



Optically Active Polyurethane/Silica Aerogel Coated Cotton Fabrics for Thermal Protection

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Application of SiO₂ aerogel in thermal protective clothing has been limited due to its brittle nature, ordinary mechanical properties, and poor film forming performance. This work is aimed to develop thermal protective cotton fabrics by coating blended OPU/SiO₂ aerogel with enhanced mechanical properties and thermal protection performance. The OPU/SiO₂ aerogel composites with different ratio were applied onto cotton fabrics by knife-coating. The morphology, chemical component, crystalline structure, thermal stability and compression strength were characterized by scanning electron microscopy (SEM), Fourier transfer Infrared spectroscopy (FTIR), X-ray diffraction (XRD), thermogravimetric analysis (TG) and compression test, respectively. Besides, the warmth retention performance and heat protection performance together with air and moisture permeability of the coated fabrics were studied. The results showed that OPU/SiO₂ aerogel were successfully coated onto cotton fabrics with enhanced mechanical properties and thermal stability together with better film forming capacity. The heat transfer coefficient of the coated cotton fabrics was distinctly dropped due to the synergistic effect of OPU and SiO₂ aerogel, which resulted in higher warmth retention. The OPU/SiO₂ aerogel coated fabrics exhibited obvious heat insulation performance with its surface temperature almost 4 °C than the uncoated fabrics. This work demonstrates a new strategy of fabricating stronger thermal insulating textiles using OPU/SiO₂ aerogel composites.

Keywords: optically active polyurethane, SiO₂ aerogel, coating, thermal insulating textiles, cotton fabrics

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INTRODUCTION

Thermal protective clothing is essential for wearers who are subject to threats from environments such as extremely cold weather, special low temperature industrial environment and/or geometrically challenging locations (e.g., the polar region of the earth; Kothari and Chakraborty, 2016; Udayraj et al., 2016a,b; Bogerd et al., 2017; Su et al., 2020). Heat is generally transferred via thermal conduction, thermal radiation and thermal convection (Abbasi et al., 2016; Hayat et al., 2016; Dai et al., 2018; Ge et al., 2019). The clothing system serves as the barrier between environment and human body, in which the fibrous materials and fibrous structure dominate the heat transfer from body to environment. Conventional thermal protective clothing is based on thermal insulating fibers, e.g., down fiber, with highly porous structure in the clothing system (Shu et al., 2017). These thermal protective products are functionally competent for daily wearing such as winter coat. However, there is a big challenge in protecting human from extreme environmental conditions, as under extreme environmental conditions the traditional

thermal protective clothing is far from enough to ensure the safety and wellbeing of human. Thus, it is important to develop new thermal insulating materials with outstanding thermal insulation performance to withstand both thermal conduction and thermal radiation (Hu et al., 2018). Due to the limit of fiber materials in high level thermal protection, novel materials with excellent thermal protection performance are widely used in textiles to provide extra thermal protection. Coating of materials with extra performance onto textiles has been widely applied in protective clothing (Tian et al., 2016; Tao et al., 2017; Yang et al., 2017; Liu et al., 2018a; Asim et al., 2020). Active nanomaterials with excellent thermal protection have also been applied in the coated layers, providing nanocomposite solutions to the development of next-generation thermal protective clothing (Wang and Fan, 2014; Záborský et al., 2016; Kim et al., 2017; Xu et al., 2017; Pakdel et al., 2019; Zheng et al., 2019).

SiO₂ aerogel has been well noticed for its light weight, porosity and network structure, and it has shown a great potential in developing thermal insulating materials (Liu et al., 2016; Günay et al., 2018; Liu et al., 2018b; Liu et al., 2019; Lu et al., 2020). However, SiO₂ aerogel shows low thermal blocking effects against Infrared radiation. To overcome this issue, additives to blocking Infrared radiation were added into SiO₂ aerogel (Tang et al., 2016). It has always been a challenging proposition to load of SiO₂ aerogel onto textiles and realize its application (Liu et al., 2016; Ye et al., 2016). In theory, SiO₂ aerogel can achieve thermal protection due to its excellent performance. However, in practice SiO₂ aerogel coated textiles have poor durability due to the imperfect combination between SiO₂ aerogel and fibers (Rostami et al., 2021). Furthermore, SiO₂ aerogel has a different shrinkage rate in the process of drying, resulting in cracks on the surface of the coated fabrics. Adhesives are widely used to overcome these issues, but the additive itself has a high thermal conductivity. As a result, finding a proper adhesive to enhance the cohesiveness of the coated layer with fibers and to maintain the excellent thermal protection of SiO₂ aerogel is still a challenge.

