PREPARATION AND CHARACTERIZATION OF JUTE FIBER REINFORCED COMPOSITE

Kyi Win Mon[1], Thinzar Nu[2], Hnin Yu Wai[3]

Abstract

This research work is mainly concerned with the preparation of composite from 5 % NaOH modified waste jute fiber with epoxy resin as binder and the study of their characteristics. Jute fiber (JF) and epoxy resin (E) were collected from Yangon Region and this research was done at Polymer Research Department, Department of Research & Innovation (DRI), Yangon. Composites were prepared by mixing 5 % NaOH modified waste jute fiber (120 g) with various amounts (5 %, 10 %, 15 %, 20 %, 25 % and 30 %) of epoxy resin (E) using cold compressing method at 2300 psi. The prepared composites were characterized by physicochemical and physicomechanical parameters such as modulus of rupture (MOR), thickness, density, water absorption, swelling thickness as well as hardness. From the results obtained, it was found that 5 % NaOH modified waste jute fiber (120 g) with 20 % epoxy resin (E), namely (JFE 4) composite was the best quality grade composite. It possesses 1248.20 psi modulus of rupture, 0.60 cm thickness, 1.2320 g cm^{-3} density, 14.18 % water absorption, 23.35 % swelling thickness, and 68 D hardness. JFE 4 composite based on physicomechanical properties, the composite showed to possess the highest modulus of rupture value indicating that this composite was the most significant and the best among all the composites. The FT IR study confirmed the presence of cellulose, hemicellulose and lignin. In addition, Simultaneous Thermal Analyzer (STA) confirmed that the sample was thermally stable till 200 ˚C on account of the high complex structure of lignin. The melting point of composite was round about 400 ˚C studied by Simultaneous Thermal Analyzer. The surface roughness value of JFE 4 composite was 900 nm studied by Atomic Force Microscopy. The present research on making composite from waste materials can contribute to the solution of the earth's escalating environmental problems.

Keywords: Jute composite, jute fiber, epoxy resin, modulus of rupture

Introduction

The production of lignocellulose fibers-based polymer composites has become in recent years an important application for recovering, reuse and recycling a variety of by-products related to industrial exploitation of natural resources. Lignocellulose materials (wood fibers, wood flour, and agro-residues) are attractive fillers for thermoplastic polymers mainly because of their low cost and large availability. According to their botanical source, these renewable materials present large variations in their composition. Main elements are cellulose, hemicelluloses and lignin which are known to present a very complex structure (Ruxanda *et al*., 2009).

The use of natural fiber from both resources, renewable and non-renewable such as oil palm, sisal, flax, and jute to produce composite materials, gained considerable attention in the last decades, so far. The plants, which produce cellulose fibers can be classified into bast fibers (jute, flax, ramie, hemp, and kenaf), seed fibers (cotton, coir, and kapok), leaf fibers (sisal, pineapple, and abaca), grass and reed fibers (rice, corn, and wheat), and core fibers (hemp, kenaf, and jute) as well as all other kinds (wood and roots) (Faruk *et al*., 2012).

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Jute fiber is a promising reinforcement for use in composites on account of its low cost, low density, high specific strength and modulus, no health risk, easy availability, renewability and much lower energy requirement for processing. The jute fiber is an important bast fiber and comprises bundled ultimate cells, each containing spiral oriented micro- fibrils bound together (Debiprasad *et al*., 2012).

Wood fibers offer a number of advantages over the currently used reinforcing inorganic materials (e.g., glass fibers) in terms of cost on a unit volume basis, as well as flexibility during processing. Wood modification can change important properties of the wood, including biological durability, dimensional stability, hardness and Ultraviolet stability by converting hydrophilic OH-groups into larger, more hydrophobic groups. Specific wood modification processes designed for the production of wood-plastic composites may be developed having in view the properties of the wood component in the composites, but also the improved, e.g., by means of grafting processes (Ruxanda *et al*., 2009).

Composites are produced from non-chemical processed using fiber particles mixed with binder. The main objective of composite materials is their high strength and stiffness, combined with low density, when compared with bulk materials, allowing for a weight reduction in the finished part. The availability of natural fiber jutes is much abundant in Asian continent and it provides and advantages over reinforcement materials in terms of cost, density recyclability and biodegradability .Meanwhile Fiber-Reinforced Polymers (FRP) have until now been largely applied to the area of aerospace technology, these construction materials have also been used in many technical applications for achieve required strength (Abilash and Sivapragash, 2013).

