



BIOMASS WASTE-DERIVED COMPOSITE AEROGEL MATERIALS FOR SUSTAINABLE PASSIVE RADIATIVE COOLING

Scientific paper
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ABSTRACT: Passive radiative daytime cooling (PRDC) materials attract attention as today's innovative materials by mitigating global warming and lowering energy consumption without any energy input. However, the petroleum-based non-biodegradable sources of the polymers creates a negative burden on the environment. In this study, a sustainable and efficient PDRC composite aerogel was developed through freeze-drying using silk fibroin (SF) from waste silkworm cocoons, cellulose nanofiber (CNF) as the matrix components and aluminium oxide (Al_2O_3) as the particle. Solar reflectance and atmospheric window emissivity, hence cooling performance were enhanced by incorporating composite aerogel structure with Al_2O_3 nanoparticles at varying content (10 and 20 wt.% of matrix components). Besides morphological and chemical structure analysis using SEM and FT-IR, optical features such as radiative emissivity and solar reflectance were determined by FT-IR and UV-VIS-NIR spectrometer integrated with a diffuse integrating sphere, respectively. Cooling performances were determined under simulated conditions by a designed hotplate system including sun-simulator a halogen lamp and outdoor real-life tests. Results show that SF@CNF-20 Al_2O_3 exhibited the maximum cooling performance with 12.25°C and 4.14°C temperature drops for bare skin and cotton fabric according to hotplate test results. Moreover, SF@CNF-10 Al_2O_3 had the maximum outdoor cooling performance of 9.4°C and 4.1°C compared to bare skin and cotton fabric, respectively. These results suggest a sustainable and efficient cooling material through upcycling of waste silk cocoons and CNFs derived from renewable biomass with Al_2O_3 nanoparticles.

Keywords: Radiative cooling; fibroin; cellulose nanofiber; aluminium oxide; upcycling.

KOMPOZITNI AEROGEL MATERIJALI DOBIJENI IZ OTPADA BIOMASE ZA ODRŽIVO PASIVNO RADIJATIVNO HLADENJE

APSTRAKT: Materijali za pasivno radijativno dnevno hlađenje (PRDC) privlače pažnju kao današnji inovativni materijali ublažavanjem globalnog zagrevanja i smanjenjem potrošnje energije bez ikakvog ulaganja energije. Međutim, nebiorazgradivi izvori



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polimera na bazi nafte stvaraju negativno opterećenje za životnu sredinu. U ovoj studiji, održiv i efikasan PDRC kompozitni aerogel je razvijen putem liofilizacije korišćenjem svilenog fibroina (SF) iz otpadnih čaura svilene bube, celuloznih nanovlakana (CNF) kao komponente matrice i aluminijum oksida (Al_2O_3) kao čestice. Solarna refleksija i emisivnost atmosferskog prozora, a samim tim i performanse hlađenja, poboljšane su ugradnjom kompozitne aerogelevne strukture sa Al_2O_3 nanočesticama u različitim sadržajima (10 i 20 tež.% komponente matrice). Pored analize morfološke i hemijske strukture korišćenjem SEM i FT-IR, optičke karakteristike kao što su radijaciona emisivnost i solarna refleksija određene su FT-IR i UV-VIS-NIR spektrometrom integrisanim sa difuznom integrišućom sferom, respektivno. Performanse hlađenja određene su u simuliranim uslovima pomoću projektovanog sistema grejne ploče, uključujući simulator sunca, halogenu lampu i testove na otvorenom u realnim uslovima. Rezultati pokazuju da je SF@CNF-20 Al_2O_3 pokazao maksimalne performanse hlađenja sa padovima temperature od 12,25°C i 4,14°C za голу kožu i pamučnu tkaninu, prema rezultatima testa grejne ploče. Štaviše, SF@CNF-10 Al_2O_3 je imao maksimalne performanse hlađenja na otvorenom od 9,4°C i 4,1°C u poređenju sa golom kožom i pamučnom tkaninom, respektivno. Ovi rezultati ukazuju na održiv i efikasan materijal za hlađenje kroz reciklažu otpadnih svilenih čaura i CNF-ova dobijenih iz obnovljive biomase sa Al_2O_3 nanočesticama.

