REGENERATED CELLULOSE-BASED COMPOSITE STRENGTHENED WITH POST-CONSUMER POLYESTER GARMENTS

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Polyester garments have an extensive availability nowadays. However, most post-consumer garments are burned and landfilled, leading to extreme pollution and significant waste of resources. Therefore, sustainably and economically recycling this post-consumer polyester (PCP) fabric waste for essential value-added products is meaningful and necessary. In this study, waste polyester fabrics obtained from PCP garments have been utilized in the development of composite materials. First, PCP fabric was dissolved in a methanol and sodium hydroxide solution. Then, the polyester paste was regenerated on regenerated cellulose-based fabric (viscose), constructing a viscose-based composite. The physical, thermal, chemical, morphological, and mechanical properties were examined for the viscose and viscose-polyester (VP) composites. FTIR, XRD, SEM, and percent add-on confirmed the presence of polyester in the composite. In addition, an add-on percentage of 10.71, higher crystallinity of 55.20%, thermal stability, and about 21% higher tensile strength were observed. The results, as mentioned above, ensured that the PCP waste can be used as a matrix for composite materials.

Keywords: viscose fabric, post-consumer polyester fabric, composite, recycling

INTRODUCTION

In recent years, the tremendously increasing living standards and population growth have boosted consumer demand for fabric. To meet the fabric demand, the textile industry has raised annual fibre and fabric production.[1] Currently, the global yearly fibre production is 82 million tons, of which 60% is synthetic.[2] Presently, fast fashion is controlled by synthetic textiles, especially polyester, with a yearly production of polyester exceeding 55.1 million metric tons.[3] Furthermore, more than 30% of the clothes purchased in Europe have not been worn for at least one year due to fast fashion trends.[4] Thus, the higher manufacturing capacity of textile industries and extensive use of polyester leads to higher production of textile waste. Given these economic and environmental issues, a growing interest has been developed in turning textile waste into valuable goods.[5]

Pre-consumer, post-consumer, and industrial wastes are all included in textile waste streams. The European Union produces almost 5.8 million tons of post-consumer textile waste annually.[6] In addition, polyester materials generate a considerable amount of waste (fibres, filaments, fabrics, films, bottles, and other molded articles).[7] Almost half of all fibre waste comes from the spinning industry alone, which accounts for nearly all waste fibre output.[8] Similarly, waste from the apparel industry and post-consumer apparel is increasing rapidly. Global fashion waste reached 92 million tons in 2015 and is expected to reach 148 million tons by 2030.[9] Hence, the development of processes that utilize fabric and fibrous wastes and transmute them into significant products to mitigate the increasing impact of suchlike non-biodegradable material on the environment.

Polyester materials represent a threat for the environment because of their non-biodegradable nature.[2] The polyester material can pollute land, waterways, and air.[10] Polyester fabric in landfills is alarming, as it releases dyes and chemicals into the soil. This leads to soil contamination.[11] The air is also polluted through the fly-off produced by small and light polyester fibres during manufacturing.[2] Numerous studies have revealed that synthetic fibres account for a sizable portion of the microplastic found in water and are frequently cited as the leading cause of pollution. Plastic Soup Foundation has revealed that more than 4,500 fibres can release after washing one gram of garments.[12,13] Microfibers are so tiny that they can readily pass through wastewater treatment plants and easily bind with molecules of harmful elements in wastewater. These microfibers are then consumed by plankton and small fish, concentrating toxins and going up the food chain.[14-17] The effects on the human body need to be investigated and clarified. Hence, proper recycling of waste polyester material can reduce environmental and human burdens by decreasing the flow of waste polyester.

A lot of research has been performed on recycling polyester materials. Hydrolysis,[18] glycolysis along with microwave irradiation,[19,20] and electron beam radiation[21] methods have been developed for the recycling of polyester bottles. Some researchers have separated polyester from a polyester wool blend using static electricity, fusing, hydrolyzing, and enzymatic degradation methods.[22,23] Various procedures, such as using ionic solvent,[1,24] a two-step method of acid treatment,[25] and phosphoric acid,[26] have been carried out for recycling cotton polyester blends. A few studies have been performed to make composites from wasted polyester fabric. Ozen *et al*.[27] have made epoxy composites by the vacuum-assisted resin transfer molding method, using waste polyester fabric. Sharma *et al*.[2] have made cotton composites reinforced with waste polyester. These researchers used waste polyester fibre found from spinning and m-cresol as solvent. Their research focused on analyzing the mechanical properties of a cotton composite by using environmentally friendly solvents and waste polyester fibre.

