

Research Article

# Thermal Mechanical and Morphological Analysis of the Reinforced Composite Material Jute/Basalt

T Pavan Kumar<sup>1</sup>, C Udaya Kiran<sup>2</sup> and A Chennakesava Reddy<sup>3</sup>

<sup>1</sup>Research Scholar, <sup>3</sup>Professor, Department of Mechanical Engineering, Jawaharlal Nehru Technological University, Hyderabad, India

<sup>2</sup>Professor, Department of Mechanical Engineering, Bhasker Engineering College, Hyderabad, India.

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## Abstract

*In the current report, study has been conducted on the preparation and characterization of jute/basalt reinforced composites as a novel polymer matrix. The material is prepared by hand layup technique by considering the orthogonal L27 array, with 3 stages and 4 process parameters. Mechanical properties such as tensile and bending are determined and the optimal structure is evaluated by the Taguchi process. To analyze the impact of the fibre sheet, a detailed physio-chemical, thermal, morphological and mechanical characterization of the jute/basalt reinforced composite material is performed. Scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR), differential calorimetry scanning (DSC) are characterized. FTIR analysis is used to identify additives after extraction from a polymer matrix and contamination on or in a material. In order to know the amount of heat needed to raise the temperature of a sample, DSC analysis is carried out and reference is determined as a function of temperature. Similarly, DSC analysis provides information regarding Transition temperatures, glass transitions, Polymorphic transformation. The microstructural properties of the fibres appeared significantly dependent upon the relative blend ratio. DSC revealed that good interaction/miscibility existed between the two polymers. Tensile strength characterization showed that the value increased over one order of magnitude with the addition of PU in the polymer matrix, attributed to the formation of inter-fibre bonds.*

**Keywords:** Mechanical properties, DSC, FTIR, SEM

## 1. Introduction

Physical and chemical properties of materials are generally affected by conditions both external and internal, external conditions like humidity, temperature, pressure, etc and internal conditions like internal energy, enthalpy, etc. DSC analyses focuses on change in physical and chemical properties like glass transition temperature, oxidative stability, crystallization temperature and time. In Differential scanning calorimetry or DSC, analysis is made on the basis of change in temperature (thermo analytical technique) i.e. the difference in value of heat necessary to raise the temperature of sample and reference will be measured in terms of function dependent on temperature, more over the sample and reference are maintained approximately at same temperature throughout the experiment. The temperature of sample holder increases as the experiment goes on, so the program is designed likewise such that the temperature increases linearly with time.

The sample used must have predetermined heat capacity rates with respect to temperatures to be measured. Glass transition temperature can be defined as the approximate midpoint of the temperature range over which the glass transition takes place. The characterization of glass transition temperature of any material can be done easily using differential scanning calorimeter. (B. Wunderlich, *et al*, 1990) Morphological analysis is carried out to observe the fracture behavior and fiber pull out of the composite samples using scanning electron microscope. (Ershad Mistria *et al*, 2011) Castor oil was converted to maleated castor oil (MACO) without any catalyst. MACO was characterized with FTIR, NMR, molecular weight and viscosity measurement. (WU Heng *et al*, 2013) The chemical, morphological and crystal structure of the jute fibers during pre-oxidation were investigated by Fourier transform infrared spectroscopy, differential scanning calorimetry, elemental analysis, X-ray diffraction and scanning electron microscopy (Yong He *et al*, 2020) investigated the jute fiber reinforced polypropylene the combined plasticization of functionalized epoxy soybean/tung oils anhydride (ESO/TOA). Thermal and morphological study was conducted to know the material behavior.

\*Corresponding author's ORCID ID: 0000-0003-3810-9661  
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In several fields, such as aerospace, automobile and related industries, natural fibre reinforced polymer composites are used as a replacement for conventional reinforcement materials. Composite materials have essential properties, such as lower weight, low cost and competitive strength. Jute fibres have benefits such as low cost, great mechanical properties and sufficient industrial supply of all-natural fibers. Since mankind is facing the detrimental consequences of the environment, the use of natural fibers has grown over the last few decades. This sustenance Natural fibers are environmentally safe, low in weight, cost-effective, sound-absorbing and shatter-resistant. Composites consist of two or more chemically distinct components that are reinforced with other materials to make composites. The fibers serve as the main load-bearing members of composites, while the underlying matrix holds the fibers' direction and position unchanged. There are readily available cellulose fibers such as jute, banana, sisal, coir and wood. Basalt, mined from volcanic rocks, is commonly used as a replacement for e-glass and carbon fibre in the building, engineering and processing industries. Latest developments have seen a major change in the use of natural fibers in packaging products for multiple applications, taking into account growing environmental issues.