Ključne reči: Radijativno hlađenje; fibroin; celulozna nanovlakna; aluminijum oksid; reciklaža.

1. INTRODUCTION

The escalating global energy demand, particularly for cooling applications in buildings and wearable systems, has intensified the search for passive thermal management strategies that require no energy input. PRDC has emerged as a highly promising approach, leveraging the Earth's atmospheric transparency window (8-13 μm) to dissipate heat directly into the cold outer space as infrared radiation, thereby achieving sub-ambient cooling without any electricity consumption [1]. A suitable radiative cooling material must simultaneously exhibit high solar reflectance in the UV-Vis-NIR range (0.3-2.5 μm) to minimize solar heat gain and high infrared emissivity within the atmospheric window to maximize heat dissipation [2, 3]. In recent years, many research has been conducted for developing PRDC materials by designing and optimizing emissive and solar reflective coatings using photonic crystals, metal-polymer coatings, porous polymer coatings, and textiles with nanostructures [4]. However, the fact that most of these materials are derived from petroleum-based non-biodegradable polymers creates a negative burden on the environment.

Aerogels, three-dimensional porous lightweight materials, have attracted considerable attention as a platform for engineering high-performance radiative cooling materials thanks to their low density, thermal conductivity, hierarchical porous structure, and tunable



surface features [2, 3, 5, 6]. The highly interconnected porous structure endows aerogels with excellent thermal management properties, capable of multiple scattering of incident sunlight, resulting in enhanced solar reflectance, while their large specific surface area facilitates efficient mid-infrared emission [2, 3]. However, these aerogels composed of inorganic compounds are more brittle mainly through weak interconnections between nanoclusters. These poor mechanical properties can disrupt the three-dimensional pore structure of aerogels and even severely limit their thermal performance. In contrast, although polymer aerogels have better mechanical properties, the thermal conductivity will be significantly increased, and aerogels such as polyurethane [6] and polyvinyl alcohol [7] have poor thermal insulation properties at high temperatures. Therefore, there is an urgent need to develop aerogel materials with high mechanical stability and low thermal conductivity to improve heat gain and enhance radiative cooling performance.

Silk fibroin (SF), a natural protein derived from silkworm cocoons, is an attractive biopolymer for functional material fabrication due to its abundance, biocompatibility, excellent mechanical properties, and characteristic molecular vibration bands overlapping the atmospheric transparency window. In particular, the amide I, II, and III absorption bands of SF at $1650\text{--}1230\text{ cm}^{-1}$ align closely with the $8\text{--}13\text{ }\mu\text{m}$ emission window, suggesting inherent infrared radiative properties and solar reflectance [2, 3]. However, the dissolution-regeneration process of silk fibers can greatly damage the hierarchical structure and natural properties of the fibers themselves, and even reduce the stability and radiative thermal management performance of the constructed aerogels. Therefore, there is a need to preserve mechanical toughness of the fibers and enhance entanglement or cross-linking between the fibers for developing stable and highly porous three-dimensional structure in aerogels. Cellulose nanofibers (CNFs), another bio-derived nanomaterial rich in surface hydroxyl groups [8], can serve as a co-matrix to reinforce SF-based aerogels, improving structural stability and promoting homogeneous porous morphology. Despite the individual merits of SF and CNF, their combination as a composite aerogel matrix for radiative cooling applications has been scarcely explored. Incorporating functional inorganic nanoparticles into biopolymer aerogels represents a promising strategy to further enhance both solar reflectance and mid-infrared emissivity. Among candidate materials, aluminium oxide (Al_2O_3) nanoparticles are particularly noteworthy due to their wide bandgap ($\sim 9\text{ eV}$), strong Mie scattering of solar radiation, phonon polariton resonance generating broadband absorption at $10\text{--}15\text{ }\mu\text{m}$, and chemical stability [1]. These properties make Al_2O_3 a compelling additive for synergistically boosting the radiative cooling performance of biopolymer composite aerogels.

