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Effect of different lignocellulosic fibre based needle punched nonwovens on mechanical properties of bio-reinforced composite

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Bio-reinforced composites have been prepared using polyester resin matrix and cross-laid needle-punched nonwoven fabrics of different lignocellulosic natural fibres (Jute, flax, sunhemp, kenaf, ramie, sisal, coir and pineapple) as well as jute-polypropylene and jute-acrylic blend reinforcement. The composite properties, such as tensile strength, flexural strength and impact strength, have been evaluated. The findings show that the tensile strength and stiffness are very high in case of sisal (211 MPa and 21 GPa) and flax (304 MPa and 30 GPa), which is almost comparable with e-glass. Properties of sunnhemp, coir, pineapple, and kenaf are found superior to ramie, jute and jute blend. The impact strength of pineapple composite (751 kJ/m^2) is found very high. The mechanical properties of woven fabric composite are much lower than nonwoven fabric. Cross- direction of composite from nonwoven shows superior mechanical properties as compared to that of the machine direction. Synthetic fibre shows low stiffness (1 GPa) and flexural modulus (1.2 GPa) but very high impact strength (655 kJ/m^2). Hence, most of the lingo-cellulosic fibres are suitable for composite reinforcement, especially where the high performance of glass-reinforced plastic is not essential.

Keywords: Bio-reinforced composite, Fibre-reinforced plastic, Lignocellulosic fibre, Mechanical properties, Natural fibres, Nonwoven, Unsaturated polyester resin

1 Introduction

Fibre-reinforced composite is gaining popularity as a substitute of metal or wood due to its light-weight and superior mechanical property[1]. Manmade fibres are predominant in this field. Presently, natural materials are getting importance day by day due to their ecological aspects[2] and potential to replace synthetic fibre reinforced plastics at a lower cost, across a wide range of applications with improved sustainability[3]. Natural fibre compositions, surface properties, mechanical behavior, and variability have a great effect on the properties of fibre reinforced plastic. Hence, fibre selection, processing, orientation and fibre-resin interfacial strength affect the composite properties. Natural fibre types are commonly categorized based on their origin viz plant, animal or mineral[4]. Mineral-based fibres are now avoided due to associated health issues and banned in many countries[5]. Generally, the strength and stiffness of animal fibres are much lower compared to plant fibres. Moreover, some of them are costly and are less readily available.

Fibre-reinforced composite with high specific stiffness and strength can be produced by adding the tough and light-weight natural fibre into polymer[6]. On the other hand, natural fibres are not free from problems and show notable deficits in properties[7]. The natural fibres structure permits moisture absorption from the surroundings which causes weak bindings between the fibre and polymer. Furthermore, the couplings between natural fibre and matrix are considered a challenge because of the chemical structures of both fibres and matrix. Accordingly, natural fibre modifications using specific treatments are certainly necessary. Extensive research has been carried out to achieve improved interfacial bonding in natural fibre composites which can be largely divided into physical and chemical approaches. Physical approaches include corona, plasma, ultraviolet (UV), heat treatments electron radiation and fibre beating. Chemical treatments include alkali, acetyl, silane, benzyl, acryl, permanganate, peroxide, isocyanate, titanate, zirconate and acrylonitrile treatments and use of maleated anhydride grafted coupling agent[8-10]. These modifications are generally centered on the utilization of reagent functional groups which have the ability for responding of the fibre structures and changing their composition. As a result, fibre modifications cause a reduction in moisture absorption of the natural fibres which leads to an

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improvement in compatibility between the fibre and the matrix [10, 11].