(Mohanty AK, *et al*.) A significant focus and substantial research effort is currently dedicated to exploring ways to minimize the environmental effect on the manufacture and use of composite materials, resulting in increased sustainability of composite materials. (Fowler PA, *et al*, 2006) An important primary driver is new environmental laws and changing governmental perceptions, encouraging research into more environmentally sustainable goods and processes [6]. (Wambua P, *et al*, 2003; Santulli C, *et al*, 2005) Natural fibres (such as jute, flax, cotton, kenaf, oak, bamboo, etc are primarily explored as an alternative to synthetic fibres, requiring absolute or partial replacement, as a reinforcement (mainly to glass, since carbon and Kevlar offer more specific properties, in terms of mechanical performance) [7,8]. (Cicala G, *et al*, 2009; Panthapulakkal S, *et al*, 2008) The aforementioned 'partial substitution' of glass fibres is normally achieved by hybridization, usually by stacking glass fibre reinforced layers with other vegetable fibre reinforced layers, such as hemp, jute, etc..

Basalt fibres have often been proposed as an alternative to glass in recent years, given some important advantages: these include the fact that basalt is spun directly from the molten rock and then finished with the application of sizings that are not different from those applied to glass fibres. (Santulli C, *et al*, 2007) In particular, the basalt fibre surface includes ion-exchange groups, such as hydrogen-bound silanol, which form active adsorption sites and are able to interact with components of the sizing agent. (Zhang

XQ, *et al*, 2012; Qin W, *et al*, 2015) Tensile characterization was investigated on basalt fibre rods and ropes. Comparisons have been made with the FRP findings of both materials. BFRP rods have been shown to have better mechanical properties relative to FRP rods and can thus be used in the building industry. (K Arun Prasath, *et al*, 2013) A fiber-reinforced composite content analysis was regarded by considering basalt, jute and polyester. Specimens are cut to ASTM specifications and subjected to tensile, flexural and impact checks by compression moulding processes with different stacking matrixes. It was concluded that pure basalt shows stronger tensile and flexural properties relative to pure jute fibre because it has low impact properties. (Fiore V, *et al*, 2015) The basalt fibre production technology was compared to glass fibre production technology. It has been concluded that, relative to glass fibre, the basalt processing process is non-hazardous and environmentally safe. In addition, it was observed that basalt absorbs less energy than fibre glass. (V. Fiore, *et al*, 2016) Jute-reinforced and jute/basalt-reinforced composites have been manufactured to accelerate hygro-thermal stress and UV radiation ageing. For 14, 28, 56 and 84 days, each human specimen is examined. In compliance with the ASTM standard, mechanical tests such as quasi-static flexural tests, Charpy effect tests and dynamic mechanical tests were conducted.

One of the main polymer products in the plastic family is polyurethane (PU). In recent decades, structural and non-structural applications of PUs and their varieties have become standard and efficient methods for the repair, reinforcement, restoration and maintenance of civil engineering infrastructure. The highest growth field in construction methods is called polymer materials. (M. Miller, 2005; P.K. Saxena, *et al*, 2013) Polymers can be represented, in simple terms, as extremely long molecules, which typically consist of several thousand repeating units. These products are currently being used in various fields in the building industry and many other applications, such as construction, automobile, furniture and bedding, appliances, electronics, footwear, packaging, textiles and apparel. (A.J. Raychura, *et al*, 2018; M.M. Alrashed, *et al*, 2019) PU coatings also have a large market in the coatings industry due to their unique properties, such as quick-curing properties (low gel time), corrosion resistance, high gloss. (S. McCreath, *et al*, 2018; Y. Boutar, *et al*, 2018) In addition, PU adhesives are commonly applied in construction, automobile, and decoration industries because of their self-supportive excellent bond strength, fast cure time, and weather resistance.. In this experimental work jute, basalt, polyurethane and orientation are considered as parameters to fabricate the sample and mechanical properties like tensile and flexural are observed. Further water absorption and thermal tests are carried to study the influence of polyurethane.

