Broomcorn Stalk Fiber in Nonwoven Reinforced Polylactic Acid Matrix Composites

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The broomcorn plant (*Sorghum bicolor* (L.) Moench) is one of the main cereal crops and its grains are used in food and feed sectors while its stems are used in broom production and as a building material. Polylactic acid (PLA) is a biobased polymer that is widely used as a matrix material in natural fiber-reinforced composites. In this study, the aim is to use broomcorn plant stems, which are agricultural waste, as reinforcement in composite production. For this purpose, fiber was obtained by purifying broomcorn plant stems from woody cells with enzyme and NaOH. To easily comb the fibers, 10 wt% cotton was added and blended and turned into nonwoven fabric *via* needle punching. Then, PLA was combined with the matrix using the hot press method to produce single and double-layered composites. To characterize the broomcorn fiber reinforced composite material, strength, elongation, scanning electron microscopy, and Fourier transform infrared instrumental methods were used within the standards. According to the analysis results, broomcorn fiber has a high potential as a new reinforcement material suitable for composite production.

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INTRODUCTION

The majority of polymer materials used in daily life are derived from petroleum and are produced using environmentally harmful methods. A new trend in material science is using biocomposite materials reinforced with plant fibers, where natural components as raw materials are converted into products using clean production methods (Ağırgan *et al.* 2022). Plant fibers are replacing synthetic fibers as environmentally friendly renewable reinforcement materials in order to develop biopolymer composites (Aaliya *et al.* 2021). The ideal procedure for designing these composites is to use a bio-based polymeric matrix together with renewable and environmentally friendly plant-based lignocellulosic fibers (Abhiram *et al.* 2021).

Vegetable fibers are classified according to the plant region from which they are obtained, such as seed (cotton, kapok), stem or stalk (flax, jute), leaf (sisal, banana), fruit (coco fiber), or grass and reed (bamboo) (Ighalo *et al.* 2021). Sorghum (*Sorghum bicolor* (L.) Moench var. *technicum* (Körn.), commonly known as sweet sorghum, is a species from the Poaceae family. It is characterized by long, fibrous, flexible, and sturdy panicles, and only the stem of the plant is used in broom production. This plant, which originates from Central Africa, is mostly cultivated in Mediterranean countries (Balkan 2008). Agriculture uses 38% of the global land area (FAO Reports 2024). Broomcorn stalk is a stalk type like wheat and rice. It is the most cultivated plant among the C4 energy plants that are slowly becoming widespread in the world (Öktem *et al.* 2021; Sadighfard and Geren 2022). It is a plant that needs a growth period of 90 to 140 days to mature and has a very effective root system; therefore, it resists drought, has good adaptability, and has a high biomass yield (Koppen *et al.* 2009).

Cellulose and lignin are the chemical components of plant fibers. Cellulose is the primary component of bast fibers, constituting 60 to 75% of plant cell walls, and it imparts properties such as strength and stability to the fibers (Kiruthika 2017). Natural fibres contain cellulose, hemicelluloses, and lignin. In order to consider the behaviour of agrifibres, it is necessary to take into account the structures of each of the major components present. Hemicellulose, second to cellulose in abundance, differs greatly from cellulose. The molecules are shorter and are built up of different heteroglycan sugar units, depending upon the species from which they are obtained. Hemicellulose constitutes 25 to 40% of woody cells (Fan and Fu 2017).

Lignin is considered a binding agent responsible for holding the cellulose content together (Li *et al.* 2020). When the hydrophobic lignin substance is removed from the fiber, the cellulosic structure remains.

A minor portion of the cellulose is present as a non-crystalline structure composed of microfibers, which are bonded by hydrogen bonds along the fiber and form macro fibers. (Ighalo *et al.* 2021). Due to low interfacial bonding, some polymer composites that are reinforced with natural fibers have low strength values. However, they are biodegradable and cheaper than synthetic fibers (Staiger and Tucker 2008). The interfacial bonding created by controlling the moisture absorption of the fibers or matrix increases the strength of the composite material. A good interfacial interaction between the fiber and the matrix helps to strengthen the interfacial bond between the fibers and the matrix by pre-treatment (Kozłowski 2012). During some pretreatment steps, non-cellulosic components and impurities as by-products of the chemical reaction are removed, providing a better load transfer capacity at the fiber-matrix interface (Syduzzaman *et al.* 2020). Vasques *et al.* (2016) investigated the effect of concentration amount and time on the mechanical properties of broom handle fibers by applying NaOH treatment. They concluded that tensile strength values were inversely proportional to the alkali concentration applied to the fiber and directly proportional to the reaction time, which makes a reinforcement material highly promising for composite material production.