In the present work, we presented a technique for using polyester fabric waste collected from end-users of polyester garments and manufacturing a composite by chemically treating the garment waste. This procedure offers a new method of composite manufacturing and decreases the effect of polyester garment waste. Thus, a suitable solvent was used first to liquefy the polyester fabric. A viscose fabric was then covered with a regenerated layer of this solution. The polyester particles take a position on the inner and outer surface of the viscose fabric, giving high abrasion and washing resistance. The environmental and health risks were considered when choosing the solvent.

EXPERIMENTAL

Materials

The 100% viscose plain-woven fabric used in constructing the composite was collected from the weaving setup of a textile plant. The structure of the plain ($\frac{1}{1}$) fabric was (75 × 70 / 32 × 30) × 56, 140 gram per square meter (GSM), dyed with reactive dyes. The regenerated cellulose-based (viscose) fabrics had a thickness of around 0.6 mm. PCP garments, treated as post-consumer textile waste, were also collected for this study. Methanol was utilized to dissolve the PCP garments; acetic acid and sodium hydroxide pellets were bought from M/S Nasir Chemicals, Dhaka, Bangladesh. The composite development processes and drying with a small-scale framework for holding the treated fabric were brought to pass on a laboratory-scale rubber-coated padding mangle and Mathis drying machine, respectively.

Methods

Specimen preparation

Dark blue dyed viscose fabric and white coloured PCP garment were taken to identify polyester components in the viscose-polyester (VP) composite. First, the viscose fabric was cut into pieces with dimensions of 10×10 inches and washed for one hour at 50 °C. Thus, dust was removed and the extra dye was washed out from the fabric surface. Next, the collected polyester garments were washed with detergent to remove dust, dirt, and oil from the apparel and thoroughly cleaned with tap water. Subsequently, the washed fabric and garment were sun-dried for approximately 48 h. After that, the dried PCP garment was cut into small pieces. An image of the dried chopped PCP is presented in Figure 1 (b).

Composite development

Chopped polyester fabric, *viz.* 3% (w/v) was dissolved in acetic acid (CH_3OH) and sodium hydroxide (NaOH) solution according to the AATCC test method 20A-2017.[28] Amounts of 18 g of NaOH and 200 mL of CH_3OH were added in a 250 mL Erlenmeyer flask. The solution was heated to 65 °C for mixing thoroughly. The specimen was then placed in the Erlenmeyer flask and stirred using a magnetic stirring bar for 5 min. After that, the solution was poured into a Petri dish to introduce viscose fabric into

the methanol solution for about a minute. Figure 1 (d) shows the liquefied polyester in methanol solutions. Afterward, the viscose fabric was removed from the solution and run through a rubber-coated padding mangle to eliminate the residual solution and allow the solution to penetrate deeply into the viscose fabric. The padding procedure was repeated four times to acquire appropriate solution penetration inside the viscose fabric and ensure a four-dip-four-nip process.

Once outside the padding, the treated VP composite was attached to a fixed tray in a stretch condition to permit the utmost opening of the VP composite in the stretched condition. A Mathis curing machine was used to dry and cure the fabric.[29] The fabrics were treated at 80 °C for 3 min and 110 °C for 2 min for drying and curing, respectively. Then, the fabrics were released from the stretch condition and kept for one hour at room temperature for relaxation. The VP composite fabric was then cleaned with regular water and neutralized with a 0.1% CH_3COOH solution. In addition, the VP composite was submerged into a solution with 2 g/L neutral detergents at 100 °C for about 10 min. Following a normal wash, the treated sample was dried in an electric dryer.[30] Figure 1 depicts the stages of the composite preparation procedure.

