**ORIGINAL ARTICLE** 



# Effect of optimized alkali-silane treatment on mechanical and fatigue behavior of maize husk fiber epoxy composites: a strength factor approach

R. Kirubagharan<sup>1</sup> · S. Dhanabalan<sup>1</sup> · T. Karthikeyan<sup>2</sup>

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#### Abstract

This study examined the role of maize (corn) husk fiber and its surface treatment pattern in an epoxy matrix by assessing its mechanical characteristics and fatigue behavior. The main objective of this study was to analyze the effect of alkali-silane treatment on the composite's characteristics utilizing strength factor methods. Varying weight percentages of base and silane durations were employed to create a variety of composite combinations. The laminates used in this investigation were fabricated by hand layup process and characterized by the American Society for Testing and Materials (ASTM) standards. From the outcomes of the study, it is observed that the strength factor shows the highest rank of 95.06 for composite designation EA21, which is base and silane treated for 4 h and 4 wt.%. It is observed that the 8 h of base treatment with 4 wt.% of 3-aminopropyltriethoxysilane (APTES) results in the lowest mechanical properties for composite EA32. The fatigue behavior gives a significant result with 2 h of base treatment and 2 wt.% of amino silane coupling agent (APTES). The composite designation EA11 shows a maximum fatigue life count of 27,942. The lowest recorded fatigue life count for a composite designation with 8 h of base treatment and 2 wt.% silane was about 21,408. But composite designation EA21 also shows a fatigue life count of about 25,318, which is nearby to the fatigue life of composite designation EA11. Thus, it is clear that 4 h of base treatment and 2 wt.% of silane coupling agent in silane solution would be the optimistic surface treatment process for corn husk fiber epoxy composites. These improvements in properties of corn-epoxy composites could be beneficial in a variety of engineering applications starting from structural to aerospace.

Keywords Mechanical properties · Natural fiber · PMC · Silane treatment · Strength factor

# 1 Introduction

Synthetic fiber polymer composites such as kevlar, glass, and carbon fiber reinforcement with polymers have been widely employed in the automotive, space sector, construction, leisure, and sports industries for a very long time. Because they are less expensive and have better mechanical qualities than aramid and carbon fibers, glass fibers are utilized extensively. However, these fibers have a number of significant

 R. Kirubagharan kirubagharanr@rediffmail.com
S. Dhanabalan dhanabalan69@gmail.com

<sup>1</sup> Department of Mechanical Engineering, M.Kumarasamy College of Engineering, Karur 639113, Tamilnadu, India

<sup>2</sup> Department of Mechanical Engineering, Sri Eshwar College of Engineering, Coimbatore 641202, Tamilnadu, India disadvantages such as high density, expense, high energy consumption, non-renewability, and non-disposability [1]. However, natural fiber-reinforced composites have evolved over the past 10 years into feasible substitutes to conventional glass fiber composites in several technical purposes. The world is looking for insane eco-friendly materials; as a result, researchers around the world are concentrating on inventing new materials that will enhance the environmental quality of products [2]. This demand for new eco-friendly materials has led to the use of composites consisting of raw natural fibers and polymer matrices, which have become one of the most intensively researched areas in recent years. Natural fiber composites are an alternative to synthetic materials that are damaging to the environment and aid in reducing emissions [3]. In addition, they are inexpensive, offer superior mechanical qualities, and need little energy to produce. It is also possible to increase sustainably by limiting construction wastes by utilizing such materials in construction projects [4].

In the past 10 years, natural plant fibers (jute, sisal, coir, banana, hemp, kenaf, flax, etc.) have attracted the interest of numerous researchers all over the world who aim to use them as an alternative reinforcement for synthetic fiber-reinforced polymer composites [5]. Because of their lightweight, low cost, reduction in carbon emission, superior thermal and mechanical qualities, high specific strength, and biodegradability, these fibers are becoming an attractive alternative to traditional fibers (such as glass, carbon, and aramid) [6]. Some alkali treatments were required to strengthen the fiber matrix interfacial bonding and decrease the fibers' moisture absorption, hence improving the physical and chemical properties of such fiber-reinforced composites [7]. It is well known that starch is the most common polymer, and that it is frequently utilized as a matrix for composite materials. In addition to being abundant, it is also renewable, affordable, and biodegradable. Corn (maize) is the primary source of readily available starch, and each corn granule includes more than 70% starch in addition to minor components such as lipids, crude protein, sugar, and ash [8].

