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TENSILE, FLEXURAL AND IMPACT BEHAVIORS OF PARTICULATE/CALOTROPIS GIGANTEA STEM FIBER REINFORCED EPOXY COMPOSITES

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ABSTRACT

Increasing demand for the development of alternative materials to plastics and depleting wood stock ushered into the development of `new polymer composite materials. These polymer composites are either fiber reinforced or particles impregnated or hybrid of these two materials as well. The natural organic matter present in the reinforcement material contributed significant development in polymer composite's mechanical characteristics. The particulate and fiber reinforced composites was fabricated and tested independently. On comparison, the characteristics of epoxy composites reinforced with four different reinforcements including Chitosen, Red Mud, Rice husk particles and Calotropis Gigantea (CG) stem fiber were carried in the recent investigation. The improved value of characteristics and their respective composites were identified and suggested for industrial applications.

Keywords: chitosen, red mud, rice husk, calotropis gigantea, epoxy, mechanical characteristics.

1. INTRODUCTION

Scientists are exploring the various opportunities of combining bio fibers like hemp , sisal, flax, banana , jute, wood and grasses from renewable resources with polymer matrices to form composite materials and make the natural fiber composite uprising revolution into a reality, as reported by Sanadi et al (1986). Natural fibers are one of the renewable sources among the developing countries as they pose no health hazards, Cheaper and enables a solution to pollution by defining new applications of waste materials. Moreover, natural fiber reinforced polymer composites formulate a different new variety of material that has potential to be an alternative to wood and wood based materials that are scarce and are more needed for structural applications (Saheb *et al,* 1990).

The researcher, Pothan *et al* (1997) examined the flexural, tensile and impact behaviours of fiber reinforced polyester composites of short banana reinforcement in relation with the impact of fiber length and content. The fiber length of 30-40 mm and fiber loading of 40 % were found to have increased the mechanical characteristics. Further, they had suggested that banana fiber reinforced composites have smoother surface and expected to be a better alternative material for recpacing the conventional wood.

Rice is one among the cereal grains that yield hull fibers. In recent times Corn, wheat, oats, rye and other cereal crops can yield fibers. Khalil *et al.* (2001) investigated the mechanical characteristics of polymer composites filled with rice husk and derived the co-relation between coupling agent, loading of fiber, process ability, hybridization effect and hydrothermal aging on the mechanical characteristics of the composite. Singleton *et al* (2003) examined the mechanical- physical characteristics of natural flax fiber and also done the recycling of polyethylene composite of higher density, which was man-made by means of hand lay-up followed by compression- molding technique. The mechanical characteristics of were evaluated under impact loading and tensile loading as well. The noteworthy improvement of toughness of the composite has been qualitatively explained based on the deformation and the failure mechanisms recognized using the microstructures obtained from optical & scanning electron microscope.

Sain Mohini & Suhara Panthapulakkal (2006) studied the properties like agro residue like corn stalk, wheat straw, and corn cob reinforced high density polyethylene composites as an alternative to wood fibers. The demand increase in the field of composites has increased the usage of synthetic materials in various engineering applications. Jayabal *et al* (2010) investigated on natural fiber reinforced composites and they highlighted the coir fiber composites act as a substitution for synthetic fiber composite in automobile applications. The lower cost & higher-modulus weight ratio provides the application of coir fibers to be used as fiber reinforcement in the applications of fiber-reinforced-composite.

Alewoopuada *et al* (2015) described the effect of particle size and concentration on the mechanical characteristics of polyester with data palm seed particulate composition. The use of cellulosic material as reinforcement in composites data palm seed particle size with variables 0.5, 2.0 and 2.8 mm and particle loading variable upto 5% to 25% wt. was studied. The results of the various types of mechanical tests of the composite indicated the use of data palm seed particles as reinforcement can enhance the characteristics of polyester composites.

Kaimeng Xu *et al* (2016) delibtrated the effect of environmental-friendly- modified rubber seed shell with the physical characteristics of high density polyethylene rubber seed shell composites. Further they stated that the various particle size of rubber seed shell mesh and conducted the mechanical and thermal characteristics. Average size of seed particles upto 105µm composites was produced and investigated for tensile test, and it conformed to the experimental tensile characteristics. This study is the main reason for new polymeric composite for engineering application.