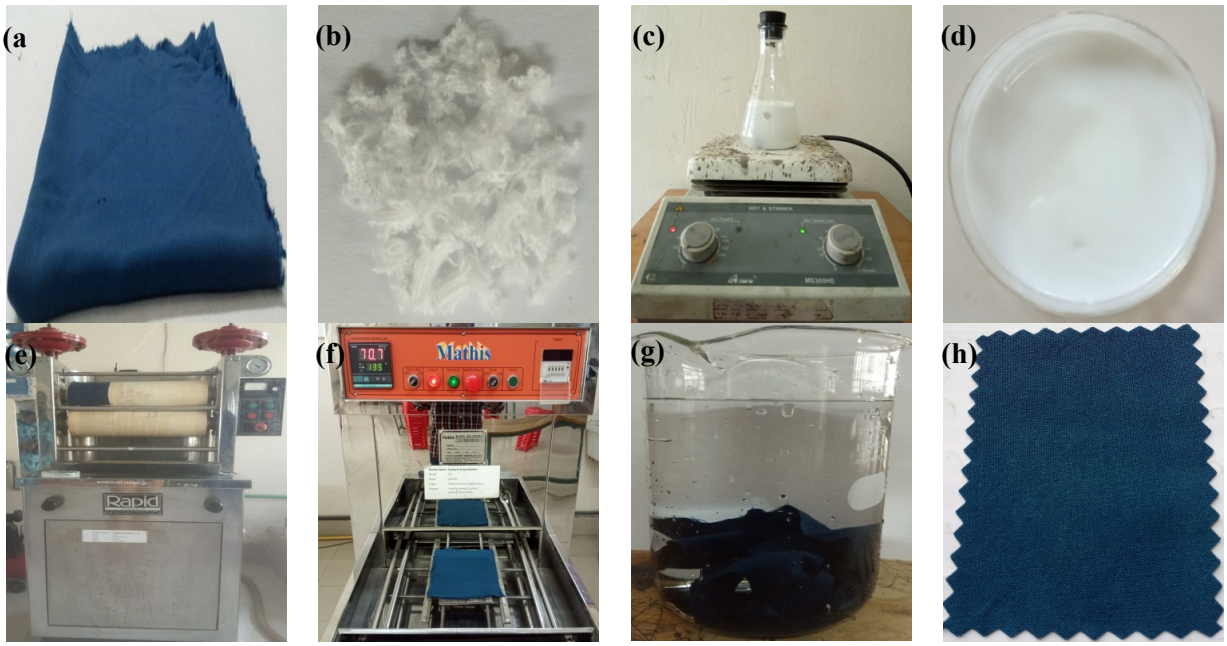

Figure 1: Viscose fabric (a), cut polyester pieces (b), polyester dissolution (c), dissolved polyester paste (d), padding mangle (e), drying and curing (f), neutralization (g), and VP composite fabric (h)

Characterization

Weight change of the fabric

The weight change of the viscose fabric was determined from the difference in gm^{-2} (GSM) before and after the composite preparation. A GSM cutting device from James H. Heal & Co. Ltd., England, was used to determine the fabric's GSM according to the ASTM-D-3776 method.[31]

Percent add-on

The VP composite was developed with ratios of reinforcing viscose fabric and polyester matrix. The composite fabric weight was measured after curing, and the add-on percentage of the matrix was obtained based on the oven-dry weight of the viscose fabric:

The add-on percentage of the polyester-coated viscose fabric was established using Equation (1):

$$Add-on\ (\%)\ of\ Matrix = \frac{100 \times [Treated\ fabric\ weight - Initial\ fabric\ weigth]}{Initial\ fabric\ weight} \quad (1)$$

Spectroscopic characterization (FTIR)

Fourier transform infrared (FTIR) spectroscopy was performed to observe how viscose fabric and polyester interacted. FTIR spectra of viscose, polyester, and VP composite were recorded in the wavenumber range of 4000 to 400 cm^{-1} at the resolution of 2.0 cm^{-1}, with 32 scans for each specimen, using a FTIR-8400S (Shimadzu Corporation, Japan) spectrometer.[32]

X-ray diffraction (XRD) analysis

The XRD of the viscose fabric and VP composite was performed on a Bruker AXS D8 diffractometer, Germany, using Cu Kα radiation (λ = 1.5405 Å). The operating conditions were the following: V = 40 kV and I = 40 mA in symmetrical reflection within the (2θ) range of 10° to 50°.

