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Preparation and Characterization of Polyester Composites Reinforced with Bleached, *Diospyros perigrina* (Indian persimmon) Treated and Unbleached Jute Mat

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Abstract

In this research work, jute mat reinforced polyester matrix composites have been developed by cold compression molding technique with varying process parameters, such as fiber condition (untreated, bleached and Indian persimmon juice treated). The developed jute fiber reinforced composites were then characterized by tensile test, flexural test, water absorption test, IR spectroscopy, thermal test and scanning electron microscopy. The results shows that tensile strength, flexural strength and value of elastic modulus increases in case of treated and bleached jute mat in compared to untreated fiber. The highest change in tensile strength, flexural strength and elastic modulus has been observed for bleached jute mat-UPR composite which means that the bleached jute mat-UPR composite is stronger than the other two. The water absorption property for the bleached jute mat-UPR composite is the lowest than the other two. The bulk density value shows that the bleached jute mat-UPR composite is denser than the other two. The thermo gravimetric analysis and thermo mechanical analysis also show that the bleached jute mat-UPR composite is more thermally and thermo mechanically stable. Microscopic observation suggests that the fracture behavior is brittle in nature and maximum fiber pull out has been occurred for unbleached jute mat-UPR composite.

Keywords: Jute mat reinforced polyester matrix composite; Tensile test; Flexural test; Water absorption test; IR spectroscopy; Thermal test; Scanning electron microscopy

Introduction

Natural fiber-reinforced composites have attracted great research and economic interests because of their outstanding 'green' characteristics compared with glass fiber-reinforced composites [1]. But the superior resistance of glass fibers to environmental attack made glass-fiber-reinforced polymers more attractive for marine products and in the chemical and food industries [2-5]. On the other hand, the manufacture, use and removal of traditional composite structures usually made of glass, carbon and aramid fibers are considered critically because of growing environmental conscious [6,7]. Scientists have been faced with the difficult challenge of lowering the human impact on their surroundings while maintaining or even bettering the quality of life. The potential environmental and economic benefits of using natural renewable resource based materials have therefore, led to increased interest in the development of novel bio-composite materials. As a result, lingo cellulose fibers which are a natural fiber have been identified as a potential substitute for commonly applied man made synthetic fibers in the preparation of composites due to their lightweight, low cost, no hazardousness, biodegradability, renewability and above all environmental friendly characteristics [8-14]. However, some of the infirmities of natural fiber such as wet ability, non-compatibility with some polymeric matrices, low thermal stability, and hydrophilic nature of fiber surface, poor adhesion with the matrices and swelling and maceration of the fibers due to moisture absorption have prevented complete replacement of synthetic fibers. But the scientific and industrial path of Fiber Reinforced Polymer (FRP) composites was successful primarily because these materials was able to offer structural efficiency and strength to weight ratios over traditional materials such as metals, plastics and wood. Cellulose fibers from kenaf, hemp, ramie, flax, sisal, coir and jute are also being used as reinforcement and they have some advantages over synthetic fibers such as low abrasion, multi

functionality, low density, low cost, unlimited availability and no waste disposal problems which encourage their applications in composite. One of the vastly available natural fibers is jute which is frequently used as reinforcing agent because it is non-abrasive, has low density and high strength. Jute appears to be a promising material because it is relatively inexpensive and commercially available in the required form. It has higher strength and modulus than plastic and is a good substitute for conventional fibers in many situations. Jute composite can be used as particleboard, ceiling, blocks for building construction and furniture.

There are several key factors that serve as a motivation for me in the development of bio-composite materials. Firstly, bio-composite materials offer high strength and stiffness at lighter weights than most traditional materials such as metals, glass fiber reinforced polymer (GFRP) composites and wood products. The seemingly endless supply of agricultural resources to serve as feedstock for bio-composite materials also allows for lower costs and makes them less subject to economic fluctuations. From an environmental and sustainability standpoint, bio based composites from renewable resources are superior to traditional FRP composites as they do not contribute to the depletion of the

world's natural resources, require less energy and chemicals to produce [15-18], emit less greenhouse gases and toxins during their production, use and disposal [16,17] and have the potential to be biodegradable, recyclable or used for energy harvesting upon incineration [15,17].

In this research work, several experimental works were done on three different composites as well as comparative studies on the experimental works were done to find the best reinforcing agent for making a convenient composite.

Materials and Methods

The raw materials for preparing the samples were unsaturated polyester resin (UPR), Monomer (Styrene), Jute mat, juice of *Diospyros perigrina* (Indian persimmon) and Hardener (Methyl ethyl ketone peroxide). The three different types of composite specimen are then prepared by the following way-

First the jute mat (bleached, unbleached) were cut into 31-21 cm pieces, then they are dried. After that Indian persimmon juice is hand-lay up on bleached jute mat which is again dried in an oven. Finally three types (bleached, unbleached and treated) of three layered composite is produced by open molding technique. Then different properties are measured for these three types of composite like Bulk Density, Water absorption, Tensile properties, Flexural properties, Hardness, Thermal, Thermo-mechanical, Chemical, Fracture properties etc.

Bulk density was calculated using following formula,

$$D = \frac{W_s}{V}$$

Where, D=Density of the specimen in kg/m³,

Ws=Weight of the specimen in kg and V=Volume of the specimen in m³.

For water intake measurement, the specimen was prepared according to ASTM-D570-98.The test specimen was 76 mm length, width above 25 mm.

The total water absorption was calculated following the rules given below.

Absorption %=100 $(W_s - W_d) / W_d$

Where, W_d= Conditioned weight of the specimen, W_s=Saturated weight of the specimen after submersing in distill water.

Tensile specimen was prepared according to ASTM-638M-91a.

