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Cellulose Extraction and Synthesis of Epoxy Polymer Composite

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ABSTRACT:

The global demand in industries leads to the development of cellulose reinforced polymer (NFRP) composite and its manufacturing has been an eclectic area of research because of its high strength to weight ratio. It is a preferred choice due to its wide applications in automotive industry. Several natural fibers such as jute, sisal, flax and hemp have been used directly or indirectly with the polymer to form the NFRP. Previous studies are upon how to introduce the natural fiber as the reinforcement in polymer composite. Recently instead of natural fibers, cellulose which was the basic structural element of natural fiber has gotten the interest of researchers and engineers as a reinforcement material in cooperating into polymer matrix to form cellulose reinforced polymer composite (CRPC). This investigation aims to develop high performance materials using cellulose of jute and sisal fibers as reinforcement and epoxy L-12 as matrix. The cellulose from raw jute and sisal fiber were extracted using chemical processing and epoxy is taken as matrix. Mechanical characterization was done to examine its tensile, impact and flexural strength at three different proportions of matrix and epoxy. The investigation shows that CRP composite is a promising replacement for NFRP composite in the manufacturing of automotive components due to its high strength to weight ratio and hardness. This work may increase the demand of CRPC in automotiveindustry.

Keywods: NFRP, Epoxy, Fiber treatment, cellulose reinforcement, Epoxy composite.

INTRODUCTION

In the current scenario of materials engineering and design field, the environmental awareness brought life to several composite materials in the market for automotive and other applications. Even though the environmental factors have a valiant role in the composites properties, also the mechanical aspects should be considered purposefully. The researchers experimented with various materials such as polymers, composites, glass reinforced polymer composite all along the way to ecofriendly production regarding the needs to be done. These experiments with materials finally resulted in natural fiber reinforced polymer composites (NFRP), polymer matrix but along the natural reinforcement. Several investigations were carried out with these NFRP to anchor the facts that they are the so called revolutionized composites in the materials market. The industries

focus on the development of composite materials using natural -fibers like jute, coir, sisal, pineapple, ramie, bamboo, banana etc., for the applications in low-load conditions. Natural Fiber Reinforced Polymer (NFRP), the wondermaterial composite replaces the conventional materials because of its properties such as lightweight, high strength-to- weight ratio and stiffness. The replacement of steel with composite can influence the 60-80 percentage of component weight and 20 - 50 weight percentage with aluminium components [1]. The attractive mechanical and tribological characteristics of NFRP make it as a promising material in the aerospace as well as automotive industry. Among the various material category, the natural fiber composites are the

dominant evolving with wide range of applications from day to day products to complex

niche applications. In the last decade, these composites have experienced rapid growth in the European automotive market, and this trend appears to be global in nature [3]. The benefits and characteristics obtained from FRPs are high strength - light weight, corrosion resistance, dimensional stability, parts consolidation and tooling minimization, minimum finishing required. design flexibility, moisture low absorption [2]. The automotive industry is one of the major consumers of natural compositematerialintheirproductswhichareusedfor interiorapplications, such as door panels and trunkline rsetc.About

