF), banana bunch (BBF), sisal (SIS) and ebari plant (EPF) were selected and surface treated with a mixture of 4 % NaOH and 2 % Na₂SO₃ solutions for 24 h at room temperature. They were removed, sun-dried for 8 h and in an oven at 60 °C for 1 h to remove moisture.

Single fibre tensile test

The fibres were separated into single strand by hand. and fibres of about 0.1-0.3 mm diameter were selected using a hand-held optical microscope and a scale rule. The two ends of the fibre were attached to a piece of thick board paper measuring 25 mm x 100 mm with provision of 50 mm gauge length properly secured with fevicol adhesive. The single fibre strand tensile test was done using a Hounsfield Tinius universal testing machine H5KS model according to ASTM D3379-75 [11] at 50 mm/min load speed and an approach speed of 1 mm/min and within a stress range of 0- 500 MPa.

Water absorption test

In this test, bundles of the fibres were glued together using natural rubber latex, dried under sun for 24 h and in an oven at 60 °C for 1 h. The dried samples were cut to dimensions and weighed using Afcoset FX400 electronic balance, and the weight (W_o) was recorded. Thereafter, the samples were immersed in water for 24 h, removed, dried with towel and then allowed to

dry in an open air for 30 min at room temperature and the weight (W_t) recorded. The percentage of water absorption capacity M_t was calculated according to ASTM D570-98 [12] as follows:

$$M_t = \frac{W_t - W_o}{W_o} \times 100 \quad \text{----- (1)}$$

Where W_t is the wet weight and W_o is the dry weight of the samples

Thermal analysis

The TGA and DTA are generally described as derivatives of thermogravimetric curves which indicates the thermal degradation range (weight loss) of the fibre or resin with increasing temperatures [13-14]. The thermal characterization of the fibres was carried out using a TA instruments Q600-SA for simultaneous TGA/DTA at a heating rate of 10°C/min and in a temperature range of 50-800°C. The TGA and DTA were used to investigate the thermal decomposition of the fibres under intense nitrogen environment.

Surface adhesion study

To study the surface adhesion of the fibres, two different bonding agents- natural rubber latex and polyvinyl acetate (fevicol) were used to mat the fibres. The samples were cut to size and gold-coated for 5 min in a JOEL-JFC-1200 fine coater. The micrograph of the surface morphology was observed using scanning electron microscope JOEL-JSM-6100 at a voltage of 15 kV at X50 of 500 µm magnifications.

Results and Discussions

Results

Single fibre tensile test

The Young's modulus, ultimate tensile strength, maximum elongation, stress at break and the

average diameter and elongation at break values of the untreated and treated banana stem., banana bunch, sisals, and *ebari* fibres denoted by BSF, BSFT, BBF, BBFT, SIST, SIS, EPF, EPFT respectively are presented in Table I. The data were computed from the values of 10 specimens in each category and their average recorded.

Table I :Tensile test result of the fibres *

Sample code	Treatment	Failure stress σ_f (MPa)	Elongation at max. Stress ϵ_{max} (%)	Average fibre diameter Φ (mm)	Elongation at break ϵ_f (%)
BSF	Untreated	88.00	2.63	0.3	2.63
BSFT	Treated	103.58	1.93	0.2	1.98
BBF	Untreated	136.76	8.11	0.3	8.11
BBFT	Treated	109.09	4.55	0.3	8.47
SIS	Untreated	144.39	5.99	0.3	6.07
SIST	Treated	74.70	3.70	0.3	3.70
EPF	Untreated	98.34	4.20	0.1	4.26
EPFT	Treated	116.76	2.58	0.1	2.58

Table values are mean of N=10

Fig 1 shows the tensile properties and the Young's Modulus in a bar chart for the various samples. The heterogeneous nature of natural fibres and the inherent errors associated with manually measured diameters of the fibres using only the optical microscope and a graduated steel rule are typical problems; however, the results were computed from 10 different readings and are believed to be fair enough to establish the true reflection of response of the fibres.