In this study, composites have been made using polyester resin matrix and cross-laid needle-punched nonwoven fabrics of different lignocellulosic natural fibres, such as jute, flax, sunhemp, kenaf, ramie as stem/bast fibres, sisal, pineapple as leaf fibre, and coir as seed fibre as well as jute-polypropylene and jute-acrylic blends. These natural fibres are strong, coarse and rigid with low extensibility, which makes them suitable to act as reinforcing material in the fibre reinforced plastic composites. Furthermore, plant fibres are process friendly (less wear and tear) and grown in many countries. Composites have been prepared by hand lay-up technique. Composite properties concerning tensile strength, flexural strength and impact strength have been evaluated. A attempt has also been made to compare the performances of polyester reinforced composites based on different natural fibres as well as synthetic and glass fibre blends.

2 Materials and Methods

2.1 Materials

Eight Indian origin commercially available natural fibres, viz jute (*Corchorus olitorius*) , flax (*Linum utisasimum*), sisal (*Agave sisalana)*, sunhemp (*Crotaleria juncea*), coir (*Cocos nucifera)*, kenaf (*Hibiscus cannabinus*), ramie (*Boehmeria nivea*) and pineapple (*Ananas comosus*) as well as three synthetic fibres viz polypropylene, acrylic and E-glass were collected for using as reinforcing material. General purpose unsaturated polyester (Industrial grade, FR brand, pale colour liquid supplied by Yash composite solutions, New Delhi) resin was used as matrix of jute reinforced plastics (JRP). For composite preparation, commercial grade methyl ethyl kitone peroxide, cobalt napthanate, waxpol and polyvinyl alcohol were used as catalyst, accelerator and mould releasing agent respectively.

2.2 Methods

2.2.1 Evaluation of Fibre Properties

All the above-mentioned natural fibres were processed in the jute spinning system to make carded sliver and then tested for properties of fibres from sliver. Fibre strength, modulus, and elongation-at-break were evaluated on Instron Tensile Tester (Model No. 5567) following ASTM D3822-01 (test length, 20 mm; crosshead speed adjusted to break in 20±3s). Linear density was tested using the gravimetric method (ASTM D1577-01). Moisture content was calculated considering the bunch of sample in the standard atmosphere and oven-dry condition (ASTM D2495-07). Diameter has been measured using a projection microscope (Radical Projection Microscope by Radical Scientific Equipments Private Limited).

All the tests were conducted at 65±2% relative humidity and 27±2 ^{0}C temperature after conditioning. Average of 30 tests of all properties was calculated (Table 1). The load elongation diagrams of those fibres and resin are shown in Fig. 1.

2.2.2 Measurement of Contact Angle and Work of Adhesion

The phenomenon of wetting or non-wetting of a solid by a liquid is better understood by studying the contact angle[12, 13]. The drop of liquid forming an angle may be considered as resting an equilibrium by balancing the three forces involved, namely the

Table 1 — Properties of natural and synthetic fibres

Fibre	Linear density tex	Bulk density g.cm $^{-3}$	Average length mm	Tensile strength MPa	Young's modulus GPa	Breaking extension %	Specific strength MPa/gcm^{-3}	Specific young's modulus GPa/gcm^{-3}	Moisture content %
Ramie	0.72	1.52	1075.6	670.6	86.3	2.97	441.2	57.8	8.43
Sisal	21.05	1.47	894.3	695.3	23.5	2.33	473.0	16.0	11.14
Hemp	10.62	1.50	39.8	883.8	65.8	1.61	589.2	43.7	10.54
Coir	27.95	1.21	72.8	179.5	5.6	21.72	148.3	4.6	9.20
Flax	2.67	1.50	581.3	1131.4	63.2	2.18	754.2	42.1	7.02
PALF	3.29	1.52	56.1	835.3	34.3	8.33	549.5	22.5	9.26
Kenaf	2.41	1.45	50.6	664.4	35.8	1.67	458.2	24.7	12.08
Jute	2.29	1.45	51.5	619.5	38.4	1.68	427.2	26.5	12.46
Polypropylene	1.70	0.91	100.0	39.7	15.5	42.03	43.6	17.0	0.09
Acrylic	1.70	1.17	100.0	31.4	21.8	38.32	26.8	18.6	1.00
E- glass	-	2.53	Continuous	2463.4	70.0	2.50	973.7	27.7	0