The review of literature provided wealth creation through waste as well as agro products by reinforcing fiber and particles of agro products for industrial applications. As stated by most of the researchers, the 30 % of weight content provided optimum reinforcement in polymer composites. If particles are used, it is around 20% for getting better value of mechanical characteristics. The particle size of 50 µm gives better surface to volume contact in micro level particle composites. Keeping these observations, the present investigation is focused on the development of new variety of composites using red mud, chitosen and rice husk particles.

2. MATERIALS AND METHODS

The constituent materials, fiber testing, characterization of fibers, fiber treatment and manufacturing of composites using compression moulding technique are deliberated in this present work. The investigation of tensile strength, flexural strength, impact energy of red mud, chitosen, rise husk particle and CG-Fiber reinforced epoxy composites is also delineated.

2.1 Materials

2.1.1 Red mud particles

Red mud is generated from MALCO (Madras Aluminium Company limited) at Salem, India. It is enriched more with the Iron oxide followed by silicon di oxide and aluminum oxide. Instead of going as industrial waste, it is effectively used as the reinforcement for strengthening matrix in epoxy composites in the investigation. The presence natural organic matter in red mud provided enhanced value of mechanical characteristics when reinforced with epoxy matrix.

2.1.2 Chitosen particles

The use of calcium oxide enriched particles can improve the static and dynamic mechanical strength of the composites. In this experiment, calcium enriched bio - particles were selected as particle reinforcement for epoxy matrix composite. Natural calcium material is selected as particle reinforcement because it is copiously available and being wasted as sea dumps. The use of natural calcium will help in using bio waste effectively for engineering applications. The size of the natural calcium particles used in the present investigation is 50 µm.

2.1.3 Rice husk particles

Rice husk (RH) is one of the waste materials generated in agricultural practices and available abundantly in rice based agricultural countries. It is a natural waste sheath that is formed over rice grains during its development and the same is detached at the time of filtering of rice and so removed husk is considered a insignificant material in commercial applications. Rice husk is a fibrous material and it helps as a filling material during the making of light weight polymer composites. It is enriched with natural silica that influenced better value of characteristics.

2.1.4 Calotropis gigantea (CG) fibers

Calotropis Gigantea (CG) fiber belongs to the family of Apocynaceae and is inhabitant to China, Malaysia, India, Cambodia, Indonesia and various regions of the world. This plant will grow up to 3-5 m height and produces waxy-flowers in cluster. Furthermore, it contains pale green leaves in oval shape and stem is milky in nature, which get enriched with more amounts of fibers. The fibers extracted from the stem of Calotropis Gigantea plant are used for composite manufacturing. The chemical analysis is carried out on the extracted CG fibers as per Technical Association of the Pulp and Paper Industry (TAPPI) Standard. The cellulose content of 73.8 % and hemicelluloses of 20.8 % were observed in the CG fibers during the chemical test.

2.1.5 Epoxy matrix

In thermoset matrix materials, epoxy, polyesters, cynate ester and vinyl esters are frequently used for marine, automotive, chemical, and electrical applications as matrix applications. Epoxy is utilized effectively as a laminating resin and also as an adhesive for most of the engineering applications. Epoxy yields 3.5% to 4.5% elongation with tensile force, during failure in comparison typical polyester resins having 1% - 2% elongation. It shows wonderful moisture-obstruction quality, in composites. It binds very well to fibers for creating fiber reinforced composites. Polymerization reaction is done for the transformation of liquid resin to solid phase is conducted by adding minimal quantity of a reactive curing agent just before incorporating fibers into the liquid mix. One such curing agent is amine based hardener (HY 951) and is utilized for preparing the composites on a mixing ratio of 9:1 as recommended. The epoxy resin (LY 556) and the hardener were supplied by M/S Covai Seenu & Company, Tamilnadu, India.

2.1.6 NaOH pellets

Sodium hydroxide pellets supplied by M/S Spectrum Reagents and Chemicals Private Limited, Edayar, Cochin, India were used for the alkaline treatment of CG fibers. Approximately 30 grams of CG fiber is soaked in 800 ml of NaOH alkaline solution. The fibers are separated, rinsed with pure water multiple times and rinsed with distilled water finally, to eliminate any NaOH presence on the fiber. Chemical tests are conducted on CG fiber after the treatment with concentrations of 5 % of NaOH solution.

