Exploration of Mechanical Attributes, Thermal Behaviors and Atomic Force Analysis of Alkali Treated Hybrid Polyester Composites for an Engineering Application

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Abstract: This work exposes the mechanical attributes, characterization and thermal behaviors of alkali treated banana fiber (BF), coir fiber (CF) and palm fiber (PF) of Asian palmyra reinforced with polyester composites are expressed. Due to the high tensile attributes of banana fiber, in this experimental work banana fiber has considered as a base material, palm and coir fibers are utilized as fillers. The natural fiber composite specimens were fabricated through injection moulding method by varying the weight percentages of fibers. The mechanical behaviors were determined by tensile, flexural, impact, hardness tests. The Scanning Electron Microscope (SEM) morphological examines were carried out on the mechanical tested specimens to expect the fracture failure mechanisms. The thermal stability/degradation was observed by Thermogravimetric Analysis (TGA). Furthermore, Atomic Force microscopy (AFM) used as a tool to estimate the surface forces and roughness for treated fibers. The results revealed that the inclusion of three different fibers enhanced the overall mechanical, thermal features and reduced the surface roughness. This type of composite can be useful for various engineering domains.

Keywords: Banana, Coir, DMA, FTIR, Palmyra palm

Introduction

Natural fiber composites join plant-determined filaments with a plastic fastener. The natural fiber segments might be wood, sisal, hemp, coconut, cotton, kenaf, flax, jute, abaca, banana leaf fibers, bamboo, wheat straw or different stringy material. The utilization of common filaments decreases weight by 10 % and brings down the vitality required for creation by 80 %, while the expense of the part is 5 % lower than the equivalent fiber glass-reinforced segment [1]. The automotive and other industry has uncovered more tendency toward these materials on account of the preferences offered by them. Characteristic fibers composites offer different preferences over synthetic fiber composites, for example, minimal effort, biodegradability, low thickness, worthy explicit properties, protecting and better warm properties and low vitality utilization during handling, among others [2-4]. Numerous researchers in the past have created composites utilizing Natural filaments, for example, bamboo [5], coir [6], jute [7], sisal [8], and banana [9] to name the meager few. The natural composites by and large have just one sort of fortification and named as mono composites. Hybrid composites comprise of more than one sort of fiber in a single matrix material. Hybrid composites have extraordinary highlights that can be utilized to meet different plan prerequisites in a more conservative route than natural composites [10]. A few researchers have been occurred toward this path. The mechanical features of banana-glass fiber reinforced polypropylene hybrid composites were determined by Samal et al. [11]. The hybrid composite outcomes are contrasted and banana fiber fortified polypropylene composites, the results demonstrated that the most extreme enhancement in the attributes is seen at 30 wt% of fiber stacking. The static and dynamic mechanical characteristics of banana/glass woven texture fortified polyester composites were studied by Pothan et al. [12]. Successful fabrication of unidirectional banana, jute, and banana/jute fiber reinforced epoxy based hybrid composites through hand lay up method The longitudinal tensile and flexural strength of the composite is increased up to 30 wt% of fiber loading and then decreased with the increase in fiber loading [13]. Hybrid polymer composites of epoxy (E) reinforced with sisal (SF), banana (BF), coir (CF) and sisal/ banana/coir (SBCF) fibers were fabricated by compression moulding process and maximum tensile, flexural strength observed in SF reinforced epoxy composites [14]. Samal, and Pradhan [15] have examined the sisal, jute and jute, banana fibers are used as reinforcement with epoxy resin to form hybrid composite specimen. The Flexural strength was increased with increased in fiber content [15]. Boopalan et al. [16] have studied the jute and banana fibers were set up with different weight proportions and afterward joined into the epoxy matrix. This examination shows that expansion of banana fiber in jute/epoxy composites of up to 50 % by weight brings about expanding the mechanical and thermal characteristics and diminishing the moisture assimilation attribute. Nagarjun et al. [17] have explored the mechanical ascribes of the coir fiber fortified epoxy composites through expansion of high-quality sisal and palmyra palm strands. The results uncovered that the half breed composite have preferable mechanical highlights over the coir fiber composites because of synergistic impact of fortifications.

