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Mechanical Properties of LLDPE Composites Reinforced with Woven Horsehair Fabrics

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ABSTRACT

This study aims to enhance the mechanical properties of linear low-density polyethylene (LLDPE) based composites by utilizing fabrics woven from horse tail hair as reinforcement. The warp and weft thread comprised bundles containing 20 hairs each in fabric production. The warp density was kept constant while two different weft densities, 5 bundles/cm and 7 bundles/cm, were used. Plain and 2/1 twill were selected as the weave type. All fabrics, except for the reference sample, were coated with ethylene vinyl acetate (EVA). The coating increased the tensile strength of the composite in the weft direction by 11.69% compared to untreated fabrics. As the fabric's weft density increased, the tensile and flexural strength of the composites in the weft direction improved. A slight decrease in tensile strength was observed in the composites with the 2/1 twill weaving structure compared to the plain weaving, while an increase in flexural strength was noted with the plain weaving structure.

At Kıllı Dokuma Kumaşlar ile Takviye Edilmiş LLDPE Kompozitlerin Mekanik Özellikleri

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Anahtar Kelimeler
At kılı
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Doğrusal düşük yoğunluklu polietilen

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ÖZ

Bu çalışma, takviye malzemesi olarak at kuyruğu kılından dokunmuş kumaşlar kullanarak doğrusal düşük yoğunluklu polietilen (LLDPE) bazlı kompozitlerin mekanik özelliklerini iyileştirmeyi amaçlamaktadır. Kumaş üretiminde çözgü ve atkı iplikleri olarak her biri 20 kıldan oluşan demetler kullanılmıştır. Çözgü sıklığı sabit tutulurken, iki farklı atkı sıklığı (5 demet/cm ve 7 demet/cm) kullanılmıştır. Dokuma tipi olarak 2/1 dimi ve düz dokuma seçilmiştir. Referans numune hariç tüm kumaşlar etilen vinil asetat (EVA) ile kaplanmıştır. Kaplama işlemi, işlenmemiş kumaşlara kıyasla kompozitin atkı yönündeki çekme dayanımını %11,69 oranında artırmıştır. Kumaşın atkı sıklığı arttıkça, kompozitlerin atkı yönündeki çekme ve eğilme dayanımları iyileşmiştir. 2/1 dimi dokuma yapısına sahip kompozitlerde, düz dokumaya kıyasla çekme dayanımında hafif bir azalma gözlemlenirken, eğilme dayanımında ise düz dokuma yapısıyla artış kaydedilmiştir.

1. INTRODUCTION

Fiber-reinforced polymer composites have attracted significant interest across various industries due to their excellent mechanical properties, low weight, and resistance to environmental degradation. Glass, carbon, and aramid fibers are extensively utilized as reinforcement materials in fiber-reinforced polymer (FRP) composites due to their exceptional mechanical properties and have long been applied in the aerospace, automotive, and marine industries [1,2]. The increasing awareness of environmental issues associated with synthetic materials has catalyzed a significant shift towards utilizing natural fibers in composite production. Natural fibers offer ecological advantages over synthetic fibers, such as biodegradability, renewability, and recyclability, as well as lower density, high flexibility, high impact resistance, comparable specific tensile properties, non-abrasiveness to equipment, and reduced energy consumption [2,3]. Natural fibers are categorized into plant-based, animal-based, and mineral-based fibers. Plant-based fibers such as flax, hemp, sisal, kenaf, jute, coconut, banana, and bamboo are commonly used in composite production [4-8]. Studies have shown that the specific strengths of flax, hemp, and jute fibers, which have particularly high mechanical properties, are competitive with glass fiber [1,9]. Using animal fibers in composite production is considerably lower than plant-based fibers [10]. Animal fibers include hair or wool, silk, and feather fibers. The growing emphasis on sustainability has made special fibers, such as horsehair, a research subject. Horsehair, sourced from the tails and manes of horses, is utilized in various industries due to its properties, including elasticity, compression resistance, and high strength [11]. However, research on using horsehair as a reinforcement material in composite production remains limited [12-16].

Natural fibers present considerable challenges when incorporated into polymer composites despite offering several advantages. A primary issue is the inherent incompatibility between hydrophilic natural fibers and hydrophobic polymer matrices, which results in poor interfacial adhesion and, consequently, reduces the mechanical properties of the composites. This incompatibility is aggravated by the moisture absorption characteristics of natural fibers, which can further weaken the fiber-matrix bond. These incompatibilities are resolved by various solutions, including chemical treatments such as alkali treatment, which enhance the surface properties of the fibers [17,18]. Coupling agents and compatibilizers play a crucial role in enhancing the interfacial bonding between fibers and the polymer matrix, thereby improving the overall mechanical performance of composites. Although coupling agents are often used directly during the mixing process, they can also be grafted onto the hydroxyl groups of the fibers or impregnated into the fibers before the production process [19,20].

