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염기 처리 대마 섬유로 강화된 셀룰로오스 충전 에폭시 하이브리드 복합재의 기계적 물성

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Mechanical Properties of Cellulose-filled Epoxy Hybrid Composites Reinforced with Alkali-treated Hemp Fiber

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Abstract: There is a limit for deforestation in order to keep the environmental cycle undisturbed. The heart of the paper is to replace the wood to a maximum extent to obtain a sustainable environment. This research aims at new natural composites in which treated hemp fiber used as reinforcement, synthetic cellulose used as particulate to improve the adhesion between matrix - fiber interface and Epoxy LY556 acted as matrix fabricated by hand layup technique. The density, water absorption, tensile properties, impact strength, hardness, flexural properties and compressive properties have been evaluated under ASTM standards and compare the results with existing materials such as wood, aluminium, etc., The composite hemp fiber reinforced polymer (HFRP) could be exploited as an effective replacement for wood and it would be suitable for automotive applications by comparing results.

Keywords: biodegradable polymers, mechanical properties, treated hemp fiber, hybrid green composites.

Introduction

A fiber reinforced polymer (FRP) is a composite material comprising of a polymer matrix embedded with high strength fibers, such as glass, aramid and carbon.[1,2] Generally, polymer can be classified into two classes, thermoplastics and thermosetting. Thermoplastic materials currently dominate, as matrices for bio-fibers; the most commonly used thermoplastics for this purpose are polypropylene (PP), polyethylene, and poly(vinyl chloride) (PVC); while phenolic, epoxy and polyester resins are the most commonly used thermosetting matrices[3]. In the recent decades, natural fibers as an alternative reinforcement in polymer composites have attracted the attention of many researchers and scientists due to their advantages over conventional glass and carbon fibers.[4] These natural fibers include flax, hemp, jute, sisal, kenaf, coir, kapok, banana, henequen and many others.[5] The various advantages of natural fibers over manmade glass and carbon fibers are low cost, low density, comparable specific tensile properties, non-abrasive to the equipments, non-irritation to the skin, reduced energy consumption, less health risk, renewability, recyclability and biodegradability.[3] These composites materials are suitably applicable for aerospace, leisure, construction, sport, packaging and automotive industries, especially for the last mentioned application.[3,6] However, the certain drawback of natural fibers/polymers composites is the incompatibility between the hydrophilic natural fibers and the hydrophobic thermoplastic matrices. This leads to undesirable properties of the composites. It is therefore necessary to modify the fiber surface by employing chemical modifications to improve the adhesion between fiber and matrix.[3]

There are many factors that can influence the performance of natural fiber reinforced composites. Apart from the hydrophilic nature of fiber, the properties of the natural fiber reinforced composites can also be influenced by fiber content/

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amount of filler. In general, high fiber content is required to achieve high performance of the composites. Therefore, the effect of fiber content on the properties of natural fiber reinforced composites is particularly significance. It is often observed that the increase in fiber loading leads to an increase in tensile properties.[7,8] Many researchers in the past have developed composites using natural fibers such as bamboo,[9-11] coir,[12,13] sisal[14-17] and banana.[18-21] Hybrid composites are materials made by combining two or more different types of fibers in a common matrix and vice versa. Hybridization of two types of materials having different properties offers some advantages over the use of either of the material alone in a single matrix. Most of the studies are on the hybridization of natural fibers with glass fibers to improve the properties.[22-28]

The main parameters which affect the mechanical properties of the composites are fiber length, weight ratio, fiber orientation and interfacial adhesion between fiber and matrix.[29] The effect of fiber volume fraction in the strength properties of short fiber reinforced cement was studied by Karam[30] and he modified the existing model in order to calculate the strength of the composites.

The hybridization approach is used to make cost effective composites. The objective of this work was to predict the mechanical properties (strength and modulus) of treated hemp fiber reinforced manmade cellulose particulate hybrid epoxy composites by using experiments. Comparison of the experimental results with other materials was also done.

Experimental

Materials. Hemp fiber mats were purchased from Sheeba fibers and handicrafts, Poovancode, Tamilnadu, India whose properties are given in Table 1. The epoxy resin employed in the present study is LY556 and the hardener HY951 was purchased from the Modern Scientific Pvt Ltd, Chennai, Tamilnadu, India. LY556 resin is a bi-functional epoxy resin i.e., diglycidyl ether of biphenyl-A (DGEBA) and HY951 is an aliphatic primary amine, viz., triethylene tetramine - TETA with the mixing ratio is 10:1 w/w. Lyocell Powder (1.7 decitex) with around 12 μm was supplied by Lab Agencies, Navi Mumbai, India. Chemicals used for the surface modification of fiber are commercial sodium hydroxide which was kindly supplied by Lab agencies, Navi Mumbai, India. The properties of fiber, resin and cellulose powder used were given in Tables 1 and 2, respectively.