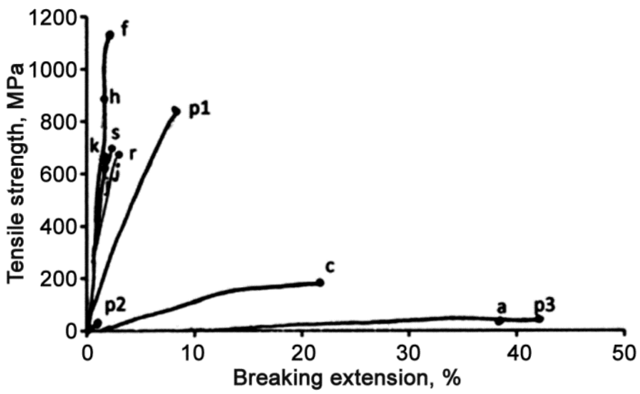

Fig. 1 — Strength-elongation curves of fibres and resin (r-ramie, s- sisal, h- sunhemp, c-coir, f-flax, p1 – pineapple, k- kenaf, j- jute, p3 – polypropylene, a- acrylic, p2 –polyester resin)

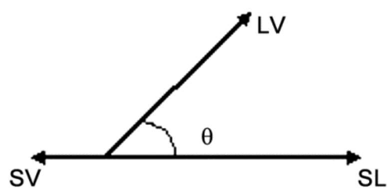

Fig. 2 — Balancing of forces for the equilibrium of liquid drop

interfacial tensions between solid and liquid (SL), between solid and vapour (SV) and between liquid and vapour (LV). Contact angle (θ) is the angle included between the tangent plane to the surface of the liquid and to the surface of the solid, at any point along their line of contact (Fig. 2).

The contact angle may be related to the surface energies (γ's) of the three interfaces by Young's equation, as shown below:

$$\cos \theta = (\gamma_{sv} - \gamma_{sl}) / \gamma_{lv}$$

The contact angle was measured by Sissilidrop method [14] using Wild Leitz Projection Microscope and a goniometer eyepiece. Liquid drops for contact angle measurement of some natural fibres are shown in Fig. 3.

A thermodynamic parameter work of adhesion (W_{SL}) was determined by using the following Young-Dupre equation [14] for low energy surfaces:

$$W_{SL} = \gamma_{lv} (1+ \cos \theta)$$

where γ_{lv} is the surface tension between liquid and vapour (dynes/cm); θ, the contact angle (degree); and W_{SL}, the work of adhesion between solid and liquid surfaces (ergs/cm^2)

The degree of wetting, called specific wettability, was expressed by $\gamma_{lv} \cos \theta$. The spreading coefficient was calculated using the following relationship:

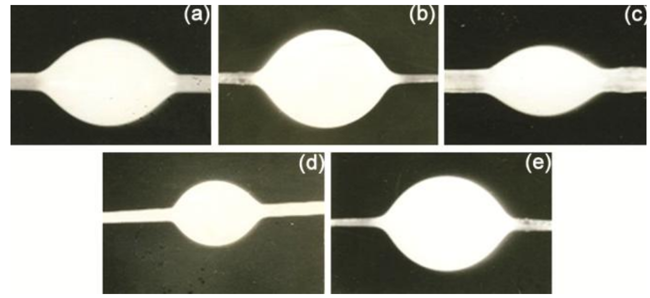

Fig. 3 — Liquid drops for contact angle measurement of (a) jute, (b) ramie, (c) flax, (d) sisal and (e) sunhemp

$$S_{SL} = W_{SL} - 2\gamma_{lv}$$

where S_{SL} is the spreading coefficient; W_{SL} , the work of adhesion; and γ_{lv} , the surface tension. Surface tension of binders was measured by tensiometer.