Corn husk is an easily available bio waste product and it can be converted into natural fiber. Several research studies have been performed on the use of thermoset polymeric composites with corn husk fiber in the making of biocomposite [9]. Ratna et al. [10] researched on advances and prospects of corn husk as a sustainable material in composites and other technical applications. The authors conclude that utilizing corn husk fiber for technical applications could become a major breakthrough in this regard and play an important role for the formation of new economy. Alshahrani et al. [11] analyzed the mechanical, fatigue, and DMA behaviors of high-content cellulosic corn husk fiber and orange peel biochar epoxy biocomposite. The presence of cellulose content in fibre offered high toughening effect and reduced the extreme brittleness of epoxy. In addition to this, the composite has maximum mechanical properties with 166, 191 MPa of tensile and flexural strength, 6.64 J of impact toughness, 91 of shore-D hardness, and 28,554 of fatigue life counts.

Corn husk fiber-reinforced polyester composites were studied by N. H. Sari et al. [12] for their tensile strength qualities, water absorption, and morphological behavior. After being soaked in water for 24 h and 72-h pouring, the results indicated that the composites' water absorption characteristics increased with increasing fiber. Composites' tensile strength and modulus of elasticity tend to rise from 20 to 30% fiber content after 24 h of immersion, and then fall as fiber content and soaking period increase because of bonding interferences.

Similarly, Herlina et al. [13] investigated the effects of water immersion and fiber content on the characteristics of a thermoset polyester composite reinforced with corn husk fibers. This study investigates the effect of corn husk fiber (CHF) content on the mechanical characteristics, water absorption behavior, and swellability of CHF/polyester (PE) composites utilized in aquatic settings. The mechanical properties of submerged composites (tensile, bending, and compression strengths) were thoroughly studied. The results suggest that composites with higher CHF content and longer immersion period are more sensitive to having inferior mechanical characteristics. The considerable amount of water absorbed by the composite diminishes the bonding contact between CHF and PE, which causes damage.

However, there are a few investigations in the field that further discuss chemically treating corn husk fibers, but the articles were unable to identify any articles that examine the effect of optimized alkali-silane treatment on mechanical and fatigue behaviors of corn husk fiber epoxy composites. In the case of corn husk fibers, no literature relevant to chemical modification other than alkalization treatments has been discovered. Hence this study is focused on alkalisilane treatment on corn husk fiber within an epoxy matrix and their effect on mechanical and fatigue properties. The laminates were prepared with the help of the hand layup method and characterized according to the respective ASTM standards. Such composites materials could be used in structural, defense, automotive sector, and domestic appliances.

# 2 Experimentation

#### 2.1 Materials

In this investigation, a liquid bisphenol-A type araldite epoxy (LY556) was acquired from Huntsman India Ltd., Mumbai, with an equivalent molecular weight of 195 g/mol, a viscosity of 12,000 cps, and a density of 1.17 g/cm<sup>3</sup>. Samples were cured with the TETA curing agent (HY951) obtained from Huntsman India Ltd., Mumbai, which had a viscosity of 20 cps and a density of 0.98 g/cm<sup>3</sup>. The 3-aminopropyltrimeth-oxysilane (APTMS) surface modifier was purchased from Sigma Aldrich, USA. Corn husk fibers with a density of 0.74 g/cm<sup>3</sup>, a diameter of approximately 300–400 µm, and a length of 60–80 mm were acquired from metro composites.

#### 2.2 Single fiber extraction

The harvested corn husks from nearby farms were collected and used as reinforcement. Additionally, the husks were washed and immersed in water for 2 weeks to induce microbial decomposition so that they would become soft. After separation of the cellulosic and other particles, they were washed and dried under the sun to eliminate any residual moisture. After the fleshy layer had been wilted, unwanted particles were removed by combing [14]. The extracted fibers from the corn husk were allowed to dry naturally for a second time before being cut to the desired length of 60–80 mm. Utilizing standard testing procedures, their physical, chemical, and mechanical properties were determined. The chemical properties of this fiber are like cellulose 44%, holo-cellulose 23%, lignin 6%, wax 0.8%, hemi-cellulose 24%, and char 2.2%. Similarly, the physical properties were mentioned above. The steps followed in corn husk fiber preparation are shown in Fig. 1.

