Aerogel Embedded High-performance Fibrous Materials

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SUMMARY OF THE THESIS

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Abstract

The objective of this thesis was to develop aerogel embedded high-performance fibrous materials for thermal insulation application and investigate their performance.

Layered nanofibrous web/silica aerogel/ nonwoven materials were prepared via laminating method by using low melting powder as thermal binding material. The effect of aerogel and thermal adhesive on thermal insulation performance and air permeability was examined. A series model was considered for thermal resistance, the theoretical predicted and measured results were compared and analyzed. Results revealed that novel techniques to combine silica aerogel with high porous textiles with less use of binding materials should be considered.

A novel aerogel-encapsulated fibrous material without using any binding material to bond aerogel particles was developed by using laser engraving technique and laminating method. Thermo Camera, Alambeta device and KES-FT-II Thermolabo were employed to measure thermal performance. Compression test was performed to examine the compression recovery which determines the sustainability of thermal insulation ability. Moreover, a laboratory-made dynamic heat transfer device was used to figure out convective thermal behavior under different airflow velocities and heating conditions. The real-time temperature values of the selected materials were collected and compared. The temperature difference and convective heat transfer coefficient under continuous heating condition were calculated and investigated. The findings could contribute to new developments in flexible aerogel-embedded high-performance textile materials for both industrial and clothing applications.

Flexible polyurethane and polyvinylidene fluoride nanoporous membranes embedded with silica aerogel were prepared by electrospinning technique. Thermal properties and air permeability were evaluated and compared. It was concluded that nanofibers embedded with aerogel are good for thermal insulation in cold weather conditions. Thermal insulation battings incorporating nanofibers could possibly decrease the weight and bulk of current thermal protective clothing.

Statistical analysis software, Matlab_R2017a were used to conduct all the statistical results in this study. The findings are significant and can be used for further study in the areas of aerogel embedded high-performance fibrous materials for thermal insulation in building, industrial and protective textile fields.

Keywords: fibrous structure; silica aerogel; thermal measurement; compression recovery; nanofiber

Abstrakt

Cílem této práce bylo vyvinout vysoce výkonný aerogel spojený s vlákennými materiály pro tepelnou izolaci a prověřit jeho výkon. Vrstvená nanovlákenná síťovina /aerogel /netkané materiály byly připraveny použitím laminovací metody s práškem s nízkým bodem tání jako tepelně spojujícím materiálem. Byl zkoumán účinek aerogelu a tepelného lepidla na tepelnou izolaci a propustnost vzduchu. V úvahu byl vzat sériový model pro tepelný odpor, byly porovnány a analyzovány teoretické předpoklady a naměřené výsledky. Bylo navrženo, že by měly být vzaty v úvahu nové techniky kombinující aerogel s vysoce porézními textiliemi s menším využitím pojivových materiálů.

Nový aerogel obalený vlákenným materiálem bez použití jakéhokoliv pojivového materiálu, který spojuje částice aerogelu, byl vyvinut pomocí techniky laserového gravírování a laminovací metody. Pro měření tepelného výkonu byly použity termální kamera, zařízení Alambeta a KES-FT-II Thermolab. Byla provedena tlaková zkouška ke zkoumání kompresního zotavení, které určuje udržitelnost tepelné izolace. Dále bylo použito laboratorně vyráběné dynamické zařízení pro přenos tepla pro zjištění konvektivního tepelného chování těchto vícevrstvých materiálů, jakož i netkaných textilií obalených aerogelem, při různých rychlostech proudění vzduchu a zahřívání. Byly porovnány teplotní křivky v reálném čase z měření předehřátých podmínek. Byly vypočteny a posouzeny hodnoty teplotních rozdílů a konvektivního součinitele přestupu tepla za podmínek kontinuálního ohřevu. Zjištění by mohla přispět k novému vývoji pružných vysoce výkonných textilních materiálů se zabudovaným aerogelem pro průmyslové i oděvní aplikace.

Pružné polyuretanové a polyvinylidenfluoridové nanoporézní membrány kombinované s aerogelem na bázi oxidu křemičitého byly připraveny elektrostatickým zvlákňováním. Tepelné vlastnosti a propustnost vzduchu byly hodnoceny a porovnávány. Byl učiněn závěr, že nanovlákna s aerogelem jsou vhodná pro tepelnou izolaci za chladného počasí. Tepelně izolační nosiče obsahující nanovlákna by mohly případně snížit hmotnost a objem tepelně ochranného oblečení.

Pro provedení a vyhodnocování všech statistických výsledků byl v této práci použit statistický a analytický software Matlab_R2017a. Dosažené výsledky jsou významné a mohou být použity pro další studium v oblasti tepelných vlastností vysoko pevnostních vlákenných materiálů s aerogelem, které mohou být využity například pro tepelnou izolaci budov či ochranné oděvy.

Klíčová slova: vlákenná struktura; aerogel; měření teploty; kompresní zotavení; nanovlákna

Summary

1 Introduction

Nonwoven fabrics possess plenty of functional properties such as high bulkiness and resilience, great compressional resistance, good filling properties and excellent thermal-insulating properties.[1] As conventional thermal insulators used in technical applications, their impact on thermal insulation performance is determined by the physical and structural parameters of the fibrous structures. Especially, their thermal insulation ability strongly depends on the fabric thickness. Generally, the thermal insulating properties increase with the increasing of thickness. However, when this thickness is limited to a few milli-meters, the insulating properties are quite limited.

Silica aerogel can be defined as a coherent, rigid three-dimensional network of contiguous particles of colloidal silica, which can be prepared by the polymerization of silicic acid or by the aggregation of particles of colloidal silica.[2-3] Due to its extraordinary small pore size and high porosity, silica aerogel exhibits superior thermal insulation performance with extremely low thermal conductivity (0.015 W/ m · K), low bulk density (0.1 g/cm^3) and high specific surface area (1000 m^2/g).[4-6] Nowadays, silica aerogel has well been acknowledged as one of the most attracting thermal insulating materials. However, since silica aerogels have poor mechanical stability, such as low strength and high brittleness, they are usually incorporated with lightweight textile structure for applications dealing with heat transfer problems for protective textiles, building and industrial facilities.

A lot of experimental studies confirmed that the present aerogel in fibrous structures would significantly improve the thermal performance of the fibrous materials, however, the application of aerogel granules has so far been limited in a few methods such as coating, padding and impregnation. In the obtained aerogel-embedded materials, aerogel granules are exposed or filled into the void space of the fibrous structure, the porous space between fibers is partly filled by additive binding agent, the thermal performance of the final product is thus reduced since the porous space which is essential to entrap air pockets is decreased. Meanwhile, the probable infiltration of the binding materials into the pores of the aerogel definitely eliminates the attractive properties of the aerogels.[7] Furthermore, the prepared materials may lack compression resilience, causing reduced recovery after exposure to external forces, which may influence the final use and the sustainability of thermal-insulating function. However, these problems were not considered in designing aerogel based fibrous materials.

Moreover, existing works were mainly focused on pure fibrous materials in absolutely flat state and with a simple airflow. There appears a gap in study on the convective heat transfer through multi-component fibrous structure system or under more complicated condition. These materials could be used for building, industrial facilities and protective textiles, which are not always in absolutely flat state. However, there is sparse information available regarding natural and forced convection through aerogel-based nonwoven. Although numerical simulation has been applied to evaluate the heat flux, temperature distributions, and convective heat transfer coefficients of aerogel-embedded fibrous materials, thermal performance of aerogel-based nonwoven under convection conditions is still not well understood.[8-9]

2 Purpose and the aim of the thesis

The research aims to develop silica aerogel-embedded fibrous materials for thermal insulation application and evaluate their thermal and non-thermal properties via different evaluation techniques. The purpose of this study is to understand the thermal performance of aerogel-embedded fibrous materials. The major objectives of this research are as follows:

2.1 Studies on the effect of silica aerogel and binding material on the transport properties of composite textiles

Researchers have stated that the application of aerogel in textile structure may cause adverse effect on thermal insulation enhancement since the porosity of textile fabric is reduced by the adhesive, but this was not experimentally studied. In this work, layered nanofiber web/silica aerogel/nonwoven materials with varying aerogel content were prepared via laminating method by using low-melting temperature powder as binding material. The effect of aerogel and thermal adhesive on transport properties of these composite textiles were investigated. Especially, the influence on thermal insulation performance were analyzed and discussed. Moreover, a series model was considered for thermal resistance of the layered systems, the theoretical results were compared with measured data.

2.2 Development of aerogel-encapsulated fibrous structures by using laser engraving technique

From previous work, it was concluded that novel techniques to combine silica aerogel with highly porous textiles with less use of binding materials should be considered. Thus, a new approach to apply silica aerogel into fibrous structure without using any binding material to bond aerogel particles was proposed in this work. To take benefit of air trapping potential in porous materials, highly porous nonwoven fabrics as well as sponge foam were selected as support layers to produce air pockets by laser engraving, aerogel granules could be applied into these pockets, together with laminating a thin fabric sheet to the support layer. Since both surfaces of the support layer were covered by a flexible fabric sheet to achieve a closed fibrous system and the adhesion of aerogel with this support structure was not involved, the resultant multi-layered materials could have light weight, excellent thermal insulation ability and good flexibility simultaneously.

2.3 Performance evaluation of novel multilayered fibrous materials in terms of thermal properties

This work attempts to explore the potential of using laser engraving to develop aerogel-encapsulated fibrous materials for cold condition use. Thus, thermal insulation function of these novel developed materials is the main point we have to concern. Thermal performance was measured by using different evaluation techniques. Alambeta device and KES-FT-II Thermolabo were employed to measure thermal conductivity, thermal resistance and coefficient of heat retention ability, the results from these two evaluation devices were compared and analyzed. Thermo Camera was used to measure infrared thermography. In addition, a laboratory-made dynamic heat transfer device was used to figure out convective thermal behavior of these multi-layered materials under different airflow velocities and heating conditions. The real-time temperature values of different materials were compared.

The temperature difference and convective heat transfer coefficient under continuous heating condition were calculated and investigated.

2.4 Study on compression performance and air permeability of novel multilayered fibrous materials

In practice, fibrous insulators are usually subjected to external forces such as compression stress. In this case, the materials readily undergo structural deformations due to their high degree of flexibility and compressibility, resulting in unrecoverable changes of pore characteristics and fabric structural parameters to some degree. Thus, compression performance plays an important role in determining thermal properties of fibrous materials. Meanwhile, air permeability is one of the most important performance characteristics of fibrous porous materials, and thermal behavior of fibrous materials under convection is related to air permeability. Thus, compression performance of the novel multilayered fibrous materials, such as compression resistance, compression resilience and thickness loss after compression test, were examined as well as air permeability.

2.5 Electro-spun nanofibrous membranes with aerogel granules and their performance evaluation

Electrospun fibrous materials with interconnected pores have the potential for use as thermal insulation materials. In this research, electrospinning process was used to produce polyurethane and polyvinylidenefluoride nanoporous membranes embedded with silica aerogel. Presence of aerogel granules were confirmed through microscopic examination. The transport behavior of these samples was evaluated and results were analyzed. Thermal properties such as thermal conductivity and thermal resistance were tested and compared. Thermal stability was investigated using thermogravimetric analysis and differential scanning calorimetry.

2.6 Comparative analysis of high-performance fibrous materials using different evaluation techniques

Several non-conventional techniques, such as KES-FT-II Thermolabo, thermal camera, and a laboratory-made new instrument, were adopted to evaluate the transmission behavior of aerogel-embedded fibrous materials. The results were evaluated statistically, the precision of evaluation techniques was analyzed and compared.

3 Overview of the current state of the problem

Aerogel-embedded fibrous materials are mainly made of fibers, aerogel and air. The effective thermal conductivity of these materials essentially depends upon the volume fraction of fiber, aerogel and air inside the composites.[10-11]

Two general strategies were usually used to prepare silica aerogel-embedded fibrous materials. The first technique involves immersing the nonwoven fabric into the sol-gel solution or impregnating a fiber network by such a mixture and followed by supercritical drying to produce silica aerogel-fiber composites in the form of blankets.[12-15] However, the fine dust particles of silica aerogels could result in an unpleasant feeling when using silica aerogel-based products. The second one is the incorporation of existing aerogel beads into the nonwoven fibrous web before bonding of the fibers or by using proper binding materials to prepare silica aerogel-polyester nonwoven materials. This method is widely used due to the lower production costs of aerogel granules, cost-effective manufacture and great flexibility of the processes and products.[16]

Silica aerogel nanoparticles have been incorporated in wool-Aramid blended fabrics by coating, results showed that the coating of aerogel nanoparticle could increase thermal resistance by up to 68.64%.[17] A study of coating silica aerogel on cotton woven fabrics indicated that the thermal properties of coated high-density cotton fabric were strongly influenced by finishing agent concentration.[18] The fibrous structure density and the aerogel present in the polyester/polyethylene fibrous nonwovens with silica aerogel impregnation have significant effect on thermal properties of the overall structures.[19] Meanwhile, thermal insulation of aerogel-embedded nonwoven was observed to strongly dependent on the weight and compressional properties of the fabric.[8] The modeling and simulation of heat transfer for aerogel-embedded nonwoven fabric has confirmed the improvement of thermal behavior when treated with aerogel.[9] University of Leeds developed Hydrospace™ fabric from a carded and cross-laid web. This fabric enables the formation of moulded voids within the cross-section of hydroentangled fabrics and simultaneous filling of these voids with loose aerogel particles composed of amorphous silica.[20] The size, shape and frequency of the filled voids can be varied depending on the dimensions of the encapsulated materials. Electrospun nanofiber supported aerogel composites were successfully synthesized via electrospinning and sol-gel processing.[21] Yin song Si[22] have prepared silica nanofibrous membranes via an electrospinning technique with a sol-gel solution containing NaCl. It opens a controllable way to improve and engineer the mechanical properties of the aerogel composites with low thermal conductivity via combining the aerogels by using electrospun nanofibers.

