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A simple and efficient method for the preparation of SiO_2/PI/AF aerogel composite fabrics and their thermal insulation performance

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ABSTRACT

As a new type of insulation material, aerogels are characterized by a high specific surface area, high porosity, low density and low thermal conductivity, which makes them a new alternative to the use of traditional insulation materials. In this paper, a simple method for preparing aerogel insulation materials is proposed. Specifically, SiO_2/PI/AF (aramid fiber) aerogel composite fabrics were successfully obtained by combining coating technology and finishing processes to use tetraethoxysilane (TEOS) as the precursor, polyimide (PI) powder as the reinforcing agent, and nonwoven AF as the substrate. These composite fabrics were characterized using field-emission scanning electron microscopy (FESEM), tensile testing with an Instron 5967, Fourier transform infrared spectroscopy (FT-IR) and thermal infrared imaging. The results show that the composite fabrics exhibited excellent performance and could effectively block heat transfer. Moreover, the thermal conductivity of the front decreased from 4.08 to 3.91 (W/cm·°C) $\times$ 10^{-4}. This work provides a novel method for the structural design of thermal insulation clothing.

1. Introduction

Thermal insulation materials refer to insulating materials or composites that shield from the effects of heat flow [1]. They are generally lightweight, highly porous and poorly thermally conductive and can sometimes be employed under freezing and low temperature conditions. Therefore, thermal insulation materials are also referred to as cold insulation materials [2,3].

Thermal insulation materials have been widely applied to prevent heat loss in industrial thermal equipment and pipelines because they save energy, maintain a constant temperature and insulate from heat. For example, thermal insulation materials for residential buildings show advantages in attenuating sound propagation, maintaining indoor temperature stability, and reducing greenhouse gas emissions [4]. In addition, high-performance insulation materials have been adopted by the aerospace industry to isolate the heating environment in spacecraft during hypersonic flight and ensure flight safety [5]. Futhermore, heat insulation materials also play an important role in protective clothing as the main raw material of the insulation layer.

Heat insulation materials cover many categories in our daily lives, and their performance varies greatly. Insulation materials normally include traditional insulation materials, the most advanced insulation materials (vacuum insulation boards, inflatable insulation boards, aerogels, and phase change materials) and possible future insulation materials (gas insulation materials, nanoinsulation materials, and dynamic insulation materials) [6]. Traditional insulation materials can be divided into two categories according to their composition: organic insulation materials and inorganic insulation materials. Organic insulation materials include natural plant fiber [7], polyurethane foam [8], polystyrene foam [9] and extruded polystyrene foam [10]. Inorganic insulation materials mainly refer to rock wool/mineral wool [11,12], expansive minerals [13,14] and foam geopolymer [15]. Organic insulation materials have low thermal conductivity [16] and excellent thermal insulation performance but poor fire resistance and durability [16–19]. They burn easily and release large amounts of toxic gases, resulting in many fire accidents and high manufacturing costs [20–23]. Inorganic insulation materials have the advantages of being nonflammable, inexpensive, durable, and good flame retardants and can be used to avoid the shortcomings of organic materials [24–26]. However, their thermal conductivity and apparent density are ordinarily less than those of

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organic insulation materials [27]. For example, although the thermal conductivity of microporous calcium silicate board is small, it exhibits high brittleness, high water absorption and poor corrosion resistance. Commonly used foam glass shows good thermal insulation performance, waterproofing, and imperviousness, but it suffers from low impact strength, fragility, and poor weather resistance, among other shortcomings. In addition, the insulation performance of aluminum silicate fiber, mineral cotton and other inorganic fiber products decreases significantly at high temperatures, despite their good mechanical properties, high specific modulus, fatigue resistance and water absorption.

The emergence of aerogels has solved the problems of traditional insulating materials. Since Kistler [28,29] discovered aerogels in 1931, the excellent properties of aerogels have attracted great interest from researchers. Aerogels are unique porous materials consisting of cross-linked solid nanoparticles (the particle size is usually 2–5 nm) [30] and nanopores (the average pore size is generally 10–100 nm) [31] that are characterized by a large specific surface area, high porosity and low density. Aerogels are multifunctional nanomaterials that insulate from heat and sound, protect against fire and have become a potential alternative to traditional thermal insulation materials [32]. In recent years, the development of aerogel blankets, aerogel-filled sandwich plates, aerogel embedded glass and aerogel coating materials resulted in promising advancements in the areas of building heat insulation and energy savings [31,33,34]. For example, Jia et al. [32] attempted to add aerogel as an insulation aggregate to cement-based composites to prepare activated carbon. The results showed that the aerogel could efficiently reduce the thermal conductivity of cement-based composites and hinder thermal conductivity. Liu et al. [35] utilized a SiO_2 aerogel coating to improve the fire resistance of tunnel linings, whereas Naeem et al. [36] identified an aerogel as an insulation substrate in protective clothing.

