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APPLICATION OF COATING MIXTURE BASED ON SILICA AEROGEL TO IMPROVE THERMAL PROTECTIVE PERFORMANCE OF FABRICS

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Abstract:

The main aim of this research is to improve the protective thermal performance of fabrics. Flame-resistant fabrics characterizing comparable thermal properties were chosen, cotton fabric with a flame-retardant finish and Nomex® fabric. To improve thermal parameters the coating mixture, based on silica aerogel, was applied on one side of the sample surface. Parameters such as the thermal conductivity, resistance to contact, and radiant heat were determined based on the standards, which set high expectations for the protective clothing. Analysis of the coated fabrics surfaces was conducted based on confocal microscopy. It was found that the coating mixture caused a decrease in thermal conductivity. All the modified fabrics reached 1st efficiency level of protection against contact and radiant heat. The best sample from the point of view of protection against contact and radiant heat was modified cotton fabric with a flame-retardant finish. The coating mixture contained 45 wt% of silica aerogel. Moreover, better adhesion of the coating mixture to the cotton fabric compared with Nomex® fabric was observed.

Keywords:

silica aerogel; woven fabrics; thermal properties; protective clothing; hot work environment

1. Introduction

The sol-gel processing method is very popular and used to synthesize materials, especially metal oxides. It consists of the system transition from a liquid sol to a solid phase in the form of a gel. The sol-gel process includes the following stages: solution formation, gelling, aging, drying, and thickening [1–3]. The most important advantages of the sol-gel process are its simplicity and the fact that the process is an effective method for the production of high-quality materials such as aerogels. Aerogel is a gel made up of a microporous solid, in which the dispersed phase is gas. Among different kinds of aerogels [3, 4], silica aerogels SiO_2 are popular, because they exhibit unique properties. Silica is a natural environmentally-friendly ingredient. The aerogels are characterized by small particle size, from 2 nm to 4,000 mm (depending on a form, powder, or granules) [1, 3] and pore diameter, ~0.02 mm [1,5], and contain 80–99.8% of air [6]. The materials have a very low density, 120–150 kg/m^3 [7–9]. Specific surface area is in the range of 500–1,000 m^2/g [1, 6, 10]. Silica aerogels have a melting point of ~1,200°C [11]. They have low thermal conductivity, 0.004–0.018 W/(m·K) [7–9, 12]; a low refractive index, 1.0–1.1; a low dielectric constant, ~1.1 for a density of 0.1 g/cm^3 [5, 11]; and also low speed of sound, 100 m/s for a density of 0.07 g/cm^3 [11].

Generally, silica aerogels are fragile and brittle because of the interparticle connections and make them inapt for load-bearing applications [11]. Aerogels cannot be easily applied due to their weak strength resulting from their high porosity and cracking along the drying process. A quite effective solution is combining aerogels with textile structures [8].

Due to the wide potential of aerogel-based materials, its applications can be found in all industrial sectors. Aerogels can be used in construction as thermal and acoustic materials [12–14]. Properties of aerogels have made them a new material for aerospace applications, sensors and coatings, energy generation and storage, and biomedical devices and implants [11, 13, 15, 16]. They were also used in the textile sector [7,9,16–19]. This material, created as a result of the use of aerogel granules between glass fiber and Kevlar fabrics, enables protection against laser radiation [7]. Aerogel blankets were produced as a composite consisting of silica aerogel and fibrous reinforcement [16]. The applied reinforcement made it possible to change the fragile aerogel into a durable and flexible material. The blanket manufactured by Aspen Aerogels Inc. was produced by depositing a thin layer of aerogel on the fabric surface.

The most commonly used solution, especially in the protective clothing protecting against extremely low temperatures, is that of nonwoven composites consisting of a silica aerogel matrix and a reinforcing material, which is a nonwoven fabric [20]. The manufactured products are sometimes laminated to eliminate undesirable aerogel dust particles. Scientists have shown that by incorporating silica-based aerogels into nonwovens, the thermal properties of firefighters' protective clothing can be significantly improved. Another example of the use of aerogel in textiles is insoles for shoes, which were used by climbers [1]. Low thermal conductivity makes silica aerogels desirable for insulating applications such as cover layers for the protective clothing used in a hot work environment. As research was shown [6] aerogels can be used successfully in the construction of garments protecting against thermal factors to improve their protective properties by increasing thermal insulation. The silica aerogel was used in textile packaging intended for implementation in protective clothing [21]. Three different packaging designs were made in the form of removable inserts filled with aerogel and assessed for thermal resistance. The increase of resistance to radiant heat and convection was observed. Silica aerogel was also used for the modification of the basalt fabric surface [22]. The aerogel layer improved resistance to contact heat and thermal radiation.