Thermal analysis

Thermogravimetric analysis (TGA) of the samples was performed to determine the thermal behavior of

the samples, using an SDT650 thermal analyzer, TA Instruments, USA. 5 mg samples were used for the TGA experiment. The TGA was performed under N_2 (g) atmosphere with 30 mL/min of gas flow, a constant heating rate of 10 °C/min, in the temperature range from room temperature to 600 °C.[33]

Moisture content

After conditioning at 105 °C, the water content of the VP composite specimen was measured using a moisture analyzer, according to the standard method ASTM D2495. A 5 g specimen was used according to the standard atmospheric conditions for testing – at 20 °C ± 2 °C and 65% ± 2% RH. With a 15 min interim, the weight of the specimen was measured until there was 0.1% less variation in sample weight between gradual weighing.[34] The weight variations of the sample between testing under atmospheric conditioning and after oven drying determine the moisture content of the composite. The following equation was used to compute the moisture content percentage of the samples:[35]

$$M = \frac{W_1 - W_2}{W_1} \times 100 \quad (2)$$

where M, W_1, and W_2 denote the moisture content in percentage and specimen weight before and after drying in grams, respectively.

Morphology analysis

The surface morphology of the viscose fabric and VP composite samples was examined with a Zeiss Evo 18 Scanning Electron Microscope. Before testing, the specimens were coated with a thin layer of gold, during 3 to 4 min, using a sputter coater at electron accelerating voltage of 10 kV. Likewise, a Jiusion microscope was used for the optical analysis of the specimens (1000x), according to standard ISO 25178.

Mechanical properties

The tensile strength, Young's modulus, elongation at break, and energy at break of the viscose fabric and VP composite were calculated as per the ASTM D3822–07 standards, using an H10KS testing machine (Hounsfield, UK), with a crosshead speed of 20 mm/min.[36] Ten samples were tested, and the mean results were recorded. Statistical analysis of tensile strength was performed by Weibull probability distribution using Minitab 18 software. The tensile strength was determined by Equation (3):

$$\sigma = \frac{F_b}{S_0} \quad (3)$$

where σ, F_b, and S_0 denote the specimen tensile strength, maximum force at break, and cross-sectional area, respectively. The elongation at break and Young's modulus were determined from the test data.

Spectrophotometry analysis

Colour difference tests of viscose and VP composite fabric were performed using a computer integrated reflectance spectrophotometer. The CIELAB colour coordinates were determined: CIE L*, a*, b*, C*, h*.[2,31] The colour difference, ΔE^*_{ab}, was measured using Equation (4):

$$\Delta E^*_{ab} = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2} \quad (4)$$

where ΔE^* is the total colour difference on the CIELAB chart; Δa, Δb, and ΔL are the difference in red/green, yellow/blue, and lightness/darkness axis, respectively.

RESULTS AND DISCUSSION

Weight change and add-on percent

The changes in fabric weight (GSM) and add-on percent were determined to confirm the incorporation of the polyester particles into the viscose fabric. Based on the obtained results shown in Table 1, it can be noticed that the fabric GSM increased from 140 to 155 in the composites formed utilizing 3% concentrations of polyester in the solution, compared to the neat viscose fabric. This weight change is likewise related to the increase in the add-on % to 10.71%. During the first phase of padding, the dissolved polyester penetrated into the spaces among the fibres due to the squeezing operation accomplished using the padding mangle. Subsequently, the polyester solution gets enduringly sealed onto the viscose fibre. This is reflected in the high add-on percent of the coated fabric, compared to the viscose fabric.

Spectroscopic characterization (FTIR)

The FTIR analysis was used to determine whether polyester particles were present in the composites, and the spectra recorded for the materials are presented in Figure 2. The bands at 3424 and 1015 cm^{-1} are attributed to intermolecular -OH bound to C=O groups and OH out-of-plane bending in the terminal carboxylic groups in the polyester chains, respectively.[37] The band at 1710 cm^{-1} is attributed to the presence of the carboxylic group. Anhydride groups are responsible for the polyester absorption band that emerges at 1953 cm^{-1}.[38]