According to the literature [37], the aerogel preparation process is well developed and easy to operate. During the preparation of aerogels, nanoparticles or macromolecules in the material cross-link to form wet gels in the dispersion process, and the solutes in the wet gels are then removed via the corresponding finishing techniques to ultimately yield aerogel solids [38]. Aerogels have many advantages, such as their light weight, good heat insulation performance and simple preparation, and they are expected to be applied in the field of heat insulation protective clothing.

In traditional heat-resistant clothing, the insulation layer fabric is generally made of flame-retardant fiber felt, such as flame-retardant cotton fiber, flame-retardant wool fiber, aramid fiber, polysulfonamide fiber, polybenzimidazole fiber and other high-performance fibers [39]. To date, most protective garments rely on increasing the number of fiber layers to insulate from high temperatures, but this results in protective clothing that is too thick, inhibits the movement of wearers and may even generate thermal stress reactions [40].

In this paper, we present a simple and efficient method to prepare SiO_2/PI/aramid fiber (AF) aerogel composite fabric via the sol-gel method. Because nonwoven AF exhibits favorable thermotolerance, it was chosen to serve as the substrate of the heat insulation layer and was combined with SiO_2/PI composite aerogels. The obtained composite fabrics exhibited greatly improve the thermal protection at higher temperatures. Consequently, our design of porous hierarchies offers ample opportunities to pursue the great heat insulation properties of thermal protective clothing that is convenient to wear in high- and low-temperature environments.

2. Experimental

2.1. Materials

The length and width of the nonwoven AF were set to 10 cm × 10 cm. Tetraethoxysilane (TEOS), ethanol (EtOH, AR, 99%), hydrochloric acid (HCl), ammonium hydroxide (NH_4OH), n-hexane (95%) and trimethylchlorosilane (TMCS, 98%) were used as precursors, solvents, catalysts, solution displacers and surface modifiers, respectively. All reagents were chemically pure (China). Polyimide (PI) powder was selected as the reinforcing material for the aerogel because of its good mechanical properties, high temperature resistance and excellent thermal stability.

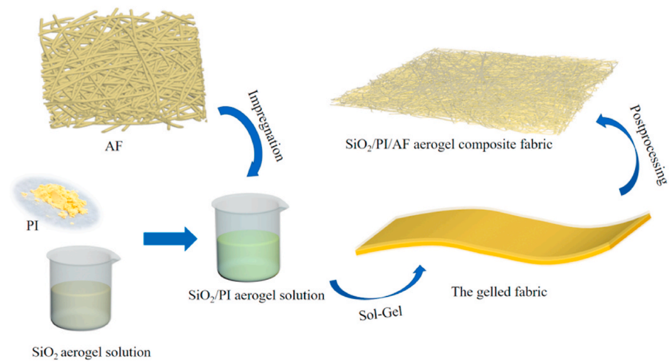

Fig. 1. Schematic of SiO_2/PI/AF aerogel composite fabric preparation.

2.2. Preparation of silica aerogel/aramid fiber composite

The flow chart of the preparation of the SiO_2/PI/AF aerogel composite fabric is shown in Fig. 1, which indicates that the formation of the composite fabric primarily consists of two steps. First, a SiO_2/PI aerogel aqueous solution was prepared. Second, nonwoven AF were impregnated with SiO_2/PI aqueous solution. Thereinto, the primary approach for forming silica composite aerogels was the sol-gel method [41]. In the first step, TEOS, EtOH, H_2O and HCl were mixed at a given proportion, heated and stirred at 60 °C for 90 min. The obtained mixture was called solution A. In the second step, the solution of EtOH, H_2O, and NH_4OH was denoted solution B. Solutions A and B were blended and stirred for a period of time to form a silica aerogel aqueous solution after hydrolysis and polycondensation. PI was then uniformly dispersed in the silica aqueous solution to obtain SiO_2/PI solutions containing 0.6%, 0.8%, 1.0%, 1.2%, and 1.4% PI. Subsequently, nonwoven AF were impregnated with different concentrations of the SiO_2/PI mixtures. The gelation lasted for 48 h, yielding the SiO_2/PI/AF fabric. This composite was allowed to age for 12 h with EtOH, exchanged with n-hexane solvent for 12 h, and modified with TMCS/n-hexane solution for 12 h. Finally, the SiO_2/PI/AF aerogel composite fabric was obtained by drying at 60 °C. The properties of AF and SiO_2/PI/AF aerogel composite fabrics were characterized, and their heat insulation properties were compared.