Polyurethane has been widely used as coating layers for various objects, and it can be simply coated on textiles for protection against different threats from environment, e.g., wind, water, chemicals and fire (Moiz et al., 2016, 2017). However, the protection as provided by polyurethane is not enough for some professionals due to the limit of polyurethane in high-level protection against different threats from either environment or working locations. Thus, modification of polyurethane, such as nanocomposites (Lee and Saito, 2018), combining with other mechanisms and surface modification, has been the research topic in the area of protective clothing. As an example, optically active polyurethane (OPU) has been synthesized to enhance the thermal stability especially the thermal radiative protection of polyurethane. OPU has evident blocking and refracting effects to Infrared and visible lights, resulting in thermal radiative protection performance to the coated materials (Sedlacik et al., 2019). OPU has the potential to be an ideal adhesive film on textiles to serve different purposes.

In this study, thermal protective cotton fabrics were fabricated by coating blended OPU/SiO₂ aerogel with enhanced mechanical properties and thermal protection performance.

OPU has great film formation capacity, which makes up for the weakness of SiO₂ aerogel poor mechanical properties. SiO₂ aerogel has high thermal stability, and the combination with OPU improves the thermal stability and thermal protection performance of polyurethane. The OPU/SiO₂ aerogel paste was then applied onto cotton fabrics by knife coating. The morphology, chemical component, crystalline structure, thermal stability and compression strength were characterized, and the warmth retention performance, heat protection performance and air and moisture permeability of the coated fabrics were all investigated in detail. The combination of OPU and SiO₂ aerogel has synergistically enhanced the performance of both polyurethane and aerogel, resulting in a thermal insulating layer on cotton fabrics. The research results provide a technical basis for the preparation of aerogel insulation coated fabric and propose an approach to improve the thermal insulation performance of the coated fabric.

EXPERIMENTAL

Materials

The sample fabrics were pure cotton in woven structure. Optically active polyurethane and SiO₂ aerogel were self-prepared according to the method as reported (Gurav et al., 2010; Yang et al., 2016). Dispersion agent SN-5040, wetting dispersant PE-100, thickening agent ASE-60 and defoaming agent were purchased from Kexingde Chemical Co., Ltd. (Yixing, China).

Preparation

First of all, 450 g deionized water, 5 g dispersion agent and 3 g wetting dispersant were added to a flask followed by high-speed dispersion for 10 min. SiO₂ aerogel was then added under high-speed stirring for 1 h. After that, OPU was added under stirring at 40°C for 1 h. Then, thickening agent and defoaming agent were added to the mixture under low speed stirring for 1 h, resulting in OPU/SiO₂ aerogel blended paste. The weight ratio of SiO₂ aerogel and OPU in the blend was adjusted to be 1:5, 1:10, 1:15, 1:20, and 1:25, coded as SiO₂/OPU-1, SiO₂/OPU-2, SiO₂/OPU-3, SiO₂/OPU-4, and SiO₂/OPU-5, accordingly.

Secondly, the cotton fabrics were knife-coated with the OPU/SiO₂ aerogel blended paste. Cotton fabrics were immersed in the OPU/SiO₂ aerogel blended paste under mechanical stirring at 40°C for 30 min. The fabrics were then placed on a Teflon molding sheet and the remaining OPU/SiO₂ aerogel blended paste was poured on the surface of the fabrics. A knife was moving along the surface of the fabrics to ensure an even coating. The Teflon sheet with coated fabrics were then placed in an oven at 40°C for drying. In the end, the coated fabrics were peeled off from the molding sheet. The fabrication process of OPU/SiO₂ aerogel coated fabrics is illustrated in **Figure 1**.

Characterization and Measurements

SEM images were obtained from Nova NanoSEM 450 field emission scanning electron microscope. The samples were gold-coated in a sputter coater before observation.

