



Novel Hybrid Structural Biocomposites from Alkali Treated-Date Palm and Coir Fibers: Morphology, Thermal and Mechanical Properties

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Abstract

In this concern study, the surging demands for regulating the environmental aspects associated with controlling a massive generated synthetic wastes in the field of structural applications, has motivated researchers to synthesize green products using agricultural wastes. Therefore, in this work, a novel hybrid epoxy composites based on alkali treated-date palm fiber and native coir fiber at different weight fractions were prepared using hand layup technique and investigated the hybridization effect of the resultant composites. Hybrid composites were characterized using several analytical techniques i.e. X-ray diffraction analysis, thermogravimetric analysis, contact angle measurement, dart impact test and tensile test. Reported results explored that the alkali treatment on date palm fiber had a reliable impact on the experimentally evaluated mechanical, hydrophobic and thermal properties of the resultant hybrid composites. The required structural properties for 50 wt% coir fiber incorporated in 50 wt% treated-date palm fiber based epoxy composites are 76.51 MPa tensile strength, 2.77% elongation limit, 832.2 J/m impact strength, and 100.2° contact angle, respectively possess highest value as compared to all other hybrid composites. Hence, the incorporation of 50 wt% treated-date palm fiber in 50 wt% coir fiber based epoxy composite has the best structural properties in terms of mechanical, water resistant and thermal properties for green rigid applications.

Keywords Composites · Agricultural wastes · Epoxy · Hand layup

Introduction

The extensive concerns to regulate the environmental aspects and controlled the pollution created by non-biodegradable materials have triggered the scientists to fabricate more convincing biodegradable novel hybrid green materials in order to meet the growing demands in various ranges of sectors. Therefore, ecological aspects have motivated researchers to synthesize hybrid biodegradable green composites products having synergetic characteristics i.e. biodegradable in nature, low-cost, easy to fabricate, light-weight, good as a thermal insulator and having high hygroscopic for various industrial sectors like automotive and structural applications [1]. So, this is the eye opener for the industrialist to fabricate agro-waste fiber based hybrid structural composites due to their providing high mechanical stability with remarkable stiffness that is comparable with synthetic

structural composites. Hybrid composites, as an imperative class in the field of composites, represent those composites in which one kind of reinforcing material is blended in the combination of different matrix [2, 3]. The idea of hybrid composites with impressive combination of characteristics i.e. high specific properties, low density and excellent thermal stability is fulfilling the requirements of medium density fiberboards, insulation boards and other automotive applications while understanding the needs of cost reduction and waste minimization. Therefore, the perfect use of plenty available natural fibers like a banana [4], cotton [5], coir [6], sisal [7], date palm [8] and jute [9, 10] for structural applications is the need of the hour.

Agricultural wastes like date palm, coir etc. fibers are mainly composed of cellulose (33–45%), hemicellulose (59.5–74.8%) and lignin (14–27%) and cotton coir is composed of cellulose (36–43%), hemicellulose (0.15–0.25%) and lignin (41–45%) [11, 12]. Due to the recalcitrance nature of biomass, the existence of undesirable materials (hemicellulose and lignin) cause weak compatibility found between natural fibers and polymer matrix [13]. Many authors have adopted reliable pre-treatment techniques in order to

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improve the suitability of biomass for blending in polymer matrix. Many researchers have used chemical pre-treatment for making biomass more promising for polymer adhesion. The main purpose of using chemical pre-treatment is to destruct the biomass body and create some voids opening at the surface. Generally, in alkali treatment, a significant delignification process followed by the removal of a small branched chain of carbohydrate that is hemicellulose from natural fiber [14–16]. It has been found that the alkali treatments enhanced the suitability of agricultural wastes for polymer adhesion [17].