2.2 Extraction and Characterization of Fibers

Calotropis Gigantea is a big shrub containing bunches of waxy flowers in either lavender or white in color. The plant consists of oval and pale green leaves and milk stem. Calotropis Gigantea yields a fiber which is very durable in nature and can be used for fishing nets, ropes, sewing thread and carpets. The pollination of this plant is mainly done by wind and insects. The current examination is dedicated for the extraction and processing of CG fiber from the stem.

2.2.1 Fiber extraction

The CG fibers are extracted from stem of the plant of one year age. First, the leaves are removed. Second, the required length of stems is shaped by cutting the stem. Then, it is allowed to get dry in room temperature for 5 days. The fibers are separated from the dried stems manually by hand de-cortication method which is the removal of outer bark initially followed by extraction of fiber through scraping with a sharp knife and dried in atmosphere. Finally the fibers are allowed to dry at room temperature for 2 days and cut into 30 mm in length for the making of composite plates. The photographic image of CG plant along with extracted fibers is shown in Figure-1.

Figure-1. Photographic image of CG plant and fibers.

2.2.2 Surface modification

The CG crown fiber is washed with water and gets dried out at room-temperature for 2 days. The surface modification is done by alkali treatment of the fiber with 5 % of NaOH concentration for 30 minutes. This type of treatment removes partial quantity of lignin, oil and wax covering the external surface of the fiber cell wall. It has been stated that treatment with alkaline shows couple of effects on fiber. It improves roughness, ensuring improved mechanical interlock with increasing the cellulose visible on the surface of fiber, thereby improving the number of possible reaction sites. Now, the fiber surface is adequately rough and has better adhesion characteristics than polymers. The fiber could be utilised as a better reinforcement in polymeric composites based on the analysis of tensile strength of raw and treated fibers by single filament testing.

2.3 Single Filament Testing

The fibers thus extracted are subjected to mono filament test for evaluating the tensile characteristics of the fiber. The monofilament of each fibers is tested using the Instron Model 5500 R machine. The rate of loading of 5 mm/min until the fiber fractures at a temperature of 21 ± 1^0C and at humidity of 55 ± 2 %. To obtain a statistically significant result for each condition, 20 fibers from each case are tested to evaluate the average tensile strength and average tensile elongation. The raw fiber can with stand up to 8 N force whereas the 5 % NaoH treated fiber can with stand up to 9 N force during mono filament testing.

2.4 Composites Manufacturing

ACE make hydraulic compression molding machine with a capacity of 30 tons is utilized to construct the CG fiber-epoxy composite plates for the dimensions of 30 × 30 × 0.3 cm. The appropriate weight content of CG fiber and epoxy were mixed using mechanical stirring at 20 rpm for 10 minutes at the room temperature. The particulate content of 20 % and fiber content of 30 % were selected as reinforcement percentage as per the literature review. The fiber length of 30 mm was selected during the fabrication of short CG fiber reinforced epoxy composites. The fabricated composites maintained with pressure of 2.6 MPa and temperature of 80°C for 0.45 hrs facilitated proper curing. The environmental conditions of 28^0 C temperatures with relative humidity of 55% are documented at the time of manufacturing of composites. The photographic image of compression molding machine set up is shown in Figure-2.

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Figure-2. Photographic image of 30 Ton compression molding machine.

2.5 Mechanical Testing of Composites

2.5.1 Tensile testing

The Dual Column Digital Universal Testing Machine (Tinius Olsen H10KL) equipped with a 5KN load cell is used for tensile testing according to standard namely ASTM D 638.