Working in a hot environment is often associated with heat loads resulting from both high temperatures and increased physical activity of the employee. At workplaces, the employee is exposed to hot factors in the form of a flame, thermal radiation, or contact heat. There is a hot microclimate, which, in combination with the protective clothing and physical effort used, results in an unfavorable hydrothermal condition under the clothing, which causes a feeling of discomfort. Reduction of discomfort can be achieved through appropriately selected undergarments and underwear, taking into account materials supporting the collection of heat, water vapor, and liquid. Scientists are constantly striving to produce innovative textile materials intended for personal protective equipment, which will show good protective and mechanical properties, and are also characterized by good comfort of use [8, 10, 23]. Therefore, the aerogel layer is a promising solution for thermal insulation and protection [9, 10, 18].

The main aim of the study is to improve the thermal protective performance of fabrics using a coating mixture based on the silica aerogel. Flame-resistant fabrics characterizing comparable thermal properties were chosen. However, they don't meet the requirements described in the standards [24–26]. To improve thermal parameters (thermal conductivity, resistance to contact, and radiant heat) the coating mixture based on aerogel was applied on the sample surface.

2. Materials

Two twill-weave fabrics used in the production of protective clothing intended for use in a hot work environment were selected for the tests. The Nomex® fabric by DuPont (Wilmington, USA) and the cotton fabric with a flame-retardant finish by XinXiang YuLong Textile (Xinxiang, China) are flame-retardant and arc-proof materials. The raw material composition and selected parameters are presented in Table 1.

Three samples of each fabric with dimensions of 24 cm x 10 cm were prepared and acclimated in the temperature of 20°C ± 2°C and the relative humidity of 65% ± 5% [27].

A coating mixture was prepared, which consisted of aerogel, adhesive mean, and flame retardant. The base of the mixture was the Enova® Aerogel by Cabot Co. USA. The selected silica aerogel is described in detail in Table 2. The aerogel enables the production of coatings with a smooth and uniform finish, high abrasion resistance, low thermal conductivity, and safe contact with the skin.

The Bonatex® KCB adhesive mean was used to improve the adhesion of the coating mixture to the substrate. It is a dispersion adhesive mean based on water dispersions of special synthetic resins and polymers. The adhesive mean does not contain any organic solvents. It is designed for bonding textiles, natural and synthetic leather, some plastics, as well as cork, cardboard, and similar materials. It contains a mixture of 5-chloro-2-methyl-4-isothiazol-3-one and 2-methyl-4-isothiazolin-3-one and may produce an allergic reaction.

The Burnblock® fire-retardant preparation was used to improve the thermal properties of the coating mixture. It is a harmless, non-toxic, ecological, and colorless product. It protects material capable of absorbing various types of liquid, such as fabrics, carpets, carpets, paper, and raw wood. The impregnated material becomes stiffer. Burnblock® firestop fluid has a limited ability to be absorbed by artificial materials, made of 100% PP, PE, PVC. It protects fabrics for five years if the fabric is

Table 2. Characteristics of the silica aerogel [18].

Particle size	2–40 μm
Pore diameter	~20 nm
Particle density	120–150 kg/m^3
Surface chemistry	Hydrophobic
Surface area	600–800 m^2/g
Thermal conductivity at 25°C	0.012 W/(mK)

Table 1. Characteristics of selected fabrics.

Woven fabric	Raw material composition	Thickness, mm	Mass per square meter, g/m^2
Nomex® fabric	93% m-aramid, 5% p-aramid, 2% antistatic fibers P140	0.37	266
Cotton fabric	100% cotton fibers, fabric with the flame-retardant finish	0.66	376

not subjected to any wear processes (abrasion, chemical, or mechanical processes).

The coating mixture was prepared in two variants, which differed from each other in the content of individual ingredients. It was assumed that the percentage of aerogel in the mixture is 45 wt% and 60 wt% for variants A and B, respectively, and the particle density equals 120 kg/m^3. The remaining part is adhesive mean and flame-retardant preparation in a 75:25 ratio composition of adhesive mean/flame-retardant preparation. The variants of the coating mixture are presented in Table 3.