1.5 million of vehicles are already using vegetable fibers such as jute, hemp, kenaf, as а reinforcement of thermoplastic and thermosetting polymers [8]. The natural fibers show scatter in their properties, because of their inherent properties and the conditions of processing. To ensure the quality of fibers used in automotive industries, fiber quality assurance protocols are used, that is by ensuring that both fiber nonuniformity and dimensional variability between production batches does not affect he mechanical properties [7]. Natural fibers have the advantage of low density, reduction in tool wear. hightoughness, ease of separation, stiffness, impact resistance, flexibility and modulus [8]. Allnatural fibers have complex surface morphologies and good mechanical/physical properties. Some natural fibers have complex surface morphologies like piassava fiber which show different bonding characteristics at the polymer fiber interface. Mostly natural fibers are lingo cellulosic in nature and isgood to know the individual concentration of these components while utilizing in the composites. To ensure better adhesion of fibers in the matrix, the ash content and the wax should be minimum. The presence of hydroxyl groups of the cellulose in natural fibers imparts the hydrophilic nature which promotes the moisture absorption [9]. Among the various natural fibers, jute has a wood like characteristics as it is a bast fiber. They have a high aspect ratio, high strength to weight ratio and good insulation properties. The jute fiber reinforced polymer composite has tested for door, window, furniture, corrugated sheet, I-shaped beam, trenchless rehabilitation of underground drain pipes, water pipes, false roofing and floor tiles [12].Recently Volvo has put an initiative by producing cellulose based cargo floor tray which results in improvised noise reduction [10]. As in the case of FRP composite the bonding interfacial adhesion between the natural fiber and the polymer matrix is affected by mechanical interlocking, attractive forces, and the chemical bonds between the natural fiber and the resin. The development of natural fiber reinforced thermoplastic system composites is inhibited by the upper temperature at which the fiber can be processed and significant difference between the polymer matrix and the surface energy of the fiber. Discoloration, volatile release, embrittlementof the cellulose content and poor interfacial adhesion willoccur due to the high temperature introduction of composites [3]. Thermosetting polymers undergo some chemical change on heating and convert themselves into an infusible mass. These thermoset resins offer excellent resistance to water, organic solvents and alkalis. They are also designed for low and high temperature applications [2]. The commonly used thermoset resins for automotive applications are polyester, vinyl esters and epoxies. Epoxy resins offer high performance and resistance to environmental degradation and these have wider appeal in the automotive industry. The extended range of mechanical properties can make them the cost /performance choice [2]. Apart from that, they also offer high strength, low shrinkage, effective electric insulation and low toxicity. In general, the epoxy resin consists of base resin, curatives and modifiers. The base resin is thought as a molecule containing three membered rings, consisting of one oxygen atom and two carbon atoms. Curatives are the materials to which the epoxy reacts and most commonly used curatives are amines amine derivatives and anhydrides. Modifiers are used to provide specific physical and mechanical properties in both cured and uncured resins. The general types of modifiers used are diluents, flame retardants, fillers,

pigments and dyes [6].Researchers are now focusing on the development of cellulose reinforced polymer composite(CRPC) because of its rapidly growing demands in the automotive industry. Cellulose is the basic structural units of all the fibers and can be easily process from the raw fiber. Cellulose is light weight, high strength weight ratio, biodegradable and have the functional capacity to replace natural fibers and glass fibers. For the development of sustainable, low-cost and renewable sources is necessary to meet the developing environmental concerns and energy demands. Most lignocellulosic biomass contains mainly cellulose, hemicellulose and lignin [16].From the past decade, the researches for cellulose as a reinforcement material are going on. The principal reasons for this are high specific strength and modulus, and its reinforcing potential. One of the primary challenge that arisen with the idea of cellulose reinforcement in nano range is the poor dispersion in the polymer matrix because of the fiber agglomerations results in hydrogen bonding within the cellulose fibrils but nowadays several techniques are introduced to eliminate these defects. The cellulose fibrils are embedded as a multi layered structure that cell wall network requires chemical or/ mechanical treatment. Many methods of separation have been used so far including chemical treatment to generate holocellulose and further mechanical through cryocrushing, shearing either homogenization, and ultra-sonication [18]. The thermoset resins polymers can be fabricated into composites with any of the moulding techniques as compression mouldingor injection such moulding. Compression moulding using thermoset matrix is an effective technique to manufacture parts in automotive mass production. The process is simple but with high production rate [13]. The primary advantage of the process is low fiber attrition and process speed. Many variations of compression moulding have been developed that are suitable for automotive application and recent developments combine extrusion to and compression of thermoplastic composites [3]. In this investigation cellulose reinforced polymer composite was developed by reinforcing jute and sisal cellulose into the matrix of epoxy lapox L-12. Cellulose can be extracted from fiber by chemical processes such as alkali treatment, steam explosion and bleaching. Then processed by sonication and lyophilser treatments for a making it as a fine, evenly distributed reinforcement in the epoxy matrix. CRP composites can be prepared by the compression moulding technique in three different proportions of matrix- cellulose as 85-15, 80-20, 70-30 respectively. The mould required for compression is prepared by stainless steel having a capacity of 200×200×3 mm³.Mechanical characterization was done for analyzing the feasibility of CRP composite in automotive applications at three different proportions. Strength properties such as tensile, impact and flexural strengths were investigated according to ASTM standards. Surface morphology of the samples were studied using a field emission scanning electron microscope for analyzing the fractured surface during impact loading. The SEM analysis helps to identify the type of failure in the reinforcement as well as matrix. The experimental results show that NCRP composites possess far better mechanical properties than the NFRP composites. This works also aims to compare the mechanical properties of sisal CRPC with jute CRPC for the same matrix – cellulose ratios. Even though both composites assumed to possess good mechanical characteristics than NFRP composites.