Treatment of Fiber. First the received hemp 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 hemp fibers.

Table 1. Properties of Untreated Hemp Fibers Used as Reinforcements[53]

Density (g/cm^3)	1.47
Elongation (%)	2-4
Tensile strength (MPa)	690
Elastic modulus (GPa)	70

Table 2. Properties of Epoxy Resin LY556[54]

Density (g/cm^3)	1.1-1.4
Elongation (%)	1-6
Elastic modulus (GPa)	3-6
Tensile strength (MPa)	35-100
Compressive strength (MPa)	100-200
Cure shrinkage (%)	1-2
Water absorption (%)	0.1-0.4
Izod impact strength (J/m)	0.3

Bleaching Treatment: For this treatment 25 g hemp 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 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.[31] Then the fibers are designated as bleached hemp fibers.

Chemical Treatments-Alkaline Treatment: Alkaline treatment or mercerization is one of the most used chemical treatments of natural fibers when used to reinforce thermoplastics and thermosets. The important modification done by alkaline treatment is the disruption of hydrogen bonding in the network structure, thereby increasing surface roughness. This treatment removes a certain amount of lignin, wax and oils covering the external surface of the fiber cell wall, depolymerises cellulose and exposes the short length crystallites.[32] Addition of aqueous sodium hydroxide (NaOH) to natural fiber promotes the ionization of the hydroxyl group to the alkoxide.[33] A water-ethanol solution (80:20) is prepared (6% of

NaOH) and stirred continuously for 1 h. Later, fiber mats were immersed one by one in the solution. Finally after immersing all the fiber mats the object is left undisturbed for nearly 3 h. Then the fiber mats are washed several times with distilled water followed by drying it at 80 ºC for 5 h in a hot air oven.[34-37]

Composite Fabrication. The composite material is fabricated by using hand layup technique.[38] Composite fabrication using double weave and non woven hemp mats (150 mm× 150 mm×1 mm) was carried out in a square mould of volume 350×350×3 mm.[3] Initially, the mould was polished and mould releasing agent was applied on its surface. Resin, hardener mixture and synthetic cellulose powder (10:1:4.3) are spilled for every layer. Figure 1 shows the treated hemp fiber mat used for the material preparation.

Initially the fibers are dried in sun light to remove the moisture. The mould surface is cleaned and releasing agent (wax) is applied. A thin layer of resin is also applied on the board. The woven roving natural fiber reinforced polymers (NFRP) are then completely filled with resin mixture, rolled to remove the entrapped air and to uniformly spread the mixture. In this way three layers of woven roving are placed one over the other to obtain top and bottom layers. A curing time of 24 h at room temperature is given for the structures to obtain good strength. After curing for 24 h, the required composite was obtained. By the same fabrication procedure, composites of different configurations by varying the ratio of cellulose powder and epoxy resin were fabricated. The same fabrication process was then carried out for fibers which were alkali treated. Thus, untreated and treated composites were prepared. Figure 2 shows the finished composite material.

All the tests were carried out as per ASTM standards at Japan Polymer Analytical and Research Laboratory India Private Limited, Chennai an ISO/IEC 17025:2005 - NABL Accredited Laboratory.

Tensile Test. The fabricated hybrid composite is cut using a saw cutter to get the dimension of the specimen for tensile testing as per ASTM: D638 standards.[39] The test was carried out using a universal testing machine at a room temperature with 40% relative humidity. The tensile stress is recorded with respect to increase in strain. Seven different types of specimens are prepared and tested. They are shown in the Figure 3. The specimen was placed in the grip of the tensile testing machine and the test is performed by applying tension until it undergoes fracture. The corresponding load and strain obtained are plotted on the graphs.

Compression Test. For compression test, the specimen was placed within the grip of compressometer. The strain gauge readings and the load cell readings were obtained, while the axial cross head movement rate imposed on the specimen was 5 mm/s. The test was carried out according to ASTM D 695[40] standards and size of the specimen is 70×19×3 mm^3. The

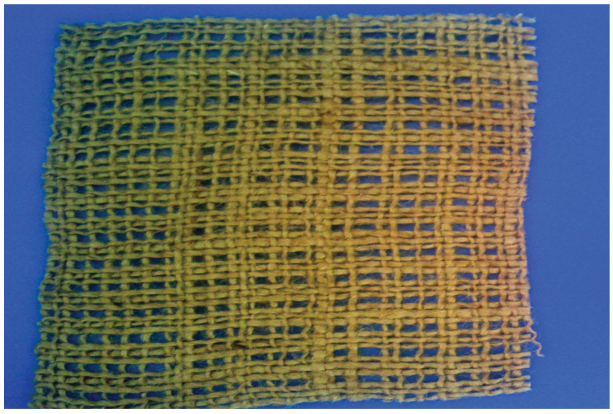

Figure 1. Treated hemp fiber mat.