2.2.3 Preparation of Fabrics

Grey natural fibres were scoured with sodium hydroxide (1%) and Ultravon JU (2 mL/L) at the boil for 60 min using 1:20 material-to-liquor ratio. After drying, the scoured fibres were sprayed with 30% water and processed through jute softener, breaker card, finisher card following the conventional jute spinning system. Fibres from finisher card was used to make the needle punched nonwoven and fed to Dilo nonwoven plant consisting of carding machine, cross lapper and needle loom for mechanical entanglement. Finally, 500 g/m^2 fabric was made using 25 gauge RB (regular barb) foster needles with 100 punch/cm^2 punch density and 8 mm depth of needle penetration. Synthetic fibres were hand opened and evenly fed to lapper of nonwoven card. Hand-opened synthetic fibre and finisher carded jute fibre were blended before feeding to nonwoven card to make blended fabric.

2.2.4 Preparation of Composites

Fabrics were dried in a hot-air oven up to approximately 3±1% moisture content. Composite sheet of (30×30) cm was prepared by hand lay-up technique using a male-female type mould, tighten by the screw. Required layers of needle-punched nonwoven were cut into the desired dimension and then put on the mould after applying mould releasing chemical. Then polyester resin was spread on nonwoven as evenly as possible along with 2% catalyst and 2% accelerator. The curing time was 30 min at 60 °C.

2.2.5 Evaluation of Composites

The tensile properties were evaluated on the Instron Tensile Tester following the ASTM standard

(D683-86). The gauge length was 50 mm, test width 6 mm and crosshead speed 5 mm/min. Average of 20 tests was reported. Tensile strength and modulus were calculated using the following equation:

Tensile strength (MPa) = Maximum load (N) /Initial cross-sectional area of specimen (mm^2)

Tensile modulus (GPa) = [Stress (MPa)/Strain] × 10^{-3}

A three-point loading system utilizing center loading on a simply supported beam was used to evaluate flexural properties on Instron Material Testing System following the ASTM Standard (D790-81). Sample dimension 80×10 mm, support span 64 mm and rate of crosshead speed 1.7 mm/min were used. Following parameters (average of 10 tests) were calculated:

Flexural strength (MPa) = $3PL/(2bd^2)$
Flexural modulus (GPa) = $L^3m/(4bd^3) \times 10^{-3}$
Maximum strain (%) = $6Dd/L^2 \times 100$

where *P* is the load at rupture (N); *L*, the support span length (mm); *b*, the width of the specimen at the centre of support span (mm); *d*, the depth or thickness of specimen at the centre of support span (mm); *m*, the slope of tangent drawn at the initial portion of load-deflection curve (Nmm^{-1}); and *D*, the maximum deflection at the centre of specimen (mm).

The impact strength was evaluated on IZOD-Type Cantilever Beam Impact Tester following the ASTM standard (D 256-88). Average of 10 tests was reported. Water adsorption was tested by complete dipping a (15×15) cm block of composite in water for 60 days. The sides were completely blocked by a coating of resin. Average of 5 tests was reported.

3 Results and Discussion

3.1 Fibre Properties

Table 1 shows the properties of some natural and synthetic fibres including the main type of glass fibre (E –glass). It can be seen that the linear density of fibres varies from 0.72 tex to 27.95 tex. The polypropylene and acrylic are fine fibres. The ramie is finer to those synthetic fibres. Coir, hemp, and sisal are very coarse with linear density more than 10 tex. All the lingo-cellulosic fibres have almost similar density except coir. Coir is lighter because it has numerous hollow spaces inside the fibre cross-section. Synthetic fibres are further lighter but glass fibre density is highest, i.e. 2.53 g/cm^3. Ramie, sisal, and flax are very long fibres followed by synthetic fibres. Average length of other fibres is lower than 100 mm. Specific mechanical properties are the highest for flax and glass, but other fibres also have very good properties which are suitable for composite except synthetic fibres and coir. The moisture content of synthetic fibres is very low, whereas those of sisal, hemp, kenaf, and jute are higher than 10% (measured in oven dry method). The properties of polyester resin (density 1.35 g/cm^3, tensile strength 23.1 MPa, strain 0.95%, tensile modulus 3.1 GPa, flexural strength 64.4 MPa, displacement 7.32% , flexural modulus 3.2 GPa, surface tension 37.13 dynes/cm), which is used as a matrix, show low strength and low modulus brittle material.