Mochane et al. reviewed the various natural fibers such as jute, flax, sisal, banana, hemp based polymer composites for structural applications [18]. Otto et al. synthesized chemically modified based lignocellulosic fibers reinforced polyurethane composites and observed benchmark elastic property showing higher potential applications for padding material and shock absorption [19]. Sanjay et al. studied mechanical and physical characteristics of natural fiber incorporated glass fiber based polymer composites using hand layup technique for engineering applications [20]. Alavudeen et al. synthesized chemically modified banana and kenaf fibers reinforced polyester composites using hand layup technique and studied the mechanical stability of resultant composites for structural applications [21]. The authors investigated the mechanical behavior of unidirectional flax/synthetic fibers hybrid composites [22]. AL-Oqla et al. confirmed the reliable use of date palm fiber based polymer composites for automotive applications on the basis of mechanical stability [23]. In this study, the authors fabricated a new composite material based on sisal-jute fiber and demonstrated the perfect replacement of synthetic glass fiber reinforced polymer composites with bio composites [24]. Sanjay et al. [16] reviewed various techniques for improving suitability of natural fibers in polymer composites. Asim et al. [25] compared the mechanical properties of the silane modified pine leaf fiber/phenolic composites with silane treated-kenaf fiber/phenolic composites for green applications. The author of this study investigated the dynamic mechanical properties of the bamboo/woven kenaf fiber based composites for building applications [26]. Ozben et al. [27] studied drop weight technique for analyzing the impact behavior of hybrid composites for structural applications. This study demonstrated the impact of date palm fiber at different loading in epoxy composites on the basis of mechanical and thermal properties for engineering applications [28]. Safri et al. [29] reviewed the impact behavior for various hybrid composites synthesized from natural fibers for structural applications. Muhammad et al. [30] demonstrated the effect of kenaf fiber in glass fiber based composites and found highest impact strength in the resultant hybrid composites. However, till today no research work has been published on the combination of alkali treated natural fiber with native fiber in epoxy

matrix in order to understand their hybridization effects for the structural properties of polymer composite.

In this research work, different combinations of natural fibers i.e. alkali treated-date palm fiber and native coir fiber based epoxy novel composites were prepared using hand layup method. Further, authors of this paper have demonstrated the characteristics of all synthesized hybrid composites in terms of Physico-chemical stability using various kinds of analytical techniques like X-ray diffraction, Thermogravimetric analysis, tensile test, impact test and Contact angle measurement for green reinforcement applications.

Materials and Methods

Materials

The coir and jute fibers are procured from a local farmer near BHU, Varanasi, Uttar Pradesh. Fibers are washed with distilled water and kept in an oven at a temperature of 70 °C for 8 h. Further, fibers are ground in a powder form. The epoxy resin (LY 556) and the hardener HY-951 are acquired from Green products Pvt. Ltd. Epoxy LY556 of density 1.15–1.20 g/cm³, mixed with hardener HY951 of density 0.97–0.99 g/cm³ was used to synthesize the composite laminate. The epoxy and hardener were mixed in the ratio 2:1 on a weight basis. Sodium hydroxide pellets were purchased from Green products Pvt. Ltd. It is highly soluble in water and was used for natural fiber surface pretreatment to enhance bonding between fibers and polymer.

Alkali Pretreatment

The external sheath of lignin and to some extent hemicelluloses restricts the cellulose to make the effective interfacial bond with the polymers. Thus to overcome poor adhesion, several pretreatment methods are to be incorporated to improve the bonding between the hydrophilic fiber and hydrophobic matrix [31]. In order to get improved mechanical and thermal characteristics of the composites, date palm fiber was subjected to the NaOH treatment process. Alkali treatment provides porous structure of fiber that is increase the interfacial bonding between treated and non-treated fibers. A 5 g weight of sieved agricultural waste was treated with 9.1% (W/V) NaOH aqueous solution for 24 h at room temperature followed by drying in an oven at 70 °C for 12 h [16, 32].

