

Mechanical and dimensional stability behaviours of alkali-treated calotropis gigantea fibre-reinforced bio-particles impregnated epoxy composites

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ABSTRACT

Calotropis gigantea fibre has been identified as suitable reinforcement fibre in polymer composites by current researchers. Epoxy matrix provided better interfacial bonding with the fibres and particulates to enhance the mechanical properties of pure polymer substances. If elements such as chitosan, rice husk and red mud have been added in epoxy system, it improves the mouldability and fills the missed portion of fibre reinforcement in composites. Alkaline treatment of natural fibres produce conversion of cellulose and production of short length crystallites which enhances surface roughness on the fibre surface for developing better adhesion between constitution materials of composites. The new variety of natural fibre extracted from stem of Apocynaceae family plant has been used in epoxy matrix along with bio and industrial residues in the present investigation. The three point flexural strength, tensile behaviour and izod impact strength have been evaluated and dimensional stability characteristics are identified for the calotropis gigantea fibre reinforced particulates impregnated epoxy composites.

1. Introduction

Evolvement of the world lead the researchers in finding new materials by waste management strategy, and one among them is producing new sustainable eco-friendly materials from bio and agro waste. Many studies have been carried out in producing natural fibre composites for a very long period of time. The composites of Natural fibre are observed to be ecologically superior to glass fibre composites in many circumstances because of the subsequent reasons; (a) Natural fibre manufacturing has lesser ecological effects in comparison to Production of the glass fibre, (b) the composites of natural fibres

possess greater fibre elements for equal functional performance, decreasing the base polymer content which contributes to more pollution, (c) these composites increase fuel effectiveness because of its lighter-weight and decrease emissions, particularly in the applications of auto-mobile sector, and (d) the finish of lifetime incineration of natural fibres effects in improved carbon credits and energy [1].

The thermo-physical performance of PALF(pineapple leaf fibre) and glass fibre reinforced polyester composites was assessed, intended for a consistent total fibre loading, the outcomes exposed that

the interaction of fibres with chemical environment decreases the Heat resistance of composite [2]. The trials that test the ability to absorption of water were conducted to determine the gain of mass and specified that fibre composites treated with alkali absorbed extra water than untreated or silane treated composites [3]. Natural fibres are strong, lightweight and also very cheap composites are established and the mechanical behaviour was valued. These effects how that the coir can be utilised as potential reinforcing material for manufacturing small load bearing thermoplastic composites [4].

An experimental analysis was conducted to examine the flexural, dielectric and tensile characteristics of composites prepared by incorporating vakka as an innovative fibrous material on a matrix of polyester resin. For the purpose of insulation in electrical applications, the fibre composite of vakka is recommended because of its dielectric property [5]. Recently scientific researchers and engineers are attracted by natural fibres as it can be served as a substitute reinforcement material for FRP (fibre reinforced polymer) composites. Owing to the low price, equally decent mechanical characteristics, great strength, eco-friendly, biodegradability and un-abrasive characteristics, these materials are utilised as an auxiliary replacement for the orthodox fibre, for example aramid, carbon and glass [6].

A comprehensive overview was provided in literature on various treatments on surface of natural fibres for innovative applications using composites [7]. The best ratio of the blend of chemical additives is required in order to accomplish a stability between durability and strength [8]. There are many challenges faced at the time of compression and injection moulding, the best methods for establishing natural fibre composites [9].

Based on the fibres like bamboo, coir and flax, the compressive characteristics of three various natural fibre composites was analysed. As far as compression is concerned, bamboo out performs as the best among other fibres. While comparing the compressive characteristics to tensile it is evident that bamboo and flax composites extent between 80% and 60% of tensile values, which is very reassuring. While comparing compression with tension, Coir fibres perform better [10].