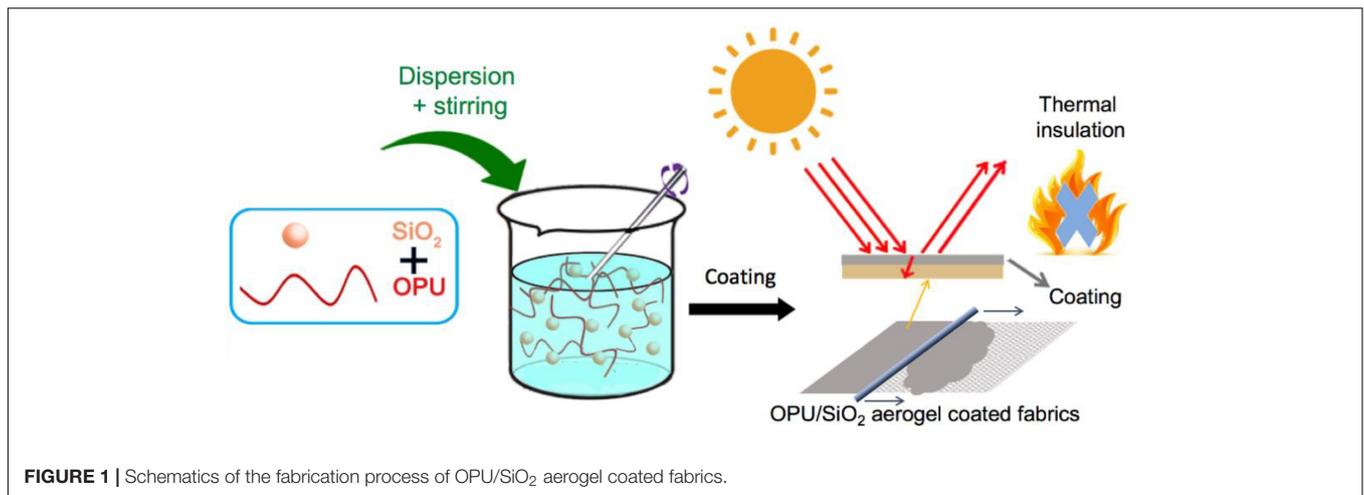


FIGURE 1 | Schematics of the fabrication process of OPU/SiO₂ aerogel coated fabrics.

FTIR spectra were obtained from NEXUS-670 FTIR spectroscopy with ATR, wavelength range was 400–4,000 cm⁻¹ and resolution <0.09 cm⁻¹.

XRD patterns were obtained from X'Pert3 Powder X-ray diffraction tester (PANalytical, Netherland), with $2\theta = 5^\circ\text{--}80^\circ$.

TG curves were obtained from STA 449C TG-DSC thermal analyzer with nitrogen protection and temperature rising rate of 10°C/min in the range of 20–1,000°C.

Compression module was tested on RGD-5B compression module testing machine. Samples were in cylinder with radius of 20 mm and height of 20 mm, and the load was 0.5 kN/s. The force-displacement curves were drawn from the extracted data in a data analysis software (Origin).

The warmth retention of coated fabrics was tested on a plate warmth retention tester (YG606, Ningbo Textile Equipment Company, Ningbo, China). In the test, the size of the sample was 30 cm × 30 cm, and the temperature was 38 ± 0.5°C. The test was repeated for 5 times and the results were averaged. The warmth retention rate was calculated as the difference (in percentage) of heat loss of a hotplate due to covering of the coated fabrics, according to the testing standard GB11048-89.

The heat protection test was performed on a purpose-built apparatus in which the fabrics were mounted on the hotplate with a constant temperature of 46°C, and the surface temperature of fabrics was monitored during the test. The environment conditions were set as 25 ± 2% RH and 20°C.

The air permeability was tested on a fabric permeability tester (YG-461E, Ningbo Textile Equipment Company, Ningbo, China). The size of nozzle was 0.8 mm, power of suction ventilator 1,100 W, flow rate 120 m³/h. The size of sample was 20 cm² and the pressure difference was 200 Pa. The air permeability was tested as the volume of the transported air through the unit area of the fabrics in a given timeframe (unit mL/cm²·s).

The moisture permeability was tested on the YG(B)216-II fabric moisture permeability tester (Wenzhou Darong Textile Equipment Co., Ltd., Wenzhou, China). The size of sample was 90 mm in diameter (round shape) and the environment conditions were 38°C and 90% RH with airflow speed of

0.3–0.5 m/s. The moisture permeability was expressed as the vapor diffusion rate through a unit of the surface of the tested fabric (unit g/m²·d).

RESULTS AND DISCUSSION

Morphology

The OPU/SiO₂ aerogel coating layer was formed on the surface of cotton fabric with the knife. The organic/inorganic blending of OPU and SiO₂ aerogel has greatly enhanced the mechanical properties and membrane forming of the coated layer on cotton fabrics. Polyurethane has great film formation capacity to form an intact membrane on the surface of fabrics and to trap the SiO₂ aerogel particles inside without falling off during usage.

Figure 2 shows the SEM images of pure SiO₂ aerogel and OPU/SiO₂ aerogel with different OPU concentrations. Pure SiO₂ aerogel shows very small particle size with uniform distribution (**Figure 2a**). Coating of OPU/SiO₂ aerogel on cotton fabrics has resulted in a membrane on the surface of the fabrics. However, larger particles of SiO₂ aerogel can be observed in the blends exhibits, suggesting a low level of aggregation of SiO₂ aerogel in the blends. As a typical phenomenon of nanoparticles in composites, the particles tend to aggregate due to the phase separation and poor interface connections in the composites. The SiO₂ aerogel was subjected to extensive stirring in accompany with dispersion agent and water. Si-OH groups were subsequently formed on the surface of aerogel and the instability of these groups forced the aerogel to move to areas with lower surface tension, resulting in coupling between Si-OH groups and aggregations.