In Myanmar, study on preparation and characterization of composite boards derived from natural fibers (sisal fiber) (Na-Nat-Shaw)(Zin Mar Aung, 2008), preparation and characterization of composite (plaster) board derived from renewable resource, non-wood plant fiber (Bagasse) (Myat Myat Nwe, 2008) and Particleboards Derived from Rattan Fiber Waste (Hnin Yu Wai, 2011) in Yangon University was reported. In the preparation of composites, the two main components involved are binder and the other is a fibrous material. The binder plays an important role to obtain good strength of the board and also for low cost products. This research work intends to use the epoxy resin in making composite with an agricultural waste fiber, the jute fiber. The prepared composites were characterized according to the physicochemical and physicomechanical properties as well as Simultaneous Thermal Analyzer and Atomic Force Microscopy analysis.

Materials and Methods

Composites were produced by using 120 g each of waste jute fiber with various concentrations of epoxy resin. This research work intends to use the epoxy resin in making composite with an agricultural waste fibers, more precisely using the jute fiber. The prepared composites were characterized according to the physicochemical and physicomechanical properties as well as Simultaneous Thermal Analyzer and Atomic Force Microscopy analyses. All the experimental procedures involved in this research. All necessary research facilities were provided by the Polymer Research Department, Department of Research and Innovation. All specific chemicals used were described detail in each experimental section. In all the investigations, the recommended and standard procedures of both conventional and modern

techniques were employed. The experiment was carried out in the National Analytical Laboratory, Department of Research and Innovation, Yangon Region.

Collection of Samples

In the experiments, the waste jute fibers collected from (Kyaw Htet Kyaw) company limited and epoxy resin from shine company limited, Yangon Region, Myanmar.

Preparation of Waste Jute Fiber

Raw samples were cut by cutting machine. After cutting, the fiber obtained in size as about 0.5-2.0 cm. The fibers were taken and dried in an oven at 70 ºC.

Preparation of Modification of Waste Jute Fiber

The fibers (*ca*. 400 g) were immersed in 6 L solution of (5 % w/v) NaOH (pH~ 13) for 24 h at room temperature, after which the fibers were washed thoroughly with plenty of water until drained water became neutral. After treatment, they were dried in the sunlight and then in an oven at 70 °C for 3 h.

Physicochemical Properties of Modified Waste Jute Fiber

The physicochemical properties (moisture content, ash content, solubility in hot water, solubility in 1% NaOH) of modified waste jute fiber were determined by conventional methods.

Physicochemical Properties of Epoxy Resin

The physicochemical properties (moisture content, ash content, viscosity, pH, gelation time) of epoxy resin were determined by conventional methods.

Characterization of Waste Jute Fiber and the Modified Waste Jute Fiber

FT IR Analysis

The sample was directly placed on the Spectrum 400 FT IR and FT NIR Spectrometer, diamond plate of Universal Attenuated Total Reflectance (UATR) detector over a 400-4000 cm^{-1}, scanning 5 min and wave number range at a resolution of 4 cm^{-1}.

STA Analysis

Thermal properties were monitored by simultaneous thermal analyzer. The sample (*ca*. 7 mg) was cut and placed in a ceramic crucible. The lid was placed over the sample. The system should be built with SaTurnA™ sensor and measured the temperature range of the system must be 50 °C to 950 °C or better. Temperature accuracy should be ±0.8 °C. The system must cool down less than 15 min. The flow rate of nitrogen gas wast 20.00 mL/min. The prepared modified waste jute fiber was performed by simultaneous thermal analyzer with nitrogen atmosphere.

AFM Analysis

Topography of the surfaces on the molecular scale was investigated by N 8, Rados Atomic Force Microscopy, Bruker, Germany. As the AFM probes the atomic interaction between surfaces, it can also be used to map the adhesion, friction, stiffness or other kinds of interaction such as magnetic or electric forces. In each AFM experiment, several scans were made to check the reproducibility of images and the absence of surface damage. Surface roughness and morphology can be seen and analyzed.

Characterization of Epoxy Resin

FT IR Analysis

FT IR analysis was performed in order to characterize the functional groups of the epoxy resin Perkin-Elmer Spectrum, Thailand was used for FT IR analysis.

STA Analysis

The prepared epoxy resin was performed by Simultaneous thermal analyzer with nitrogen atmosphere.