2.3. Characterization

A field emission scanning electron microscope (FESEM) was applied to observe the microstructures of the surface of the SiO_2/PI/AF aerogel composite fabric. Furthermore, a Instron 5967 universal testing machine (Instron Ltd., America) was used for the tensile test. Fourier transform infrared spectroscopy (FT-IR) confirmed the surface functional groups of SiO_2/PI/AF and AF. The temperature on the fabric surface was detected with a thermal infrared imager at a given temperature. The thermal conductivity was measured at room temperature with a contact cold-warm sense tester.

3. Results and discussion

3.1. Microstructures

Fig. 2 presents the microscopic morphologies of AF and the SiO_2/PI/AF aerogel composite fabrics with PI concentrations of 0.6%, 0.8%,

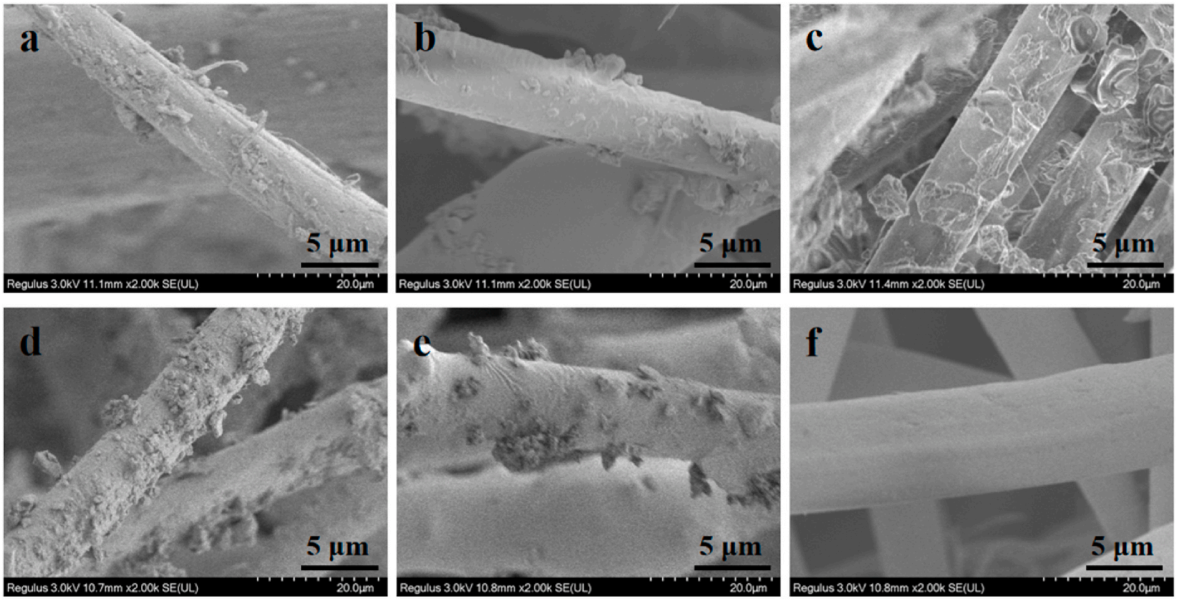

Fig. 2. FESEM images of SiO_2/PI/AF aerogel composite fabrics with PI concentrations of 0.6% (a), 0.8% (b), 1.0% (c), 1.2% (d), 1.4% (e) and FESEM image of AF (f).

