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ORIGINAL ARTICLE

Development of thermally reduced corn stover biochar and its satin weaved sisal-reinforced vinyl ester composites

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Abstract
The aim of this research is to develop thermally reduced corn stover biochar and combined it with the satin weaved sisal-reinforced vinyl ester composites. This investigation focus on mechanical, wear, DMA, fatigue, and hydrophobic properties of this composite, characterized according to ASTM standards. Composites are made after silane surface treatment on reinforcements and fabricated by hand layup process. Results show that the composite with the 5.0 vol.% biochar had the highest values of mechanical properties, which were 134 MPa, 4372 MPa, and 4.74 J for tensile strength, flexural strength, and Izod impact, respectively. But further increased in biochar up to 5.0 vol.% shows the reduction in mechanical properties. On the other hand, the composite designation VS3 was discovered to have the maximum storage modulus and lowest loss factor with inclusion of 5.0 vol.% of biochar particles as well as it shows the better wear characteristics of about 0.28 coefficient of friction and 0.009mm^3/Nm sp. wear rate. However, maximum fatigue life counts of about 28,813 were observed by addition of 3.0 vol.% of biochar. The composite material designated VS3 exhibits the highest recorded contact angle, which is around 71°, indicating that it is hydrophobic in nature. SEM fractography demonstrates better fiber-to-matrix adhesion as a result of surface treatment on reinforcing materials. Furthermore, such composites could be used in industrial and domestic applications.

Keywords Satin weaved sisal fiber · DMA · Biochar · Fatigue life counts · Mechanical properties

1 Introduction

Composites are made up among different materials blended together while still retaining the unique characteristics of each material. Together, these parts work to provide the necessary toughness and mechanical qualities to composite materials [1]. It is made up of two or more separate phases (the matrix part and the dispersed section), each of which has large volume characteristics that are noticeably different from the others. The major phase with a continuous property is the matrix section, which is typically more ductile [2]. Fiber or reinforcements distribute the load equally and hold the secondary phase, also known as the scattered phase. The matrix contains a discontinuous form of the

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phase separation. This phase is also known as the reinforcement agent because it is typically stronger than the matrix [3]. Vinyl ester is one of the most durable alternatives for regions subjected to such problems due to its great chemical, thermal, and mechanical resistance. For increased endurance, the majority of vinyl ester systems have several layers. However, over the last few hundreds of years, natural fibers such as cotton, bamboo, jute, hemp, flax, and sisal have been utilized as substitute reinforcing fibers in wide applications of polymer composites due to several advantages over synthetic fibers, e.g., minimal cost, minimal wear rate throughout processing, light weight, ecologically friendly, biocompatible, and affordable from biomass resources [4].

Among all-natural fiber, sisal fiber is one of the prominent reinforcements which deliver excellent mechanical properties to the composite materials. The sisal fiber is non-toxic, relatively inexpensive, easily accessible, and has a low density and increased specific strength and modulus [5]. Chaitanya et al. [6] researched on the recyclability of PLA/Sisal fiber biocomposites. Up to the third recycle, the tensile strength of injection molded biocomposites decreased by 20.9%. Beyond the third recycling, the dynamic mechanical investigation demonstrated a significant fall in storage and loss modulus. Morphological and thermal evaluation of recycled biocomposites revealed significant fiber and matrix disintegration. Murugan et al. [7] examined the mechanical and physical properties of banana/sisal fiber–reinforced interpenetrating polymer networks (IPN) composites. In all physical tests, the sisal fiber–reinforced IPN laminate demonstrated a significant rise except the moisture absorption properties. However, compared to other woven types, satin woven fabrics are strong due to the large number of threads employed, yet the lesser interlacing gives flexibility and wrinkling resistance. Satin textiles are almost usually warp-faced and made of glossy filament yarns with a very low twist [8].