The above fibre properties are mainly governed by its physical and chemical structures. Table 2 shows that all the natural fibres considered here are cellulose-based and having hydroxyl functional group. The second-largest component is adhering material lignin having phenolic methoxyl and hydroxyl functional groups. Wax in sisal and PALF are 2-3%.

Generally, higher performance is achieved with varieties having higher cellulose content and with cellulose microfibrils aligned more in the fibre direction, which tends to occur in bast fibres (e.g. flax, hemp, kenaf, jute, and ramie) that have higher structural requirements in providing support for the stalk of the plant[15] (Table 2). Strength and stiffness of natural fibres are generally lower than that of glass fibre. However, the specific properties compare more favourably; specific Young’s modulus can be higher for natural fibres and specific tensile strength can be compared well with lower strength E-glass fibres.

Although fibres are normally stronger and stiffer than the matrix, strength and stiffness of the composite are generally found to increase with increased fibre content (Table 3). Strength –elongation curves of fibres and resins are shown in Fig. 1.

3.2 Structure of Fibres

The surface morphology of reinforcing material plays a great role in composite properties. Most of the natural fibres have a rough surface. Sisal and pineapple leaf fibre (PLAF) surface are smoother than others. PALF has a scaly, cellular structure with vegetable matter intact. The rough surface of ramie fibre is characterized by small ridges, striations, and deep fissures. The cells of sunhemp fibre are cylindrical, and are marked here and there by joints. Coir fibre morphology reveals cracks, voids and

Table 2 — Chemical composition of natural fibres [26, 27]

Fibre	α-Cellulose %	Lignin %	Hemicellulose %	Pectin %	Ash %	Waxes %	Microfibrillar angle deg
Ramie	68.6–91.0	0.6–0.7	5.0–16.7	1.9	—	0.3	7.5
Sisal	47.0–78.0	7.0–11.0	10.0–24.0	10.0	0.6–1.0	2.0	10–22
Hemp	57.0–77.0	3.7–13.0	14.0–22.4	0.9	0.8	0.8	2.62
Coir	37.7- 39.1	31.4 -32.3	24.4- 24.7	0.5	1.4	1.1	-
Flax	71.0	2.2	18.6–20.6	2.3	—	1.7	5–10
PALF	67.1–69.3	14.5–15.4	-	1.2	0.9	3.2	
Kenaf	37.0–49.0	15.0–21.0	18.0–24.0	—	2.0–4.0	—	—
Jute	41.0–48.0	21.0–24.0	18.0–22.0	—	0.8	0.5	8

Table 3 — Properties of composites

Reinforced fibre	Fibre content %	Tensile strength, MPa	Stiffness GPa	Flexural strength, MPa	Flexural modulus, GPa	Izod impact strength, kJ/m^2	Bulk density g. cm^{-3}
Ramie	43.35	43.59	1.8	47.41	2.2	49.3	1.159
Sisal	32.14	211.63	20.7	290.48	22.6	129.5	0.954
Sunnhemp	31.93	52.42	4.9	86.73	4.4	210.3	1.305
Coir	46.33	101.68	3.8	114.81	4.1	56.2	1.024
Flax	35.72	304.37	30.5	218.26	18.3	157. 8	1.147
PALF	31.03	170.62	6.2	80.62	3.7	751. 1	1.362
Kenaf	39.30	46.18	5.6	58.70	4.6	39.8	1.202
Jute (m/c)	40.58	52.45	2.2	54.67	2.7	58.4	1.194
Jute (cross)	40.58	57.63	2.5	58.52	2.2	96.7	1.194
Jute (woven)	36.73	39.52	1.5	50.94	1.7	76.5	1.080
Jute (nonwoven +woven)	37.71	54.57	3.1	45.29	1.8	70.2	1.109
Jute:PP (1:1)	42.37	40.30	1.5	52.66	1.1	318.6	0.840
Jute:acrlic (1:1)	43.27	46.38	2.0	56.50	2.1	121.8	0.916
Polypropylene	48.17	21.87	1.0	27.37	1.2	655.1	0.877
E-glass	60.42	695	31	450	21	107	1.841