Polylactide or polylactic acid (PLA) is a leader in the bioplastic market with its demand and affordable production costs. It is an aliphatic (straight-chain) polyester produced from lactic acid and is obtained from corn. The PLA is a thermoplastic material with hardness and transparency in polyester structure with different areas of use such as flexible and rigid film packaging, injection molding, and extrusion coating. The processing, crystallization, and degradation properties of PLA, the composition and structure of the polymer chains, depend on the L- and D-isomer ratio of lactic acid. This stereochemical structure of PLA can be changed by adding L-lactide and meso-, or D-lactide mixtures with a copolymerization reaction, resulting in a high molecular weight amorphous or semi-crystalline polymers with a melting point in the range of 130 to 185 °C. The PLLA homopolymer shows a semi-crystalline structure (Ağırgan 2013). In the literature, natural fiber reinforcement composites have been used as matrices along with reinforcement materials containing many natural cellulosic fibers such as abaca, bamboo, banana, flax, sisal, cotton, rice straw, bagasse, coir, ramie, jute, coconut, pineapple, hemp, wheat straw, *etc*. (Rajeshkumar *et al.* 2021).

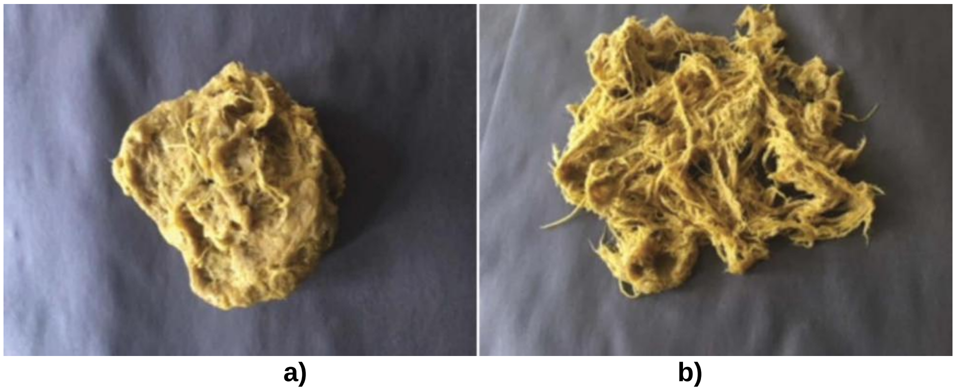
The sorghum plant is used in many areas such as grain, silage, green and dry feed, broom, and wall covering. In recent years, the widespread use of vacuum cleaners in workplaces and homes and the emergence of brooms made of nylon fibers have led to the atrophy of the broom making craft and the decrease in sorghum cultivation. Today, broom making is done in very small numbers. Composite materials are generally called high value-added products because they are used in places that require high levels of material performance. This study introduces a new value-added product to the literature and presents a potential alternative to current materials utilized in various fields, including machine components, vehicle bodies, equipment, sports equipment, and the furniture industry. The objective of the research is to develop composite materials by reinforcing sorghum plant stems with a polylactic acid (PLA) matrix.

EXPERIMENTAL

Materials

Sorghum (*Sorghum bicolor* (L.) Moench) stalks used in the study were purchased from commercially grown products used for broom production in the Uzunköprü district of Edirne province (Turkey). Broom stalks fibers (BSF) were treated at room temperature for 15 days in a 70 × 50 cm^2 polypropylene-based bathtub using 20% NaOH, 2% Bioprep 3000 L enzyme and 2% wetting agent at a 1:10 bath ratio to separate woody cells (Avecilla-Ramírez *et al.* 2020; Agirgan and Taskin 2020). Then, as shown in Fig. 1, the remaining alkali on the surface was removed using nonionic detergent and softener at 40 °C for 90 min.

