

*Fabrication and characterization of raw and bleached
treatment coir fiber
Reinforced polymer composite*

**A THESIS SUBMITTED IN PARTIAL FULFILMENT OF THE
REQUIREMENTS OF THE DEGREE OF**

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CERTIFICATE

This is to certify that the thesis entitled “Fabrication and characterization of raw and bleached treatment coir fiber reinforced polymer composite” is submitted by Snehamayee das in partial fulfilment for the requirements for the award of Master of Science degree in physics department at National institute of technology, Rourkela is an authentic work carried out by her under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other institute/university for the award of any degree.

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ABSTRACT

The bleached treatment of coir fibre composites were prepared using treated coir fiber and epoxy resin using handmade mould. XRD patterns confirm that degree of crystallinity decreases by the treatment of coir fibre with H₂O₂. SEM image shows roughness of surface structure of composites. It confirms from the SEM, that the adhesion is increased after treatment. FTIR study gives us information about the functional group present in the composite .The 3 point bending system or instron analysis tells about the flexural strength of the composite.

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CHAPTER-1

INTRODUCTION:-

1.1 OVERVIEW OF COMPOSITE:-

In the last thirty years composite materials like fibers, glass, plastics and ceramics has been the dominant emerging materials. The features of composite materials have grown continuously, penetrating and conquering new markets relentlessly. The composites industry has started to recognize that the commercial functions of composites offer much larger business opportunities than the aerospace sector due to the sheer size of transportation industry. There for the shift of composite applications from aircraft to other commercial uses has become prominent .in some applications, the use of composites in place of metals has in fact resulted in savings of both cost

Composites are now being used for rehabilitation or strengthening of pre-existing structures that have protected them from earth quake like natural calamities. Properties of composite material (e.g. stiffness, thermal expansion etc.) can be changed continuously over a large range of values under the control of the designer can be made considering the structural aspects.

1.2 DEFINITION OF COMPOSITE:-

A composite is a type of material which consists of two or more combined constituents with different physical & chemical properties.

These are combined at a microscopic level & are not soluble in each other .One constituent is called reinforcing phase & the other in which it is embedded is called matrix.

(i) Reinforcing phase

(ii) Matrix

The reinforcing phase materials may be in the form of fibers, particles, or flake.

The matrix phase materials are generally continuous. The role of matrix in composite part is to give shape to the composite, protect the reinforcements from the environment, transfer load to the reinforcement.

1.3 ADVANTAGES OF COMPOSITE:-

Advantages of **composite** are the ability to meet diverse design requirements with significant weight savings as well as strength-to-weight ratio. Some advantages of composite materials are as follows:

- Tensile strength of composites is four to six times greater than that of steel or aluminum (depending on the reinforcements).
- It has improved tensional stiffness and its tensile strength is very good
- Light weight
- Corrosion & chemical resistance
- It possesses lower embedded energy compared to other structural metallic materials
- Non conductivity
- elasticity

DISADVANTAGE:-

- temperature limit
- cost

Composite materials are classified into three groups according to the matrix material. They are:

- Metal Matrix Composites (MMC)
- Ceramic Matrix Composites (CMC)
- Polymer Matrix Composites (PMC)

Among these three matrixes material polymer matrix composite is again divided in to two types

- Fiber reinforced polymer (FRP)
- Particle reinforced polymer (PRP)

Fiber is a class of materials that are continuous filaments or are discrete elongated pieces similar to long threads. It is important for both plant & animals.

Fiber reinforced polymer:-

Common fiber reinforced composites are composed from fibers and a matrix. These fibers carry the loads along their longitudinal directions. Common fiber reinforcing agents include carbon graphite fibers, beryllium, beryllium carbide, beryllium oxide, molybdenum, aluminium oxide, glass fibers, natural fibers etc. Similarly common matrix materials include epoxy, phenolic, polyester, vinyl ester etc. Epoxy, which has larger adhesion and lower shrinkage than matrix comes in second for its high cost.

Particle Reinforced Polymer:-

These are used for reinforcing ceramics and glasses such as small mineral particles, metal particles such as aluminium and amorphous materials, including polymers and carbon black. Particles are used to increase the modulus of the matrix, and to decrease the ductility of the matrix, Particles are also used to reduce the cost of the composite.