parallel ridges. Jute fibre cell-surface is smooth, but disfigured here and there by nodes and cross-markings. The fibres are coated with a layer of woody material. Flax fibre cells are transparent, cylindrical tubes which may be smooth or striated lengthwise and without any convolutions. There are swellings or 'nodes' at many points, and the fibres show characteristic cross-markings. The tie marks on the fibre surface of each bundle consist of several fibrils[16-24].

3.3 Structure of Needled Nonwoven

During needling, barbed needles are continuously pushed into and through web material. Some fibres are held by barbs, and their orientation is altered as they transfer into the vertical plane of the resulting fabric. This orientation of some fibres into a vertical plane and the continued presence of some fibres in both planes produce a coherent structure. Many fibres that are reoriented remain partly in the horizontal plane. It is thought that such behaviour is important in the realisation of maximum fabric strength. Photographs of individual fabric surface and the composites made from those fabrics are shown in Figs 4 and 5 respectively.

3.4 Effect of Natural Fibres as Reinforcement

Table 3 shows that when fibre content is lower, the resin content is higher, and this is possible with high resin absorbency of the fibre. It affects the bulk density of composite. It is also substantiated by the specific wet ability data (Table 4). The changes in contact angle are mainly concerned with chemical and physical characteristics of fibre surface and the surface energy of polyester resin. A surface roughness brings about a reduction in contact area, leading to reduced strength and formation of air pockets.

Interfacial bonding between fibre and matrix plays a vital role in determining the mechanical properties of composites. Since stress is transferred between the matrix and fibres across the interface, good interfacial bonding is required to achieve reinforcement. Tensile strength and stiffness are very high in case of sisal and flax which is almost comparable with e-glass. It

Fig. 4 — Surface photographs of nonwoven from natural fibres (in Nikon coolpix A 10 camera without magnification)

Fig. 5 — Surface photographs (top view) of natural fibre- polyester composites (in Nikon coolpix A 10 camera without magnification)

Table 4 — Wetting characteristics of fibres with polyester resin[a]

Fibre	Contact angle (θ), deg	Work of adhesion mJ/m^2	Specific wettability mJ/m^2
Ramie	63.06	53.95	16.82
Sisal	42.95	64.31	27.18
Sunhemp	42.30	64.59	27.46
Coir	64.95	52.85	15.72
Flax	47.84	62.05	24.92
PALF	40.86	65.21	28.08
Kenaf	56.71	57.51	20.38
Jute	55.74	58.03	20.90
PP	67.06	51.60	14.47
Acrylic	64.11	53.34	16.21
E-glass	72.29	48.42	11.29

[a]Surface tension of polyester resin 37.13 mJ/m^2.

can be substantiated by high specific wet ability (Table 4), fibre mechanical properties (Table 1) and cellulose content (Table 2). For bonding to occur, fibre and matrix must be brought into intimate contact; wettability can be regarded as an essential precursor to bonding. As per mechanical properties, sunhemp, coir, pineapple, and kenaf are superior to ramie, jute, and jute blend. Fibre mechanical properties are mainly responsible for this.

The impact strength of PALF is very high due to fineness and strength of fibre and better resin absorbency. Polypropylene-polyester composite impact strength is also very high due to fibre fineness and elongation. Jute-pp blend as reinforcement also shows good impact strength, which is better than jute but inferior to all-polypropylene.