4 Methods used, studied materials

4.1 Materials

Details of the samples are presented in Table 1. Six types of layered nanofibrous web/silica aerogel/ nonwoven samples with different aerogel content (sample A0, A1, A2, A3, A4 and A5) were prepared via laminating technique by using low melting powder as thermal binding material. Twelve multi-layered samples with novel structures were developed by laser engraving and laminating technique. These samples are prepared on the basis of three highly porous Struto nonwoven fabrics and one polyurethane foam which were selected as middle layers to produce air pockets by laser engraving. Sample P1, P2 and P3 correspond to multilayered materials with different middle layer structures, respectively regular Struto nonwoven P, Struto nonwoven P with air pockets and aerogel-encapsulated Struto nonwoven P. Sample Q1, Q2, Q3 and sample X1, X2, X3 follow the same configurations. Sample Y1, Y2 and Y3 are multi-layered materials with the three different structures of polyurethane foam as middle layers. Sample B1, B2 and B3 are 50:50 ratio compositions of polyester/polyethylene nonwoven fabrics treated with aerogel. The aerogel particles were added during thermal bonding of the nonwoven web.

Table 1. Description of samples

Sample No.	Aerogel content g/m^2	Fabric thickness mm	95% confidence interval	Areal density g/m^2	95% confidence interval	Fabric density kg/m^3	95% confidence interval
A0	0	1.25	1.25±0.01	62.01	62.01±2.12	49.61	49.61±1.57
A1	1.25	1.30	1.30±0.06	63.75	63.75±1.97	49.41	49.41±1.52
A2	2.50	1.37	1.37±0.05	67.28	67.28±2.44	49.11	47.72±1.73
A3	3.75	1.42	1.42±0.09	69.97	69.97±1.85	49.27	49.27±1.30
A4	5.00	1.48	1.48±0.11	71.77	71.77±1.46	48.49	48.49±0.97
A5	6.25	1.53	1.53±0.04	73.21	73.21±1.23	47.85	47.85±0.83
B1	1.38	3.50	3.5±0.02	278.6	278.6±1.02	79.6	79.6±0.51
B2	9.31	6.60	6.6±0.07	440.2	440.2±1.05	66.7	66.7±0.32
B3	13.48	6.20	6.2±0.01	498.5	498.5±1.10	80.4	80.4±0.62
P1	0	12.80	12.80±0.13	420.32	420.32±7.30	32.83	32.83±0.57
P2	0	12.92	12.92±0.22	386.58	386.58±9.40	29.92	29.92±0.73
P3	26.76	12.88	12.88±0.19	415.25	415.25±10.4	32.24	32.24±0.81
Q1	0	9.88	9.88±0.09	363.41	363.41±9.50	36.78	36.78±0.96
Q2	0	10.11	10.11±0.11	344.75	344.75±8.90	34.10	34.10±0.79
Q3	38.42	10.18	10.18±0.17	381.36	381.36±7.72	37.42	37.42±0.78
X1	0	12.50	12.50±0.23	279.57	279.57±5.79	22.37	22.37±0.46
X2	0	12.64	12.64±0.17	262.38	262.38±7.48	20.76	20.76±0.59
X3	29.33	12.64	12.64±0.15	293.65	293.65±7.72	23.23	23.23±0.61
Y1	0	7.09	7.09±0.03	287.81	287.81±3.64	40.59	40.59±0.52
Y2	0	7.15	7.15±0.05	272.15	272.15±3.28	38.06	38.06±0.46
Y3	14.98	7.13	7.13±0.05	292.08	292.08±4.95	40.96	40.96±0.69

4.2 Sample preparation

4.2.1 Fabrication of layered nanofibrous web/silica aerogel/ nonwoven materials

Layered nanofibrous web/silica aerogel/ nonwoven materials with varying aerogel content were prepared by laminating technique, using low melting powder as thermal binding material. The low melting powder used here was 10 g/m^2, which was determined by previous studies. The layered system was subsequently held together on a heated plate at 110℃ at a given pressure, and the layered fabric was obtained as it cooled down. A layered fabric without aerogel granules was also prepared as control sample.

4.2.2 Fabrication of silica aerogel-encapsulated fibrous materials

Three types of Struto nonwoven fabrics and a polyurethane (PU) sponge foam were selected as support layers to prepare novel multilayered fibrous materials. Struto nonwovens and PU foam were treated by a commercial pulsed CO_2 laser system GFK Marcatex FLEXI-150 to create perforations as the laser beam vaporizes the surface.[23] During the laser engraving process, the laser head moves back and forth to engrave a series of dots in one line at a time, the dot pattern will form the designed image.[24] The dot pattern file designed in grey scale by

Photoshop CS4 graphic software and typical image of laser-engraved sample are shown in Figure 1.

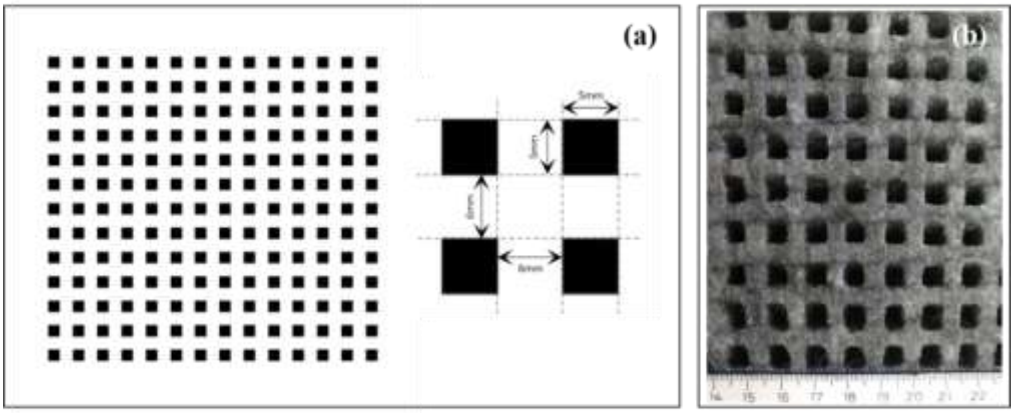


Figure 1. Dot pattern used for laser engraving: (a) - Designed dot pattern, (b) - Typical image of laser engraved sample.

Thermo-bonded nonwoven fabric sheet with thickness 0.16 mm was laminated onto support layers. Aerogel granules were subsequently injected into these laser-engraved holes. After that, another fabric sheet was combined to form a closed fibrous composite system. The fabrication process is illustrated in Figure 2. Different cases of middle layers including untreated support materials and support layers with air pockets were prepared as control samples.

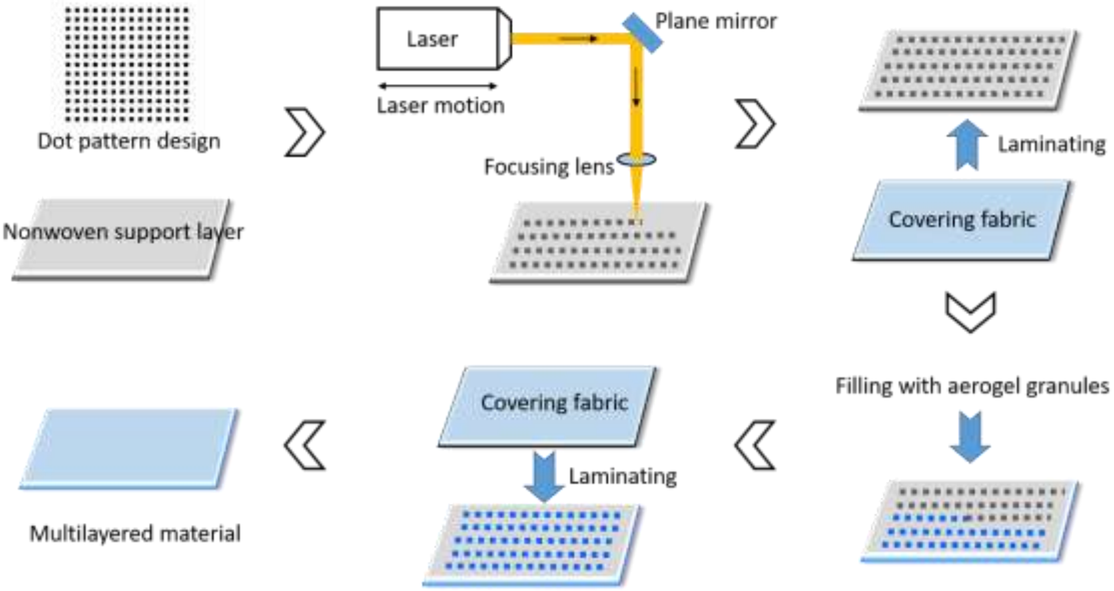


Figure 2. Fabrication process of aerogel-encapsulated fibrous materials

4.2.3 Electrospinning of nanofibrous membranes embedded with aerogel

In order to investigate the mechanisms of heat transfer through nanofibrous materials, flexible electrospun nanofibrous membranes embedded with silica aerogel was produced via electrospinning process. The electrospun PUR and PVDF nanofibrous microstructures were fabricated and then used to reinforce the SiO_2 aerogel. The PUR and PVDF were separately dissolved in DMF at a concentration of 18 wt. %. The solutions were stirred in a magnetic stirrer for 2 hours at room temperature. Subsequently, silica aerogel particles were added into the solution. These mixtures were stirred again for 3 hours at room temperature prior to electrospinning. Electrospinning was carried out using Nanospider technology, which is a modified electrospinning method based on the possibility of creating Taylor Cones and the subsequent flow of material not only from the tip of a capillary, but also from a thin film of a polymer solution.[25-26]

4.3 Measurement methods

4.3.1 Evaluation of thermal properties

Alambeta instrument was used to measure thermal conductivity and thermal resistance, according to EN 31092 standard. The measuring head of the Alambeta contains a copper block which is electrically heated to approximately 32℃ to simulate human skin temperature, which is maintained by a thermometer connected to the regulator. The lower part of the heated block is equipped with a direct heat flow sensor which measures the thermal drop between the surfaces of a very thin, non-metallic plate using a multiple differential micro-thermocouple.

Examination of heat retention ability and thermal conductivity is performed with KES-FT-II Thermolabo device. Thermal conductivity is measured by steady heat flow method using the BT box and water box. The heat source plate-BT box is set at a constant temperature 33°C to simulate a human skin temperature. The water box is kept at 23°C, the specimen is placed at the top of water box. The heat source plate is shifted onto the sample, thus the direction of heat flow agrees with the direction of acceleration of gravity. The temperature difference between BT box and water box is 10°C. The heat lost from the hot plate is then indicated directly on the display. According to the thickness of the specimen, heat loss, temperature difference between BT box and water box, the thermal conductivity can be calculated. Coefficient of heat retention ability is determined with the help of the wind column. The BT plate is heated to 33˚C and the heat loss is measured. A constant air flow rate of 0.3 ms^{-1} is present in wind column, with a constant air temperature of 23 ˚C ± 2 ˚C. Loss of heat flow in both conditions, without fabric on BT plate and with fabric on BT plate, are measured. From the obtained values of the heat flow loss, the coefficient of heat retention ability can be calculated.

Figure 3. Image of BT box, T box and water box

4.3.2 Measurement of infrared thermography

The thermography measurement was conducted by using FLIR ThermaCAM TVS300 thermal camera as shown in Figure 4. Tests were carried out in a test chamber with an opening on its upper surface, the dimension of the chamber was 45 cm × 32 cm × 12 cm. A guard hot plate with constant temperature 33˚C was located in the chamber to provide uniform thermal radiation, the thermal camera was placed in the air space with a distance of 40 cm from the hot plate, facing to the opening of the chamber as well as the hot plate. When the specimen was placed on the hot plate, pictures were taken every 5 second up to when the heat transfer reaches steady state. The room temperature was kept at 23± 2°C. The specimen size used for measurements was 20 cm × 20 cm, three tests were carried out for each sample. The temperature of the surface was calculated using Avio Thermography Studio 2007 software in which each pixel of the picture was allocated to one temperature value.

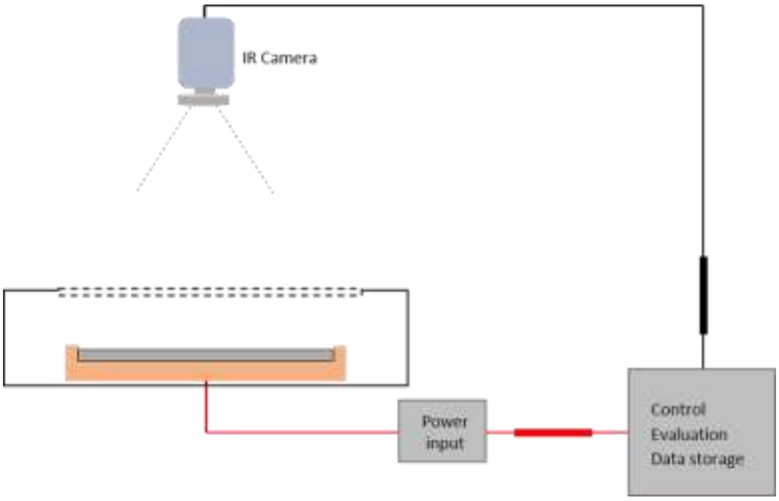


Figure 4. Setup of thermography measurement

4.3.3 Measurement of dynamic convective heat transfer behavior

A dynamic heat transfer device was made in our laboratory to evaluate thermal performance of multilayered samples and aerogel treated nonwovens under different airflow velocities. The schematic diagram of the laboratory made device is presented in Figure 5. The main part of the measuring section consists of a subsonic wind tunnel with a square cross section of 20cm×20cm, an electric heating rod with a diameter of 2cm and length 20cm placed perpendicular to the airflow direction in the wind tunnel, and a fan ventilator connected in the right corner of the lower duct. The heating rod, composed by a Ni-Cr 80-20 heating wire with a stainless-steel shell, was connected to a control unit and powered by a Sefram DC power supply. The input power was adjusted by control unit. For mounting the heating rod in the testing section, two holes were drilled on the duct wall, the heating rod was able to be inserted into the testing section. The fan ventilator, acting as a suction fan, inlets airflow velocities at room temperature. Different airflow velocities can be achieved by switching the fan speed.