1.4 CHARACTERISTICS OF COMPOSITE

A composite material consists of two phases. It consists of one or more discontinuous phases embedded in a continuous phase. The discontinuous phase is usually harder and stronger than the continuous phase and is called the “reinforcement” or “reinforcing material”, whereas the continuous phase is termed as the “matrix”. The matrix is usually more ductile and less hard. “Matrix” is composed of any of the three basic material type that is, polymers, metals or ceramics.

1.5 NATURAL FIBER REINFORCED COMPOSITE:-

For making composite of reinforcement polymer, we use natural fibers; it includes those produced by plants animals, & geological process. Now there is much interest in natural fiber-reinforced polymer composite materials because of their industrial applications and fundamental research. These are renewable, cheap, completely or partially recyclable, and biodegradable. Plants, such as flax, cotton, hemp, jute, sisal, pineapple, ramie, bamboo, banana, etc., are used as a source of lignocellulosic fibers, and are more and more often applied as the reinforcement of composites. Their availability, renewability, low density, and price as well as satisfactory mechanical properties make them an attractive ecological alternative to glass, carbon and man-made fibers used for the manufacturing of composites. The natural fiber-containing composites are more environmentally friendly, and are used in transportation (automobiles, railway coaches, aerospace), military applications, building and construction industries (ceiling panelling, partition boards), packaging, consumer products, etc.

1.6 CLASSIFICATION OF NATURAL FIBER:-

Natural fiber can be classified into three categories according to their origin.

- ❖ Animal fiber
- ❖ Mineral fiber
- ❖ Plant fiber

Animal fibre: Animal fibre consists largely proteins: instances are spider silk, sinew, catgut, wool and hair. Animal hair taken from animals or hairy mammal's for example sheep's wool, goat hair, horse hair etc. And silk fibres are collected from dried saliva of bugs or insects during the preparation of cocoons. Avian fibres are the fibres from birds, e.g. feather fiber. Animals or hairy mammals. Silk from silk worms.

Mineral fibre: Mineral fibres comprised abestors, which is only naturally occurring long fibres. This one of the categorized of mineral fiber. Ceramic fibres includes glass fibres i.e. glass wood and quartz.

And others are aluminium oxide, silicon carbides, and borocarbide. Metal fibres consist of aluminium fibres.

Plantfiber: plant fibres are consists generally cellulose. Cotton, jute, flax, ramie, sisal and hemp are the examples of plant fiber. In the manufacture of paper and cloth cellulose fibres is required. These fibres are classified as:

- Seed fiber, those are collected from the seed and seed case e.g. Kapok and cotton
- Leaf fiber is the fibre collected from the leaves e.g. Agave and sisal.
- Skin fibers are collected from the skin or blast surrounding the stem of their respective plant. .
- Fruit fibers are the fibres collected from the fruit of the plant, e.g. coconut (coir) fibre
- Stalk fiber are the fibres are actually the stalk of the plant.e.g straws of wheat, rice, barley. Bamboo and grass are also included in this type of fibre. Tree wood is also considered as plant fibre.

The tensile strength of plant fiber is higher than other fibers. So for durable yarn, fabric, packaging, and paper these fibers are mainly used. Banana, flax, jute, hemp and soybean are the examples of plant fiber. We can use the natural fibers to reinforce both thermosetting and thermoplastic matrices. And these composites require high performance applications. They provide sufficient mechanical properties, with fix stiffness and strength, at acceptably low cost levels. Considering the ecological aspects of material selection, replacing synthetic fibers by natural ones is only a first step. CO2 gas which causes the emission of green house effect into the atmosphere can be restricted and an increasing awareness of the completeness of fossil energy resources are leading to developing new materials that are entirely based on renewable resources.