Preparation of Composites

The composites were fabricated by hand lay-up technique. The inner cavity dimension of the mould is 15.4 cm x 15.4 cm. First of all, a release gel is wiped on the mould surface to avoid the sticking of epoxy resin to the surface. Thin plastic sheets are used at the top and bottom of the mould plate to get a good surface finish of the product. The 5 % sodium hydroxide modified fiber was further with epoxy resin in liquid form was mixed thoroughly in suitable proportion with a prescribed hardener (curing agent) and poured onto the surface of mat already placed in the mould. The epoxy resin was uniformly spread with the help of the brush. Composites of different compositions with constant fiber loading are made. The composites from the cold press were kept at room temperature for 24 h. The prepared composites were cut for testing conforms to the dimensions of the specimen.

Physicochemical and Physicomechanical Properties of Composites

Modulus of Rupture

The composites were cut into pieces (14 cm × 2.54 cm), and modulus of rupture of individual composites was measured by Electro-hydraulic tensile tester according to the procedure. All of the samples were measured at least five references. Two references were taken from one and three references were taken from another of the same ingredient of composites.

Thickness

The composites were cut into pieces (14 cm × 2.54 cm), and thickness of individual composites were measured by veneer clipper at least four points of each bundle.

Water Absorption

The composites to be tested were cut into 2.54 cm × 2.54 cm in size, weighed and placed in a desiccator. Then, the test specimens were immersed in fresh clean water for 24 h. After soaking in water, each test piece was withdrawn from the water, wiped with absorbent paper to remove water on the surface and reweighed to the same degree of accuracy as before. Five replicated specimens of individual composites were tested and the results were presented as average of the tested specimens.

Swelling Thickness

The composites to be tested were cut into 2.54 cm × 2.54 cm in size, the thickness of test pieces were measured and placed in a desiccator. These pieces were immersed in clean water at room temperature. They were covered by approximately 1 inch of water. After 24 h of water soaking, these pieces were removed from water and wiped with absorbent paper to remove water

on the surface and allowed to stand under normal room condition for 2 h. The thickness of each test piece was premeasured with screw gauge and increase was recorded.

Density

The composites to be tested were cut into 2.54 cm × 2.54 cm in size. The length, width and thickness of each test piece were measured to an accuracy of ±0.01 cm. The weight in grams of each test piece was determined to an accuracy of ± 0.01 g.

Hardness

Durometer hardness readings were performed according to ASTM D 2240. The test was carried out by first placing a specimen on a hard, flat surface. The pressure foot of the instrument was pressed on to the specimen, making sure that it was parallel to the surface of the specimen. The durometer hardness was read within one second after the pressure foot was in form contact with specimen. Each specimen was subjected to durometer hardness readings, at designated positions on the sample bases.

Characterization of JFE 4 Composite

STA Analysis

The thermogram of JFE composite was analyzed by simultaneous thermal analyzer.

AFM Analysis

Topography of JFE 4 composite surface and roughness was obtained with the help of Atomic Force Microscope.

Results and Discussion

Table 1 shows that the physicochemical parameters of modified waste jute fiber. It was found that moisture content is 7.83 % , ash content is 3.34 %, solubility in hot water 4.32 % solubility in 1 % NaOH is 26.38 %.

Table 1 Physicochemical Properties of Modified Waste Jute Fiber

Characteristics	Observed Values (%)
Moisture content	7.83
Ash content	3.34
Solubility in hot water	4.32
Solubility in 1 % NaOH	26.38

Physicochemical Properties of Epoxy Resin

Table 2 shows that the physicochemical properties of epoxy. It was found that moisture content is 0.58 %, ash content is 0.88 %, viscosity is 4319 cp, pH is 6.8 and gelation time is 20 min.

Table 2 Physicochemical Properties of Epoxy Resin

Characteristics	Observed Values
Moisture content (%)	0.58
Ash content (%)	0.88
Viscosity (cp)	4319
pH	6.8
Gelation Time (min)	20

Characterization of the Modified Waste Jute Fiber

FT IR Analysis

The FT IR spectrum of modified waste jute fiber shows has especially hydroxyl and carbonyl stretching because of the presence of cellulose shown in Figure1and the description data in Table 3 are given.