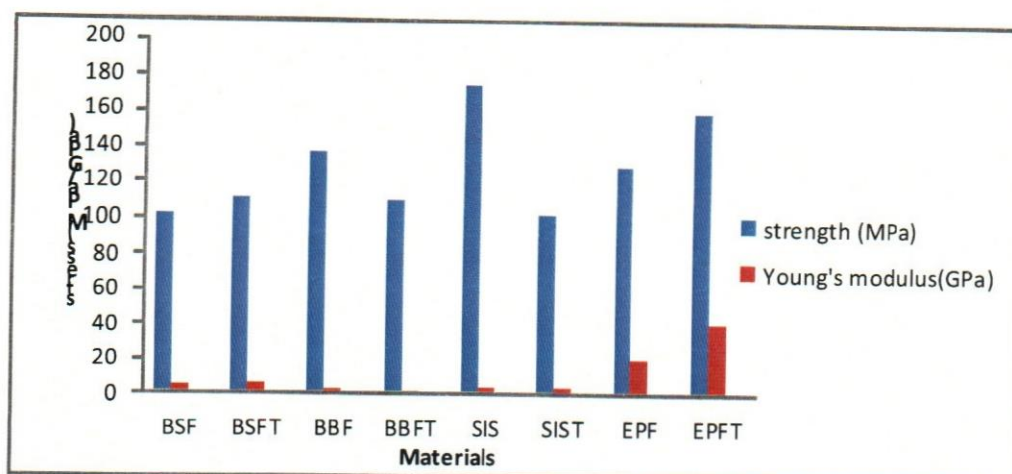


Fig 1: Ultimate tensile strength and elastic modulus of treated and untreated banana stem, banana bunch, sisals and ebari fibres

Water absorption test

The result of the water absorption test of the samples after 24 h of immersion are shown in Fig 2. It can be seen that surface treatment of all the fibres reduced their water absorption capacity. From literatures[15], it is well known that the amorphous part of natural fibres absorb dyes and water easily. The surface treatment of natural

fibres therefore attacks and reduces the amorphous part of the fibre consisting of the hemi-cellulose and the lignin. This means that the treatment of the fibres eliminated the hydroxyl groups and reduces its tendency to absorb water, which will increase their adhesion with the hydrophobic matrix.

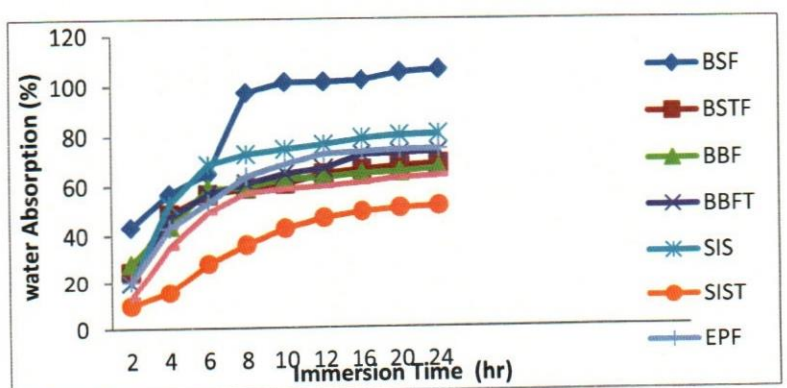


Fig 2: Effect of immersion time on the water absorption capacity of the natural fibres

TGA/DTA analysis

The TGA/ DTA of treated and untreated banana stem fibres as generated and plotted automatically by the equipment software are presented in Fig 3a & 3b respectively. From the

graphs, the major weight changes can be directly read with the sharp changes in their slope as the pointers.