Tensile strength = Applied loadCross sectional area of the load bearing area

σ = PA

= PA in Pa

Tensile strain is calculated according to ASTM D-638M - 91a.

Tensile strain = $\frac{l - l_0}{l_0}$

Where, l_0= Original length of the sample.

l = Length of the material after stretching

Flexural specimen was prepared according to ASTM D790M, 3 point loading.

The Flexural strength may be calculated for any point of the load deflection by means of the following equation-

$$S = 3PL / 2bd^2$$

Where,

S = stress in the outer fibers at midspan in MPa, P = load at a given point on the load – deflection curve in N, L = support span in mm,

b = width of specimen tested in mm,

d = depth of tested specimen in mm.

Flexural strain may be calculated for any deflection using the following equation-

$$\varepsilon_f = \frac{6Dd}{L^2}$$

Where, ε_f = Main strain in the outer surface in mm/mm, D= Maximum deflection of the center of the beam in mm, L= Support span in mm, d= Depth in mm

Microhardness of the samples was measured by using following formula:

$$HV = 1.854 \frac{F}{d^2} \text{(approximately)}$$

Where, F= Load in Kgf, d = Arithmetic mean of the two diagonals, d_1 and d_2 in mm, HV= Vickers hardness

Leeb Rebound Hardness value is derived from the energy loss of a defined impact body after impacting on a metal sample, similarly to the Shore scleroscope.

Thermal analysis of the specimen includes a group of techniques where some physical property of the sample is monitored under controlled conditions with variation of temperature at a programmed rate. When the mass change is monitored the results, which indicated chemical reactions, are called Thermo gravimetry (TG). When heat absorption monitored, the result indicate crystallization, phase change etc. as well as reactions. This is called Differential Thermal Analysis (DTA). Together, they are a powerful method of analysis.

SEM analyses have been carried out in the Centre for Advanced Research in Sciences (CARS) situated in university of Dhaka. This is a JEOL JSM-6490LA Scanning Electron Microscopy for image analysis. For SEM analysis, the fracture surface of the three different types of composite need to be analyzed. First the non-conducting samples were treated with powdered platinum for making the sample conductive and then set on a carbon tape for analysis.

IR test was done by using Shimadzu IR prestige-21 Fourier Transform Infrared Spectroscopy (FTIR) technique.

Thermo-mechanical analyses of composites were carried out by using an S II-TMA/SS6300 analyzer.

Results and Discussion

From Figure 1 we can clearly see that for bleached jute mat-UPR (Unsaturated Polyester Resin) composite, the value of Bulk Density, Water absorption, Tensile strength, Elastic modulus, Flexural strength , Leeb hardness value is the highest among the three different types of composite. For Indian persimmon treated composite these properties are moderate in nature. The Indian persimmon treated composite has the lowest percentage of elongation Figure 1a-1c, which means that it has the least ductility among the three different types of composite. From Figure 1d-1h we also see that the unbleached jute mat-UPR
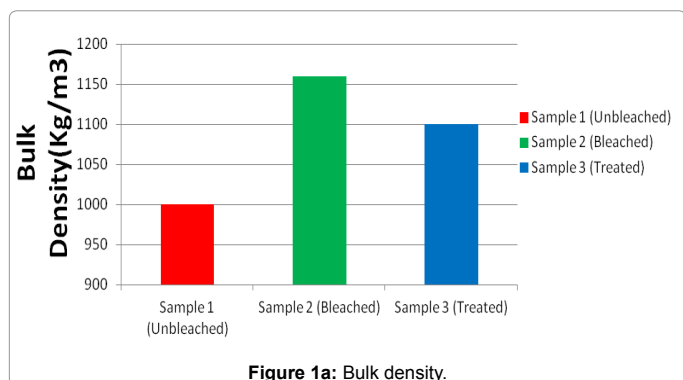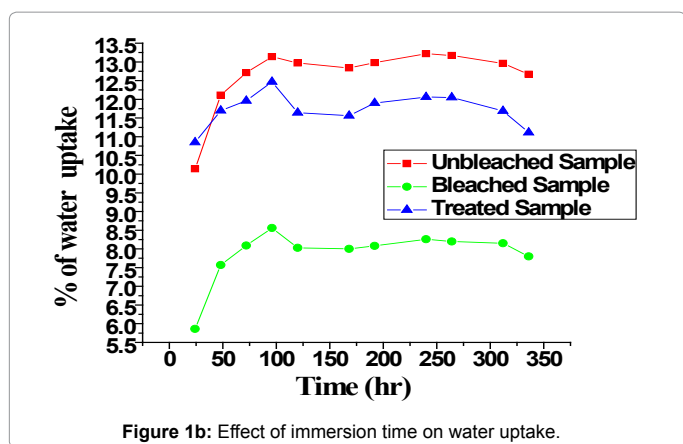
Figure 1a: Bulk density.

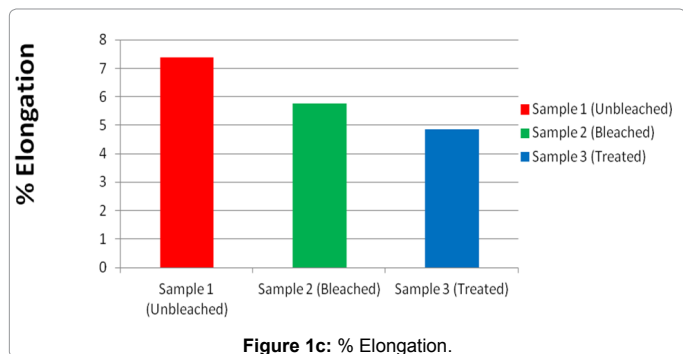**Figure 1b:** Effect of immersion time on water uptake.

Figure 1c: % Elongation.