EXPERIMNTAL DETAILS Materials

The raw sisal and raw jute fibers for the cellulose extraction or the reinforcement preparation process, 1kg of each fibers were purchased from Enterprises, Alappuzha. The various S.R chemicals utilized for cellulose extraction and preparation are NaOH, Acetic acid, Sodium hypocholorite, Oxalic acid, etc and all are from Venad Chemicals Kollam, India. Epoxy resin L-12 is selected for this investigation to prepare matrix for the composite. L-12 (Lapox), a bifunctional epoxy resin consists of diglycidyl ether of bisphenol- A (DGEBA). The hardener used for the L-12 is K-6, known as tri ethylene tetra amine (TETA). The K6 is a room temperature curing agent. The epoxy L-12 and hardener are purchased from Atul Indialtd.

Table 1: Properties of Sisal fiber[9]

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Properties	Values
Diameter	100-300 µm
Density	1.450 g/cm ³
Cellulose	65%-70%
Hemicellulose	10%-14%
Lignin	9.9%
Elongation at break	2%
operation of Jute fiber[0]	

Table 2: Properties of Jute fiber[9]

1	
Properties	Values
Density	1.45 g/cm^3
Elastic modulus	10-32 GPa
Tensile strength	0.45-0.55 GPa
Elongation at break	1.1%-1.5%

Table 3: Properties of Epoxy L-12 [34]

Properties	Values	
Weight per epoxide	187.51 gm	
Viscosity	10000-12000 MPa-s	
Density	1.15-1.20 g/cc	
Shelf life	24 months	

Chemical Treatments

Alkali treatment

The process was a very effective and commonly carried out as a low-cost surface treatment of natural fibers as an initiative programme towards cellulose extraction. In this case, 5% of NaOH was used. 400 gmof each fibers, sisal and jute are taken and separated in 4 beakers by 100 gm in each beaker. For completely soaking the fibers in the beaker 500 ml of NaOH solution is prepared by using 25 gm of NaOH pellets in 500 ml water and stirred well to dissolve the pellets completely. The alkali treatment of sisal and jute fiber was done for a total of 12 hours, but on a period of 2 hour freshly prepared solutions are put on to thebeakers.

Steam explosion

Steam explosion was carried out by using a laboratory autoclave which have the capacity to generate 20 lbs pressure and 150° C. The treatment is done for one hour with a temperature and pressure of 120° C and 15 lbs pressure. The steam for the process is obtained by heating the water

present $(1/3)^{rd}$ of the autoclave chamber.

Bleaching

Before bleaching procedures to start the fiber samples are washed well with water to get it completely neutralized. Then for bleaching a mixture of solutions are needed. First solution was prepared with 26.5 gm of NaOH with 73.5ml of acetic acid added to 900 ml of water to make the up the solutions for 1000 ml. The second solutions 250 ml of sodium were prepared with hypochlorite in 750 ml of water for the 1000 ml solution and the solutions are mixed according to 1:1 ratio. Aftert he bleaching, if all the hemicellulose parts are removed, it will become whitish in color, which is an indicates that cellulose is extracted.



Fig 1: Photograph of alkali treatment



Fig 2: Photograph of autoclave at BT lab, SBCE If necessary, acid treatment can be done followed by bleaching with 5% of oxalic acid after completely neutralizing the bleached fiber. Apart from that, one more steam explosion can be given to the extracted fiber. The handling of fibers and take-over of the processes are carried out with ensuring safety measures.



Fig 3: Photograph of bleaching process Composite Preparation

The preferred matrix Lapox L-12 and the cellulose as the reinforcement, the composites are prepared with the proportions of 85%-15%, 80%-20% and 70%-30% using compression moulding technique. The compression mouldingtechnique are effectively and habitually used for natural fiber reinforced thermoset polymer composites. Thus, in this scenario compression moulding are utilized and was done on Neoplast compression moulding machine.



Fig 4: Neoplast compression moulding machine at CBPST, Cochin

The machine consists of a mould with a dimension of $(200 \times 200 \times 3)$ made up with stain less steel.