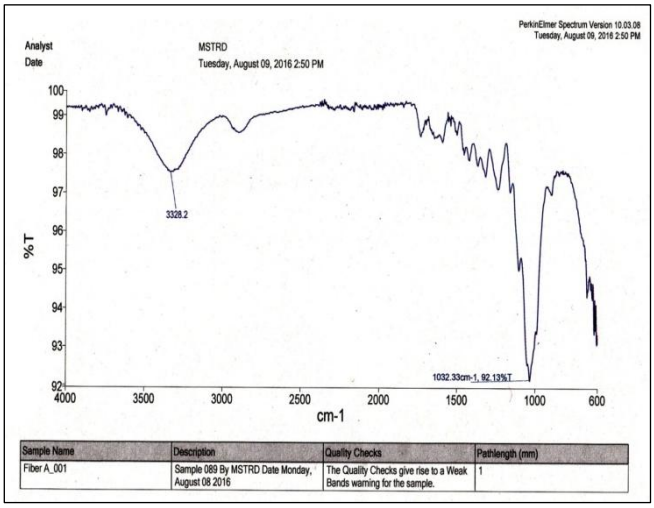


Figure 1 FT IR spectrum of the modified waste jute fiber

Table 3 FT IR Spectral Data of the Modified Waste Jute Fiber

Frequency (cm^{-1})	Band Assignments	Band Assignments
Modified Waste Jute fiber	**Literature ***	
3328	3330-3270	O-H stretching
1032	1320-1000	C=O stretching

* as Win Lab software of FT IR machine

STA Analysis

Simultaneous Thermal Analyzer (STA 6000) is a thermal method that measured the weight loss as a function of temperature or time. The STA scan of waste jute fiber is shown in Figure 2 and the description data in Table 4 are given. In the case of waste jute fiber, 8.03 % of weight loss indicated the evaporation of moisture content evinced by the presence of hydroxyl content in the fibers and other volatile matter. The major weight loss 62.22 % occurs at

365.28 °C. Endothermic peak was observed in the temperature range 200 ºC and 500 ºC that is attributed to the thermo oxidative degradation of cellulose and hemicellulose that takes place by the degradation of its lignin. The weight loss 27.29 % was gradually degraded of lignin by the breakage of its subunits.

The STA scan of the modified waste jute fiber is shown in Figure 3 and the description data in Table 5 are given.The first stage was from about 50 °C to 200 °C. In this stage of temperature, a loss in weight about 4.61 % indicated dehydration due to surface water and moisture. After that the second stage, the loss in weight about 64.61 % was observed at the temperatures range from 200 °C to 500 °C. The thermograph of individually modified waste jute fibers peak was 362.59 °C was degradation of cellulose and hemicellulose. The third stage, loss in weight about 27.69 % at temperature range of 500 °C to 800 °C was decomposition of lignin. Modified waste jute fibers were completely burnt around 462.34 °C.