Polypropylene hybrid composites are created by means of coir fibres and coir shell elements with entire

weight content of 20%. The structural and morphological effects exposed that the coupling agent improved filler/matrix interfacial bond. The blend of coir shell and coir fibres elements possess a progressive result over the Young's moduli through hybrid composites presenting transitional values among non-hybrid composites (progress in the range of 35 and 50%) [11]. The properties of pine needle and mustard cake on impact energy, abrasion and tensile strength of sisal fibre made polymer composites was examined. 9 various samples was made prepared using various sisal fibres reinforcements (In the wt% range of 20, 30 and 40) in matrix of polyester along with hybridization of those on agro-waste mustard cakes and with natural pine needles at a fixed amount (5 wt%) each for adapting the wear and mechanical Characteristics. The blend of pine needles and mustard cakes improved the wear and mechanical characteristics of polyester composites based on Sisal along with improved outcomes gained with the fillers of pine needles than with mustard cakes. Amongst all, the hybridized composites (polymer) made up with sisal (40 wt%) and pine needles (5 wt%) provided higher mechanical and wear characteristics [12]. A superficial pyrolysis process was reported to get an ecologically-friendly and also bearable oil absorption material by means of *calotropis gigantea* (CG) which is plant fibre with consists of a hollow-tubular arrangement as a preliminary base input material. The consequential carbon-fibre preserves the hollow arrangement still, despite shrinking diametrical shape and size of curl edge. While usage on an application of absorbent, carbon fibres could efficiently and quickly absorbs different oils [13].

As a new attempt as raw material, CG is used the manufacturing of components like helical springs. Keeping shock absorber as die, the poring is turned in the lathe after the fibre is dipped on a matrix. The outcomes gained are streamlined with FEA (Finite Element Analysis) and found that under axial loading condition (coil surface) the specified material is isotropic in nature at transverse direction [14]. In order to lower the hygroscopic and hydrophilic nature, surface treatment was carried out with NaOH (sodium hydroxide) to develop the characteristics of the CG fibres. Determining the appropriate NaOH concentration in the treatment solution is essential to reduce the hydrophilicity of the fibres while retaining its mechanical characteristics [15]. By referring previous studies, the CG has been identified as alternative source to synthetic fibres as reinforcements in polymer composites in recent years. With this, the focus was

made on fabrication and testing of mechanical and dimensional stability behaviours of alkaline treated CG fibre reinforced particles impregnated polymer composites.

2. Materials, Fibre treatment, and Manufacturing

2.1 Materials

Red mud is collected from MALCO (Madras Aluminium Company Limited) at Salem, India. It encompasses a rich amount of Iron oxide followed by aluminium oxide and Silicon dioxide. Rather considered unproductive, it can be used as the reinforcement for strengthening the matrix in epoxy composites in this study. The use of calcium oxide enriched particles can improve the static and dynamic mechanical strength of the composites Plates. In this research, 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 unused as sea dumps. Rice husk (RH) is a waste material generated in agricultural execution and available abundantly in rice cultivating countries. It is a natural waste sheath that is formed over rice grains during its development and the same is removed during the refining paddy into rice and consequently detached husk has no viable attention.

The fibres extracted from the stem of CG plants are used to fabricate the composite. The chemical analysis is carried out on the extricate CG fibres as per the TAPPI (Technical Association of the Pulp and Paper Industry) Standard. The cellulose content of 73.8 % and hemicelluloses of 20.8 % were observed in the CG fibres during the chemical test. Epoxy yields tensile elongation of 3.5% to 4.5% at the time of failure in comparison with polyester resins (1% to 2%). It offers excellent moisture obstruction qualities when used in composites. One of the curing agents is an amine-based hardener (HY 951) and is utilized for preparing the composites on a mixing ratio of 9:1 as endorsed. The epoxy resin (LY 556) and the hardener were supplied by M/S Covai Seenu and Company, Coimbatore, Tamilnadu, India.