Additionally, the different types of particles are used as fillers in the composite materials which may improve the material's thermal, mechanical, damping, wear, and fatigue properties [9]. To make these composites more thermally stable and highly toughened and to enhance the efficiency of natural fiber composite, biochar has been introduced in recent studies [10]. Numerous researchers have attempted to develop novel eco-friendly biofillers for enhancing the matrix phase's characteristics using convenient domestic materials [11]. Due to the high rate of maize production, after harvesting, its wastes are massive in India and worldwide. These corn cobs and corn stover are frequently burned or discarded as agricultural waste, causing major environmental problems such as air pollution and soil contamination. Alternate techniques must be implemented in order to conserve the environment; hence, the utilization of corn stover for biochar preparation has been taken into consideration [12]. A new polymer composite for biosensors based on activated biochar was developed and characterized by Sobhan et al. [13]. Corn stover was used to create biochar, which was afterward activated through the steam. Using a solvent casting process, activated charcoal (ABC) was created using polylactic acid (PLA). When PLA contents rose from 15 to 50%, the ABC/PLA composite film's tensile strength (TS) and Young's modulus raised from 0.81 to 3.04 MPa and 56.31 to 102.69 MPa, correspondingly. Xu Zhou et al. [14] researched on environment-friendly corn stover/poly (butylene adipate-co-terephthalate) PBAT's biopolymer composites. While maintaining the same CS content, reducing CS size improves particle dispersion, boosts PBAT's degree of crystallinity, and greatly enhances the CS/PBAT composites' thermal stability and tensile characteristics.

Hence, based on previously published literature studies and research circumstances, the fabrication of thermally reduced corn stover biochar and its satin weaved sisal-reinforced vinyl ester composites was analyzed. The mechanical, wear, DMA, fatigue, and hydrophobic properties have been characterized according to ASTM standards for this composite laminates. The composites from corn stover biochar and satin weaved with vinyl ester were fabricated by hand layup process. However, the simplest and most traditional open molding technique for making composites is hand layup. Initially, dried fibers in the form of woven or bond fabrics are manually introduced to the mold, and the resin matrix is applied to the reinforcing material with a brush. Such composite materials could be used in aerospace, automobile, and industrial sectors as well as for domestic appliances.

2 Experimental procedure

2.1 Materials

Metro Composites, Chennai, India, provided a commercially available vinyl ester with a viscosity of 350cps at room temperature. As accelerators, catalysts, and promoters, N-dimethylaniline, methyl ethyl ketone peroxide, and cobalt naphthenate are used. Metro Composites, Chennai, India, also supplies 220GSM (Grams per Square Meter (g/m^2)) sisal fiber with a satin weaving pattern as a reinforcing element. For filler content, 1–3-μm biochar particles with a density of 0.98gm/cc made from locally available corn stover. Sigma-Aldrich sells 3-aminopropyltrimethoxysilane (APTMS) for surface modification.

2.2 Biochar preparation

Figure 1 depicts the step-by-step synthesis of corn stover biochar. The slow pyrolysis (bio-carbonization) technique of charcoal manufacture converts corn stover waste into