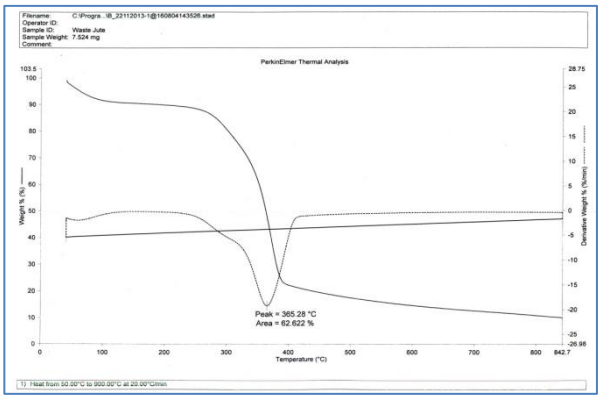


Figure 2 STA thermogram of waste jute fiber

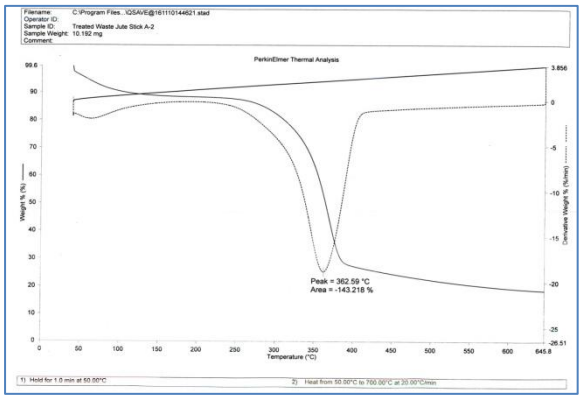


Figure 3 STA thermogram of modified waste jute fiber

Table 4 Thermal Analysis Data of Waste Jute Fiber

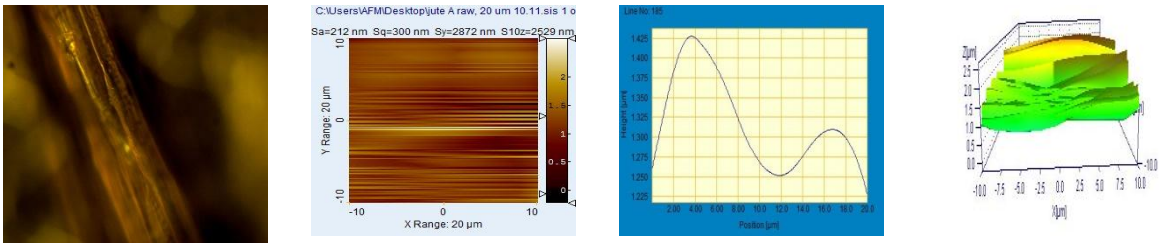
Sample	Melting point value (°C)	Temperature range (°C)	Weight loss (%)	Nature of peak	Remarks
Waste Jute fiber	-	50-200	8.03	-	Dehydration due to surface water and moisture content
	365.28	200-500	62.22	endo	Degradation of cellulose and hemicellulose
	-	500-800	27.29	-	Decomposition of lignin of the jute fiber

Table 5 Thermal Analysis Data of the Modified Waste Jute Fiber

Sample	Melting point value (°C)	Temperature range (°C)	Weight loss (%)	Nature of peak	Remarks
Modified waste jute fiber	-	50-200	4.61	-	Dehydration due to surface water and moisture
	362.59	200-500	64.61	endo	Degradation of cellulose and hemicellulose
	-	500-800	27.69	-	Decomposition of lignin of the jute fiber

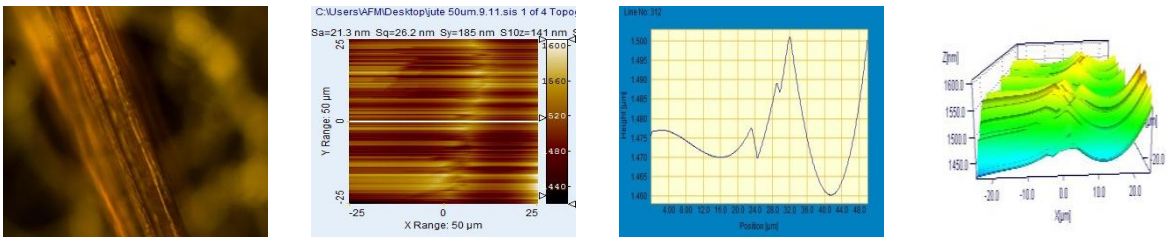
AFM Analysis

The surface roughness of the waste jute fiber and modified waste jute fiber were also studied using Atomic Force Microscopy. AFM microphotographs of waste jute fiber and the modified waste fiber are shown in Figures 4 and 5 and the description data in Table 6 are given. It was obvious from the image that significant smoothing occur in jute fiber surface. AFM was employed to monitor the topological changes of the heterogeneous surface morphologies and the roughness of waste jute fiber. A cantilever with a sharp force sensing tip interacted with the surface. The vertical position of the scanner at each (x,y) data point in order to maintain a constant "set point" amplitude was stored by the computer to form the topographic image of the sample surface. The surface roughness values were observed as 1.425 µm and 1.500 µm because of a layer of pectin and waxy materials which covered the surface of fiber.



(a) **(b)** **(c)** **(d)**

Figure 4 AFM microphotographs of waste jute fiber (a) morphology (b) topography (c) line profile (d) 3 Dimension



(a) **(b)** **(c)** **(d)**

Figure 5 AFM microphotographs of modified waste jute fiber (a) morphology (b) topography (c) line profile (d) 3Dimension

Table 6 Microscopic Analysis Data of Waste Jute Fiber and Modified Waste Jute Fiber

Sample	Surface roughness (µm)	Remarks
Waste jute fiber	1.425	The outer layer of pectin and waxy materials which covered the surface of fiber
Modified waste jute fiber	1.500	The outer layer of lignin was removed during alkali treatment

Characterization of Epoxy Resin

FT IR Analysis

The FT IR spectrum of epoxy resin is shown in Figure 6 and the description data in Table 7 are given.

