# WASTE SILKWORM COCOON-BASED COMPOSITE COATING FOR PASSIVE RADIATIVE COOLING

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***ABSTRACT:*** *Personal thermal management technologies based on passive radiative cooling materials has attracted attention thanks to the great potential in reducing energy consumption with low carbon emission. Today, endowing common fabrics with radiative cooling capability especially using simple/scalable production method and also renewable and degradable radiative cooling materials, is an effective strategy. In this study, viscose (CV) nonwoven fabrics were coated by silk fibroin (SF) particles-based composite having high mid- infrared emissivity and solar reflectance, from waste silkworm cocoons through simple spraying method. Besides SEM and FT-IR analysis, solar reflectance and passive cooling performance under simulated conditions were measured by UV-VIS-NIR spectrometer integrated with a diffuse integrating sphere and self-made hotplate system, respectively. Also, wearability performance of the fabrics was determined by measuring physical, mechanical, and permeability performance. Coated especially SF composite coated CV fabrics enable the achievement of cooling performance with an average temperature drop of 6.4°C and 20.22°C, respectively compared to reference and bare skin as a result of high infrared emittance arising from abundant chemical bonds and also solar reflectance around 69.43%. Besides passive cooling performance, these coated fabrics had convenient physical, mechanical, and air permeability performance for practical usage. Thanks to combination passive cooling and wearability performance through upcycling of waste silkworm cocoons towards to SF particle, coated CV nonwoven would be a sustainable textile for passive personal thermal management.*

***Keywords:*** *Silk fibroin, personal thermal management, passive radiative cooling, upcycling, viscose nonwoven, thermoplastic polyurethane.*

# KOMPOZITNI PREMAZ NA BAZI ČAURE SVILENE BUBE ZA PASIVNO RADIJATIVNO HLAĐENJE

**APSTRAKT:** *Tehnologije ličnog termalnog upravljanja zasnovane na pasivnim materijalima za radijativno hlađenje privukle su pažnju zahvaljujući velikom potencijalu u smanjenju potrošnje energije uz nisku emisiju ugljenika. Danas je efikasna strategija davanje uobičajenim tkaninama mogućnosti radijativnog hlađenja, posebno korišćenjem* *jednostavnih/skalabilnih metoda proizvodnje, kao i obnovljivih i razgradivih materijala za radijativno hlađenje. U ovoj studiji, netkane tkanine od viskoze (CV) su presvučene kompozitom na bazi čestica svilenog fibroina (SF) sa visokom emisivnošću u srednjem infracrvenom zračenju i solarnom refleksijom, dobijenim iz otpadnih čaura svilene bube jednostavnim metodom prskanja. Pored SEM i FT-IR analize, solarna refleksija i performanse pasivnog hlađenja u simuliranim uslovima merene su UV-VIS-NIR spektrometrom integrisanim sa difuznom integrišućom sferom i samostalno napravljenim sistemom grejnih ploča. Takođe, performanse habanja tkanina su određene merenjem fizičkih, mehaničkih i performansi propustljivosti. Prevučene, posebno SF kompozitne CV tkanine sa prevlakom omogućavaju postizanje performansi hlađenja sa prosečnim padom temperature od 6,4◦C i 20,22◦C, respektivno, u poređenju sa referentnom i golom kožom, kao rezultat visoke infracrvene emisije koja nastaje usled obilnih hemijskih veza, a takođe i solarne refleksije oko 69,43%. Pored pasivnih performansi hlađenja, ove presvučene tkanine imale su pogodne fizičke, mehaničke i performanse propustljivosti vazduha za praktičnu upotrebu. Zahvaljujući kombinaciji pasivnog hlađenja i performansi nošenja kroz reciklažu otpadnih čaura svilene bube u SF čestice, presvučeni CV netkani materijal bio bi održivi tekstil za pasivno lično upravljanje toplotom.*

***Ključne reči:*** *Svileni fibroin, lično upravljanje toplotom, pasivno radijaciono hlađenje, reciklaža, viskozna netkana tkanina, termoplastični poliuretan.*