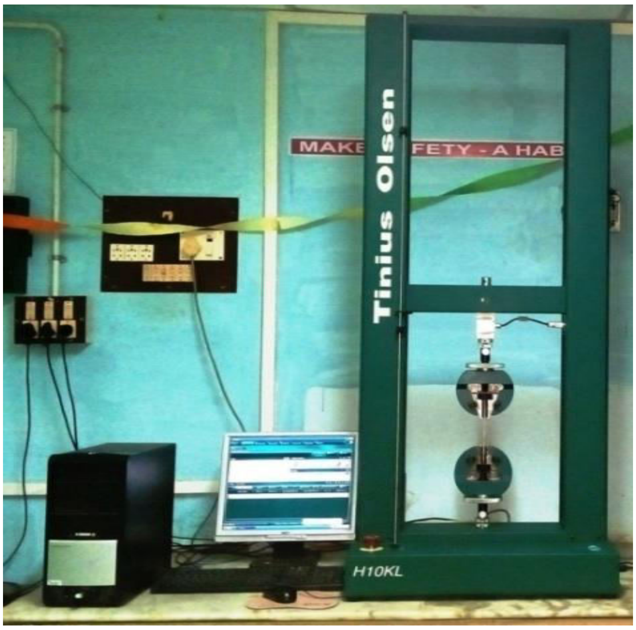

Figure-3. Photographic image of Tinius Olsen H10KL tensile testing set up.

Five samples with size 165 x 13 x 3 mm^3 are tested for each case, setting the cross-head speed to 1 mm/min and the average value is noted as the tensile strength of respective composite samples. From the load-time record of the test, the tensile characteristics are determined by using the following Equation (1).

$$\text{Tensile strength, } \sigma_t = \frac{Force at failure}{Cross sectional area of the specimen} \quad (1)$$

Five samples have been tested and the average value is reported. The photographic image of tensile testing set up is shown in Figure-3.

2.5.2 Flexural testing

Flexural tests (Three point bending) are done using Tinius Olsen H10KL (Column Digital Universal Testing Machine) which equipped with a load cell of 5 KN, as per ASTM D 790 standards. 5 prismatic samples with a length of span of 48 mm are tested with crosshead speed of 2.5 mm/min. The flexural testing set up is shown in Figure-4.

Figure-4. Photographic image of Tinius Olsen H10KL flexural testing set up.

A three point flexural test is conducted for the specimen dimensions of 125 × 12.5 × 3 mm^3 and five samples of flexural specimen are tested to obtain statistically significant result for each condition. The flexural strength in a three-point bending test is given by

$$\text{Flexural strength, } \sigma_f = \frac{3}{2} \times \frac{Failure load \times specimen length}{specimen width \times (specimen thickness)^2} \quad (2)$$

2.5.3 Impact testing

Izod impact tests are carried out using a Tinius Olsen (Model 104) impact tester according to ASTM D 256. The machine is equipped with a pendulum of a potential energy of 2.57 J. Five un-notched samples of size

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64 x 12.7 x 3 mm^3 are tested for each case and the average value is noted as the impact strength. The specimens for impact test are cut from the manufactured composite and finished to the accurate size using emery paper. The maximum pendulum capacity of impact tester is 25 J and maximum impact velocity is 3.46 m/s.

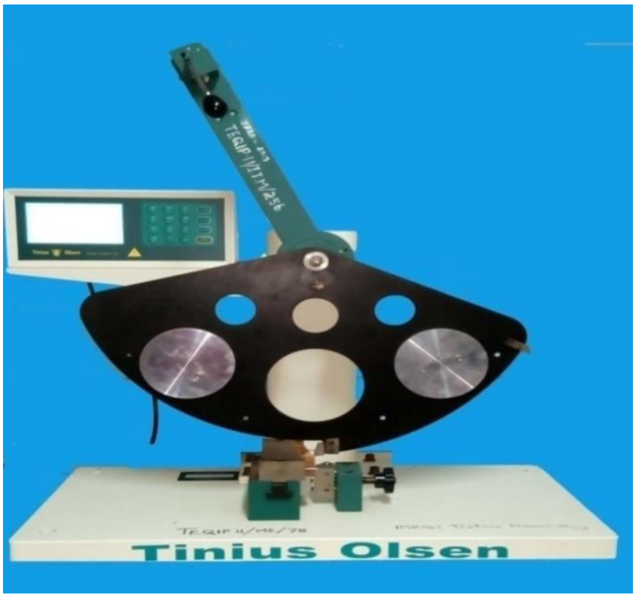

Figure-5. Photographic image of 104 impact tester.

The test specimen is supported as a vertical cantilever beam and broken by a single swing of a pendulum. The pendulum strikes the face of the sample and a total of 5 samples are tested; the mean value of the absorbed energy is taken. Energy absorbed (*U*) by the specimen is calculated using the following formula:

$$U = \frac{1}{2} \times \frac{\text{Weight of striking head}}{\text{Acceleration due to gravity}} \times (\text{velocity before impact}^2 - \text{velocity after impact}^2) \quad (3)$$

The photographic image of 104 impact testing set up is shown in Figure-5. All the mechanical tests are conducted in the atmospheric conditions of 23±2^0 C temperature and relative humidity of 50 ±5 %.