## 2.3 Surface treatment

#### 2.3.1 Base treatment

The corn husk fibers were treated with alkali previously to the silane surface treatment process. The primary objective of alkali treatment was performed to remove lignin and hemicellulose from the fiber's outer wall. During this procedure, the fiber was submerged in a 1-N NaOH solution for approximately 12 h. After 2, 4, and 8 h, the fiber diameter and surface crystalline changes were examined using SEM and FT-IR. Figure 2 depicts the SEM picture of alkali-treated corn husk fibers for 2, 4, and 8 h. The diameter of the fibers significantly reduced as the treatment duration increased [15]. For treatment times of 2, 4, and 8 h, the diameter measured was 246, 214, and 202 µm, respectively, as shown in Fig. 2. The as-received fiber shows a diameter of about 284 µm and is represented in Fig. 2(a) and further base-treated corn husk fibers' SEM fractography is illustrated in Fig. 2(b) for 2 h, (c) for 4 h, and (d) for 8 h. This diameter reduction occurs due to the elimination of lignin and semi-cellulose. As semi-cellulose and lignin have limited adhesion to lumen, they are easily removed from the fiber surface [16]. Observations show that the diameter reduction is nearly uniform up to 4 h. Only 44 µm is reduced over a period of 2–8 h, indicating the disappearance of amorphous cellulose and the development of crystallized micro fibrils in the cellulosic layer [17].

#### 2.3.2 Silane treatment

The base-treated corn husk fibers were subjected to a further hydrolysis process to create silane-treated fibers; the fibers were then immersed in an ethanol–water-silane solution [18]. A mixture of 95% ethanol and 5% water was stirred for 10 min. A varying amount of silane coupling agent, approximately 2 and 4 wt.% concentration separately, was added drop by drop to obtain a homogenous mixture, followed by 5 min of gentle stirring. Ten minutes was spent immersing the fibers in the ethanol–water solution. The surface-treated fibers were extracted from the aqueous solution by gently pouring the remaining solution. The surface-treated fibers were briefly rinsed with ethanol to remove excess silane and dried in an oven at 110 °C for 10 min to eliminate moisture [19].

Figure 3 depicts the Fourier transform infrared spectroscopy for the as-received and silanized fibers of corn husk fibers. Utilizing FTIR spectroscopy, the presence of silanized fiber was investigated (Bruker Alpha ATR mode, Germany). Samples obtained from silane treatment were placed in a KBr pellet container, and reference spectra were examined. Figure 3(b) displays the frequency versus observed peaks of silanized corn husk fibers. The signal at 3498.29 cm<sup>-1</sup> indicates the presence of a N–H stretch as an amine group on the surface of the fiber. The signal at 2916.37 cm<sup>-1</sup> indicates the presence of a C–H stretch, which is a propyle group bonded to the surface of corn husk fibers. The signal at 1519.91 cm<sup>-1</sup> indicates that C=C is an alkaline bond. The signal at 894.97 cm<sup>-1</sup> indicates that the surface of the fiber contains a condensed Si–O–Si structure. Figure 3(a)

**Fig. 1** Corn husk fiber preparation process





displays the spectrum of as-received corn husk fibers, confirming the lack of functional groups on the fibers' surface. Consequently, the investigation of FTIR spectra revealed that the surface modification process generates an  $NH_2$  function group on the cellulose surface, which improves adhesion and the bonding mechanism between fiber and matrix materials [20].

## 2.4 Composite making

The bio-composites were fabricated by the hand layup process to make a composite laminate. The required amount of epoxy (LY556) resin was taken into the beaker and mixed with a hardener (HY951). Then, a 3-mm thickness (as per ASTM) mould was prepared by applying a thin layer of wax for easy removal of the composite laminate. This ready mix was further poured in the wax-coated mould. After pouring the resin surface, treated corn husk fibers were immersed by the hand layup process. The excess resin was wiped out by cotton and a cotton roller was used to make its thinness uniform. The composites were cured at RT for 24 h and post cured at 120 °C for 48 h [21]. The same process is followed for making different composite compositions as shown in Table 1.