## 1. INTRODUCTION

Nowadays, next-generation textile materials providing personal thermal-moisture management with low carbon emission has attracted attention by taking into account the environmental concerns due to global warming and extreme heat weather [1]. In this manner, passive cooling textiles using common fabrics with scalable materials and methods have emerged as a promising solution owing to sustainable and energy-free cooling technologies [2]. To get efficient temperature reduction hence cooling performance, these materials should have both higher solar reflectance (0.3-2.5 µm) and mid-infrared emissivity (8-13 µm) without any energy input [1]. In this regard, many researchers have been developed numerous materials in a wide variety of forms, including inorganic materials [3], multilayer photonic crystal/coating [4, 5], inorganic/polymer hybrid composite materials [1]. On the other hand, most of these materials were manufactured using complicated and costly process which makes these materials unsuitable for large-scale production. At this point, imparting common fabrics with radiative cooling capability draws attention as a high potential solution method in recent years [6] by applying mid-infrared emissive polymers and/or solar reflective particles. However, using of harmful organic solvents and also complex process restricts the use of these materials. Lately, some natural polymers, including cellulose [7] and silk [6] have gained popularity for developing environmentally friendly passive cooling materials.

Silk provides an alternative sustainable option for developing passive radiative cooling materials with its higher mid-infrared emissivity arising from protein backbone (i.e., amide groups) of SF protein and also solar reflectance as well as low cost and biodegradability [1, 6]. SF-based passive cooling materials could be developed using electrospinning [8-10] and imparting to common fabric by coating method [6]. In this study, the potential of upcycling of waste silkworm cocoons towards to SF particle and using as composite coating with thermoplastic polyurethane (TPU) polymer onto common CV nonwoven fabric through simples spraying method to impart passive cooling based thermal management.

## 2. MATERIALS and METHODS

### 2.1. Materials

CV nonwoven fabric (Spunlace, 65 g/m$^2$, 0.6 mm, Mogul Tekstil, Türkiye) which a kind of renewable cellulose having good breathability, dyeability, and smooth features. Waste silkworm cocoons were provided from Umurbey Silk Production and Design Center in Bursa, Türkiye. Sodium carbonate ($Na_2CO_3$) (ZAG Chemical, Türkiye), anhydrous calcium chloride ($CaCl_2$) (Merck, Germany), and ethanol (EtOH) (Merck, Germany) were used without purification for fibroin extraction. TPU pellets (Elastollan 1185A) were supplied by BASF Co., Ltd. N, N-Dimethylformamide (DMF) (Merck, Germany) and acetone (Merck, Germany) were as solvent and co-solvent, respectively. All chemicals and materials were used as received without further purification.

### 2.2. Silk fibroin extraction and particle preparation

To prepare SF particles from waste silkworm cocoon, firstly silk cocoons were broken into pieces and cleaned from the dead silkworm residues. Afterwards, the cleaned silkworm cocoon shells were boiled in 0.5 wt.% $Na_2CO_3$ solutions at 95°C for 1 h (liquor ratio: 70:1) to extract sericin, to remove the sericin, followed by separating the silk fiber through washing in deionized water at 60°C for three times and drying at 50°C. For the preparation of SF particles, the degummed silk fibers were dissolved in $CaCl_2$:EtOH:$H_2O$ solution with a molar ratio of 1:2:8 at 60°C for 6 h. Finally, the obtained dispersions of SF particles were centrifuged at 10.000 rpm for 15 min and samples were previously freeze-dried at -80°C and lyophilized at - 55°C for 72 h (Labconco Centrivap Micro IR). The obtained particles milled into micrometer diameter by milling into powder using a micro powder pulveriser (Fritsch Pulverisette, Germany), then sieved through a 40 µm sieve. These SF microparticles were kept at 4°C until using.