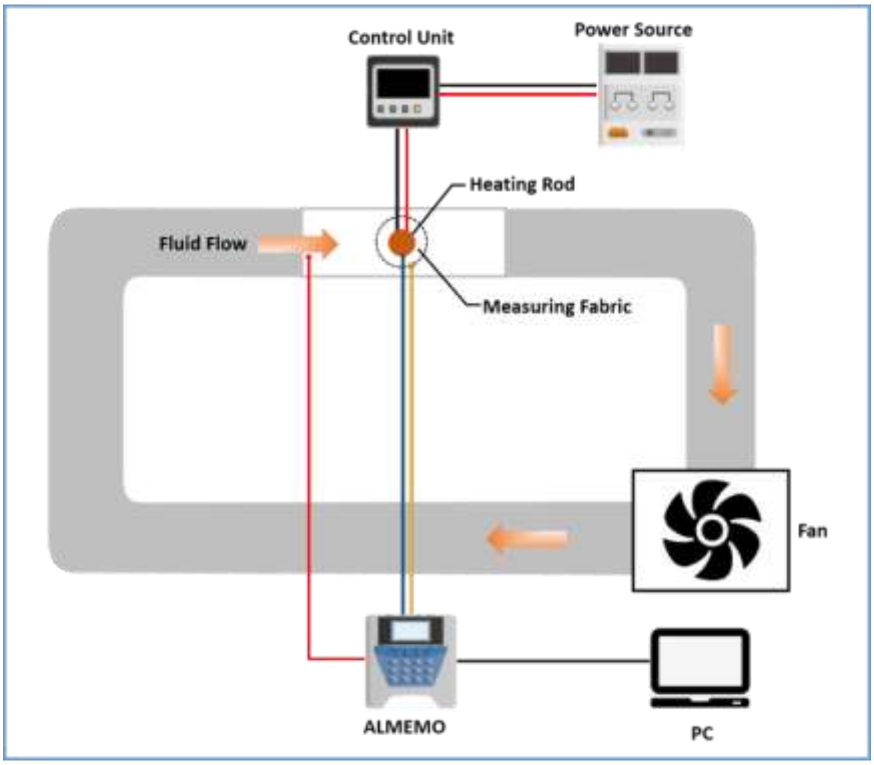


Figure 3. Schematic diagram of the measurement device

The experiments involve measurements of real-time temperature values of the heating rod as well as the fabric insulator, wind velocity, wind pressure and the temperature of airflow before entering the testing section. Temperatures of the heating rod and fabric were measured by using T-type copper constantan thermocouples. Two thermocouples were respectively attached on the heating rod surface and fabric surface with the help of high temperature RTV silicon. A thermoanemometrické sensor FVAD35TH4 was mounted in the upstream to monitor the velocity, pressure and temperature of the free stream. All these thermocouples and sensor were connected through ALMEMO 2590-2A - 2 datalogger to a PC device.

A fabric specimen with size of 20 cm ×8 cm was wrapped on the heating rod (as seen in Figure 6) by using insulating rubber tape to seal the lateral gap. Any gaps between the fabric and the heating rod were eliminated. The tests were carried out at different values of air velocity under two different heating conditions, preheated and continuous heating conditions, to investigate how the fabric help prevent heat loss from the heating rod. The preheated condition refers to preheat the heating rod to a specific temperature value 60°C and switch off the heating power to let the system cools down with a selected air velocity. The continuous heating condition involves non-stop heating during the whole measuring process. The voltage and current supplied from DC power for heating were 37 V, 0.33 A for each measurement. Five airflow velocity levels, 0, 1, 5, 10 and 15 m/s were used to study the effect of different airflow velocity on the heat loss rate of different fabrics by convection. The sensed data of temperature values, airflow pressure and velocity were collected at an interval of 3.2 second.

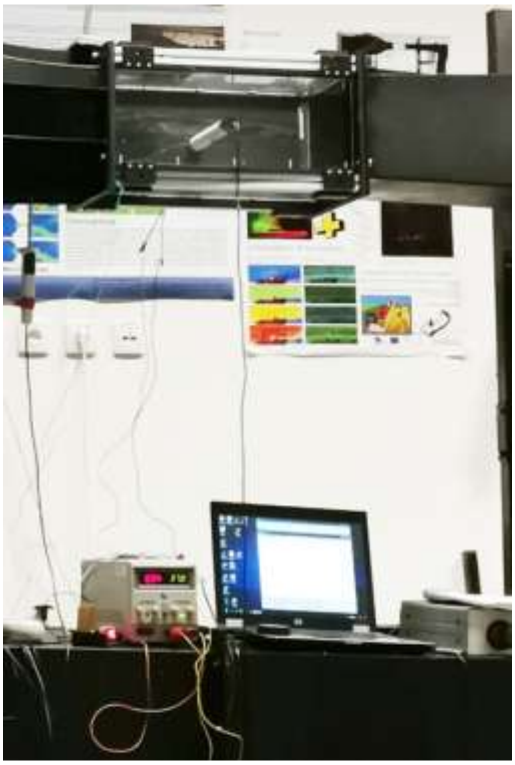


Figure 4. Image of the main testing section

4.3.4 Measurement of compression properties

ORIENTEC STA-1225 universal testing device was used for compression testing. The circular pressure pads with foot area 36.3 cm^2 and diameter 68 mm were used. The maximum pressure was set at 560 gf/cm^2. The sample size used in compression test was 20 cm × 20 cm, the loading speed was set at 2 mm/min. The obtained compression behavior of a fabric is described by the relationship between the applied force per unit area and the resulting fabric thickness, in the form of compression load-displacement curve. From compression hysteresis curves, compression resistance, compression resilience and thickness loss can be calculated.

4.3.5 Measurement of air permeability

Air permeability is described as the rate of air flow passing perpendicularly through a known area, under a prescribed air pressure differential between the two surfaces of a material. Tests were performed according to standard ISO 9237 using a FX-3300 air permeability tester. The fabric sample is fixed as an obstacle in a flow of air by the clamping holder. A pressure difference between both sides of the fabric sample develops as a consequence of hydraulic losses. The pressure difference is recorded by using of the manometer. The measured value is a speed of air in meter per second or a volume rate of the flow in liter per hour.

4.4 Statistical analysis

Statistical analysis software, Matlab_R2017a were used to conduct all the statistical results in this study. The findings are significant and can be used for further study in the areas of aerogel embedded high-performance fibrous materials for thermal insulation in building, industrial and protective textile fields.

5 Summary of the results achieved

5.1 Thermal properties of layered nanofibrous web/silica aerogel/ nonwoven

Thermal conductivity and thermal resistance of the layered materials are presented in Figure 7. The aerogel-embedded materials showed lower thermal conductivity and much higher thermal resistance, indicating that the aerogel content present in the fabrics plays a major role and gives better thermal insulation performance. Thermal conductivity and thermal resistance of the layered fabrics were directly proportional to the areal density of aerogel.

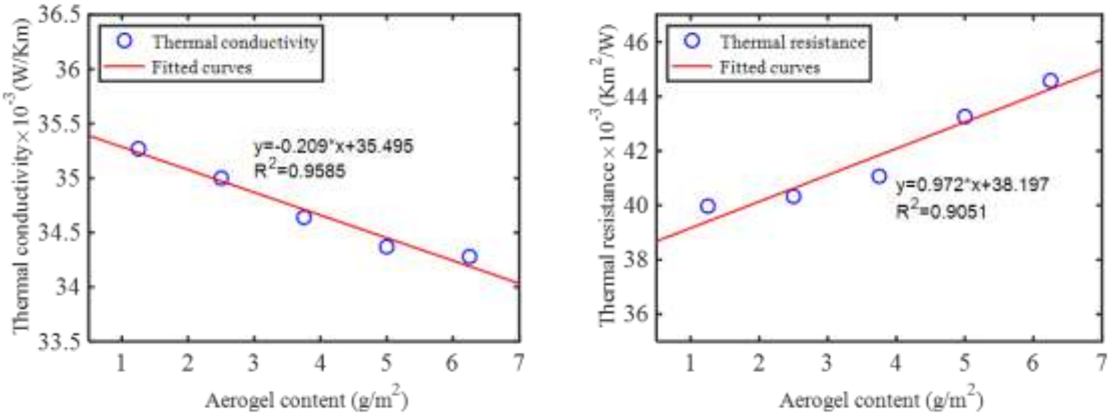


Figure 5. Thermal conductivity and thermal resistance of layered nanofibrous web/silica aerogel/ nonwoven

The thermal conductivity of layered nanofibrous web/silica aerogel/ nonwoven increases with the increase in fabric density as seen in Figure 8, this is attributed to the less porosity in higher density fabrics. Especially, the thermal resistance is directly proportional to fabric thickness, indicating fabric thickness is the primary factor contributing to the thermal insulating ability of a fibrous material.

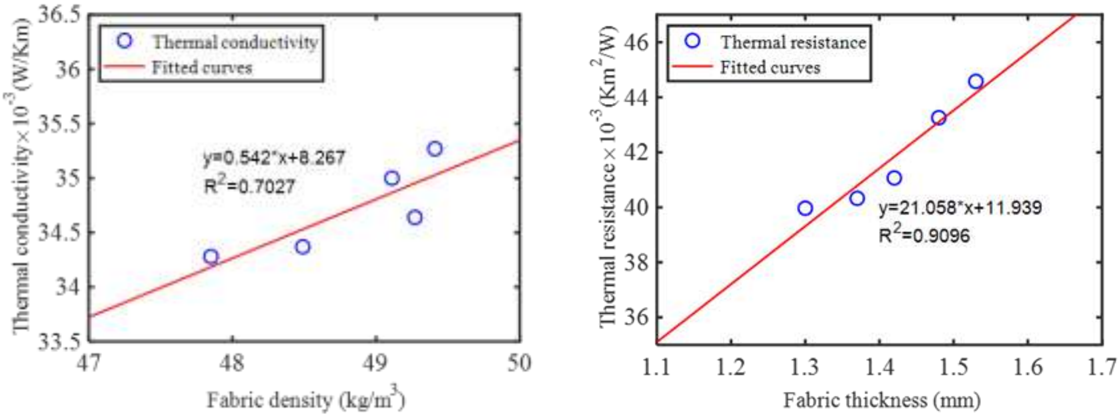


Figure 6. Effect of fabric density and thickness on thermal performance

For multilayered fabric systems, the layers are considered to set as a series of thermal resistance, according to electrical analogy with conduction heat transfer, the total thermal resistance is the sum of thermal resistance of each layer. However, the use of thermal binding powder in the layered structure will improve thermal conductivity, this will cause some reduction in total thermal resistance since the fabric thickness is not proportionally decreased. This reduction in thermal resistance, ΔR [$m^2 \cdot K/W$], can be obtained by

$$\Delta R = R_S + R_N - R_{A0} \quad \#(1)$$

where R_S is thermal resistance of the nonwoven fabric [$m^2 \cdot K/W$], R_N is thermal resistance of the nanofiber web [$m^2 \cdot K/W$], R_{A0} is thermal resistance of the layered fabric without aerogel [$m^2 \cdot K/W$].

In this study, the reduction in thermal resistance ΔR is 4.36×10^{-3} $m^2 \cdot K/W$, which accounts for 13.65% of the total thermal resistance. This indicated that the use of adhesive in textile structure has significant effect on the final thermal insulation performance, which is noteworthy for the fabrication of textile composites as thermal insulators.

The decrease of thermal resistance induced by adhesive strongly depends on the amount of thermal binding powder and its distribution. In this study, the amount of thermal binding powder in each layered fabric is totally identical, if the thermal binding powder is assumed to uniformly spread only in the middle layer consisted by aerogel granules and air, then the reduction in thermal resistance in each layered system can be approximately considered to be the same. Therefore, the total thermal resistance of a layered fabric with aerogel can be calculated by

$$R_t \approx R_s + R_M + R_N - \Delta R \#(2)$$

where R_M is thermal resistance of the middle layer [$m^2 \cdot K/W$].

The thermal performance of this middle layer is determined by aerogel granules and the air space in the structure. Assuming it is a homogeneous layer without aerogel loss from this layer, then the thermal conductivity of this layer λ [W/ m·K] can be obtained by using

$$\lambda = \lambda_0(1 - f_V) + \lambda_{aer} f_V \#(3)$$

where f_V, the volume fraction of aerogel granules, is

$$f_V = \frac{\rho_s}{\rho_V \cdot h} \#(4)$$

where λ_0 is the thermal conductivity of stagnant air (0.024 W/K·m at 25°C), λ_{aer} is the thermal conductivity of aerogel granules [W/K· m], ρ_s is the areal density of aerogel in layered fabric [g/m^2], ρ_V is the bulk density of aerogel [kg/m^3] and h is the thickness of the middle layer [mm].

Thus, the total thermal resistance of a layered fabric R_t can be expressed as

$$R_t = R_S + R_N + \frac{\rho_V \cdot h^2}{\lambda_0 \cdot (\rho_V \cdot h - \rho_S) + \lambda_{aer} \cdot \rho_S} - \Delta R \ \#(5)$$

Thermal resistance values obtained from experiment and series model are shown in Figure 9. Results showed a good agreement between the values from experiment and those from series model. Average error was about 2.07 %. The error showed a decreasing trend with the increasing of aerogel, this could be explained by the assumption that the thin middle layer between nanofiber web and nonwoven substrate is a uniformly continuous layer, if the aerogel particles are too less to form a continuous layer, the thermal resistance will be underestimated by theoretical model since the thickness of the middle layer is decreased.

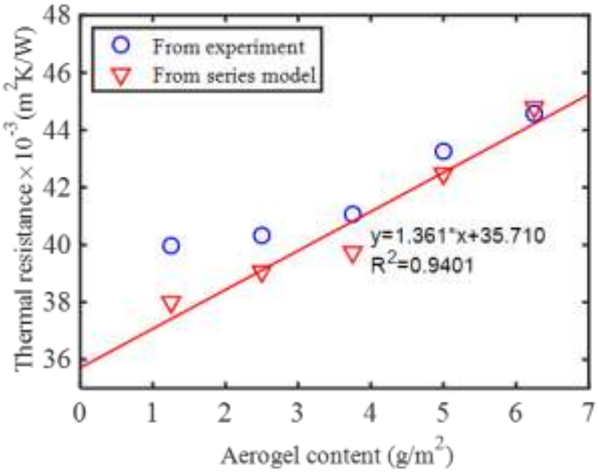


Figure 7. Theoretical and experimental values of thermal resistance

5.2 Thermal properties of novel multilayered fibrous materials

Thermal conductivity and thermal resistance of novel multilayered fibrous materials from Alambeta device are shown in Figure 4. 10. Apparently, in each sample group, air pockets give slightly decrease to thermal conductivity value and small rise to thermal resistance as compared to regular materials. Aerogel-encapsulated structure give decrease to thermal conductivity while the thermal resistance significantly increases. For the aerogel-encapsulated materials, the large open voids are filled by nanoporous structural aerogel, this will further prevent the convection current transfer through the structure. Furthermore, since the pore size in the aerogel granules is lower than the mean free path of air molecules, according to Knudsen effect these extremely small pore size will cause a very low gaseous thermal conductivity, resulting in less heat transfer through the structure and significant improvement of thermal insulation performance.