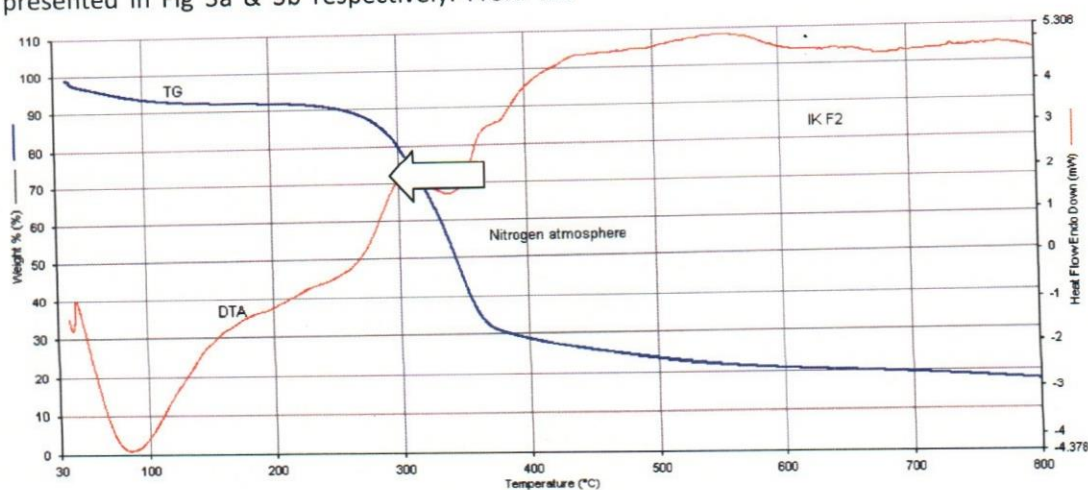


Fig 3a: TGA/DTA of treated banana stem fibres

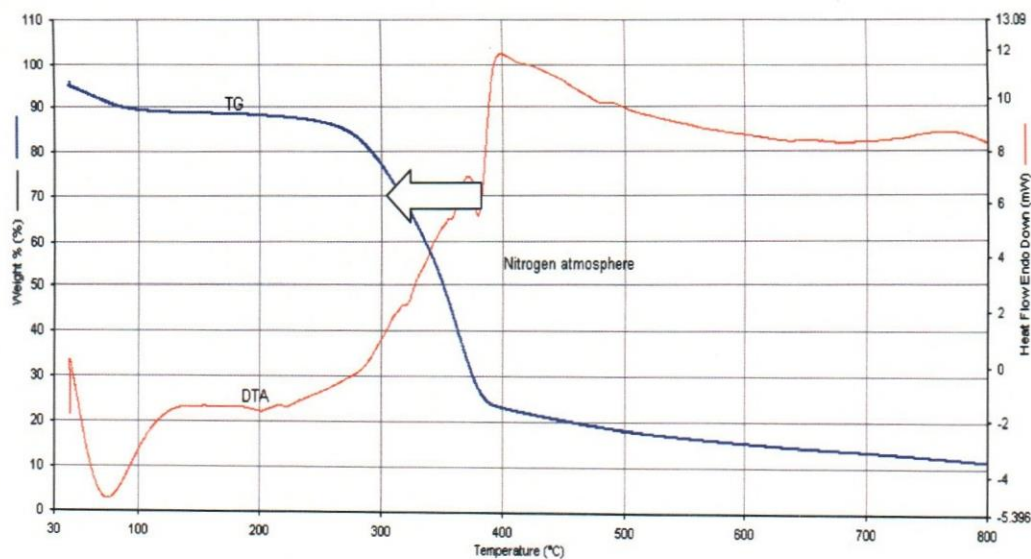


Fig 3b: TGA/DTA of untreated banana stem fibres

Surface Morphology

The micrograph and surface morphology of the natural rubber latex bonded BSFT, BBFT, SIST, EPT

fibre bundles are shown in Fig 4 a,b,c and d respectively while that of fevicol bonded BSF and BSFT are shown in figure 4 e and f respectively.