### 2.3. Preparation of silk fibroin composite coated fabrics

The coated fabrics were prepared by simple spraying and drying process using CV nonwoven fabrics and TPU-SF particle solutions. For TPU and also TPU-SF coating solutions, firstly, a 10 wt.% TPU solution was obtained by dissolving TPU pellets in DMF: acetone binary solvent system with a ratio of 1:1 using mechanical stirrer at 60°C, 6 h, and 400 rpm. Subsequently, fibroin particles at a rate of 10% of the polymer was added into TPU solution and mixed mechanical stirring for another 1 h followed ultrasonic mixing for 30 min. The resulted TPU and TPU-SF composite solutions were sprayed onto the CV nonwoven fabric surface using a simple spraying system (Alfajet W-77S, Türkiye) at a pressure of about 0.3 MPa and a distance of 50 cm with a solution amount of 0.01 mL/cm$^2$. At last, coated fabrics were dried at room temperature for 24 h. The samples were coded as

CV (reference), CV-TPU (TPU coated ones), and CV-TPU-SF (TPU-SF composite coated ones).

### 2.4. Characterizations

Scanning electron microscopy (SEM) was conducted with a Fei Quanta FEG 250 instrument at an acceleration voltage of 20 kV, 10 µA current, and 500-1000 magnification for observing surface morphology of the refence and coated CV fabrics. Fourier transform infrared (FT-IR) spectra were recorded using a JASCO FT/IR 4700 spectrometer equipped with a diamond ATR (Attenuated total reflectance) in the 400-4000 $cm^{-1}$ range, 2 $cm^{-1}$ resolution, and 16 scan at room temperature. Also, spectral solar reflectance and transmissivity of the samples in the wavelength range of 190-2700 nm were recorded on a UV-VIS-NIR spectrometer (JASCO V-770 UV-Vis-NIR) integrated with a diffuse integrating sphere. The average values of solar reflectance were calculated according to Equations (1).

$$\bar{\rho}_{solar} = \frac{\int_{0.3\ \mu m}^{2.5\ \mu m} I_{solar}(\lambda)\cdot\rho(\lambda)\mathrm{d}\lambda}{\int_{0.3\ \mu m}^{2.5\mu m} I_{solar}(\lambda)\mathrm{d}\lambda} \qquad (1)$$

Passive cooling performance under indoor simulation of the samples was determined by recording the temperature changing of the lower surface of the samples using a self-made hotplate system simulating skin with heating sheet, 8 K-type sensors, a sun simulator halogen lamp (600 W $m^{-2}$), and a data acquisition system. After 1 min stabilizing period, temperature data for cooling performance of the samples placed on the hot plate under lamp’ switched-on and switched-off period for 30 min, respectively via recording one data per ten second. The average temperature variation on the lower surface of the sample were determined by performing least three replications for each sample.

A universal material tensile tester (Llyod Instruments LR5K+) was used to determine mechanical properties of the reference and coated fabric with test length and speed of 75 mm and 100 mm/min in uniaxial tests mode according to ASTM D5035-95 (2003) standard method. Moreover, physical (weight) properties were determined according to ASTM D3776/D3776M-20 to evaluate coating effect. Air permeability of the fabrics was tested using a FX Textest 3300 (James Heal Corp., UK) automatic fabric permeability meter according to TS 391 EN ISO 9237 standard under 200 Pa pressure with at least ten replicates. All test results were statistically evaluated by SPSS 26 for Windows statistical software (IBM, Armonk, USA) using Tukey post hoc test. *p* value less than 0.05 was deemed to be significant for all test results.

## 3. RESULTS

In this study, the potential of obtaining SF particles from waste silk cocoons and using as coating material to endow common CV nonwoven fabric with passive radiative cooling performance due to the high infrared emittance and solar reflectance characteristic, were investigated. Microstructure and also surface morphology of the reference as well as coated

CV nonwoven fabrics with TPU and TPU-SF composite, were visualized using SEM analysis and images were shown in Figure 1. As seen in Figure 1 ($a_1$) and ($a_2$), the reference CV nonwoven fabric had a smooth, porous, and open network structure with fiber entanglement. TPU and especially TPU-SF composite coating resulted in rougher surface structure, which can be seen in Figure 1 ($b_1$)-($b_2$) and ($c_1$)-($c_2$), respectively. Through surface coating of the CV fabrics by simple spraying method, TPU solution formed very thin and more blurred coating while TPU-SF particle composite solutions created more thicker coating on the fabric surface and among fiber/yarns. Also, there are many bright spots on the surface of the CV-TPU-SF ones owing to SF particles. SF particles and particle agglomeration resulted in rougher surface can be seen in surface of CV-TPU-SF ones (Figure 1 ($c_1$)-($c_2$)). More rougher surface structure in coated especially TPU-SF composite coated samples could allow sunlight reflection and mid-infrared emission thereby passive cooling based thermal management.