Synthesis of Green Composites

Initially, a mold of dimension 220 mm × 220 mm × 3 mm was cleaned and dried before the fabrication process. The releasing wax was also applied to mold releasing sheet and

Table 1 Compositional analysis for synthesized green composites

Composites	Chemical pretreatment	Date palm fiber (wt%)	Coir fiber (wt%)
E1	–	–	100
E2	Date palm fiber treated with NaOH	50	50
E3	Date palm fiber treated with NaOH	25	75
E4	Date palm fiber treated with NaOH	75	25
E5	Date palm fiber treated with NaOH	100	0
E6	–	50	50
E7	–	100	0
E8	–	25	75
E9	–	75	25

fibers were mixed with the resin inside the mold using hand layup technique. Further, the mixture was allowed to set inside the mold for a period of 24 h at room temperature. In this work, different combinations of treated-date palm/coir/epoxy are prepared which are shown in Table 1. The specimens were finally cut into the appropriate dimensions for characterization.

Characterizations for Treated-Date Palm/Coir Based Epoxy Composites

XRD analysis was used to analyze the crystalline changes after incorporation of natural fibers in epoxy polymer using X-ray diffractometer (model Minifux II, Rigaku). The crystallinity index (CI) of the composite samples was calculated by using the formula:

$$CI = \frac{I_{max} - I_{am}}{I_{max}} \times 100 \quad (1)$$

where I_{max} is the maximum intensity of diffraction on the crystallographic plane at a 2θ angle and I_{am} is the intensity of diffraction of the amorphous material taken at a 2θ angle from where the peak originates.

In order to visualize the thermal stability for all green composites, TGA analysis is performed from 30 to 800 °C temperature at a scan rate of 10 °C/min in an inert atmosphere using Perkin Elmer instrument, USA and weight changes is recorded as a function of temperature. The sessile drop method (KRUSS DSA25 Series, Germany) was used to check water repelling characteristics for all green composites. The tensile test of the composites was conducted in INSTRON 1195 testing machine as per the ASTM D3039 standards. This property is determined in accordance with the ASTM D3039. The specimen was cut in the required dimension 50 mm × 20 mm × 3 mm. Further, the test was conducted at a constant strain rate of 2 mm/min and

demonstrated the mechanical stability of the material. Dart impact strength for all hybrid composites was analyzed using Dart impact tester (Asian Test Equipments, Hapur, India). In this test, a dart was free fell on the surface (220 mm × 220 mm × 3 mm) with 13.2 m/s striking velocity and calculated impact strength using following equation.

$$\text{Impact energy}(KE) = \frac{1}{2}mv^2 \quad (2)$$

$$\text{Impact Force} = \frac{KE}{d} \quad (3)$$

where d (m) is the thickness of hybrid composite and m (g) is dart impact failure weight.

Results and Discussions

XRD Analyses for Hybrid Composites

XRD analysis was used to estimate crystalline changes in the hybrid composites. In this test, XRD analysis for E1, E2, E3, E4, E5, E6, E7, E8 and E9 was conducted and shown in Fig. 1. There is one major peak found in XRD spectra of all composites which are 20.68° for E1, 20.52° for E2, 20.74° for E3, 20.46° for E4, 20.86° for E5, 20.07° for E6, 20.19° for E7, 19.83° for E8 and 19.59° for E9, respectively. The higher intensity peak observes in E2 hybrid composite at 20.52° 2 theta. The crystallinity index value is 84.48% for E1, 88.37% for E2, 84.32% for E3, 85.92% for E4, 85.74% for E5, 85.61% for E6, 85.82% for E7, 84.61% for E8 and 84.76% for E9, respectively.