Well-defined bands with typical cellulose structures can be seen in the FTIR spectrum of the viscose fabric at 3327, 2903, 1423, 1365, 1162 and 1016 cm^{-1}.[36,37] All characteristic peaks of the viscose fabric experienced red shift intensity after the incorporation of polyester. Although the peak at 1718 cm^{-1} assigned to the carbonyl group was absent in the spectrum of the viscose fabric, it was redshifted in the spectrum of the VP

composite.[39,40] Strong C-H bending (721 cm^{-1}), modest C-H stretching (2956 cm^{-1}), and relatively significant C-C out-of-plane bending vibrations of the benzene rings are all visible in the FTIR spectrum of polyester (871 cm^{-1}). A characteristic peak at 837 cm^{-1} in the VP composite and polyester FTIR fingerprint region was similarly absent from the spectrum of the pure viscose fabrics. In this approach, the composite fabric began to behave similarly to polyester upon the incorporation of polyester particles into the viscose. The decreased peak intensity at 1112 cm^{-1}, which is characteristic of viscose, also demonstrates the reduced viscose-like behavior.

X-ray diffraction (XRD) study

The crystalline and amorphous characteristics of the base viscose and of the VP composite were determined using X-ray diffraction analysis. The recorded XRD patterns are shown in Figure 3. The peaks typical of viscose are recorded at 2θ = 15.38 and 29.74.[41] As regards the composite, the diffraction peak at 2θ = 21.5° and plane 002 are usually characterized by a narrowing of the peak intensity, typically indicating a highly ordered crystalline area.[2] The results revealed that the crystallinity of the VP composite was higher (55.20%) than that of the viscose fabric (50.22%). This can be explained by the fact that, upon the addition of polyester particles, they formed crystal structures after regeneration in the amorphous regions of the base viscose fabric. In this way, the incorporation of polyester improved the crystallinity of the initial viscose.

Table 1
Physical and mechanical properties and moisture content of viscose and VP composite

Sample	Physical properties			Moisture content (%)	Mechanical properties			
	GSM	Matrix weight (g)	Add-on (%)		Tensile strength (MPa)	Elongation at break (%)	Young's modulus (N/mm^2)	Energy at break (J)
Viscose fabric	140	-	-	11.14	113	19	8.55	0.4064
VP composite	155	15	10.71	13.10	136.5	17.5	8.5	0.38575

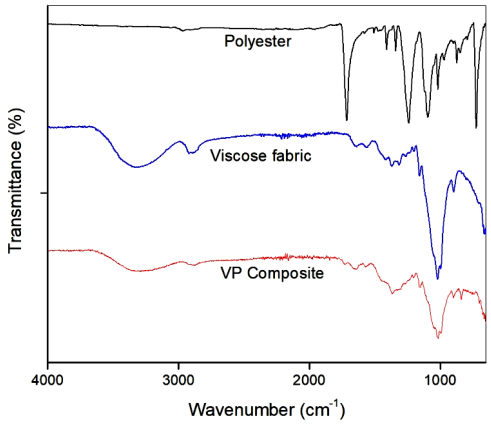

Figure 2: FTIR spectra of polyester, viscose and VP composite fabrics

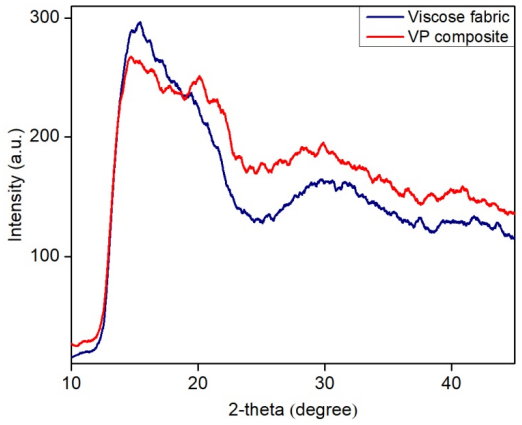

Figure 3: XRD patterns of viscose and VP composite fabric

Thermal analysis

The thermal stability of the viscose and its composite was investigated by thermogravimetric analysis (TGA) and derivative thermogravimetry (DTG), as shown in Figures 4 (a and b), respectively. The thermal degradation of the VP composite is impacted by polyester deposition.[2] According to the TG curves, an intial weight loss was observed in both samples in the range of 50–250 °C, which was due to the evaporation of the absorbed moisture from the viscose. This weight loss represented 29% and 13.18% for the base viscose and polyester-incorporating viscose composite, respectively. The difference in the weight loss percentage is explained by the hydrophobic nature of polyester, as opposed to the hydrophilicity of the viscose. The results also show that the viscose sample and VP composite started to degrade at about 225 °C and 289 °C, respectively, with significant thermogravimetric

peak maxima occurring at about 359.5 °C and 311.5 °C. The thermal stability of the viscose fabric was high due to the cellulose content.[42] Also, the weight losses of the base viscose and the VP composite at T_{dmax} were of 59.46% and 11.98%, respectively.