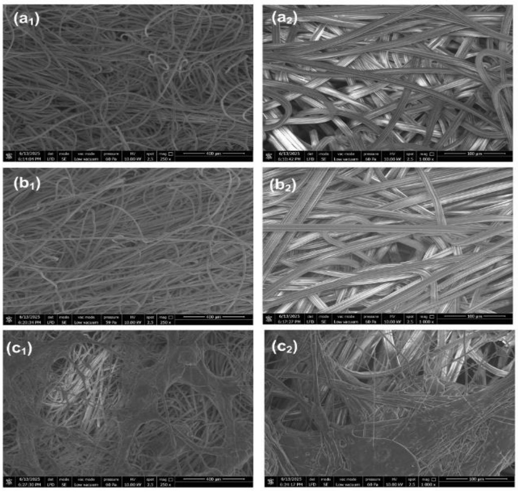

**Figure 1:** Surface SEM images of the reference CV ($a_1$)-($a_2$), CV-TPU ($b_1$)-($b_2$), CV-TPU-SF ($c_1$)-($c_2$) at 250x and 1000x magnification

In order to evaluate molecular structure and also molecular vibration-based selective mid-infrared wavelength emission inside the atmospheric transparency window, the functional groups of the samples were analyzed using FT-IR spectra. Based on Kirchhoff's Law of thermal radiation, materials which have molecular vibration overlapping the atmospheric transparency window (8-13 μm) corresponded exactly to the absorption at 1250-780 $cm^{-1}$, could exhibit selective mid-infrared emission hence passive cooling performance. As seen in Figure 2, the stretching vibrations of the N-H bond, alkene-CH stretching vibrations, and C–O–C stretching vibrations were detected at 3578-3348 $cm^{-1}$, 2922 and 2855 $cm^{-1}$, and 1074 $cm^{-1}$ which are characteristic peak of TPU. In addition to these peaks, TPU polymer had two characteristic peaks at 1731 and 1701 $cm^{-1}$ are attributed to the free C=O and H-bonded C-O, in turn. The characteristic peak of SF particle such as amide I, amide II, and amide III were observed at 1651-1593, 1535, and 1233 $cm^{-1}$, arising from C-N stretching, C-N and N-H deformation, respectively [11, 12]. Also, characteristic peak of polypeptide corresponding to peptide bands were observed in the range 800-1200 cm$^{-1}$. -OH of alcoholic and carboxylic and -C = O bands were represented at 3200-3500 and 1655 cm$^{-1}$ in the IR spectrum of the reference CV fabrics. The coated especially TPU-SF composite coated samples exhibit strong infrared absorption due to the mentioned molecular vibration peaks overlapping the atmospheric transparency window contribute to higher thermal emittance thereby passive cooling performance.