**2. Materials and Methods**

**2.1 Manufacturing process**

Jute fiber, Basalt fiber, Polyurethane in powder form, Epoxy Hardener (HY951) and Epoxy resin (CY230) are used for sample manufacture. Taguchi L27 orthogonal array is considered to fabricate the samples, where 4-parameters followed by 3-levels are chosen. Composite laminates were produced by a hand layup procedure,

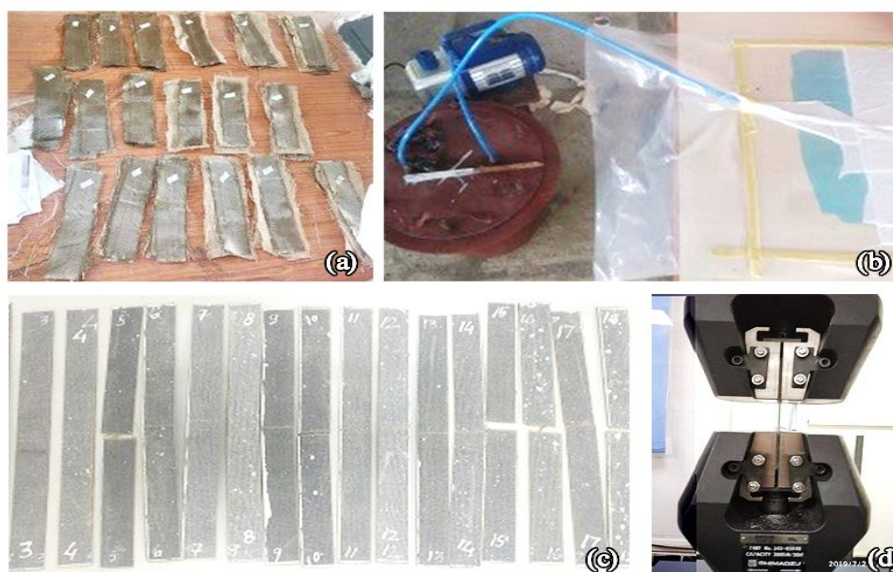
stacking dry fabric layers by hand onto planar support to form a laminate stack. Resin was applied to the dry plies after layup, where the orientation of the jute fiber will differ. After lay-up the samples are vacuum-bagged for proper curing, internal voids can be removed, uniform distribution of resin is possible and fine surface finish will be achieved. In practice, laminates underwent an initial 24 hours curing at environmental conditions then subjected to mechanical tests.

**Table 1:** Mechanical properties of process parameters

Parameters	Density (gm/cm <sup>3</sup> )	Young's modulus (N/mm <sup>2</sup> )	Poisson's ratio
Basalt	2.65	86	0.26
Jute	1.5	25	0.3
Polyurethane	0.12	0.033	0.33
Resin	1.54	3.5	0.33

**Table 2:** Selection and set of parameters for processes.

S. No	Process Parameters	Level-1	Level-2	Level-3
1	Jute	8	10	12
2	Basalt	2	3	4
3	Polyurethane	3	6	9
4	Orientation	00	450	600



**Fig 1:** (a) Selected material (b) vacuum bag with pump setup (c) samples (d) UTM setup

**2.2 Samples**

The samples are ultimately cut to ASTM specifications. Table 2 displays the working set of process parameters

selected. A minimum of five samples was tested in the case of tensile and flexural tests with planar dimensions 250x25 mm for tensile tests, 120x15 mm for flexural tests and in case of hardness three

indentations was made, an average of those were considered. Size of the sample observe for hardness test is 50x50 mm.

### 2.3 Mechanical Characterizations

#### 2.3.1 Tensile tests

The tensile strength test was conducted on Computerized Universal Testing Machine. Loading was done in displacement control mode using a 1 mm/minute velocity. From the maximum load supported until failure, the ultimate tensile strength of each sample was calculated, while the yield tensile strength of each sample was evaluated by the 0.2 per cent yield offset method: the device automatically measures the 0.2 per cent deviation from linearity, while also setting two points for the region of the stress-strain curve in which Young's measurement is measured.

#### 2.3.2 Flexural tests

Using the Computerized Universal Tester, three-point flexural tests were performed according to the ASTM D790-10 standard. Upper cylindrical support of 12.7 mm in diameter was used in the flexural rig, while the lower supports had a diameter of 6.35 mm each. 120x15 mm is the planar dimensions of the samples. From the full load carried prior to failure, the ultimate flexural strength of each sample is determined. Using a 2 mm/min velocity, loading was carried out in displacement control mode. Every sample's yield flexural strength was also determined by the 0.2 per cent yield offset process.

