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Mechanically Robust, Responsive Composite Membrane for a Thermoregulating Textile

Md Anwar Jahid, Jinlian Hu,* and Suman Thakur

Cite This: ACS Omega 2020, 5, 3899–3907

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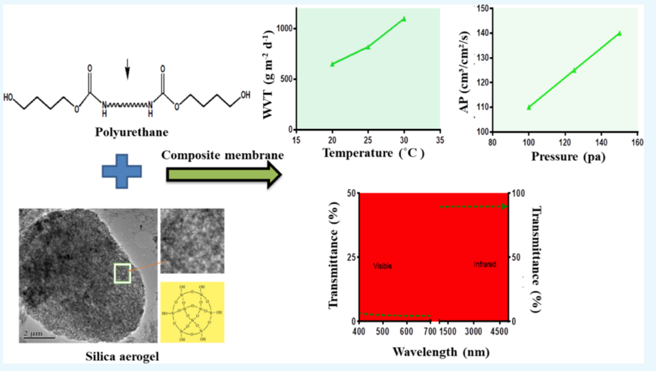
ABSTRACT: The human body releases heat via four mechanisms: conduction, convection, evaporation, and radiation. The normal core temperature of the human body is around 37 °C, and metabolism may be negatively affected and enzymes/proteins may be destroyed if the core temperature rises above 45 °C. To prevent such overheating, we developed an evaporative–radiative–convective fabric which can control the personal microclimate of the human body through a cooling mechanism (evaporation of perspiration, air convection, and emission of heat radiation directly into the environment). In this work, we fabricated a thermo–moisture sensitive polyurethane/silica aerogel composite membrane which showed super evaporative and radiative effects and which can facilitate the convection process in the human body. We also fabricated a sensitive membrane-based textile which can cool down the human body by releasing body heat. The developed material possessed robust mechanical properties for the longevity of the material, high water-evaporative ability, and air permeability to provide comfort to the wearer. Microclimate-controlled clothing can release most of our body heat to the environment.



1. INTRODUCTION

Climate change is one of the fundamental challenges for human civilization, with the current challenge being to keep global warming below a rise of 2 °C. To keep within this limit, deployment of negative emissions technologies is required.[1,2] According to the Hong Kong Observatory, climate change is making Hong Kong warmer; it was reported that the temperature and relative humidity during the summer could be around 35.4 °C and 95%, respectively. Such hot weather can have a negative impact on the human body, such as heat stroke, dehydration, elevated heart rate, and so forth.[3,4] Personal cooling technology could play a vital role in controlling body heat in hot environments.

The requirement for a personal microclimate controlling system has existed for a long time. Active cooling, passive cooling, and combo cooling are the three major types of cooling process. Combo cooling is the combination of active and passive cooling. Active cooling includes ventilated air cooling such as liquid supplies or external air connections; active cooling reduces both thermoregulatory and cardiovascular strain.[5−9] Phase-change materials act as passive cooling materials and can reduce thermal stress and improve thermal comfort.[10] Thermophysiological comfort is the ideal state between the environmental atmosphere and physical and emotional harmony of the body.[11] Thermoregulation is the biological process which keeps the body temperature within the correct range when the outdoor temperature can vary. If the human body cannot maintain a normal core temperature and it increases significantly above normal temperature, then hyperthermia can occur when the core temperature rises above 45 °C. During hyperthermia, proteins and enzymes may be destroyed, which can lead to death. On the contrary, when the core temperature decreases below normal temperature, then hypothermia occurs at a core temperature lower than 35 °C. Hypothermia can slow the metabolism process of the human body. During exercise, the body's ability of thermoregulation is affected. Metabolism (metabolism maintains the reactions which occur in the human body) produces heat as a byproduct. The human body is 25% efficient; therefore, we lose approximately 75% of energy as heat.[12,13] According to Guyton and Hall, the hypothalamus in the brain controls the body temperature.[14]

The hypothalamus responds to different temperature receptors in the body; it uses thermophysiological adjustment to maintain a comfortable core temperature. For example, when the surroundings are hot, the skin will pass the signal on to the hypothalamus to adjust the core temperature by increasing radiation through the sweat rate (evaporation), convection, and conduction. The heat exchange between the human body and the environment plays an important role in

Received: October 3, 2019
Accepted: February 4, 2020
Published: February 17, 2020