Moisture content

The moisture content of the polyester-incorpotating composite fabric was influenced by the hydrophobic polyester incorporated in the viscose fabric. Thus, the moisture content of the samples was reduced from 13.10% to 11.14%, upon incorporation of the polyester particles. This signifies that the hydrophilic viscose fabric has marginally turned hydrophobic, and the normally characteristic hydrophilic property of the viscose is not considered for the composite.

Morphology analysis

SEM micrographs of the base viscose and VP composite specimens are presented in Figure 5 (a) and (b). It may be seen that the incorporation of polyester into viscose fabric modifies the surface morphology of the fibres. The viscose fabric initially shows typical smooth longitudinal fibrils, with some light deposits of white particles, probably due to chemical residues (Fig. 5 (a)). On the other hand, the surface of the polyester-incorporating composite material reveals larger deposits of polyester particles, which make the surface appear rougher (Fig. 5 (b)). Similar results were also found in optical images taken with a laser microscope (Fig. 5 (c), (d)). The viscose fabric serving as the base material has a striking dark blue colour, while polyester particles are white. As a result, the composite VP fabric appears to have a lighter shade than the base viscose fabric.

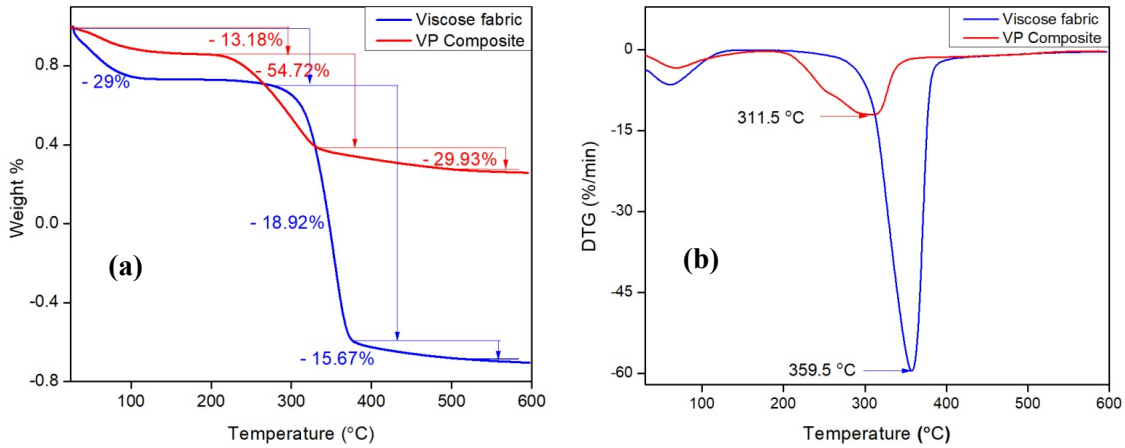

Figure 4: TGA (a) and DTG (b) curves of viscose fabric and VP composite samples

Mechanical properties

The stress *vs* strain and tensile strength elongation percentages are shown in Figure 6 (a) and (b), respectively, for the initial viscose and composite fabric samples. As expected, the addition of polyester boosted the tensile strength, while reducing the elongation at break of the initial viscose. It is clear from Figure 6 (a) that the tensile strength of the composite increased by 20.80%, compared to the initial viscose specimen (113.1 MPa), reaching a maximum tensile strength of 136.6 MPa. The elongation at break, on the other hand, droppted from 19% (for the viscose) to 17.50% (for the VP composite). This can be explained by the presence of the polyester particles on viscose fabric, which helped to improve the tensile strength and reduce the elongation percentage, while also enhancing the crystallinity of the initial viscose, as discussed above.