Herein, a sustainable and efficient PDRC composite aerogel was developed using SF from waste silkworm cocoons, CNF as the matrix components and Al_2O_3 as the particle through freeze-drying. The influence of Al_2O_3 nanoparticle content on the microstructure, chemical composition, solar reflectance, mid-infrared emissivity, and passive cooling performance was systematically investigated. Both indoor simulations using hotplate system and outdoor real-life cooling tests were conducted to evaluate practical applicability. This work demonstrates a sustainable route toward high-performance, biomass-derived radiative



cooling aerogels with potential applications in thermal management for varying cooling systems.

2. MATERIALS and METHODS

2.1. Materials

Waste silkworm cocoons were kindly provided by Umurbey Silk Production and Design Center in Bursa, Türkiye. For SF extraction, sodium carbonate (Na_2CO_3) (ZAG Chemical, Türkiye), anhydrous calcium chloride (CaCl_2) (Merck, Germany), and ethanol (EtOH) (Merck, Germany) were used without purification. CNFs (Length: 1-3 μm , Dia: 4-10 nm) and Al_2O_3 particles (Alpha, Purity: 99.9+%, Size: 100 nm) were supplied from Nanografi, Türkiye. All chemicals were used as received without further purification. Deionized water (Millipore, 18.25 $\text{M}\Omega\cdot\text{cm}$) was used for preparing all aqueous solutions.

2.2. Preparation of SF and composite aerogel

For SF extraction, the cleaned waste silkworm cocoons in small pieces were degummed by boiling in 0.5 wt.% Na_2CO_3 solutions at 95°C for 1 h (liquor ratio: 70:1) then washed with deionized water at 60°C for three times and dried at 50°C. The degummed silk fibers were immersed in a $\text{CaCl}_2\text{:EtOH:H}_2\text{O}$ solution at 60°C for 6 h [9]. The separated SF fibers were obtained by centrifugal separation at 10.000 rpm for 15 min and drying. To prepare composite aerogel, SF fibers at 0.4 wt.% and 1 wt.% CNF as matrix components were dispersed in deionized water by mechanical mixing and stirring for 3 h. Subsequently, Al_2O_3 particles at varying content (10 and 20 wt.% of matrix components) were added to the mixed solution to fully dispers by additional mechanical mixing for 3 h and bath sonication for 30 min. The resultant mixtures were poured into petri dishes and rapidly frozen at -80°C for 24 h. The frozen aerogel samples were subsequently subjected to freeze-drying in a lyophilizer for 48 h. The obtained aerogel samples were names as SF@CNF and SF@CNF- $x\text{Al}_2\text{O}_3$, respectively. x denotes the concentration of Al_2O_3 particles.

2.3. Characterizations

The microstructure of the aerogel surface was visualized by a scanning electron microscope (SEM, Fei Quanta FEG 250) instrument at an acceleration voltage of 20 kV, 10 μA current, and 250-1000 magnification. Fourier transform infrared (FT-IR) spectra were recorded to analyze compositional structure of the samples by 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. The solar reflectance spectra of the aerogel samples in the UV-Vis-NIR (190-2700 nm) range were recorded using a JASCO V-770 spectrophotometer equipped with an integrating sphere. The average values of solar reflectance were calculated according to Equations (1).

$$\rho_{\text{solar}} = \frac{\int_{0.3 \mu\text{m}}^{2.5 \mu\text{m}} I_{\text{solar}}(\lambda) \cdot \rho(\lambda) d\lambda}{\int_{0.3 \mu\text{m}}^{2.5 \mu\text{m}} I_{\text{solar}}(\lambda) d\lambda} \quad (1)$$



Also, reflectance ($\rho(\lambda)$) and transmittance ($\tau(\lambda)$) in the range of atmospheric window wavelength (8-14 μm) were measured using a Fourier transform infrared spectrometer (Bruker VERTEX 70v) equipped with MIR integrating sphere in the diffuse reflectance mode. The average values of atmospheric window emissivity calculated according to Equations (2).

$$\varepsilon_{IR} = \frac{\int_{8\mu m}^{14\mu m} I_{BB}(\lambda) \cdot \varepsilon(\lambda) d\lambda}{\int_{8\mu m}^{14\mu m} I_{BB}(\lambda) d\lambda} \quad (2)$$

where ε : emissivity, ρ : reflection, τ : transmittance, and λ : wavelength, $I_{solar}(\lambda)$: global solar intensity spectrum according to ASTM G173, I_{BB} : spectral radiance of a blackbody defined by Planck's law at temperature T .