### 2.4. Design of experiments

Using Minitab software, statistical analysis is performed after the experimental procedure and the signal to noise ratio values of those values are tabulated. The S/N ratio can be an efficient representation in order to find the significant parameter by measuring the minimum variance. To optimize the design of experiments, the Taguchi method is simple, systematic and efficient. It is a better technique than traditional experimental design, which reduces the number of experiments, time and expense. The orthogonal array-based Taguchi technique offers a series of balanced experiments. An L27 orthogonal array was chosen for the present work, consisting of 27 rows and 13 columns. Table 1 displays the operating parameters and level of operation. The experiments consisted of 27 orthogonal array (OA) experiments.

### 2.5. Fourier-transform infrared (FTIR)

FTIR is performed on a NEXUS-570 spectrophotometer in a range of wave numbers from 4000 to 700  $\text{cm}^{-1}$  with the resolution and scanning number of 4  $\text{cm}^{-1}$  and

32 time, respectively. All samples for FTIR analysis were prepared by pressing with KBr.

### 2.6. Differential scanning calorimetry (DSC)

To maintain the sample and the reference at same temperatures, heat is needed to flow through the sample. The main reason for it is change in phase of sample in terms of temperature. Similarly when a sample is subjected to exothermic processes, less amount of heat is required to raise the temperature of sample. By observing the difference in heat flow between the sample and reference, differential scanning calorimeters are able to measure the amount of heat absorbed or released during such transitions. DSC may also be used to observe more subtle physical changes, such as glass transitions. The DSC curves are recorded on a TA instrument Q10 thermal gravimetric analyzer (TA Instruments). The measurements were conducted under a nitrogen atmosphere. The samples were first heated up from 10 to 200°C at a heating rate of 10°C/min, and then maintained at 200°C for 5 min, and finally cooled down to 0°C at a cooling rate of 10°C/min.

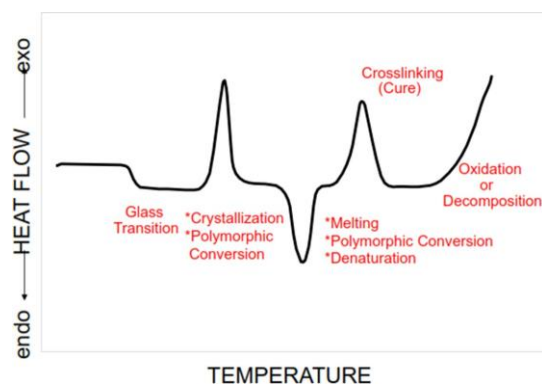


Fig. 2. General DSC curve.

### 2.7. Scanning electron microscopy (SEM)

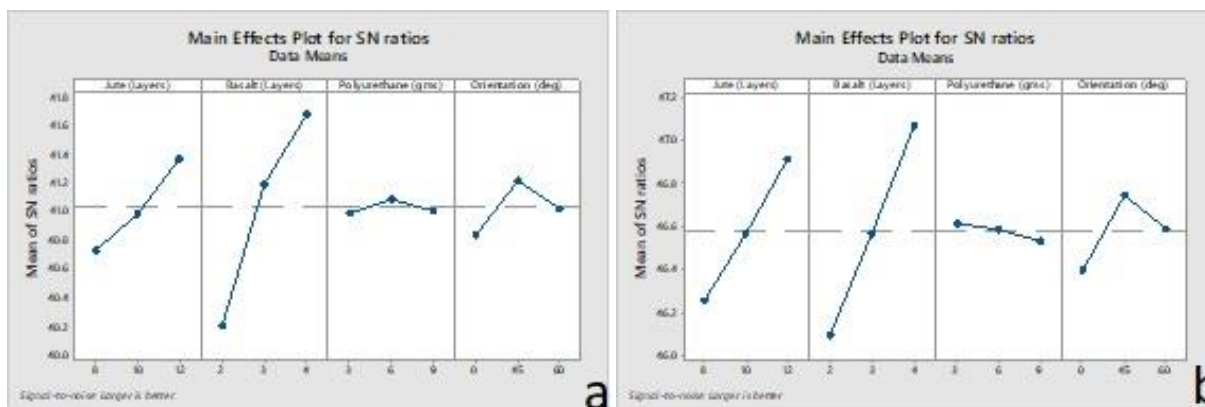
KYKY-EM6200 scanning electron microscope (KYKY Technology Development Ltd., Beijing, China) was used to image the fracture surface of the jute/basalt composites with the accelerating voltage of 25 kV. The morphology of the composites was obtained at different magnifications.