Figure 2. Fabricated HFRP specimen.

Figure 3. Specimens for tensile test.

compression test was carried out at 28 °C with 40±2% relative humidity and three samples were tested for each test.

Flexural Test. The flexural test is performed on the same tensile testing machine as per the ASTM: D79041 standards. It is performed at room temperature and close to 40% relative humidity for seven different types of specimens as shown in Figure 4. In this test, the specimen to be tested is subjected to a load at its midway between the supports and until it fractures and breaks. This test determines the behaviour of the specimen when it is subjected to simple beam loading. Flexural test determines the maximum stress induced in the outer most fiber.

Impact Test. The impact tests were carried out using an Izod digital impact tester according to ASTM D 256[42] standards. The specimen 64×13×3 mm^3, notched; seven specimens were tested, to determine the impact resistance and impact strength of composites at room temperature.

Hardness Test. Shore A durometer helps us to determine the hardness of rubber materials and Shore D is used for composites. The hardness value is determined by the penetration of the durometer indenter foot into sample. The test will be conducted as per ASTM D 2240.[43] A measure of the indentation resistance of elastomeric or soft plastic materials based on the depth of penetration of a conical indentor. Hardness values range from 0 (for full penetration) to 100 (for no penetration). Full penetration is between 2.46 and 2.54 mm (0.097 and 0.100 in) depending on the equipment used. It is recommended that Durometer D tests be used when durometer A results greater than 90 and durometer A tests be used when Durometer D results are less than 20. Durometer A values less than 10 are inexact and are not reported.

Moisture Absorption Test. The specimens are cut as per the ASTM D 570[44] standards for moisture absorption test. The specimens are cleaned and weighed in an electronic balance of 0.00001 g accuracy to monitor the mass during the aging process. The specimen is 76×25×5 mm^3, six specimens were tested, and the average was calculated. Moisture absorption tests were conducted by immersing specimens in a distilled water bath at room temperature (28 °C) and boiling water at 60 and 100 °C. At room temperature and boiling temperature, the absorption was monitored at 24 h, 2 h and 30 min interval for five repeats. During the immersion process, the specimens are allowed to rest on one edge at the bottom and entirely immersed. For each observation at each stipulated interval, the specimens are wiped with a clean, dry cloth to remove the excess moisture immediately and then weighed. In the boiling water immersion process, after each immersion, the specimens are brought to room temperature by dipping in cold water for 10 min and then processed further as mentioned above. The moisture content $M(t)$, absorbed by each specimen is calculated as follows[45]:

$$M(t) = \frac{W_t - W_o}{W_o} \quad (1)$$

Where W_t & W_o – Wet & Dry weights of the specimen

Morphological Study (Scanning Electron Microscope-SEM). To illustrate the effect of alkali treatment of the fiber, the failure surfaces of the specimens subjected to test were analyzed using a JEOL scanning electron microscope (SEM). In SEM a fine probe of electrons scans the surface of the sample and the signals emanating from the incident site are processed and quantized. All specimens were sputtered with 10 nm layer of gold prior to SEM observations. Each specimen was mounted on the aluminium holder of the microscope using double sided electrical conduction carbon adhesive tabs. The accelerating voltage of 5-15 kV was employed. The SEM analyses of both raw and alkali treated fibers composites were compared.

Figure 4. Specimens for flexural test.

Results and Discussion

The seven specimens having different compositions with three samples each were tested in the various testing machine to find the mechanical properties (Table 3).

Tensile Properties. The Z HFRP (treated hemp fiber reinforced synthetic cellulose filled epoxy composites) specimen is found to withstand more loads (3016.4 N) with a displacement of 1 mm when compared to that of other specimen (Figure 5).

Table 3. Compositions of Different Specimens

S.No	Specimen	Hemp (%)	Cellulose powder (%) (1)	Epoxy resin (%) (2)	Hardener (%) (3)	Proportion 3:2:1
1	U HFRP	8	34.25	52.5	5.25	10:1:6.5
2	V HFRP	8	31.5	55	5.5	10:1:5.7
3	W HFRP	8	28.75	57.5	5.75	10:1:5
4	X HFRP	8	26	60	6	10:1:4.3
5	Y HFRP	8	23.25	62.5	6.25	10:1:3.7
6	Z HFRP	8	20.5	65	6.5	10:1:3.2
7	UX (Untreated) HFRP	8	26	60	6	10:1:4.3