2.2 Fibre Treatment

Amongst various treatments used on natural fibres, alkaline treatment is more familiar, which is termed as mercerization. These kind of alkaline treatments will encourage elimination of partial amorphous components like waxes, lignin, hemicellulose which are soluble on alkaline solution thereby minimizing the level of aggregation of fibre along with changing the surface to rough. Throughout the treatment, OH groups which is

present in fibre reacted with NaOH. As a resultant of treatment, the positioning of filled crystalline cellulose order is changed and by swelling the wall of fibre cell, it formulates an amorphous region. This offers additional entrance to the diffusion of Chemical substances. Hydrogen bonds which are sensitive to alkalis exist amongst the breakdown of fibres and newly formed hydrogen bonds formed amongst molecular chains of cellulose. Due to this, hydrophilic hydroxyl clusters were partly detached and the moisture fighting character is enhanced. In addition, it releases an assured share of lignin, wax, hemicelluloses, pectin, and oil cover as an effect along with cleaning the fibre surface. In a nut shell, fibre superficial surface turn into more uniform surface due to the exclusion of micro-voids and hence stress transfer capacity among the ultimate cells increases. In addition to this, it reduces fibre diameter and thereby increases the aspect ratio. This results in better fibre-matrix interfacial adhesion. The mechanical and thermal behaviour of the composite material can be enhanced considerably with this type of treatment. In the case of more alkali concentration than the required optimal level, additional delignification of fibre takes place and creates damage to the fibre. The frees that are treated fibres possess little amount of lignin, limited removal of wax and oil cover along with expanding crystalline order of cellulose.

2.3 Manufacturing

A 30-ton capacity ACE-make hydraulic compression moulding machine is used to fabricate the CG fibre-epoxy composite plates for the dimensions of 300×300×3 mm³. The appropriate weight content of reinforcement and matrix (30 % fibre and 70 % resin) was stirred mechanically at 20 rpm for 10 min at ambient temperature. The fabricated composites maintained with a pressure of 2.6 MPa and a temperature of 80°C for 45 min facilitated uniform curing of composite sheets. The atmospheric conditions of 28°C temperatures and relative humidity of 55% are recorded during the composite manufacturing. The Photographic image of compression moulding machine is shown in Fig. 1.

3. Mechanical and Dimensional stability Testing

3.1 Mechanical Tests

According to ASTM D 638 standard, The Dual Column Digital Universal Testing Machine (Tinius Olsen H10KL) equipped with a 5 kN load cell is utilized for tensile tests. Five samples with size 165×13×3 mm³ are used for testing each case. The cross-head speed is set in

the range of 1 mm/min, and the tensile strength of respective samples is calculated using average value.