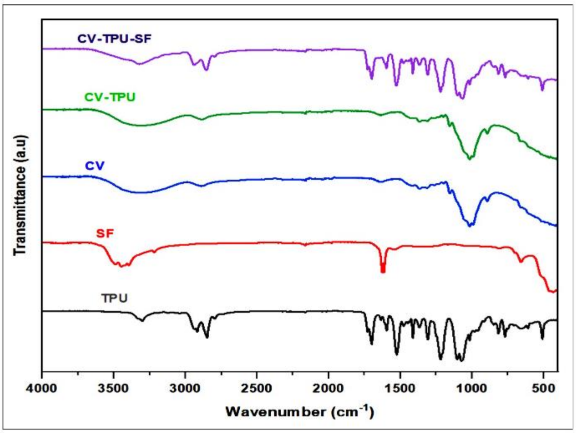

**Figure 2:** FT-IR spectra of TPU polymer, SF particle, reference and coated CV fabrics

High solar reflectance and also emissivity from atmospheric windows are fundamental parameters for achieve efficient passive cooling performance in outdoor thermal management. In order to evaluate solar reflectance performance of the samples, solar reflectance spectra of the samples in corresponding wavelength were measured and average solar reflectance values are calculated. As shown in Figure 3, TPU and TPU-SF composite coating enhances the solar reflectance of the CV nonwoven fabric. The reference CV nonwoven samples showed an average reflectance of 51.23% as this value increased with TPU and reached up to maximum value of 69.43% in CV-TPU-SF ones. Rougher surface and also SF particles on the fabric surface as seen in related SEM images resulted in sufficient reflection and Mie scattering of sunlight [1, 6]. The average solar reflectance value obtained through coating especially including SF particles is consistent with similar study about coating of cotton fabric with only SF particles [6]. These results indicate that SF particles have enhanced solar reflectance and mid-infrared emissivity hence passive radiative cooling performance.

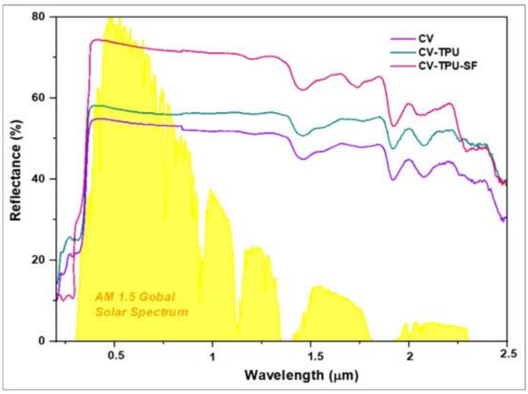

**Figure 3:** Solar reflectance spectrum of the reference and coated CV fabrics

Passive cooling performance of the samples were measured using a designed hotplate system with heating sheet which simulating human MIR emission, 8 K-type sensors for recording the real-time temperature of the samples, halogen lamp (600 W m$^{-2}$) simulating sunlight exposure, and a data acquisition system. Real time temperature graphic and also temperature variation of the samples between each other and also bare skin are given in Figure 4, Table 1, and Table 2, respectively. As seen in Figure 4 and Table 2, the temperature of the all samples gradually increased and reached maximum for each sample after 30 min of sunlight exposure. Thanks to the higher solar reflectance and mid-infrared radiation emission, CV-TPU-SF samples finally displayed a temperature of 49.86°C, lower than the all other fabric samples (54.1°C and 56.28°C for CV-TPU and reference CV, respectively) and also bare skin (70.08°C). Under a solar intensity of 600 W m$^{-2}$, passive cooling effect were detected for TPU and TPU-SF composite coating with a temperature drop of 2.18 and 6.41°C (Table 1), in turn compared to reference ones. Compared to smooth fiber surface in reference fabrics, rougher fiber surface in TPU and TPU-SF composite coated ones as seen in SEM images resulted in higher solar reflection and Mie scattering hence passive cooling performance. Although, the average reflectance values of the TPU and TPU-SF composite coated samples are close, the temperature drop, hence the cooling effect enhanced with SF particle up to 6.41°C thanks to higher mid-infrared emission based on strong molecular vibration peaks such as amide I, amide II, and amide III arising from N-H, C=O, and C-O in SF molecules [6, 8] overlapping the atmospheric transparency window (as seen in FT-IR spectra) as well as solar reflectance. The same samples exhibited lower temperature by an average of 20.22°C as compared to the bare skin simulating the body without clothing under simulated conditions, which is higher than differences of reference and TPU coated samples from bare skin with lower temperature value of 13.80°C and 15.98°C (Table 1), respectively. The temperature drop obtained in the mentioned sample is higher than the studies using SF particles for thermal management [6, 8]. In a passive radiative cooling material, the minimum temperature increase under the lamp simulating solar ($\Delta T_{solar}$) is targeted, while the maximum temperature decrease ($\Delta T_{shadow}$) after the lamp is turned off is the ideal situation for it to cool down as quickly as possible. As seen in Table 2, CV-TPU-SF samples had the minimum $\Delta T_{solar}$ and maximum $\Delta T_{shadow}$

with value of 16.36 and 49.86, respectively as well as lower temperature values. These results indicate that coated fabrics especially with TPU-SF composite have enhanced solar reflectance and mid-infrared emission, which is beneficial for personal thermal management through passive radiative cooling.