The tensile strength of the composite is influenced by the strength and modulus of the fibers.[46] The Z HFRP specimen was capable to provide some resistance to continued elongation and most of the specimen displayed brittle failure at a maximum stress of 52.9 MPa. Further the true stress-strain of the composite materials were analyzed and the results are shown in Figure 6 which indicates clearly that the treated continuous fiber composite gives better strength than untreated continuous composites. Thus, tensile strength and tensile modulus of UX HFRP are 25% less than that of the Z HFRP specimen. In the case of the alkali treated fiber, the fiber pull-out length is considerably shorter than that noticed for the untreated fiber, due to the fiber failed by tearing. The deprived tensile strength exhibited by the untreated fiber reinforced epoxy composite specimen may have resulted from poor adhesion between fiber and matrix. The mechanical properties concern with the bonds existing between the different components of the fiber.[47] Alkali treatment causes removal of hemicelluloses and swelling of the fiber thus helps stress transfer between ultimate fiber cells.[48] In addition, the formation of hydrogen bonds of the cellulose chains improves chemical bonding between the fibers in composites. Thus the alkali treatment of hemp fibers and increase of epoxy resins leads to changes in mechanical properties like tensile strength, displacement, tensile load and tensile modulus.

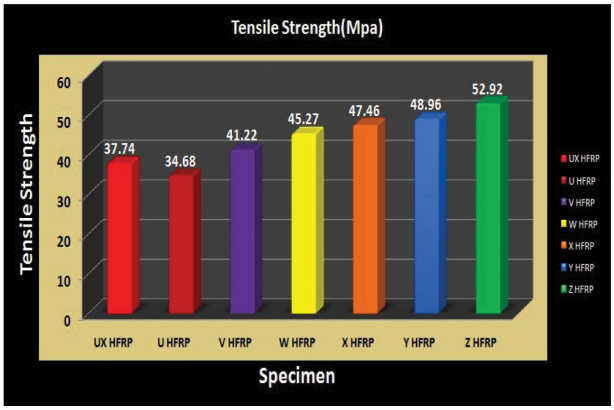

Figure 5. Tensile strength of treated and untreated HFRP.

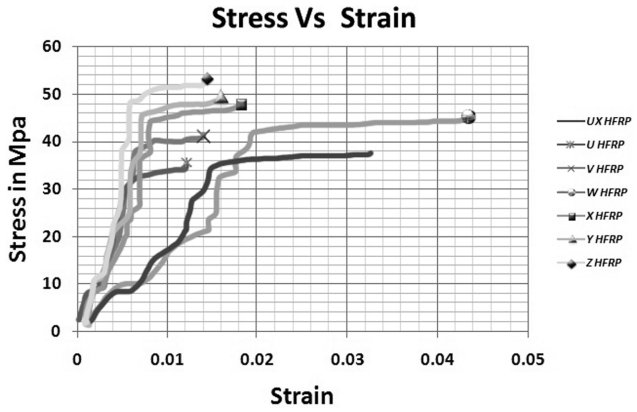

Figure 6. Stress strain analysis.

Compression Properties. The compression strength of the hemp fiber-epoxy composites are shown in Figure 7. The compression strength increases 20% because of the alkali treatment thus enhancing the mechanical interlocking. When the alkali treated fiber is used, a gradual increase in the compression

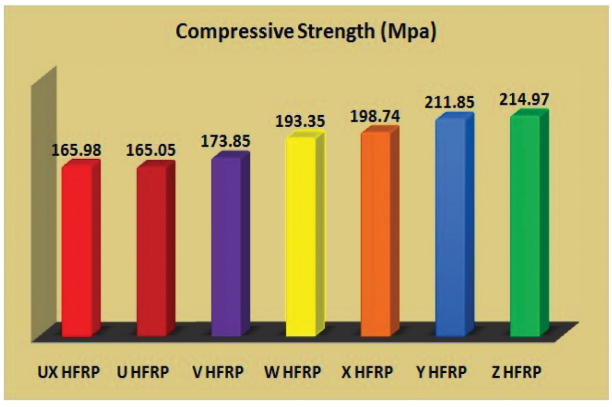

Figure 7. Compressive strength of treated and untreated HFRP.

force upon varying percentage of deflection was observed. The NaOH reacts with hydroxyl groups of the natural fiber hemicellulose, and it brings on the destruction of the cellular structure and the fibrillation increases effective surface area available for contact with the matrix.[49] Figure 7 shows an evaluation of the compression force of samples and the Z HFRP shows better result than other composites. This increment is attributed to chemical interaction between the fiber and the matrix. It is observed in this study that the compression moduli of Z HFRP samples are 214.9 MPa and UX HFRP samples are 165 MPa. It is well known that the fiber reinforcing effect is most efficient along the fiber axis orientation.