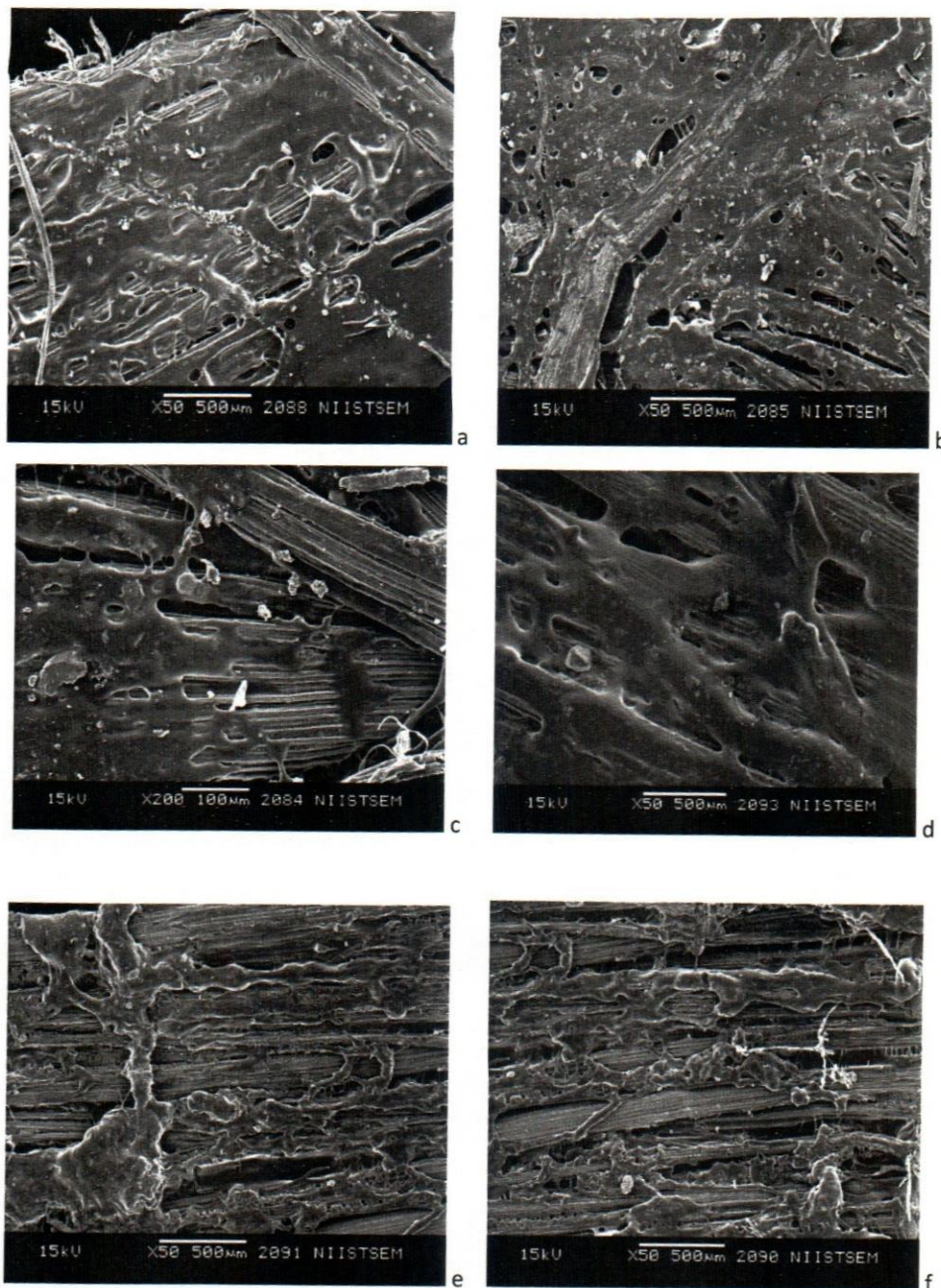


Figure 4: Micrograph of (a) NR bonded BSF (b) NR bonded BSFT (c) NR bonded BBFT (d)NR-bonded EPFT (e)PVA(fevicol) bonded BSFT and (f) PVA(fevicol) bonded BBFT at magnification X50 within a distance radius of 500um.

Discussion

From Fig 1, it can be seen that the treated and *untreated* ebari (EPF) fibres has the highest modulus of elasticity of 39.56 GPa and 19.41 GPa respectively, with the least diameter of 0.1mm. The treated banana stem fibre (BSFT) had a modulus of elasticity of 5.7 GPa. It can be deduced from the data that the treatment of the fibres with a mixture of NaOH and Na₂SO₃ reduced the strength and modulus of the fibre contrary to the report made by [13], except for banana stem fibre (BSFT) where an increase was recorded. A typical case is that of BBF which showed a decrease from a strength of 136.76 MPa for the untreated to a very low value of 57.30 MPa for the treated. Again that of sisal decreased from 174.18 MPa for the untreated to 101.52 MPa for the treated. It is therefore possible that the treatment did not cause any significant degradation of the hemi-cellulose and pectin giving rise to high amorphous part of the fibre, which will lead to no improved strength and modulus. This is in line with the reports that fibre treatment is done for the distension of the crystalline region and elimination of the hydrophilic hydroxyl groups and removal of surface impurities (waxy substances) [14-15].