The structure and arrangement of the reinforcement material influence the properties of fiber-reinforced composites. Woven fabric structures allow for a more compact arrangement of fibers, providing composite materials with high strength, impact resistance, and flexibility. The study used plain and twill woven fabrics made from horsetail hair to reinforce the linear low-density polyethylene (LLDPE) matrix. To improve adhesion between the reinforcement material and LLDPE, the horsehair woven fabrics were coated with ethylene vinyl acetate (EVA). EVA is a copolymer composed of a hydrophobic (nonpolar) polyethylene (PE) monomer and a flexible, hydrophilic (polar) vinyl acetate (VA) monomer. It provides low-temperature flexibility, toughness, and strong adhesion to various materials. The VA group, which provides flexibility and adhesion to the copolymer, is expected to interact with the hydrophilic -OH group on horsehair, while the PE group bonds with LLDPE. The thickness, weight, morphological structure, mechanical properties, and thermal behavior of horsehair-woven fabric-reinforced EVA/LLDPE composites were examined.

2. MATERIALS AND METHODS

2.1. Materials

LLDPE, with a density of 0.92 g/cm³, a melt flow index of 1.0 g/10 min, and a melting point of 120°C, was sourced from Ozugur Plastik Ltd. Sti. EVA, with a vinyl acetate content of 18% (REPSOL PRIMEVA® P1820C), a density of 0.937 g/cm³, a melt flow rate of 2 g/10 min (at 190°C and 2.16 kg), and a melting point of 87°C, was purchased from Resinex. Chloroform (99%, EMSURE® ACS, ISO, Reag. Ph Eur) as a solvent was bought from Sigma-Aldrich. Washed and disinfected horse tail hairs were obtained from Barış Fırça Ind. Trade. Co. Ltd.

2.2. Production and Pre-Treatment of Fabrics

For fabric production, horsetail hair was grouped into bundles of 20 each for warp and weft use. The fabrics were woven on a handloom with two bundles/cm fixed warp density. Plain fabrics were produced with two planned weft densities of 5 bundles/cm and 7 bundles/cm, while twill fabrics were woven with 7 bundles/cm. To prevent the fabrics from fraying during composite production, the edges of the 18 × 25 cm² test area were secured with double stitching, 1 cm inward, using a Singer sewing machine. The weave type, the measured weft density, thickness, and weight of the produced fabrics are presented in Table 1.

Table 1. The physical parameters of horsehair woven fabrics

Sample codes	Weave type	Weft density (bundles /cm)	Thickness (mm)	Weight (g/m^2)
P5	Plain	4.87	1.41	487
E-P5	Plain	4.77	1.41	511
E-P7	Plain	7.30	1.79	674
E-T7	Twill (2/1)	7.80	2.17	696

The woven fabrics were coated with EVA to enhance compatibility with LLDPE. Before the coating process, the fabrics were conditioned in an oven at 40°C for 24 hours, and their initial weights were recorded. EVA (40 wt%) was dissolved in chloroform with a magnetic stirrer for 30 minutes at 60°C and 80 rpm. The solution, prepared at a 50:1 liquid-to-fabric ratio, was poured over the fabric, placed on a plate, and left sealed at room temperature for 15 minutes. The fabric, with one side coated, was dried in an oven at 40°C for 24 hours. The same procedure was applied to the other side of the fabric. The weight of the treated fabrics was measured, and the amount of EVA coating was determined relative to the initial dry weight of the untreated fabric. Except for the reference fabric coded as P5, all other fabrics were coated with EVA and are represented by the letter "E" in the fabric code.

2.3. Production of Composite Samples

The composites were fabricated by compression molding using a laboratory hot press (GULNAR MAKINA, Istanbul, Türkiye), where the fabrics were placed between two LLDPE sheets and compressed at 125°C and 4 bar pressure for 15 minutes using a 2 mm thick mold. The composites were allowed to cool slowly to room temperature under pressure for 6 hours. The LLDPE sheets were produced by placing them in a 1 mm thick mold and heating them at 130°C under 4 bar pressure for 15 minutes. Before the composite production, the fabrics were conditioned in an oven at 40°C for 24 hours (Figure 1).