## 3. Results and Discussion

The proposed composite is developed and tested for mechanical behavior. The optimum composition is captured through Taguchi analysis. Characterization of the high strength exhibiting composition is conducted through FTIR and DSC analysis. From the experimental results, it is noted that the material is exhibiting superior strength when the orientation is maintained at 45°. The highest tensile and flexural strength are observed for the same level and parameter. The experimental results are tabulated below.

**Table 3:** Taguchi Orthogonal array with experimental results

Jute (layers)	Basalt (layers)	Polyurethane (gms)	Orientation (deg)	Tensile Strength (Mpa)	Flexural Strength (Mpa)
8	2	3	0	96.75	189.32
8	2	6	45	103.24	198.05
8	2	9	60	97.23	192.32
8	3	3	45	113.06	209.93
8	3	6	60	110.87	206.23
8	3	9	0	108.69	198.35
8	4	3	60	117.38	219.96
8	4	6	0	115.03	215.88
8	4	9	45	119.42	221.63
10	2	3	0	97.93	198.92
10	2	6	45	105.69	206.47
10	2	9	60	101.99	200.58
10	3	3	45	116.32	218.68
10	3	6	60	114.09	212.21
10	3	9	0	112.97	206.25
10	4	3	60	121.35	226.69
10	4	6	0	118.08	221.02
10	4	9	45	122.12	227.85
12	2	3	0	104.24	206.53
12	2	6	45	109.18	213.29
12	2	9	60	106.61	211.41
12	3	3	45	119.98	226.58
12	3	6	60	120.54	221.37
12	3	9	0	116.16	218.66
12	4	3	60	125.42	234.58
12	4	6	0	124.88	227.97
12	4	9	45	128.92	235.96



**Fig. 3.** Main effects plot for (a) Tensile (b) Flexural

**3.1. Effect of process parameters on mechanical properties of the composite material**

From the experimental results, it is observed that a trend is followed through the results, where the highest tensile and flexural strengths are noted when the orientation is at 45°. There is a slight improvement in the strength with the addition of polyurethane. Basalt is the highest effecting factor above all other parameters, where each layer of basalt improve the strength by 10-12N/mm<sup>2</sup>. The highest strength exhibiting composition for both tensile and flexural are 12 layers of jute, 4 layers of basalt, 45° orientation and less amount of polyurethane like 6 grams for tensile and 3 grams for flexural is observed.

**3.2. Differential calorimetry scanning (DSC) analysis**

Result of any DSC experiment is depicted in the form of a graph having both axes as temperature and rate of heat flux. Depending upon the technology used in the experiment, there are two kinds of conventions, exothermic reactions in the sample subjected to test are represented with a positive or negative peak. The curve obtained can be used for representing transition enthalpies. Integrating the peak value with transition gives this value. The enthalpy of transition can be shown by equation below:

$$DH = KA$$

Where DH is transition enthalpy, K is the calorimetric constant, and A is the area below the curve. Calorimetric constant value varies with variation in instrument, moreover it can be found by analysis of sample with transition enthalpy which has already been determined.

The DSC curve of jute/basalt fibers in nitrogen atmosphere is shown in Fig. 4. The decomposition behavior of major components of jute fibers can be seen on the curve. An endothermic peak appears due to moisture loss. Fig. 4 shows the DSC curve of jute/basalt composite. The oxidation behavior of jute fibers is clearly shown on this curve. The values observed at the peak are 508cel at 0.846w having -2804mj/mg.

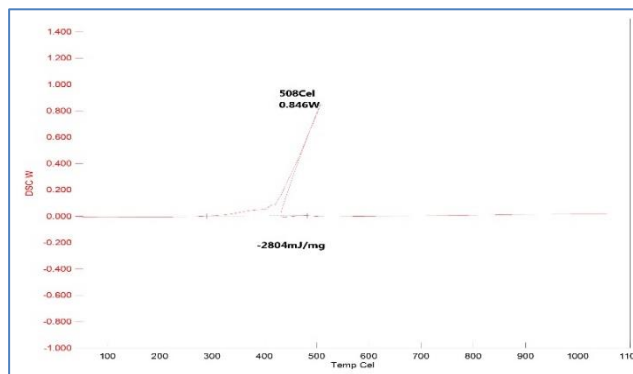


Fig. 4. DSC curve of jute/basalt fiber in nitrogen atmosphere

### 3.3. Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR of jute/basalt fibers processed at different wavenumbers are shown in Fig. 5. The % transmittance is linear initially in the later stage the curve peak positions are varying due to temperature change. The proposed fibers are exhibiting superior changes in the chemical structure.