Figure 1d: Ultimate tensile strength.

Thermal Analysis (DTA) and Differential Thermo Gravimetry (DTG) curves of bleached, treated and unbleached jute mat-UPR composite respectively. The top (blue) one is the TG, bottom (red) one is the DTG and middle (black) one is the DTA curve. The major degradation occurs in one stage. The lighter substances remove initially and then heavier material removed. Initially, the composites loss their weight due to moisture removal. Figure 2d shows the overall Thermo Gravimetric (TG) curves of bleached, treated and unbleached jute mat-UPR composite. The top (blue) one is for bleached comp., bottom (green) one is for unbleached comp. and middle (red) one is for treated comp. The TG curve reveals that the onset temperature of unbleached, treated and bleached jute mat-UPR composite is 333.4°C, 313.5°C and 307.7°C respectively which means the unbleached jute mat-UPR composite is
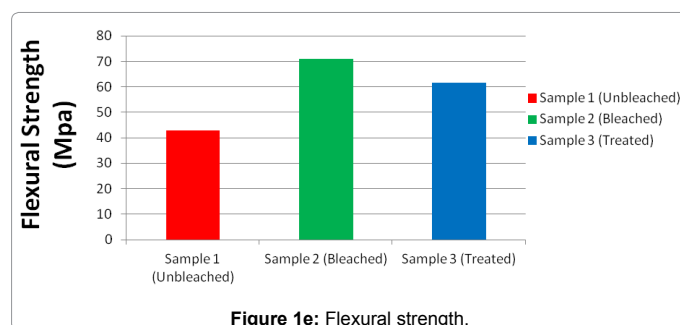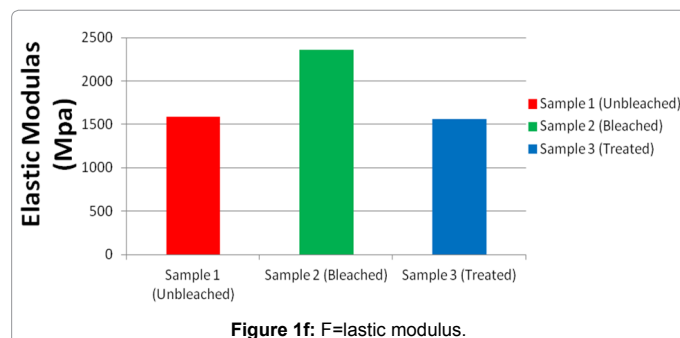

Figure 1e: Flexural strength.
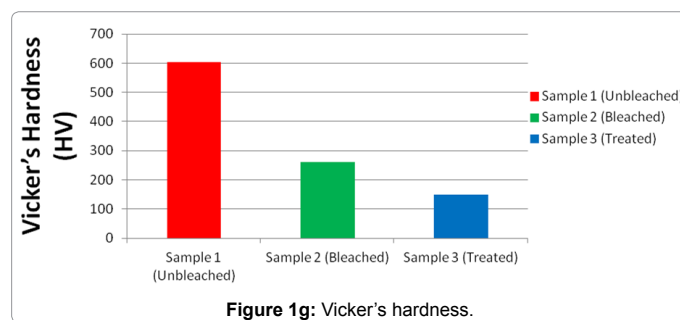

Figure 1f: F=lastic modulus.

Figure 1g: Vicker's hardness.

Figure 1h: Leeb Rebound Hardness Value (HL).

composite has the highest Vicker's hardness value among the three different types of composite. From the above discussion we can say that the unbleached jute mat-UPR (Unsaturated Polyester Resin) composite is harder and stiffer than the other two composite (bleached and treated one).

Figure 2a-2c shows the Thermo Gravimetric (TGA), Differential
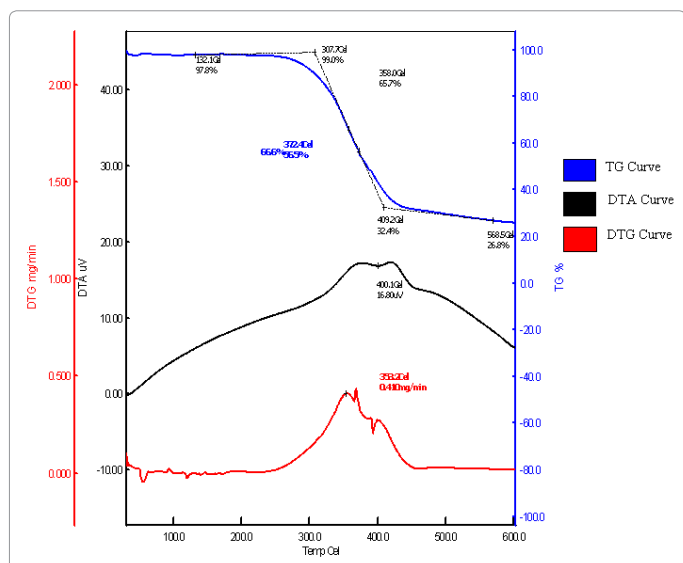
Figure 2a: TG, DTG and DTA curves of bleached jute mat + UPR composite.
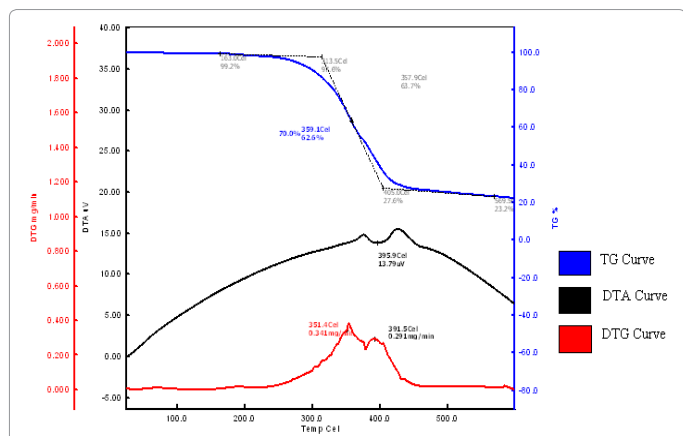