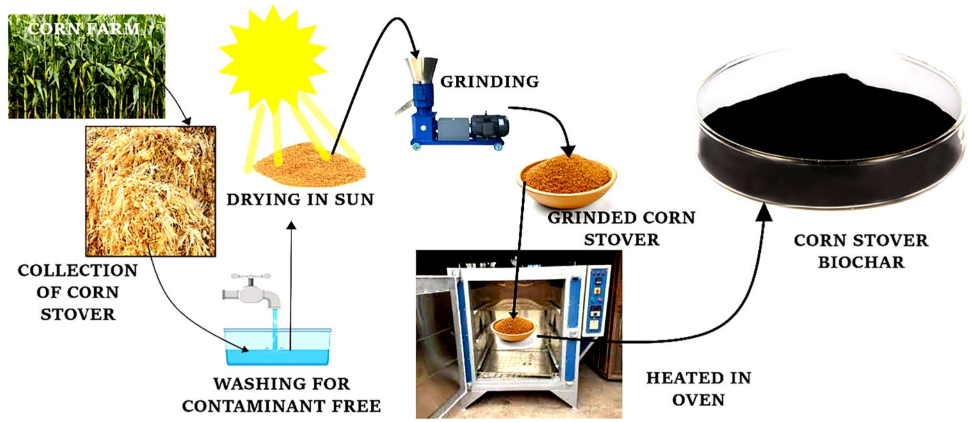

Fig. 1 Corn stover biochar preparation

biochar. To begin, raw corn stover waste was continuously washed in water for 2 h to eliminate any contaminants from the stover. To remove any remaining moisture, the stover was washed and dried in the sun for 48 h. The cleaned corn stover was then passed through the pyrolysis process [15]. The three-stage procedure is carried out with the help of multi-mode, specially designed pyrolysis equipment. The feedstock was first lit with kerosene oil, but as it began to burn, the supply was cut off. The first phase entailed completing the pre-pyrolysis procedure in a controlled air environment (nitrogen 0.32 l/s) for roughly 10 min at temperatures ranging from room temperature (RT) to 200°C. The incoming moisture and light volatiles were eliminated during this phase. In the second stage, which ran from 200 to 500°C, the hemicellulose and cellulosic components of the biomass were quickly destroyed. This stage takes roughly 30 min to complete [16]. The temperature was then raised to 500–800°C to break down the char component and free it from lignin and other organic debris with stronger chemical bonds. This stage took roughly 20 min to complete. All stages were heated at a rate of 50K/min for a total residence duration of 60 min. The thermally damaged biochar is separated at the end of the operation using fine sieves. The average particle size was determined using a particle size analyzer to be 1–3μm (having pore size 10–15 nm). As a consequence, the biomass of corn stover was transformed into fine-sized biochar [17].

2.3 Surface modification for fibers

The surface of sisal fiber with a satin weave was treated individually using silane treatment. At the first stage in making the silane solution, 95% ethanol is placed in a glass beaker and well agitated before adding 5% distilled water. Second, to regularize the pH of the resultant solution, which has a pH range of 4.5 to 5.5, acetic acid is added. Third, the silane drops must be progressively added to the ethanol-water solution until saturation is achieved. Fourth, the solution was then thoroughly mixed by stirring to dissolve all of the silanol groups and remove the methoxy group from the silane material. Finally, after 10 min of soaking, the fiber and biochar are removed from the silane solution to form a Si-O-Si structure [18].

2.4 Composite preparation

The layup surface must be ready as part of the mold preparation process prior to the fabrication of the composite, and wax is then coated on the cleaned surface to assist demolding laminate. If any edge gaps are found, silicon-rubber is enclosed around the perimeter of the laminate to designate the working area. Following that, a precise volume of vinyl ester resin is collected in a cleaned glass beaker to which corn stover biochar has been added [19]. The accelerator dimethylaniline (1.5%), catalyst methyl ethyl ketone peroxide (1.5%), and promoter cobalt naphthenate (1.5%) are combined with vinyl ester resin and corn stover biochar combination. The layers of satin weaved sisal fiber are placed on the mold, and the resin mixture is poured on top of each layer of fiber in a progressive manner. The hand layup technique is used to incorporate the resin into the fiber layup. Because of the slower polymerization at room temperature induced by the resin mixture ratio, the cure duration at room temperature is 24 h. A composite with a thickness of up to 3mm was created using satin weaved sisal fiber and vinyl ester with corn stover biochar [20]. Numerous laminates were made utilizing the hand layup technique and the components are listed in Table 1.

3 Characterization of composite

Visual inspection is performed on the vinyl ester–based corn stover biochar satin woven sisal fiber composite to detect any recognizable surface flaws. In accordance with ASTM requirements, three test specimens are drowned out of the

Table 1 Various combinations for different composite designation

Composite designation	Resin (vol.%)	Fiber (vol.%)	Biochar (vol.%)
V	100	-	-
VS	65	35	-
VS1	64	35	1
VS2	62	35	3
VS3	60	35	5

laminate using abrasive water-jet equipment from Maxiem water jets 1515, KENT, USA. The machine parameters were 220-psi maximum jet pressure, 0.3g/s abrasive flow rate, 1.1-mm nozzle diameter, and 3-mm SOD throughout the processes. Surface flaws on the specimen faces were eliminated by polishing them using P320 grit emery paper [21]. Table 2 was used to evaluate the mechanical wear, DMA, fatigue, and hydrophobic properties of the composites.