Fig. 1. Enzymatic treatment



a) b)

Fig. 2. a) Flushing process, and b) Drying

Detergent and softening wash were applied to BSF (Fig. 2). The purpose of this process was to facilitate the combing of broom plant fibers, which possess a rigid and brittle structure when dry. Broomcorn stalk fibers were cut into 5 to 10 cm lengths and blended with 10% cotton in a laboratory type carding machine to obtain carding band (Ağırgan *et al.* 2022). Figure 3 shows the laboratory type carding machine (Mesdan SpA, Puegnago del Garda, Italy) and carding belt used to card broom stalk fibers (Ağırgan *et al.* 2020).

Fig. 3. Laboratory-type carding machine and carding band made of broomcorn stalk fibers

Prepared BSF card bands were used in a needle punching machine (Dilo Machines GmbH, Eberbach, Germany) using a 36 gauge Triangular 3 type needle. The punching speed was set at 800 rpm, punch density was set at 25 punches/cm^2, punching depth was set at 3.5 mm, and feeding speed was set at 3 m/min to produce nonwoven fabric. A standard PLA polymer (Total Corbion LX175, Gorinchem, Netherlands) mentioned in Table 1 was used as the matrix material to produce composite materials. This polymer has other properties such as high viscosity, transparent resin, amorphous and is suitable for injection molding processes.

Table 1. Properties of Used Polymer

Material	Number	Density	Purity	T_m	T_g
Poly(L-Lactic Acid)	PLLA 101 LX 175	1.24 g/cm^3	96%	155 °C	55 °C

PLA sheet production

A hot hydraulic press (HÜRSAN Hydraulic hot press C type, Konya, Turkey) was used in the production of PLA sheet and biocomposite, and a chiller system was used to cool the molds.

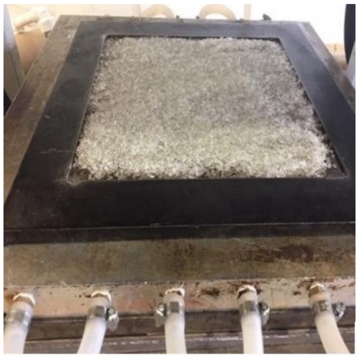


Fig. 4. Granule PLA in a steel mold

The system reached 200 °C in 25 min. About 430 g of PLA granules were poured in a 300 × 300 mm^2 steel mold and pressed with 90 bar pressure for 10 min. The PLA granules were cooled down to 55 °C (Fig. 4). At the end of the process, PLA sheets of 300 × 300 mm^2 , with 4-mm thickness were obtained (Ağırgan *et al* 2022).

Single-layer and double-layer BSF/PLA bio composite material production

During PLA /BSF nonwoven/PLA biocomposite production, the layers were placed in the steel mold overlapping each other. Single-layer BSF/PLA biocomposite material was produced by being pressed at 200 °C at 90 bar pressure for 10 min. The plates were placed in the steel press mold so that they overlapped in the order of PLA/BSFN/PLA/BSFN/PLA. The structure in a hot press machine at 200 °C at 90 bar pressure for 10 min.