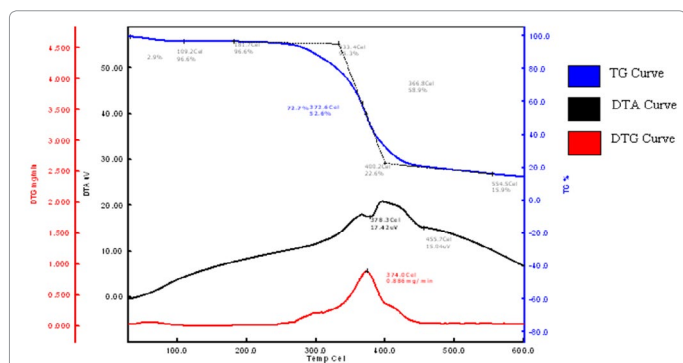**Figure 2b:** TG, DTG and DTA curves of treated jute mat + UPR composite.

Figure 2c: TG, DTG and DTA curves of unbleached jute mat + UPR composite.

and bleached jute mat-UPR composite is 333.4°C, 313.5°C and 307.7°C respectively which means the unbleached jute mat-UPR composite is more thermally stable than the other two. Figure 2f shows the Differential Thermo Gravimetric (DTG) curves of bleached, treated and unbleached jute mat-UPR composite. The top (green) one is for unbleached comp., bottom (red) one is for treated comp. and middle
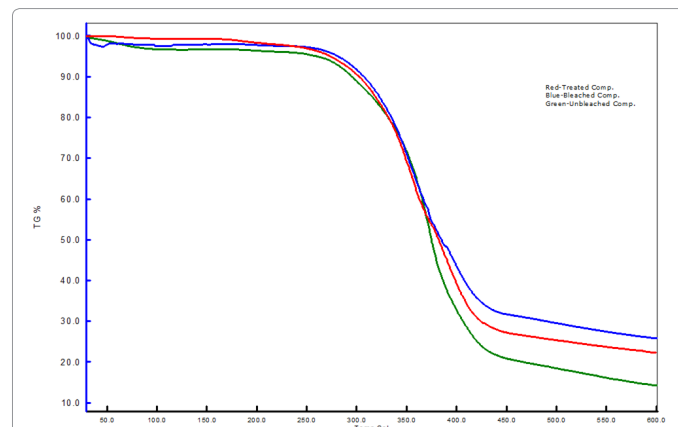

Figure 2d: Thermo Gravimetric Analysis of differently treated jute mat + UPR composite.
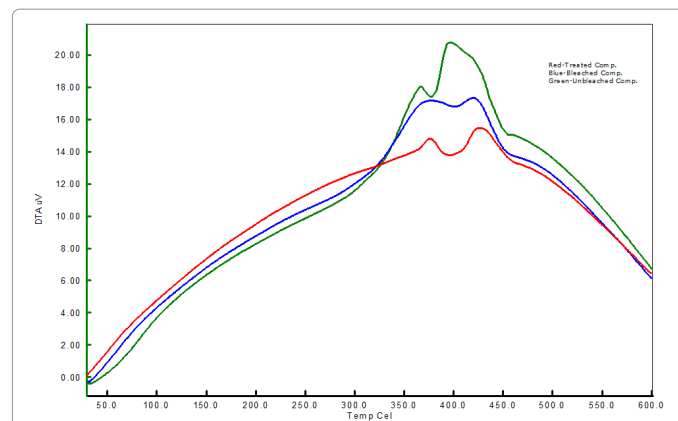

Figure 2e: DTA curves of differently treated jute mat + UPR composite.

Figure 2f: DTG curves of differently treated jute mat + UPR composite.

more thermally stable than the other two.

Figure 2d-2e shows the Thermo Gravimetric Analysis (TGA) curves of bleached, treated and unbleached jute mat-UPR composite. The top (blue) one is for bleached comp., bottom (green) one is for unbleached comp. and middle (red) one is for treated comp. Then the TG curve reveals that the onset temperature of unbleached, treated

(blue) one is for bleached comp. Then the DTG curve reveals that the maximum degradation rate of unbleached, treated and bleached jute mat-UPR composite is 0.886 mg/min at 374°C, 0.341 mg/min at 351.4°C and 0.410 mg/min at 353.2°C respectively which also means the unbleached jute mat-UPR composite is more thermally stable than the other two.

Figure 2e shows the Differential Thermal Analysis (DTA) curves of bleached, treated and unbleached jute mat-UPR composite. The top (green) one is for unbleached comp., bottom (red) one is for treated comp. and middle (blue) one is for bleached comp. The DTA curve yields one endothermic peak for treated and bleached jute mat-UPR composite which is 395.9°C and 400.1°C respectively. The DTA curve yields two endothermic peak for unbleached jute mat-UPR composite is 378.3°C and 455.7°C respectively.