Three ingredients of coating mixture—aerogel, adhesive mean, and flame-retardant—were prepared in the amount specified in Table 3 for chosen variant. Next, the ingredients were mixed in the vessel until a homogeneous mixture was obtained. The coating mixture of 90 ml was divided into three equal parts. The mixture was applied to one side of the surface of each of the three fabric samples using a woody spatula. The designations of all obtained samples variants are summarized in Table 4.

The drying process was carried out at a temperature of 40°C and for 20 minutes as recommended [28].

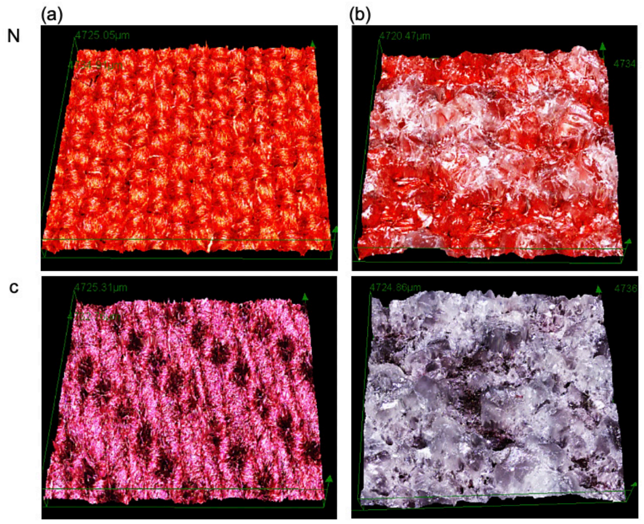

Figure 1. Woven fabric surfaces: (a) unmodified; (b) modified with coating mixture.

The image of the Nomex® fabric and cotton fabric with a flame-retardant finish before and after the application of the coating mixture was taken with the laser confocal microscope Olympus LEXT OLS5100 (Tokyo, Japan) at the total visual magnification of 100 x and shown in Figure 1.

3. Methods

3.1. Thermal conductivity

The thermal comfort of clothes depends especially on thermal conductivity. Thermal conductivity defines the energy flow, in the form of heat, through the mass of the sample due to an external temperature difference. It is a material property that characterizes the material in stationary heat flow conditions.

Thermal conductivity measurements of samples were made on the Alambeta device (Sensora, Liberec, Czech Republic) [29]. The test is limited to measuring the amount of heat that flows through the tested sample of material placed between two metal plates. The top plate is heated to 35°C and reflects the body temperature. The temperature of the bottom plate had the ambient temperature. The modified sample is placed between the plates so that the heat flow is through the fabric toward the aerogel layer. The plates have adhered to the tested sample with a pressure of approximately 200 Pa. Measurements were conducted under normal climate conditions [27].

3.2. Resistance to contact heat

The test method based on the ISO 12127-1:2016-02 [24] standard consists of placing the sample on a calorimeter and then putting it in contact with a heating cylinder heated to a temperature, with a value in the range of 100°C –500°C. In the case of the modified sample, it was placed on the calorimeter on its unmodified side.

The threshold time t_t, i.e., the time from the moment of the first contact with the heating cylinder until the temperature of the calorimeter increases by 10°C from the initial value, is recorded. The EN ISO 11612:2015 [25] standard distinguishes three efficiency levels of protection against contact heat: the

Table 3. Variants of the coating mixture.

Ingredients	Variant A		Variant B	
	ml	wt%	ml	wt%
Aerogel	41	45	54	60
Adhesive mean	37	41	27	30
Flame-retardant preparation	13	14	9	10
Total	90	100	90	100

Table 4. Samples destinations.

Sample	Before application of coating mixture	After application of variant A coating mixture	After application of variant B coating mixture
Nomex® fabric	N	NA	NB
Cotton fabric	C	CA	CB

first efficiency level, when $t_t \in [5.0, 10.0)$ s; the second, when $t_t \in [10.0, 15.0)$ s; and the third, when $t_t \geq 15.0$ s.

Single-layer or multi-layer garments for which protection against contact heat is declared and tested in accordance with ISO 12127-1:2016-02 [24] at a temperature of 250°C should meet at least the first efficiency level.