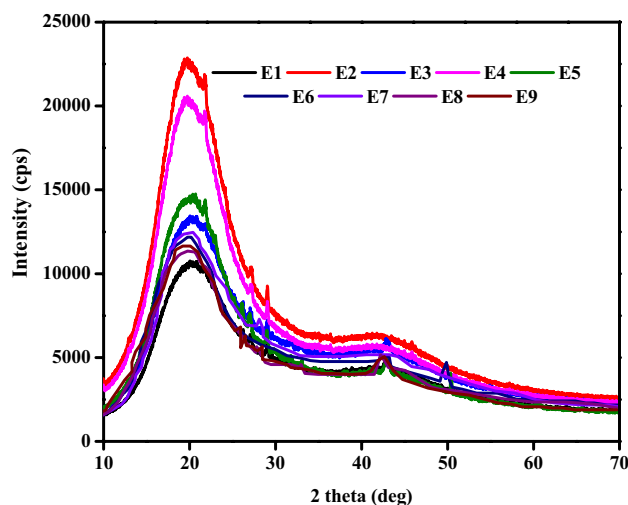


Fig. 1 XRD analyses for E1, E2, E3, E4, E5, E6, E7, E8, and E9 hybrid composites

Some shifting of 2 theta peaks observes due to biomass addition in epoxy matrix. The presence of effective peak at around $21^{\circ} 2$ theta in XRD spectra attributes to the crystalline structure of cellulose that is present in the hybrid composite. The higher intensity peak observes in the E2 hybrid composite shows better compatibility of fibers with epoxy matrix assuring remarkable mechanical and thermal stabilities which confirms in mechanical and thermal tests. A similar result that is higher intensity peak concept in composites is also explained by Laadila et al. [33] in their published article.

Thus it can be concluded that 50% treated date palm fiber/50% coir fiber based epoxy composite has a higher crystalline stable structure as compared to other hybrid composites showing its suitability for green applications.

TGA Analyses for Hybrid Composites

Thermal stability analyses for coir fiber incorporated treated-date palm fiber/epoxy composites have been studied using TGA analysis. In this analysis, thermal stability tests for E1, E2, E3, E4, E5, E6, E7, E8 and E9 were performed in a programmed temperature range of 30–800 °C (Fig. 2). The primary, main and final degradation temperature ranges for hybrid composites have been determined in this analysis. Small degradation is mainly observed in E1, E3, E6, E7, E8 and E9 from 100 to 250 °C temperature range. The main degradation starts from 250 °C around at 14% weight loss for E1, 5.13% weight loss for E2, 4.71% weight loss for E3, 4.86% weight loss for E4, 4.87% weight loss for E5, 7.31% weight loss for E6, 5.51% weight loss for E7, 8.41% weight loss for E8 and 9.97% weight loss for E9, respectively. In addition, degradation ends at around 522 °C with 92%

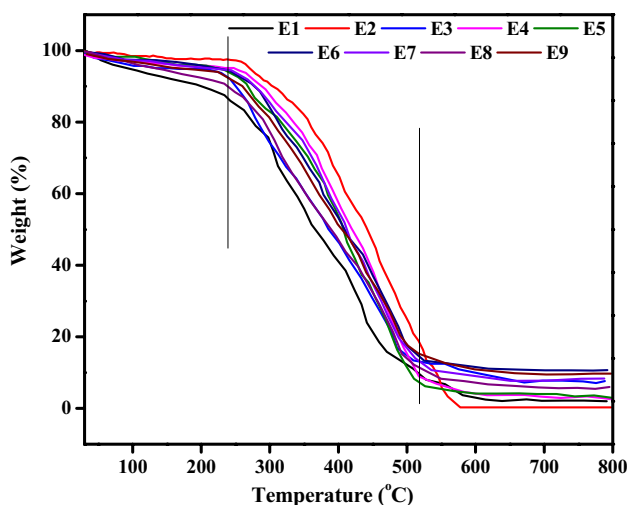


Fig. 2 TGA analyses for E1, E2, E3, E4, E5, E6, E7, E8, and E9 hybrid composites

weight loss for E1, 560 °C with 95% weight loss for E2, 511 °C with 86% weight loss for E3, 517 °C with 93% weight loss for E4, 526 °C with 94% weight loss for E5, 515 °C with 86.44% weight loss for E6, 518 °C with 87.81% weight loss for E7, 519 °C with 89.29% weight loss for E8, 521 °C with 89.79% weight loss for E9, respectively.