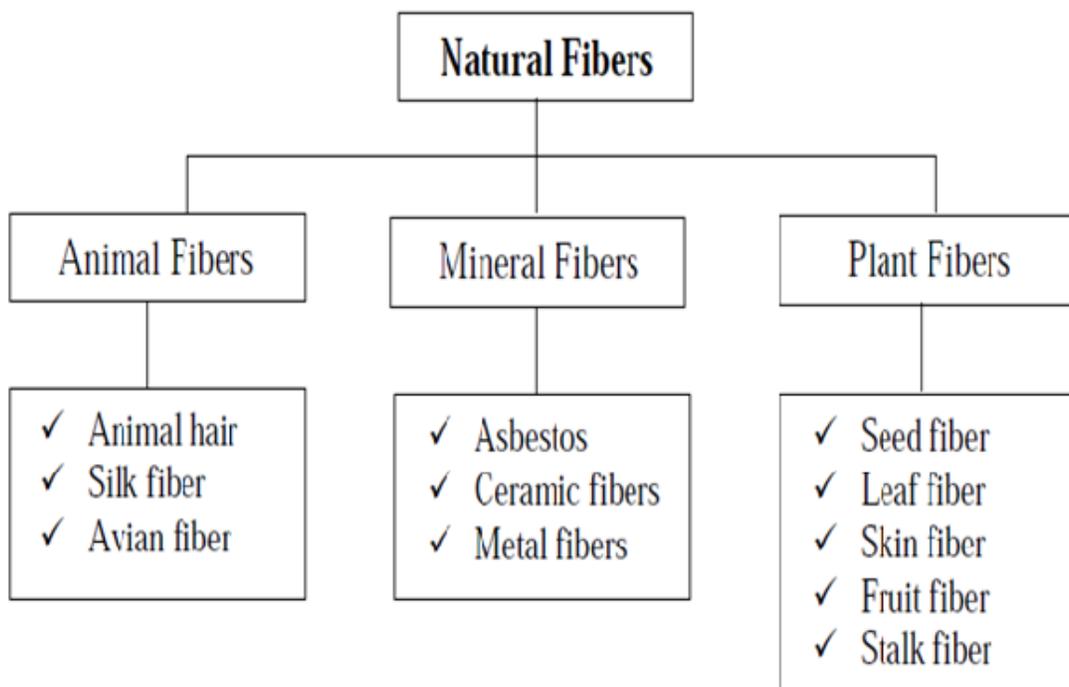


Figure 1.1 Classification of natural fibers

1.7 APPLICATION OF NATURAL FIBER:-

- These are used in building and construction industry for partition and false ceiling, partition boards, wall, floor, window frames and door, roof tiles, mobile, fabricated buildings which can be used in times of natural calamities, like flood, cyclone etc
- It produces Furniture like chair, table, shower, bath units, etc.
- These are used in electric devices like electrical appliances, pipes and Storage devices like post-boxes, grain storage silos, bio-gas containers, etc
- It is also used in everyday applications such as lampshades, suitcases, helmets, etc.
- It uses in transportation automobile and railway coach, boat, etc
- It possesses low density which may lead to a weight reduction of 10 to 30%.

1.8. Advantages of Natural Fiber Composites:-

The natural fiber composite mainly have the following advantages

- Its low specific weight, higher specific strength and stiffness than glass fiber.
- It is a renewable source & it is biodegradable
- We can produce these composites with low investment at low cost, which makes the material, an interesting product, for low developing countries.
- It possesses reduced wear of tooling, healthier working condition, and no skin irritation.
- Here thermal recycling is possible
- It has Good thermal conductivity and acoustic insulating properties
- Its low cost is one of the advantages of the fiber
- It behaves friendly to the environment

Chapter-2

2.1 Literature survey:-

As a result of increasing demand for environmentally friendly materials & the desire to reduce the cost of traditional fibers, reinforced composites have been developed. Researchers have been focused on natural fibers composites.

Harishet.al. [2] developed coir composite & mechanical properties were evaluated .scanning electron microscope obtained from fracture surface were used for a qualitative evaluation of the interfacial properties of coir or epoxy and compared with glass fiber.

Wang & huang [1] taken a coir fiber stak characters of the fibers were analysed .length of he fiber with the length range between 8to 33 mm.The fibers amount with the range of 15~145mm was 81.95% of all measured fibers. Weight of the fiber with the length range of 35~225mm accounted for 88.34% of all measurement. The average fineness of the coir fibers was 27.94.longer fibers usually had higher diameters composites boards were fabricated by using a heat press machine with the coir fiber as the reinforcement and the rubber as matrix .Tensile strength of composite was investigated.

Nilza.et.al [3] use three Jamaican natural cellulose fibers for the design and manufacture of composite material. Samples were subjected to standardized tests such as ash and carbon content, tensile strength, elemental analysis, &chemical analysis. Bilba [4] examined four fibers from banana tree & coconut tree, before there incorporation in cementitious matrixes in order to prepare insulting material for construction. These fibers possess thermal degradation in between the range 200c to 700c under N₂ gas flow. It was observed practically that temperature is a parameter for pyrolysis. The solid residues obtained were computed by classical elemental analysis, FTIR and were observed by SEM .This study has shown the relation between botanical, chemical composites with both localization of fiber in the tree & tree type.