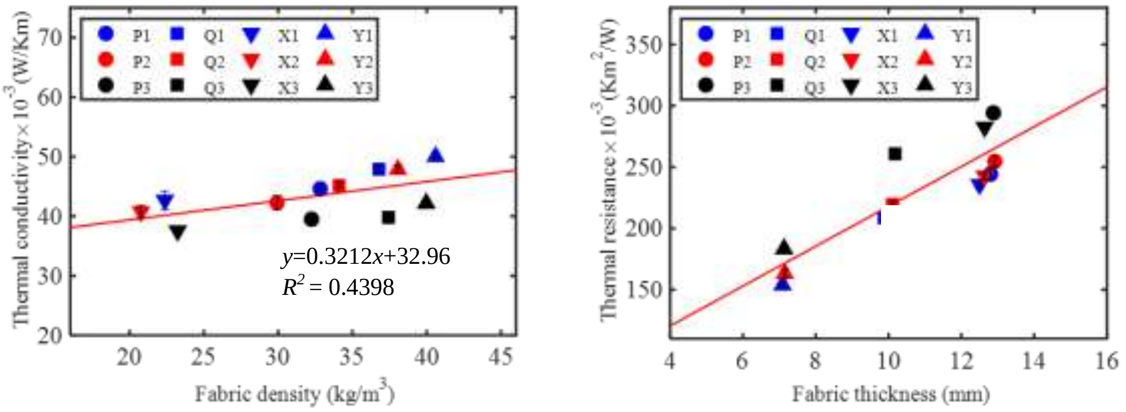


Figure 8. Thermal conductivity and thermal resistance of multilayered fibrous materials from Alambeta

Analysis of variance was performed for each group using t- test method to determine whether there are any significant differences in thermal insulating properties. Results showed that for nonwoven based multilayered fibrous materials the variance of thermal conductivity values within groups are all significant ($p\text{-value} < 0.05$), the differences between air pockets and aerogel-encapsulated structure are found to be very significant ($p\text{-value} < 0.01$). For foam-based materials the thermal conductivity difference between regular sample and laser-engraved one is observed to be highly significant ($p\text{-value} < 0.00001$). With respect to thermal resistance, in each group all the values from different structures show very significant difference ($p\text{-value} < 0.01$). Variance analysis indicates that the aerogel-encapsulated structure has significant effect on thermal insulation enhancement while the air pockets can improve thermal insulating properties to some degree.

The heat retention coefficient and thermal conductivity from KES-FT-II Thermolabo are illustrated in Figure 11. It is apparent that the materials with air pockets had better ability to retain heat compared to regular samples. The highest heat retention ability was observed from materials with aerogel-encapsulated structure. It also can be observed that with the increase in fabric thickness, the heat retention ability of the multi-layered fabric increases in the same way as thermal resistance. Similar to thermal resistance, the coefficient of heat retention ability was linear related to the thickness of multilayered fibrous material. As the fabric density increases, the measured thermal conductivity increases as well. Aerogel content present in the fabrics gives lower thermal conductivity. This agrees well with the results from Alambeta device.

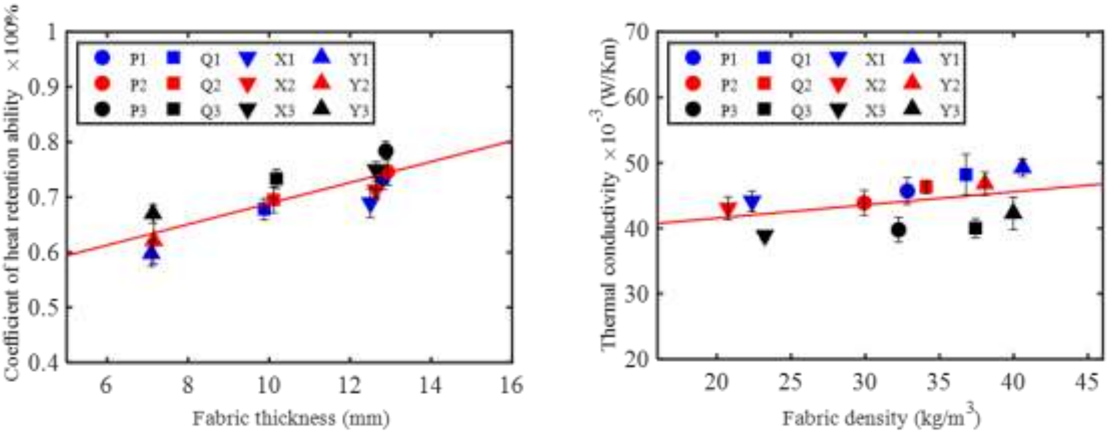


Figure 9. Heat retention coefficient and thermal conductivity of multilayered fibrous materials from KES-FT-II Thermolabo

Since thermal resistance and coefficient of heat retention are both related to fabric thickness, the correlation of thermal resistance and heat retention ability is illustrated in Figure 12. The two parameters correlate well with adjusted coefficients of determination 0.915. The correlation validates the accuracy of the measured results and proves that the two instruments are both suitable for measuring thermal properties of multi-layered fibrous materials. Results of thermal conductivity from both instruments were found to be well correlated with adjusted coefficients of determination 0.9353.

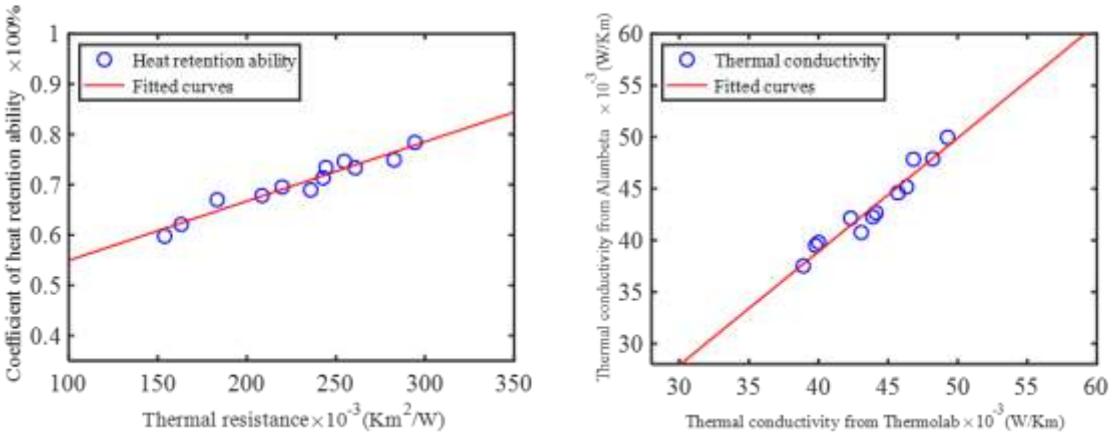


Figure 12. Correlation of thermal properties from KES-FT-II Thermolabo and Alambeta

Figure 13 illustrates the actual infrared thermography images under steady state. It is clear that for each group, temperatures for samples with air pockets are lower than that from regular samples, and the lowest values are observed from aerogel-encapsulated materials. Results also reveals that the Struto nonwoven based materials have better thermal performance in comparison with PU foam-based samples. Still, the aerogel particles and air pockets present in the porous PU foam could contribute to the improving of thermal performance. Generally, under infrared radiation the aerogel-encapsulated materials demonstrated lowest temperature values, with 1-1.5°C lower than regular multilayered samples when the temperature difference between hot plate and environment is 10 °C.

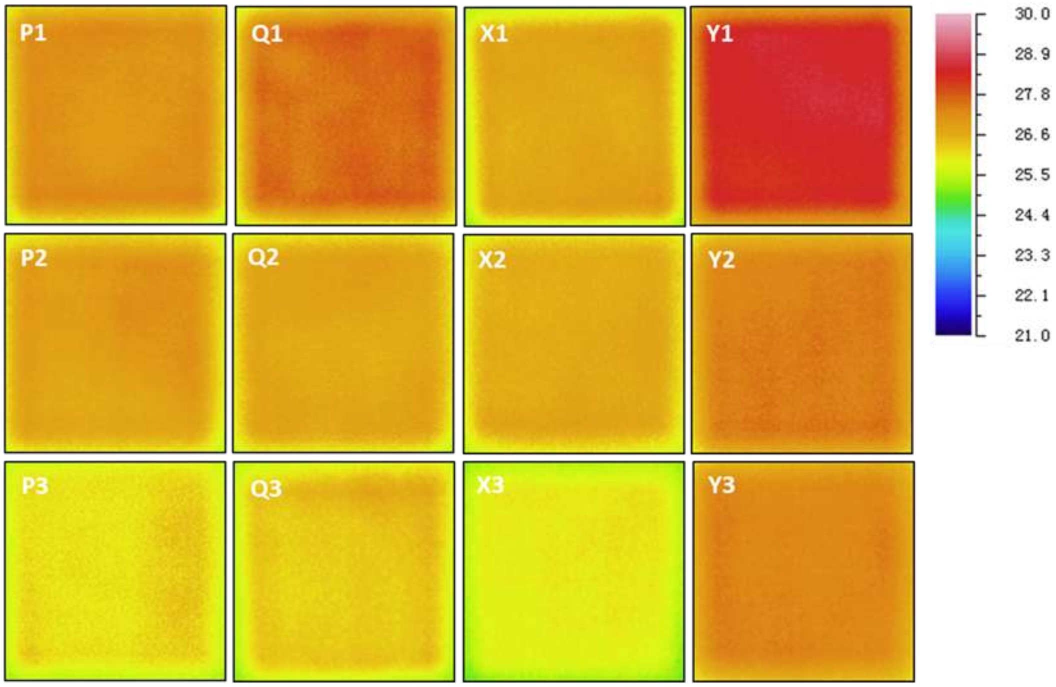
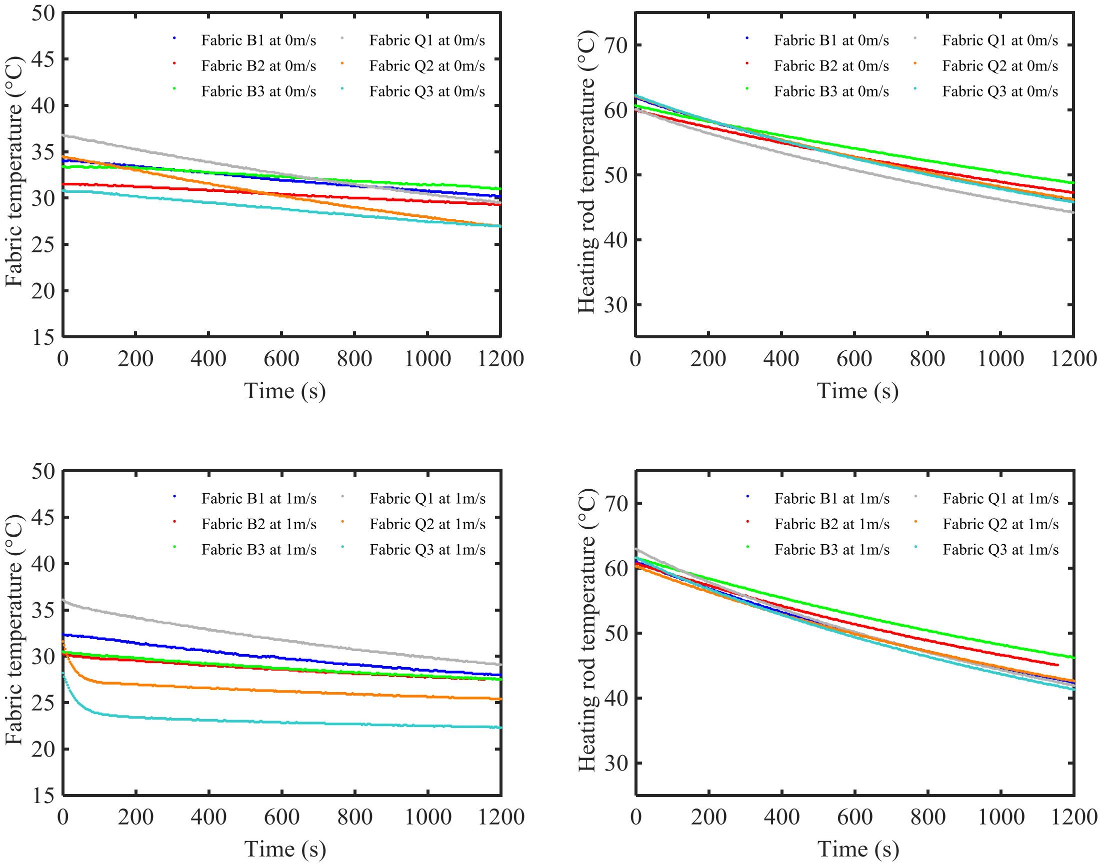


Figure 13. Infrared thermography images of multilayered fibrous materials

5.3 Thermal behavior of multilayered fibrous materials under convection

Figure 14 illustrates the real-time temperature curves from different fabrics. The fabric temperature depends on not only thermal characteristics of the fabric, but also external factors such as airflow temperature, airflow velocity, heating rod temperature and so on. Multilayered samples demonstrate rapid decreases in fabric temperature in the very beginning of the cooling curve while very small temperature drops are observed for aerogel-treated samples. This could be explained by that the multilayered samples exhibit lower density and their heat loss rate is more influenced by airflow, leading to obvious fabric temperature drop at airflow velocity over 1m/s.

With respect to the heating rod temperatures, it is clear that the heating rod temperature curves shift to the lower part with the increasing of airflow velocity, showing obvious temperature decrease trend. Generally, the heating rod with aerogel-treated nonwoven B2 and B3 give higher temperature in comparison with sample B1, and the temperature gradient is more significant at high airflow velocities. As for multilayered fibrous materials, since the samples having very similar characteristics of permeability and fabric density, the heating rod temperature curves under the same airflow velocity are quite close.