Determining the optimized blending ratio of SiO₂:OPU is the key to ensure that both OPU and SiO₂ aerogel can play their roles in the composites. From **Figures 2b–f** it is clear that the aerogel turns to distribute more evenly under a high OPU concentration. As seen from **Figures 2b,c**, the aerogel exists mainly in aggregation form with a very uneven distribution. With the increase of OPU content in the composites, the size of aerogel became smaller with a more even distribution, without

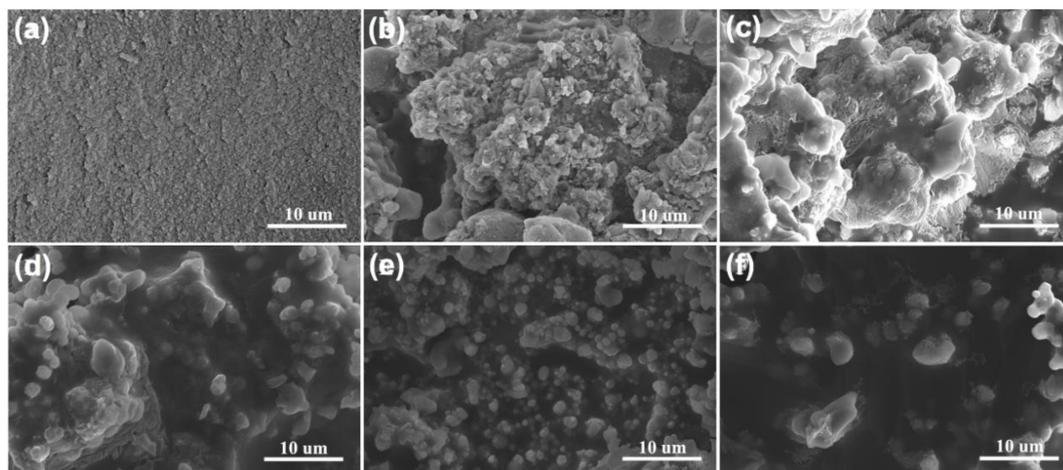


FIGURE 2 | SEM images of pure SiO₂ aerogel (a), SiO₂/OPU-1 (b), SiO₂/OPU-2 (c), SiO₂/OPU-3 (d), SiO₂/OPU-4 (e), and SiO₂/OPU-5 (f).

evident aggregation (Figures 2d–f). The reason is probably due to the stronger coupling effect between -NCO and H hydron as compared to that with Si-OH, which disperses the aggregated aerogel during the preparation stage of OPU/SiO₂ aerogel. Besides, under a high OPU ratio the excellent film forming capacity of polyurethane can slightly overcome the aggregation of aerogel with an improved film forming capacity of the coated layer. However, the viscosity of OPU/SiO₂ aerogel increased gradually with the increase of the ratio of OPU in the composites. A ratio of higher than 1:25 for SiO₂:OPU would result in too viscous blend for subsequent coating applications. From morphology study it can be found that a ratio of 1:20 is more suitable for preparing OPU/SiO₂ aerogel with uniform distribution of aerogel and without aggregation.

FTIR Spectra

Figure 3A shows the FTIR spectra of OPU/SiO₂ aerogel coated cotton fabrics with different blending ratios. All the spectra show a similar pattern with characteristic peaks of SiO₂ and polyurethane. The obvious peak at 1,800 cm⁻¹ is due to the stretching of Si-O-Si. The peak at 800 cm⁻¹ is due to Si-C stretching, and the one at 1,245 cm⁻¹ is corresponding to the C-H in Si-CH₃. The peaks between 1,537 and 1,704 cm⁻¹ are attributed to the absorbing peaks of benzene ring. The peak around 930 cm⁻¹ is the absorbing peak of Si-H vibration, whereas the one at 2,963 cm⁻¹ is the absorbing peak of the antisymmetric stretching vibration of C-H from aerogel. Besides, the absorbing peaks at 1,245 and 3,415 cm⁻¹ are due to the stretching vibration of N-H, C=O and C-O from the urethane groups. FTIR analysis has revealed that both the chemical components of OPU and SiO₂ aerogel can be detected from the coated fabrics, suggesting that both OPU and SiO₂ aerogel were successfully loaded onto the fabrics.

XRD Patterns

Figure 3B shows the XRD patterns of OPU/SiO₂ aerogel coated cotton fabrics with different blending ratios. Overall,

the XRD patterns suggest the amorphous crystalline structure of the coated fabrics, which is typical crystalline structure for fibrous materials. From the XRD pattern curves, there is a broad non-crystalline characteristic peak at around 20°, and the intensity of the peak has found to be decreased with the increase of the amount of OPU in the blend. Apart from the peak at 20°, there are no other obvious crystalline peaks as observed from the patterns. Therefore, adding SiO₂ aerogel to OPU hasn't altered the amorphous crystalline structure of OPU. The coupling among Si-OH groups or between -NCO and H hydron only resulted in changing of the size and aggregation form of aerogel but not the overall crystalline structure of OPU/SiO₂ aerogel. Coating as a physical method to apply extra materials onto fabrics has little effect on the crystalline structure, thus the XRD patterns suggest no evident changes after coating.