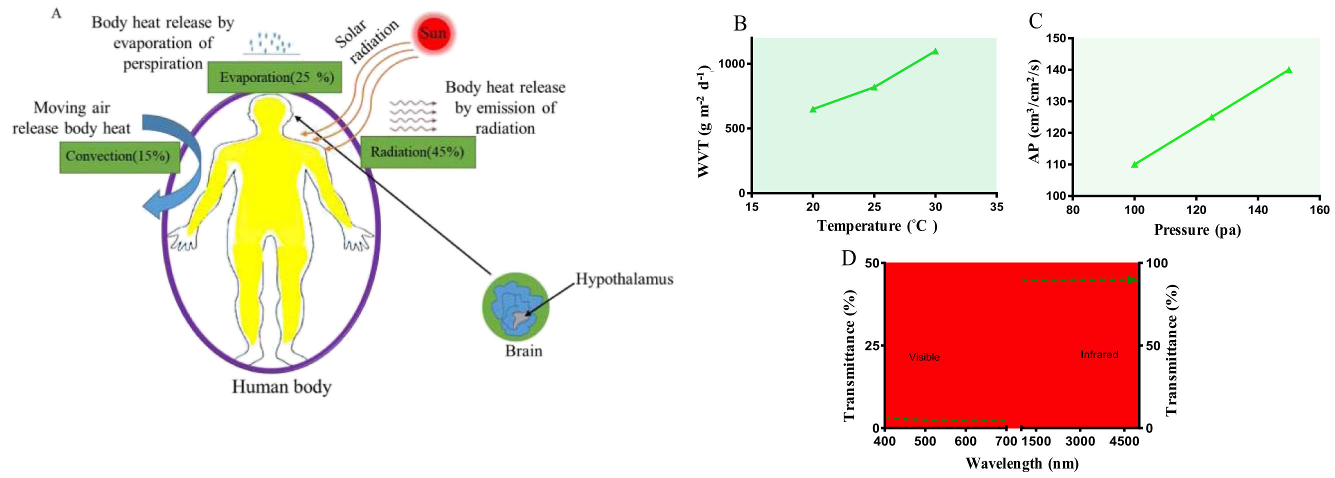


Figure 1. Thermal management of the human body. (A) Way the human body releases heat (emission of radiation, evaporation of perspiration, and releasing heat by air convection), (B) water vapor transmission (WVT) of laminated fabric, (C) AP of laminated fabric, and (D) transmittance of visible light and IR radiation by the composite membrane.

the body's thermoregulation.[15−17] The human body releases heat by evaporation, radiation, conduction, and convection. Sweat evaporation is one effective way by which to cool the human body; evaporation of sweat from the skin to the environment provides effective body cooling for individuals exposed to hot/dry environments.[18] Heat from the skin converts sweat (water) to sweat vapor, and body heat can be released by sweating. In high-intensity heat, the human body loses up to 1 L of sweat per hour. Temperature and relative humidity play a key role in controlling the sweating level. Water and electrolytes (sodium, potassium, and chloride) produce sweat; the hypothalamus in the brain senses when the core temperature is high and responds by stimulating the sweat glands to maintain a normal core body temperature. The emission of radiation is the most effective way to release heat from the skin into the environment; most of the body heat is released by radiation. Tong et al. developed an infrared (IR)-transparent−visible-opaque fabric which provides passive cooling through the transmission of thermal radiation from the body to the environment.[19] It does not provide evaporative and convective behavior, which is related to thermoregulation of the human body. Hsu et al. demonstrated nanoporous polyethylene (nanoPE) which is transparent to mid-IR human body radiation but opaque to visible light because of the pore size distribution. They then developed a textile that promotes effective radiative cooling;[20] however, the mechanical properties of nanoPE are inferior compared to those of PE or cotton. For thermal comfort of the human body, air conditioning (convection) plays a vital role. A significant amount of body heat can be transferred to the environment by convection. Convective cooling of the human body in hot weather improves thermal comfort,[21] and convective cooling facilitates the hypothalamus to control core temperature. Nowadays, most people keep their house warm in winter and cool in summer via air conditioning; however, this consumes a lot of energy.[22,23] Passive personal body cooling may be a better solution to overcome such a problem. Currently, there are several personal body cooling clothing materials available on the market. However, such clothing is fabricated for high performance applications, such as protective clothing and sports clothing, which are not suitable for regular use.[24−27]

According to the definition of thermoregulating textile, it is a textile which helps regulate the inner temperature of a textile by releasing or absorbing heat energy as the ambient temperature changes.[28−30] In this study, we mainly focused on the releasing heat of the human body through the textile as the surrounding temperature increased. Therefore; we developed an evaporative−radiative−convective fabric (ERCF) that provides passive cooling by means of evaporation of perspiration, air convection, and emission of radiation directly into the environment. The microclimate-control fabric plays a very vital role in controlling human body temperature by transferring body heat into the environment in different ways during the summer season when the surrounding temperature is too high (Figure 1A−D). One of these methods is evaporation; the prepared composite membrane was super evaporative, and most textile fibers are either water absorbent or resistant, but the ERCF we prepared is water vapor permeable. Therefore, this kind of evaporative behavior may provide evaporative cooling to the wearer. A limited range of body heat can be transported from the skin into the environment by air convection; the ERCF has air permeability (AP) behavior which can provide air convection to the human body. When there is no wind, convective cooling occurs by temperature or density difference, which can be called "free convection". Usually, convective cooling during high speed winds is better for comparing temperature or density difference. Emission of radiation is the most effective way to release body heat, transporting the heat via electromagnetic waves. Radiative cooling provides an effective way to surpass conventional ways of microclimate controlling. Radiative thermal management can be achieved by controlling transmissivity, emissivity, and reflectivity.[31−34] The ERCF provides the necessary body cooling for an individual to feel comfortable at different temperature levels. The proposed super evaporative composite membrane was prepared using a hydrophilic segmented polyurethane (PU) solution along with hollow silica aerogel. Different weight percentages of silica aerogel were added (0.5 & 1 wt %, named composite 0.5% & composite 1%, respectively). This kind of responsive PU has the capability to respond to specific changes in its surroundings (temperature and relative humidity). Hydrophilic segmented PU is composed of soft (polyol) and hard segments (di-