On considering the volume of the mould and the density of the epoxy 1-12, the total mass of the composite, which is the amount of matrix and reinforcement, are calculated and it as around 151 gm. According to various proportions of epoxy matrix and cellulose reinforcement selected the epoxy, hardener and the reinforcement are as follows;

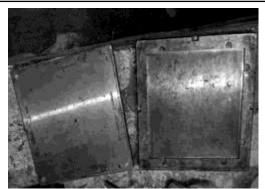


Fig 5: Mould – stain less steel ($200 \times 200 \times 3$) mm³

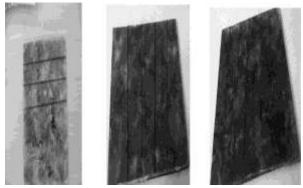


Fig 6: Prepared jute epoxy composite for 85%-15%,80%-20% and70%-30%

Table 4: Amount of matrix and reinforcement forvarious proportions

Descention	Enour	Hondonon	Cellulose Fiber
Proportion	Ероху	Hardener	(Jute/Sisal)
85%:15%	96gm	32gm	23gm
80%:20%	90gm	30gm	41gm
70%:30%	79gm	26gm	46gm

The mould is prepared and the based on each proportion, the epoxy and the reinforcement are weighed then the hardener is mixed with the epoxy before pouring into the mould. The first layer of the epoxy is applied by pouring into the mould. followed by adding reinforcement distributed over the layer of epoxy matrix. Again, the final layer of epoxy - hardener mixture is poured over the reinforcement. The mould is closed and compression pressure is applied automatically until the normal holding pressure attains. 83.5 bar pressure is applied for 3 hours for effective compaction. Then the composite was taken out of the mould and 24 hours room temperature maturation is carried out. Each remaining proportion of the NCRP Composite are made out on similar manner.

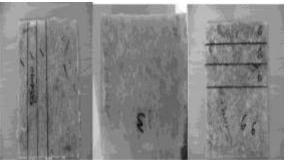


Fig 7: Prepared sisal epoxy composite for 85%-15%,80%-20% and70%-30%

Mechanical Testing

The three proportions of the Sisal cellulose reinforced epoxy composite and jute cellulose reinforced epoxy composite are prepared, and they are denoted as E85SC15, E85JC15, E80SC20, E80JC20, E70JC30, E70JC30. In order for the characterization of the composite mechanical testing and microstructure study must carry out. Mechanical characterization such as tensile test, impact test, flexural test and hardness test were done according to ASTM standards. The composites were taken, and the specimens are prepared from each sample for testing. The samples were cut by using semi- automatic hack saw. The tensile test was carried out on WDW200 universal testing machine, the samples prepared according to the ASTM standards. ASTM d 638 are used for preparing the sample specimen, the specimens from each sample are placed at the support ends. In order to measure the impact strength of the samples notched izod impact test has been carried out. The samples for the impact test are prepared according to the ASTM d 256 standards, the specimen is clamped into the pendulum impact test fixture with the notched side facing the striking edge of the pendulum. The pendulum is released and allowed to strike through the specimen. If breakage does not occur, a heavier hammer is used until failure occurs. The test was carried out on 3 samples of each composite are done on the Tinius Olsen impact testing machine. It has a load range of 0 N to 50 N with an accuracy of 0.1 N. The flexural strength of the composite is found out by doing three-point bending test with samples prepared according to the ASTM d 790 standards, the three-point

bending test was carried out on WDW 200 universal testing machine at CBPST, Cochin, for three-point bend test the specimens are supported on two supports at two of the ends and from another support through the center the load is applied and on failure the value is noted. The Shore (A) hardness was to be done for the composite. The preferred standard for the test are ASTM d 2240, hardness test was dropped out on composite to find out most hardened sample. The test was carried out with Mitutovo hardnessdurometer.

Material Characterization

The fractured surface of the impact samples was analyzed by using a field emission scanning electron microscope. This analysis helps to analyze the fracture during impact loading.



Fig 8: Field emission scanning electron microscope at NIT Calicut

The mode of failure of the reinforcement and matrix can be observed through SEM analysis. Thus, to study the ability of the cellulose to absorb impact energy. The fractured samples are prepared and have dimension of $1 \text{ cm} \times 1 \text{ cm} \times 0.3 \text{ mm}$ and well dried was first subjected to gold sputtering pre-phase to the SEM analysis. Then the samples are loaded into the FESM and the images are obtained for the magnifications of 50SE, 100SE and 1.00kSE.