The earlier degradation is observed in E1, E3, E6, E7, E8 and E9 composites. This may be lot of lesser amounts of some impurities like hemicellulose, pectin and wax present in treated-date palm fiber. Gheith et al. [28] synthesized 50% date palm fiber based epoxy composites and observed similar initial degradation corresponds to water molecules and impurities. This characteristic associated with poor interfacial bonding between the fiber and polymer ascribe to less mechanical stability of natural fiber based composites. Nair et al. [34] also observed similar result sisal fiber based polystyrene composites for reinforcement applications. Puglia et al. [35] demonstrated the effect of alkali and silane treatments on phormium tenax fiber and observed initial degradation in native fiber based composites due to wax and pectin present in the material. In addition, it shows lower thermal stability than the higher cellulose containing composites. The main degradation starts from 250 to 550 °C due to the degradation of cellulose and lignin in the composites. The thermal stability of E2 seems higher as compared to all other composites. This is the main effect of hybrid composites synthesized from equal amounts of native coir fiber and treated-date palm fiber based epoxy composites. Gheith et al. [28] prepared date palm fiber at different weight fractions based epoxy composites and found highest thermal stability in 50% date palm fiber based epoxy composites. Chee et al. [26] prepared 50% bamboo fiber incorporated 50% kenaf fiber based epoxy hybrid composites and observed highest dimensional stability of the resultant composite. Boopalan et al. [36] used equal percentages of jute and banana fibers in epoxy matrix and observed higher thermal stability as compared to other hybrid composites. This behavior reveals the strong adhesion present in treated fiber incorporated native fiber based epoxy composites and assuring E2 composite suitability for structural applications.

Wettability of Hybrid Composites

The wettability of hybrid composites was evaluated using the sessile drop method as shown in Fig. 5. The sessile drop method is based on the surface tension between liquid-solid interfaces. In this analysis, the sessile drop is permitted to fall on the surface of hybrid composites and observe the wettability of the surface with time. Higher contact angle (greater than 90°) represents the less wettability of the surface and confirms the hydrophobic characteristics of the synthesized hybrid composites. Generally, fibers are hydrophilic in nature and have higher wettability characteristics. Authors

of this paper have prepared different combinations of treated and untreated fibers in epoxy matrix and observed its contact angle in order to attain the higher hydrophobic quality among all synthesized hybrid composites. The contact angle value is measured progressively with time as shown in Fig. 3. The averaged contact angle value is 29.09° for E1, 101.78° for E2, 54.50° for E3, 73.56° for E4, 61.88° for E5, 42.16° for E6, 38.99° for E7, 33.07° for E8, and 40.43° for E9, respectively. It is clear from the graph that the contact angle decreases as a function of time. So, the length of the interface between liquid drop and epoxy hybrid composites increases as time elapsed. This surface characteristic explores mainly due to liquid spreading over the hybrid composite surface. The sessile drop volume decreases due to liquid penetrate into the surface of date palm/coir fiber based epoxy composites. Zhang et al. [37] explained the

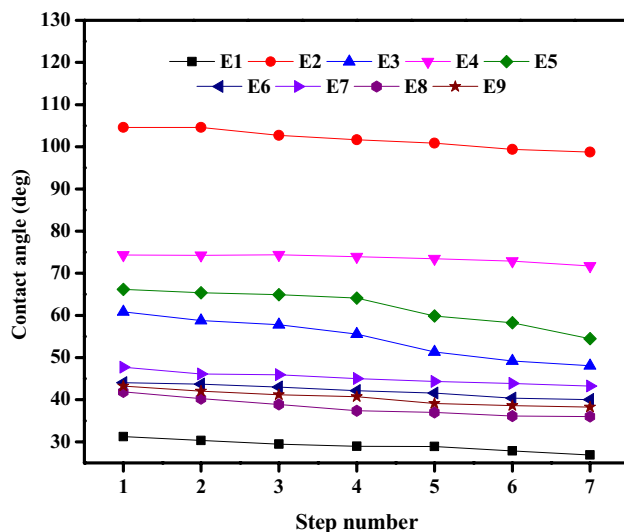


Fig. 3 Wettability analyses for E1, E2, E3, E4, E5, E6, E7, E8, and E9 hybrid composites

wettability behavior of rice straw/coir fiber based composite and observed similar results in their published article.