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Sathees Kumar [18] have reported the effects of sorghum bicolor, sisal fiber and jute reinforced with polyester composites. The results explored, by increasing the sisal and jute fiber wt% and decreasing sorghum contents the ductile property of the composites is increased. Apparently, without a doubt, not many writings revealed the properties of hybrid composites created utilizing three distinctive natural fibers. Nobody has considered the three different fibers such as banana, coir and palm filaments in their exploration work. In this experimental work, three different fibers were utilized to investigate the mechanical attributes, thermal degradation of composites examined through TGA. The morphological images were utilized to identify the fracture behavior mechanism of the natural fiber composites through SEM. AFM used as a tool to evaluate the adhesion strength and surface potential of the materials.

Experimental

Materials

The polymer used in this work development was unsaturated terephthalic polyester resin in the pre-accelerated form, produced by Royal Polímeros under the commercial name of Denverpoly 754. The matrix material was utilized in this study Araldite-LY 556 with Amine Hardener HY 951, supplied by New scientific Chemicals, Hyderabad, India. The natural fibers were purchased from Ebenezer fiber products, Coimbatore, Tamil Nadu, India.

Extraction of Natural Fibers

Banana Fibers

The extracting measures for the natural consistent banana fiber comprise of two significant components. Initially, the banana fiber is preoccupied from the completely developed trunk after the natural product has been culled so as to dodge the organic products getting squandered. At that point, the banana trunks are put under the daylight for the drying cycle inside about fourteen days. After the banana trunks are dried, they are absorbed the water for an additional fourteen days. In the event that under about fourteen days, the fiber will not be isolated from the un-utilized cell, though if the drenching time is over about fourteen days, the handling time will be squandered.

Coir Fibers

Raw coconut coir filaments were first exposed to Soxhlet extraction with acetone for around 24 h, followed by cleaning with distilled water and air-drying. The fibers were then separated in a 1:2 (vol./vol.) blend of ethanol and benzene for 72 h to dewax the sample, trailed by washing with distilled water and air-drying. The fibers along these lines obtained were alluded to as dewaxed or defatted filaments.

Palmyra Palm Fibers

The gathered palmyra filaments were absorbed water for

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Fibers	Density (g/cm ³)	Diameter (µm)	Tensile strength (MPa)	Tensile modulus (GPa)	Elongation at break (%)
Palm	1.45	200	89-222	1.09	14-23
Coir	1.2	43.3-140	271-281	8.87-8.9	15-40
Banana	7.5-9.5	10	529-914	27-32	1-3

Table 1. Physical properties of natural fibers [20]

about 24 h and strands were extricated physically from the stems. The extricated strands are treated with 1 % NaOH solution for 30 minutes and afterward altogether washed in distilled water to eliminate undesirable particles. The washed strands were dried at room temperature for two days. Further, the strands were kept in hot air broiler for 2 h at 70 °C to guarantee that most extreme dampness was eliminated, and afterward cut into 20 mm length.

Alkali Treatment of Natural Fibers

The natural fibers were absorbed 5 and 10 % NaOH arrangement at 31 °C. The fibers were kept drenched in the alkali solution for the two times of 24 and 48 h. The treated natural fibers were then washed a few times with distilled water. Any hints of NaOH, staying on the fiber surface, were neutralized with 2 % sulfuric acid during 10 min. The fibers were washed again with refined water until acquiring a pH= 7. Accordingly, the strands were dried at 60 °C for 6 hours. Researchers thusly found that alkalization likewise positively affects the mechanical attributes of fiber-strengthened composites, yielding noteworthy upgrades in the interfacial performance [19]. Physical attributes of natural fibers as shown in Table 1.