Passive cooling properties of the aerogel samples under indoor simulation were determined by recording the temperature by thermocouples of a hotplate system simulating skin with heating power of 104 W/m². The hotplate system possesses simulated skin whose temperature was set at 34±0.5°C, a sun simulator halogen lamp (600 W/m²), and a data acquisition system. After 1 min stabilizing period, temperature data of the samples were recorded with 10-second frequency during 30 min switch-on and switch-off periods. At least three replications were carried out for each sample.

Meanwhile, outdoor real-life cooling performances were tested using a home-made test setup including an aluminum foil coated polystyrene foam box in size of 20 × 20 × 12 cm³ and temperature data logger system (KIMO KTH 320). The top opening of the insulating foam box was covered with samples, and K-type thermocouples were fixed to the bottom of the samples for recording temperature with 5-second frequency. The outdoor passive cooling tests were conducted at West Campus of Süleyman Demirel University, Isparta, Türkiye (37° 49' 42" N, 30° 31' 34"E) by taking measurements from 09:40 am to 13:45 pm in July 2025. The environmental conditions of the outdoor test were determined as 18-39°C temperature, 26-47% relative humidity, and 7.6-15.1 km/h air velocity. The solar irradiance was ranged 714-815 W/m².

3. RESULTS

In this study, biomass waste-derived composite aerogel samples with SF from waste silkworm cocoons, CNF, and Al₂O₃ as the particle through freeze-drying were produced to develop efficient and sustainable passive radiative cooling material. Microstructure and surface morphology of the SF@CNF and SF@CNF-Al₂O₃ composite aerogel samples, were visualized using SEM analysis and images were shown in Figure 1. As seen in Figure 1 (a), SF@CNF aerogel structure has been successfully produced with porous geometry. Adding CNF with rich hydroxyl groups, as co-matrix material may enhance affinity of SF and improve the connection SFs, resulting in a stable aerogel hence hierarchical porous structure [3]. Adding Al₂O₃ particles into SF@CNF matrix led to an increase in pore size, which directly proportional to the amount of nanoparticles (Figure 1 (b, c)). Relatively macroporous structure in SF@CNF-Al₂O₃ composite aerogel could derived from

disrupting high entanglement between SFs and CNF based on serving abundant hydroxyl groups on the surface of them as binding sites [2, 3] for Al_2O_3 nanoparticles.

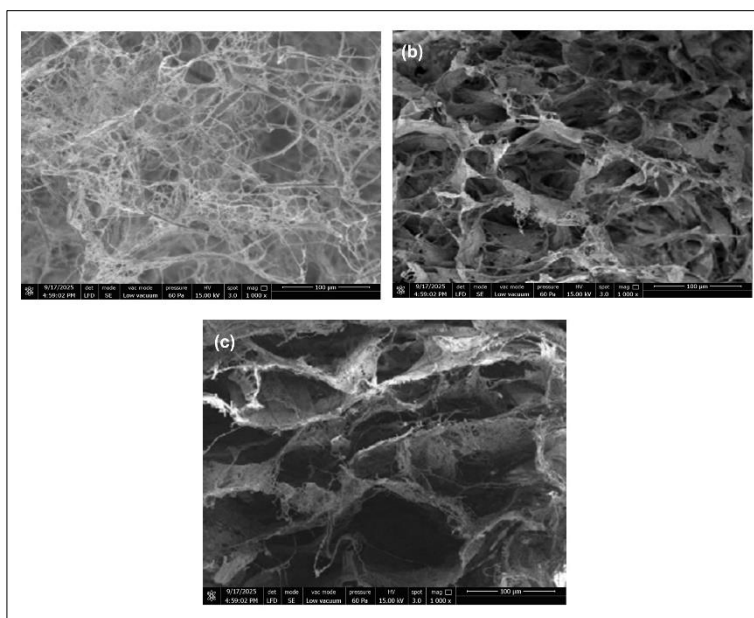


Figure 1: Surface SEM images of the SF@CNF (a), SF@CNF-10 Al_2O_3 (b), and SF@CNF-20 Al_2O_3 (c) composite aerogel at 1000x magnification