RESULT AND DISCUSSIONS

Cellulose reinforced polymer composite has been developed with jute and sisal cellulose as reinforcement and epoxy, Lapox (L-12) as matrix. The composites are prepared in various

2018

proportions of epoxy matrix and cellulose. The different proportions are 85%-15%, 80%-20%, 70%-30% respectively. The composites were prepared by compression moulding technique and the various mechanical properties such as tensile strength, impact strength, flexural strength and hardness were investigated. In addition to that the surface morphology of the fractured surfaces the impact sample were analyzed using SEM. The obtained discussed the results are in followingsections;

Tensile test

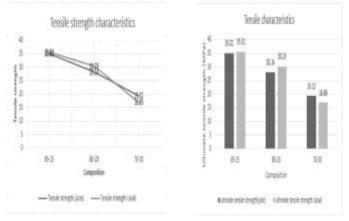
The test process involves placing the test specimen in the testing machine and slowly extending it until it fractures. During this process, the elongation of the gauge section is recorded against the applied force. On testing a maximum ultimate load of

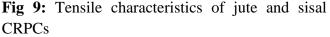
3.5 KN is applied and each the specimen, tensile strength was noted. Table 5 shows the ultimate tensile strength values of both JNCRP and SNCRP composite prepared in different compositions;

Table 5: Tensile strength values for jute and sisalCRPCs

Sampla	Ultimate Tensile Strength (MPa)			
Sample	Specimen 1	Specimen 2	Specimen 3	Average
E85SC15	39.34	33.04	25.2	35.52
E85JC15	34.63	38.75	31.70	35.02
E80SC20	30.0	37.47	29.4	30.29
E80JC20	27.90	27.4	29.2	28.16
E70JC30	27.74	14.81	13.76	19.32
E70SC30	20.93	14.32	15.42	16.89

From the table 5, it is evident that the tensile strength of CRP composite is more for 85% -15% proportion for both jute and sisal Nano cellulose reinforced composites. The former has an ultimate tensile strength of 35.52MPa and the later shows MPa. There is slight increase in ultimate tensile strength of sisal CRPC when compared with jute CRPC for the entire three matrix-cellulose compositions. The lowest value is obtained for the composition 70-30 and is 19.32 MPafor sisal based CRPC and 16.89 MPafor jute basedCRPC.





The Fig. 9 shows a comparison of the tensile properties of both sisal and jute based CRP composites in the three-different matrix –cellulose composition. From the graph, it is evident that even though there is a slight increase in ultimate tensile strength of sisal CRP composite, there is no significant variation in tensile properties of sisal CRPC with jute CRPC in the three-different matrix -cellulose ratio. The results indicate that the tensile strength decreases with increase in cellulose concentration. The increased concentration of cellulose reduces the ductile behavior of the composite material and which results in the failure of cellulose followed by matrix failure. The failure may be due to the cellulose pullout and breakage, caused by the nonuniform stress transformation due to random orientation of cellulose in the matrix. Random distribution may result in the cellulose agglomeration in the matrix. CRP composite with three matrix-fiber ratios shows the entire tensile characteristics appreciable than the reported values of NFRP composite with the same proportions. In this investigation CRP composite with 85-15 composition shows more promising tensile characteristics than the other two compositions.

Impact test

The impact test results of both jute and sisal based NCRP composite material at different proportions is shown in the table 6 Notched Izod test was carried out for analyzing the impact characteristics of specimens. **Table 6:** Impact strength values for jute and sisalCRPC

Sample	Izod Impact Strength (Kj/M ²)
E85SC15	2.86
E85JC15	2.26
E80SC20	3.45
E80JC20	2.36
E70JC30	4.38
E70SC30	7.02

From the table 6, it is clear that the cellulose absorbs more impact energy than fibers and impact strength increase when the cellulose concentration in the matrix increases. E70SC30 composite absorbs 7.02 KJ/m² energy during impact loading. This is a noticeable result when compared with E70JC30 and the other two proportions of both sisal and jute CRPC shown in the table6.

The role of cellulose concentration in the epoxy matrix during impact loading can be analyzed with figure 10. From the figure, it is evident that sisal CRPC absorbs more impact energy than jute CRPC in all matrix-cellulose composition. When the cellulose concentration increases more energy will be absorbed and which resists the failure of the matrix. Cellulose pull out and sudden breakage may be the reasons for initiating the failure which leads to the failure of the matrix. In this investigation, it is found that CRPC possess characteristics better impact than NFRP composites and these appreciable results of CRPC enhances its opportunities in the manufacturing of automotive components where impact strength is more critical in design.