Fabrication of Samples Through Injection Moulding

Granulated material was dried in a stove at 70 °C for 2 h before injection moulding. This is to forestall the arrangement of air rises in the samples which may bring down their mechanical attributes. The materials aggravated were injection moulded into ductile test samples, utilizing an Injection forming machine. Table 2 shows the injection moulding machine set boundaries for the fiber/Polyester composites. The stack comprising of polymer was packed in a rectangular formed shape (160 mm×140 mm×4 mm) under a tension of 10 MPa and at a temperature of 190 °C for 10 min. A similar weight was kept up for 15 min during the cooling stage. The materials compacted were shaped as level test examples fabrication process of composite as shown in Figure 1. Composition of fabricated specimens as shown in Table 2.

Mechanical Characterization

Tensile Strength

The tensile strength of a material is the maximum amount of tensile stress that it can take before failure, for example breaking. The tensile specimen prepared is shaped into the required dimension using a hand cutter, and the edges are polished using a salt paper. It is prepared according to the



c. Palmyra tree and Fibers

Figure 1. Fabrication process of hybrid composite specimens.

Table 2. Composition of hybrid fabricated specimens

Designation	Banana	Coir	Palm
А	90	5	5
В	80	10	10
С	70	15	15
D	60	20	20
Е	50	25	25

ASTM D638 standard. The tensile test is conducted on a Tinius Olsen 10 KN Universal testing machine (UTM) with a gauge length of 75 mm and the crosshead speed of the machine is set at 5 mm/min. The specimen size for the tensile test is 115 mm×20 mm×3 mm according to ASTM D638. The process involves placing the test specimen in the UTM and applying tension to it until the fracture of the material. Furthermore, load the specimen into tensile grips and attach the extensometer to the specimen. Begin the test by separating the tensile grips at a constant rate of speed. The speed depends on specimen shape and the range form 0.05-20 inches per minute. Then the applied force is recorded as a function of the increase in gauge length [21]. During the application of tension, the elongation of the gauge section is recorded against the applied force. The tensile force is documented as a performance of the expansion in gauge length. The experiments are conducted four times in each composition and the average values are taken for results. The fabricated hybrid composite tensile specimen as shown in Figure 2.



Figure 2. Fabricated hybrid composite tensile specimen.

Flexural Strength

In this experiment, a 3-point bending method is used for finding out the flexure of a specimen. Flexural tests have been conducted by the same tensile testing machine. The size of specimen for flexural test is 110×15×3 mm according to ASTM D790. The 3-point flexural test is the most common flexural test and used in this experiment for checking the bending strength of the composite materials with a support span length of 70 mm and the crosshead speed of the machine was set at 2 mm/min. The testing process involves placing the test specimen in the UTM and applying force to it until it fractures and breaks. The measurements were taken at five magnitudes of the constant load for all specimens. The 3-point flexural test is the most common flexural test and used in this experiment for checking the bending strength of the composite materials with a support span length of 70 mm and the test speed is 0.05 to 0.1 in/min and adjustable span distance of 10-200 mm. The testing process involves placing the test specimen in the UTM and applying force to it until it fractures and breaks. The measurements were taken at five magnitudes of the constant load for all specimens. Fabricated



Figure 3. Fabricated hybrid flexural composite specimen.





hybrid composite flexural specimen as shown in Figure 3. *Impact Strength*

The ability of a material to absorb mechanical energy in the process of deformation and fracture under impact loading. The impact test is conducted on a Tinius Olsen testing machine and the test specimens are prepared according to the required dimension following the ASTM-D 256 standard. The Izod test specimen as per dimensions are 65 mm×15 mm×3 mm. The Izod impact test consists of a pendulum with a determined weight at the end of its arm swinging down and striking the specimen while it is held securely in a vertical position. Izod normally refers to a notched specimen impact, the notch is placed in the clamp and a pendulum is released that impacts the bar, measuring the energy required to break the sample. Fabricated hybrid impact test specimen as shown in Figure 4.