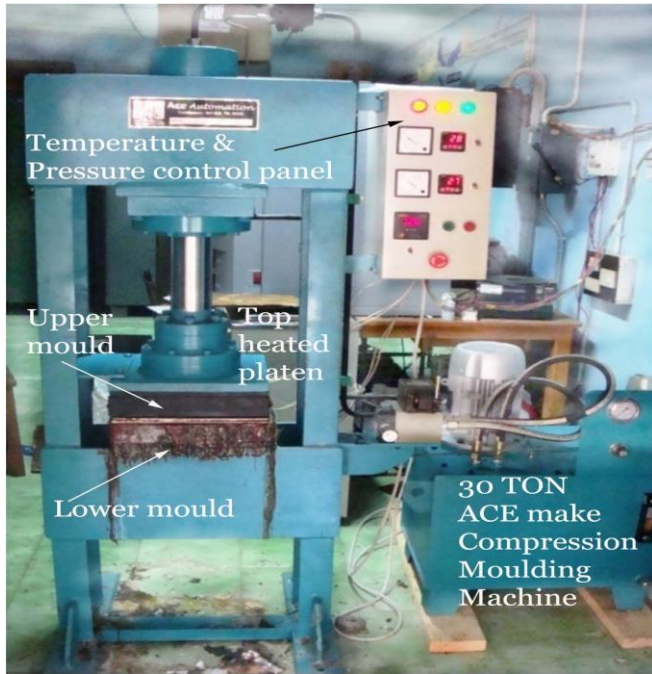


Fig. 1. Photographic image of ACE make Compression Moulding Machine

Flexural tests (Three-point bending) are executed by using a Column Digital Universal Testing Machine (Tinius Olsen H10KL) equipped with a load cell of 5 kN, according to ASTM D 790 standard. Five prismatic samples taking a distance length equal to 48 mm are tested for each case, setting the crosshead speed of 2.5 mm/min. A three-point flexural test is conducted for the specimen dimensions of 125×12.5×3 mm³ and five samples of flexural specimens are tested to acquire statistically significant results for each state.

Izod impact tests are conducted using a Tinius Olsen (Model 104) impact tester according to ASTM D 256. The machine is armed with a pendulum of a potential energy of 2.57 J. Five un-notched samples of size 64 x 12.7 x 3 mm³ are tested for each case and the average value is noted as the impact strength. The specimens for the impact test are severed from the manufactured composite and refined to the accurate size using emery paper. The maximum pendulum capacity of the impact tester is 25 J and the maximum impact velocity is 3.46 m/s. The Photographic image of Flexural, Tensile and Impact machine set up is shown in Fig. 2.

3.2 Dimensional Stability Testing

The water absorption characteristics of the particles reinforced CG fibre-epoxy composites were determined

according to ASTM 570-10 standard with the specimen dimensions of 75×25×3 mm. The specimens from each type of composition were taken and dried in an oven for 24 h at a temperature of 60°C and submerged in distilled water at ambient temperature for about 24 h. The variation of weight between dried and submerged samples was measured time after time using a high precision weight balance. The dimensional stability characteristic test set up is shown in Fig. 3.



Fig. 2. Photographic image of Flexural, Tensile and Impact machine setup

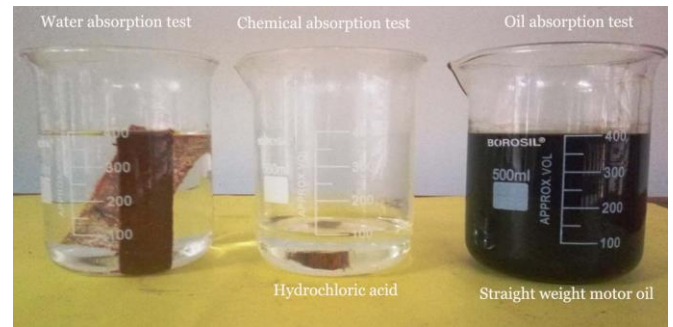


Fig. 3. Photographic image of dimensional stability characteristic test set up

The chemical stability of particles reinforced CG fibre-epoxy composites was fixated as per ASTM D 543-06 with a specimen dimension of 10 × 5 × 3 mm by dipping in the Hydrochloric acid for a duration of 24 hrs. This experiment covers the assessment of polymer materials for resistance to chemical reagents, boosting performance in possible end surroundings. Chemical reagents can include cleaning agents, lubricants, foods, ink or anything different that the test sample was expected to come in contact with. The weight loss/gain was obtained using the following formula.

$$\text{Weight loss/gain} = \frac{\text{Final weight} - \text{Original weight}}{\text{Original weight}} \times 100$$

4. Results and Discussion

The three-point flexural, tensile and izod impact strength of particulates impregnated CG-Epoxy composites are given in Table 1. In all cases, the better value of strength was obtained in alkaline treated CG fibre reinforced rice

husk particles impregnated epoxy composites. The microfibrils are surrounded by hemicelluloses and lignin in natural fibre which provides strength to the natural fibre. The chemical treatment removes wax, oil and other substances which covers the external surface of the fibre and reduces cellulose hydroxyl group in the fibre matrix interface. The sticking together of reinforcement and matrix are better thus enabled better strength in mechanical testing.