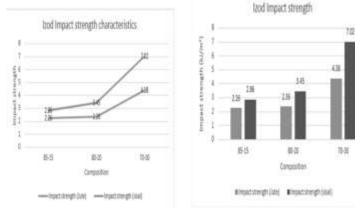


Fig 10: Impact strength characteristics of sisal and jute CRPCs

SEM Analysis

The surface morphology of the fractured tensile samples were observed through a scanning electron microscope and the images were taken in 50 SE and 1000 SE magnifications, the failure reasons and impact fracture characteristics are noted.

E85SC15

The figure 13 shows the cellulose breakage and matrix fracture during impact loading. From the figure, it can be inferred that the cellulose absorbs the impact energy and failed due to sudden

breakage during impact loading and which leads to the failure of composite. Matrix crack can be also observed from the SEM images.

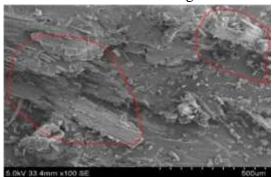


Fig 11: SEM images for impact fractured E85SC15 sample

E85JC15

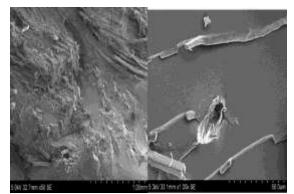


Fig 12: SEM images for impact fracture E85JC15 samples

E80SC20

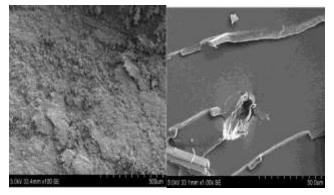


Fig 13: SEM images for impact fractured E80SC20 samples

E80JC20

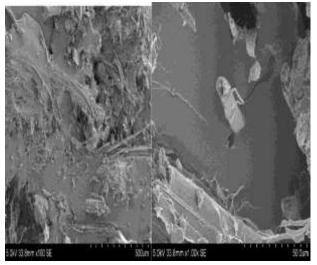


Fig 14: SEM images for impact fractured E80JC20 samples

E70JC30

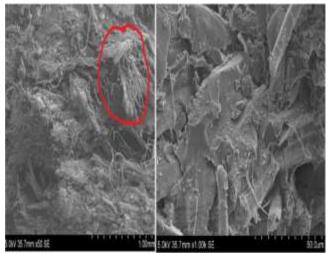


Fig 15: SEM images of impact fractured E70JC30 samples

E70SC30

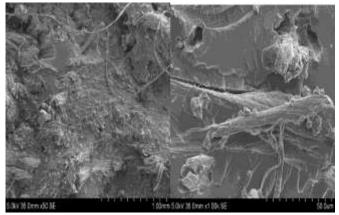


Fig 16:SEM Images of E70SC30

Matrix crack can be also observed from the SEM images. The figures 14-18 shows the same impact characteristics during SEM analysis. From the SEM images, it is evident that cellulose content absorbs more impact energy than fibers.

When cellulose concentration in the matrix increases impact strength also increases and failure is mainly due to the cellulose pullout and can be evident from the figure 16 to figure 18. Increased cellulose content offers more resistance to impact loads and which give more strength to the composite. The matrix crack can also be observed in all the figures and which leads to the failure of matrix. Agglomerations can be observed in figure 14, figure 16 and figure 17 which will result in non-uniform stress distribution and will lead tofailure.

CONCLUSION

In this investigation cellulose reinforced polymer composite was developed using jute and sisal cellulose as a reinforcement and epoxy Lapox-L12 as the matrix. the mechanical and material characteristics were investigated. In view of the analysis the following conclusions were eached.

The tensile properties are maximumfor CRP composite with 85-15 matrix cellulose composition and tensile properties decreases with cellulose increase in concentration. CRP composites possess better tensile characteristics than NFRP composite in all three-matrix celluloseratio.

Impact properties increase in fiber concentration

since the cellulose absorbs more energy during impact loading and is maximumfor sisal CRP composite with 70-30 matrix cellulose ratio. Since the cellulose content resist the impact loading the failure was observed due to cellulose pullout and cellulose breakage. The noticeable impact properties of sisal CRP composite opens a door to the automotive industry where the components require high impactproperty.

Even though the cross-linking nature of the matrix possesses better strength characteristics, cellulose failure weakens the bond between the matrix and the reinforcement finally it leads to failure of the composite.

Random distribution results in non-uniform stress concentration an agglomeration of cellulose which results in poor properties at somepoints.

Failure prediction models can be developed by continuumapproach.

The infusible property of the cross-linking structure enhances the application of cellulose reinforced polymer composite to a wider extend.

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