Hardness

A common way of measuring hardness of plastics is through Shore durometer testing. Shore is a measure of the resistance of the plastic material to indentation. The depth of surface penetration is measured using a durometer gauge which uses predetermined geometry. Durometer is used as a stiffness indicator since the depth of the penetration is directly related to the force required to make the penetration. Each hardness test specimen is prepared for the size of 35 mm×15 mm×3 mm [22-25]. Both force and deflection are measured. The ASTM D2240 standard specimen was used for hardness test in shore (Durometer) hardness tester (Make-Hiroshima) 1.40 mm diameter and 30 °C one indenter extension up to 2.54 mm with an applied force of 44.45 N. The device consists of a hardened steel rod with a truncated cone at the tip. The steel rod is spring-loaded and actuates a dial with a scale of 0 to 100. The test specimen is placed directly underneath the truncated cone, and the device is pressed down onto the part until the flat metal plate on the bottom is flush with the rubber specimen. The test specimens were kept under the indenter of shore D testers and the deflection on the scale was noted. The more the cone deforms the rubber material, the lower the hardness measurement. The less the cone deforms the material, the higher the hardness measurement. The indentation value reflects the resistance to local deformation, which is a complex property and related to modulus, strength, elasticity, plasticity and dimensional stability.

Scanning Electron Microscopy (SEM)

The morphology of the PA6 composites is observed using a Scanning Electron Microscopy (SEM) on a JEOL equipment model JSM-5300LV with 10 kV of voltage acceleration.

Thermogravimetric Analyzer (TGA)

Thermogravimetric analysis is performed on a EXSTAR TG/DTA 6300 TG instrument with the temperature accuracy \pm 5 °C to examine the thermal degradation behaviors of different specimens. Approximately, 5 mg of milled samples is placed in an alumina crucible with a diameter of 5 mm and is heated under dynamic linear rate of 10 °C/min, with a 50 cm³/min nitrogen flow from 50 °C to 600 °C.

Atomic Force Microscopy

Commercial atomic force microscope (AFM) (NT-MDT, Russia) was used for the present investigation. Gold-coated cantilevers with Si3N4 tip CSG 10, NT-MDT with a radius of curvature of the tip 35 ° and the cantilever elastic constant 0.1 N/m is used for this experiment without any further functionalization. AFM can offer improved resolution up to nano scale and give information about surface roughness parameters such as average surface roughness (Ra), Root mean square roughness (Rq or Rrms), Ten-point average roughness (Rz), skewness (Rsk), kurtosis (Rku) and maximum peak-to-valley height (Rt). This analysis was carried out by Park XE-70 Model AFM with XEI image processing software to analyse the data. The scanner scale range is 10 μ m×10 μ m in x, y direction with the 70 μ m resolution in the z direction.

Results and Discussion

Tensile Attributes

It tends to be seen from Figure 5. that ductile strength and ductile modulus of the polyester composite increments with common fiber stacking in all cases. The alkali base treated composites A, B, C and D show continuous upgrade of flexible quality. The mixture composites uncovered a superior impact up to the weight% of BF (60 %), PF (20 %) and CF (20 %) in example D. From the Figure 5 at the point when the equivalent load of fiber substance of PF and CF are broadened the rigidity and malleable modulus of the composites. In E example, the malleable quality was bit by bit lessened, because of the high expansion of (25 % PF and 25 % PCF) strands. Nonetheless, the low consideration of BF likewise liable for negative impact. Low substance of BF (50 %) traded the better flexible property into brittleness.

Hybrid Composites



Figure 5. Tensile properties of hybrid composite specimens.

The trial consequences of example D has upgraded the general flexible conduct contrasted and different examples, for example, A, B, D and E. The examination esteems are seen in percentages are 9.34 %, 7.43 %, 5.68 %, 2.43 % individually. Besides, the ductile modulus of NF composites was improved consistently from A, B, C and E samples. In E sample the quality was diminishing because of the failure of the fiber to help stresses moved from the polymer matrix [26,27]. The tensile modulus of sample D contrasted and remaining samples, such as, A, B, C and E. The examination modulus esteems are concentrated in rates 25.34 %, 12.36 %, 5.51 % and 4.72 % respectively. Due to the load transfer between the polyester matrix and reinforced natural fibers were enhanced in specimen D.

