IJESRT: 7(12), December, 2018 ISSN: 2277-9655

International Journal of Engineering Sciences & Research Technology

(A Peer Reviewed Online Journal)
Impact Factor: 5.164

IJESRT

IJESRT
INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY

CHARACTERIZATION AND SYNTHESIS OF NANO SISAL FIBER REINFORCED COMPOSITES

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DOI: 10.5281/zenodo.2526231

ABSTRACT

There has been a growing interest in utilizing natural fibres as reinforcement in polymer composite for making low cost construction materials in recent years. Economic and other related factors in many developing countries where natural fibers are abundant demand that scientists and engineers apply appropriate technology to utilize these natural fibres as effectively and economically as possible to produce good quality fibre reinforced polymer composites for housing and other needs. Among the various natural fibres, sisal is of particular interest in that its composites have high impact strength besides having moderate tensile and flexural properties compared to other lingo cellulosic fibres.

The present work aims is to compare the tensile properties of sisal nano fibre reinforced polymer composites to glass fibre reinforced polymers composites. Initially sisal fibers are chemically treated with sodium hydroxide (NaoH) and hypo chlorite (Naclo) to extract the cellulose content present in the fibers. Chemically treated fibre size is reduced to nano level by using planetary ball milling machine. Ball milling was carried out for 10 hours. Chemically treated fibers are characterized by X-ray powder diffraction to measure their crystallite size. And to find the morphology and inorganic materials of the nano powder, scanning is performed by scanning electron microscope (SEM).

Composites were fabricated using Hand Lay-up process. Glass fibre reinforced polymer composites and sisal nano fibre reinforced polymer composites were fabricated. The tensile properties of composites are evaluated experimentally. It is observed the tensile strength of sisal nano fibre reinforced polymer composites have high strength compared to glass fibre reinforced polymer composites.

1. INTRODUCTION

1.1 Composite materials:

Composite materials are those that are formed by the combination of two or more materials to achieve properties to achieve properties that are superior to those of its constituents. Generally, a composite material is composed of reinforcement (fibers, particles, flakes, and/or fillers) embedded in a matrix (polymers, metals, or ceramics).The matrix holds the reinforcement to form the desired shape while the reinforcement improves the overall mechanical properties of the matrix. When designed properly, the new combined material exhibits better strength than would each individual material. Most commonly, composite materials have a bulk phase, which is continuous, called the matrix, and one dispersed, non- continuous, phase called the reinforcement, which is usually harder and stronger.

1.2 Fiber Reinforced Polymer (FRP) Composites:

Fibers are the important class of reinforcements, as they satisfy the desired conditions and transfer strength to the matrix constituent influencing and enhancing their properties as desired. Glass fibers are the earliest known fibers used to reinforce materials. Ceramic and metal fibers were subsequently found out and put to extensive use, to render composites stiffer more resistant to heat. Fibers fall short of ideal performance due to several factors. The performance of a fiber composite is judged by its length, shape, orientation, and composition of the fibers and the mechanical properties of the matrix.

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1.3 Sisal Fibre:
Sisal fibres have been the leading material for agricultural twine because of its strength, durability, and ability stretch.

Fibers obtained from the various parts of the plants are known as vegetable fibers.

Fig.1.1.Sisal Plant

Fig 1.2.Sisal Fibres.

Sisal fibre is mainly used for the manufacture of ropes for use in marine industry and agriculture, for making twines, cords, padding, mat making, fishingnets, fancy articles such as purses, wall hangings, table mats etc. The use of sisal fibre asa textile fibre by mankind began with Weindling's work during forties.

2. LITERATURE REVIEW

Joseph ,et.at[1](1999). carried out a research work in the field of sisal fibre reinforced polymer composites with special reference to the structure and properties of sisal fibre, processing techniques, and the physical and mechanical properties.. Thus it can be concluded that with systematic and persistent research there will be a good scope and better future forsisal fibre polymer composites in the coming years.

V.N.P. Naidu,et.al, [2] (2011).studied about Sisal/Glass fibre hybrid composites and the compressive and impact properties of these hybrid composites. It is observed that the compressive and impact strength of sisal/glass fibre hybrid component is higher than sisal fibre reinforced composite, but lower than the glass reinforced composite.

G.R.C. Reddy, et.al, [3] (2011) prepared hybrid composites of unsaturated polyester based sisal/glass fibre hybrid composites. The sisal is a natural fibre which isalso biodegradable and glass fibre is a synthetic fibre. Itis observed that the thermal conductivity of sisal/glass fibre hybrid component is higher than sisal fibre reinforced composite, but lower than the glass fibre reinforced composite.