In order to evaluate the compositional structure of the samples, the functional groups were analyzed using FT-IR spectra. The infrared spectra of SF, SF@CNF, and SF@CNF- Al_2O_3 composite aerogel with varying particle content were illustrated in Figure 2. In the spectra of SF and all aerogel samples, 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 [10, 11]. Also, SF@CNF and SF@CNF- Al_2O_3 composite aerogel samples show characteristic cellulose characteristic peaks of (-OH) stretching band at 3200-3400 cm^{-1} . Although there is a noticeable difference with Al_2O_3 particles, the broad band at 665 and 845 cm^{-1} can be assigned to bending mode of O-Al-O and Al-O stretching mode, respectively in the composite aerogel samples. The resulted composite SF@CNF- Al_2O_3 aerogel samples exhibit strong infrared absorption due to the mentioned molecular vibration peaks overlapping the atmospheric transparency window (8-13 μm) corresponded exactly to the absorption at 1250-780 cm^{-1} , contribute to higher thermal emittance thereby passive cooling performance.

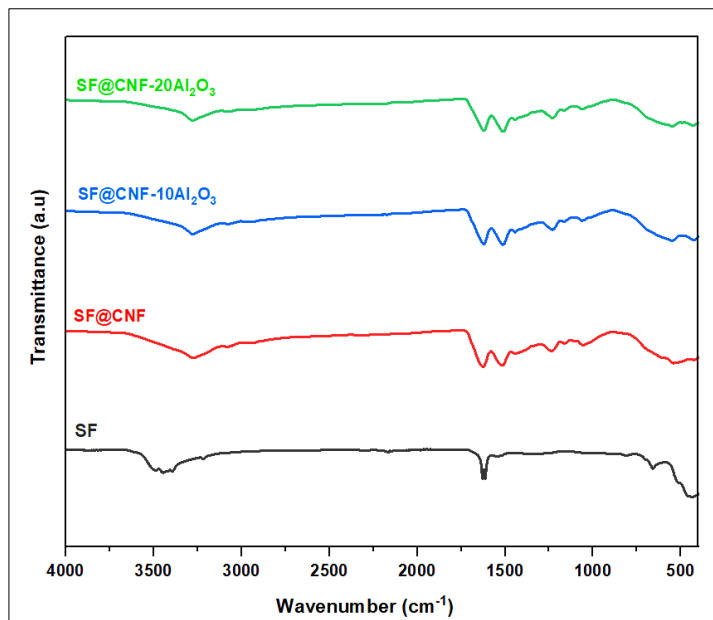


Figure 2: FT-IR spectra of degummed SF, SF@CNF, SF@CNF-10Al₂O₃, and SF@CNF-20Al₂O₃ composite aerogel

High solar reflectance and emissivity from atmospheric window are crucial for materials to get efficient passive radiative cooling. In order to evaluate solar reflectance performance, reflectance spectra of the aerogel samples in corresponding wavelength were measured and average solar reflectance values are calculated, respectively. As seen in Figure 3, Al₂O₃ particle at varying content enhanced the solar reflectance of the porous aerogel. Although the solar reflectance of the SF@CNF aerogel without particle is relatively high with an average reflectance of 88.82%, this value increased with Al₂O₃ particle and reached up to maximum value of 92.28% for SF@CNF-10Al₂O₃. The close-packed porous structure of SF@CNF aerogel without particle may resulted in relatively higher reflective interface and scattering ability inside the aerogel, thereby decreasing the thermal radiation absorption ability [12]. For composite aerogels including Al₂O₃ particle, the high light scattering properties of Al₂O₃ particles [12, 13] and porous structure of the aerogel resulted in sufficient reflection and Mie scattering of sunlight. On the other hand, increase of Al₂O₃ particle content led to decrease in the solar reflectance with a value of 91.32% for SF@CNF-20Al₂O₃, which may be due to the decrease of light scattering caused by the excess Al₂O₃ particles hindering the pore structure of the aerogel.