3.3. Resistance to radiant heat

Thermal radiation resistance was tested according to ISO 6492:2002 [26] and method B, with the use of thermal radiation density equal to 20 kW/m^2. The test is carried out by placing a sample of the tested material on the face of a copper calorimeter and subjecting it to thermal radiation with a flux density equal to 20 kW/m^2, recording the time of temperature rise of the calorimeter behind the sample by 12°C and 24°C. In the case of the modified sample, it was placed on the face of a calorimeter on its unmodified side.

The relative heat transfer index $RHTI_{12}$, expressed in seconds, is the time for the calorimeter temperature to rise by 12°C, while the relative heat transfer index $RHTI_{24}$ is the time for the calorimeter temperature to rise by 24°C. The basic protective parameter is the index $RHTI_{24}$. The efficiency levels of protection are described in standard EN ISO 11612:2015 [25]. The first efficiency level of protection against radiant heat is obtained when $RHTI_{24} \in [7.0, 20.0)$ s, the second one when $RHTI_{24} \in [20.0, 50.0)$ s, the third one when $RHTI_{24} \in [50.0, 95.0)$ s, and the fourth one when $RHTI_{24} \geq 95.0$ s.

3.4. Characterization of surface texture

Assessment of surface texture (especially surface roughness) can be carried out by a surface profile and areal surface methods [30]. Surface profile measurement is achieved by measuring a line across the surface and representing that line mathematically as a height function with lateral displacement. Areal measurements provide data points that are used to receive an areal map that is a closer representation of the real surface. Some particular parameters were used to assess irregularities of woven fabrics surfaces based on standards ISO 4287 [31] and ISO 25178-2 [32].

The maximum height of profile R_z is the sum of the maximum peak height Z_p, and the maximum valley depth Z_v of a profile within a sampling length l_r. The sampling length is used for identifying the irregularities characterizing the profile under evaluation and is numerically equal to the characteristic wavelength of the profile filter λ_c. The profile filter defines the intersection between the roughness and waviness components and is described in detail in [31]. The evaluation length l_n is the length in the direction of the x-axis used for assessing the profile under evaluation.

The arithmetic mean deviation of the assessed profile R_a is the arithmetic mean of the absolute ordinate values $Z(x)$, the height of the assessed profile at any position x, within a sampling length l_r. This parameter is expressed by the following formula:

$$R_a = \frac{1}{l_r} \int_0^{l_r} |Z(x)|\, dx. \tag{1}$$

The maximum height of the scale-limited surface S_z is the sum of the maximum peak height value and the maximum pit height value within the definition area Ω. The pit is a point on the surface that is lower than all other points within a neighborhood of that point. The definition area Ω is a portion of the evaluation area for defining the parameters characterizing the scale-limited surface.

The arithmetic mean height of the scale limited surface S_a is the arithmetic mean of the absolute of the ordinate values within a definition area Ω. This parameter is expressed by the following formula:

$$S_a = \frac{1}{\Omega} \iint_\Omega |Z(x, y)|\, dx\, dy, \tag{2}$$

where (x, y) are the coordinates of the definition area Ω.

The surface L-filter can be used to remove large-scale lateral components from the primary surface or S-F surface described in detail in [32].

4. Results and discussion

Thermal parameters obtained for woven fabrics before applying the coating mixture are presented in Table 5. Next to the mean values of quantities is their standard deviation, given in parentheses.

As can be seen from Table 5, the parameters characterizing the thermal properties of the fabrics before the mixture is applied to their surface are comparable.

The relative heat transfer index $RHTI_{24}$ obtained for fabrics before the coating mixture above 12 s is applied indicates that the fabrics meet the first efficiency level of protection. Due to the low value of the threshold time t_t, fabrics can't be used as materials protected against contact heat.

Samples on the surfaces of which the mixture in variant A or B was applied were tested. Results of thermal conductivity obtained for samples are presented in Figure 2. The coefficient of variation did not exceed 2.7%.

A decrease in thermal conductivity was observed (Figure 2). The parameter is directly related to the structure of textile material.

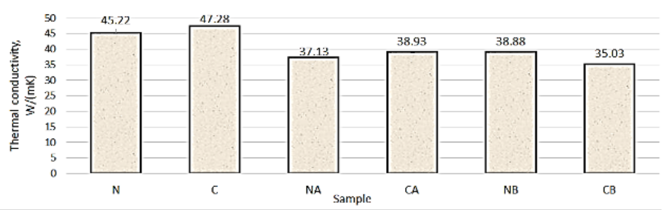

Figure 2. Thermal conductivity for all samples.