Dixit S.and VermaP.[4]**(**2012) evaluated experimentally the effect of hybridization on Mechanical properties on coir and sisal reinforced polyester composite(CSRP), coir and jute reinforced polyester composite (CJRP), jute and sisal reinforced polyster composite (JSRP). It has been studied that the tensile properties of natural fibre composites can be significantly improved by natural fibres in a sandwich construction.This work also demonstrates the potential of these hybrid natural fibre composite materials for use in a number of consumable goods.

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Yan Li, et.al. [5] (2000) developed sisal fibre and its composites. The properties of sisal fibre itself interface between sisal fibre and matrix, properties of sisal-fibrerein forced composites and their hybrid composites have been reviewed. It is observed Sisal and glass fibres can be combined to produce hybrid composites which take full advantage of the best properties of the constituents. Almost all the mechanical properties show `positive' hybrid effects.

SusheelKalia, et.al [6] (2011) studied about benzoylation and grafting of sisal fibers were reported using benzyl chloride of different concentrations and methyl acrylate monomer, respectively. Thermal stability and percentage crystallinity of sisal fibers were enhanced in both the chemical treatments.

3. SYNTHESIS AND CHARACTERIZATION OF SISAL NANO FIBRE

3.1 Chemical analysis:
To find out the cellulose content in the fiber about 90gm of sisal fiber are taken ina dish containing 1200cc of 1500 of 8% NaoH. The solution is boiled for 8 hours while boiling regress evaporation of solution is avoided so as to keep the concentration of the solution constant. Theoretically small quantity of water is added to the solution. After 8 hours of boiling the contents are filtered and fibers are washed with distilled water and dried in a oven 30degrees per two hours then those fibers are cleaned with 5% of Hypo solution to remove lignin completely. After that the fibers are washed with distilled water and dried so as to remove water completely

Preparation of chemical analysis:
Used materials are:
Sisal fiber was cut into 5mm, 2lt Beaker, Filter papers, Digital Weighing machine, Burnel, Initial weight of the fiber is 90g so take 3 times more than the fiber weight.
Solution for 8% **Noah**:

- 100ml of solution= 100ml of distilled water +8gm of NaoH
- 300ml of solution = 8g(Noah) x3=24gms of Noah+ 300ml of distilled water.

Solution for 5% **hypo**:

- 500ml of solution= 5g (hypo) x 5=25gms of hypo+ 500ml of distilled water.

Surface lignin plays an essential role in the surface characteristics of fibre and the interfacial behavior of composites prepared with cellulosic materials. The sisal fib resurface is covered with large amounts of lignin, and relatively few cellulose micro fibrilsare exposed. These conditions do not favor effective bonding at the fibre/resin interface.

Fig. 3.1 chemical analysis

The significant removal of lignin from the fibre surface is promoting the fibre/resin interface by increasing the interlocking strength, chemical bonding strength between fibre and resin, and eliminating the weak interface.

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3.2 Ball milling machine:

A ball mill is a type of grinder used to grind materials into extremely fine powder for use in mineral dressing processes, paints, pyrotechnics, and ceramics.

Fig. 3.2.Planetary Ball Milling Machine

4. FABRICTION AND TESTING OF SISAL NANO FIBREREINFORCED POLYMER NANO COMPOSITES

4.1 Materials Used:

Many materials are required to prepare a composite laminate**.**

Following is the list of materials used while preparing the laminate:

- Die, Resin. (Ly 556), Hardener. (Hy 951), Glass fibers. (E-glass), Ply, Sisalnano powder, Other accessory materials.

4.2 Preparation for composites:

Preparation for making a composite material is done in two phases.

Phase1: Ply and laminate preparation

Phase2: Test specimen preparation

4.3Manufacturing method for composite:

Hand lay –up:

It is manual fabrication process. It involves building up layers of chopped glass or woven glass mat impregnated with catalyzed resin around a suitable mold. The reinforcement is then rolled for better wet-out and removing trapped air. Resin is mixed with a hardener if working with epoxy, otherwise it will not cure (harden) for days/weeks. Next, the mold is wetted out with the mixture. The sheets of fiberglass are placed over the mold and rolled down into the mold using steel rollers. The material must be securely attached to the mold; Air must not be trapped in between the fiberglass and the mold.