4 Result and discussion

4.1 Mechanical properties

Figure 2 represents the mechanical performance of corn stover biochar with satin weaved sisal fiber–reinforced vinyl ester composite. Figure 2 illustrates (a) the tensile behavioral traits, (b) the flexural properties, and (c) the Izod impact values. The composite designation "V" has tensile strength, flexural strength, and Izod impact values of roughly 61MPa, 1672MPa, and 0.41 J, respectively. The explanation for these lower results is the pure resin content of the composite designation "V," which is usually highly brittle and exhibits flat cracks throughout the surface [22]. Furthermore, including 35 vol.% satin weaved sisal fiber in pure vinyl ester resin improves the mechanical performance of the composite designation "VS." Tensile strength, flexural strength, and Izod impact values for composite designation "VS" enhanced to 96MPa, 2947MPa, and 3.86J, respectively. This large improvement was achieved by using silane-treated satin sisal fibers in vinyl ester resin, which improved the adhesive bonding between the fiber and the matrix [23]. As well as sisal fiber has strong wetting ability with matrix hence improved mechanical strength is observed. Similarly, corn stover biochar particle insertion of 1.0, 3.0, and 5.0 vol.% increased mechanical properties for composites designation "VS1," "VS2," and "VS3." But maximum values of mechanical properties were observed for composite designation VS2 with 3.0 vol.% of biochar about 148 MPa, 4519 MPa, and 4.98 J for tensile strength, flexural strength, and Izod impact respectively. Biochar particles were evenly dispersed in matrix and improved bonding is observed due to pores structures of biochar particles. These pore structures infiltrate the resin through it and enlace the bonding mechanism [24]. But it is observed that further increase in biochar vol.% up to the 5.0 vol.% composite designation VS3 shows decreased values for tensile strength, flexural strength, and Izod impact (134 MPa, 4372 MPa, and 4.74 J correspondingly). This decrease in values is a result of the cluster formation of biosilica particles, which provides composite's a brittleness and causes a failure.

4.2 Dynamic mechanical analysis

The DMA performance of corn stover biochar with satin-reinforced vinyl ester composites is shown in Fig. 3. The composite designation "V" has the lowest storage modulus characteristic, which is about 2.1 GPa. When the free volume grows with temperature, vibration also rises, showing that the matrix molecules rotate quickly and have a low storage modulus. The loss tangent for pure vinyl ester resin similarly shows lower values because the molecules cannot keep the energy delivered as stress while rotating at high temperatures and frequency [25]. The storage modulus appears to be 3.5 GPa when 35 vol.% satin weaved sisal fibers were

Table 2 ASTM standards with machine specification

Sr. no.	Tests	ASTM standards	Machines used
1	Tensile	D3039	INSTRON 4855, UK Traverse speed of 1.1 mm/s
2	Flexural	D790-17	
3	Izod impact	D256–10	Krystal equipment Ltd., India Maximum load capacity of 20 J
4	DMA	D4065	Dual cantilever mounted DMA analyzer with sweep mode
5	Wear properties	G99–17	Du-com instruments Pvt. Ltd., India Pin-on-disc setup
6	Fatigue	D3479	MTS Landmark 370 load frame, USA Working stress values are 30 %, 60 % and 90 % of UTS frequency of 5 Hz, stress ratio of 0.1 and temperature of 28 °C
7	SEM	-	HITACHI, S-1500, JAPAN