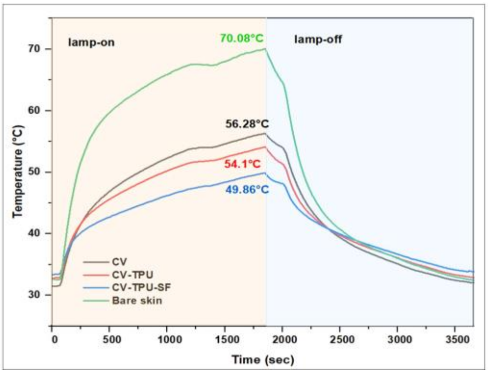

**Figure 4:** Real-time temperature curves of the films and bare skin.

**Table 1:** Comparative cooling performances of the reference and coated CV fabrics

| **$\Delta T_{max}$ (°C)** | | |
|---|---|---|
| **1-2** | **1-3** | **2-3** |
| 2.18 | 6.41 | 4.24 |
| **Bare skin-fabric samples** | | |
| 13.80 | 15.98 | 20.22 |

*1: Reference CV nonwoven, 2: CV-TPU, 3: CV-TPU-SF

**Table 2:** Temperature changes of the reference and coated CV fabrics under sunlight exposure and shadow

| **Sample** | **$\Delta T_{max}$ (°C)** | | | |
|---|---|---|---|---|
| | **CV** | **CV-TPU** | **CV-TPU-SF** | **Bare skin** |
| $T_0$ | 31.54 | 32.87 | 33.46 | 32.51 |
| $T_{maximum}$ | 56.27 | 54.10 | 49.86 | 70.08 |
| $\Delta T_{solar}$ | 24.67 | 21.16 | 16.36 | 34.43 |
| $T_{end}$ | 32.03 | 32.89 | 33.83 | 32.47 |
| $\Delta T_{shadow}$ | 56.53 | 54.04 | 49.86 | 69.94 |

To evaluate wearability performance of the samples in practical applications as a clothing or medical textile materials; physical, mechanical, and permeability tests were conducted. Weight (physical) values and mechanical properties of the reference and coated CV fabrics were given in Table 3. According values in Table 3, TPU and TPU-SF composite coating resulted in significantly ($p$=0<0.05) weight increasing and the maximum values belonged to the coated samples with TPU-SF composite coating. Conversly, the coating type such as TPU or TPU-SF composite ones did not effect sample weight. Mechanical performance such as tensile stress-strain properties were examined and the effect of coating with TPU and TPU-SF composite was summarized in Table 3 and Figure 5, respectively. As shown in Table 3 and Figure 5(a), the tensile strength values of the samples in both direction increased significantly via TPU and TPU-SF composite coating. In MD, the maximum tensile strength values belonged to CV-TPU-SNF samples with 92.76 N while for CD direction this value was was 21.45 N and belonged to CV-TPU ones. In both direction, the minimum tensile strength values detected in reference CV ones. TPU and TPU-SF composite coating increased the mechanical strength of the samples by chemical interaction of hydroxyl on the amino acid of the SF molecular chain with the amino (-NH) from TPU as seen in FT-IR spectrum. Also, strong interfacial interaction with fabric surface by developing more hydrogen bonds with the TPU matrix due to the hydroxyl groups in SF particle structures enhanced the mechanical properties. On the other hand, elongation values at break reduced with TPU while increased with TPU-SF composite coating in the both direction as compared to the reference CV samples (Figure 5(b)). Consequently, the resulting composite fabrics coated with TPU and TPU-SF composite, had convenient mechanical performances.