TG Analysis

OPU has a better thermal stability than polyurethane due to its evident blocking and refracting effects to Infrared and visible lights (Sedlacik et al., 2019), as a result OPU/SiO₂ aerogel composites are supposed to exhibit better thermal stability than widely used PU/SiO₂ aerogel. Figure 3C shows the TG curves of both OPU/SiO₂ aerogel (1:15 ratio) and PU/SiO₂ aerogel. Both curves show a weight loss at around 60°C owing to the evaporation of water and solvents. The main weight loss (47%) of PU/SiO₂ aerogel occurred between 154 and 254°C, and in this stage the polymer chain of PU broke up and decomposed. However, the main weight loss (38%) of OPU/SiO₂ aerogel occurred at the temperature of 385–490°C. The significance difference is due to the excellent thermal stability of OPU, which leads to a higher decomposing temperature of OPU/SiO₂ aerogel. At a given weight loss of 60%, the decomposing temperature for PU/SiO₂ aerogel and OPU/SiO₂ aerogel is 280 and 485°C, respectively. These results show the enhanced thermal stability of OPU/SiO₂ aerogel than SiO₂/PU aerogel.

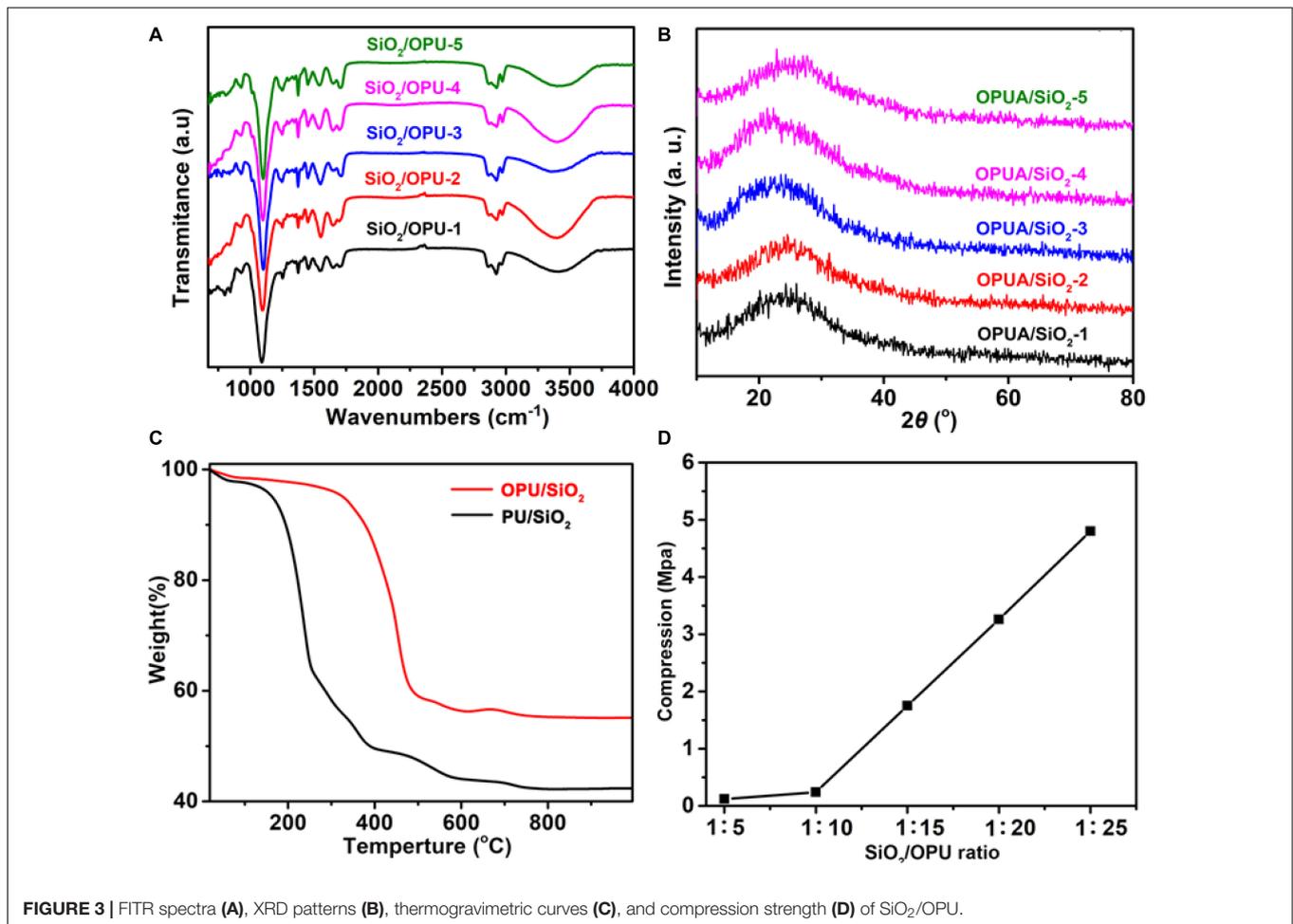


FIGURE 3 | FTIR spectra (A), XRD patterns (B), thermogravimetric curves (C), and compression strength (D) of SiO₂/OPU.