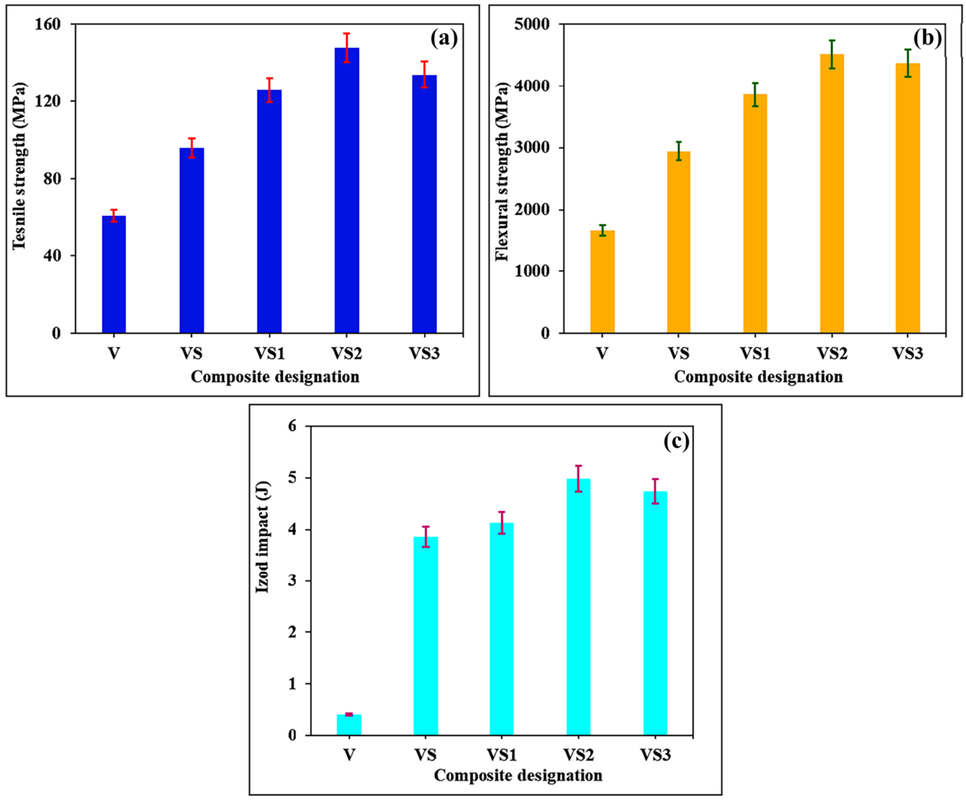

Fig. 2 Mechanical properties for various composite designation

added to pure vinyl ester resin. The layer-sequence stacking of fibers into vinyl ester resin results in an increase in storage modulus. The free space in the matrix, surrounded by fiber volume fraction, increases the storage modulus due to the surface-treated fiber bonding mechanism preventing the rotation of the matrix molecules. Comparable improvements in storage modulus values and a decrease in loss factor are seen when thermally reduced corn stover biochar was incorporated in composite by 1.0, 3.0, and 5.0 vol.%. However, with inclusion of 5.0 vol.% of biochar particles, the composite bearing the name VS3 was found to have the highest storage modulus and the lowest loss factor. This is because the addition of biochar strengthens the interaction between the fiber and the resin's secondary molecules. Hence, the inertia to rotate about the primary chains is rapidly increased due to the added biochar and more heat energy is required to activate the same [26].

4.3 Wear properties

Due to the pure vinyl ester resin as the main content element of the specimen, which exposes its surface in a large area to the wear disc, the composite designation "V" exhibits a higher wear rate of roughly 0.48 COF and sp. wear rate of 0.022 mm^3/Nm (Fig. 4). But the inclusion of silane-treated satin weaved sisal fibers enhances the wear resistances up to 0.41COF and a specific wear rate of 0.018 mm^3/Nm. The insertion of fiber decreases the contact angle of the matrix