# **3** Characterizations

The created films were inspected by naked eyes to identify the existence of any flaws and cleansed with the help of a cotton fabric. The testing specimens were cut in accordance with ASTM standards by following Table 2 and shown in Fig. 4. In all the test methods, a minimum of five samples were tested to compute the average [22].

# 4 Results and discussion

## 4.1 Mechanical properties

The mechanical properties for various composite designations are shown in Table 3. It shows that the highest observed strength factor is 95.06 for composite designation EA21, which means this composite designation gives optimum results among all of the composite designations. The reason behind such results is the 4 h of base treatment and 2 wt.% of silane treatment process. The 4-h base treatment reduces the semi-cellulose and lignin, and silane coupling agents modify the cell walls of corn husk fiber surface, which improves the adhesion and moisture resistance properties [23, 24], as shown in Fig. 5(a). By calculating the normalized strength,



Fig. 3 FTIR spectra for as-received and silane treated fibers

Table 1 Different composite designations for various compositions

Composite	Epoxy	Corn husk	Condition	
designations	resin (vol. %)	fiber (vol. %)	Base treat- ment	Silane treatment
Е	100	-	-	-
EA11	70	30	2 h	2 wt.%
EA12	70	30	2 h	4 wt.%
EA21	70	30	4 h	2 wt.%
EA22	70	30	4 h	4 wt.%
EA31	70	30	8 h	2 wt.%
EA32	70	30	8 h	4 wt.%

the strength factor was determined. The ranking of individual composite designations was then determined by adding up all of the normalized strengths. Similarly, composite

Table 2 ASTM standards for tests with specification

Sr. No	Tests	ASTM	Machines
1	Tensile	D-3039	INSTRON 4855, UK,
2	Flexural	D-790–17	Traverse speed of 1.1 mm/s
3	Izod impact	D256–10	Krystal Equipment Ltd., India, maximum load capacity of 20 J
4	Hardness	D 2240	Durometer (shore-D)
5	Fatigue	D 3479	MTS Landmark 370 load frame, USA
6	SEM	-	HITACHI, S-1500, Japan

designation EA22 shows the closest rank to the composite designation EA22 due to the 4 h of base treatment, but a slight change is observed because of the 4 wt.% of silane



Fig. 4 Test samples as per the ASTM standards

coupling agent addition. However, the lowest observed strength factor after the treatment of fibers is about is about 80.1 for composite designation EA32, which is treated for 8 h of base treatment with 4 wt.% of silane agent. The cause of this is the enriching of silane concentration during silane treatment that induced a minor increase in hemicellulose content [25, 26]. The pure epoxy composite designation E shows very poor mechanical properties like 64 MPa for tensile strength, 98 MPa for flexural strength, 0.34 J for Izod impact, and 86 shore-D for hardness. This is due to the absence of reinforcements and only the plain epoxy as main consistent of this composite [27, 28]. Further inclusion of corn husk fiber with 2-h base treatment and 2 and 4 wt.% of silane solution shows the tensile strength, flexural strength, Izod impact, and hardness values of about 88 MPa, 64.7 MPa, 3.64 J, and 86 hardness for composite designation EA11, respectively, as well as 102 MPa, 132 MPa, 3.82 J, and 87 shore-D for composite designation EA12. The addition of corn husk fibers improves the strength of composite materials due to load bearing ability of the reinforcements, as shown in Fig. 5(b). This value further gradually increases maximum up to the composite designation EA21 and starts to reduce for further surface treatment, which gives minimum observed outcomes for composite designation EA32. The increment in base treatment hours reduces the fiber diameter as discussed previously, and higher concentration of silane solution shows the raise in relative amount of cellulose, as elaborated in Fig. 5(c) [29].