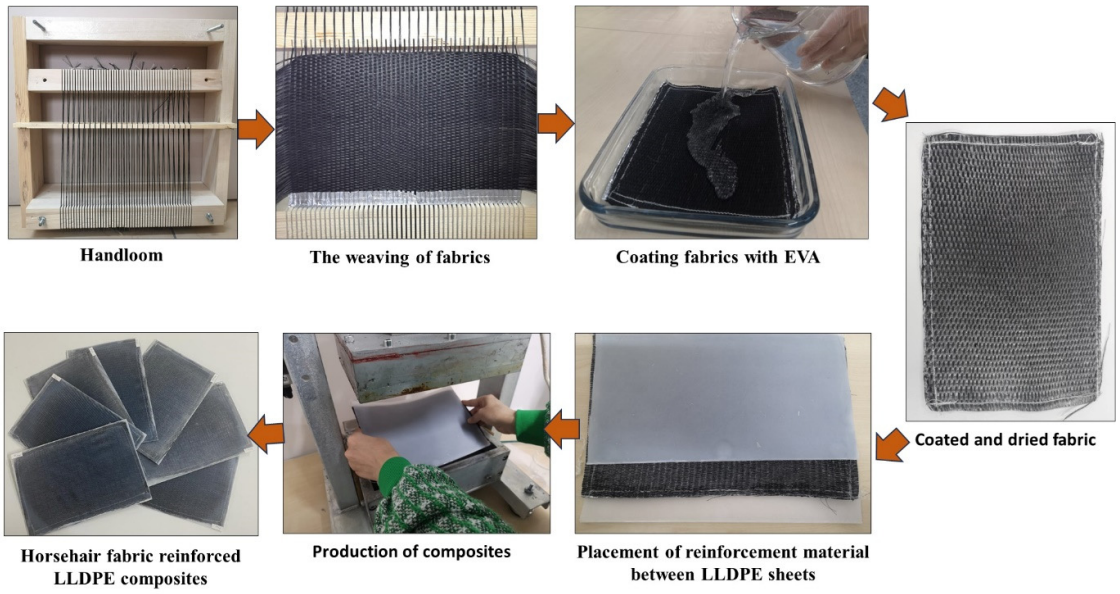

Figure 1. The production stages of composite samples

Table 2 presents the thickness, weight, and composition ratios of the composite samples. "P" represents plain weaving, while "T" indicates twill weaving. The numbers 5 and 7 correspond to the planned weft densities. "E" refers to the coating process with EVA, and "L" denotes the LLDPE matrix. For example, "LE-T7" indicates a composite structure reinforced with twill fabric made from horsehair treated with EVA and embedded in LLDPE sheets.

Table 2. The physical parameters of composite samples

Composite codes	Horsehair (%)	LLDPE (%)	EVA (%)	Thickness (mm)	Weight (kg/m^2)
LLDPE sheet	0.00	100.00	0.00	2.31	1.94
L-P5	21.83	78.17	0.00	2.55	2.55
LE-P5	22.34	74.33	3.33	2.60	2.60
LE-P7	28.28	65.54	2.18	2.85	2.85
LE-T7	27.56	69.87	2.57	2.90	2.90

2.4. Characterization of Composite Samples

Thermogravimetric analysis (TGA) and derivative TGA (DTGA) were performed using a thermogravimetric analyzer (Perkin Elmer, DIAMOND). The tests were conducted under a nitrogen atmosphere with 5 mg samples, heated from 23°C to 600°C at a flow rate of 10°C/min.

Field Emission Scanning Electron Microscopy (FESEM, ZEISS, GEMINI 500) was employed at an acceleration voltage of 5 kV to analyze the fracture surfaces of the composites and investigate the hair/matrix interactions. Before analysis, the sample surfaces were coated with an Au/Pd alloy under vacuum using a sputter coater.

The tensile properties of the developed composites were evaluated using a 50 kN capacity universal testing machine (Shimadzu AGX, Japan) following ASTM D 638-14 standards. Rectangular specimens (165 × 13 × thickness mm3) were tested at a crosshead speed of 5 mm/min with a grip separation of 100 mm. Flexural testing was carried out according to standard ASTM D790-10. The samples (13 × 125 × thickness mm³) were subjected to a three-point bending test, where the ram was loaded at a rate of 2 mm/min until fracture occurred.

A hair pull-out test was conducted on Shimadzu AGX to evaluate the hair and LLDPE interface characteristics. In the samples prepared according to the structure shown in Figure 2, the section labeled "a" was opened, and a single hair from the middle part of the sample was attached to the upper grip of the machine. The section labeled "c" was attached to the lower grip. The hair was pulled at a rate of 2 mm/min under a load of 5 kN, and the evaluation was based on the detachment or breakage of the hair from the structure.