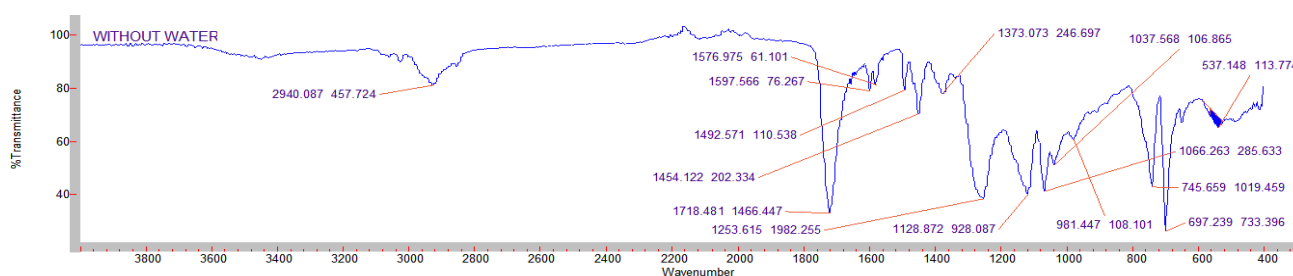


Fig. 5. FTIR of jute/basalt fibers at different temperatures

### 3.4. Scanning electron microscopy (SEM)

The sample after subjected to mechanical tests morphology study is performed to know the fiber behavior and surface of the sample. The images are captured at different magnifications and sides of the sample. The SEM images are also captured after conducting FTIR and DSC analysis.

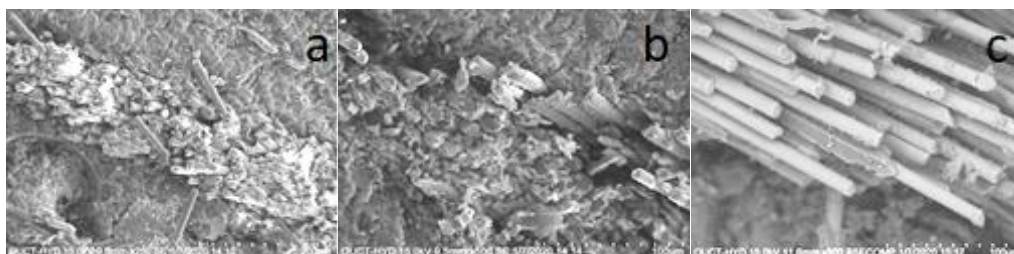


Fig.6. SEM images samples subjected to Tensile strength (a,b,c sample sides)



Fig.7. SEM images of samples subjected to Flexural strength (a,b,c sample sides)



**Fig.8.** SEM images of samples subjected to DSC & FTIR (a,b,c sample sides)

## Conclusion

In this work, for the developed hybrid composites FTIR, DSC analyses and morphology study were carried out to find the thermal stability. The following results were observed.

Fourier transform infrared spectroscopy (FTIR):

The proposed sample molecular composition and structure is determined at various wavelengths.

FTIR spectrum is recorded between 4000 and 400  $\text{cm}^{-1}$ . Which is used to obtain infrared spectrum of absorption, emission, and photoconductivity of the sample.

Differential scanning calorimetry (DSC):

The Transition temperature of the composite increases with increase in amount of fibre.

It has been found out that the Transition temperature of sample is superior.

Adding fibres to the already existing composites (while synthesis) decreases the transition temperature but increases the recrystallization temperature.

During the phase of recrystallization, it has been found that the exothermic energy of sample is least.

Product having high onset temperature and low exothermic energy is best suited in the experiment.

Scanning electron microscopy (SEM):

After each examination, microscopy images were taken to study the conduct and intactness of internal and external fibers within two layers. Similarly, samples were also subjected to thermal analysis with superior texture, which is evident from the images.

When it comes to thermal analysis, polyurethane serves as a superior element. In addition, the properties of the proposed jute/basalt reinforced composite can also be strengthened by PU, such as adhesion and resistance to high-temperature deformation. Thermal experiments on composites have shown that good thermal stability is displayed by the proposed composite material. The development of natural fiber reinforced polymer composites with a wide range of industrial applications, in view of these few superior marks?

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