3. RESULTS AND DISCUSSIONS

The tensile, flexural and impact characteristics of epoxy composites reinforced with particulates/CG fiber are given in Table-1. The chitosen-epoxy composites revealed lower value of mechanical characteristics whereas rice husk-epoxy composites exhibited enhanced value of mechanical characteristics. The sample identification S1, S2, S3 and S4 are given for epoxy composites reinforced with Chitosen, Red mud, Rice husk and CG fiber respectively.

Table-1. Tensile, flexural and impact characteristics of epoxy composites reinforced with particulates/CG fiber.

Sample ID	Particulate /Fiber	Resin	Flexural Strength (MPa)	Tensile Strength (MPa)	Impact Strength (kJ/m^2)
S1	Chitosen	Epoxy	27.2	20.2	36.8
S2	Red mud	Epoxy	31.5	23.0	39.1
S3	Rice husk	Epoxy	34.5	34.2	47.2
S4	CG fiber	Epoxy	80.3	64.3	56.2

3.1.1 Tensile properties

The tensile characteristics of the four samples of composites are given in the table and the corresponding graph (Figure-6). The tensile strength of 20.2 MPa, 23 MPa, 34.2 MPa and 64.3 MPa were obtained in chitosen - epoxy, red mud-epoxy, rice husk - epoxy and CG fiber-epoxy composites. In general, the mechanical characteristics are better in natural fiber composites as compared with micro particles impregnated agro residue composites. The same pattern is observed in this investigation also and the rice husk - epoxy composites revealed better value of mechanical characteristics in particulate composites.

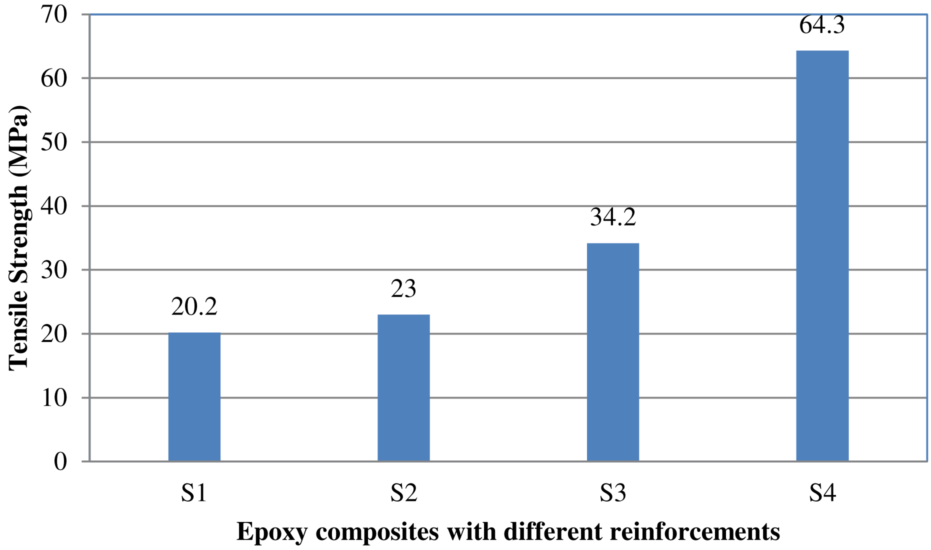

Figure-6. Tensile behaviours of epoxy composites reinforced with particulates/CG fiber.

3.1.2 Flexural properties

The flexural behaviours of epoxy composites reinforced with particulates/CG fiber are shown in Figure-7. Flexural strength is important for many products, especially composites used in demanding applications. The flexural strength of CG stem fiber- epoxy composites are more than twice that of riche husk-epoxy composites. This is due to the transfer of load to the continuous reinforcement in terms of fibers instead of particles through matrix.