The contact angle value is higher for E2 hybrid composite. This may be due to strong adhesion present between treated-date palm fiber and untreated-coir fiber. Generally, contact angle greater than 90° explores between the sessile drop and hybrid composite surface is very weak. This characteristic is enabling E2 composite for hydrophobic structural applications.

Tensile Test for Hybrid Composites

The mechanical stability test is playing a pivotal role in visualizing the ability of hybrid composites to withstand force. The tensile test for E1, E2, E3, E4, E5, E6, E7, E8, E9 and E10 hybrid composites were performed in this analysis (Fig. 4). Tensile strength, elongation limit and young modulus have been summarized in Table 2. The mechanical stability of hybrid composites depends on fiber-polymer interaction, fiber percentage and distribution of fiber in the matrix. Stress represents the maximum stability of composites to withstand force and strain represents the change in length of material with respect to original length of the material. The stress vs. strain curve is depicted in Fig. 5c. The tensile strength is 54.03 MPa for E1, 76.51 MPa for E2, 55.65 MPa for E3, 61.04 MPa for E4, 59.89 MPa for E5, 49.02 MPa for E6, 50.01 MPa for E7, 45.85 MPa for E8, 46.75 MPa for E9 and 48.84 MPa for E10, respectively. The tensile strength is higher for E2 (50% treated date palm fiber/50% untreated coir fiber/epoxy composite) hybrid composites due to strong interfacial bonding present between fibers and polymer. Further addition of fiber causes a decrement in the mechanical stability of the resultant hybrid composites. Boopaln et al. [36] explained similar results in jute/banana fiber based epoxy composites in their published literature. Asim et al. [25] compared the mechanical property of treated-fibers based composites and observed highest tensile strength

Table 2 Dart impact velocity, dart impact height, dart impact strength, dart impact failure weight, tensile strength, elongation at break (%), tensile modulus for E1, E2, E3, E4, E5, and E6 hybrid composites

Hybrid composite	Dart Impact velocity before strike (m/s)	Dart impact height (m)	Dart impact failure weight (g)	Dart impact strength (J/m)	Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (MPa)
E1	3.63	0.66	310	709.56	45.85	1.34	3421
E2	3.63	0.66	380	832.2	76.51	2.77	2762
E3	3.63	0.66	335	733.65	55.65	2.53	2199
E4	3.63	0.66	360	788.4	61.04	2.61	2388
E5	3.63	0.66	350	766.5	59.89	2.34	2599
E6	3.63	0.66	325	711.75	49.02	0.75	6535
E7	3.63	0.66	330	722.70	50.01	0.49	10206
E8	3.63	0.66	315	689.85	46.75	0.62	7540
E9	3.63	0.66	320	700.8	48.84	0.55	8880

(≈ 46 MPa) in 50% kenaf fiber based phenolic composites for engineering applications. Chee et al. [26] also observed highest mechanical stability in 50% bamboo/50% kenaf fibers based epoxy composites.

The elongation limit is 2.25% for E1, 2.77% for E2, 2.53% for E3, 2.61% for E4, 2.34% for E5, 0.75% for E6, 0.49% for E7, 1.34% for E8, 0.62% for E9 and 0.55% for E10, respectively. The higher percentage of flexible limit is observed in the case of E2 hybrid composite due to strong adhesion present between the fibers and polymer. Authors also observed similar result in 50% pineapple leaf based composites for green applications [25].