Compression Strength

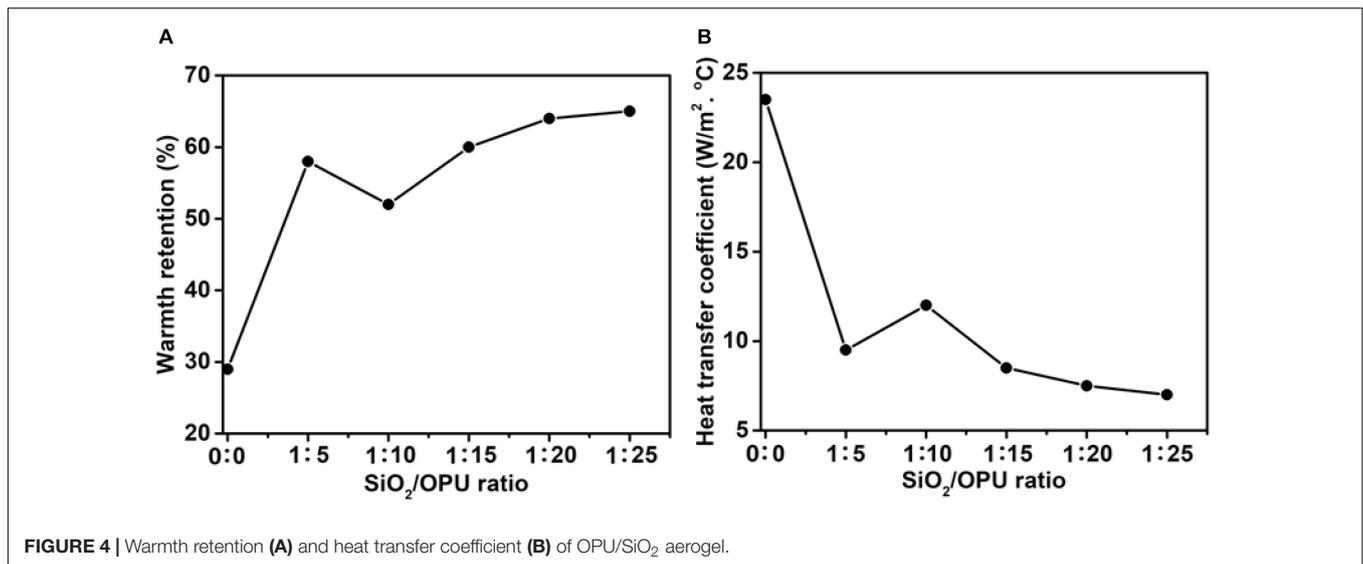
The compression modulus of OPU/SiO₂ aerogel was investigated, as shown in **Figure 3D**. It can be seen that the compression modulus of OPU/SiO₂ aerogel has increased almost linearly with the increase of OPU ratio from 1:10 to 1:25. The compression modulus of OPU/SiO₂ aerogel at 1:25 ratio is almost 5 MPa, which is almost 150 times of the compression modulus of pure SiO₂ aerogel.

In OPU/SiO₂ aerogels, the deformation is large whereas the compressive strength is small during the early compression process, which mainly due to the macroscopic pores in OPU and SiO₂ aerogels. The subsequent continuous compression may cause the collapse of SiO₂ aerogel framework and chain break, resulting in crack and structure damage, and the compression strength and compressive strength gradually increase. After that, all the pores in the composite aerogel disappeared and the compressive strength showed a linear maximum. Besides, OPU also shows outstanding tenacity to further withstand external pressure. The OPU withstands the force to protect the brittle SiO₂ aerogel in the composites, leading to a greatly enhancement of mechanical properties for OPU/SiO₂ aerogel. Besides, OPU has tenacity to further withstand external pressure.

Warmth Retention

Fabrics coated with OPU/SiO₂ aerogel showed excellent warmth retention with a low heat transfer coefficient. As presented in **Figure 4A**, the warmth retention rate for uncoated fabrics is 29.05%. However, the warmth retention rate of OPU/SiO₂ aerogel coated fabrics is higher than 50%. Besides, the warmth retention increases gradually with the increase of OPU ratio in OPU/SiO₂ aerogel. On the other hand, the heat transfer coefficient drops distinctly when the fabrics have been coated with OPU/SiO₂ aerogel, and the coefficient decreases gradually with further increasing OPU ratio (**Figure 4B**).