Flexural Properties. The flexural strength of the cellulose filled hemp fiber reinforced epoxy composites are shown in Fig. 8. It is observed that the flexural load increased from 476 to 547 N for UX HFRP to Z HFRP composites. Flexural strength increased in the Z HFRP samples from 10% to 15% and it is an interesting note that the alkali treated fiber showed more shrinkage and toughness that inbuilt the chemical strength between the fiber and the matrix. The Z HFRP sample was considered to have 19% higher flexural strength than the UX HFRP sample. The fiber surface alkali treatments had a significant effect on the flexural modulus; similar to the observations made for the tensile properties.[50] The enhanced interfacial area of contact was favourable for the flexural strength.[51] The flexural modulus is used as an indication of the material's stiffness in static bending condition. The flexural modulus showed a similar behaviour as the tensile modulus and the values of stiffness measured were 12.8 GPa for UX HFRP and 15.3 GPa Z HFRP in the longitudinal direction. This indicates a better fiber/matrix contact, the increase in areas of contact between the fiber and the matrix improves the adhesion by incorporation of mechanical components of adhesion for the fiber-cellulose-epoxy interfacial strength.

Impact Properties. The influence of surface modification on the impact strength of composites is shown in Figure 9. The impact strength of the Z HFRP sample improved to 25-30% than that of UX HFRP samples. The shrinkage of the fibers during alkali treatment affects the impact strength of the composites and it helps to improve the interfacial strength between fiber and matrix. The impact resistance of Z HFRP and UX HFRP composites is 0.85 and 0.64 KJ/m, respectively. There was significant difference in the impact strength of the composites and the alkali treated fiber reinforced composites showed slightly superior values. The improvement in the impact strength of Z HFRP samples is related to the chemical effect of the fiber-cellulose, epoxy interface. The alkali treated fiber composites showed maximum improvement in impact strength compared with untreated fiber composites.[49] A strong interface has an effect on the impact strength as energy absorption is from the fiber-matrix debonding and fiber pullout. The impact failure of hemp fiber reinforced cellulose filled, epoxy composites reflects a process involving crack initiation and growth in the resin matrix. It is well known that the impact response of fiber composites is highly influenced by the interfacial bond strength, the matrix and the fiber properties.

Hardness Test. As known, hardness implies a resistance to indentation, permanent or plastic deformation of material. In a HFRP composite material, filler weight fraction significantly affects the hardness value of the composite material. Hardness values measured on the Shore D[47] scale showing the effect of weight percentage of cellulose powder, epoxy and hemp fibers on the hardness values of HFRP composites are presented in Figure 10. It is found that hardness of the neat epoxy resin is 57.8. The hardness of the fabricated composite made of epoxy

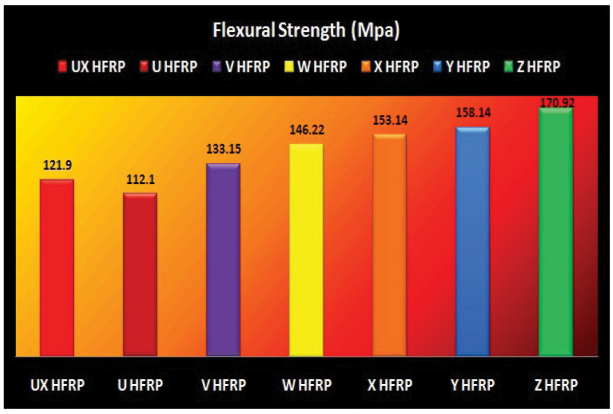

Figure 8. Flexural strength of treated and untreated HFRP.

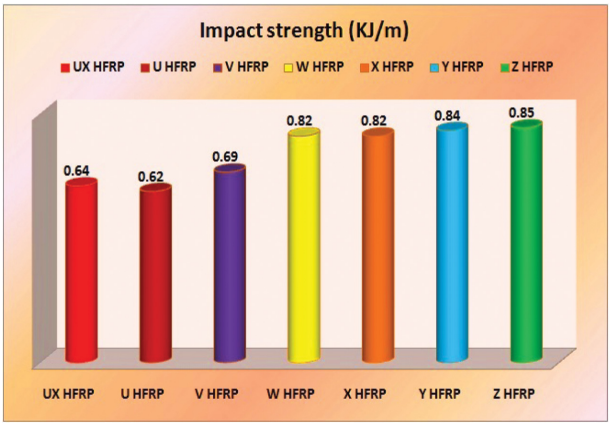

Figure 9. Impact strength of treated and untreated HFRP.

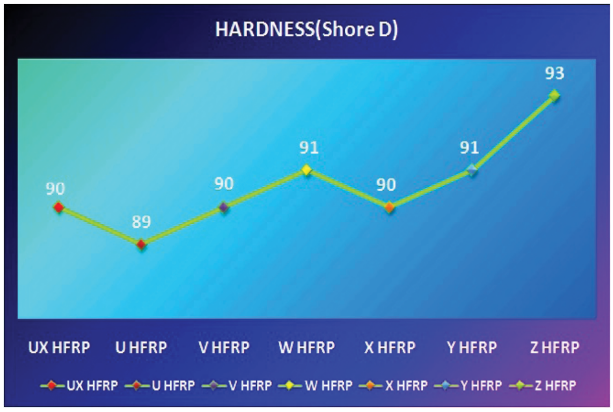

Figure 10. Hardness of treated and untreated HFRP.