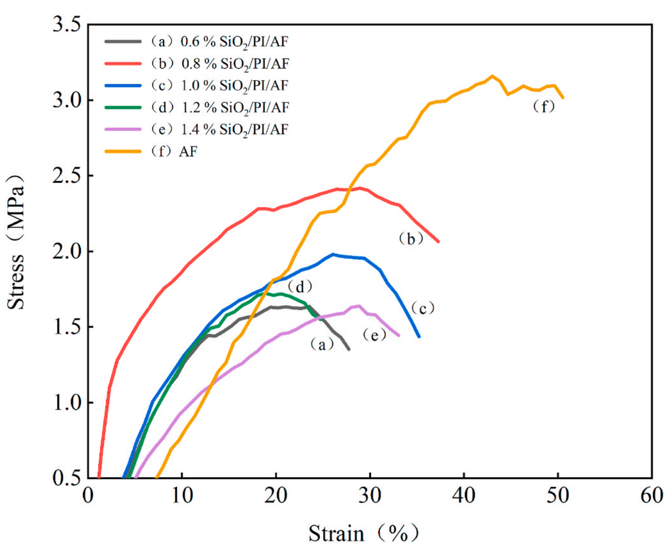

Fig. 3. Stress–strain curves of composite fabrics with different PI concentrations.

Table 1
The breaking stress and strain of composite fabrics with different PI concentrations.

	Breaking stress (MPa)	Breaking strain (%)
0.6% SiO_2/PI/AF	1.60 ± 0.14	20.89 ± 2.72
0.8% SiO_2/PI/AF	2.43 ± 0.29	25.28 ± 3.12
1.0% SiO_2/PI/AF	1.77 ± 0.43	21.48 ± 4.01
1.2% SiO_2/PI/AF	1.64 ± 0.23	26.54 ± 3.32
1.4% SiO_2/PI/AF	1.67 ± 0.18	28.85 ± 2.41
AF	3.02 ± 0.54	39.71 ± 3.35

1.0%, 1.2% and 1.4%. Fig. 2 a-e show that a layer of SiO_2/PI aerogel is attached to the surface of the gel-impregnated AF. Of these fabrics, the fiber surface in Fig. 2b is relatively smooth. Fig. 2f displays the surface morphology of AF.

Fig. 4. Digital photograph of the SiO_2/PI/AF aerogel composite fabric laid on a flower.

3.2. *Mechanical properties of composites*

The stress–strain curves for composite fabrics with different PI concentrations are exhibited in Fig. 3. These data show that the mechanical properties of the SiO_2/PI/AF aerogel composite fabrics are much worse than those of nonwoven AF. However, among the five fabrics containing SiO_2/PI composite aerogels, the 0.8% SiO_2/PI/AF featured the best tensile performance. As shown in Table 1, the breaking stress and breaking strain of the 0.8% SiO_2/PI/AF are 2.43 ± 0.29 MPa and 25.28 ± 3.12%, respectively. These mechanical properties may be related to the surface morphology of the AF. According to Fig. 1, the fiber surface of AF is the smoothest, followed by the 0.8% SiO_2/PI/AF aerogel composite fabric. This feature may be mainly due to the large size of SiO_2/PI aerogel particles attached to the AF, which increases the friction between the fibers and causes fiber sliding. Second, the low intensity and brittleness of the SiO_2/PI aerogel may lead to the poor adhesion of aerogel particles and interfacial adhesion. Therefore, the aerogel adhered to the fiber surface after impregnation will weaken the

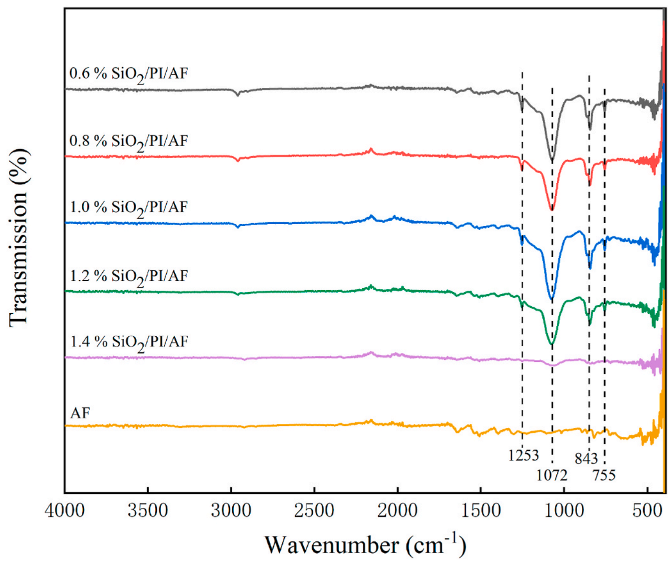

Fig. 5. FT-IR spectra of composite fabrics with different PI concentrations.

fiber and make it brittle. Third, in the SiO_2/PI/AF aerogel composite fabric, the AF itself bears major external force, whereas the SiO_2/PI aerogel is only used as filler, jointly forming a dense structure and increasing the stiffness of the SiO_2/PI/AF. In summary, the mechanical properties of the composites are decreased [42].