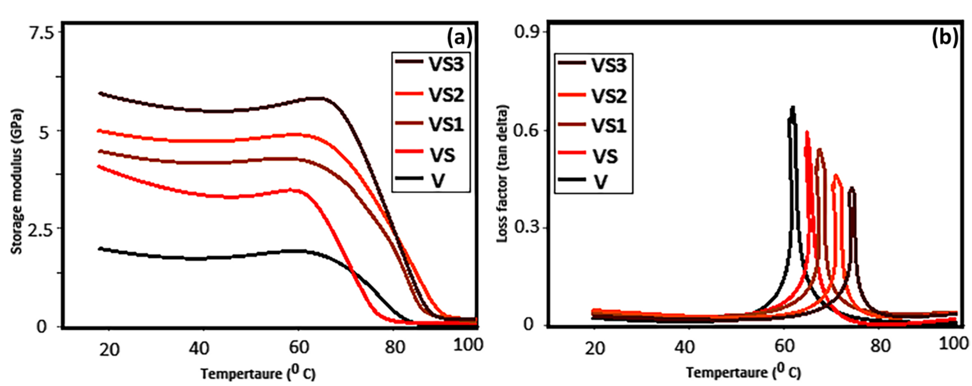

Fig. 3 DMA for various composite designations

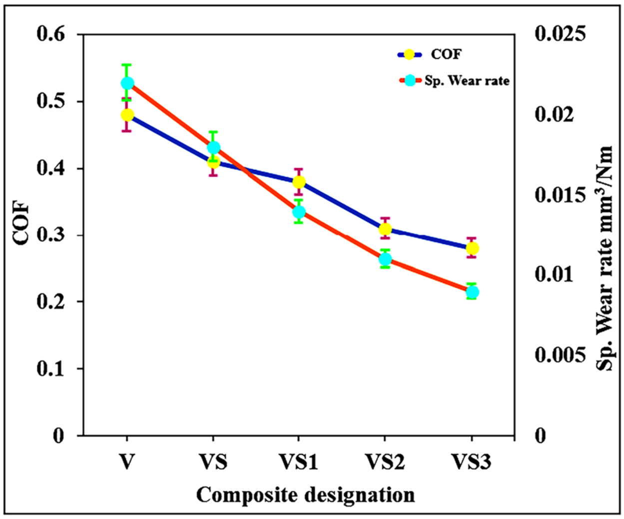

Fig. 4 Wear properties for various composite designation

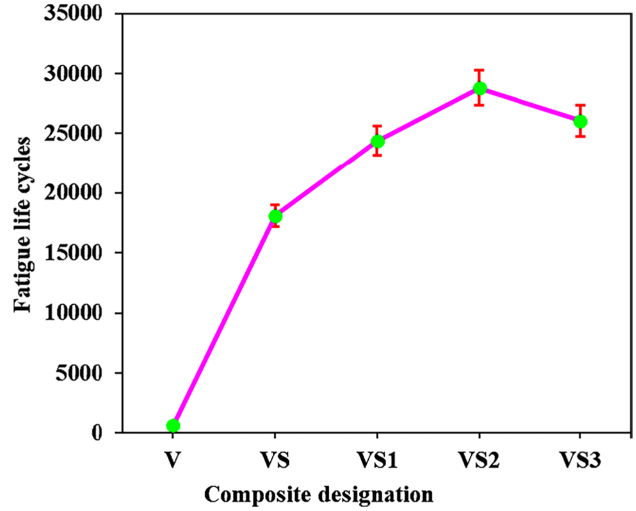

Fig. 5 Fatigue behavior for various composite designation

to the wear disc and enhances the resistances against abrasion [27]. Furthermore, with the incorporation of silane-treated biochar particles by 1.0, 3.0, and 5.0 vol.%, the wear resistance of composite designations VS1, VS2, and VS3 steadily rises. This increase is due to silane-treated biochar particles dispersed evenly in the matrix. The best values for this property were 0.28 COF at 0.009mm^3/Nm for the composite designation VS3 with 5.0 vol.% biochar particles and 35% satin weaved sisal fiber. It is due to the lubricant packet formation which occurs when biochar comes in contact with wear disc [28].

4.4 Fatigue behavior

Figure 5 demonstrates the fatigue behavior of corn stover biochar satin weaved sisal fiber–reinforced vinyl ester composites. At 50% of UTS, which is around 624 cycles, the composite designation "V" provides the lowest fatigue life numbers. Figure 5 a depicts the greater brittleness of pure vinyl ester resin as a result of the decreased fatigue life count with flat brittle fracture. The justification for this is the preservation of plastic strain in the vinyl ester molecular chain [29]. Additionally, the inclusion of 35% sisal fiber increases the fatigue life counts for composite designation "VS" by around 18,118 cycles. This phenomenon could be due to the silane surface treatment on satin weaved sisal fiber improving chemical bonding and mechanical interlocking between the treated fibers and matrix. Similarly, biochar particle insertion of 1.0, 3.0, and 5.0 vol.% increased fatigue life counts such as 24,385, 28,813, and 26,094 cycles with respect to composite designations "VS1," "VS2," and "VS3." This improvement is because of the improved dispersion and adhesion of particle in matrix, which reduce the crack propagation [30].