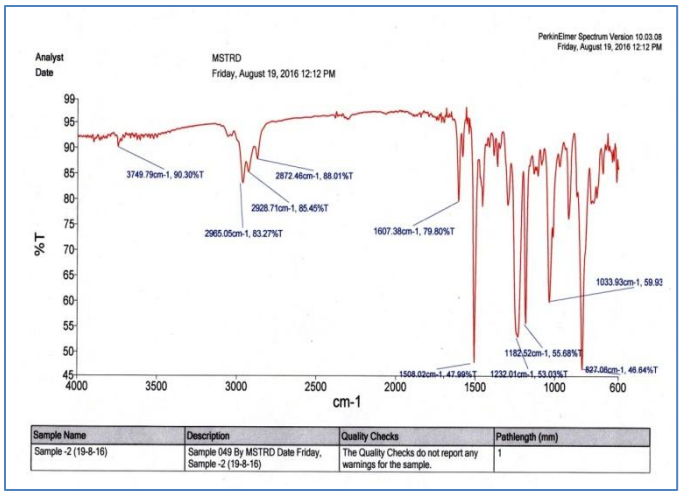


Figure 6 FT IR spectrum of epoxy resin

Table 7 FT IR Spectral Data of Epoxy Resin

Frequency (cm^{-1})		Band Assignments
Epoxy Resin	**Literature ***	
2965	3100-3000	C-H stretching
1607	1600-1585	C-C stretching(ring aromatics)
1508	1500-1400	C-C stretching(ring aromatics)
1232	1335-1250	C-H in plane bending
1033	1250-1020	C-H bending

* as Win Lab software of FT IR machine

STA Analysis

Thermal stability of epoxy resin sample was investigated by STA analyzer. The thermograph of individually epoxy resin is presented in Figure 7 and Table 8. Temperature range was occurring from 50 to 800 °C. The initial region (onset) of the STA profile at about 373.79 °C and the major endothermic peak was observed around 394.34 °C. It was completely burnt around 460.68 °C and about 93.50 % was weight loss.

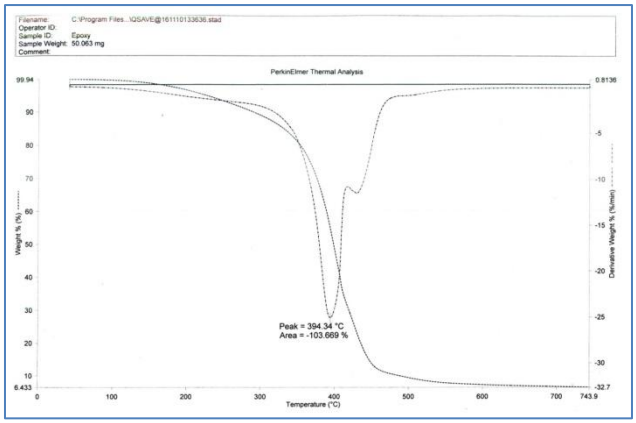


Figure 7 STA thermogram of epoxy resin

Table 8 Thermal Analysis Data of Epoxy Resin

Sample	Melting point value (°C)	Temperature range (°C)	Weight loss (%)	Nature of peak	Remarks
Epoxy resin	394.34	200-500	93.50	endo	Decomposition of cross-linked polyepoxides and graphitization

Preparation of Composites made from Jute with Epoxy Resin

Each modified waste jute fiber 120 g and various weight percentages (5, 10, 15, 20, 25 and 30 %) of epoxy resin were mixed by Henschel mixer for 2 min. The complete mixture was laid in mold. Later, this mat was carefully transferred to the hydraulic press machine for 15 min. The photographs of prepared jute fiber epoxy composites (JFE) are shown in Figure 8**.**