A series of batch adsorption experiments were conducted with the initial phenol concentrations, ranging from 100 to 500mg l⁻¹, adsorbent loading of 0.2 g and the adsorption process was maintained at 30±1°C. Chemical reaction was found to be a rate-controlling parameter, to this phenol-CS850A batch adsorption system due to strong agreement with the, pseudo-second-order kinetic model. Adsorption capacity for CS850A was found to be, 205.8mg g⁻¹. Ra.et. al [5] aims at introducing new natural fibers used as fillers in a polymeric matrix enabling production of economical and lightweight composites for load carrying structures. A research of the extraction procedures of coconut, bamboo fibers has been undertaken. The cross-sectional area, the density and tensile strength of these fibers, along with established fibers like sisal, banana, coconut and palm, are determined practically under similar conditions. The fibers introduced now a days could be used as an effective reinforcement for preparing composites, which gives an advantage of being lightweight.

2.2 Objective of the research work:-

The main aim of this project work is

- Fabrication of coir fiber reinforced epoxy based polymer composite
- .Determination of mechanical properties like (roughness, flexural, hardness, etc.)
- In this work it is expected to introduce a new class of polymer composite that might find many engineering applications.

CHAPTER-3

EXPERIMENTAL

3.1 Materials & method:-

In this chapter we will discuss about the experimental procedure & processing of the composite material. For preparation of coconut composite the required raw materials are:-

- (i) Coconut fiber
- (ii) Epoxy
- (iii) Hardner

Coconut fiber is a fruit fiber, it is extracted from the outer cover of the fruit. It is water proof & has balanced ph. It is 100% organic. It is biodegradable renewable resource & strong air porosity.

Chemical composition:-

- Lignin-45.84%
- Cellulose-43.44%
- Water soluble-5.25%

Physical composition:-

- Breaking elongation-30%
- Moisture elongation-65%
- Density-1.40gm/cc
- Diameter-16micron

The unmodified liquid epoxy resin based on Bisphenol A, of grade LY 556 along with hardener HY 951 is provided by B. Mukesh & Co., Kolkata, India. The density of the resin is 1.15 g/cc. whereas the hardener density is 0.98 g/cc.

3.2 Treatment of fiber:-

First the received coconut fibers are washed with distilled water to remove the surface dirt present in the fibers and then the fibers are dried in an air circulating oven at a temperature of 100°C until it gains a fixed value of weight. Then the fibers are named as raw coir fibers.

Bleaching treatment:-

For this treatment 25g Coconut fibers were added to a 2 L solution containing 320 mL (30%; w/w) hydrogen peroxide and 1 g sodium hydroxide. And heated at 85°C for 1 h [6]. During this process the fibers are cooked in the solution under gradual rise and fall of the temperature of the bath from 30°C to 85°C. This process of heating and cooling was done for a period of 1 h. Finally, the cooked fibers are removed from the mixture at a temperature of 30°C. In order to remove excess mixture, the fibers are washed with distilled water. After washing, the fibers are again dried in an air circulating oven at a temperature of 100°C until it gains constant weight. Then the fibers are designated as bleached coconut fibers.

3.3 Fabrication of composite plate:-

In this part a handmade wooden mould is designed, for the fabrication of the randomly oriented raw coir fiber reinforced epoxy composite (RCFREC) and bleached fiber-reinforced epoxy composite (BCFREC). First, we use a releasing plastic & it is spread over the bottom of the wooden mould. Then heavy duty silicon spray is applied to the plastic sheet in order to remove the composite plate easily [7]. After this the fibers are cut into 3cm length and spread over uniformly at the bottom of the mould area, which is prepared before. Fifteen percentage of the fiber volume is used for the fabrication of the reinforcement composite. Initially, epoxy and hardener are being weight in the proportion of 10:1 ratio by a weight machine & mixed together to form a matrix. The matrix is poured over the fibers evenly then pressed and pushed down with the iron roller to avoid and eliminate the air bubbles. Finally, load is given to it to remove excess matrix and left for curing at room temperature for 24 h.

3.4 Characterisation:-

In order to find out the effect of treatment on the micro molecular structures of coir fiber, wide angle X-ray diffraction (WAXD) spectra are collected by PHILIPS PAN analytical PW1830 with Cu-K α radiation from 10° to 45° with a scan speed of 0.04m/s. The crystallite sizes of the fibers are determined by modified Scherer's formula whereas the degree of crystallinity is computed by comparing the areas under crystalline peak and amorphous curve i.e., the area under the crystalline peak around 22.5° is compared with sum of the areas of amorphous peaks around 15° and 34° by peak fit software [8].