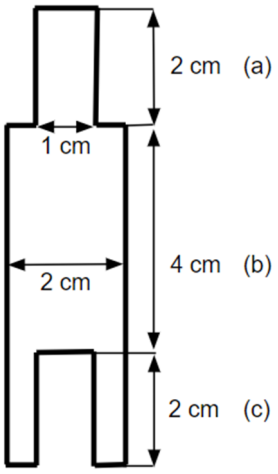

Figure 2. The sample dimensions prepared for a hair pull-out test to evaluate interface properties

3. RESULTS AND DISCUSSION

3.1. TGA/DTGA Analysis

The TGA and DTGA curves for the LE-P5 composite and its components are shown in Figure 3. The degradation of horsehair and EVA occurs in two distinct steps. The first step of horsehair degradation, likely due to the water loss in the structure, occurs at approximately 220°C with an initial weight loss of about 8%. The second step, which is taken in the temperature range of 220-500°C, results in a weight loss of approximately 64%. At this stage, the complete degradation of keratin is expected to occur. Due to the carbon content and mineral components in horsehair, about 30% of the residue remains at 800°C. EVA remains thermally stable up to 280°C. The first mass loss of 22%, observed between 280°C and 380°C, corresponds to the deacetylation of the VA group. This step results in the release of acetic acid into the gas phase. The second mass loss step of 77% (between 400 and 497°C) is attributed to the oxidation of hydrocarbons resulting from the degradation of the main polyethylene chain, which leads to their volatilization [21,22]. LLDPE maintains thermal stability up to 400°C, after which it loses 98% of its weight in the temperature range of 400-500°C.

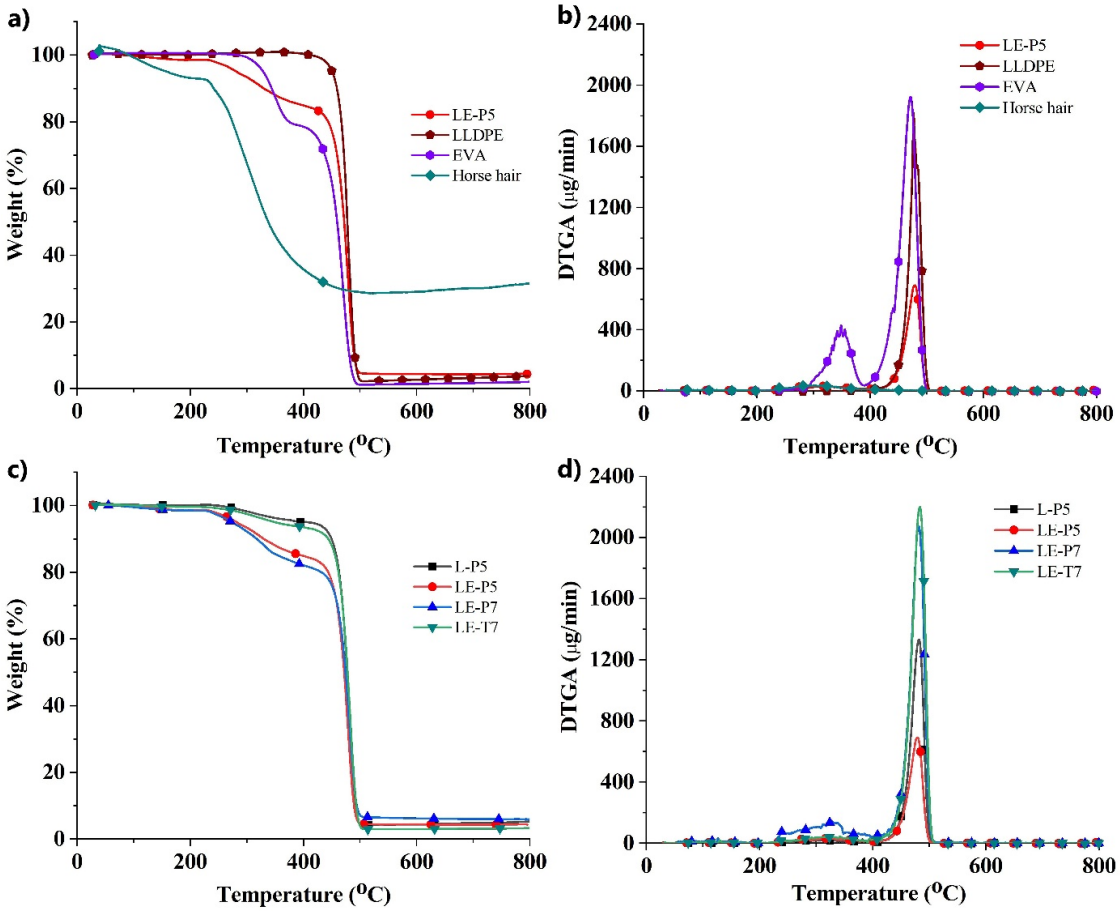

Figure 3. TGA (a, c) and DTGA (b, d) curves for composite samples and their components

The LE-P5 composite degrades in three steps. The first step occurs at 233°C, where the weight decreases by 2%. This step corresponds to the removal of water molecules present in the horsehair component. The initial weight loss is negligible since the fabrics were dried before being incorporated into the composite. The second step, occurring between 233-433°C, causes a 16% weight loss and corresponds to the first degradation stage of EVA and the second degradation stage of horsehair. The third step, between 433°C and 500°C, results in about 78% weight loss, representing the final degradation phase of EVA and LLDPE. Other composite structures (LE-P5, LE-P7, and LE-T7) show similar thermal degradation stages (Figure 3c and 3d). The inclusion of EVA in the composite generally reduces the thermal stability. However,

composite containing twill weaving fabrics (LE-T7) maintain their thermal stability despite the addition of EVA.