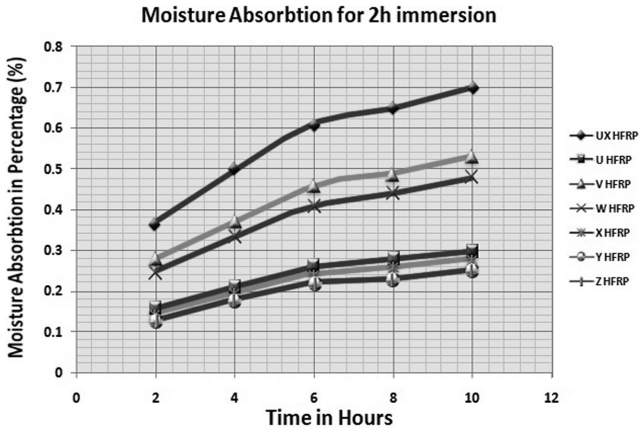

Figure 12. Moisture absorption for 2 h immersion.

resin and untreated UX HFRP is 90 and maximum of Z HFRP is 93. The hardness increases with the presence of epoxy with the treated hemp fibers reflecting the reinforcement formed in the hybrid composite.

Moisture Absorption Properties. The percentage of moisture absorption of the hemp fiber reinforced epoxy composites was evaluated as the difference between the dry weight and wet weight of the specimen. Figure 11 shows the percentage of weight gain as a function of time for hemp fiber reinforced cellulose filled epoxy composites samples (UX HFRP to Z HFRP) immersed in distilled water at room temperature and boiled water at 30 min interval. It can be inferred that water absorption is more at boiling temperature than at room temperature. The results of moisture absorption of the samples in boiling water (100 °C) and room temperature at 2 h duration are shown in Figure 12. When the temperature of immersion is increased, the moisture saturation time is significantly shortened. This shows that water absorption at room temperature takes fairly long period to reach equilibrium than absorption at boiling temperature. When weight gain is less, water molecules interlocked in the composites is also less. Thus the water molecules can't get chances to actively attack the interface, resulting in bonding of the fiber and the matrix internally is good in the composite.

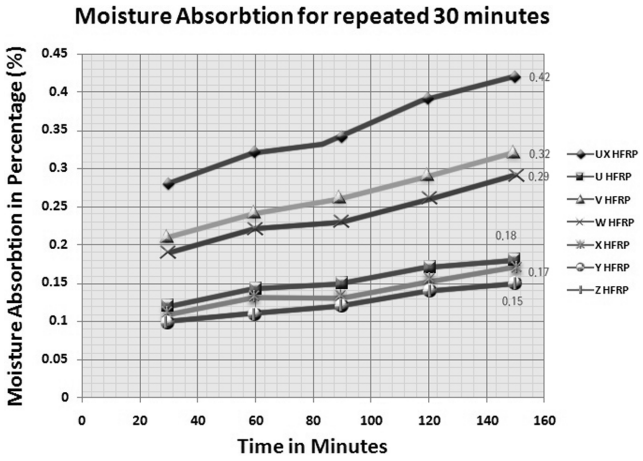

Figure 11. Moisture absorption for repeated 30 min.

Morphological Analysis. Morphological analysis was done using SEM. The surface characteristics of the composite material were studied through SEM after conducting tests. Subsequently the specimens were inspected by a scanning electron microscope. The interfacial adhesion between matrix and the fiber is clearly seen from scanning electron micrographs.

The SEM micrograph of the UX HFRP composite is shown in Figure 13. Even though the manufacturing of the composite was done with care, it is seen that there is intra fiber delamination predominantly present in the hemp fibers which reduces the strength of the composite. The top portion of the image shows the hemp fibers in vertical direction and the centre part shows the fiber in horizontal direction. Since the loading for tensile test is done in horizontal direction, the fibers are found to be damaged in that direction more than the other direction. Figure 14 shows the point of fracture of hemp fibers in the X HFRP composite subjected to impact testing. The fibers are fractured due to the sudden impact and no trace of fatigue failure is observed. Moreover due to the woven nature of the fiber, it is clear that there is uniform distribution in the matrix and interfacial adhesion is also present to a decent level.[38]

Figure 15 shows the adhesion of fiber and resin in Y HFRP composite which is subjected to tensile testing. In general, the adhesion is good although there are a few defects like air bub-