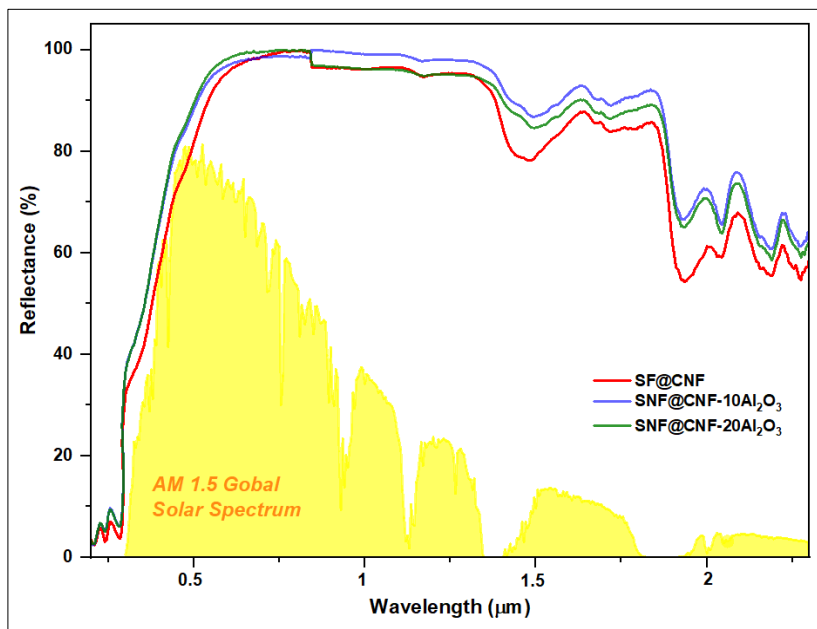


Figure 3: Solar reflectance spectrum of SF@CNF, SF@CNF-10Al₂O₃, and SF@CNF-20Al₂O₃ composite aerogel

In harmony with solar reflectance, the emissivity of the aerogel samples within the atmospheric window range (8-13 μm) enhanced with Al₂O₃ particle addition. As seen in Figure 4, SF@CNF aerogel without particle exhibited an average emissivity value of 86.63% while Al₂O₃ particle incorporation leads to an increase in infrared emissivity, reaching to a maximum value of 89.05% at nanoparticle concentration of 20%. As stated before, SF@CNF and also SF@CNF-Al₂O₃ aerogel samples had molecular vibration peaks overlapping the atmospheric transparency window (8-13 μm) corresponded exactly to the absorption at 1250-780 cm⁻¹, may resulted in higher infrared emissivity values. Compared with with similar studies including SF and CNF-based cooling materials [1-3, 12, 13], the aerogels especially SF@CNF-Al₂O₃ composites has comprehensive competitiveness, presenting the high infrared emissivity and solar reflectivity, hence passive radiative cooling performance.