It is noted that there is a fluctuation in both curves with the ratio increasing from 1:5 to 1:10. This is probably related to the heat transfer ways and the distribution together with the aggregation of SiO₂ aerogel in the coated layer. When the OPU ratio is low, the thermal retention is mostly determined by SiO₂ aerogel. The distribution of the aerogel has great impact on the overall thermal retention. As shown in the morphology study (**Figure 2**), evident aggregation can be found at the ratio of 1:5–1:10, leading to an uncertain thermal retention capacity. Besides, SiO₂ aerogel has no evident thermal protection against radiant transfer, thus the heat transfer is slightly high when the OPU ratio is low. With the increase of OPU in the SiO₂ aerogel,



the thermal retention capacity is largely determined by OPU. OPU has an evident blocking effect to infrared, leading to its excellent thermal protection against radiation. Furthermore, the distribution of SiO₂ aerogel becomes more even without obvious aggregation at a higher OPU ratio. As a result, the thermal protection effect of SiO₂ aerogel has been strengthened. In the end, both OPU and SiO₂ aerogel work collaboratively to generate excellent thermal retention capacity as shown in Figure 4.

Heat Protection

Figure 5 shows the surface temperature of OPU/SiO₂ aerogel coated fabrics when they are attached to the hotplate of 46°C. It can be found that the surface temperature of the control fabrics increases immediately from 23.9 to 31.2°C after 10 min, and then the temperature further increases gradually to 41.1°C after 30 min. A similar trend can be found for the surface temperature of OPU/SiO₂ aerogel coated fabrics. In the first 5–10 min, the surface temperature of OPU/SiO₂ aerogel coated fabrics is almost the same and sometimes higher than that of the control fabrics.

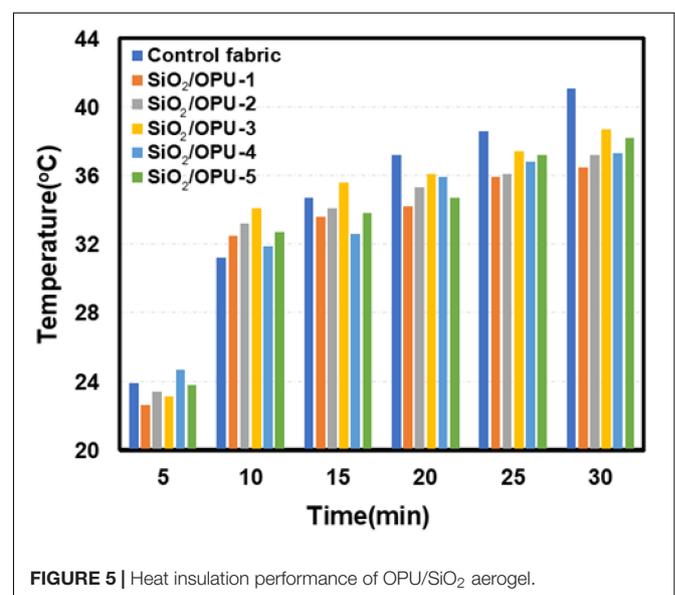
However, the surface temperature of coated fabrics increases much slower than the rate for the control fabrics. As a result, the surface temperature of coated fabrics is lower than that of the control fabrics, and the difference becomes bigger and bigger with the increase of testing time. For the control fabrics, the surface temperature is 41.1°C after 30 min, a difference of 4.9°C to the hotplate (which is 46°C). But for the coated fabrics, the surface temperature can be as low as 37.2°C after 30 min, suggesting a difference of 8.8°C. It can be concluded that the surface temperature of coated fabrics is almost 4°C lower than that of uncoated fabrics. These results show that the OPU/SiO₂ aerogel coated fabrics exhibit obvious heat insulation performance.

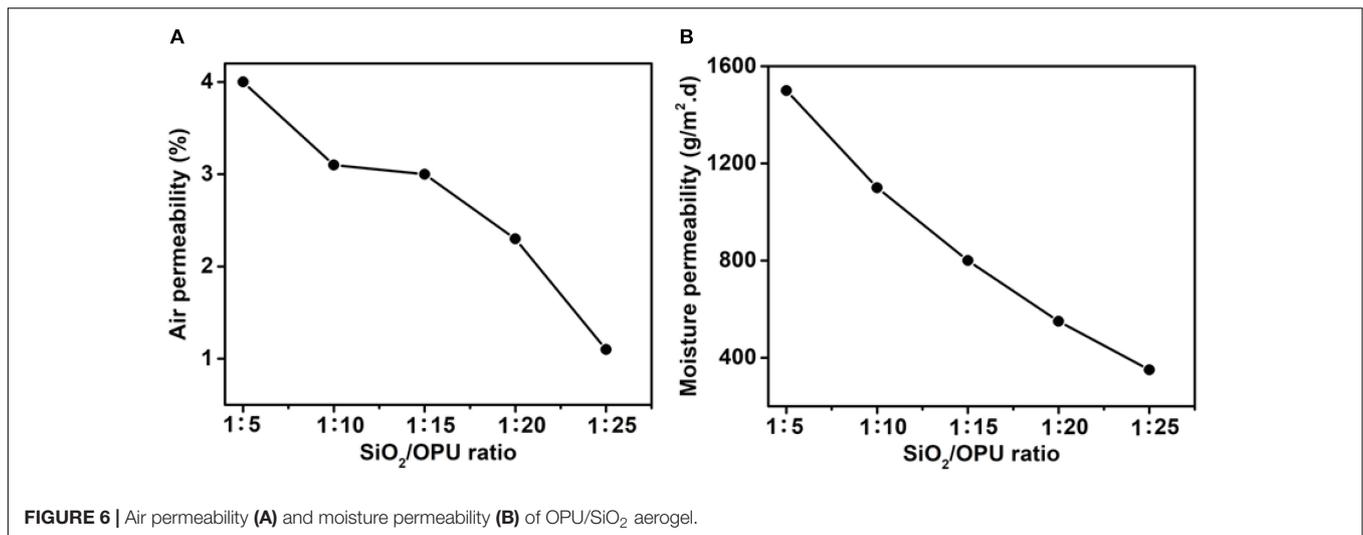
It is evident that the coated fabrics showed excellent warmth retention and heat protection as per the results. The mechanism of thermal protection performance of the coated fabrics can be explained as a combined effect of OPU and SiO₂ aerogel. The membrane forming capacity of OPU has provided