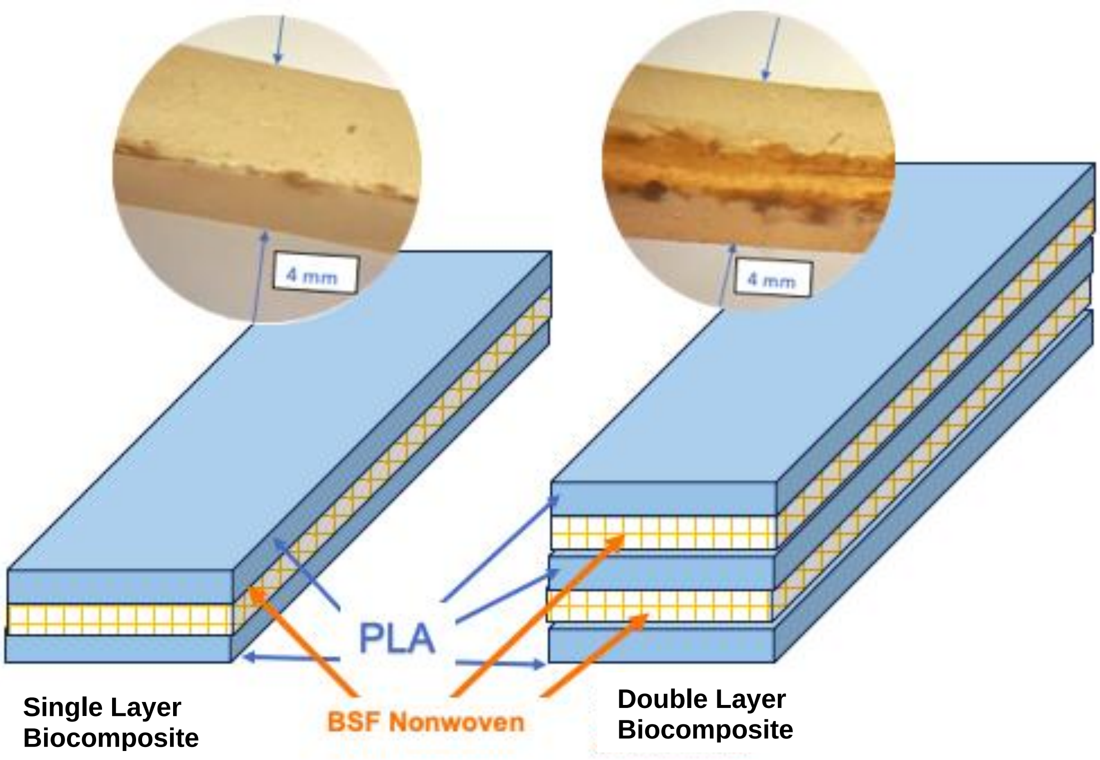


Fig. 5. Single and double-layer biocomposite

Cutting process

Produced biocomposite materials were cut with a water jet cutting machine according to ASTM D 638 (2005) and ASTM D790 (2000). Composite sheets produced in the size of 300 × 300 mm^2 were cut according to the needs of tests and analyses. These samples were then used for tensile, three-point bending testing, scanning electron microscopy (SEM), and Fourier transform infrared (FTIR) analyses.

Methods of Analyses

A scanning electron microscope (Carl Zeiss Microscopy GmbH, Jena, Germany), secondary electron (SE), and backscattered electron (BSE) detectors with a working range of 37× to 1,000,000× were used. The obtained signals provide the characteristic properties of the material, including crystal structure, chemical content, morphology, and orientation.

Fourier transform infrared analysis allows the recognition of the chemical structure of substance by showing the chemical bonds that constitute the structure of the substance, the bond vibrations of the molecules, or the changes in the rotational energy levels. A Spotlight 400 FTIR device (Perkin Elmer, Shelton, CT, USA) was used in the wavelength region between 4000 to 500 cm^{-1}.

Dumbbell-shaped test samples and Instron 4411 Strength Testing Machine (Instron, Bucks, UK) were used according to ASTM D 638 (2005) to test the tensile properties. The tensile test was performed by applying force to the test sample prepared according to the standards in a single axis direction and pulling it at a certain tensile speed and constant temperature with a stroke speed of 10 mm/min until the material broke.

The ASTM D790 (2000) standard test method was used to evaluate bending properties of reinforced and unreinforced plastics and electrical insulation materials. During the test, the material was supported at 2 points and a force was applied to the sample from the middle point. The force at which the permanent deformation in the sample begins or the sample breaks gives the resistance it shows against bending.

RESULTS AND DISCUSSION

SEM Analysis

Scanning electron microscopy was used to obtain images from the surface of the produced single-layer and double-layer composite materials. The images obtained from electron-material interactions provided information about the morphology of the composite materials.