Figure 3a shows the Thermo Mechanical Analysis (TMA) and Differential Thermo Mechanical Analysis (DTMA) curves of treated jute mat-UPR composite. The top (blue) one is for DTMA, bottom (green) one is for TMA. The TMA curve shows that the onset of contraction occurs in the range of 24.6 to 51.6°C, the value of which is 7.53 μm. From DTMA curve we can see that there are two stages of contraction occurs at a rate of 0.719 μm/°C at 63.6°C and at a rate of 0.847 μm/°C at 96.2°C. Figure 3b shows the Thermo Mechanical Analysis (TMA) and Differential Thermo Mechanical Analysis (DTMA) curves of bleached jute mat-UPR composite. The top (blue) one is for DTMA, bottom (green) one is for TMA. The TMA curve shows that the onset of contraction occurs in the range of 23.9 to 61°C, the value of which is 5.20 μm and there is also an expansion occurs in the range of 79.9 to 109.9°C the value of which is 0.55 μm. The maximum contraction occurs at 67°C, the value of which is 9.41 μm. From DTMA curve we can see that there is one contraction occurs at a rate of 0.705 μm/°C at 66.9°C. Figure 3c shows the Thermo Mechanical Analysis (TMA) and Differential Thermo Mechanical Analysis (DTMA) curves of unbleached jute mat-UPR composite. The top (blue) one is for DTMA, bottom (green) one is for TMA. The TMA curve shows that the onset of contraction occurs in the range of 28.1 to 58.5°C, the value of which is 6.68 μm. The maximum contraction occurs at 66.9°C, the value of which is 16.05 μm. From DTMA curve we can see that there is one contraction occurs at a rate of 1.111 μm/°C at 66.6°C

Figure 3d shows the Thermo Mechanical Analysis (TMA) curves of bleached, unbleached and treated jute mat-UPR composite. The top (green) one is for bleached jute mat-UPR composite, bottom (red) one is for treated jute mat-UPR composite and middle (black) one is for unbleached jute mat-UPR composite. The TMA curve shows that for bleached, unbleached and treated jute mat-UPR composite, the onset of contraction occurs at 23.9°C, 28.1°C and 24.6°C respectively, the value of which is 8.20 μm, 6.68 μm and 7.53 μm respectively which means that bleached one is thermo mechanically unstable than the other two composite. Figure 3e shows the Differential Thermo Mechanical Analysis (DTMA) curves of bleached, unbleached and treated jute mat-UPR composite. The top (black) one is for unbleached jute mat-UPR composite, bottom (green) one is for bleached jute mat-UPR composite and middle (red) one is for treated jute mat-UPR composite. From DTMA curve we can see that there are two stages of contraction occurs for treated jute mat-UPR composite at a rate of 0.719 μm/°C at 63.6°C and at a rate of 0.847 μm/°C at 96.2°C. For bleached and unbleached jute mat-UPR composite we can see that there is one contraction occurs at a rate of 0.705 μm/°C at 66.9°C and 1.111 μm/°C at 66.6°C respectively.

Figure (S-1-S-3) shows the FTIR spectrum of the treated, bleached and unbleached jute mat-UPR composite. The intense peak for

ester group appeared at wavenumber of 1728 cm⁻¹ for bleached and unbleached jute mat-UPR composite. For treated jute mat-UPR composite ester group appeared at wavenumber of 1724.36 cm⁻¹. The C=O absorption band of saturated aliphatic esters with intense appearance is presenting the 1750-1725cm⁻¹ region. The more intense peak for C-O appeared at wavenumber of 1041.56,1043.49 and 1039.63 cm⁻¹ for bleached, treated and unbleached jute mat-UPR composite

Figure 3a: TMA and DTMA curves of treated jute mat + UPR composite.

Figure 3b: TMA and DTMA curves of bleached jute mat + UPR composite.

Figure 3c: TMA and DTMA curves of unbleached jute mat + UPR composite.

Figure S1: IR analysis of treated jute mat + UPR composite.

Figure 3d: TMA curves of differently treated jute mat + UPR composite.

Figure S2: IR analysis of bleached jute mat + UPR composite.