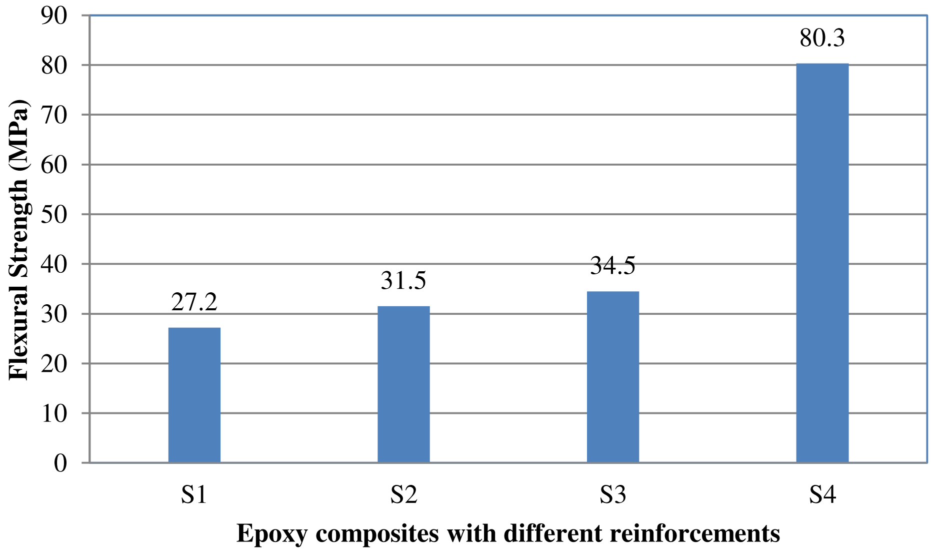

Figure-7. Flexural behaviours of epoxy composites reinforced with particulates/CG fiber.

3.1.3 Impact properties

Impact strength is the capability of the material to withstand a suddenly applied load and is expressed in terms of energy. The un-notched Izod impact test was carried out on the newly developed epoxy composites. The better value of impact strength of 56.2 kJ/m^2 was obtained in CG stem fiber reinforced epoxy composites. Among the particulates, rice husk-epoxy composites exhibited the better value of impact strength of 47.2 kJ/m^2. The rich content of SiO_2 materials in rice husk particles provided better mechanical characteristics. Next to that the presence of SiO2, MgO, Fe_2O_3 and $CaCO_3$ in red mud particles provided significant mechanical characteristics. The 8 Impact behaviours of epoxy composites reinforced with particulates/CG fiber is shown in Figure-8.

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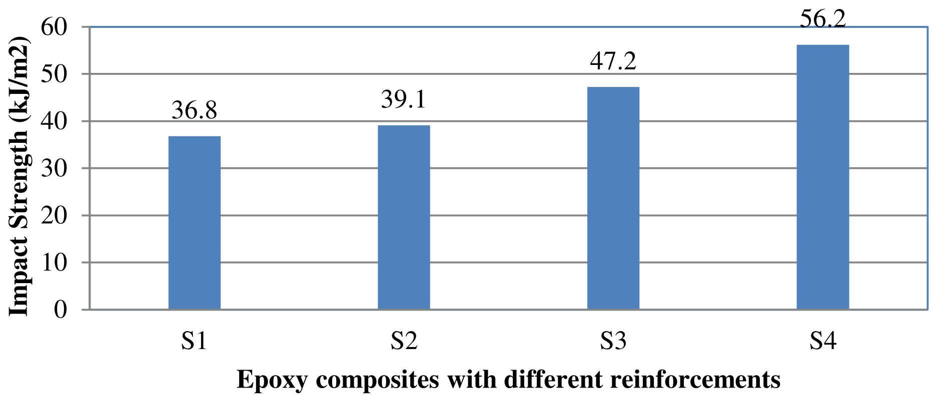

Figure-8. Impact behaviours of epoxy composites reinforced with particulates/CG fiber.

CONCLUSIONS

The tensile, flexural and impact characteristics of Chitosen, Red-mud, Rice Husk and CG fiber – epoxy composites were investigated in the present work. It was observed that the value of CG fiber -epoxy composite is high in all mechanical characteristics recorded. Next to CG fiber-epoxy combination, Rise Husk particle reinforced epoxy composite stands better than other two combinations. From this it's a clear statement that a new composite can be made by reinforcing CG fiber and Rice Husk particles with epoxy which would bear much better sustainable mechanical characteristics. The characteristics obtained for the rice husk-epoxy composites and CG stem fiber - epoxy composites are significantly comparable with the other particulate/fiber composites. Thus a novel approach of producing composites from agro waste products is highlighted.

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