4.5 Contact angle

Figure 6 shows the contact angle between composite materials and water drop. The pure vinyl ester resin has a greater contact angle of around 86°. This matrix material repeals the OH molecules and gives highest contact angle. Further incorporation of sisal fiber by 35vol.% reduces the contact angle up to 82°. Natural fiber has tendency to attract water molecules; hence, this increment in water absorption and reduction in contact angle is observed for composite designation "VS." Moreover, adding 1.0, 3.0, and 5.0 vol.% biochar particles decreases the contact angle between composite materials and water molecules to 77°, 74°, and 71° for composite designations VS1, VS2, and VS3, respectively. The biochar particles also easily bind the OH molecules,

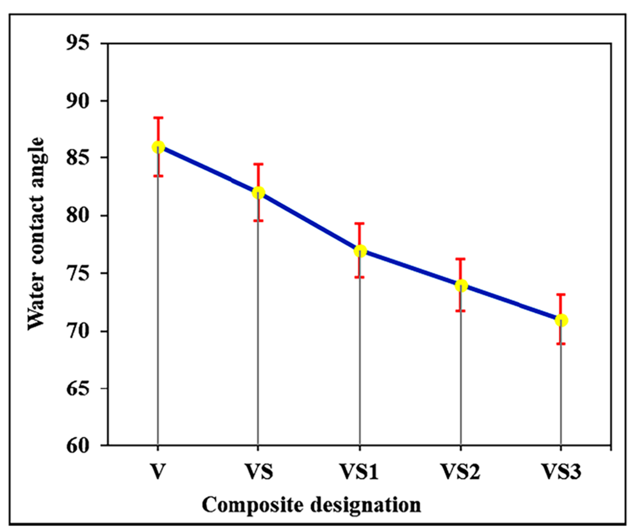

Fig. 6 Water contact angle for composite designation

although silane treatment limits this to some amount; thus, the measured contact angles are only similar to pure vinyl ester [31].

4.6 SEM fractography

Figure 7 demonstrates the scanning electron microscopy for various fractured composites. Figure 7 a shows the flat fracture with brittle surface for the composite designation V. The absence of reinforcements and the pure epoxy contents of the composite designation V are the major cause of such fracture. Improved adhesive bonding between the fiber and matrix is demonstrated in Fig. 7 b and c for composite designations VS and VS1, respectively. This is a result of the Si-O-Si structure obtained by the surface treatment on satin weaved sisal fiber making strong bonding [32]. Figure 7 d shows a strong bonding process with a highly reactive fiber phase in composites. This is because the inclusion of biochar and its even dispersion throughout the matrix improved the adhesion property for composite designation VS2. Figure 7 e shows the enhanced bonding mechanism for composite designation VS3 due the pore structure of biochar, which