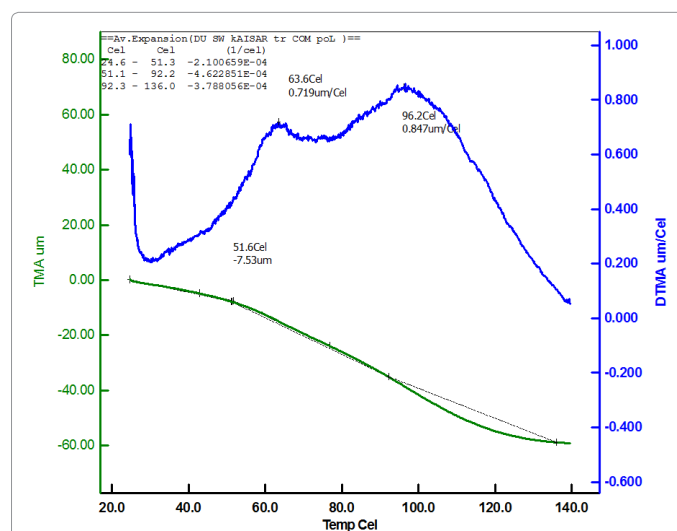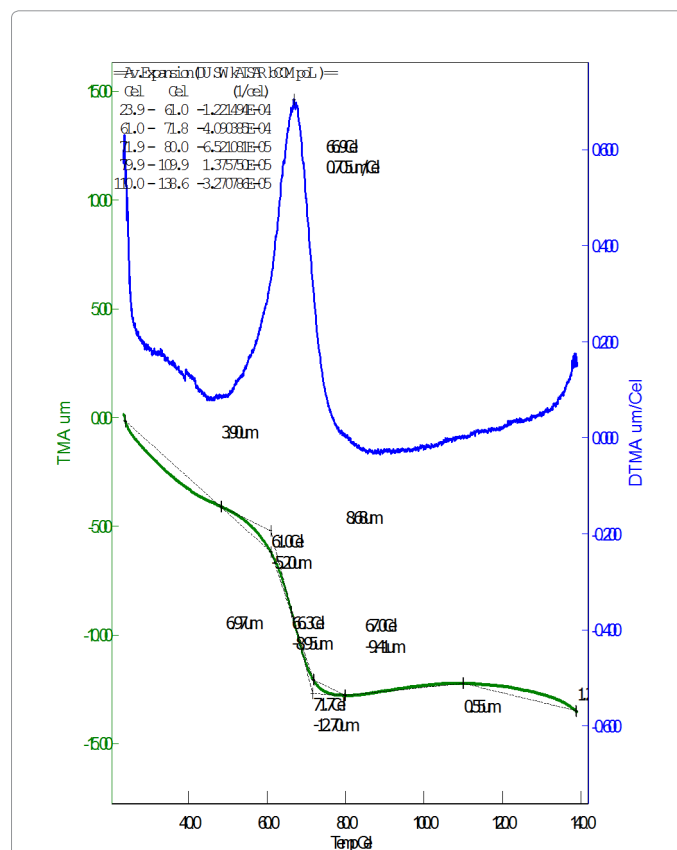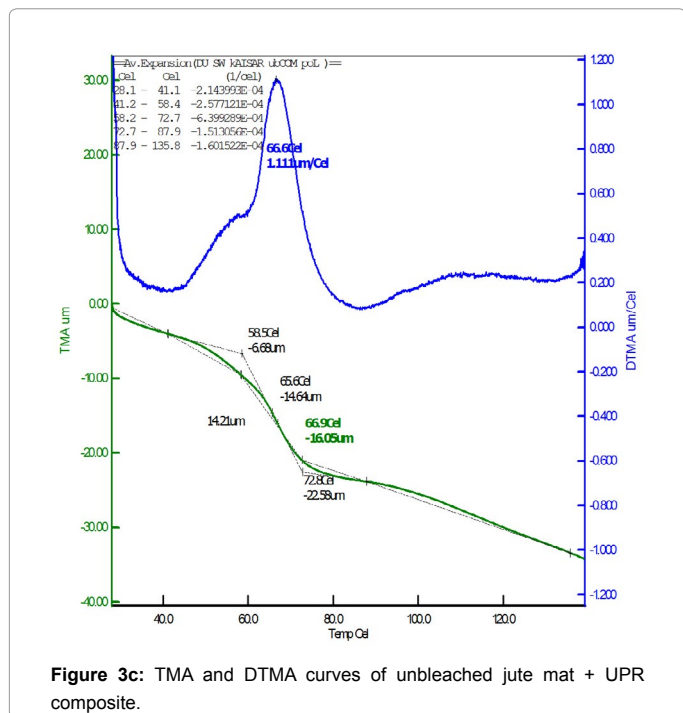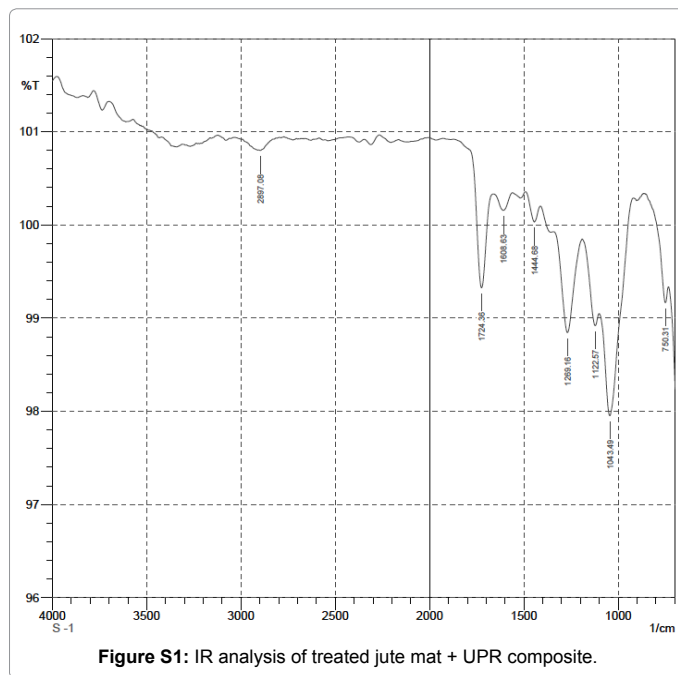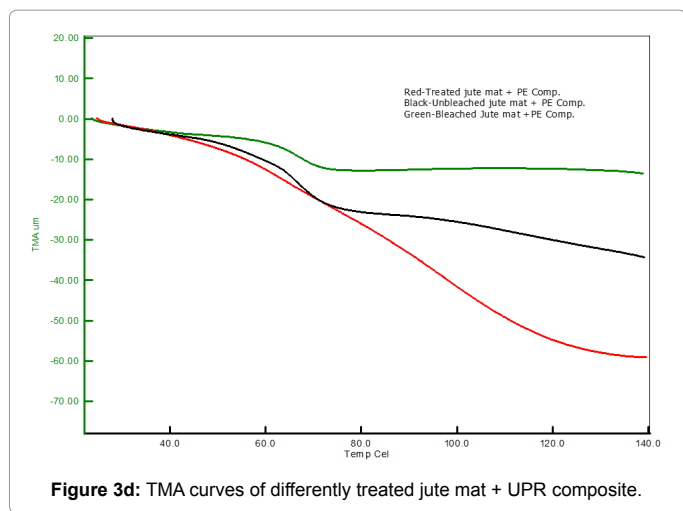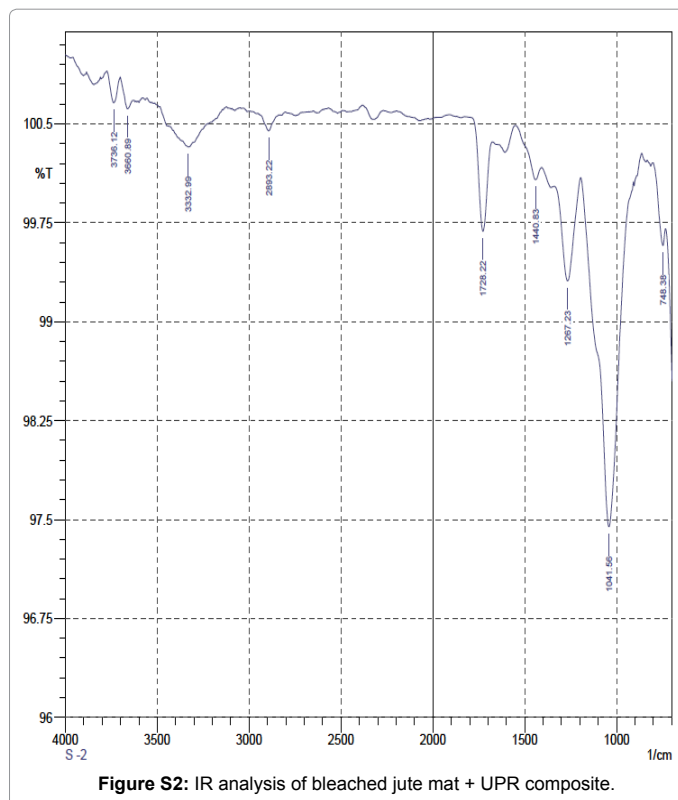**Figure 3e:** DTMA curves of differently treated jute mat + UPR composite.