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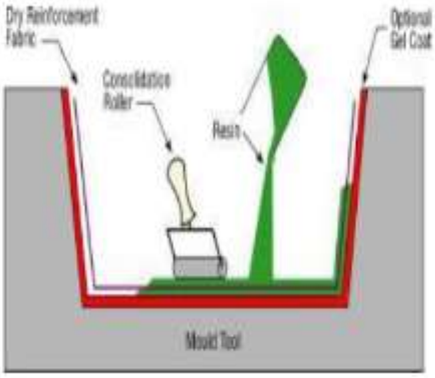

Fig.4.1, Hand Lay-Up

Various curing times can be achieved by altering the amount of catalyst employed. It is used for large diameter structure and custom shapes like asymmetric shapes and for bonding two or more modules.

Fig4.2. Sisal Nano Fiber Reinforced Polymer Composite.

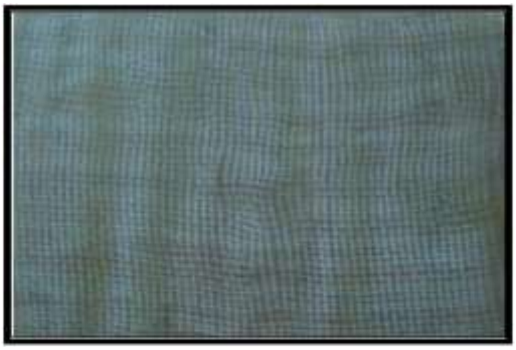

Fig.4.3. Glass Fiber Reinforced Polymer Composite.

<u>Phase 2:</u>

Preparation of test specimens:

After laminate is prepared it needs to be cut to specified dimensions according toASTM-D-3039 standards and nature of test to be conducted. The test conducted is tensile test. The dimension for tensile specimen is 25.4cm x 2.5 cm x 2mm.

According to standard composite materials was cut into 3 pieces. And for gripping purpose 2.5cm square rubber materials was attached at the end of the specimens. There are two varieties of composites the above three are sisal nano fiber reinforced polymer composite and below three are glass fiber rein forced polymer composite.

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Fig.4.4. Tensile test specimens

5. RESULTS AND DISCUSSIONS

5.1 Chemical Analysis:
The sisal surface is covered with large amount of lignin. Surface lignin plays an essential role in the surface characteristics of fibre and the Interfacial behavior of composites prepared with cellulosic materials. These conditions do not favor effective bonding at the fibre interface.
Initial weight of the sisal fiber is = 90g.
After chemical analyses final weight of the sisal fiber is =65.18g.
The cellulose content in the fiber is =72%.
Lignin content in the fiber is =28%.

5.2 Planetary Ball Mill:
- Total material taken is 65gms.80 balls are taken for grinding.
- The speed of the ball mill is 240r.p.m and the medium is dry.
- The grinding time of the material is 10 hours. By 10 hours of grinding machine sisal fibers are reduced to nano level.

5.3 X-ray Powder Diffraction (XRD):
X-ray powder diffraction is used for phase identification of a crystalline material. X-ray diffraction is now a common technique for the study of crystal structures and atomic spacing.

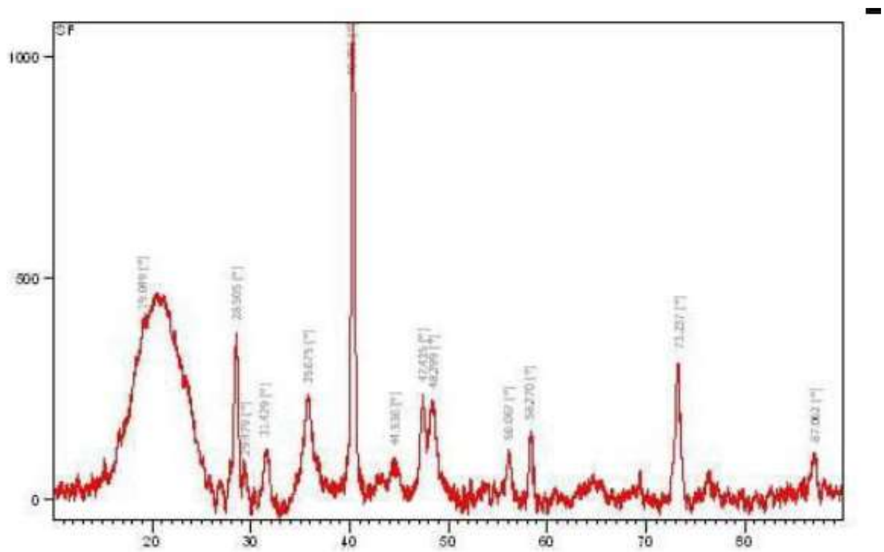

Fig 5.1 X-ray Powder Diffractogram

These X-rays are directed at the sample, and the diffracted rays are collected. A key component of all diffraction is the angle between the incident and diffracted rays. Peak position occurs where the X-ray beam has been diffracted by the crystal lattice. Peak position occurred at 40.29 degrees 2-theta.