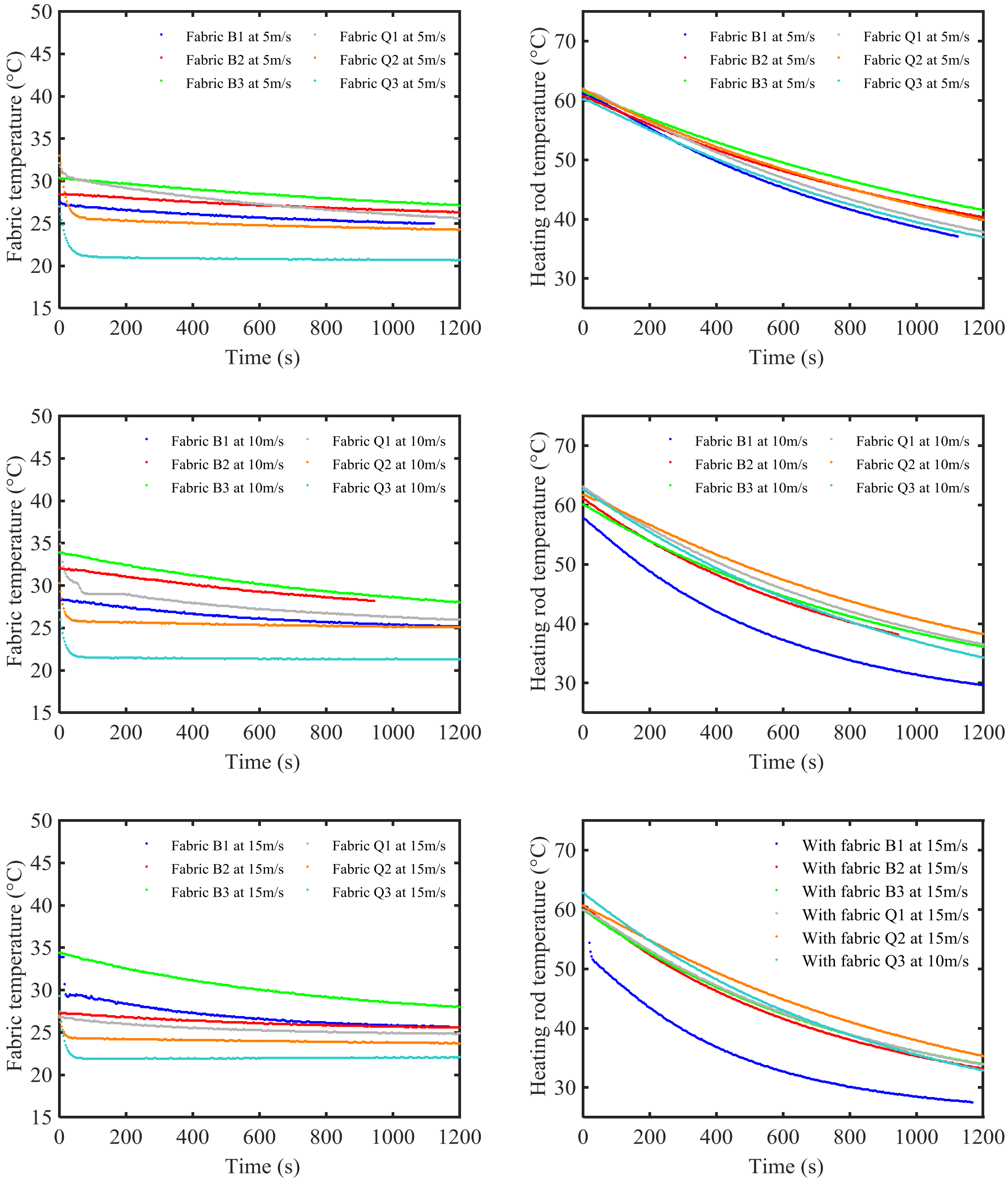


Figure 14. Comparison of real-time temperature curves under preheated condition

The real-time temperature curves of the heating rod and different fabrics under continuous heating are presented in Figure 15. In comparison with the heating rod, all the fabrics maintain relatively stable temperature values under continuous heating condition. Sample Q3 shows the lowest temperature values under continuous heating, which is benefited from the aerogel-encapsulated structure. Sample Q2 exhibits lower temperature due to the structure with laser-engraved air pockets. Among the aerogel-treated nonwoven fabrics with the same structures, samples with higher thickness and aerogel content, demonstrate better ability to prevent against heat loss from the heating rod. Especially at high airflow speed the heating rod is able to achieve quite high temperature values, meaning better thermal protection given by these fabrics.

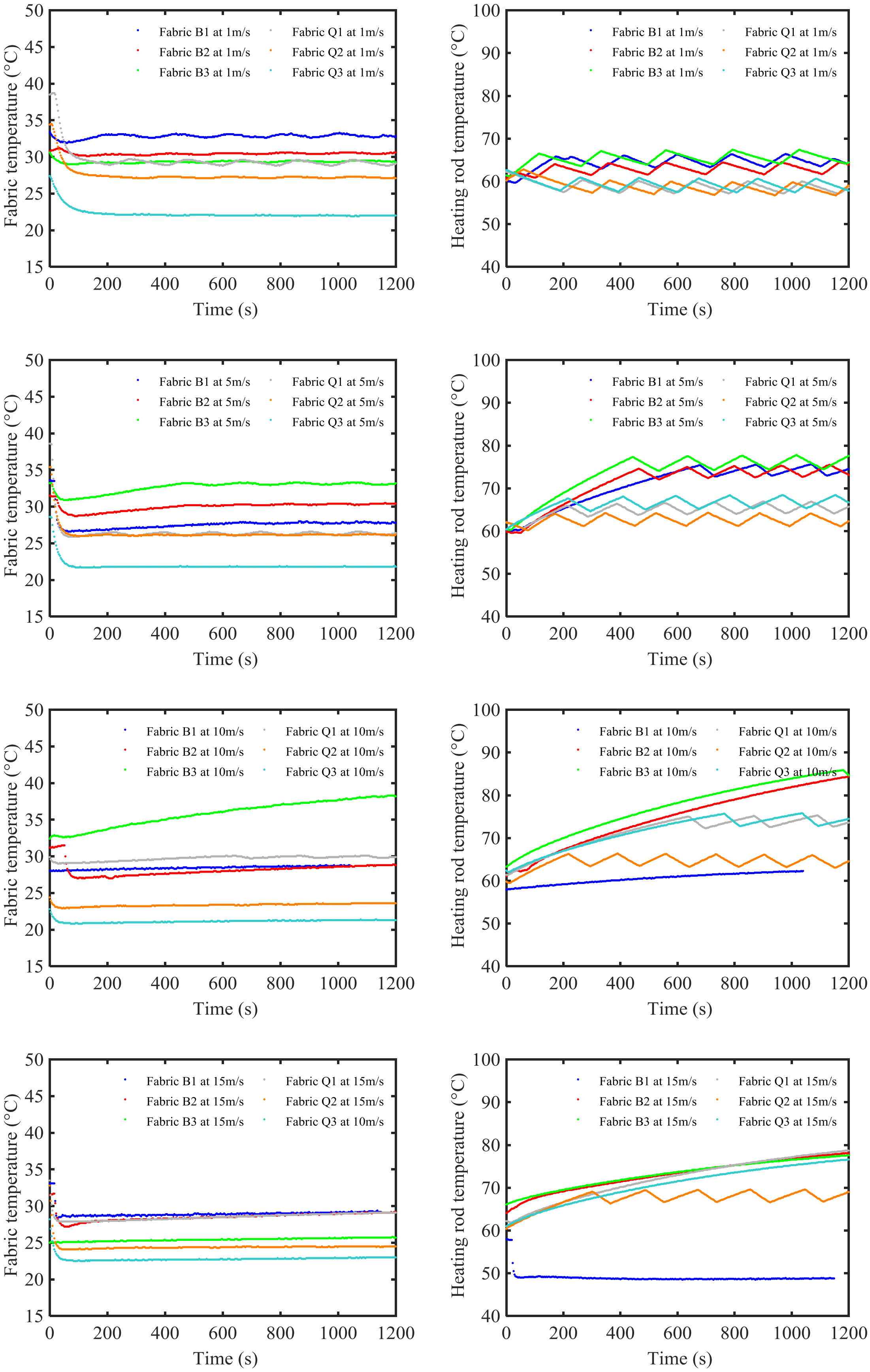


Figure 10. Comparison of real-time temperature curves under continuous heating

With respect to the heating rod temperatures, slight fluctuations are observed for the temperature curves at low airflow speed (1 m/s and 5 m/s). These fluctuations are well consistent with the current switch. The heating rod with multilayered sample generally demonstrates lower temperature value. This may be attributed to the air permeable characteristics of the multilayered materials which enables considerable heat loss from the heating rod. A gentle increasing trend in the heating rod temperature is observed for aerogel-treated nonwoven fabrics when the airflow speed is 10 m/s and 15m/s. Meanwhile, it is notable that the heating rod with aerogel-treated nonwoven B1, has rapid temperature drop from 58℃ to 48.8 ℃when the airflow with velocity of 15m/s is induced. This implies that aerogel content and fabric thickness are very important factors in protecting against heat loss at high airflow velocity.

Figure 16 illustrates the heating rod to air temperature difference under continuous heating. Apparently, the temperature difference of the heating rod without fibrous material tends to dramatically decrease with the increase in airflow velocity, while this trend is reversed when a fibrous material is used as insulator. For all the samples, the temperature gradient increases as the airflow velocity increases. Among different materials, aerogel-treated nonwovens with higher aerogel content, and multilayered materials with higher fabric density, demonstrated higher temperature gradient.

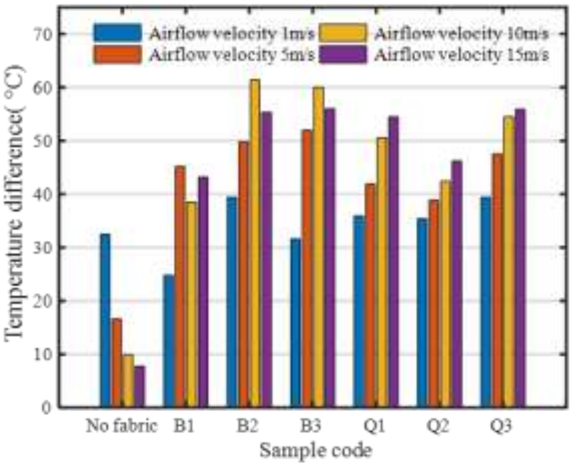


Figure 11. Heating rod to air temperature difference based on insulating materials

The Reynolds number is important in predicting flow patterns in different fluid flow situations for convective heat transfer problems. The heating rod to airflow temperature difference is plotted for different Reynolds number as seen in Figure 17. For the heating rod without fabric, temperature difference lies in the range of 32℃-8℃, showing obvious increase in heat transfer rate with the increasing of Reynolds number and considerable decrease in the temperature of heating rod as expected. The heating rod to air temperature difference increases with the Reynolds number when applying a fibrous insulator.

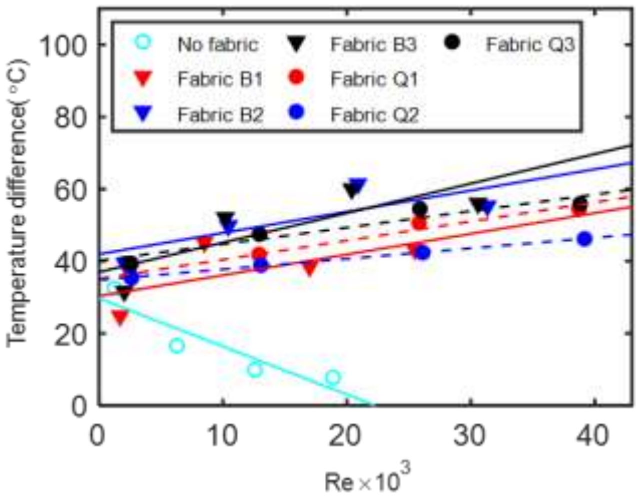


Figure 17. Heating rod to air temperature difference vs Reynolds number

Figure 18 shows the heat transfer coefficient of different samples. The coefficient value tends to increase with the increase in airflow velocity. Multilayered fibrous materials demonstrate higher heat transfer rate in comparison with aerogel treated nonwoven fabrics. Aerogel-treated nonwovens B2 is observed to have relatively stable heat transfer coefficients, this could be attributed to the present aerogel particles in the low-density fibrous structure.

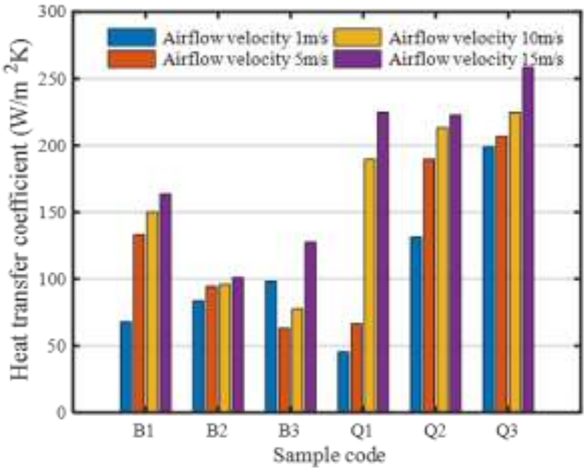


Figure 18. Heat transfer coefficients of different samples

The heat transfer coefficient increases with Reynolds number as seen in Figure 19. The data fall in distinct groups depending on the fabric density and permeability of the material. A flat upstream trend is observed from aerogel-treated nonwoven B2 and B3. For multilayered materials and aerogel-treated nonwoven B1, the coefficients significantly increase with the increase in airflow velocity and Reynolds number.

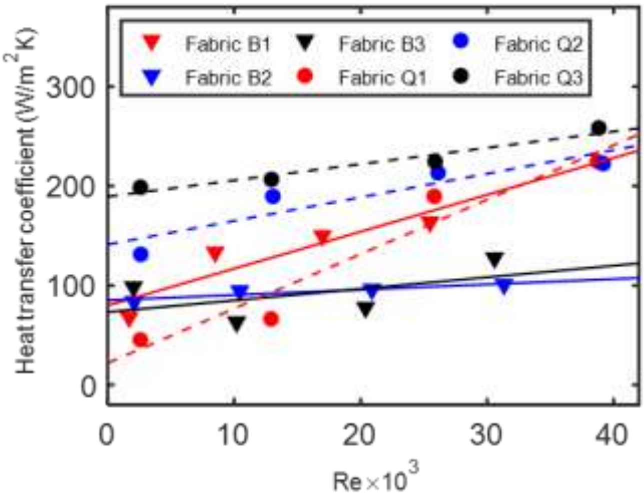


Figure 19. Average heat transfer coefficient vs Reynolds number

5.4 Thermal properties of PUR and PVDF electrospun nanofibrous layer embedded with silica aerogel

Thermal conductivity of PUR and PVDF nanofibrous layer embedded with silica aerogel is shown in Figure 20. The results showed that thermal conductivity of the nanofibrous layer decreased with increase in density. The superfine fibers in the web have better radiation absorption and extinction since their higher surface-area-to-volume ratio leads to decrease in the thermal conductivity. Decrease in the average nanofiber diameter leads to lower limit of conductivity. Moreover, smaller pore size between nanofibers decreases the mean free path for photon movement resulting in lower radiative energy transfer.