Chemical compositions of both the raw and bleached coir fibers are measured by the Perkin Elmer FTIR spectrometer spectrum RX-1 in the mid IR range i.e., from 400 to 4000 cm. Fourier Transform Infrared Spectroscopy (FTIR) is an analytical technique which is used to find out the organic materials present in the composite. FTIR analysis results in absorption spectra which gives information about the chemical bonds and molecular structure of a material [9, 10].

The fractured surfaces of coir fibers and composites are examined by SEM (JEOL JSM-6480 LV).

In order to find out the flexural strength, of the composites three point bending test are done by INSTRON 1195. The randomly oriented RCFEPC and DCFREC specimens are cut as per the ASTM D790 to measure the flexural strength. The sample size for flexural measurement is 130*30*50mm with a crosshead speed of 2 mm/min with a gauge length of 50 mm [11]. The reported data is the average of the five successful tests.

Result and discussion:-

4.1 XRD ANALYSIS:-

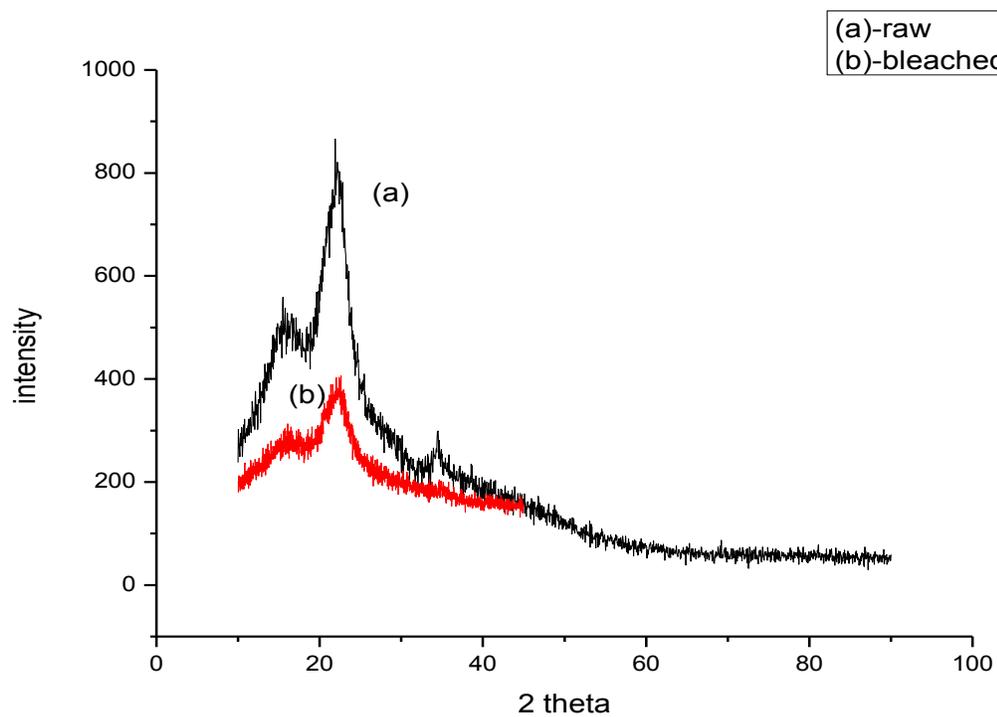


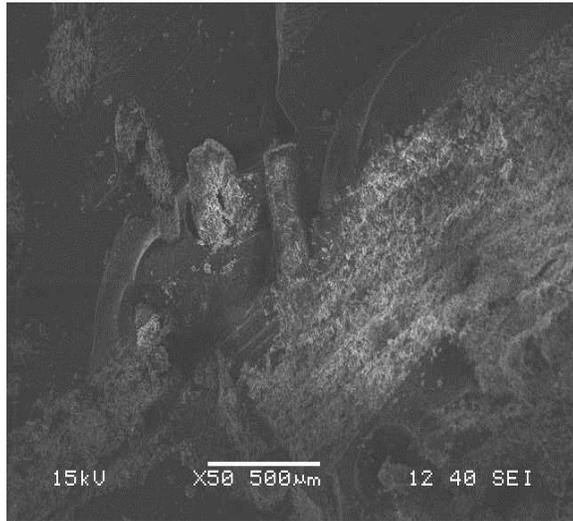
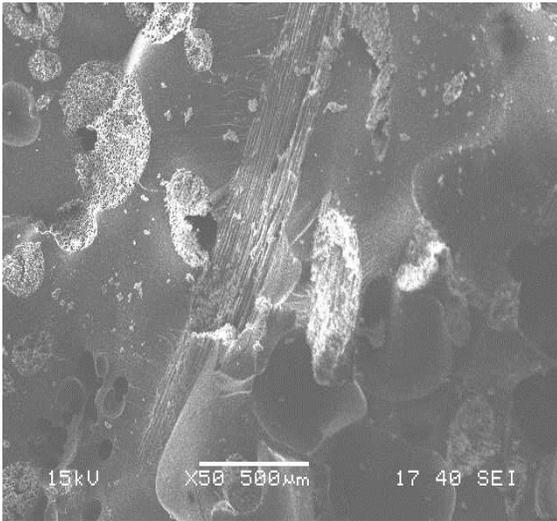
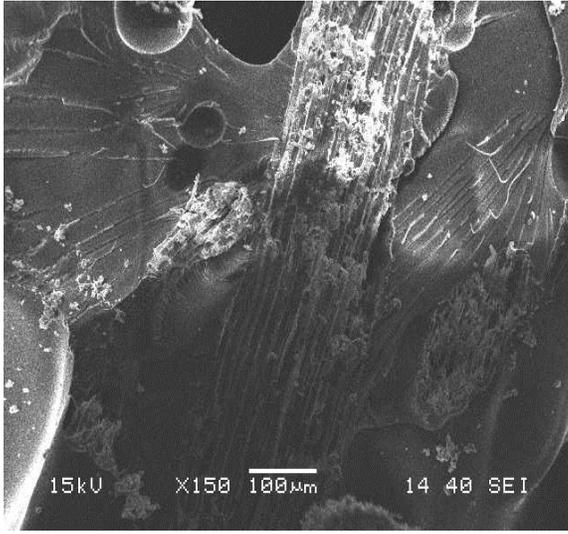
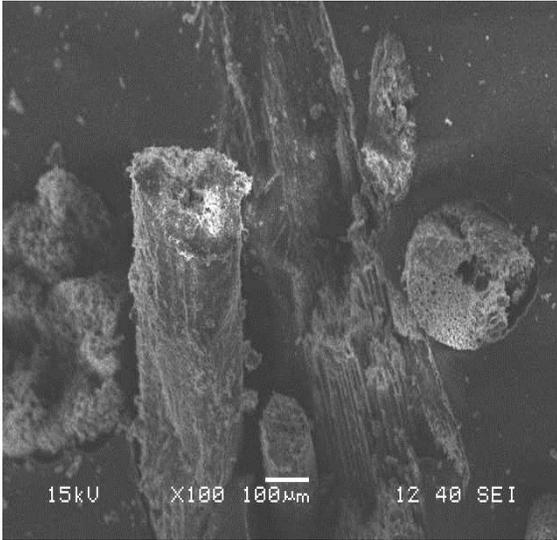
Figure-A

Coir fiber	Degree of crystallinity
Raw fiber	71.5
Bleached fiber	66.6

Table1 given above shows the crystallite size and degree of crystallinity of bleached and raw coir fibers. It shows that, after bleaching the degree of crystallinity of the fiber has been decreased, which may be due to the rearrangement of the cellulose chain. The changes in the crystalline peaks in Figure-A of bleached fiber in comparison with raw fiber denote the changes in lignin contain & crystalline size [12]. This may be occurred due to prolonged acid treatment, which not only removed the amorphous portion of cellulose fibers but also partly destroyed the crystalline ones.