https://dx.doi.org/10.1021/acsomega.9b03268
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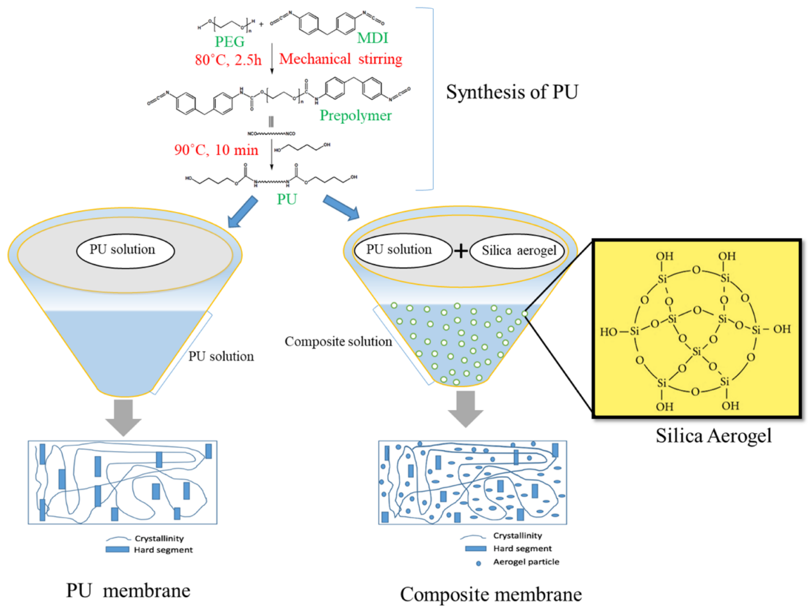

Figure 2. The fabrication process of PU and composite membranes.

isocyanate and chain extender). Soft segment (polyol) is the responsive part of the PU and the hard segment gives the PU its mechanical properties.

2. EXPERIMENTAL SECTION

2.1. Materials. 4,4-Methylene(di-isocyanate) (MDI), polyethylene glycol (PEG, molecular weight 1000 g/mol), 1,4-butanediol (BDO), and *N*,*N*-dimethylacetamide (DMAc) were purchased from Alfa Aesar (USA). Silica aerogel particles (size—10 μm) were obtained from Hengqiu Graphene Technology (Suzhou) Co., Ltd.

2.2. Synthesis of PU. Synthesis of PU was carried out in a two-step process. First, the macroglycol (PEG) was taken in a three-necked round bottom flask equipped with a thermometer, mechanical stirrer, and vacuum pump. PEG was degassed at 80 °C for 1 h under vacuum. Then, MDI was added to degassed PEG and the reaction was continued at 80 °C for 2.5 h. At the second stage, the chain extender (BDO) was added to this prepolymer and vigorously stirred to obtain segmented PU. The synthesis procedure of PU is shown in Figure 2.

2.3. Preparation of the Composite Membrane. Different percentages (0.5 and 1%) of silica particles were incorporated to fabricate the PU composite. The silica particle was added into the PU solution (DMAc was used as the solvent) and mixed by mechanical stirring for 30 min for homogeneous mixing. After that, sonication was performed in order to properly mix the silica particles in the PU matrix. The final PU composite membrane was obtained from a melt blowing machine. The thickness of the fabricated membrane was about 25−28 μm.

2.4. Fabric Details and Lamination. Scoured, bleached, and dyed 100% cotton fabrics were used in the twill weaving structure, and then, the fabric samples were cut into size 500 × 500 mm before laminating. The used fabric was −115 g/sq m and 2/2 twill fabric. The twill line can be seen from both sides of the fabric. Then these fabrics were laminated with composite membranes by using a hot press. A Digital Knight 20 16″ × 20″ digital clamshell was used for the lamination. The temperature was kept at 150 °C for the lamination and then cooled down at room temperature.

2.5. Characterization. *2.5.1. Water Vapor Transmission.* Water vapor transmission tests were conducted in a climate chamber according to ASTM E96 BW standard by using a Haida International Equipment instrument (Model-HD-E702-100-4). It is a temperature-, relative humidity-, and air velocity-controlled climate chamber. Tests were conducted at different temperatures and relative humidities. The air velocity in the chamber was 0.2−0.03 m/s. The test cup was half filled with water and the laminated sample was fixed on the top of the cup with grease. The test cup was placed into the test chamber for 4 h and then weight change was taken to calculate the WVT rate using the following eq 1.

$$\mathrm{WVT} = G/tA \tag{1}$$

where G is the weight change in grams, t is the duration of the test in hour, and A is the test area in m^2.

2.5.2. Air Permeability. To observe the breathability of the membrane, the AP testing of the laminated fabric was performed according to ASTM D737-96 by using SDL international textile testing equipment.