**Table 3:** Physical and mechanical properties of the efference and coated CV nonwoven fabrics

| Sample | Weight ($g/m^2$) [S.D] | Tensile strength, MD (N) [S.D] | Tensile strength, CD (N) [S.D] | Elongation, MD (%) [S.D] | Elongation, CD (%) [S.D] |
|---|---|---|---|---|---|
| **CV** | $64.96^{a}$<br>[1.69] | $64.06^{a}$<br>[2.44] | $15.11^{a}$<br>[0.84] | $17.22^{b}$<br>[0.89] | $86.62^{ab}$<br>[3.42] |
| **CV-TPU** | $79.34^{b}$<br>[0.94] | $69.92^{b}$<br>[3.74] | $21.45^{c}$<br>[1.82] | $12.36^{a}$<br>[0.55] | $83.10^{a}$<br>[4.09] |
| **CV-TPU-SF** | $80.48^{b}$<br>[3.97] | $92.76^{c}$<br>[4.55] | $18.10^{b}$<br>[1.67] | $17.88^{b}$<br>[2.34] | $90.72^{ab}$<br>[3.35] |

*: Different superscript letters show statistically significant differences

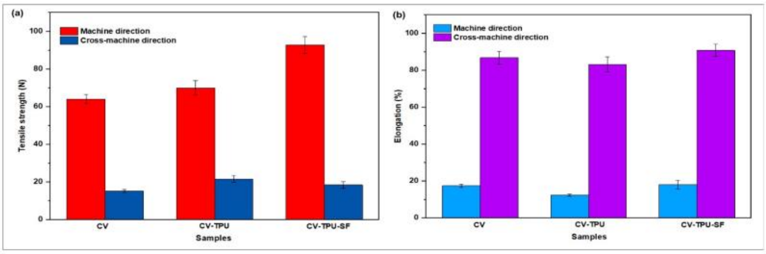

**Figure 5:** Mechanical properties of the reference and coated CV fabrics, tensile strength (a) and elongation values (b) at break.

As an another parameter of wearability and also permeability, air permeability of the samples evaluated and results were shown in Figure 6. As seen in Figure 6, air pemeability performance of the CV nonwoven fabrics significantly reduced with TPU and TPU-SF composite coating and minimum values were determined in CV-TPU ones (1313.18 l/m$^2$/s) thanks to the filling pores of nonwoven fabric with TPU polymer as can be seen in SEM inmages. Although the air permeability performance decreases with the TPU-SF composite coating, it is higher than TPU coated ones with value of 1618.64 l/m$^2$/s. Based on the values and performance metrics, it can be drawn that the physical, mechanical property, and air permeability of the CV nonwoven fabrics were found to be affected by TPU and TPU-SF composite coating owing to the filling of some interspaces between fibers by the coating.

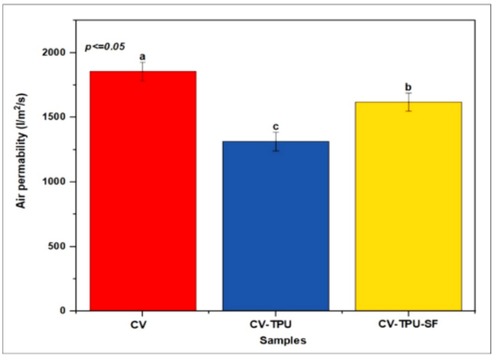

**Figure 6:** Air permeability of the reference and coated CV nonwoven fabrics.

## 4. CONCLUSION

In summary, eco-friendly passive radiative cooling fabrics are developed by coating of the CV nonwoven fabrics using TPU polymer and SF particles regenerated from waste silkworm cocoons, through simple spraying method for thermal management. Owing to advantages of the high infrared emittance and also solar reflectance of the SF particles, coated especially TPU-SF composite coated CV fabrics enables the achievement of cooling performance with an average temperature drop of 6.4°C and 20.22°C, respectively compared to reference and bare skin under simulated condition using 600 Wm$^{-2}$ solar irradiance. Also, TPU-SF composite coated CV fabrics had convenient mechanical and breathability performances ensuring its potential for applications in the field of personal thermal management. Besides its passive cooling performance and wearability features, upcycling of waste silkworm cocoons towards to SF particle and also using as coating material attracts attention as a sustainable production method for passive personal thermal management.

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