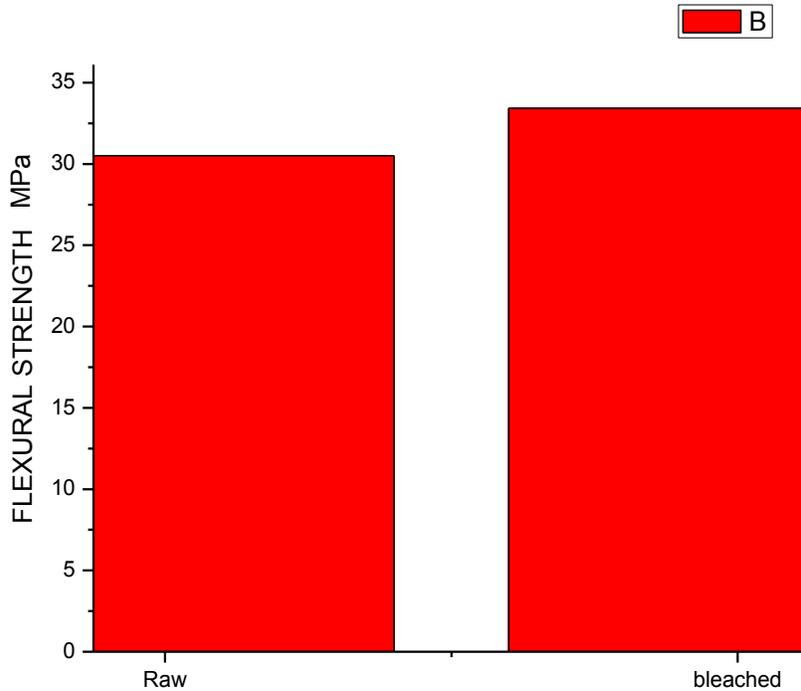
SEM ANALYSIS:-

The SEM images of fractured surfaces of raw (fig-1),(fig-4) and bleached treated coir fibre (fig-2),(fig-2) are shown in the figure. It is observed that the treatment has improved the surface roughness of the fiber as compared to the untreated raw fiber. Which increases the adhesion between fiber and matrix. Also because of the removal of the surface impurities and cementing materials like lignin and hemicelluloses, it can be observed that separation of the ultimate cells has increased in bleached fiber than raw fiber which leads to the increase in effective surface area of the fiber so it become more compatible with the matrix.



SEM surface of raw and bleached coir fiber

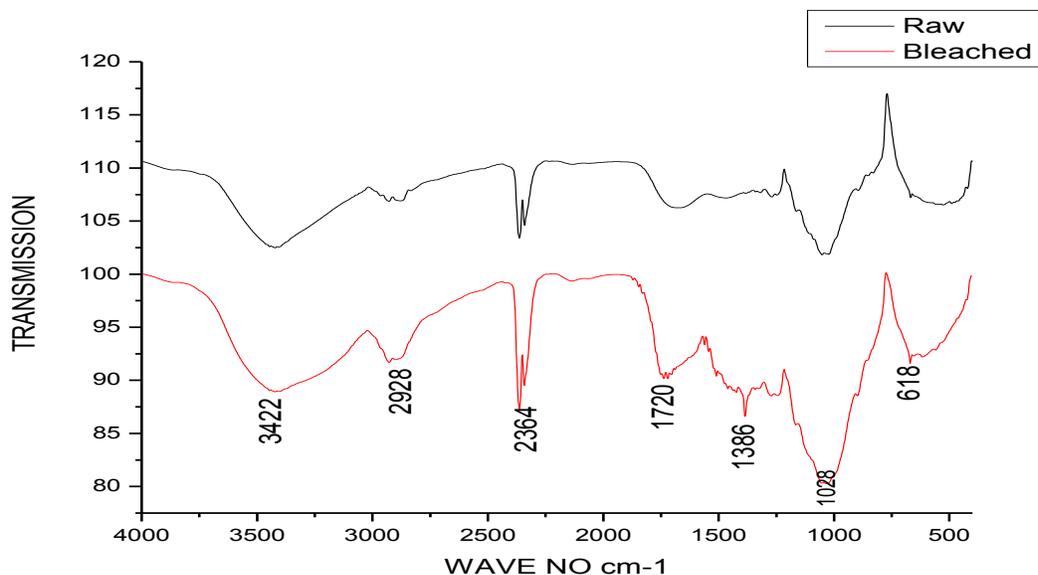
INSTRON ANALYSIS:-



INSTRON GRAPH OF RAW AND COIR FIBER

Figure-B shows the flexural strength of both raw fiber and bleached fiber. from the 3 point bend test, it is found that the strength of bleached fiber is higher than raw fiber. For the fibre reinforced composites, the interfacial zone plays a leading role in transferring the load between fiber and matrix which affects the mechanical properties such as strength [13].This finding demonstrates that flexural failure depends mainly on the fiber or matrix adhesion. The increased value of flexural strength in case of bleached may be increase in effective surface area available for contact with the matrix [14]. The flexural strength of the coir increases after bleached treatment is due to the dissolution of hemicelluloses, development of crystallinity and fibrillation thus creating superior bonding with matrix.