Table 5. Thermal properties of woven fabrics before applying the coating mixture.

Sample	Thermal conductivity	Resistance to contact heat (threshold time t_t) for 250°C	Resistance to radiant heat (relative heat transfer index $RHTI_{24}$)
	W/(mK)	s	s
N	45.22 (1.18)	4.33 (0.030)	12.65 (0.141)
C	47.28 (0.46)	4.36 (0.035)	12.55 (0.269)

The thicker the material, the greater its ability to protect against heat loss. An additional layer, in this case, is the coating mixture applied to the sample surface. Values of thermal conductivity of modified samples are comparable due to the application of the same amount of coating mixture to the sample surface. Similar conclusions were reached by other researchers [19], claiming that the thickness of aerogel is important. They found a better ability to retain heat during crossflow for the nonwoven fabric with higher thickness and aerogel content.

The results of measurements of contact heat resistance for all samples for contact temperature 250°C are shown in Figure 3. The coefficient of variation did not exceed 3.1%.

In the case of resistance to contact heat, all samples modified with coating mixture obtained the first efficiency level of protection (Figure 3). It is the minimum performance requirement for clothing protected against contact heat. The best result reached the modified cotton sample (CB) using the coating mixture in variant B containing 60 wt% of silica aerogel. The mean threshold time was equal to 6.99 s for the sample.

We noticed that application of the coating mixture in variant B caused a greater increase in the threshold time value compared with the coating mixture in variant A assuming the same substrate. Therefore, the level of improvement depends on the textile substrate. The results show that cotton fabric (C) with a flame-retardant finish is a better substrate from the point of view of resistance to contact heat than Nomex® fabric (N). The cotton fabric with a flame-retardant finish has greater thickness and surface mass than the Nomex® fabric. Moreover, due to material composition, the cotton fabric is a hydrophilic material, which may cause this phenomenon. The faster absorption of the coating mixture during application to the surface was observed just for cotton samples. On both the cotton and Nomex® fabrics surfaces, the aerogel layer was found (Figure 1b).

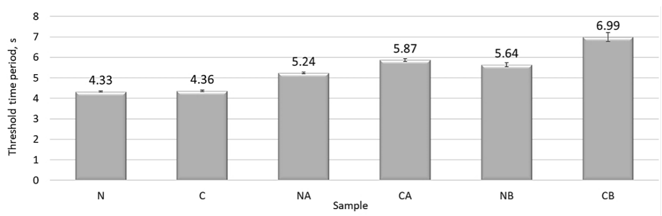

Figure 3. Contact heat resistance for all samples for contact temperature 250°C.

Results of measurements of the radiant heat resistance for all samples are shown in Figure 4. The coefficient of variation did not exceed 2.1%.

The resistance to thermal radiation was improved for all of the samples. Both unmodified and modified fabrics showed the first efficiency level of protection. The highest index $RHTI_{24}$ was obtained for modified cotton fabric (CA) and equal to 19.93 s (with a coefficient of variation of 0.5%). The result obtained for modified Nomex® fabric is also promising ($RHTI_{24}$ = 18.13 s with a coefficient of variation of 1.0%). The samples were modified with the coating mixture in variant A containing 45 wt% of silica aerogel.

The profile and areal methods to compare unmodified woven fabrics surface and fabrics modified with coating mixture were applied in compliance with ISO 4287 [31] and ISO 25178-2 [32]. The laser confocal microscope Olympus LEXT OLS5100 (Tokyo, Japan) at the total visual magnification of 100 x was used for this purpose. The analysis was subjected to the unmodified Nomex® and cotton fabrics, and the woven fabrics modified with coating mixture in one variant, A, due to the same total amount of the mixture used in variants A and B. In the case of modified fabrics, both sides of the sample were analyzed.

The sampling length l_r and the characteristic wavelength of the profile filter λ_c were equal to 1,000 µm and assumed in the surface profile method. The definition area $\Omega \cong 22$ mm^2, in the form of the square, was assumed in the areal surface method. Moreover, high-pass filtering of the surface was applied in the method, wherein the value of the L-filter was equal to 1,000 µm. Results of the confocal microscopy performed are presented in Table 6. Next to the determined R-parameters, the coefficient of variation is given. The measurements were repeated in three places for each sample. In the case of the areal surface method, S-parameters were determined for one definition area indicated on the sample surface.