Fig. 4 depicts a digital photograph of a SiO_2/PI/AF aerogel composite fabric placed on a flower. Surprisingly, the petals remain erect. This result suggests that although the strength of the SiO_2/PI/AF aerogel composite fabric is not as good as that of pure nonwoven AF, the composite fabric continues to be lightweight due to the addition of the SiO_2/PI aerogel. This lightweight nature plays a vital role in the development of high-temperature protective clothing. For instance, it can make fire clothing more portable and less cumbersome.

3.3. FT-IR spectrum

The purpose of the impregnation with SiO_2/PI solution is to make the aerogel adhere to the surface of the AF. Fig. 5 shows the FT-IR spectra of composite fabrics with different PI concentrations. The characteristic peaks of SiO_2 are attributed to the Si–O–Si antisymmetric stretching vibration located at approximately 1253 cm^{-1} and the asymmetrical stretching vibration at 1072 cm^{-1} [43,44]. In addition, the presence of an imine linkage at 755 cm^{-1} and the imide ring at 843 cm^{-1} are characteristic of polyimide [45]. These functional groups prove the presence of SiO_2/PI aerogel on the fiber and also indirectly indicates the successful preparation of the SiO_2/PI/AF aerogel composite fabrics.

3.4. Thermal infrared images

Thermal infrared images taken by a thermal infrared imaging device to describe the thermal insulation properties of fabrics are given in Fig. 6a–f. Before the experiment began, the platform was heated to 60 °C and maintained at that temperature for the duration of the test [46]. The 0.6%, 0.8%, 1.0%, 1.2% and 1.4% SiO_2/PI/AF aerogel composite fabrics and AF specimens to be tested were placed at the center of the heating platform, and the images were recorded after 10 min. As shown in Fig. 6, the actual surface temperature of 0.6% SiO_2/PI/AF was approximately 52 °C. The temperatures of the 0.8%, 1.0%, 1.2% and 1.4% SiO_2/PI/AF aerogel composite fabrics were 47.8 °C, 47.5 °C, 52.5 °C and 51.8 °C, respectively. AF had a surface temperature of 50 °C. In summary, only 0.8% and 1.0% SiO_2/PI/AF had lower temperatures than AF, indicating that their infrared thermal radiation was low. This finding also confirms that the high infrared reflectance of the SiO_2/PI aerogel contributes to improving the thermal insulation performance of the fabric.

3.5. Thermal insulating property

Table 2 lists the thermal conductivity of composite fabrics and nonwoven AF. The thermal conductivity of the front of the AF was 4.08 (W/cm·°C) × 10^{-4} and that of the backside was 4.12 (W/cm·°C) × 10^{-4}. Among the five composite fabrics, only the 0.8% SiO_2/PI/AF had lower thermal conductivity than the nonwoven AF at 3.91 (W/cm·°C) × 10^{-4} and 3.97 (W/cm·°C) × 10^{-4} on the front and back, respectively. The heat

Table 2
Thermal conductivity of composite fabrics with different PI concentrations.

Sample	Thermal conductivity ([W/cm·°C] × 10^{-4})	
	Front	Back
0.6% SiO_2/PI/AF	5.05	4.90
0.8% SiO_2/PI/AF	3.91	3.97
1.0% SiO_2/PI/AF	5.36	5.25
1.2% SiO_2/PI/AF	5.75	5.60
1.4% SiO_2/PI/AF	4.83	5.10
AF	4.08	4.12