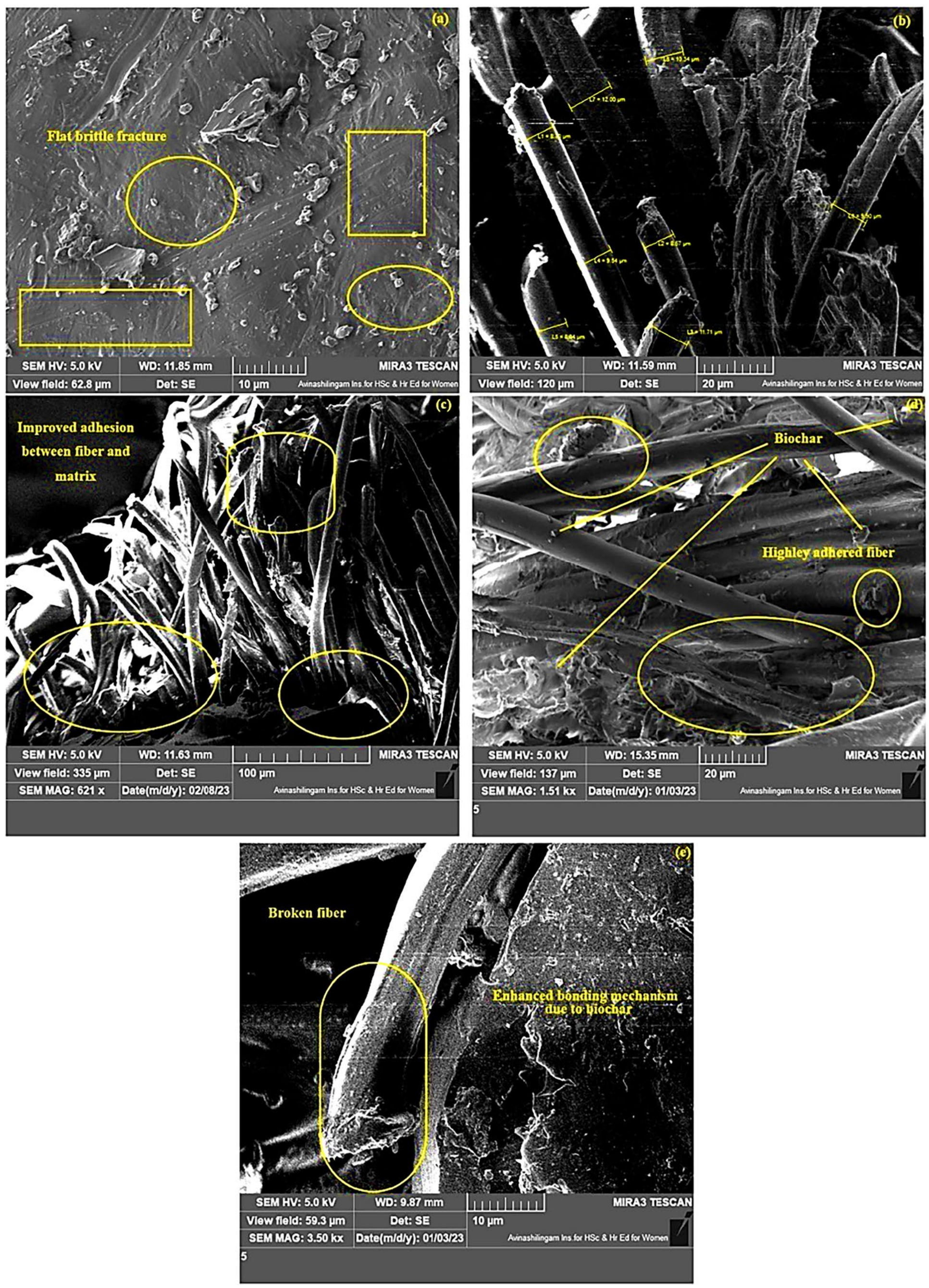

Fig. 7 SEM fractography for fractured samples

infiltrate resin through it and improved mechanical interlocking [2, 33].

5 Conclusions

The analysis and fabrication of thermally reduced corn stover biochar with the satin weaved sisal-reinforced vinyl ester composites is the aim of this study.The mechanical, wear, DMA, fatigue, and hydrophobic properties have been characterized according to ASTM standards for these composite laminates. After surface treatment on reinforcements by hand layup process, composites were fabricated from corn stover biochar and satin weaved with vinyl ester. The outcomes of this study were followed as follows: maximum values of mechanical properties were observed for composite designation VS2 with 3.0 vol.% of biochar about 148 MPa, 4519 MPa, and 4.98 J for tensile strength, flexural strength, and Izod impact respectively. But it is observed that further increase in biochar vol.% up to the 5.0 vol.% composite designation VS3 shows decreased values for tensile strength, flexural strength, and Izod impact (134 MPa, 4372 MPa, and 4.74 J correspondingly). However, with inclusion of 5.0 vol.% of biochar particles, the composite bearing the name VS3 was found to have the highest storage modulus and the lowest loss factor. The best values for wear properties were 0.28 COF at 0.009mm^3/Nm for the composite designation VS3 noted by addition of 5.0 vol.% biochar particles and 35% satin weaved sisal fiber. On the other hand, biochar particle insertion by 1.0, 3.0, and 5.0 vol.% increased fatigue life counts such as 24,385, 28,813, and 26,094 with respect to composite designations “VS1,” “VS2,” and “VS3.” The highest observed contact angle is about 71° for composite designation VS3 which indicates the hydrophobic nature of composite material. SEM fractography shows the improved adhesion between fiber and matrix due to the surface treatment on reinforcing materials, as well as because of the silane surface treatment corn stover biochar distributed evenly within the matrix.

Author contribution P. Sivamurugan, M. Mareeswaran—concept design and experiment. S. A. Muhammed Abraar, Savita Verma—concept design, experiment, and testing. Neha Verma, Bipin Kumar Srivastava—concept design, experiment, and funding. D. Vinay Kumar and I. S. Chakrapani—concept design. B. Ramesh—correspondence.

Data availability Nil

Declarations

Ethical statement NA

Competing interests The authors declare no competing interests.

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