3.2. Tensile Properties

In the production of horsehair woven fabrics, weft density was chosen as a variable parameter, while warp density was kept constant. The tensile stress-strain curves of the composite samples in the weft direction are illustrated in Figure 4, while their respective values are listed in Table 3. Figure 4 demonstrates that pure LLDPE undergoes ductile rupture, whereas composites with reinforcing materials fail in a brittle manner due to rapid crack propagation. The LLDPE sheet was determined to have a tensile strength of 10.18 MPa, an elongation at break of 34.27%, and a modulus of 0.31 GPa. The reinforcement of the LLDPE sheet with horsehair woven fabrics generally increased the tensile strength and elastic modulus and decreased the elongation at break in the weft direction. The addition of untreated horsehair fabric with a weft density of 5 bundles/cm (L-P5) resulted in a 93.23% increase in tensile strength, a 414.56% decrease in elongation at break, and a 141.3% increase in elastic modulus in the weft direction. To improve the adhesion between the horsehair woven fabrics and the matrix material, all fabrics except the L-P5 coded fabric were coated with EVA. The 200X and 40X magnified images of untreated (a, b) and coated (d, e) fabrics are shown in Figure 5. The FE-SEM images demonstrate that the fabric surface is completely coated. The sample reinforced with EVA-coated fabric, having a weft density of 5 bundles/cm (LE-P5), exhibits a tensile strength of 21.97 MPa and a tensile modulus of 0.64 GPa. The EVA coating increased the tensile strength of the composite in the weft direction by 11.69% compared to untreated fabrics (L-P5). The increase can be attributed to the improved interfacial bonding between the fabric and the matrix. As a more flexible and adhesive material than LLDPE, EVA likely enhances the interaction between the fabric fibers and the polymer matrix. Additionally, the coating could reduce defects or weak spots in the matrix, leading to higher tensile strength. When the fabric was coated with EVA, the elongation at the break of the composite increased by 75.23%. The coating likely enhanced the overall ductility and flexibility of the composite, improving its ability to stretch and absorb stress before failure. In contrast, the elastic modulus decreased by 14.67% with the coating. The EVA coating may have softened the overall structure of the composite, leading to a decrease in its ability to resist elastic deformation under stress. Figure 5 shows the fracture morphology corresponding to the composites reinforced with untreated fabric (L-P5) and coated fabrics (LE-P5). The L-P5 coded sample exhibits fiber fracture, pull-out, and debonding. With the fabric coating, the majority of the fibers are observed to break, while the matrix fractures in a more ductile manner. These morphological analyses are consistent with the tensile test results.

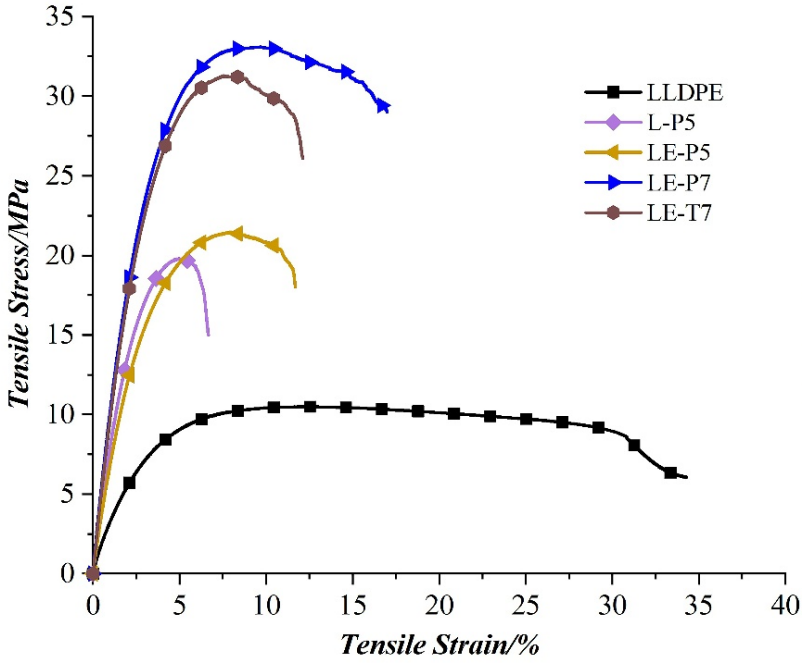

Figure 4. Tensile stress-strain curves of composite samples in the weft direction