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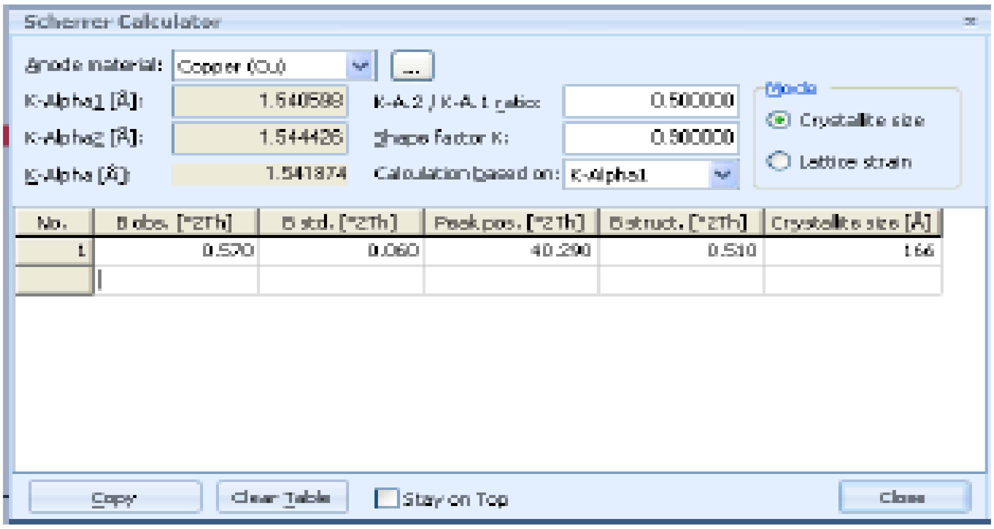

Fig 5.2.Crystallite size was measured automatically by the system software.

5.4 Scanning Electron Microscope (SEM):

A scanning electron microscope (SEM) is a type of electron microscope that images a sample by scanning. The electrons interact with the atoms that make up the sample producing signals that contain information about samples surface topography, composition, and other properties such as electrical conductivity.

The morphology of the sisal nano particle are

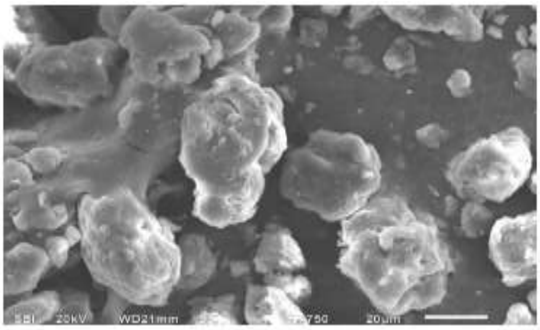

Fig. 5.3. SEM Micrograph of Sisal Nano Particle at 20 microns

5.5 Energy-dispersive X-ray spectroscopy (EDX or EDS):

Energy dispersive X-ray spectroscopy is used for the elemental analysis or chemical characterization of a sample.

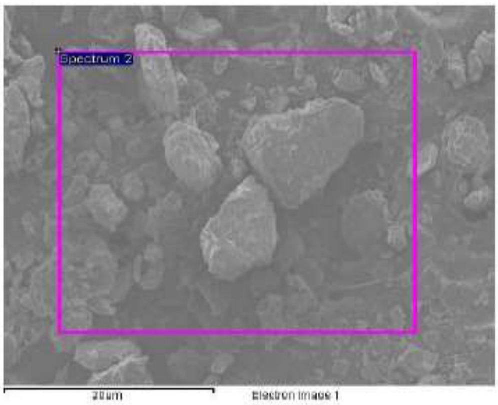

Fig. 5.4 Spectrum processing electron image at 20 microns

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Element	Weight%	Atomic%	Compd%	Formula
B K	20.70	27.23	66.66	B2O3
C K	8.90	10.53	32.60	CO2
Na K	0.03	0.02	0.04	Na2O
Si K	0.15	0.07	0.31	SiO2
Ti K	0.00	0.00	0.00	TiO2
Cu L	0.05	0.01	0.07	CuO
Ag L	0.09	0.01	0.10	Ag2O
Ba L	0.21	0.02	0.23	BaO
O	69.87	62.10		
Totals	100.00			