The values of the R- and S-parameters obtained for N and C samples confirm that the cotton fabric is thicker than Nomex®.

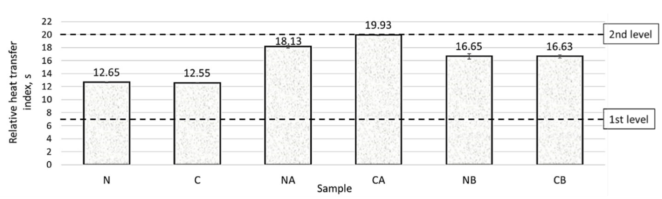

Figure 4. Radiant heat resistance for all samples.

Table 6. Results of analysis of fabrics surface roughness

Sample	R-parameters			S-parameters	
	l_n, μm	R_z, μm	R_a, μm	S_z, μm	S_a, μm
N	5,775.33 (7%)	160.69 (22%)	22.17 (8%)	487.76	26.56
C	5,773.67 (7%)	190.59 (28%)	24.13 (30%)	576.49	27.39
NA-ms(1)	5,773.67 (7%)	206.76 (16%)	38.65 (14%)	700.60	51.12
CA-ms(1)	5,777.67 (7%)	293.98 (25%)	53.64 (26%)	913.32	65.15
NA-ums(2)	5,779.33 (7%)	147.35 (20%)	24.74 (26%)	421.50	27.07
CA-ums(2)	5,782.00 (7%)	149.07 (14%)	21.65 (8%)	483.20	29.42

(1) ms: the modified side of the coated sample; (2) ums: the unmodified side of the coated sample

Moreover, if we compare the variation coefficients of R_a, we can conclude that the cotton fabric has a rougher surface.

Woven fabrics modified with a coating mixture (the modified side of the coated sample) are characterized by higher values of R- and S-parameters. The differences in the roughness of the samples NA-ms and CA-ms increased relative to the original samples, comparing parameters R_a and S_a. The cotton fabric CA-ms remained thicker than the Nomex®. It can be assumed that the coating mixture was not completely absorbed by the cotton fabric substrate. Better adhesion of the coating mixture to the cotton fabric substrate compared to Nomex® fabric was observed. It also results from the raw material composition of the used textile substrates.

The unmodified sides of the coated samples, NA-ums and CA-ums, are characterized by comparable values of R- and S-parameters. The surfaces of fabrics became smoother compared with the original samples due to the substrate on which the samples were placed after the modification process. This indicates that the mixture soaked through the cotton fabric smoothing its unmodified side. The value of the parameter S_z obtained for sample CA-ums is less than the value obtained for the original, unmodified cotton fabric with the flame-retardant finish (C).

5. Conclusions

Aerogel is quickly becoming one of the most attractive materials of the future. Traditional clothing protecting against the high temperature and heat is usually made of thick, multi-layer material systems, the effectiveness of which increases with increasing thickness, while materials with aerogels can provide adequate protection against heat and flame without an excessive increase in their thickness and hence the mass. The coating mixture based on silica aerogel causes a decrease in thermal conductivity.

In the case of resistance to contact heat for the contact temperature of 250°C, all fabrics modified with coating mixture in variants A and B showed the first efficiency level of protection. The minimum performance requirement for clothing protected against contact heat was fulfilled.

The resistance to thermal radiation was improved for all of the modified samples and the first efficiency level of protection against radiant heat was obtained.

The best sample from the point of view of protection against contact and radiant heat was CA. The following parameters were obtained: t_t = 5.87 s and $RHTI_{24}$ = 19.93 s.

The cotton sample with the flame-retardant finish was modified using the coating mixture in variant A containing 45 wt% of silica aerogel. The amount of aerogel turned out to be sufficient to achieve the first efficiency level of protection against contact and radiant heat by the sample.

The differences in the roughness of the modified side of the samples decreased relative to the original samples; better adhesion of the coating mixture to the cotton fabric (C) compared with Nomex® fabric (N) was observed.

Protective clothing is subjected to maintenance. Therefore, before the clothing reaches the end user, testing of the resistance to radiant heat and contact heat after maintenance cycles is necessary.

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