Flexural Strength

From Figure 6 that the flexural quality of the polyester composite escalates with natural fiber stacking in all samples. The composites A, B, C, D and E shows steady upgrade of twisting quality. Figure 8 indicates that the low substance of PF and CF filaments to the polyester improves the flexural quality and the diversion on Specimen D. Better holding zones among fibers and polyester resin and essentially no indications of fibers debonding, detachment or pull out systems at the treated NF composite break surface after flexural stacking. This result recommends that



Figure 6. Flexural strength of hybrid composite specimens.



Figure 7. Flexural modulus of hybrid composite specimens.

interfacial connection between the three fibers and polyester matrix has gotten essentially more certain and unending gracefully of fibers with NaOH. Flexural properties likewise upgraded with filler expansion [28]. Besides, the flexural modulus of composites as appeared in Figure 7. As the BF content goes upto 60 %, the flexural modulus grows perceptibly in polyester strengthened composites. This improvement is an away from of better successful burden move between the polyester framework and filaments. Stress move of the composites expanded by solid fiber and framework holding, permitting flexible distortion [29]. It is consequently clear that the soluble base treatment essentially grows the versatility of NF composites.

Impact Strength

Figure 8 the impact of composites enhances from A, B, C, D and E progressively. The weight of BF (60 %), PF (20 %) and CF (20 %) are extended the extreme impact of about 36 J/m^2 . Specimen E compared with other specimens like A, B, C, and D were attained the better impact of 24.13 %, 16,12 %, 12.5 %, 11.5 % and 5.88 % respectively. The expanded fiber substance will augmentation the contact surface between the fiber and matrix if there is acceptable impregnation of filaments in the resin. At higher fiber stacking the effect move ought to be more proficient [30].



Figure 8. Impact strength of composite specimens.

Significant attachment between the fiber and polyester matrix is too liable for the great protection from break spread during impact tests. The effect of strain rate on fracture and ductility of the material can be analyzed by using the impact test. Significant attachment between the fiber and polyester matrix is too liable for the great protection from break spread during impact tests.

Hardness

Figure 6 represents the hardness values of the natural fiber composites. Alkali treated fibers reveals the rapid enhancement of hardness values. In specimen A it has low value 82.3 then it has progressively enhanced upto 92.6 on specimen E. The higher content of BF and low addition of (20 % CF and 20 % PF) natural fibers boosted the hardness attributes of the polyester composites [31]. The reason for increasing the hardness is the better dispersion of fibers into the polyester matrix stronger interfacial adhesion to the fiber matrix.

Morphological Study of Tensile Fractured Specimens

The SEM morphologies of tensile fractured specimen as shown in Figure 9(a-e). Figure 9(a) represents the BF, CF and PF reinforced polyester matrix. The interfacial bonding between the fibers and polyesters were very poor. Due to improper bonding of fiber and matrix, specimen A has not sustained the maximum load and It was attained very low tensile strength. Figure 9(b). revealed the more voids and matrix fracture aspect. Few fibers were pulled out in the image. The fibers are also shortened by injection moulding. But even these short fibers not deliver significant reinforcing effect. Compared to Figure 9(a) and Figure 9(b), Figure 9(c) has explored the better interfacial bonding between the fibers and matrix. It is not visualizing the voids and agglomeration of the fibers. However, three different fibers were well trapped with matrix. Even though, it was slightly shown the misalignment fibers in the left corner of the image. Figure 9(d) represents the good interfacial and proper alignment of all fibers and matrix. Bonding between the fibers and polyester shows the better adhesion. Due to good adhesion of fibers, the high load or interfacial stress has been enhanced in the specimen D. In Figure 9(e), the fibers are not properly aligned. Furthermore, there exists an optimum concentration of NaOH and immersion time, beyond which delignification of the fiber takes place resulting in damaged fiber and leading to decrease in the mechanical properties [32,33]. Higher interfacial stress can develop, reducing the work of fracture and causing the fracture to be more brittle.