FTIR ANALYSIS:-



SPECTRUM OF RAW AND COIR FIBER

- Figure-C shows the FTIR spectra of both bleached and raw coir fiber. The broad intense peak at 3422 cm⁻¹ in raw coir fiber is due to the O–H stretching for hydrogen-bonded hydroxyl group present in polysaccharide. However, in case of bleached coir fiber this peak gets narrower and less intense, because of the reduction of the O–H group of the fiber after bleaching treatment.
- The weak peak occurs at 1386 cm⁻¹ in raw coir fiber is because of the presence of hemicelluloses which can be assigned to the group of C=O stretching, and it is increased significantly after bleaching.
- Again, due to the removal of the surface impurities and cementing material like lignin and hemicelluloses, separation of the ultimate cells has increased. This leads to the increase in effective surface area of the fiber to become more compatible with the matrix.
- The weak broad peak observed in 1720 & 1028 cm⁻¹ is associated with water absorbed in cellulose.[15]

Position of bands(cm^{-1})	Assignment
3422	O-H stretch
2928	alkyl C-H stretch
2364	WATER PEAK
1720	ketone C-O stretch
1386	C-O
618	C-H bending

5. CONCLUSION

In this project work we synthesized the natural fiber and study the nature of reinforcement composites by comparing raw and bleached coir fiber. XRD patterns confirm that the coir fiber has been modified by bleaching which results a decrease in the degree of crystallinity, and crystallite size. SEM micrograph shows that the roughness of fiber increases as compaired with raw fiber so the adhesion properties increase. FTIR spectra confirm that water content of composites decreases due to intermolecular hydrogen bonding. The 3 point bending test (INSTRON) shows that the strength of bleached fiber increases with the comparison of raw fiber. Moreover, the residual lignin content, which is controlled by the bleaching treatment, can act as a compatibilizer to hydrophobic polymer matrices.

So we conclude that, it gives better result if we use bleached coir fiber instead of raw fiber composite preparation.

CHAPTER-6

REFERENCES

1. Bledzki AK, Gassan J. Composites reinforced with cellulose based fibers. *Prog Polym Sci* 1999;24: 221–74.
2. Mwaikambo LY, Ansell MP. Chemical modification of hemp, sisal, jute, and kapok fibers by alkalis. *J Appl Polym Sci* 2002;84(12):2222–34.
3. Jochen Gassan. A study of fiber and interface parameters affecting the fatigue behaviour of natural fiber composites. *Composites Part A*: 2002;33(3):369–74.
4. Ruys D, Crosky A, Evans WJ. Natural bast fiber structure. *Int J Mater Product Technol* 2002;17(1–2):2–10.
5. Santulli C. Post-impact damage characterization on natural fiber reinforced composites using acoustic emission. *NDT&E International* 2001;34(8):531–6.
6. Morsyleide F, Rosa a,b, Bor-sen Chiou b, Eliton S, Medeiros b,c, Delilah F, Woodb, Tina G, Williams b, Luiz H.C. Mattoso c, William J. Orts b, Syed H. Imam b,*
7. Roy SC (1960) *Text Res J* 30:451 & Ratho T, Torasia S, Mohanty JC (1964) *Indian J Phys* 38:28
8. Klemm D, Heublein B, Fink H, Bohn A (2005) *Angew Chem Int Ed* 44:3358
9. Liansong W, Dongling H, Tianyao Z, Lifang Z, Chengdong X (2010) *Cryst Res Technol* 45:27
10. Trindade WG, Hoareau W, Megiatto JD, Razera IAT, Castellan A, Frollini E (2005) *Biomacromolecules* 6:2485
11. Bisanda ETN (1991) Ph. D Thesis
12. Klemm D, Heublein B, Fink H, Bohn A (2005) *Angew Chem Int Ed* 44:3358
13. Georgopoulos ST, Tarantili PA, Avgerinos E, Andreopoulos AG, Koukios EG (2005) *Polym Degrad Stab* 90:303.
14. Thielemans W, Wool RP (2005) *Biomacromolecules* 6:1895.
15. 18. Troedec ML, Peyratout C, Chotard T, Bonnet JP, Smith A, Guinebretière R (2007) In: 10th International Conference of the European Ceramic Society, Berlin, pp 451–45
16. Mohanty AK, Misra M, Drzal LT. Surface modifications of natural fibers and performance of the resulting biocomposites: an overview. *Composite Interfaces* 2001;8(5):313–43.