Table 3. Tensile strength, elongation at break, and elastic modulus values of composite samples

Samples		Tensile strength (MPa)	Elongation at break (%)	Tensile modulus (GPa)
LLDPE	-	10.18	34.27	0.31
L-P5	weft	19.67	6.66	0.75
	warp	10.89	7.14	0.40
LE-P5	weft	21.97	11.67	0.64
	warp	10.97	7.93	0.45
LE-P7	weft	32.62	16.98	0.97
	warp	9.64	10.91	0.38
LE-T7	weft	30.99	12.09	0.94
	warp	8.72	8.31	0.34

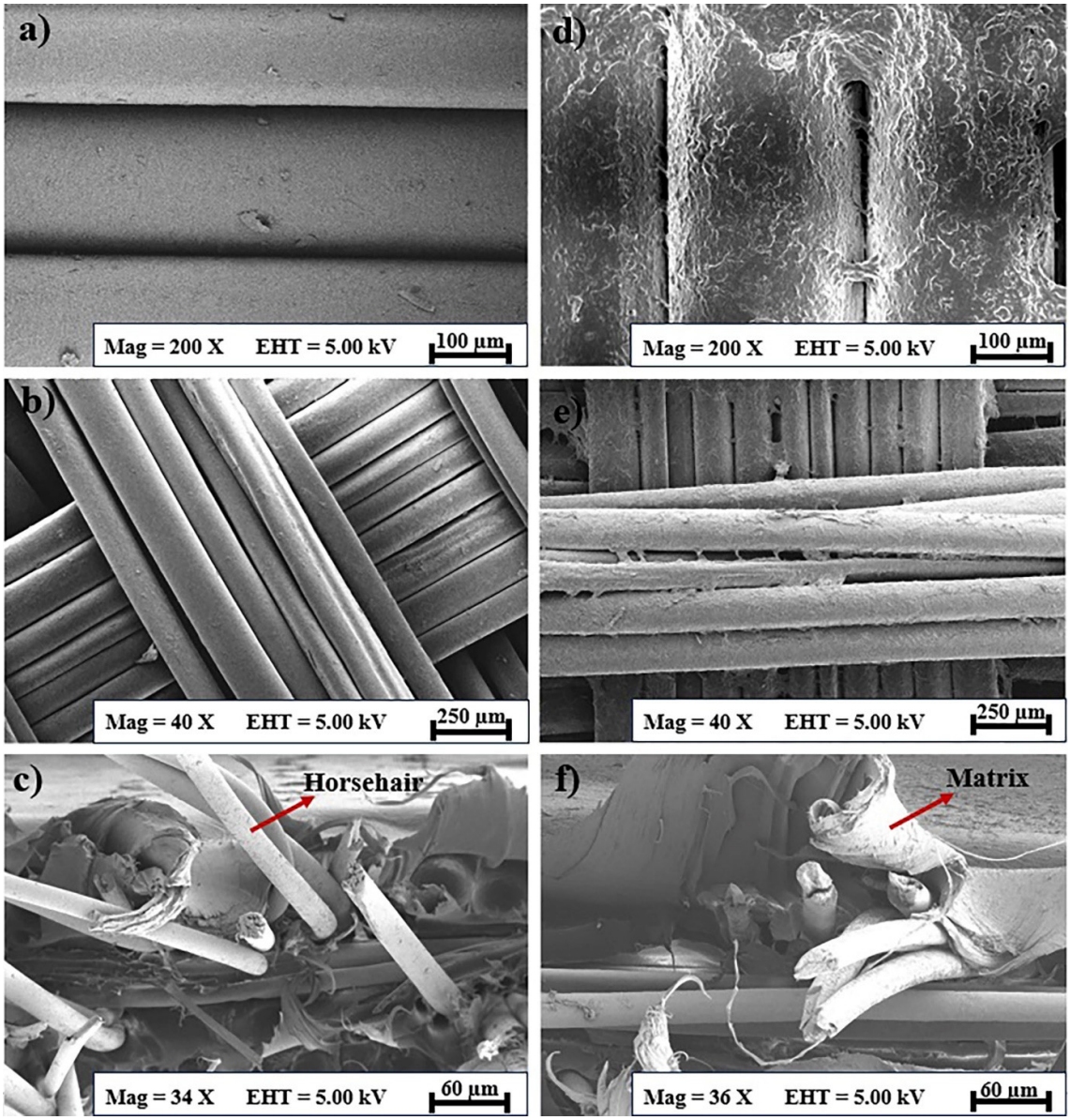

Figure 5. FE-SEM micrographs of untreated (a, b) and EVA-treated (d, e) fabrics at 200X and 40X magnifications; micrographs of tensile fracture surfaces of composites in the weft direction (c) L-P5 at 34X and (f) LE-P5 composites at 36X