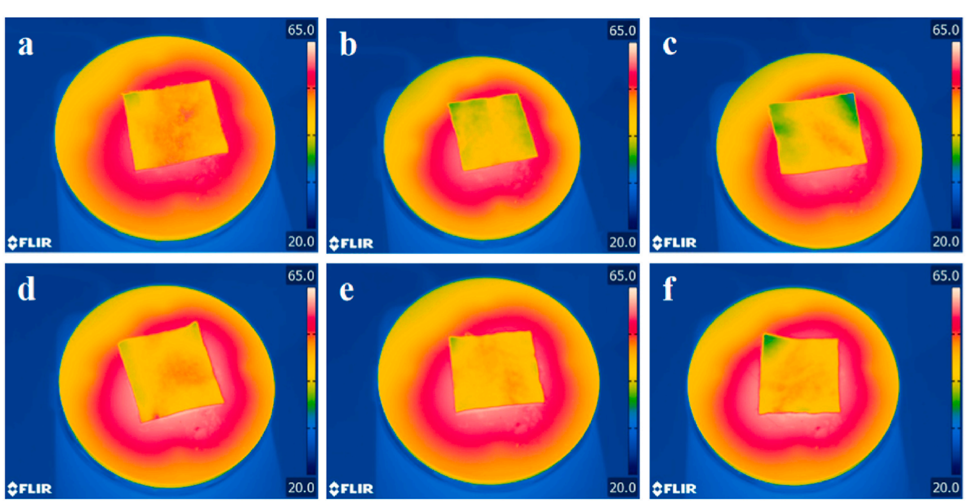

Fig. 6. Thermal infrared images of SiO_2/PI/AF aerogel composite fabrics with PI concentrations of 0.6% (a), 0.8% (b), 1.0% (c), 1.2% (d), 1.4% (e) and thermal infrared image AF (f).