Table 5.1. Inorganic Materials in the Sisal Nano Powder

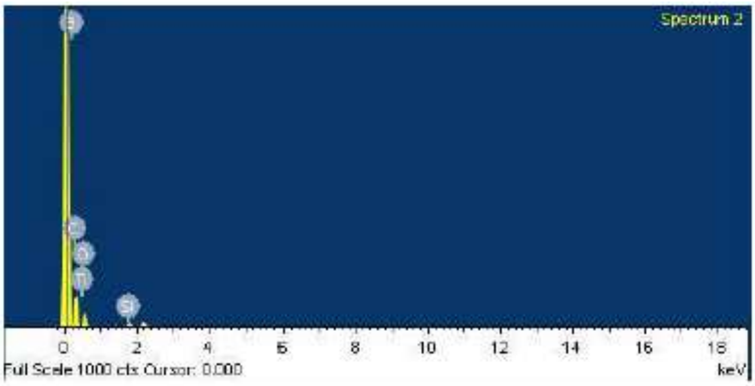

Fig. 5.5EDX Spectrum of the Sisal Nano Particle

5.6 Testing of sisal fibre composites:
Tensile test is conducted on 2 varieties of specimens. Three specimens are glass fiber reinforced polymer composites and three specimens are sisal nano fiber reinforced polymer composites and are tested for tensile properties. Tensile tests are conducted on tensile testing machine at Naval Science & Technology Laboratory, Visakhapatnam

Test results are:

Table-5.2

Specimen No.	Type of Specimen	Load at Break (KN)	Tensile Strength (MPA)
1	GFRP	2.665	117
2	GFRP	2.96	117
3	GFRP	2.945	129
4	SNFRP	3.155	138
5	SNFRP	3.2	140
6	SNFRP	3.22	141

GFRP: Glass fibre reinforced polymer composites.
SNFRP: Sisal nanofibre reinforced polymer composites.

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Table-5.3This test reports observes for each of the specimen to know the stress, strain andload variations before the specimen fails under ultimate load.

SAMPLE NO	AREA (mm2)	TEST SPEED (mm/min)	ULTIMATE BREAK LOAD (KN/mm2)	ULTIMATE STRESS (KN/mm2)	% of ELONGATION
1	22.860	15	2.665	0.117	4.606
2	22.860	15	2.945	0.129	2.458
3	22.860	15	3.155	0.138	4.691
4	22.860	15	3.220	0.141	4.9

The tensile strength values of sisal nanofibre reinforced polymer composites (SNFRP) and glass fibre reinforced polymer composites (GFRP)are compared.

SNFRP	GFRP
138	117
140	117
141	129

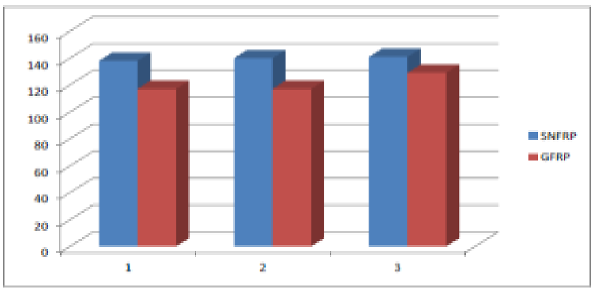

Fig 5.6.comparison between the glass fibre reinforced polymer composites (GFRP) to the sisal nano fibre reinforced polymer composites (SNFRP)

6. CONCLUSION

- By doing chemical analysis cellulose content in the sisal fibre is found 72% and lignin is 28%.
- Chemical treatment has been found to be effective in this respect by removing the deleterious constituents such as lignin, hemicelluloses which affect the bonding strength between the sisal fibres.
- The chemically treated fibers are reduced to nano level by using planetary ballmilling machine. Ball milling was carried out for 10hours.
- By 10 hours of ball milling the crystallite size is found as16 nanometers using Xray-diffracto meter.
- The morphology and inorganic materials of sisal nano particle are found by scanning electron microscope.
- Composites are fabricated by Hand lay-up process.
- Glass fibre reinforced polymers and sisal nano fibre reinforced polymers are prepared.
- The tensile properties of sisal nanofibre reinforced polymer composites and glassfibre reinforced polymer composites are evaluated by using tensile testing machine.
- It is observed that the sisal nanofibre reinforced polymer composites have highstrength compared to glass fibre reinforced polymer composites.
- Addition of sisal nano particles to the composites will improve the mechanical properties of composites.

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CITE AN ARTICLE

Saranya, G., Naidu, M. K., Mr, & Rao, P. K., Mr. (2018). CHARACTERIZATION AND SYNTHESIS OF NANO SISAL FIBER REINFORCED COMPOSITES. *INTERNATIONAL JOURNAL OF ENGINEERING SCIENCES & RESEARCH TECHNOLOGY*, *7*(12), 390-399.