From Fig. 2, it can also be seen that untreated banana stem fibre (BSF) had the highest water absorption of 106% which is an indication that it has the highest amorphous content, while untreated banana bunch fibre (BBF) has the least

absorption of 66 % which is believed also to be an indication that it has the lowest hemi-cellulose and lignin contents *when compared to others*. However, the treatment of the fibres which effected reduction in the water absorption for every other fibre considered was not observed with the banana bunch fibre, instead an increase from 66 % to 73.1% was observed. The implication of this is that a combination of NaOH and Na₂SO₃ as treatment chemical is not the very suitable choice for banana bunch fibres surface modification.

Figs 3a & 3b showed that a major change in weight (weight loss) occurred between 100 °C and 400 °C. The initial decomposition temperature (IDT) at about 110 °C corresponds to initial weight loss which is mainly due to loss of cellulose cells attached to the fibre, while the second decomposition temperature between 110°C and 190°C corresponds to hemicellulose decomposition. The very major weight loss occurring between 190°C and 380°C is due to decomposition of the crystalline cellulose, while the final weight loss is attributed to decomposition of the lignin content of the fibres. This is similar to some previous literature reports [16-17].

The TGA graphical results (Figs 3a & 3b), indicated that the surface treatment considerably reduced the weight loss of the fibres, such that at about 300°C and 380°C the loss in weight of the

treated fibre is only about 10% and 70% respectively as against the untreated which has a weight loss of about 14% and 76% respectively for the same reference temperatures. The initial weight loss of 14% of untreated fibre against 10% for treated fibre is an indication that the untreated has higher affinity for water which is in line with our water absorption test report. The main deduction from this TGA/DTA analysis is that banana stem fibre can not be processed into useful product beyond 400°C for the untreated and 550°C for the treated fibres. This is evident from the shapes of the DTA graphs.

The SEM micrographs (Fig. 4) showed that the natural rubber latex bonded fibre has a better bonding than the other sample since its covering on the fibres surfaces are more obvious, binding them together in what looks like a laminated composite thereby exhibiting a better interfacial properties than the fevicol adhesives bounded samples, where the samples are seen to be segregated from one another. The better the bonding of the fibres, the greater the interfacial cohesion and the better the mechanical properties and performance in service.

Conclusion

The percentage water absorption, thermal analysis and single fibre tensile strength (SFTS) of a strand of some selected natural fibres has been studied. The following conclusions are drawn.

1. Treated *ebari* plant fibre has the best single fibre tensile strength (SFTS) and

Young's modulus among all the fibres studied.

2. Treatment of natural fibres with a mixture of NaOH and Na₂SO₃ improved the tensile properties of the banana stem fibre and *ebari* fibre but not that of banana bunch and sisal fibre
3. Fibre surface treatment with a solution of NaOH and Na₂SO₃ reduced the water absorption capacity of all the fibres except that of banana bunch fibres
4. The degradation temperatures of the treated and untreated banana stem fibres started within the range 210°C and 380°C after the initial loss due to release of water molecules. This is within the range of polymer processing temperatures. Again, the surface treatment of the fibres reduced the weight loss due to temperature increase by about 10%.
5. The interfacial bonding property of natural rubber latex is better than that of poly-vinyl acetate (Fevicol) and hence natural rubber is preferred as a bond agent for matting of natural fibres prior to lamination.

Acknowledgments

This research was jointly funded by World Bank STEP-B under 2010 Innovators of Tomorrow (IOT) grant and by Indian National Science Academy (INSA), New-Delhi through Centre for International Co-operation in Science (CICS) formerly CCSTDS Chennai India and National Institute for Interdisciplinary Science and

Technology (NIIST-CSIR) Trivandrum, India as part of INSA-JRD-TATA FELLOWSHIP Training, June-

August, 2010. Authors are therefore very grateful to all these sponsoring organizations.