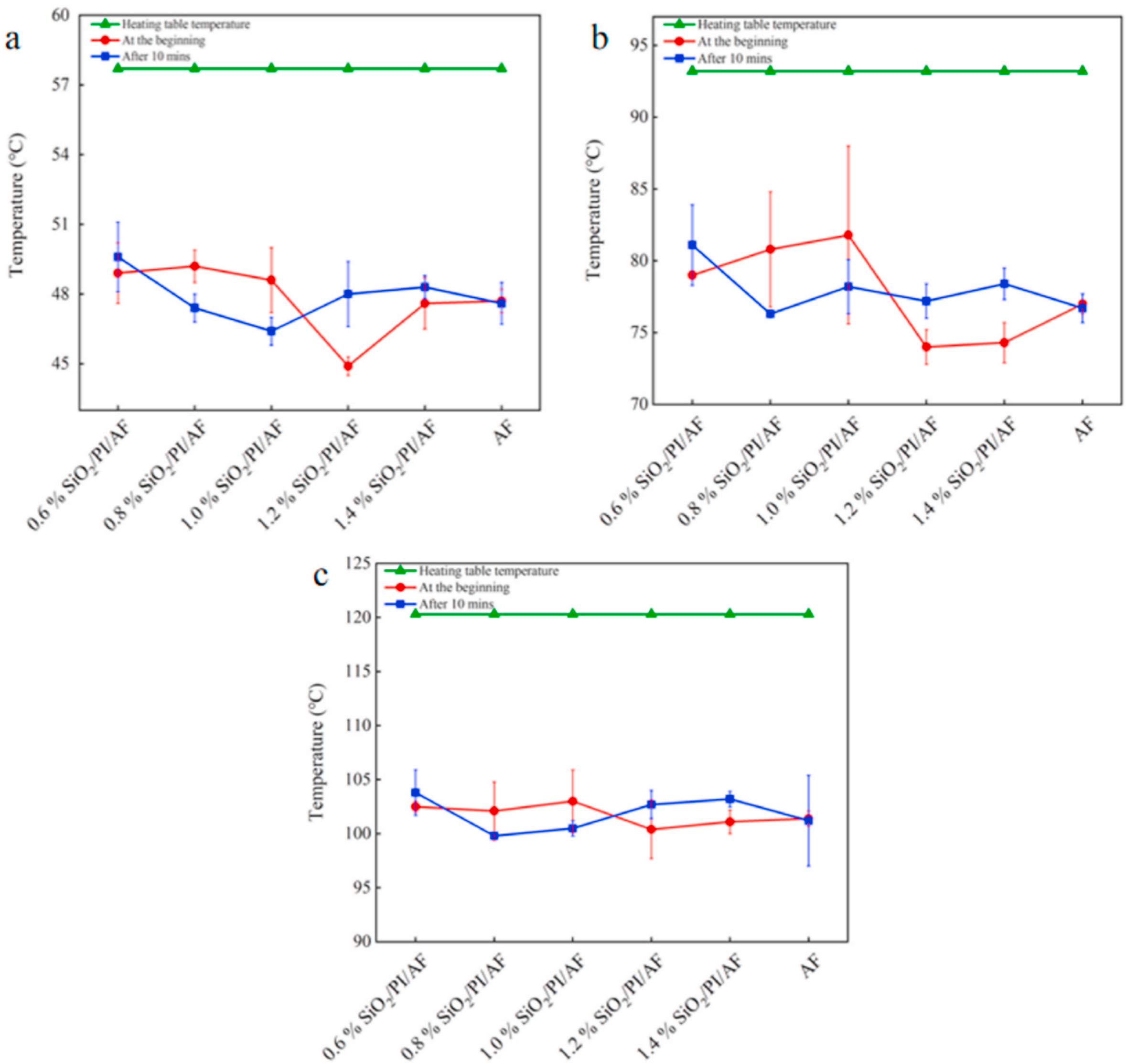

Fig. 7. The surface temperatures of composite fabrics with different PI concentrations on the heating table at 60 °C (a), 90 °C (b), and 120 °C (c).

conductivity is one of the most direct and important parameters to judge the thermal insulation performance of fabrics. Based on these data, the heat insulation capability of 0.8% SiO_2/PI/AF aerogel composite fabric is considered better than that of nonwoven AF and other composite fabrics. Moreover, the thermal conductivity of the composites prepared in this work have more advantages than other thermal insulation materials. For example, the thermal conductivity of composites fabricated by Xiong et al. [47] exceeded 4.00 (W/cm·°C) × 10^{-4}, and Akcagun et al. [48] reported a material with a minimum thermal conductivity of approximately 4.30 (W/cm·°C) × 10^{-4}. In addition, the thermal conductivity of the silica aerogel microsphere coating discussed by He et al. [49] was large, i.e., 20.00 (W/cm·°C) × 10^{-4}.

Measuring the temperature variations of the fabric surface within a given time is also an effective method to assess fabric insulation performance. Therefore, we maintained the heating table at 60 °C, 90 °C or 120 °C and placed the specimens in the middle of the heating table to observe their surface temperature. The instantaneous and stable temperatures after 10 min of the specimens were determined with an infrared temperature measuring gun. The experimental results are shown in Fig. 7a–c. We found that the temperatures of all samples were less than those of the heating table, indicating that they definitely insulate from heat. However, only the temperatures of 0.8% and 1.0% SiO_2/PI/AF decreased after stabilization. Finally, the surface temperatures of composite fabrics under three different conditions were comprehensively compared. After stabilization, the surface of 0.8% SiO_2/PI/AF was coolest, i.e., 0.8% SiO_2/PI/AF aerogel composite fabric was a better thermal insulator than aramid and other composite fabrics. This feature may be due to the following: the surfaces of fibers impregnated with the gel are smoother than those of other fabrics and the gel particles are relatively small, occupying fewer pores between the original fibers, thus retaining more still air. However, as the temperature of the heating table increased, the error also markedly increased. This finding may be due to the uneven distribution of some aerogels, which resulted in a large temperature difference between points on the composite fabric.

4. Conclusions

In short, we combined aerogel preparation and coating technology to develop a new type of lightweight and heat-resistant insulation material: SiO_2/PI/AF aerogel composite fabric. In addition, fabrics with different thermal insulation properties were obtained by adjusting the concentration of PI in the aerogel. The FESEM micrographs show that the SiO_2/PI aerogel is uniformly attached to the surface of the AF, which may insulate well from heat. That is, smaller aerogel particles result in a more uniform coating and reduced thermal conductivity. When the PI concentration was set to 0.8%, the composite fabric exhibited excellent mechanical properties and thermal insulation performance. Most importantly, the thermal conductivity of 0.8% SiO_2/PI/AF aerogel composite fabric was lower than that of pure nonwoven AF, 3.91 (W/cm·°C) × 10^{-4} and 3.97 (W/cm·°C) × 10^{-4} on the front and back, respectively. Thus, as a novel and efficient thermal insulation material, SiO_2/PI/AF aerogel composite fabrics provide endless inspiration for the structural design of thermal insulation clothing.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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