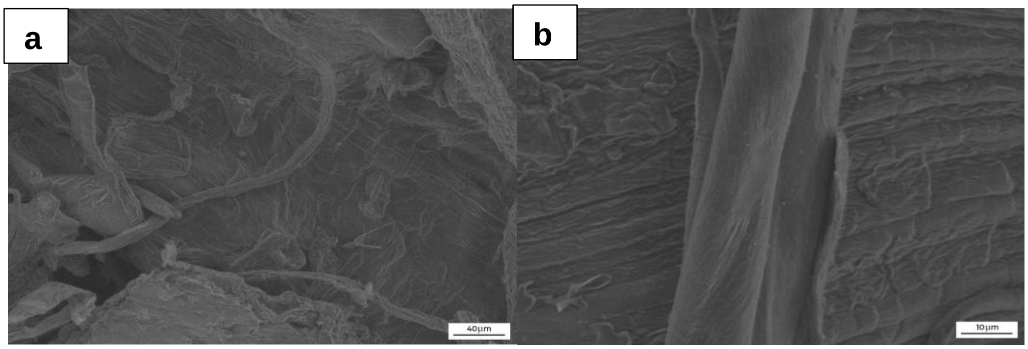


Fig. 6. Nonwoven broomcorn stalk fiber a) 40 µm b)10 µm

Broomcorn fibers contain hydrophobic and a difficult-to-process substance called lignin in their structure. Lignin is separated from the fiber by alkali pretreatment and thereby increases the roughness on the fiber surface. To evaluate the surface pore size of alkali lignin before and after enzyme treatment, the specific surface areas of the untreated and treated alkali lignin were measured using nitrogen adsorption *via* the BET method. The surface area of the treated alkali lignin was nearly five times higher than that of the untreated lignin, thereby confirming the effective degradation of alkali lignin through enzyme treatment (Zhang *et al.* 2020). After the treatment, the compatibility of the fibers with the PLA matrix increased, and the cellulose content reached up to 80%. The strength of the BSF and its bonding ability also increased at the interface. As a result of the process, a suitable environment was created for the hydrogen bonding of the cellulose molecules with the hydroxyl groups in the PLA matrix (Figs. 6a-b) (Li *et al.* 2020). In this study, alkali pretreated single-layer BSF was turned into a biocomposite by alkali pretreatment followed by thermal treatments with the PLA polymer matrix. This provided a good interface connection in the resulting biocomposite material (Figs. 7 a-b).

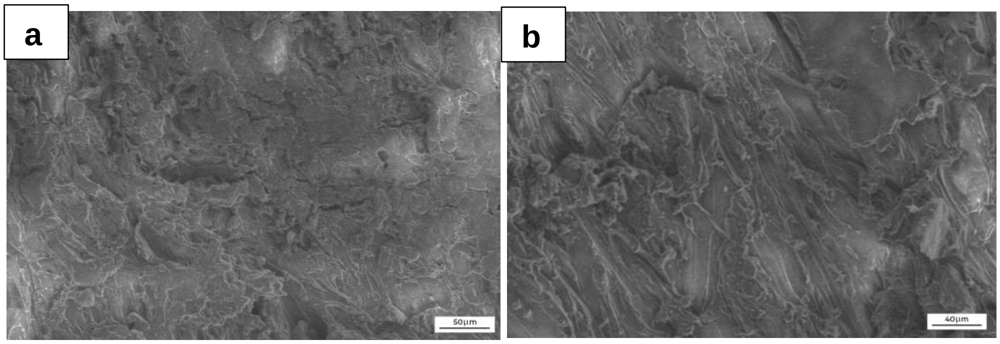


Fig. 7. SEM single-layer composite a) 50 µm b) 40 µm

In the SEM analysis of the double-layer BSF/PLA biocomposite material, it was seen that there was a good interaction between the fiber and matrix, and a good interfacial contact was achieved (Figs. 8a-b).