References

- 1 Maleque, M.A. and Behl, F.Y (2006). Mechanical Properties: Study of Pseudo – Stem Banana Reinforced Epoxy Composite. *The Arabian Journal for Science and Engineering*, 32: (2B): 359 – 364.
- 2 Satyanarayana, K.G., Pai, B.C, Sukumaran, K. and Pillai, S.G.K. (1990). Fabrication and Properties of Lingocellulosic Fiber-Incorporated Polyester Composites; Handbook of Ceramics and Composite: (Synthesis and Properties), Marcel Dekker Inc. New York, 1: pp. 339 – 386.
- 3 Amer, A.A., Azza, E., Malash, G.F. and Nahla, A.T. (2007). Extensive Characterization of Raw Barley Straw and Study the Effect of Steam Pretreatment: *Journal of Applied Science Research*. 3: (11): 1336-1342
- 4 Ticoalu A., Aravinthan T. and Cardona F. (2010). A Review of Current Developments in Natural Fiber Composites for Structural and Infrastructure Applications: Southern Region Engineering Conference, 11 – 12 November, Toowoomba, Austria.
- 5 Kaith, B.S. and Chauhan, A. A. (2008). Synthesis, Characterization and Evaluation of the Transformations in *Hibiscus sabdariffa* – Graft – Poly (Butyl Acrylate). *E-Journal of Chemistry*.5: (21): 980-986.
- 6 Roger, M. R. (2006). Advances and Challenges of Wood Polymer Composite: Proceedings of the 8th Pacific Rim-Based Composite Symposium. 20–23 November, pp. 2 – 4.
- 7 Ezema, I.C, Agbo I.U and Enetanya A.N. (2009). Natural Fibre Reinforced Composite: Initiatives in Cashew Nut Shell Liquid Development and Applications: IST International Conference on Polymer Development and Applications, University of Nigeria, Nsukka. March, 18-21, pp 51-56
- 8 Srinirasababu, N., Murali, M.R.K., Suresh Kumar J. (2009). Experimental Determination of Tensile Properties of Olera, Sisal and Banana Fibre Reinforced Polyester Composite: *Indian Journal of Science and Technology*, 2: (7): 35 – 38.
- 9 Gamstedt, E.K and Almgren, K.M.. (2007). Natural Fibre Composites—with Special Emphasis on Effects of the Interface between Cellulosic Fibres and Polymers. Proceedings of the 28th Riso International Symposium on Materials Science, Riso National Laboratory, Roskilde, Denmark. pp 1-20
- 10 Minh – Tan Ton That, Florence P.S., Johanne D; An Improved Method of Single Fiber Tensile Test of Natural Fibre; Wiley Inter-Science Online Publication.Doc. www.interscience.wiley.com/doc/20/5/2011.
- 11 American Standard for Testing and Materials (ASTM). (1975, revised 1989). Standard Test Method for Tensile strength and Young's Modulus for high modulus single filament materials. www.astm.org/standards
- 12 American Standard for Testing and Materials (ASTM). (1998). Standard Test Method for Water Absorption of Plastics. www.astm.org/standards
- 13 Gareth, B. (2007). Performance of Hemp Fibre Reinforced Polypropylene Composite Materials; *Ph.D Thesis* Submitted to Department of Materials & Process Engineering. The University of Waikato, New Zealand. Available online at <http://www.waikato.researchgateway.ac.nz/...pdf> 3/04/2010
- 14 Kabir, M. M., Wang, H., Aravinthan, T., Cardona, F., & Lau, K.-T. (2011). Effects of Natural Fibre Surface on Composite Properties: A Review. *eddbE Proceedings*, pp. 94-99
- 15 Leonard, Y.M., and Martin, P.A. (2002). Chemical Modification of Hemp, Sisal, Jute and Kapok Fibres by

Alkalisiation. *Journal of Applied Polymer Science*, 84: (12): 2222 – 2234.

- 16 Danesh, M.A, Tabari H.Z, Reza H, Noradin N and Shams M. (2012). Investigation of Morphological and Thermal Properties of Waste Newsprint/recycled Polypropylene Nanoclay Composite: *BioResources* 7(1): 936-945.
- 17 Pickering K.L, Li Y, Farell R.L and Lay M. (2007). Interfacial Modification of Hemp Fiber Reinforced Composites Using Fungal and Alkali Treatment: *Journal of Biobased Materials and Bioenergy* 1:109–11