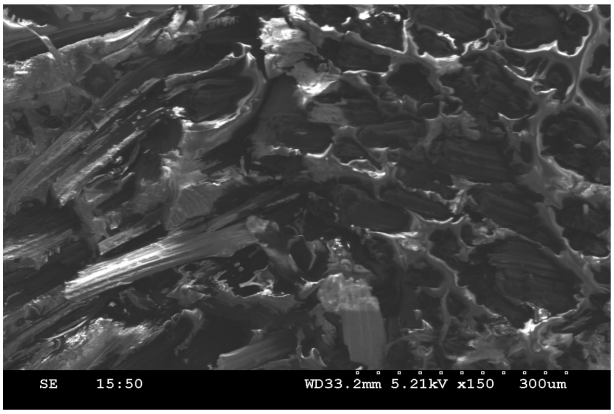

Figure 13. SEM photograph of UX HFRP.

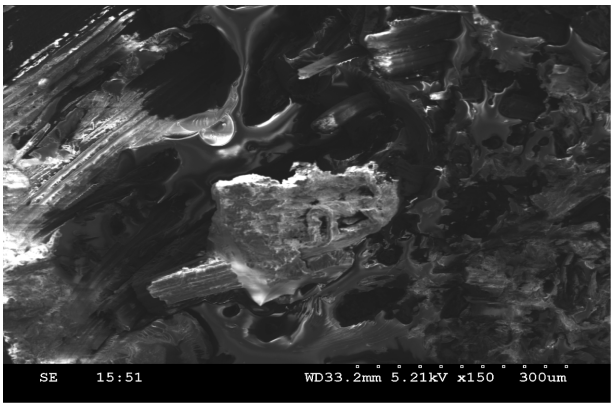

Figure 14. SEM photograph of X HFRP.

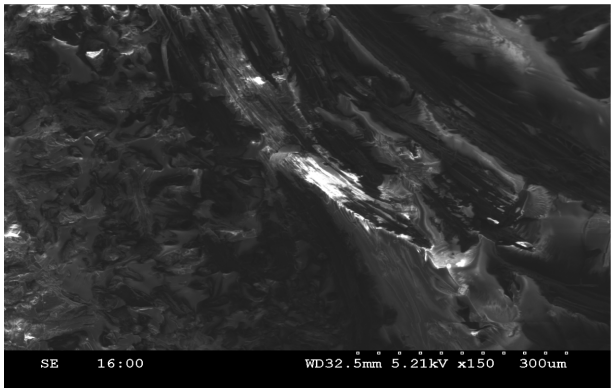

Figure 15. SEM photograph of Y HFRP.

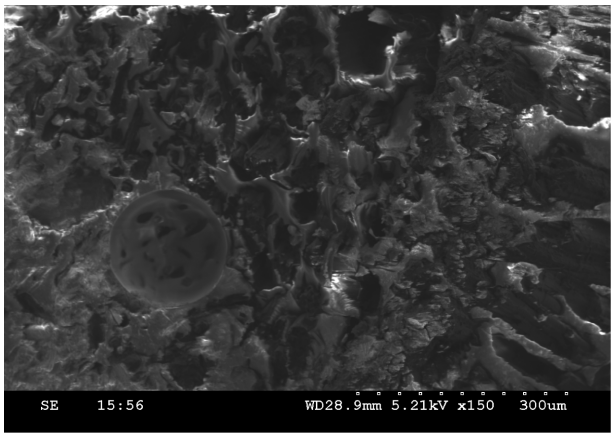

Figure 16. SEM photograph of Z HFRP.

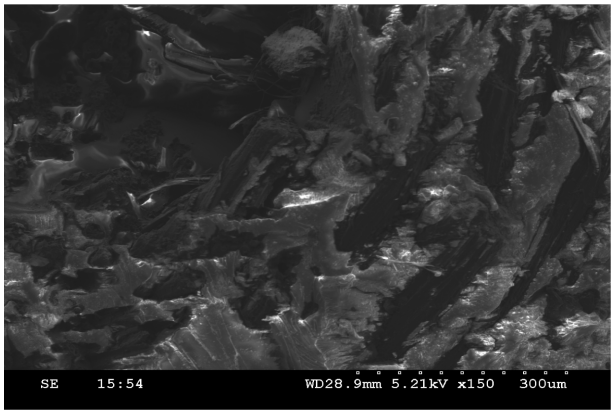

Figure 17. SEM photograph of Z HFRP.

bles and fiber draw-out. The smooth surface seen is the resin and the irregular surface is fiber. Due to the high strength of hemp fibers, they have undergone individual breakage, giving it very high strength. The effective stress transfer in the tensile direction between the fiber and matrix is supported by the high stress values obtained in the test.