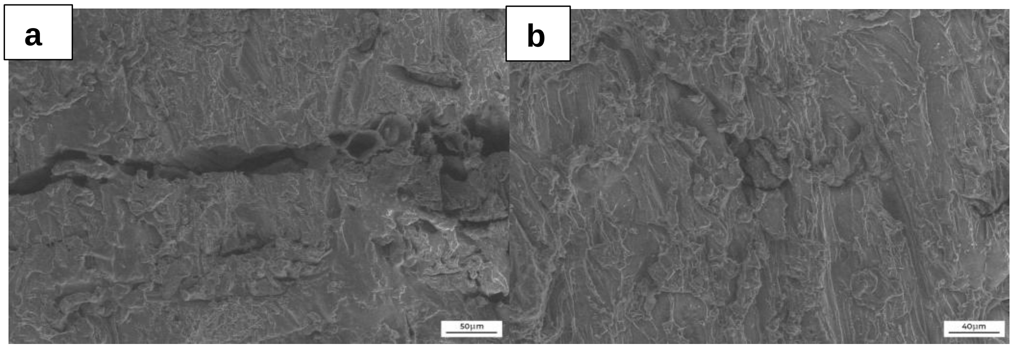


Fig. 8. SEM double-layer composite a) 50 µm b) 40 µm

FTIR Analysis

The nonwoven reinforcement material produced from BSF showed intense O-H stretching at 3323 cm^{-1} bands. The absorbance peak at 2920 cm^{-1} represented the antisymmetric stretching vibrations of CH_3– and –CH_2– groups in lignin. The intensity of this peak decreased significantly after enzymatic hydrolysis, indicating that lignin was degraded (Ohra-aho and Linnekoski 2015). The peak at 1032 cm^{-1} represented C–H vibrations of the guaiacyl unit. The decrease in the intensity of this peak indicated that the guaiacyl structure was disrupted, further indicating lignin degradation (Pandey and Pitman 2003). Based on previous studies and combined with the infrared spectra of alkaline lignin before and after enzymatic hydrolysis, the representative chemical bonds in alkaline lignin were disrupted, while the chemical bonds present in lignin degradation products increased significantly after enzymatic treatment (Zhang *et al.* 2020). A moderate C-O ether stretching band in PLA was observed at 1748 cm^{-1} and the band at 1032 cm^{-1} indicated the C=C vibrational olefinic stretching. The C-C bond peaks at 564 cm^{-1} were also observed in single and double-layer biocomposite materials (Fig. 9). The FTIR results showed that lignin in the fibers was removed by alkaline processes. This result is similar to previous studies (Mittal *et al.* 2016).

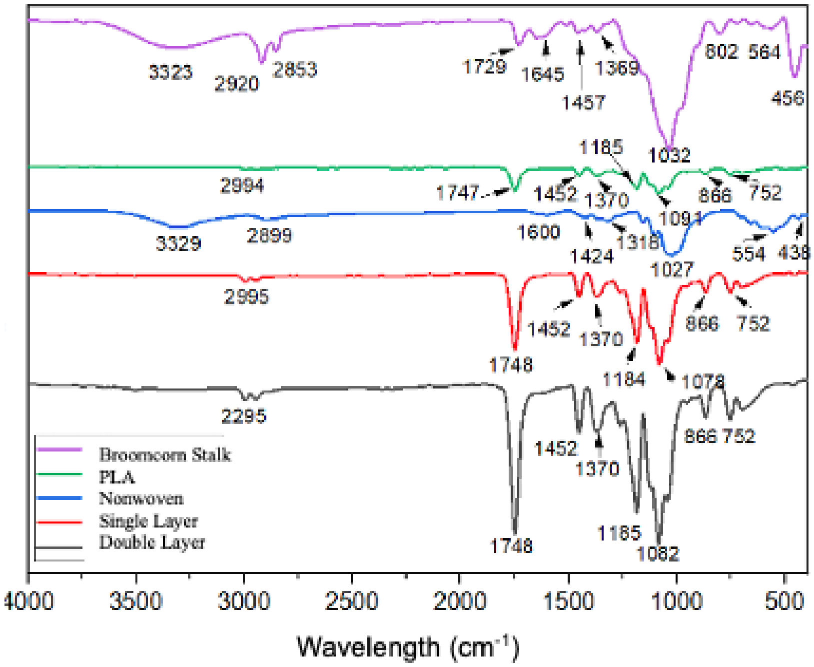


Fig. 9. FTIR graphs of BSF, non-woven, PLA, single, and double-layer biocomposites

Tensile Test

According to the tensile test results, the best value was obtained from the double-layer sample taken in the machine direction with 1820 N, 62.0 MPa, and 1.24% elongation value. The lowest value was given by PLA (cross direction) with 1180 N, 38.2 MPa, and 1.24% elongation value (Fig. 9). These values were compared with those from epoxy/jute (43 MPa), Epoxy/bamboo (392 MPa), and epoxy/cotton (72.9 MPa) tensile values obtained by Sathishkumar *et al.* (2020).