2.5.3. IR Transmittance. The IR transmittance of the membranes was tested by using Fourier transform IR spectroscopy with a PerkinElmer model Spectrum 100. The spectra of the membrane were recorded in the range of 2−18 and 7−14 μm.

2.5.4. Mechanical Property. The mechanical properties (strength and elongation at break) of the samples were tested according to ASTM D882 standard using an Instron 4411 (Boston, MA, USA). The samples were cut into 100 × 10 mm ($L \times W$) squares. Tests were performed at room temperature (25 °C).

2.5.5. Absorbency. The water absorbency of the membrane was measured in order to check the absorbability of different membranes. Testing time 4 h and condition were recorded. Absorbability was calculated according to eq 2

$$\text{Abosbency} = 100 - (\text{initial weight/final weight} \times 100) \tag{2}$$

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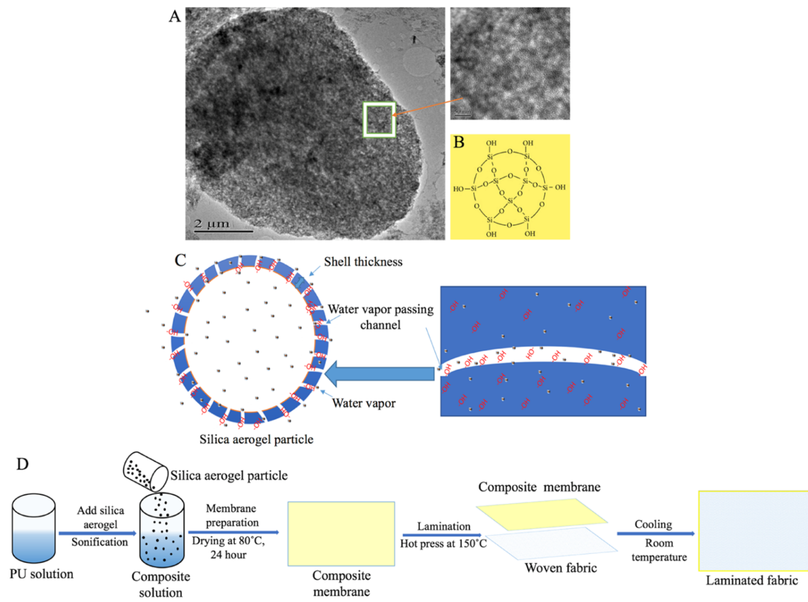

Figure 3. A) TEM image of silica aerogel particles (scale 2 μm, 50 nm), (B) structure of silica aerogel, (C) schematic of hollow silica aerogel and water vapor passing channel, and (D) the fabrication process of the laminated fabric.

2.5.6. Scanning Electron Microscopy. The surface morphology of the membrane was observed by scanning electron microscopy (SEM) with a TESCAN VEGA3. The cross-section of the above-mentioned specimens was further investigated to determine the changes in the fiber morphology and fabric properties.

2.5.7. Abrasion Resistance. For checking the hardness and adhesion properties of the laminated fabric, we have carried out a Martindale abrasion resistance test of the laminated sample according to ASTM D4966 standard; we rubbed it 1000 times (50 per min) with 9 kPa load on the upper surface, and under the lower surface, there was a plain twill fabric. After testing, we investigated the sample and found very good abrasion resistance properties, which means that it has very good adhesion properties.

2.5.8. Wicking Test. The wicking test was performed according to AAATCC TM 197. The test samples were cut into 2 cm wide stripe and dipped into distilled water. Then, the water started to climb up the testing sample due to capillary force.

3. RESULTS AND DISCUSSION

Hydrophilic silica aerogels are nanoporous and of low density with an open pore structure. Highly porous three-dimensional silica aerogel networks (Figure 3A,B) contain some extraordinary properties, such as low thermal conductivity (~0.02 W/mK) and high specific surface area (450−950 m^2/g). Porous silica aerogel contains lots of hydroxyl (−OH) groups on its backbone (Figure 3C), and such structures have led to silica aerogels being used in a wide variety of scientific and industrial applications.[33−36] After making the composite membrane, we laminated (Figure 3D) it with woven fabric (cotton). These hydroxyl (−OH) groups on the silica aerogel facilitate the achievement of water vapor permeability of the composite membrane. A self-adaptive water vapor permeability membrane has many applications such as laminated textiles, gas separation, food packaging, and wound dressing.[37,38] Porous skin covers the human body, which perspires all the time; it contains more than 60% of water at normal core temperature (36−37 °C). It would be a crucial property of a textile to have the ability to transport such perspiration from the body surface to the environment for controlling the thermophysiological comfort of the human body. Thermal comfort is controlled by body sweat in the vapor form and its transmission from inside the fabric to the environment. The sweat glands begin the production of perspiration, while heat transmission from the skin to the environment decreases. Normally, body sweat in the vapor form is termed insensible perspiration and body sweat in the liquid form is termed sensible perspiration.[11] When perspiration is transported from the skin to the environment, it contains body heat, thus adjusting the body heat level. If the water vapor transmitting ability rate is low or limited, then it increases body temperature and causes heat stress in hot environments. Water vapor permeability through fabric plays a significant role in adjusting body comfort in both hot and cold environments or during high activity levels. To provide wearing comfort, the fabric should have a high level of WVT so that body sweat can evaporate and be transmitted from the skin to the environment.