4.1 Flexural Behaviours

The rectangular beam specimen rests on two roller supports and is subject to a concentrated load at its centre provided bending behaviours. The better flexural strength behaviour was obtained in rice husk particles impregnated CG fibre reinforced epoxy composites. The presence of more amount of SiO₂ and remaining composition of Al₂O₃, Fe₂O₃, CaO, MgO, K₂O, Na₂O, and P₂O₅ in rice husk particles added strength to the epoxy system. The 2θ for the relative intensity of 100% and d-spacing of 4.00147 Å confirmed the presence of SiO₂ materials in rice husk particles. Silica is a metal oxide, which has good abrasion resistance and high thermal stability. The mixing of silica in epoxy matrix provided better interfacial bonding in addition with alkaline treated CG fibre in epoxy system. The three point bending strength characteristics of various particle impregnated CG-Epoxy composites are shown in Fig. 4.

4.3 Impact Behaviours

The izod impact strength characteristics of various particle impregnated CG-Epoxy composites are shown in Fig. 6. Impact toughness is the amount of energy that a material can withstand when it is subjected to sudden load. The better value of impact toughness of 56.2 MPa was obtained CG fibre reinforced epoxy composites which can be further enhanced by the addition of chitosan, red mud and rice husk particles.

4.4 Water, Oil, and Acid Absorption Behaviours

The Dimensional stability characteristics of particulates impregnated CG-Epoxy composites are given in Table 2. Dimensional Stability is a measurement of the dimensional change resulting from exposure to different environment. The degree to which the composite material maintains its original dimensions when subjected to exposure to water, oil and chemical were successfully studied. Compared with untreated CG fibre-epoxy composites, the NaOH untreated CG fibre-epoxy composites exhibited low value of dimensional stability behaviours.

Table 1

Flexural, tensile and izod impact strength of particulates impregnated CG-Epoxy composites

Sample ID	Composites	Flexural Strength (MPa)	Tensile Strength (MPa)	Impact Strength (kJ/m ²)
S1	Untreated CG	60.5	62.7	43.5
S2	Alkali treated CG fibre + epoxy	80.3	64.3	56.2
S3	alkali treated CG fibre + chitosan + epoxy	82.2	65.5	59.8
S4	alkali treated CG fibre + red mud + epoxy	84.7	66.8	60.5
S5	Alkali treated CG fibre + rice husk + epoxy	87.8	72.3	63.4

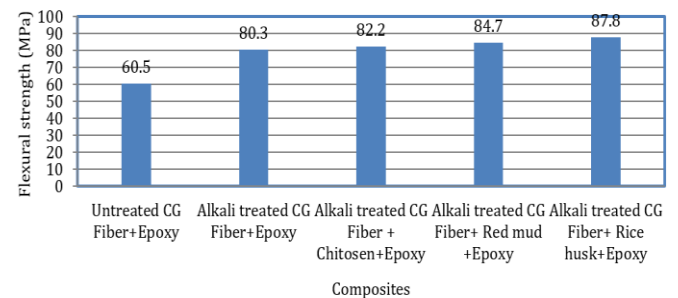


Fig. 4. Bending failure characteristics of particulates impregnated CG-Epoxy composites

4.2 Tensile Behaviours

The alkaline CG fibre reinforced epoxy composites filled with chitosan, red mud and rice husk particles exhibited better value of tensile strength of 65.5 MPa, 66.8 MPa, 72.3 MPa respectively. The tensile strength characteristics of various particle impregnated CG-Epoxy composites are shown in Fig. 5. During the tensile test, the load is transferred to the reinforcement material by epoxy matrix which provided better breaking strength due to the good adhesion between CG fibre and epoxy system. The presence Si, Mg, Fe, Ca, Al, K, Ti, Fe and their oxides in red mud particles

provided better strength to matrix system and comparable tensile strength value was obtained.