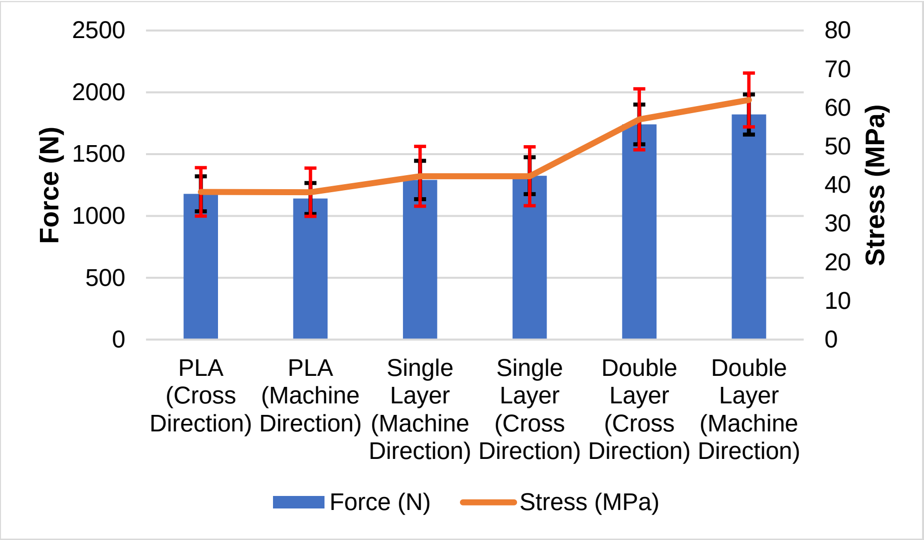


Fig. 10. Tensile test results

Therefore, the data in the literature support the results obtained in this study. In the tests of flax/PLA composite material made with 6% reinforcement, flax yarn/PLA, and nonwoven flax/PLA gave tensile values of 83.0 ± 5.0 MPa and 151.0 ± 7.0 MPa, and RS/PLA indicated 30.01 MPa, respectively (Akonda *et al.* 2018; Ağırgan *et al.* 2022). Increasing the rice fiber content of the nonwoven reinforced composite material to 10% yielded a decrease in tensile strength (Fahim *et al.* 2012).