The tensile modulus is the ratio of stress and strain. The tensile modulus is 2401 MPa for E1, 2762 MPa for E2, 2199 MPa for E3, 2388 MPa for E4, 2599 MPa for E5, 6235 MPa for E6, 10206 for E7, 3421 for E8, 7540 MPa for E9 and 8880 MPa for E10, respectively (Table 2).

The mechanical stability of E2 hybrid composite is higher as compared to other hybrid composites. This signifies the better adhesion exists between fibers and polymer assuring E2 composite suitability for green structural applications (Fig. 4).

Dart Impact Test

Dart impact strength is a valuable property in terms of structural applications. Dart impact strength explores a material ability to withstand a sudden force applied on the surface of hybrid composites and demonstrated the quality of the sample for structural industries. In this dart test, the impact strength is calculated for all hybrid composites using the free fall method. This is a reliable technique to understand the behavior of composite's impact resistance according to ASTM D1709. The thickness of the hybrid composite is 3 ± 0.05 mm. Initially, a 75 g dart is free-fell at 66 cm impact height to the surface of the hybrid composite and observed hybrid composite is partially fractured or not. The impact strength is calculated at dart impact failure weight. All characteristics such as dart impact strength, Dart impact energy, dart failure weight, and striking velocity before impact for E1, E2, E3, E4, E5, E6, E7, E8, E9 and E10 are shown in Table 2. The dart impact strength is 709.56 J/m for E1, 832.2 J/m for E2, 733.65 J/m for E3, 788.4 J/m for E4, 766.5 J/m for E5, 711.75 J/m for E6, 722.70 J/m for E7, 678.9 J/m for E8, 689.85 J/m for E9 and 700.8 J/m for E10, respectively (Fig. 5).

The impact strength depends on fiber-matrix interaction behavior and was observed highest in the case of 50% coir incorporated 50% treated-date palm fiber based hybrid composites (E2). Asim et al. [25] synthesized pine apple leaf based phenolic composites and observed similar result in 50% treated-pine apple leaf reinforced composites for green applications. So increase in impact strength was ascribed to