3.5 Effect of Reinforcement Structure

Jute cross-laid needle punched nonwoven has been used as a reinforcing agent in the composite. During laying, fibres are oriented either in 60^0 or 120^0 angle in machine direction. During needling and stretching, the average orientation is, to some extent, moved towards the machine direction. The cross- direction shows higher mechanical properties as compared to that in machine direction due to fibre length orientation. But flexural modulus shows lower value, which may be due to slippage of fibres[25]. Jute reinforcement has been made as nonwoven (needle punched), woven (hessian) and combination of woven and nonwoven fabrics. Its properties in the machine direction are reported in Table 3. It is observed that the fibre content is higher in the nonwoven fabric due to better wettability. The mechanical properties of woven fabric are much lower than nonwoven fabric due to lower fibre matrix interface and poor

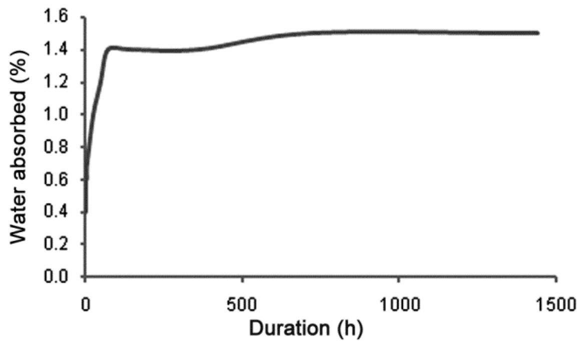

Fig. 6 — Water absorption of jute fibre composition

penetration of the matrix in the inside of yarn structure. But woven fabric shows higher impact strength due to the higher transverse strength bearing capacity of twisted fibre bundle as yarn. When these two structures are combined as layers, all the properties are improved. Bulk density of the woven fabric is lower than the density of nonwoven composite. Better wetting and higher fibre-resin interface play a significant role in better performance of composite.

3.6 Comparison with Glass Fibre Composite

When natural fibre composites are compared with glass fibre composites, mechanical properties of glass fibre are much higher than other natural or synthetic fibre composites. But the density of glass fibre composite is very high, and hence the specific mechanical properties of natural fibre composites are slightly lower than the glass counterpart (Table 3). Hence, when durability and very high mechanical properties are not required, the natural fibre composites can replace glass because of eco-friendliness, lesser abrasive damage, and low cost.

3.7 Comparison with Synthetic and its Blend with Natural Fibre Composite

Composites were made from 100% polypropylene, 50:50 blend of jute- polypropylene and 50:50 blend of jute- acrylic. If their properties are compared with 100% jute, the stiffness and flexural modulus are highest in all jute composite followed by jute-acrylic, jute- polypropylene and all polypropylene sequentially. The bulk density follows the similar trend (Table 3). But impact strength of all polypropylene is much higher and shows about 50% reduction with the introduction of jute. This is basically due to high elongation, low wettability and smooth surface of synthetic fibre.

3.8 Wetting of Jute Composite

Rate of water absorption of jute fibre reinforced composite is shown in Fig. 6. Instantaneous 0.6% of water intake is mainly the water adhered with the block and that is not absorption. Very slowly, it absorbs water and reaches to 1.4% after 72 h. Then there is no further significant absorption observed. Actual water going inside is not more than 0.9% which is well within the limit for good performance.

4 Conclusion

4.1 Tensile strength and stiffness are very high in the case of composites from sisal and flax, which is almost comparable with e-glass.

4.2 According to mechanical properties, composites from sunhemp, coir, pineapple, and kenaf are superior to ramie, jute and jute blend.

4.3 The impact strength of pineapple leaf fibre reinforced composite is very high.

4.4 Cross direction of nonwoven reinforcing material shows higher mechanical properties of the composite as compared to the machine direction. Therefore, fibre laying plays an important role in the composite properties.

4.5 The mechanical property of a woven fabric is inferior to the nonwoven fabric.

4.6 Composites from synthetic fibre & its blend with natural fibres show low stiffness and flexural modulus but very high impact strength.

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