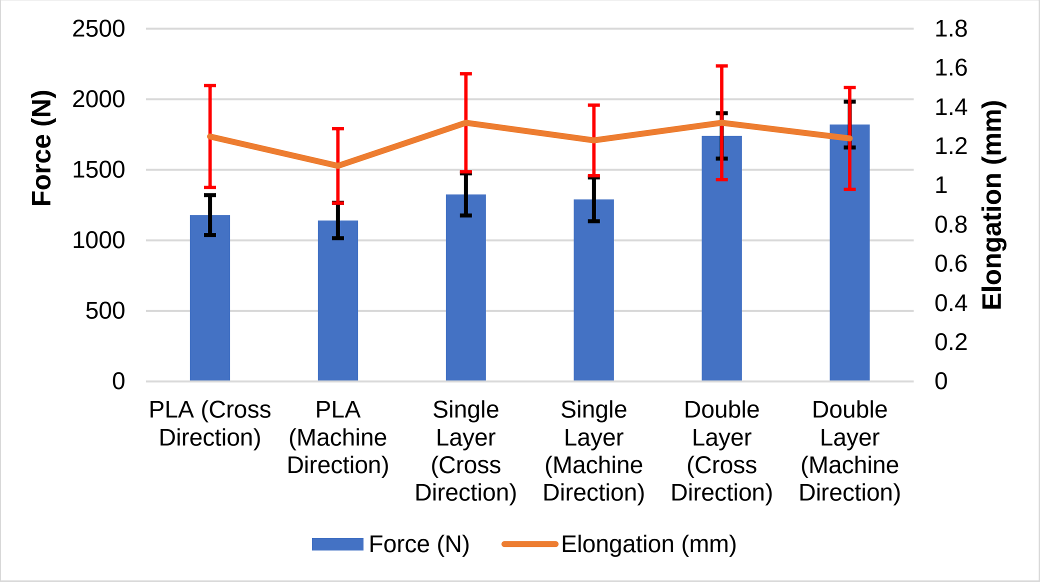


Fig. 11. Tensile test result and force-elongation values

Figure 11 shows the applied force and % elongation values at the moment of rupture as a result of the Tensile Test. Observed that the double-layer composite material (cross direction) gave the highest elongation value with 1.32 mm. Later, as the number of layers decreased, the strength and elongation values became 1.23 and 1.1 in the single-layer composite material and PLA structure, respectively.

Three-point Bend Test

The samples were prepared in the form of bars with dimensions of 100 × 10 × 4 mm^3. Three-point bend tests were applied to a total of 10 samples, 5 each in the machine direction and 5 in the opposite cross direction. The arithmetic average of the results is given in Fig. 12.

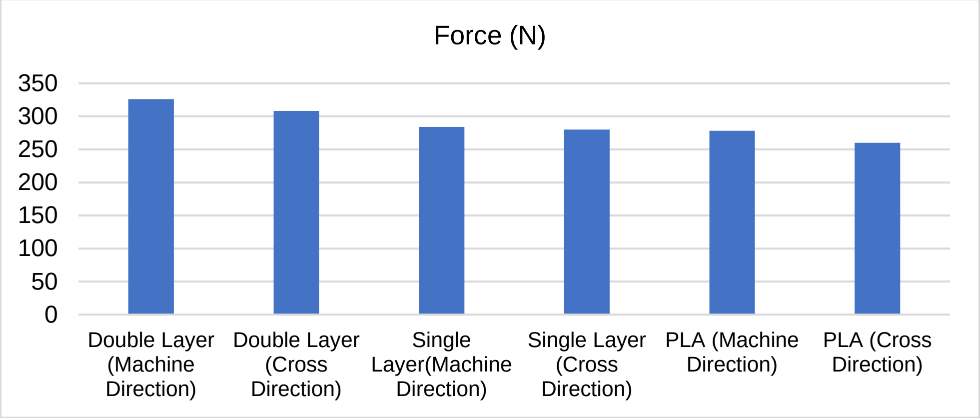


Fig. 12. Three-point bending test results

In the three-point bending test results, the highest result was seen in the double-layer in machine direction with a value of 326 N, and the lowest result was seen in the PLA in cross direction sample with 260 N. In a previous study, the following values were obtained in the three-point bending test, epoxy/jute 55.8 MPa, epoxy/bamboo 226 MPa, epoxy/cotton at 82 MPa, and RS/PLA 222 N (Sathishkumar *et al* 2020; Ağırgan *et al.* 2022).

In future studies, the strength performance of the composite might be increased by adding different reinforcing materials to the interfacial gaps and connections to improve the mechanical properties.

CONCLUSIONS

1. The woody cells on the fiber surface were reduced to a certain extent with the help of an alkali-enzyme combination, as a result of which hemicellulose and lignin-like substances were removed from the structure, and the fibrillation surface roughness formed on the fiber surface was increased. After the pretreatment process, 78% fiber was obtained by weight.
2. The fiber matrix structure was revealed in scanning electron microscope images and Fourier transform infrared (FTIR) spectra showed that the hydrophilization process was successful.
3. Although the strength values of the produced broomstalk stem fiber (BSF) nonwoven fabric were low for comparison, when reinforced with poly(lactic acid) (PLA) matrix in single and double layers, the test results showed that the tensile strength increased, depending on the number of layers.
4. According to the three-point bending test results, when the single layer and double layer materials are compared with each other, an increase in the strength value was observed with the increase in the number of layers.

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