## 4.2 Fatigue properties

Table 3 shows the fatigue properties for various composite designations. The least fatigue life cycles were shown by composite designation "E," about 548. These lower fatigue life cycles are because of the epoxy molecular store's plastic strain [30, 31]. Further incorporation of corn husk fiber improves the fatigue life cycles. The composite designation

lable 5 Sur	engin factor c	alculations of va	rious composi-	te designations							
Composite lesignation	Tensile strength (MPa)	Normal- ized tensile strength	Flexural strength (MPa)	Normal- ized flexural strength	Izod impact (J)	Normalized Izod impact (J)	Hardness (shore-D)	Normalized hardness shore-D	Fatigue life cycles	Normalized fatigue life cycles	Strength Factor
ш	64	52.9	98	63.6	0.34	8.9	86	98.9	548	2	45.26
EA11	88	64.7	118	76.6	3.64	95.3	86	98.8	27,942	100	87.08
EA12	102	75	132	85.7	3.82	100	87	100	27,311	97.7	91.68
EA21	136	100	143	92.9	3.55	92.9	86	98.9	25,318	90.6	95.06
EA22	122	89.7	154	100	3.48	91.1	87	100	26,377	94.4	95.04
EA31	94	69.1	126	81.8	3.18	83.2	86	98.9	21,408	76.6	81.92
EA32	88	64.7	102	66.2	3.26	85.3	87	100	23,555	84.3	80.1

Fig. 5 SEM fractography for

tensile fractured samples



EA11 shows a higher fatigue life cycle of about 27,942 by addition of 30 vol.% of corn husk fibers. This improvement in fatigue life cycles is a result of the ability of fibers to bear the load transferred by the epoxy matrix [32]. Furthermore, the base treatment and silane treatment result in the development of surface roughness, which increases the amount of cellulose coated on the surface of fibers, resulting in improved interfacial adhesion between corn husk fibers and the epoxy matrix and increase in the number of bonds between the epoxy matrix and the fiber surface, as shown in Fig. 6(a). However, increase in base treatment hours and increase in silane wt.% show decrement in fatigue life cycles, minimum up to 21,408 for composite designation EA31. The cause of this is reduced diameters of fibers by base treatment and shows the poor bonding [33] of corn husk fiber and epoxy matrix, as represented in Fig. 6(b). The composite designation EA21 gives fatigue life cycles of about 25,318 with 4 h of base treatment and 2 wt.% of silane treatment. The fatigue life cycles decrease due to the base treatment, which reduces the surface of fiber and weakens it, that is not able to sustain against the fatigue life cycles [34, 35] and shows fiber pullout as illustrated in Fig. 6(c). But these life cycles were about to near the highest life cycles observed for composite designation EA11.

# **5** Conclusions

The role of corn husk fiber in the epoxy matrix was studied in this investigation by finding its mechanical properties and fatigue behavior. This study aims to analyze the effect of alkali-silane treatment with a strength factor approach. The different wt.% of silane and different base treatment hours were used to make various composite combinations. The laminates for this study were prepared by the hand layup technique and characterized according to the respective ASTM standards. It is observed that the incorporation of corn husk fiber in pure epoxy gives improved mechanical properties and fatigue life. The strength factor of various composite combinations shows the highest rank of 95.06 for composite designation EA21. It indicates that composite EA21 gives overall good strength value in all aspects and produces balanced results in all parameters. It means 4 h of base treatment and 2 wt.% of silane solution as surface treatment is the



optimum combination. Similarly, the composite designation EA22 shows a 95.04 rank, which is closer to the composite designation EA21. The mechanical properties were increased with an increase in base treatment hours of up to 4 h and showed a decrement in values for further increment. On the other hand, silane treatment with 2 wt.% gives the highest results than silane treatment with 4 wt.% of silane coupling agent. The pure epoxy composite designation E shows very poor mechanical properties like 64 MPa for tensile strength, 98 MPa for flexural strength, 0.34 J for Izod impact, and 86 shore-D for hardness. The composite designation EA21 shows the maximum mechanical properties of about 136 MPa, 143 MPa, 3.55 J, and 86 shore-D for tensile strength, flexural strength, Izod impact, and hardness. It is noted that 8 h of base treatment with 4 wt.% of silane coupling agent possesses the lowest mechanical properties for composite designation EA32. However, the fatigue behavior shows diversification in results as compared to mechanical properties. Here, composite designation EA11 shows the highest fatigue life count of 27,942 with 2 h of base treatment and 2 wt.% of silane coupling agent. The lowest observed fatigue count was about 21,408 for a composite designation for 8 h of base treatment and 2 wt.% of silane solution.

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Author contribution R. Kirubagharan and S. Dhanabalan had undertaken all the experimental work and testing. T. Karthikeyan provided guidance in manuscript preparation and proofreading.

#### Declarations

Conflict of interest The authors declare no competing interests.

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