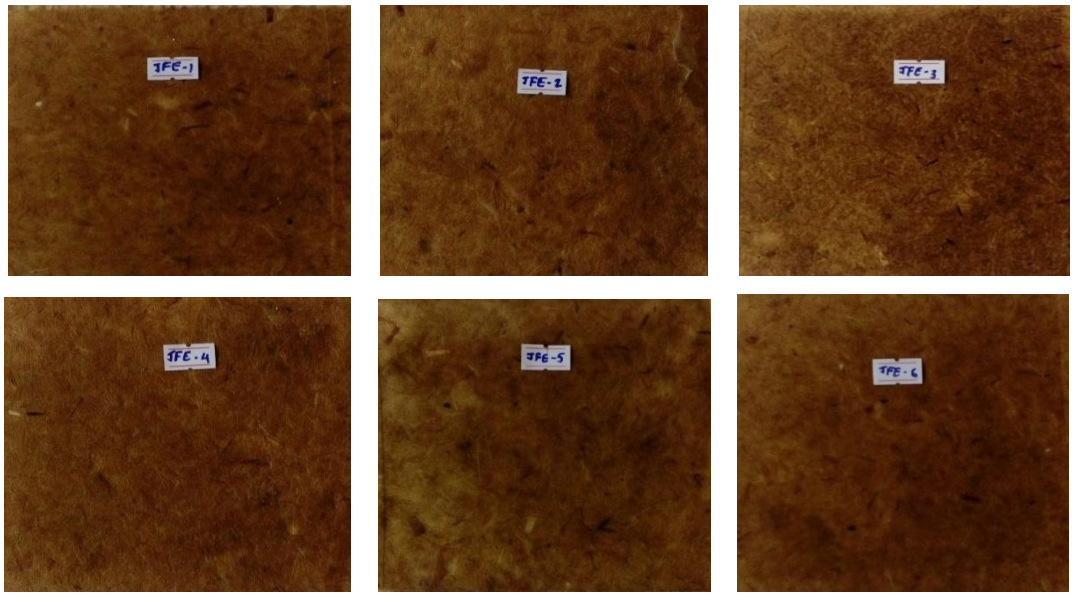


Figure 8 Photographs of jute fiber epoxy composites (JFE)

Physicochemical and Physicomechanical Properties of Composites Made From Jute and Epoxy Resin

JFE 1, JFE 2, JFE 3, JFE 4, JFE 5 and JFE 6 composites parameters such as thickness, swelling thickness, water absorption, density, modulus of rupture and hardness are presented (Table 9 and Figures 9 and 10). It can be observed that JFE 4 composite has largest modulus of rupture (MOR). The physicochemical and physicomechanical properties of composites were shown in Table 9. The quality grade composite was chosen depending on the modulus of rupture. From the results JFE 4 composite pertaining 20 % E, at applied pressure 2300 psi was higher MOR value than others. Therefore, composite JFE 4 was chosen to make the most suitable composite.

Table 9 Physicochemical and Physicomechanical Properties of Jute Fiber Epoxy Composites

Composites	E (wt %)	MOR (psi)	Thickness (cm)	Density (gcm^{-3})	Water Absorption* (%)	Swelling Thickness* (%)	Hardness Shore (D)
JFE1	5	580.34	0.72	0.7825	29.40	49.42	61
JFE2	10	785.15	0.65	0.8746	25.16	41.20	63
JFE3	15	1076.88	0.59	0.9200	22.19	33.04	64
JFE4	20	1248.20	0.60	1.2320	14.18	20.35	68
JFE5	25	1158.68	0.60	1.0962	14.99	22.08	65
JFE6	30	1099.43	0.60	0.9954	15.00	23.42	65

Applied pressure = 2300 psi
* = after 24 hour

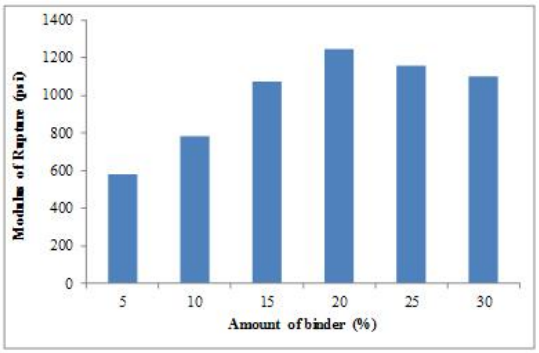


Figure 9 Modulus of rupture of JFE composites as a function of percentage of epoxy resin at 2300 psi

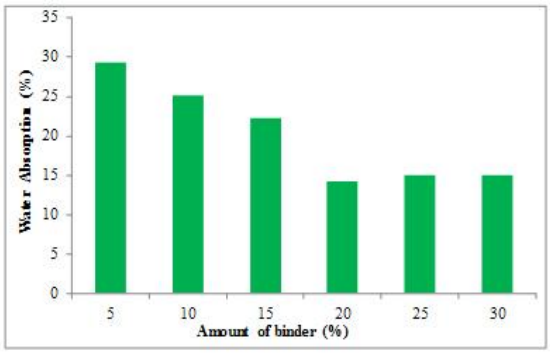


Figure 10 Water absorption of JFE composites as a function of percentage of epoxy resin at 2300 psi

Characterization of JFE 4 Composite

STA Analysis

On the basis of STA profiles, the break in temperature, corresponding dehydration, decomposition and combustion of JFE composite. The STA thermogram profiles of the thermal degradation of jute fiber epoxy composite is represented in Figure 11 and the description in Table 10. The temperature range from 50 °C to 995 °C at 20.00 °C/min. From STA curve, the

endothermic peak was observed at 361.02 °C combustion of composite. At about 500 °C the STA profiles level out, where graphitization of the residual matter may be taken place.