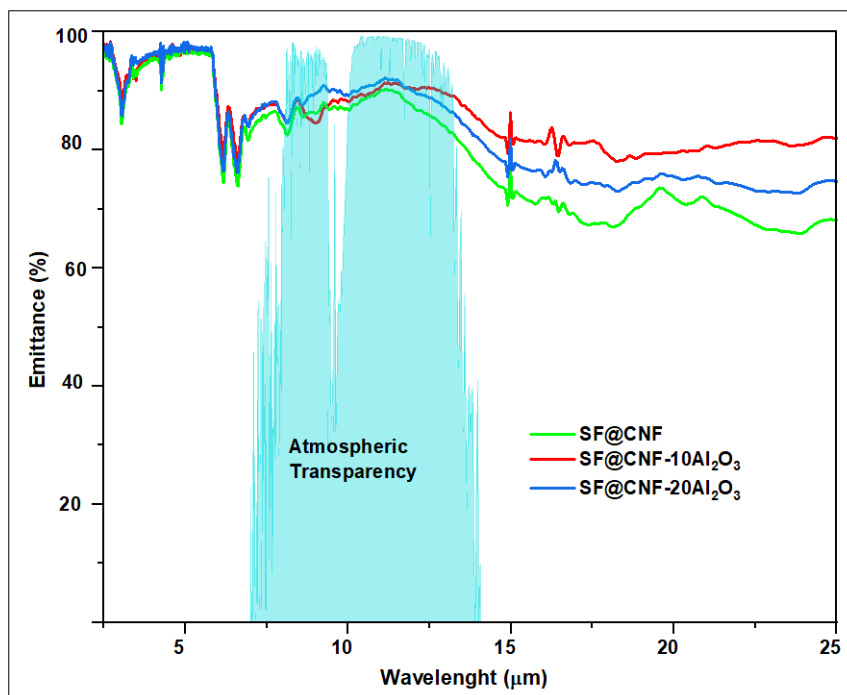


Figure 4: Mid-infrared emissivity spectra of SF@CNF, SF@CNF-10Al₂O₃, and SF@CNF-20Al₂O₃ composite aerogel

Passive cooling performances of the samples which can be calculated from the real-time temperature graphics obtained from the designed hotplate system in comparison with a single jersey cotton fabric and bare skin are given in Figure 5. According to test results, the temperature of the all samples gradually increased and reached maximum for each sample after 30 min of sunlight exposure. Benefiting from the higher solar reflectance and mid-infrared radiation emission, SF@CNF-20Al₂O₃ composite aerogel samples had a maximum temperature of 45.61°C, which is lower than all aerogel ones (49.53 and 47.87°C for SF@CNF and SF@CNF-10Al₂O₃, respectively). Under a solar intensity of 600 W/m², the maximum cooling performance of 12.25°C, 4.14°C, and 3.92°C temperature drops were detected for SF@CNF-20Al₂O₃ as compared to bare skin, cotton fabric, and SF@CNF, respectively. SF@CNF-Al₂O₃ composites, especially the one including 20% Al₂O₃ particles exhibited the maximum cooling performance thanks to its higher solar reflectance and mid-infrared emittance.