thermal protection to some extent as the pores of fabrics were largely blocked and thermal exchange between the fabrics and environment was decreased. The excellent thermal stability of SiO₂ aerogel has provided thermal protection effect to the coated layer. Besides, the even distribution of SiO₂ aerogel in the coated layer has maximized the thermal protection effect of SiO₂ aerogel.

Air and Moisture Permeability

The permeability of fabrics is greatly affected by their thickness, pore size and porous structure of the coated layer. Figure 6A shows the air permeability of OPU/SiO₂ aerogel coated fabrics. It can be seen that the air permeability decreases gradually with the increase of OPU ratio. Polyurethane has good membrane forming properties, thus the increase of OPU ratio in the OPU/SiO₂ aerogel will result in a film on the fabric surface with





most of the porous structure in SiO₂ aerogel blocked. Besides, the thickness of coated fabrics increased from 0.54 to 0.61 mm with the increase of OPU ratio in the OPU/SiO₂ aerogel. Thicker fabrics tend to exhibit a lower air permeability when other parameters are the same.

Figure 6B shows the moisture permeability of the coated fabrics. Similarly, the moisture permeability declines evidently with the increase of OPU ratio in the OPU/SiO₂ aerogel. The excellent film forming performance of polyurethane has formed an intact membrane on the surface of fabrics after coating, and the porous structure of SiO₂ aerogel has been altered with most of the pores blocked. The existence of OPU endows excellent water repellent performance to the coated fabrics. Therefore, the OPU/SiO₂ aerogel coating has led to deteriorated moisture repellent capacity as per the results.

From the above results it can be seen that the coated fabrics have excellent compression strength, partly preserved permeability, and excellent warmth retention together with heat protection. These properties and performance have granted the coated fabrics with application potential as heat protective clothing, sportswear (for cold weather) and technical textiles.

CONCLUSION

OPU/SiO₂ aerogel were successfully coated on cotton fabrics with enhanced mechanical properties and membrane forming capacity. Existence of OPU has significantly improved the film forming capacity and distribution of SiO₂ aerogel in the coated layer. OPU/SiO₂ aerogel coated fabrics showed a similar FTIR pattern with characteristic peaks of SiO₂ and polyurethane. Adding SiO₂ aerogel to OPU hasn't altered the amorphous crystalline structure of OPU as seen from XRD patterns. The excellent thermal stability of OPU has led to a higher decomposing temperature and enhanced thermal stability of OPU/SiO₂ aerogel as compared with PU/SiO₂ aerogel. The compression modulus of OPU/SiO₂ aerogel has increased almost linearly with the increase of OPU ratio from 1:10 to 1:25,

reaching to almost 150 times of the compression modulus of pure SiO₂ aerogel. The warmth retention increased gradually with the increase of OPU ratio in OPU/SiO₂ aerogel, and the heat transfer coefficient decreased gradually with the increase of OPU ratio. The OPU/SiO₂ aerogel coated fabrics exhibited obvious heat protection performance with its surface temperature almost 4°C than the uncoated fabrics. The air permeability of OPU/SiO₂ aerogel coated fabrics decreased gradually with the increase of OPU ratio. Besides, the moisture permeability of OPU/SiO₂ aerogel coated fabrics declined evidently with the increase of OPU ratio in the OPU/SiO₂ aerogel. The development of OPU/SiO₂ aerogel coated fabrics has showed new strategy of fabricating stronger thermally insulating textiles using OPU/SiO₂ aerogel composites.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/supplementary material, further inquiries can be directed to the corresponding author/s.

AUTHOR CONTRIBUTIONS

LL: data curation, investigation, and writing—original draft. ZL: methodology. CW: supervision. CW and WL: review and editing. All authors contributed to the article and approved the submitted version.

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Conflict of Interest: The authors declare that the research was conducted in the absence of any commercial or financial relationships that could be construed as a potential conflict of interest.

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