Nowadays, energy-efficient and environmentally friendly cooling systems are receiving huge attention. Air conditioning systems consume more energy, and to reduce the energy consumption of air conditioning systems, evaporative cooling can be a better alternative.[39,40] Evaporative cooling is the reduction of heat by evaporation of perspiration; this moves latent heat from the skin into the environment. Evaporative cooling is different from typical air conditioning; in evaporative cooling, the water absorbs a huge amount of latent heat during evaporation. When the core temperature of the body increases, the hypothalamus attempts to control the core temperature by releasing heat through perspiration, and body heat loss by perspiration increased with increasing temperature, while human body radiation decreased with increasing temperature and sweating.[41] To pass this perspiration from the surface of the body into the environment, we have developed an evaporative cooling fabric (Figure 4A−E); the relationship between WVT, temperature, and relative humidity is shown in

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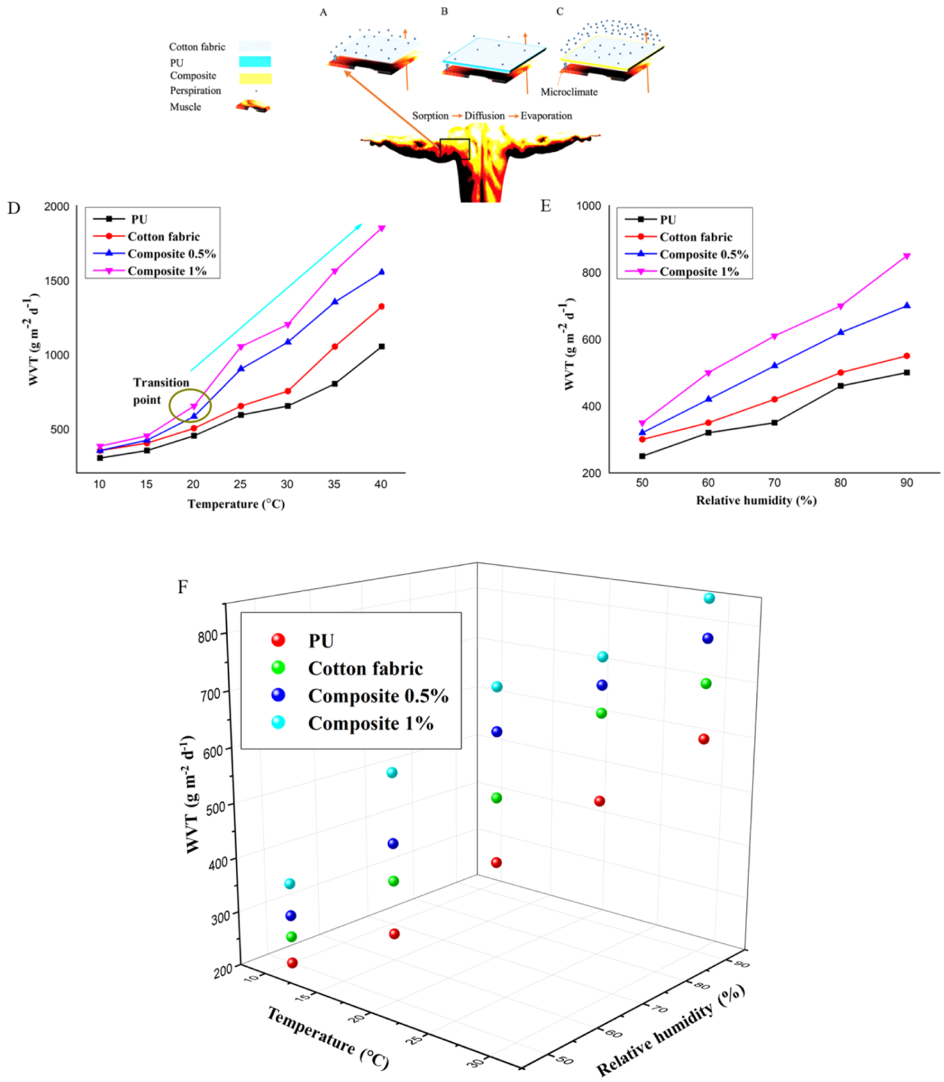

Figure 4. Mechanism of evaporation of perspiration: (A–C) evaporation of perspiration with fabric (cotton), PU layer, and composite layer; (D) WVT results of laminated fabric of PU, composite 0.5%, composite 1%, and cotton fabric as a function of temperature; (E) WVT results of laminated fabric of PU, composite 0.5%, composite 1%, and cotton fabric as a function of relative humidity, and (F) relationship between WVT, temperature, and relative humidity.