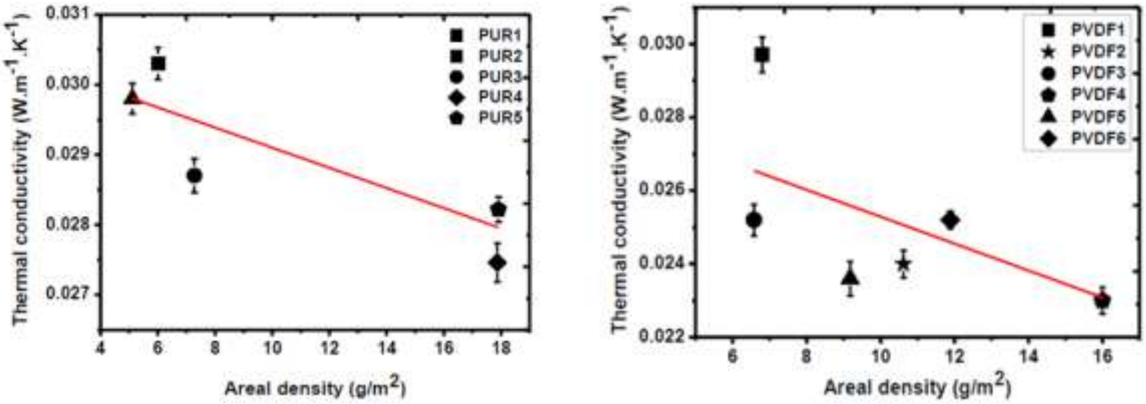


Figure 20. Thermal conductivity vs areal density of electrospun nanofibrous membranes embedded with silica aerogel

With respect to high porosity fibrous insulation materials, the combination of aerogel and nanofiber demonstrated excellent insulation per unit thickness properties. The thermal resistance of electrospun PVDF nanofibrous layer embedded with silica aerogel is higher than electrospun PUR nanofibrous layer embedded with silica aerogel.

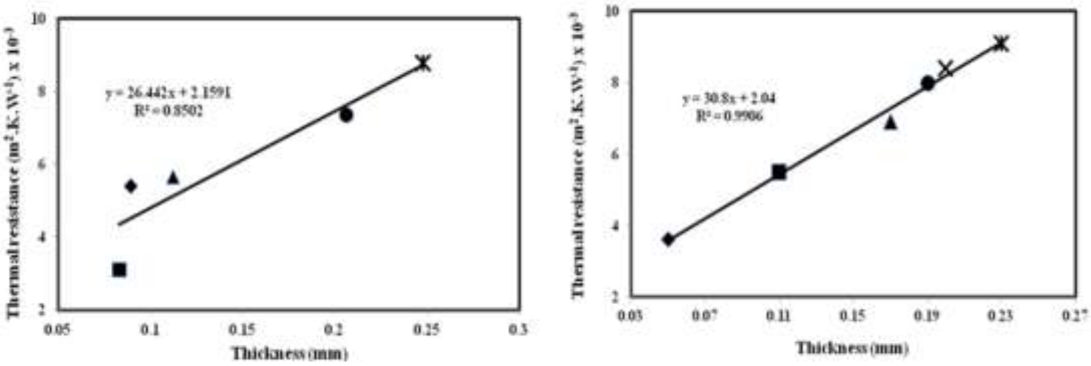


Figure 21. Thermal resistance vs fabric thickness of electrospun nanofibrous membranes embedded with silica aerogel

5.5 Thermal stability of nanofibrous membranes embedded with silica aerogel

Figure 22 shows the TGA curves of electrospun PUR and PVDF nanofibrous membranes with and without SiO_2 aerogel. The PUR nanofibrous membranes demonstrated considerable weight loss above 300℃, the electrospun PVDF layers showed significant weight loss in the temperature range of 400-45℃. The weight loss was associated with the degradation of the polymer chain structure, in agreement with previous literature.[27] The weight loss of the PUR nanofibrous membranes embedded with and without aerogel was between 90% to 95% at around 290℃and PVDF nanofibrous membranes embedded with and without aerogel were between 5% and 17% at around 430℃, respectively. Therefore, the PVDF nanofibrous membranes showed better stability than PUR nanofibrous membranes.

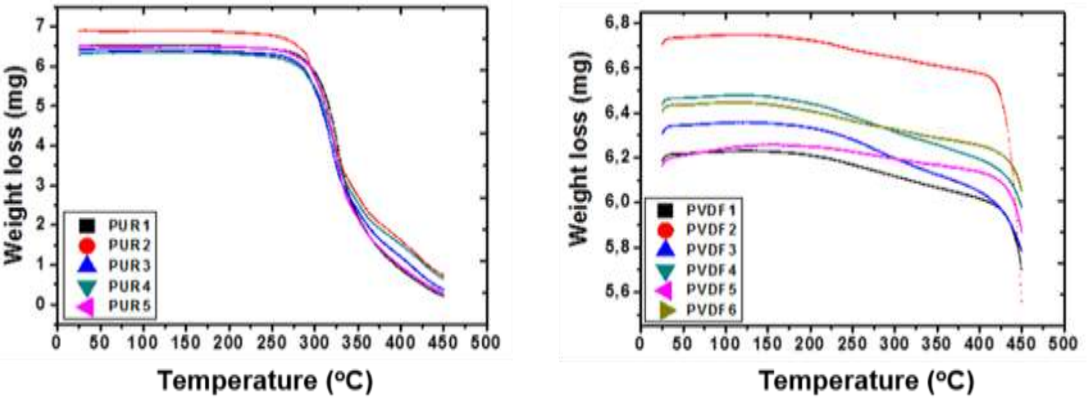


Figure 12. TGA curves of PUR and PVDF nanofibrous membranes embedded with and without aerogel

5.6 Air permeability of aerogel-embedded fibrous materials

Air permeability of nonwoven substrate was found to sharply decrease when a nanofiber web was laminated onto its surface. This is mainly because nanofiber web has a large number of smaller pores and relatively lower porosity, which covers the open pores of nonwoven substrate and prevents the air flow go through. Moreover, the thermal adhesive would reduce the pores of nonwoven substrate in some degree, this may also account for the decrease in air permeability. Aerogel showed limited influence on air permeability of layered fabrics. For a specified fabric, air permeability had a strong correlation with air pressure gradient.

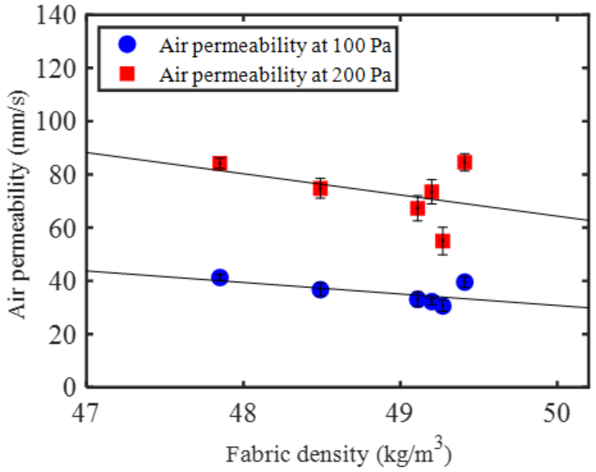


Figure 23. Air permeability of layered nanofibrous web/silica aerogel/ nonwoven

The air permeability of multilayer fabrics is influenced not only by the porosity, but also by other parameters relating the number of layers, the structural parameters of a single fabric and the multilayer structure of the system. In a multilayer fibrous system, the air permeability decreases with the increasing of the number of layers due to the increase in the resistance to flow of air through the pores of the fibrous material. The resistance to flow depends upon the alignment of pores in one layer with that of another layer, the gap between individual layers of the fabric, and the distortion of the fibers caused by the airflow.[28] As shown in Figure 24, the multilayered samples with air pockets had slightly higher air permeability, the aerogel particles present in the multilayered fibrous materials led to small decrease in air permeability because the large voids in the fibrous structure were divided into many smaller pores and partly replaced by mesopores in the aerogels. However, aerogels had negligible effect on the porosity of fibrous material since the dominant component of aerogel particles is air.

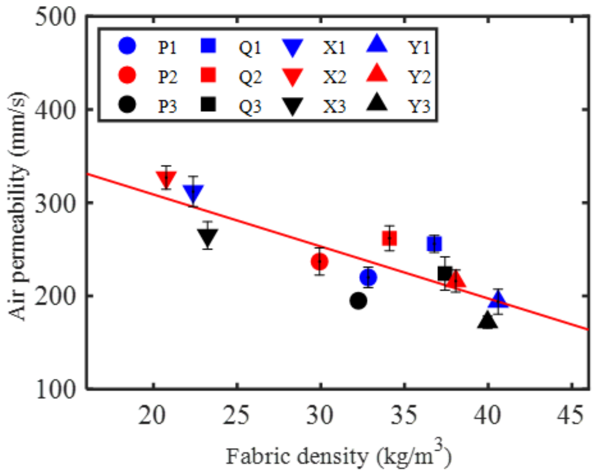


Figure 24. Air permeability of multilayered fibrous materials

5.7 Compression properties of multilayered fibrous materials

The compression resistance of multilayered fabrics is shown in Figure 25. Nonwoven-based materials with air pockets or aerogel generally have slightly lower compression resistance than regular samples. During the process of fabric compression, the inter-fiber spaces decrease continuously, the resistance force necessary to compress a fabric has to overcome the internal stresses of the fibers and the inter-fiber frictional force.[29] Thus, the compression resistance is closely related to fiber quantity, materials with less fibers have a lower compression resistance if the fiber arrangement of the fabrics are assumed to be the same. However, it is found that for foam-based materials the air pockets have insignificant effect on compression resistance while the encapsulated aerogel gives slightly increase to compression resistance.

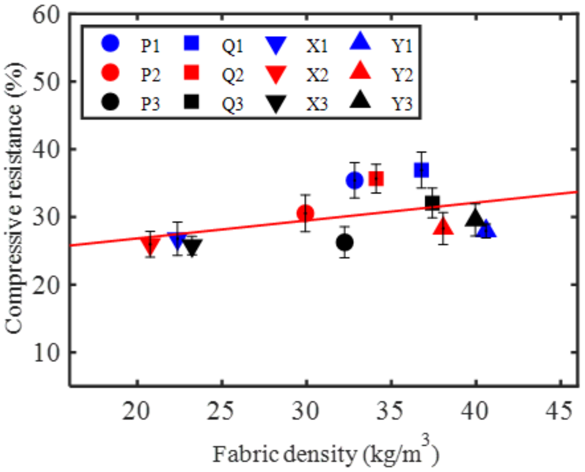


Figure 25. Compression resistance of multi-layered fibrous materials

The compression resilience values of regular materials, as shown in Figure 26, are found to lie in the range of 66.11% -76.82%, samples with air pockets and encapsulated aerogel are observed to recover slightly less than regular structures. Results also indicates that the decrease in compression resilience induced by air pockets and aerogel-encapsulated structure are very small.

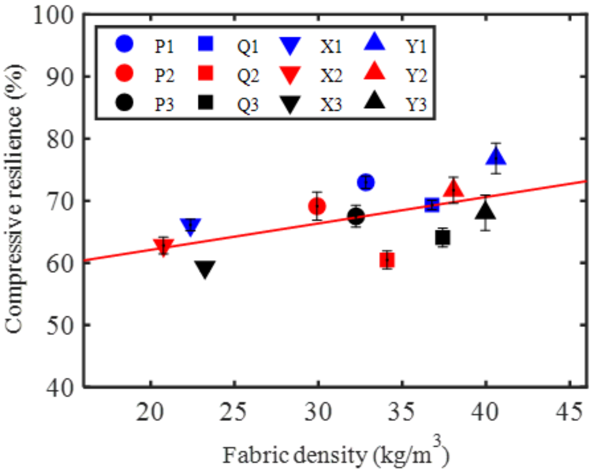


Figure 26. Compressive resilience of multi-layered fibrous materials

Nonwoven materials with air pockets and encapsulated aerogel exhibit lower thickness loss in comparison with regular samples (as shown in Figure 27), indicating these two kinds of nonwoven-based structure are able to maintain relatively higher thermal insulating ability after exposed to external forces. This is because during compression process all the fibers get compressed and trapped to each other, which will restrict the thickness recovery when the load is released. Thus, the percentage thickness loss is much higher in the case of regular structures. The sponge foam is in a reasonably consolidated state, with compression the material gets denser, resulting in better recovery after the compression pressure is released. However, this consolidated state may be partly destroyed by laser engraving treatment, which will reduce its ability to recover back to initial thickness. This is also reflected by the trend with compression resilience percentage.

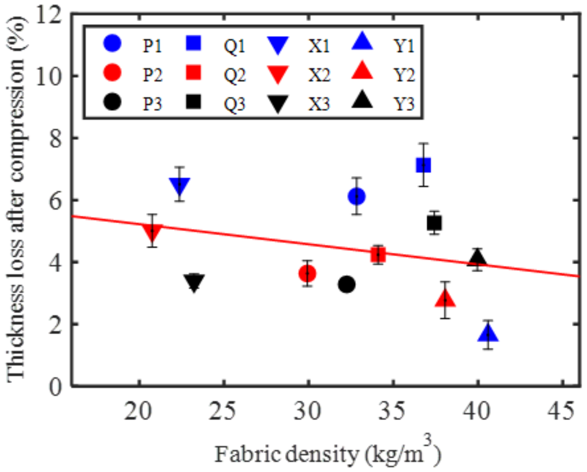


Figure 27. Thickness loss after compression test

6 Evaluation of results and new finding

In this study, layered nanofibrous web/silica aerogel/ nonwoven materials were fabricated via laminating technique by using low-melting powder as thermal binding material. Aerogel-encapsulated fibrous materials were developed based on laser engraving and laminating technique to evaluate the effect of novel structures on thermal and compression performance. Moreover, electrospun nanofibrous layers embedded with silica aerogel was produced via electrospinning process. Thermal properties of these materials were characterized by means of different evaluation techniques. Air permeability and compressibility of selected samples were investigated as well.