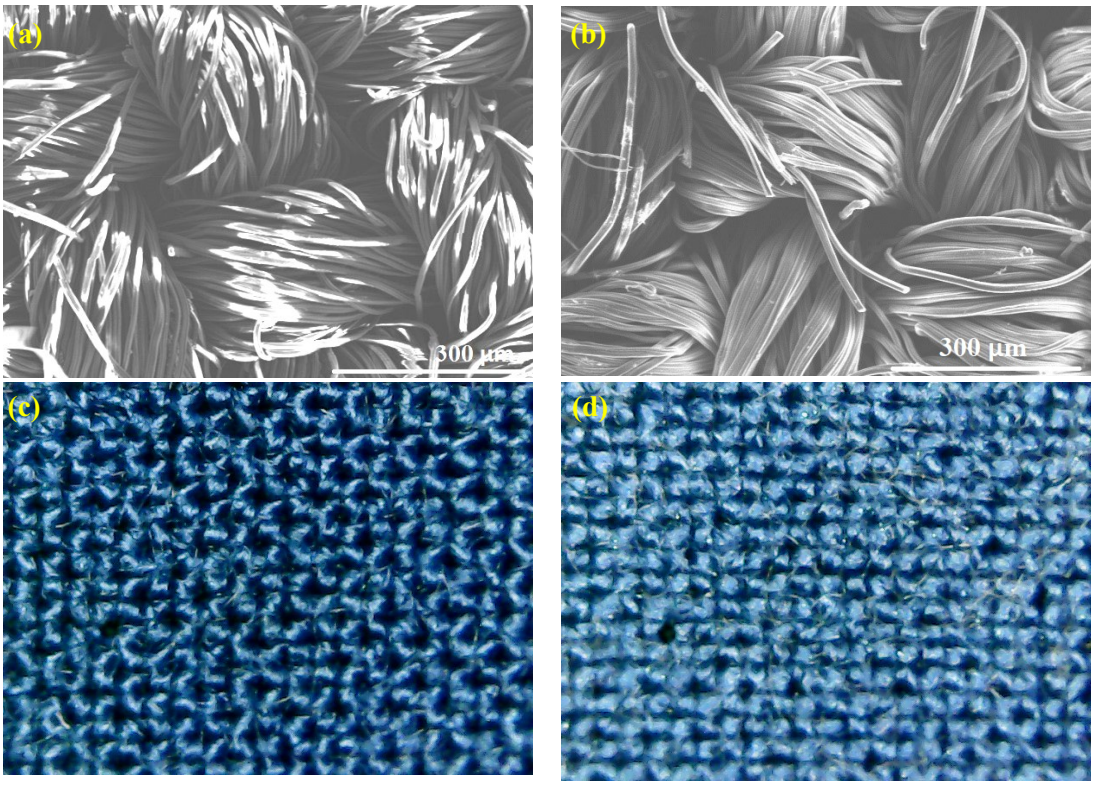

Figure 5: SEM images of viscose (a) and VP composite (b); laser microscopic images of viscose (c) and VP composite fabric (d)