which should be found in the range of 1300-1000 cm^{-1} in the case for either the C-O bond in cellulose or for C-O bond of ester in polyester. Another intense peak is found at wavenumber of 3660 and 3610 cm^{-1} for bleached and unbleached jute mat-UPR composite which is for alcohol group in cellulose.

Scanning electron microscopic (SEM) investigation of the fracture surface of the jute composites is performed to study interfacial properties between jute fiber and UPR matrix. SEM images of fracture
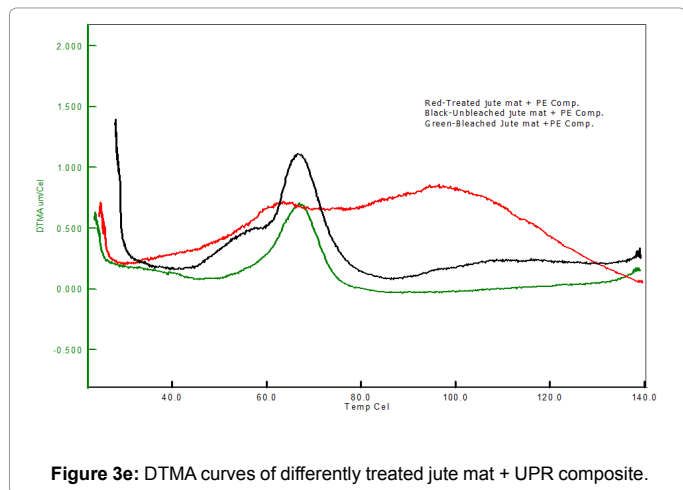
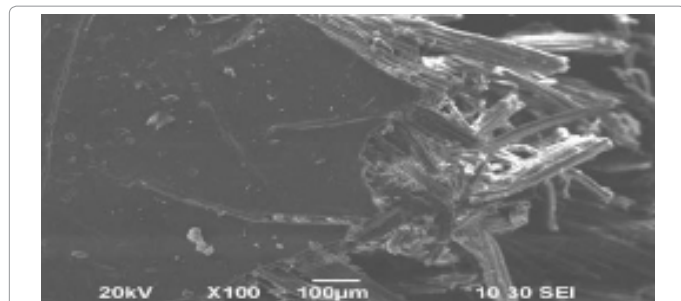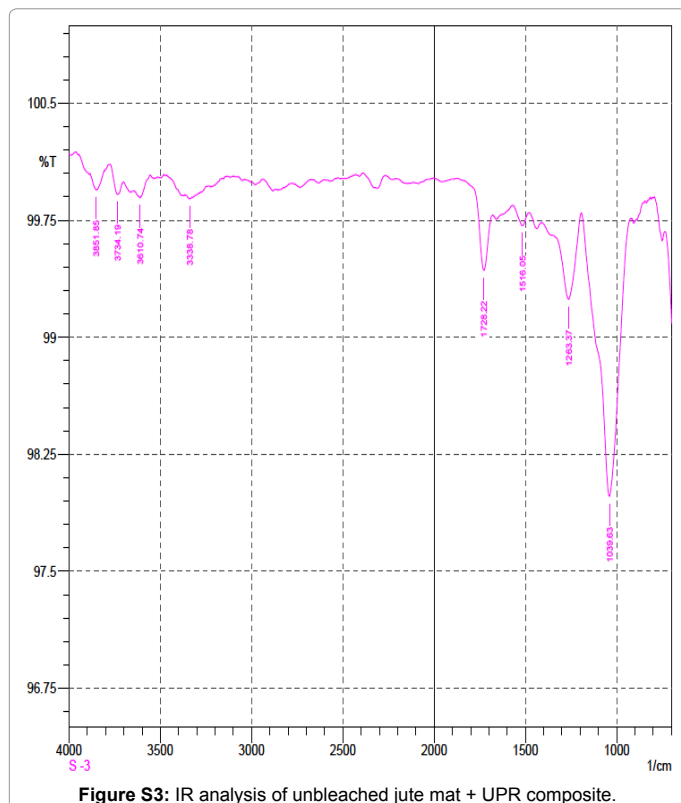**Figure S3:** IR analysis of unbleached jute mat + UPR composite.