It is found that thermal resistance of the layered fibrous material is directly proportional to aerogel content. Remarkably, the thermal adhesive powder used for combining aerogel particles with fibrous structure could cause considerable reduction in total thermal resistance. This indicated that novel techniques to combine silica aerogel with high porous textiles with less use of binding materials should be considered. Meanwhile, the thermal resistance predicted by a series model showed a good agreement with the measured thermal resistance of the layered fibrous materials.

Results of the novel developed multilayered fibrous materials revealed that the aerogel-encapsulated samples demonstrated reduced thermal conductivity, significantly increased thermal resistance and coefficient of heat retention ability. The silica aerogel present in the fibrous structure plays an important role in contributing to the thermal performance of multilayered fibrous materials. The air pockets created by laser engraving could marginally improve the thermal performance as well. Meanwhile, thermal resistance and coefficient of heat retention ability were both strongly related to fabric thickness of the multilayered materials. Infrared thermography results showed that under infrared radiation the aerogel-encapsulated structure demonstrated lowest temperature, with 1-1.5°C lower than regular samples when the temperature difference between hot plate and environment is 10 °C. Besides, Struto nonwoven based multilayered materials were observed to have lower temperature values in comparison with PU foam-based samples, which could be attributed to the lower fabric density and higher fabric thickness. Study of the compression properties of multilayered materials revealed that nonwoven materials with air pockets and aerogel-encapsulated structure exhibited slightly lower compression resistance and thickness loss after compression test. These materials could recover to a smaller extent than regular samples. However, air pockets and the aerogel present in PU foam-based multilayered materials showed insignificant effect on compression resistance.

Measurement of thermal behavior of selected aerogel-embedded fibrous materials by convection were performed on a laboratory-made device. In preheated conditions, it was found that a low airflow velocity gave a gentle temperature drop of the heating rod and fibrous insulator while a high velocity led to a rapid temperature decline and fairly high heat transfer rate. The multilayered fibrous materials with air pockets and aerogel-encapsulated structures exhibited significantly lower initial temperature. Aerogel-treated nonwoven fabrics with higher aerogel content and higher fabric thickness demonstrated better ability to prevent against heat loss from the preheated heating rod. In continuous heating conditions, the content of aerogel present in the fibrous structure and fabric thickness are very important factors in protecting against heat loss at high airflow velocity. The heating rod to air temperature difference and heat transfer coefficient were observed to increase with the increase in airflow velocity and Reynolds number. Aerogel-treated nonwoven fabrics with lower fabric thickness and aerogel content demonstrated lower temperature difference under convection. However, all the multilayered fabrics showed higher heat transfer rate in comparison with aerogel treated nonwoven fabrics.

Study of nanofibrous membranes embedded with aerogel showed that the PVDF nanofibrous membranes had better thermal stability than PUR nanofibrous membranes. The increase in duration of electrospinning leads to higher web thickness which results in considerable decrease in air permeability. Considerable improvement of thermal insulation was observed by increasing the number and the weight per unit area of nanofibrous membranes. The results confirmed increase in thermal insulation by embedding silica aerogel in nanofibrous membranes.

7 References

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8 List of papers published by author

8.1 Journal Publications

[1] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Hiroyuki Kanai, Jiri Militky, Thermal and Compression Characteristics of Aerogel-encapsulated Textiles, *Journal of Industrial Textiles*, Vol.47, 1998-2013, 2017.

[2] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Jiri Militky and Jakub Wiener, Investigation on Laser Engraving based Application of Silica Aerogel into Nonwovens, *Fibers and Polymers*, Vol.18, No. 12, 2469-2475, 2017.

[3] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Jiri Militky, Transport Properties of Aerogel-based Nanofibrous Nonwoven Fabric, *Fibers and Polymers*, Vol.17, No.10, 1709-1714, 2016.

[4] Mohanapriya Venkataraman, **Xiaoman Xiong**, Rajesh Mishra, Jiri Militky, Jaromir Marek, Juming Yao, Guocheng Zhu, Electrospun nanofibrous membranes embedded with aerogel for advanced thermal and transport properties, *Polymers and Advanced Technologies*, Vol. 29, Issue-10, 2583-2592, 2018.

[5] **Xiaoman Xiong**, Mohanapriya Venkataraman, Darina Jašíková, Tao Yang, Rajesh Mishra and Jiří Militký, Experimental investigation of convective heat transfer in aerogel-embedded nonwovens, *Journal of Materials Science*, under review.

[6] **Xiaoman Xiong**, Mohanapriya Venkataraman, Darina Jašíková, Tao Yang, Rajesh Mishra and Jiří Militký, Thermal performance of aerogel-encapsulated fibrous materials by convection, under submission.

[7] Tao Yang, **Xiaoman Xiong**, Rajesh Mishra, Jan Novak and Jiri Militky, Sound Absorption and Compression Properties of Perpendicular-laid Nonwovens, *Textile Research Journal*, doi: 10.1177/0040517517753634, First Published January 18, 2018.

[8] Tao Yang, **Xiaoman Xiong**, Rajesh Mishra, Jan Novak and Jiri Militky, Acoustic Evaluation of Struto Nonwovens and Their Relationship with Thermal Properties, *Textile Research Journal*, doi: 10.1177/0040517516681958, First Published December 06, 2016.

[9] Tao Yang, **Xiaoman Xiong**, Rajesh Mishra, Jiri Militky, Investigation on Acoustic Behavior and Air Permeability of Struto Nonwovens, *Fibers and Polymers*, Vol.17, No.12, 2078-2084, 2016.

[10] Tao Yang, **Xiaoman Xiong**, Mohanapriya Venkataraman, Rajesh Mishra, Jan Novák and Jiří Militký, Investigation on sound absorption properties of aerogel nonwoven blankets, *The Journal of the Textile Institute*, Accepted.

[11] Tao Yang, **Xiaoman Xiong**, Ferina Saati, Kai Yang, Rajesh Mishra, Kirill V Horoshenkov, Rajesh Mishra, Steffen Marburg and Jiří Militký, Study on sound absorption behavior of multi-component polyester nonwovens: Practical and numerical methods, *Textile Research Journal*, Accepted.

8.2 Book Chapters

[1] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Juan Huang and Jiří Militký, A Review on Nanofibrous Membranes and Their Applications, Advances in fibrous material science, ISBN 978-80-87269-48-0, 2016.

[2] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Juan Huang, T M Kotresh and Jiří Militký, Heat Transfer through Thermal Insulation Materials Part I – Nonwoven Fabrics, Recent developments in fibrous material science, ISBN 978-80-87269-45-9, 2015.

[3] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Juan Huang, T M Kotresh and Jiří Militký, Heat Transfer through Thermal Insulation Materials Part II – Nano-porous Aerogel, Recent developments in fibrous material science, ISBN 978-80-87269-45-9, 2015.

[4] Tao Yang, **Xiaoman Xiong**, Rajesh Mishra, Jan Novak and Jiří Militký, Comparative Analysis on Acoustic Properties of Different Struto-type Nonwovens, Advances in fibrous material science, ISBN 978-80-87269-48-0, 2016.

[5] Tao Yang, **Xiaoman Xiong**, Rajesh Mishra, Jan Novák, Filip Sanetrník and Jiří Militký, A Review of Acoustic Properties Measurements and Wave Propagation Models of Porous Materials, Recent developments in fibrous material science, ISBN 978-80-87269-45-9, 2015.

8.3 Conference Publications

[1] **Xiaoman Xiong**, Mohanapriya Venkataraman, Rajesh Mishra and Jiří Militký, Experimental Study on Convective Heat Transfer Through Fibrous Insulators, 46th Textile Research Symposium, Teijin Academy Fuji, Susono City, Japan, September 3-5, 2018.

[2] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Jiri Militky, Transport Properties of Nonwovens with Aerogel, CLOTECH 2017, Lodz, Poland, October 11-14, 2017.

[3] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Jiri Militky, Laser Engraving based Application of Silica Aerogel into Nonwovens for Thermal Insulation, 45th Textile Research Symposium, KIT Kyoto, September 14-16, 2017.

[4] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Jiri Militky, Investigation on Thermal and Compression Performance of Aerogel Incorporated Textiles, Textile Bioengineering and Informatics Symposium, TBIS-2017, Wuhan, China, May 18-20, 2017.

[5] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, and Jiri Militky, Thermophysiological Performance of Aerogel Embedded Electrospun Nonwoven Layers, 44th Textile Research Symposium, IIT Delhi, December 14-16, 2016.

[6] **Xiaoman Xiong**, Aerogel-based Nanofibrous Nonwoven Fabrics for Thermal Insulation, Workshop Bílá voda 2016, Harrachov, Liberec, September 20-23, 2016.

[7] **Xiaoman Xiong**, Tao Yang, Rajesh Mishra, Effect of weaving friction on yarn hairiness and mechanical properties, 8th TBIS-APCC International conference, Zadar, June 14-17, 2015.

[8] Tao Yang, **Xiaoman Xiong**, Rajesh Mishra and Jiri Militky. Relationship Between Sound Absorption Property and Transmission Behavior of Struto Nonwoven, Textile Bioengineering and Informatics Symposium, TBIS-2017, Wuhan, China, 18-20 May, 2017.

[9] Tao Yang, **Xiaoman Xiong**, Rajesh Mishra and Jiri Militky. Acoustic and thermophysiological properties of Struto nonwovens, 44th Textile Research Symposium, IIT Delhi, December 14-16, 2016.

[10] Tao Yang, **Xiaoman Xiong**, R Mishra, V. Arumugam, Mohanapriya Venkataraman and Jiri Militky. Study on Acoustic and Thermal Performance of Struto Nonwovens, 21st Strutex, december 1-2, p. 345-350, ISBN:9788074942693, Liberec, 2016.

[11] Tao Yang, **Xiaoman Xiong** and R. Mishra. 3D orthognal fabric composites from carbon fiber, 8th TBIS-APCC International conference, Zadar, 14-17 June, 2015.

[12] Tao Yang, **Xiaoman Xiong**, R Mishra, Jan Novák and Jiri Militký. Sound absorption behaviour of perpendicular-laid nonwovens, 46th Textile Research Symposium, Teijin Academy Fuji, Japan, September 3-5, 2018.

9 Curriculum vitae

XIAOMAN XIONG, M.Eng.

Date of birth: 14 July 1987 Nationality: China

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+420-776-589-928

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AREAS OF RESEARCH

- Thermal properties of fibrous materials

CURRENT RESEARCH OBJECTIVES

- Study on influence of structural parameters on thermal properties of fibrous materials
- Development of aerogel-embedded fibrous materials and performance evaluation
- Investigation on electrospun nanofibrous membranes embedded with silica aerogels

EDUCATION

Technical University of Liberec - Liberec, Czech Republic
Pursuing doctoral studies at Faculty of Textile Material and Engineering
Date: July 2014 until now
Thesis: Study of Aerogel Embedded High-performance Fibrous Materials

Wuhan Textile University - Wuhan, Hubei, China
Master of Textile Material and Engineering
Date: September 2010 to June 2013
Thesis: Effect of Weaving Friction on the Weavability of Cotton Yarn

Wuhan Textile University - Wuhan, Hubei, China
Bachelor of Textile Engineering
Date: September 2006 to June 2010
Thesis: A Study on Anti-felting Finishing of Woolen Fabric

INTERNSHIP

- 12.2015-3.2016

Faculty of Textile Science and Technology, Wuhan Textile University Wuhan, Hubei, China
Mentor: Prof. Weilin Xu

- 10.2016-12.2016

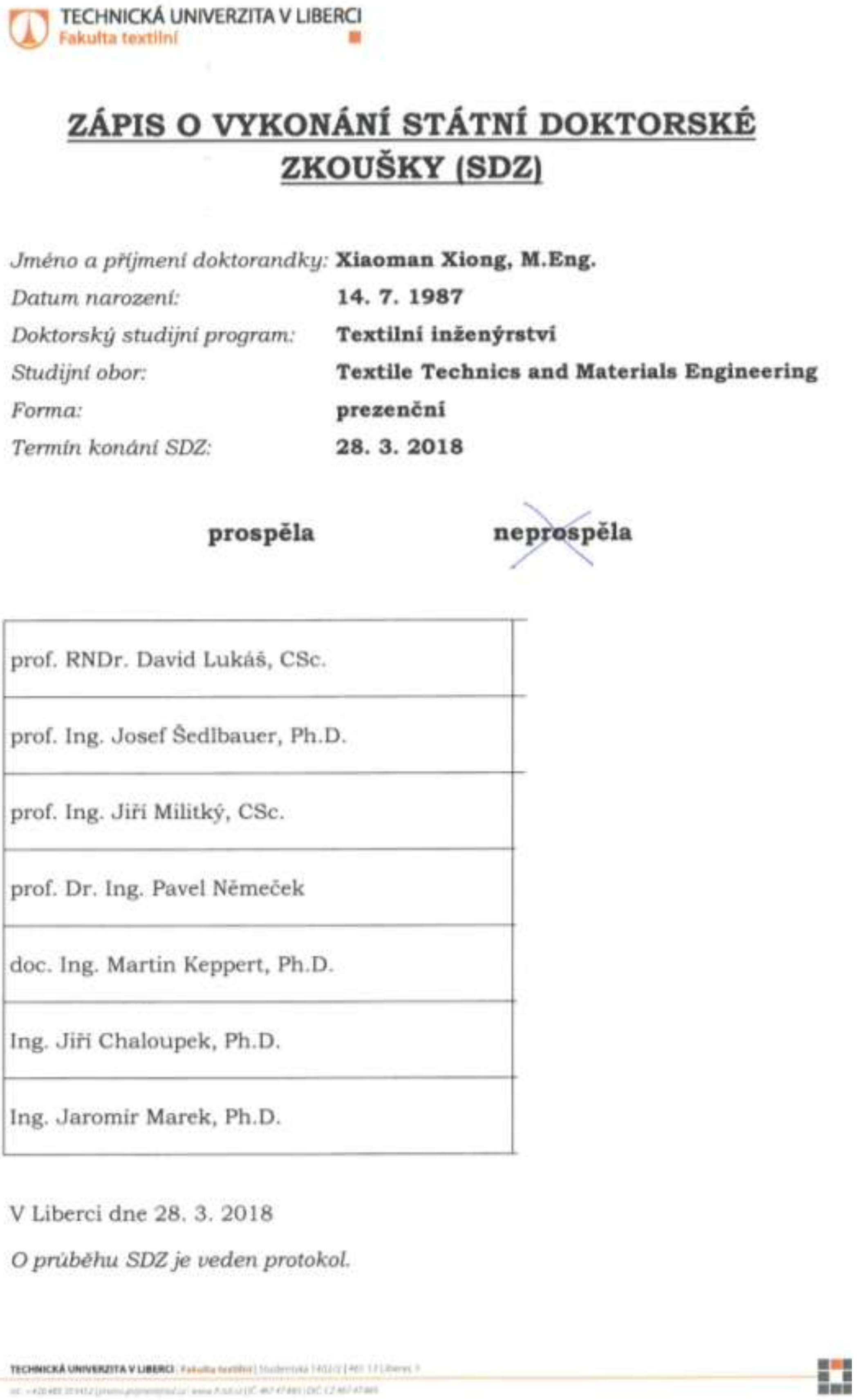
Interdisciplinary Graduate School of Science and Technology, Shinshu University, Nagano, Japan
Mentor: Prof. Hiroyuki Kanai