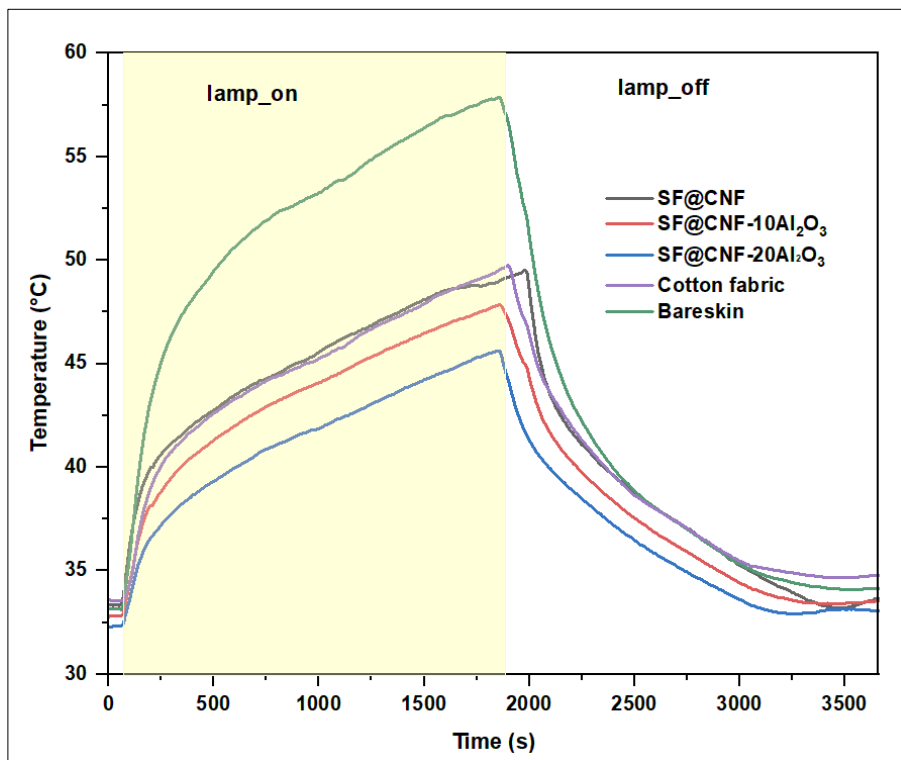


Figure 5: Real-time temperature curves of SF@CNF, SF@CNF-10Al₂O₃, and SF@CNF-20Al₂O₃ composite aerogel, cotton fabric and bare skin

The real time temperature values of the aerogel samples, cotton fabric, and bare skin, showing the outdoor cooling performances, can be seen in Figure 6. The temperature of all the samples gradually increased up to 12:15 pm and reached T_{max} for bare skin as 65°C and minimum T_{max} was recorded for SF@CNF-10Al₂O₃ as 55.6°C. T_{max} and $T_{average}$ values of the all-aerogel samples are lower than cotton and bare skin, showing their passive cooling capabilities. According to results, SF@CNF-10Al₂O₃ had the maximum cooling performance under outdoor real-life condition; namely 9.4°C and 4.1°C compared to bare skin and cotton fabric, respectively. These further reveals that SF@CNF-Al₂O₃ composite aerogel have a strong cooling effect and are expected to be a potential material for outdoor radiative cooling.

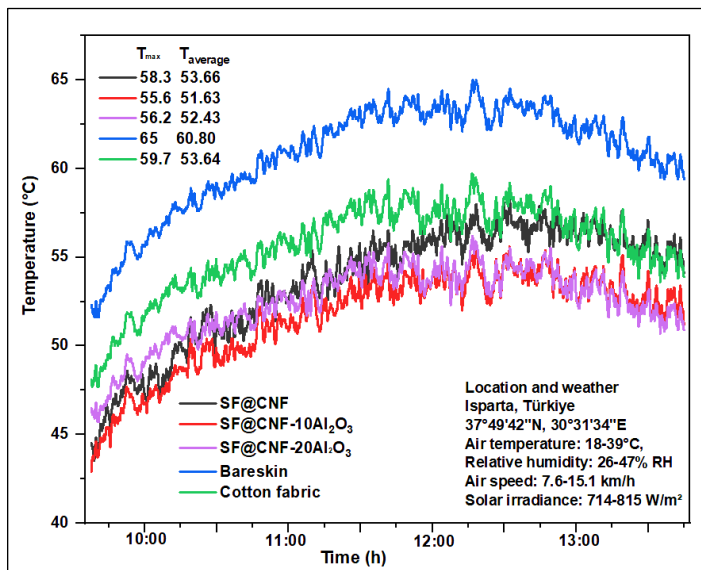


Figure 6: Outdoor-real-life cooling performance of SF@CNF, SF@CNF-10Al₂O₃, and SF@CNF-20Al₂O₃ composite aerogel, cotton fabric and bare skin

4. CONCLUSION

In this study, efficient passive cooling composite aerogels were successfully developed by combination of SF from waste silkworm cocoons-CNF and Al₂O₃ particles through freeze-drying. Solar reflectance and atmospheric window emissivity, hence cooling performance were enhanced by incorporating composite aerogel structure with Al₂O₃ nanoparticles. Under 600 W/m² solar irradiation in hotplate test, SF@CNF-20Al₂O₃ exhibited the maximum cooling performance with 12.25°C and 4.14°C temperature drops for bare skin and cotton fabric, respectively due to higher solar reflectance (92.28%) and mid-infrared emittance (89.05%). Outdoor tests demonstrated that under 714-815 W/m² solar irradiation, average temperature reduction of up to 9.4°C and 4.1°C compared to bare skin and cotton fabric, were achieved for SF@CNF-10Al₂O₃ composite aerogels. Overall, this study demonstrates that upcycling of waste silk cocoons combined with renewable biomass-derived CNF, and Al₂O₃ nanoparticles, constitutes a viable and environmentally responsible strategy for fabricating cooling materials.

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