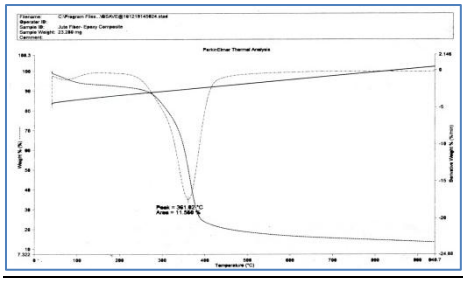


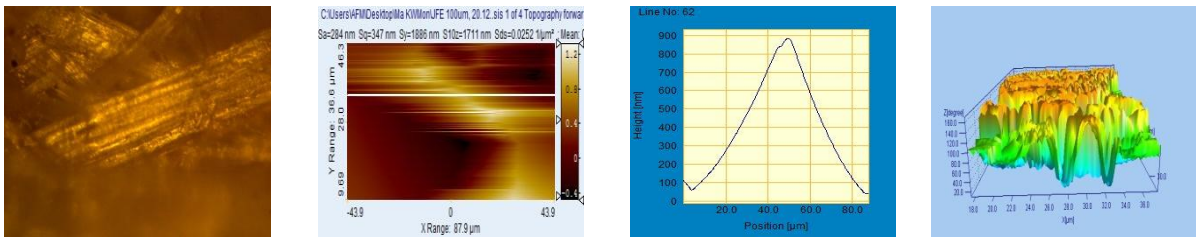
Figure 11 STA thermogram of jute fiber epoxy composite

Table 10 Thermal Analysis Data of Jute Fiber Epoxy (JFE 4) Composite

Sample	Peak Temperature (°C)	Temperature range (°C)	Weight loss (%)	Nature of peak	Remarks
	-	50-200	6.31	-	Dehydration of surface water and moisture
JFE 4	361.02	200-500	64.03	endo	Degradation and decomposition of cellulose and lignin
	-	500-800	28.06	-	Epoxy resin final residue carbon is obtained

AFM Analysis

From the AFM image of jute fiber epoxy composite, the stiffness nature of fibers can clearly observed as shown in Figure 12 and the description data in Table 11 are given. The results of epoxy surface was highlighted to build three-dimensional structure images. A roughness indicated by JFE 4 value was 900 nm which is correlated with the increasing amounts of fibrillar structure on the surface of fiber.



(a) (b) (c) (d)

Figure 12 AFM microphotographs of JFE 4 composite (a) morphology (b) topography (c) line profile (d) 3 Dimension

Table 11 Microscopic Analysis Data of Jute Fiber Epoxy (JFE 4) Composite

Sample	Surface roughness (nm)	Remarks
JFE 4	900	The stiffness nature of jute fiber

Conclusion

This study reveals that the use of lignin in the development new polymer composite materials. Improved composites were prepared from 5 % (w/v) NaOH used as surface modifiers of fibers to form modified waste jute fiber. The physicochemical properties of jute fiber (JF), epoxy resin (E) has carried out. Thermal nature and surface morphology of waste jute fiber and modified waste jute fiber were investigated according to STA and AFM techniques. According to STA thermograms, it was observed that alkali treated waste jute fiber has the most smooth surface. Composites namely (JFE) were prepared by mixing modified waste jute fiber with different proportions of epoxy resin (E) by using the cold pressing method at 2300 psi. The optimal condition influencing the preparation of composites based on the composition of binder (20 % of E), at pressure (2300 psi). It was found that the optimized JFE 4 composite possesses1248.20 psi modulus of rupture, 0.60 cm thickness, 1.2320 gcm^{-3} density, 14.18 % water absorption, 20.35 % swelling thickness, and 68 D hardness. The optimized JFE 4 composite was characterized by STA and AFM analysis. According to the STA thermogram profiles, the thermal decomposition of JFE 4 composite which can withstand the temperature of about 360 ˚C. The thermograms of STA confirmed the presence of lignin, an amorphous hydrophobic biopolymer with strong intermolecular, intramolecular hydrogen bond and cross linking of the molecules requiring more energy to breakdown resulting in good thermal stability of hybrid composites around 200 ˚C. The three dimensional structure images of composite were measured by Atomic Force Microscopy. From the experimental results revealed that the jute-epoxy exhibited better mechanical properties. It was found that these jute fiber reinforced epoxy composites were found to be quality grade composite

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