Figure 16 shows the SEM micrograph of a flexural fractured Z HFRP specimen. Interphase delamination is found at the cross-section of applied load. Presence of voids in the specimen is found to be minimal due to uniform load applied on it. The crack propagates through the natural fibers rather than the glass fiber and causes failure. Flexural strength values also indicate that there is very little stress transfer from the matrix to the fiber and hence very low values.

Figure 17 shows the Z HFRP composite specimen subjected to tensile test. The fibers are found to be intact which suggests there is good wetting of the fiber by the epoxy matrix. Adequate interfacial adhesion ensures good toughening. In its absence, there is little contribution from fiber peeling and breakage and the measured value is attributed to the toughness of the matrix alone over a reduced section caused by the presence of the non-adhering fibers. The high degree of adhesion allows a full contribution from the matrix and extra contributions from fiber.

Conclusions

The scientific world is facing a serious problem of developing new and advanced technologies and methods to treat solid wastes, particularly non-naturally-reversible polymers. The processes to decompose those wastes are actually not cost-effective and will subsequently produce harmful chemicals. Owing to the above ground, reinforcing polymers with natural fibers is the way to go. In this investigation, the effect of treated hemp fiber reinforced synthetic cellulose filled epoxy composites on the mechanical properties, water absorption property and morphological behaviours was studied. Surface treatments resulted in the removal of pectin, hemicelluloses, and other non-cellulosic substances from the fibers and the higher percentage of celluloses in the final treated fibers. Rougher surface and increased effective surface area of the chemically treated fibers facilitated better interaction between the fiber and matrix. Surface modified fibers showed better tensile properties compared to untreated fibers for the presence of higher percentage of crystalline celluloses.

Based on the investigations, Z HFRP composite showed optimum mechanical strength. The mechanical findings were corroborated with morphological evidence. Based on these studies, it can be concluded that treated hemp fibers could effectively reinforced in cellulose filled epoxy matrix when used in optimal concentration of fibers and coupling agents. Figure 18-20 show the comparison of HFRP with other natural composite materials.[33,38,51-56] Thus mechanical properties of treated hemp fiber reinforced synthetic cellulose filled epoxy composites shows excellent figures than other conventional fiber reinforced plastic materials which may extend the applicability in automotive and building products industries.

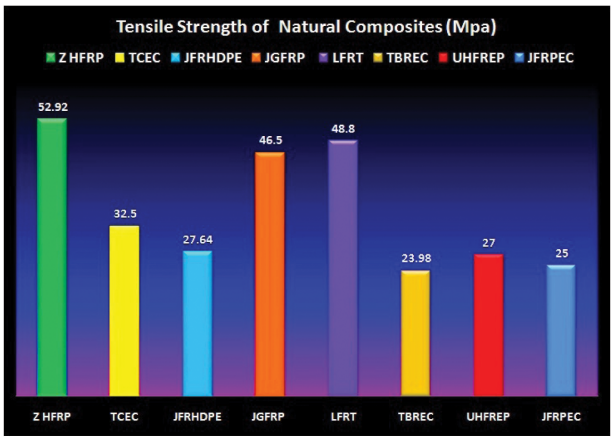

Figure 18. Tensile Strength comparison of NFRP.

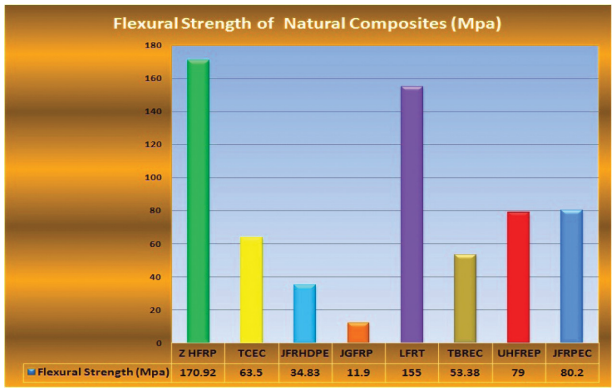

Figure 19. Flexural strength comparison of NFRP.

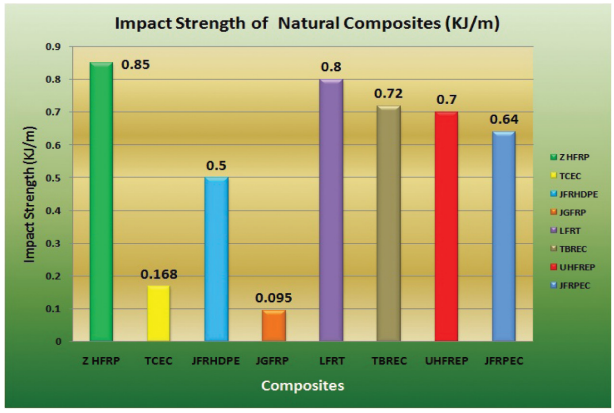

Figure 20. Impact strength comparison of NFRP.

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