The sample reinforced with EVA-coated fabric having a weft density of 7 bundles/cm (LE-P7) exhibited the highest tensile strength of 32.62 MPa and the highest elastic modulus of 0.97 GPa. The increase in weft

density enhanced the tensile strength, elongation at break, and elastic modulus in the weft direction by 48.47%, 45.50%, and 51.56%, respectively. The increased horsehair density enables the fabric to carry more load, thereby improving stress distribution and load transfer between the fabric and the matrix.

The sample reinforced with twill weaving fabric having a weft density of 7 bundles/cm (LE-T7) exhibited a slight decrease of 4.9% in tensile strength compared to plain weaving fabric. The twill weaving structure caused a 40.45% reduction in elongation at break and a 3.09% decrease in elastic modulus. The diagonal twill weaving structure creates a more irregular surface, which may have caused micro-scale voids between the fabric and LLDPE sheets during composite production. These small voids could hinder interfacial bonding between the fabric and the matrix, potentially reducing stress transfer efficiency along the interface.

Table 3 presents the mechanical properties of composites in the warp direction of the reinforcement fabric. The horsehair warp density has been constant at two bundles/cm, depending on the handloom. The tensile strength of the samples with codes L-P5 and LE-P5 in the warp direction was found to be 6.97% and 7.76% higher, respectively, compared to the LLDPE sheet. However, when the horsehair density increased in plain and twill weaving fabrics, the tensile strength was lower than in the pure LLDPE sheet. The lower warp density may limit the load-bearing capacity of the warp threads, leading to reduced tensile strength in that direction. Moreover, the crimping of the threads also affects tensile strength [23]. The increase in weft density may have increased the crimp effect, which could have limited the load-bearing capacity of the fewer warp threads.

3.3. Adhesion Degrees of the Reinforcement Material

The adhesion degree of the reinforcement material within the composite samples was evaluated based on the occurrence of breaking or debonding of a single hair underload. The hairs were selected from the center of the bundle in the weft direction. Three repetitions were performed for each sample, and the adhesion degree was determined according to the number of hairs that broke. According to the values presented in Table 4, no significant difference in adhesion degree was observed between the samples with and without EVA. However, the samples with a plain weaving structure exhibited better adhesion than those with twill weaving. This is thought to be due to the higher number of weaving intersections per unit area in plain weaving, which increases friction and improves adhesion. Additionally, this result indicates that the matrix penetrates less into the inner parts of the fabric in the twill weave structure.

Table 4. Adhesion degrees based on the behavior of hairs pulled from the samples

Samples	1	2	3	Adhesion degree
L-P5	Debonding	Breaking	Debonding	1
LE-P5	Debonding	Debonding	Breaking	1
LE-P7	Debonding	Breaking	Breaking	2
LE-T7	Debonding	Debonding	Debonding	0

The tensile strength and elongation at break values of the hairs debonded from the structure are presented in Table 5. According to these results, the EVA-coated reinforcement demonstrated higher resistance than its uncoated counterpart. As the number of bundles in the structure increased, the adhesion degree was generally negatively affected. This may be attributed to a reduction in the penetration of the matrix material into the reinforcement or an insufficient matrix-to-reinforcement ratio within the composite.

Table 5. Maximum stress and elongation values of hairs debonded from the structure

Samples	Tensile strength (MPa)	Elongation at break (%)
L-P5	0.09	17.93
LE-P5	0.12	27.69
LE-P7	0.07	20.09
LE-T7	0.04	11.48

3.4. Flexural Properties

The flexural stress-strain curves in the weft direction for fabric-reinforced composites are presented in Figure 6. Table 6 presents the flexural strength, flexural strain, and flexural modulus in both the weft and warp directions. Overall, the reinforcement material increased the flexural resistance in the weft direction. The untreated fabric with a density of 5 bundles/cm improved the flexural strength in the weft direction by 5.67%, while the treated fabric with the same density enhanced it by 8.56%. The reinforcement material generally increased the flexural modulus. The flexural modulus of the L-P5 composite sample in the weft direction was 1.30 GPa, while that of the LE-P5 composite sample was 1.19 GPa. The EVA coating slightly reduced the flexural modulus. The EVA layer gave additional flexibility to the composite structure and reduced its overall stiffness.