Figure 4F. Cotton is a highly water-absorbing cellulosic material; the moisture absorption rate of cotton is around 7–8%. To make the evaporative cooling fabric, we laminated the cotton-weaved fabric via a highly evaporative thermo–moisture-responsive PU composite membrane. The composite membrane was prepared by adding hydrophilic hollow silica aerogel with PU. Segmented hydrophilic PU was synthesized, and then, the super moisture-absorbent hollow silica aerogel was added in order to make a highly evaporative film. Our composite membrane and laminated fabric have a high water vapor permeability property to control the personal microclimate of the human body, as shown in Figure 4D; composite 1% has better WVT (1859 g m^{-2} d^{-1}) than PU alone (1065 g m^{-2} d^{-1}) or cotton fabric (1190 g m^{-2} d^{-1}) at 40 °C temperature. Furthermore, WVT of composite 1% is 859 (g m^{-2} d^{-1}), while PU has only 440 (g m^{-2} d^{-1}) at 90% relative humidity (Figure 4E). This type of microclimate-controlled cooling fabric has been developed to minimize body heat-related diseases such as heat stroke and other heat-related injuries in extreme weather. A hot environment reduces the working efficiency of the body. Evaporation of perspiration is an extremely effective way by which to cool the body in a hot environment. Moreover, when the core temperature of the body increases, it directly influences physical performance and metabolism. Uncontrolled metabolism can cause serious physical defects and cause the body to work inefficiently. Research has shown that if human thermoregulation can be controlled and body efficiency increased, then the risk of heat-related diseases is limited. Personal cooling fabrics can enhance working time by keeping the body cool. When metabolic heat is produced, it passes the signal to the hypothalamus which distributes the heat to different parts of the body via blood circulation to release body heat by evaporation of perspiration, convection, radiation, and conduction. Thus, the hypothalamus controls the core temperature of the body. Water vapor permeability through the membrane strongly depends on the microstructure and hydrophilicity of PU. The physical and chemical properties mostly depend on the soft and hard segment ratio, the molecular weight of the monomer, and processing parameters. Incorporating the hollow silica aerogel makes the PU membrane more water vapor- and air-permeable, radiative, and robust. The hollow silica aerogel