WORK EXPERIENCE

- 6.2013-2.2014 Intellectual Property Specialist

Product R & D department, Fujian Xinhua Ltd., Jinjiang, Fujian, China

10 Record of the state doctoral exam



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Fakulta textilní

ZÁPIS O VYKONÁNÍ STÁTNÍ DOKTORSKÉ ZKOUŠKY (SDZ)

Jméno a příjmení doktorandky: **Xiaoman Xiong, M.Eng.**
Datum narození: **14. 7. 1987**
Doktorský studijní program: **Textilní inženýrství**
Studijní obor: **Textile Technics and Materials Engineering**
Forma: **prezenční**
Termín konání SDZ: **28. 3. 2018**

prospěla **~~neprospěla~~**

prof. RNDr. David Lukáš, CSc.
prof. Ing. Josef Šedlbauer, Ph.D.
prof. Ing. Jiří Militký, CSc.
prof. Dr. Ing. Pavel Němeček
doc. Ing. Martin Keppert, Ph.D.
Ing. Jiří Chaloupek, Ph.D.
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11 Recommendation of the supervisor

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Supervisor's opinion on PhD thesis of Ms. Xiaoman Xiong, M.Eng.

Thesis title: **Aerogel Embedded High-performance Fibrous Materials**

Ms. Xiaoman Xiong, M.Eng., has worked for her PhD thesis under my supervision since 2014. Her research is focused on thermal and related performance of advanced fibrous materials embedded with aerogel.

In her work, she has developed several multilayered fibrous structures with silica aerogel embedded inside the layers. The effect of silica aerogel in such materials has been investigated and evaluated with sufficient statistical accuracy. She has proposed and developed novel techniques to incorporate such aerogel granules into foam and textile fibrous assemblies. Laser engraving based incorporation of aerogel has been described in much detail. Moreover she has adopted electrospraying and electrospinning techniques to produce nanoporous and nanofibrous structures with aerogel in the spinning solution. PVDF and PUR membranes with aerogel have been developed and evaluated for thermal properties. Study of nanofibrous membranes embedded with aerogel showed that the PVDF nanofibrous membranes had better thermal stability than PUR nanofibrous membranes. The glass transition and melting point was not affected by the aerogel content in the fibrous layers. The increase in duration of electrospinning leads to higher web thickness which results in considerable decrease in air permeability. Considerable improvement of thermal insulation was observed by increasing the number and the weight per unit area of nanofibrous membranes. The results confirmed increase in thermal insulation by embedding silica aerogel in nanofibrous membranes.

New devices and methods are developed to evaluate thermal transmission through convection and radiation. The ideas, experiments and data generated as part of this research, have added to the knowledge base that could be useful to define the future direction and provide insightful references to researchers.

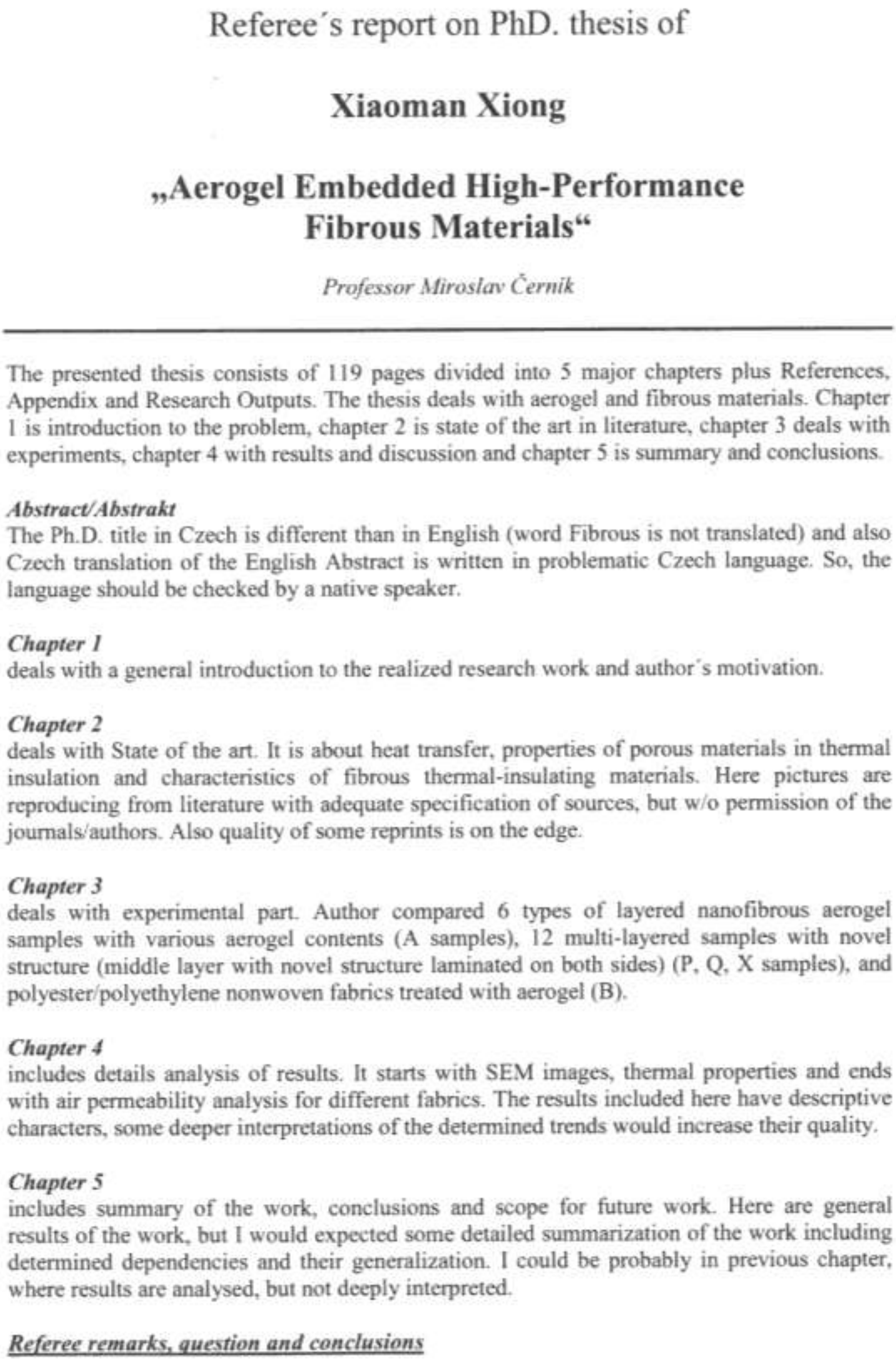
Her publication activities are in excellent level. Ms. Xiaoman Xiong has published 12 papers in international journals with impact factor. A few more are under review and expected to be published soon. She has presented more than 10 papers individually or jointly at international conferences. 5 chapters are published in reputed books. These are strong indicators for her thesis as a comprehensive work of independent research.

The dissertation work is formally correct, parts of the text or images are properly cited and all literary sources are listed in accordance with established rules. Checking of plagiarism on 16. 05. 2019 showed no relevant similarity to other work.

I therefore recommend the thesis for defense.

doc. Rajesh Mishra, PhD
Supervisor

12 Opponent's reviews



Referee´s report on PhD. thesis of

Xiaoman Xiong

„Aerogel Embedded High-Performance Fibrous Materials“

Professor Miroslav Černík

The presented thesis consists of 119 pages divided into 5 major chapters plus References, Appendix and Research Outputs. The thesis deals with aerogel and fibrous materials. Chapter 1 is introduction to the problem, chapter 2 is state of the art in literature, chapter 3 deals with experiments, chapter 4 with results and discussion and chapter 5 is summary and conclusions.

Abstract/Abstrakt

The Ph.D. title in Czech is different than in English (word Fibrous is not translated) and also Czech translation of the English Abstract is written in problematic Czech language. So, the language should be checked by a native speaker.

Chapter 1

deals with a general introduction to the realized research work and author´s motivation.

Chapter 2

deals with State of the art. It is about heat transfer, properties of porous materials in thermal insulation and characteristics of fibrous thermal-insulating materials. Here pictures are reproducing from literature with adequate specification of sources, but w/o permission of the journals/authors. Also quality of some reprints is on the edge.

Chapter 3

deals with experimental part. Author compared 6 types of layered nanofibrous aerogel samples with various aerogel contents (A samples), 12 multi-layered samples with novel structure (middle layer with novel structure laminated on both sides) (P, Q, X samples), and polyester/polyethylene nonwoven fabrics treated with aerogel (B).

Chapter 4

includes details analysis of results. It starts with SEM images, thermal properties and ends with air permeability analysis for different fabrics. The results included here have descriptive characters, some deeper interpretations of the determined trends would increase their quality.

Chapter 5

includes summary of the work, conclusions and scope for future work. Here are general results of the work, but I would expected some detailed summarization of the work including determined dependencies and their generalization. I could be probably in previous chapter, where results are analysed, but not deeply interpreted.

Referee remarks, question and conclusions

QUESTIONS

1. Table 3.1 reports values and 95% confidence interval. How many parallel measurements of these parameters were done to get these statistics?
2. The section about microstructure of nanofibrous membranes (4.1.3.) stated: "…membrane consists of firebs in the submicrometer range arranged…", but Figures which should support this structure (Fig. 4.4 and 4.3) have scale bar of 100 µm (20 µm). Could you show the structure in the batter scale?
3. What is the thickness of nanofiber web (N) used for layered fabric preparation? From Table 4.1 it look that it is only 40 nm?
4. Theoretical model (p.52-3) does not describe the experimental data for low aerogel content. The difference is decreasing with increased amount of aerogel. Author explained this by underestimation of resistance since the thickness of the middle layer is decreased. Is not possible to determine thickness of this layer independently? By microscopy? Other measurement?

Imperfections and recommendations

The language and overall arrangement of the Ph.D. thesis in good and sufficient. These are few imperfection and errors in the text.

Example of imperfections:

- Units of thermal conductivity should be [W/(m.K)] not W/m.K (page 7), similarly h_c [$W/(m^2.K)$] not [$W/m^2.K$] (p.8);
- Pictures from SEM have no scale bar (4.1; 4.2)
- The values in confidence interval must have the number of decimal digits (not 6.2±0.01)
- Is it very difficult to follow data curves in some of the Figures (e.g. 4.24, the data points specifying lines are tiny and colours difficult to see).

Referee's conclusion

The presented thesis of Xiaoman Xiong is logic, has all necessary parts and show the author understand her work and she is able to put results logically into appropriate parts. The work significantly contributes to knowledge in the subject. There are only a few recommendations for next author's work. The language is good and fully understandable.

The thesis is good and meets all criteria to be taken to the defence.

In Liberec (Czech R.) on May 10, 2019 **Professor Miroslav Černík**

Opponent´s review

Author of dissertation: Xiaoman Xiong, M.Eng.

Title of dissertation: Aerogel Embedded High-performance Fibrous Materials

The main aim of the dissertation is to study thermal performance of fibrous materials based on different types of aerogels.

The dissertation consists of 119 pages and is divided into 5 parts, appendix, references and research outputs. Work is written very well and contains only a few typing errors.

In the first part, the individual objectives of the experimental work are described and divided into 4 separate tasks. The first aim of this work is to study the thermal resistance of materials based on non-woven nanofibrous web and silica aerogel with thermal binding material. Furthermore, the author studied the use of laser engraving on the above samples without using thermal binding materials. Another group of materials studied for thermal properties was prepared using encapsulated aerogel. The last type of studied material was PUR and PVDF membranes produced by electrospinning.

In the dissertation thesis, the author set out the objective of evaluating different evaluation methods for the determination of thermal properties in the above samples. Evaluation of choosing methods of thermal performance determination is the last aim of this work. The second part of this thesis deals with research of current knowledge in the field of thermal transfer and its evaluation. Characteristics and using of selected insulating materials is also described in this part. The third chapter, experimental part of dissertation, describes the procedures used to prepare individual samples of insulators and also the procedures for evaluating not only thermal performance these samples. The following methods have been selected for material structure evaluation: SEM, digital microscope, thermal performance methods: TGA, DSC, Alambeta, IR camera ThermaCAM TV300, KES FT II Thermolabo tester, measuring of convective heat and method for compression properties testing.

Fourth part of thesis contains results, which are very well organized and discussed in detail. The fifth part summarizes the partial conclusions from previous part and contains suggestions for future of research in this area.

In this dissertation many experiments were carried out with different types of materials. The author appropriately selected the evaluation methods. This work gives a very good overview of the possibilities of using materials based on aerogel in the field of thermal resistant materials. The author's publication activity is sufficient. The results of her thesis were published in 11 journals, 5 chapters of books and presented at 7 international conferences.

Questions for defense:

1. Why did you choose as a preheat temperature at 60 °C?
2. Why the thermal behavior under convection were determined only for samples B and Q? I am missing results for other samples based on Struto nonwovens.

In conclusion I recommended this dissertation for the defense.

In Pardubice 14th May 2019

Ing. Michal Černý, Ph.D.