The composite reinforced with coated fabrics having a weft density of 7 bundles/cm exhibited a flexural strength of 11.06, a bending strain of 3.69%, and a flexural modulus of 0.83 GPa in the weft direction. The flexural strength and bending strain improved as the horsehair density increased, while the flexural modulus slightly decreased. The LE-T7 coded twill fabric with a weft density of 7 bundles/cm was found to have the highest flexural modulus, measured at 12.67 MPa in the weft direction. With its diagonal arrangement of thread, twill weaving enables a more homogeneous distribution of vertical loads. Additionally, the LE-T7 sample exhibited a flexural strain of 3.43% and a flexural modulus of 1.07 GPa in the weft direction.

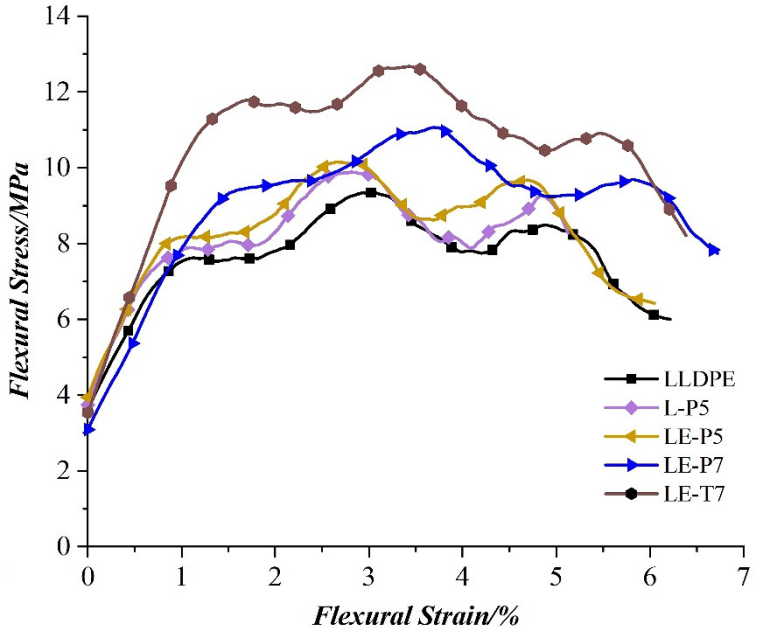

Figure 6. Flexural stress-strain curves of composite samples in the weft direction

Table 6. Flexural strength, flexural strain, and flexural modulus values of composite samples

Samples		Flexural strength (MPa)	Flexural strain (%)	Flexural modulus (GPa)
LLDPE	-	9.35	3.05	1.00
L-P5	weft	9.88	2.82	1.30
	warp	8.03	2.70	1.06
LE-P5	weft	10.15	2.88	1.19
	warp	9.34	3.12	0.98
LE-P7	weft	11.06	3.69	0.83
	warp	8.24	3.25	0.79
LE-T7	weft	12.67	3.43	1.07
	warp	6.97	4.28	0.71

The flexural strength values of the composites in the warp direction were lower than those of the matrix material. Furthermore, an increase in the weft density of the fabrics resulted in a deterioration of the flexural strength and flexural modulus of the composites in the warp direction. The low warp density and the resulting unbalanced structure may have contributed to this outcome.

4. CONCLUSIONS

The present study investigates the thermal and mechanical properties of LLDPE composites reinforced with horsehair woven fabrics, with a focus on the influence of fabric structure (plain and twill), weft density (5 and 7 bundles/cm), and surface treatment with EVA.

Including untreated and coated horsehair-woven fabrics slightly reduced the thermal stability of the composite structure. The reinforcement with horsehair-woven fabrics significantly enhanced the mechanical properties of the composites. The EVA coating enhanced the interfacial bonding between the fabric and the polymer matrix, resulting in higher tensile strength and elongation at break while reducing the tensile modulus by increasing the ductility of the composite in the weft direction. Furthermore, FE-SEM analysis revealed that composites reinforced with coated fabrics predominantly exhibited fiber breakage. Increasing the fabric's weft density improved the composites' mechanical properties in the weft direction. The composite reinforced with EVA-coated fabric with a weft density of 7 bundles/cm demonstrated the highest tensile strength of 32.62 MPa and an elastic modulus of 0.97 GPa in the weft direction. The use of twill weaving fabrics decreased tensile strength and modulus, likely due to the irregular surface structure and the formation of voids between the fabric and matrix. Despite this, the composite reinforced with twill fabrics exhibited the highest flexural strength of 12.67 MPa in the weft direction. Since the warp density of the composites is considerably lower than the weft density, no significant improvement was observed in the tensile and flexural properties in the warp direction; instead, these properties worsened as the weft density increased. The findings suggest that for optimal performance, careful selection of horsehair fabric structure and density, as well as matrix treatment, is essential for tailoring the properties of the composite material for specific applications.

5. ACKNOWLEDGMENT

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