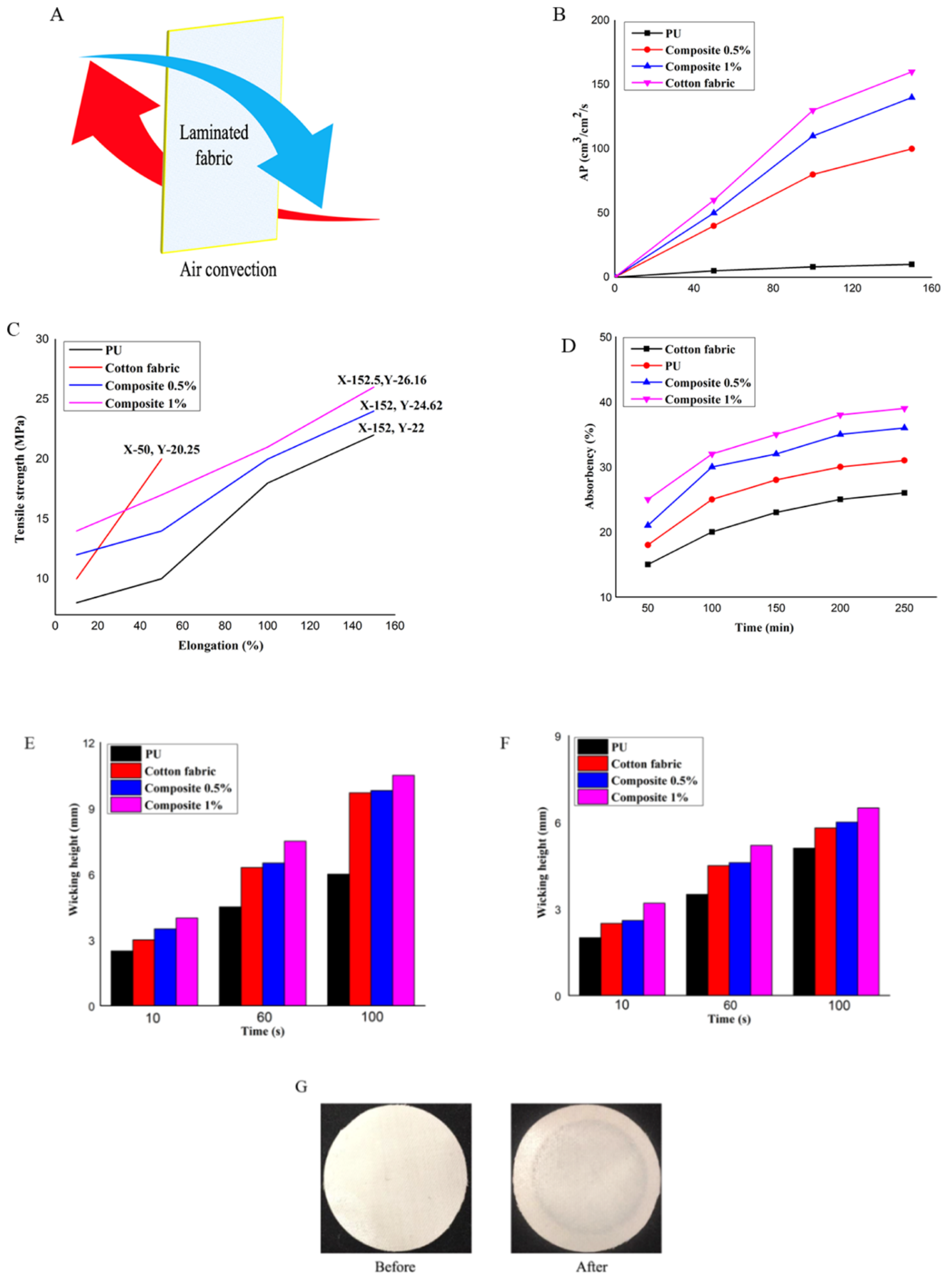

Figure 5. (A) Air convection of the laminated fabric, (B) AP result of the laminated fabric (PU, composite 0.5 and 1%) and control (fabric), (C) mechanical properties of the laminated fabric (D) water absorbency of the laminated fabric, (E) wicking properties of the laminated fabric (warp direction), (F) wicking properties of the laminated fabric (weft direction), and (G) abrasion resistance property of the laminated fabric.

has super affinity to moisture and can pass water molecules to the environment by using its hollow water vapor passing channel.

Convection is one heat transfer mechanism of the body; convection contributes to the transportation of body heat from the body's surface into the environment. During the convection process, air removes body heat from the skin to the environment and cools down the body during high activity. Heat stroke is a common reason of death during extreme weather or high activity such as marathon running; convective clothing could play a significant role in minimizing these deaths; our composite membrane and laminated fabric have the ability to cool down our body by air convection (Figure 5A,B) and warm the body during high activity levels and in extreme weather. Nowadays, heat-related diseases are increasing greatly because of global climate change, with diseases ranging from neurological diseases and cellular damage to many more heat-related illnesses, and may cause death. Bai et al. have reported the devastating effects on human health from recent heat waves in China as a consequence of extreme heat stress.[42] It has also been suggested that children and the elderly are most affected during summer because of high temperatures. Air convection play a major role in minimizing such heat-related diseases; our laminated fabric has the ability to circulate air in hot conditions. AP was measured on an instrument designed to pressure air through the laminated fabrics. The rate of air passing through the test specimen was significant, except for the PU laminated fabric. Cotton fabric has better AP (160 $cm^3/cm^2/s$ at 150 pa pressure) because of its porous woven structure, whereas composite membranes [140 $cm^3/$

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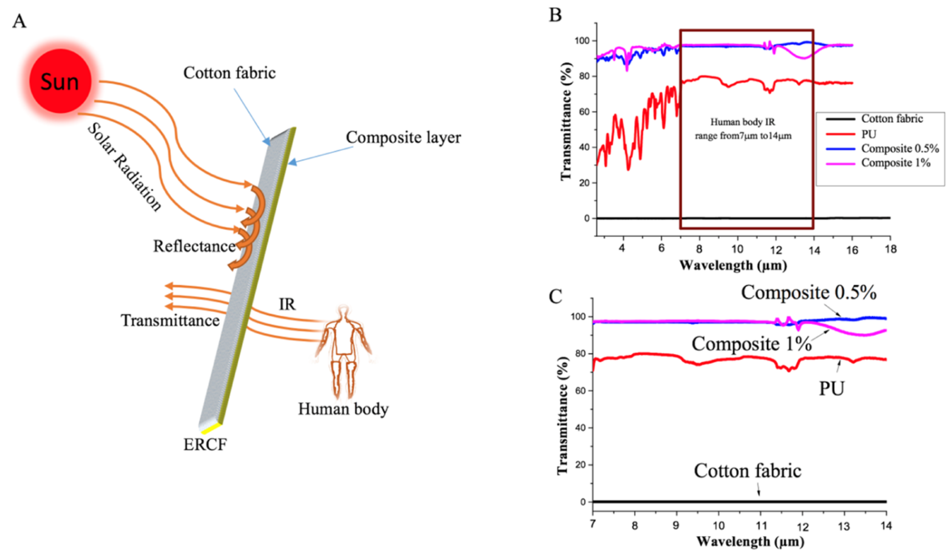

Figure 6. (A) The behavior of ERCF with solar radiation and IR radiation; (B) IR transmittance percentage of cotton fabric, PU, composite 1%, and composite 0.5% (range 3−18 μm); and (C) IR transmittance percentage of cotton fabric, PU, composite 1%, and composite 0.5% (range 7−14 μm).