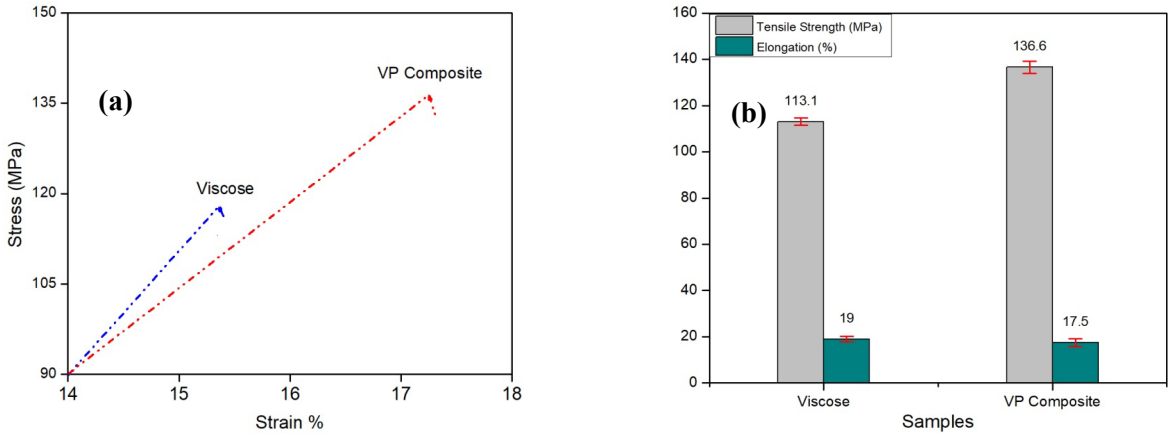

Figure 6: Stress *vs.* strain curve (a), tensile strength and elongation percentage (b) of viscose and VP composite fabrics

The region of the elongation percentage and the tensile strength of the materials have an impact on the energy at break. The viscose fabric demonstrates the necessary energy at break, which is higher than that of the composite (0.038575 J), namely 0.04046 J. It may be inferred from this experiment that the energy at break, shown in Table 1, increases with the elongation percentage.

Figure 7 shows the study of the Weibull probability distribution line for the tensile strength of the viscose and VP composite samples. The tensile strength results can be observed to be within bounds and suit the distributions entirely. The Weibull distribution confirms that the tensile strength values are close to the calculated mean values by this statistical analysis.

Visual appearance

The change of fabric colour was measured using the CIELAB computer colour-matching technique under illuminate D64 and U3500, and the results obtained are presented in Table 2.

Clearly, it may be noticed that the colour shade of the viscose fabric decreased upon the addition of polyester, as shown by the drop in the values of ΔL*, Δa*, and Δb*. Certainly, the colour of the composite material is influenced by the colour of the polyester particles, which were initially white, affecting the values of the colour coordinates determined for the composite sample.

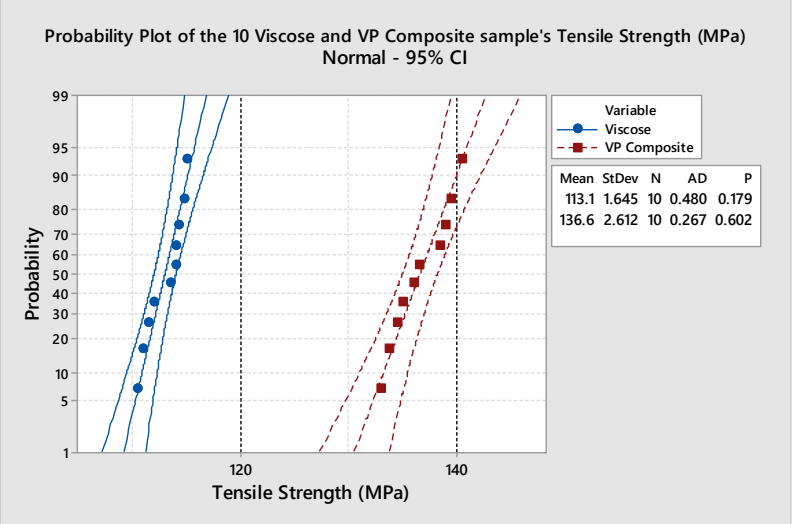

Figure 7: Weibull probability distribution plot for tensile strength

Table 2
Color characteristics of viscose and VP composite specimens

Illum/Obs	ΔL*/ISL	Δa*	Δb*	Δc*/cSc	ΔH*/SH	ΔEcmc
D65 10 Deg	-0.43	-0.37	-0.86	0.60	0.05	0.74
U3500 10 Deg	-0.59	-0.44	-1.16	0.72	0.08	0.93

CONCLUSION

This study focused on preparing viscose-based composites reinforced with PCP garment fabric and on their characterization. The test results revealed that the viscose-polyester composite was more thermally stable, with a lower weight loss compared to the initial viscose fabric. The crystallinity increased from 50.22% to 55.20% in the VP composite compared to the viscose sample. The mechanical properties of the composite were higher than those of the viscose fabric, and the tensile properties improved by almost 21%. The composite proposed in this study could serve as an alternative to commercial viscose-polyester blends considering that it contains recycled polyester waste fabrics and has good mechanical properties. The findings of the study recommend the use of waste polyester garments to be used to fabricate viscose-polyester blended composites by this new, cost-effective procedure.

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