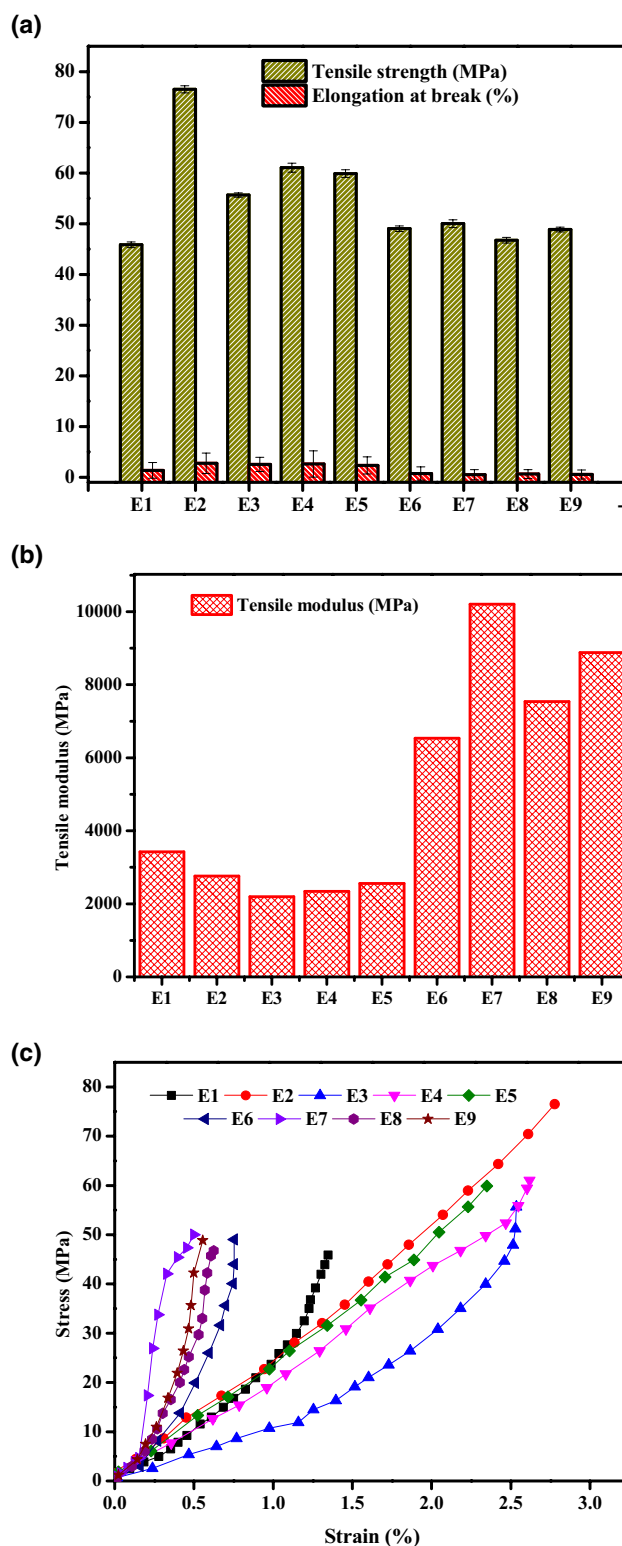


Fig. 4 **a** Tensile strength and elongation at break tests for E1, E2, E3, E4, E5, E6, E7, E8, and E9 hybrid composites. **b** Tensile modulus for E1, E2, E3, E4 and E5 hybrid composites. **c** Stress vs., strain curve for E1, E2, E3, E4, E5, E6, E7, E8, and E9 hybrid composites

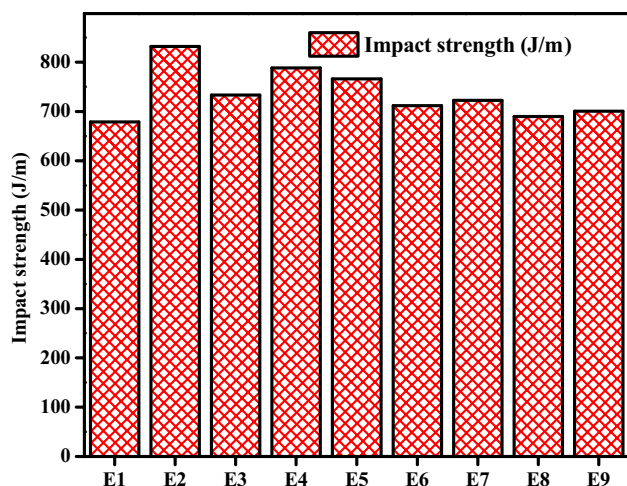


Fig. 5 Impact strengths for E1, E2, E3, E4, E5, E6, E7, E8, and E9 hybrid composites

homogeneous distributions of fibers in epoxy matrix resulting in remarkable impact strength. So, this excellent interfacial interaction presented between fibers and polymer which was proved in a stress test for green structural applications.

Conclusions

The impacts of fiber type and their different combinations on surface morphology, mechanical, thermal, crystalline and wettability characteristics were studied in this paper. The hybrid composite synthesized from treated-date palm fiber incorporated coir fiber based epoxy composite has proven as an inexpensive novel material for structural applications. The coir fiber has a remarkable impact on the thermal and mechanical properties of E2 hybrid composite, especially the wettability, load bearing ability and tensile strength. The E2 hybrid composite consists of equal weight fraction of treated-date palm fiber and coir fiber has represented the highest tensile and impact strengths with reliable elongation limit as compared to other hybrid composites at different weight fractions of fibers. The higher thermal stability with suitable less wettability of E2 hybrid composite has also managed to perform excellently for thermal stable hydrophobic structural applications. So it can be concluded that E2 had the ability to be used in structural applications which might be a possible solution of agricultural wastes.

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