cm^2/s at 150 pa pressure (composite 1%) and 100 cm^3/cm^2/s at 150 pa pressure (composite 0.5%)] also have better AP. Normally, air-permeable materials allow water vapor to pass through and therefore WVT is closely related to the air passing ability of the material. Water vapor permeability also depends on the material's chemical structure. The pore from the woven fabric structure and porous silica aerogel facilitates the AP of the laminated fabric. For thermophysiological comfort, AP is a very important property of a fabric. The first step of WVT is absorption, followed by diffusion and evaporation. Additionally, WVT mostly depends on the water absorbance ability of the material (Figure 5D). The water absorbency of composite samples is significantly higher than that of the cotton fabric and PU. As seen in Figure 5C, composite materials re-enforce the laminated fabric, and composite samples are significantly strong and elastic compared with cotton fabric. Toughness and tensile modulus also showed a similar result that composite samples are significantly stronger compared with PU (Figure S3). The wicking height rate reveals the moisture absorbency ability of the material. Normally, composite materials have better wicking height in both warp and weft directions because of their water-absorbing ability and hydrophilic group in the backbone (Figure 5E,F). Cotton fabric also has a good wicking height compared with the PU membrane. To check the adhesiveness and abrasion resistance of the laminated fabric, we performed an abrasion resistance test (Figure 5G) and found that there was almost no weight loss; there is also no yarn breakage after rubbing the laminated fabric 1000 times (50 per min) with 9 kPa load, which means that the laminated fabric has very good abrasion resistant properties.

Radiative heat emission is an effective way by which to release heat from the skin to the environment. Maximum amount of body heat is released into the environment by the emission of radiation. The human body is mid-IR radiative in the wavelength range between 7 and 14 μm.[20,43] The traditional textile is not IR-transparent and our human skin is a super IR emitter; therefore, the IR transmittance textile provides thermal comfort to the body. Because textile materials are very heat-absorbent, fabric temperature rises rapidly when exposed to thermal sources.[42] Silica aerogel and composites made by using silica aerogel can be considered as semi-transparent materials capable of absorbing, emitting, and radiating heat.[44−46] Our fabricated composite membrane successfully transmitted more than 90% of inferred radiation with a wavelength longer than 7 μm (Figure 6B,C). The schematic of the ERCF (Figure 6A) shows that the outer surface of the laminated fabric (cotton) reflects solar radiation and the inner surface of the laminated fabric (composite membrane) transmits IR from the body. The presence of the silica aerogel has improved the IR transmittance of the laminated fabric (Figure 6B,C). Consequently, PU composites showed better transmittance compared to pristine PU, whereas the cotton fabric showed almost zero transmittance. To maintain the human thermoregulation system and a constant body temperature, our developed ERCF could play a vital role.

4. CONCLUSIONS

The developed ERCF can transfer most of our body heat to the environment. Climate change is increasing the global temperature daily, and the current challenge is to keep the global temperature below a variation of 2 °C; to do so requires sustainable and significant global mitigation. Hot outdoor environments can have a negative impact on the body, such as heat stroke, dehydration, and elevated heart rate. During high activity or in a hot environment metabolism produces heat as a byproduct. The body is 25% efficient; therefore, we lose around 75% of energy as heat. To overcome such challenges, our ERCF can play an important role by providing passive cooling by evaporation of perspiration, air convection, and emission of radiation from the body directly to the environment. The ERCF has a vital role in controlling body temperature by transferring body heat through various means into the environment during the summer season or when the surrounding temperature is too high. The body heat is released by evaporation of perspiration; the evaporative behavior of the ERCF could provide evaporative cooling by releasing body heat during extreme weather. Air convection can also transport a significant amount of body heat from the skin to the environment; the ERCF has an AP behavior which can provide air convection to the human body. Emission of radiation is the

https://dx.doi.org/10.1021/acsomega.9b03268
ACS Omega 2020, 5, 3899−3907

most effective way by which to release body heat, and the maximum amount of body heat can be transported by emission of radiation through electromagnetic waves at room temperature or below. Radiative cooling provides an effective way by which to surpass conventional methods of controlling the microclimate of the human body. The ERCF we have developed provides necessary body cooling for an individual to feel comfortable at different temperature levels.

■ ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.9b03268.

Synthesis details of samples, pictures, SEM, mechanical property, DSC, and WVT results of samples (PDF)

■ AUTHOR INFORMATION

Corresponding Author

Jinlian Hu – *Institute of Textiles and Clothing, The Hong Kong Polytechnic University, Kowloon 999077, Hong Kong, China;* orcid.org/0000-0001-8914-5473; Phone: +852 27666437; Email: jin-lian.hu@polyu.edu.hk

Authors

Md Anwar Jahid – *Institute of Textiles and Clothing, The Hong Kong Polytechnic University, Kowloon 999077, Hong Kong, China*

Suman Thakur – *Institute of Textiles and Clothing, The Hong Kong Polytechnic University, Kowloon 999077, Hong Kong, China*

Complete contact information is available at: https://pubs.acs.org/10.1021/acsomega.9b03268

Notes

The authors declare no competing financial interest.

■ ACKNOWLEDGMENTS

The authors of this paper would like to acknowledge the financial support from projects fund, Development of Environmental Friendly and Self-Adaptive Breathable Polyurethane Film (GHX/001/16GD), and the Science and Technology Planning Project of Guangdong Province, China (project number: 2016A050503013).

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https://dx.doi.org/10.1021/acsomega.9b03268
ACS Omega 2020, 5, 3899−3907