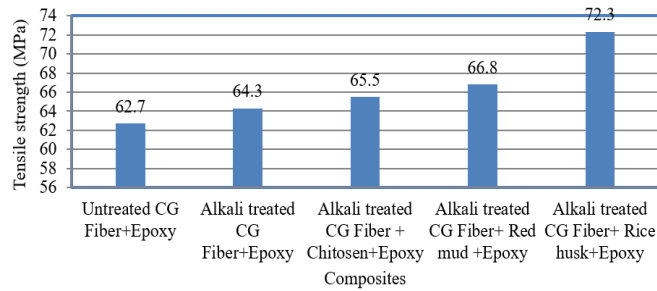


Fig. 5. Tensile failure characteristics of particulates impregnated CG Epoxy composites

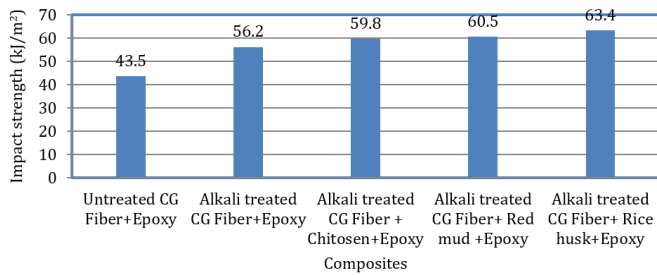


Fig. 6. Izod impact failure of particulates impregnated CG-Epoxy composites

Table 2

Dimensional stability characteristics of particulates impregnated CG-Epoxy composites

S. No.	Composites	Absorption rate (%)		
		Water	Oil	HCL
1	Untreated CG fibre + epoxy	3.9	3.1	0.82
2	Alkali treated CG fibre + epoxy	2.7	2.7	0.75
3	Alkali treated CG fibre + chitosan + epoxy	2.4	1.8	0.66
4	Alkali treated CG Fibre + redmud + epoxy	2.0	1.6	0.55
5	Alkali treated CG fibre + rice husk + epoxy	1.9	1.5	0.47

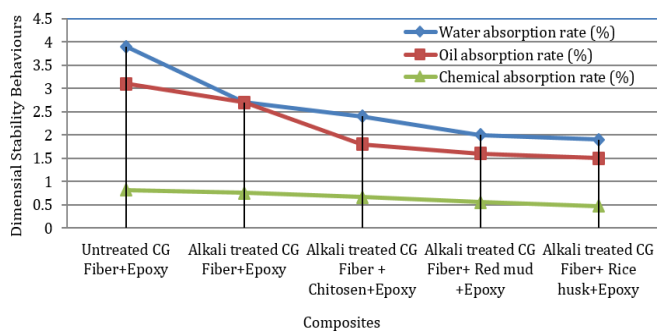


Fig. 7. Water, Oil, and Acid absorption behaviours of Particles-CG-Epoxy Composites

The same pattern was obtained in all the three dimensional stability characteristics of the fabricated composite material in the present investigation. The Dimensional steadiness influences in what weaned-composite-product will change and distort on its service and subsequently is a significant composite stuff to realize (Fig. 7).

5. Conclusion

The varieties of CG fibre reinforced epoxy composites have been developed and their mechanical and dimensional stability behaviours were studied. The better value of characteristics were obtained in rice husk impregnated CG fibre reinforced epoxy composites. It exhibited three point bending strength of 87.8 MPa, tensile strength of 72.3 MPa and impact strength of 63.4 kJ/m². It also exhibited less than 2% of absorption rate in water, oil and chemical environments. The rice husk filled the entire matrix system and acted as secondary reinforcement materials in epoxy composites. The particulates increased the mouldability of matrix and also enhanced strength behaviours. The use of nanoparticulates may be carried out to further improve the mechanical characteristics of CG fibre-epoxy composites.

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