Thermogravimetric Analysis

Thermogravimetric investigation (TGA) is valuable procedure for the investigation of the thermal strength/ deterioration of natural composite materials. The thermal stability of the fiber is a significant boundary for the preparing and utilization of these materials. The assembling of such composites needs the blending of fibers and matrix at elevated temperatures, so the degradation of the cellulosic materials can deliver bothersome impacts on the attributes. The amount of weight reduction of the sample as an element of temperature estimated to anticipate thermal conduct of the material [34].

TGA treated natural fiber was studied as a function of percentage weight loss with the increased in temperature. The mass loss increases with temperature, for a heating rate of 10 C/min, were shown in Figure 10. The treated NF



Figure 9. SEM morphologies of tensile fracture specimens; (a) specimen A, (b) specimen B, (c) specimen C, (d) specimen D, and (e) specimen E.

Hybrid Composites



Figure 10. TGA of specimen E.

indicated a comparative pattern of deterioration with one phase of weight reduction measure, which had a progress temperature that started from 235 to 325 °C and the range for definite change at 350-400 °C. From a room temperature until 32 °C, the TG thermograms (Figure 10) present the loss of mass because of the water vaporization heat in the example. In light of these outcomes, the warm strength of lignocellulosic materials was expanded by the alkali treatments. This could be credited to the evacuation of hemicellulose and lignin during the alkaline treatments, just as to the further extent of crystallinity in the material after mechanical processing. Therefore, the more prominent crystalline structure prompted a high obstruction towards warmth and increment in the greatest temperature for thermal degradation [35,36]. Thermal debasement is additionally the significant aspect in the improvement of natural fiber composites since it will emphatically influence the greatest temperature utilized in the preparing composites.

Surface Topographical Analysis of NFC

The AFM analysis do not damage the fiber's surface and also, its ability to provide high resolution images of sample's surface in both 2D and 3D. The topographical AFM images of composition of NFC are shown in Figure 11. The average surface roughness (Ra) of NFC were calculated as $0.052 \,\mu\text{m}$ and it indicates the presence of impurities and amorphous content of lignin on the surface of the NF. This observation is also strengthened by the other surface parameters such as skewness (Rsk), kurtosis (Rku), RMS roughness (Rq), average maximum height of the profile (Rz), and maximum height of the profile are $0.228 \,\mu\text{m}$, 2.214, $0.062 \,\mu\text{m}$, $0.225 \,\mu\text{m}$, and $0.143 \,\mu\text{m}$, respectively. Hence, for potential use and applicability of NFC as a reinforcement material in polyester composites are in need of surface modifications, in order to enhance the various surface parameters.

Conclusion

The banana, coir and palmyra palm natural fibers are reinforced polyester composites on different weight percentages by injection moulding method. Mechanical attributes such as, tensile, flexural, impact and hardness behaviors of hybrid composites were investigated. Thermal characterization and AFM analysis were performed in this work. From the above results, the following conclusions were drawn: The inclusion of three different fibers reinforced



Figure 11. (a) 3-D roughness surface texture, (b) 2-D roughness surface texture, (c) 2-D line diagram for roughness measurement of NF, and (d) roughness parameters.

polyester composites enhanced the tensile, flexural, impact and hardness of hybrid specimens. The over all mechanical attributes of hybrid composites were significantly developed due to the strong interfacial adhesion and uniform dispersion of fibers and polyester matrix. However, increase the banana fiber and equal sharing of coir and palm fiber loadings in polyester composites enhances the thermal and AFM characteristics. The specimen D enhanced the tensile strength and tensile modulus values were upto 25.34 % and 24.52 % respectively compared with specimen A. The flexural strength and flexural modulus of specimen E has upgraded upto 13.04 % and 21.24 % respectively compared with specimen The impact strength and hardness of specimen E has improved upto 24.13 % and 9.75 % respectively compared with specimen A. These effects are attributed to the good reinforcing effects of natural fibers and the formation of interfacial interaction to the polyester matrix. This type of composite material can be useful various engineering domains.

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