Figure 4a: Fracture surface of bleached jute mat –UPR composite shows fibre pull out and poor fibre matrix adhesion (100 magnification).
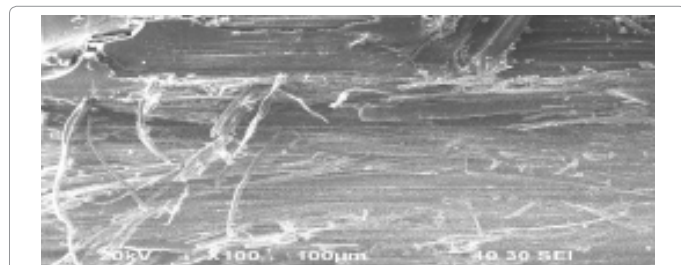

Figure 4b: Fracture surface of unbleached jute mat-UPR composite shows fibre pull out and poor fibre matrix adhesion (100 magnification).

surface of (a) Bleached jute mat + UPR composite (Figure 4a) and (b) Unbleached jute mat + UPR composite (Figure 4b) are presented in Figure 4a and 4b The Figure clearly indicates that there is considerable difference in the fiber-matrix interaction between the bleached and unbleached jute mat + UPR composites. SEM observation suggests that the fracture behavior is brittle in nature. Fiber pull-out phenomena are observed for all cases but for unbleached jute mat + UPR composite

fiber pull-out is observed in bundle form whereas in bleached jute mat + UPR composites agglomeration of the fibers into bundle form is relatively prevented. Between the bleached samples and unbleached samples (Figure 4a and 4b) shows significant change of morphology and this is effective for better mechanical bonding between fiber and polymer matrix.

Conclusions

The result of the present study reveals that composites with good strength could be successfully developed by using bleached jute mat as the reinforcing agent. From characterization of three layered jute mat-UPR show high strength but composite's SEM analysis indicating a strong adhesion between lignocellulose bleached jute mat and polyester. So from the thesis work we concluded that bleached fiber was the best one for reinforcement although medium performance Indian persimmon treated jute fiber can be used where there is a cost concern involved with the bleaching agent.

References

1. Yan ZL, Zhang JC, Lin G, Zhang H, Ding Y, et al. (2013) Fabrication process optimization of hemp fiber-reinforced polypropylene composites. Journal of reinforced plastics and composites 32: 1504-1512.

2. Wambua P, Ivens J, Verpoest I (2003) Natural Fibers: Can they replace Glass in Fiber Reinforced Plastics? Composites Science and Technology 63: 1259-1264.

3. Sharifah HA, Martin PA, Simon TC, Simon RP (2005) Modified polyester resins for natural fiber composites. Compos Sci Technol 65:525-535.

4. Bledzki AK, Gassan J (1999) Composites reinforced with cellulose based fibers. Prog Polym Sci 24: 221-274.

5. Marion P, Andreas R, Marei HM (2003) Study of wheat gluten plasticization with fatty acids. Polym 44: 115-122.

6. Sebe G, Cetin NS, Hill CAS, Hughes M (2000) RTM Hemp Fiber-Reinforced Polyester Composites. Applied Composite Materials 7: 341-349.

7. Chawla KK, Bastos AC (1979) The Mechanical Properties of Jute Fiber and Polyester/Jute Composites. Mechanical Behaviour of Materials 3: 191-196.

8. Jiang L, Hinrichsen G (1999) Flax and cotton fiber reinforced biodegradable polyester amide. Die Angew Makromol Chem 268:13-17.

9. Fan L, Du Y, Hung R, Wang Q, Zhang L (2005) Preparation and Characterization of Alginate/Gelatin Blend Fibers. Journal of Applied Polymer Science 96: 1625-1629.

10. Idicula M, Joseph K, Thomas S (2010) Mechanical Performance of Short Banana/ Sisal Hybrid Fiber Reinforced Polyester Composites. Journal of reinforced plastics and composites 29: 12-29.

11. Khan MA, Khan RA, Zaman HU, Alam MN, Houqe MA (2009) Effect of Surface Modification of jute with Acrylic Monomers on the Performance of polypropylene.A Composites. Journal of Reinforced Plastics and Composites.

12. Khan RA, Khan MA, Sultana S, Khan MN, Shubra QTH, et al. (2009) Mechanical, Degradation and Interfacial Properties of Synthetic Degradable Fiber Reinforced Polypropylene Composites. Journal of Reinforced Plastics and Composites 49:466-476.

13. Shokoohi S, Arefazar A, Khosrokhavar R (2008) Silane Coupling Agents in polymer- based Reinforced Composites: A Review. Journal of Reinforced Plastics and Composites 27:473-485.

14. Kuswaha P, Kumar R (2009) Effect of Silanes on Mechanical properties of Bamboo Fiber-epoxy Composites. Journal of Reinforced Plastics and Composites.

15. Saheb DN, Jog JP (1999) Natural Fiber Polymer Composites: A Review. Advances in Polymer Technology 18: 351-363.

16. Schloesser TP (2004) Natural Fiber Reinforced Automotive Parts. In: Natural Fibers, Plastics and Composites. Wallenburger FT, Weston NE (eds) Kluwer Academic Publishers, Norwell, MA, USA.

17. Joshi SV, Drzal LT, Mohanty AK, Aurora S (2004) Are Natural Fiber Composites Environmentally Superior to Glass Fiber Reinforced Composites? Composites Part A: Applied Science and Manufacturing 35: 371-376.

18. Patel M, Narayan R (2005) How Sustainable are Polymers and Biobased Products? The Hope, the Doubts and the Reality. In: Natural Fibers, Biopolymers and Biocomposites. Mohanty AK, Mishra M, Drzal LT (eds) CRC Press, Boca Raton, USA.