

Abstract

Sintering and interlocking of model nonwoven materials composed of a mixture of Polycaprolactone (PCL) and Polyacrylonitrile (PAN) fibers by means of forced convection of hot air through their pores is studied experimentally and theoretically. PCL has a much lower melting point than PAN, and the air temperature was sufficiently high to melt the former, while the latter stayed solid. These molten PCL fibers became a binder and conglutinated the PAN matrix, enhancing stiffness. This was demonstrated by measuring the effect of heat treatment on the resulting Young's modulus of these compound nonwovens, as well as by the corresponding micro-morphological changes revealed by scanning electron microscopy. It was also shown that heating past the melting point of the binding fibers (PCL) would not further increase stiffness of the nonwovens, neither would heating for longer periods of times. A theoretical model describing the heating process was developed and tested experimentally. The model was verified using poly(ethylene terephthalate) PET nonwovens, which revealed good agreement of the data with the theoretical predictions.

Keywords: Transient Temperature Distribution, Forced Air Convection, Sintering, Nonwovens, Modulus of Elasticity.

1. Introduction

Thermally compressed porous nonwoven mats have become a popular alternative to the use of bulk plastics in the automotive industry. The versatility of these nonwovens produced from low cost recycled fibers is an attractive feature in manufacturing stiff, lightweight porous mats for sound insulation and such load-carrying surfaces as the trunk bottom [1-2]. Thermally

compressed nonwovens are also highly formable into complex contours and their cost-to-performance ratio makes them a viable alternative to bulk plastics [1-2].

Porous nonwoven mats containing a mixture of PET fibers and bi-component core-shell fibers with shell of low-melting temperature copolymer of poly(ethylene terephthalate) PET and core of PET are used in some automotive applications. Such mixed nonwovens are heated by means of forced hot air convection through the pores. Namely, hot inlet air of constant temperature is pushed to filtrate through the sample at a constant velocity. This inlet air temperature is high enough to melt the lower melting point fibers and interlock the adjacent higher melting point fibers which stay solid [1]. The manufacturing of load-bearing functional fibrous mats is based on conglutination by means of this molten low-melting temperature copolymer of poly(ethylene terephthalate) PET shell of bi-component fibers. The melting temperature of this copolymer shell is 110 °C or 180 °C depending on the copolymer choice. It is known that the PET matrix fibers experience significant thermal [3-5] and hydrolytic [6-7] degradation at the processing temperatures of 280-300 °C [5]. Thermal degradation occurs due to macromolecular chain scission at the elevated processing temperatures starting at temperatures of 270 °C [3], reducing the molecular weight of polymer [3, 5]. In the case of hydrolytic degradation, moisture present during processing past the glass transition temperatures also induces macromolecular chain breakup [5-7] with a rate several orders higher than in the case of thermal degradation [6]. These forms of degradation have an abrupt adverse effect on stiffness and strength [8-12] and especially on the strength of thermally-bonded nonwovens [9-12]. Therefore, the working temperature range for successful conglutination of the matrix without deterioration of stiffness and strength of individual fibers should be slightly above the melting

temperature of the binder shell, either 110 °C or 180 °C (depending on the copolymer used in the shell), while the detrimental overheating should be avoided by a proper temperature control.

The inlet air temperature is important in the case of creating load-bearing fibrous surfaces. In the processes used by the industry, nonwovens are primarily heated under the conditions of a fixed hot inlet air temperature. Prior work by the other research groups has been related to heat transfer through porous media, with forced air convection parallel to the porous medium surface [13-21]. Some of these works focused solely on convective heat transfer [13-14], whereas others have also taken into account thermal diffusion under different boundary conditions [16-19]. In addition to these works, analytical solutions of the convection-diffusion equations for species migration have been explored [22]. Although model [22] offers a similar solution to non-steady state diffusion-convection, albeit under different boundary conditions, it does not account for the effect of porosity. Note also research works devoted to convection-diffusion effect in infinite porous media with a planar heat source [19], and heat transfer through a melting porous medium [21]. In addition, forced air convective heat transfer under non-Darcian filtration flow has also been explored [16, 17]. In one of these studies, empirical relationships were used to describe non-Darcian effects along with volume averaging techniques to numerically describe two-dimensional heat transfer in a porous medium [16]. Notably, much theoretical work has been aimed at forced air convection in a porous medium heated by planar heat sources [17-19]. In those cases, constant heat fluxes are introduced along the inlet face or lengths of the samples. Here, we deal with the forced convection normal to the porous medium surface, with the flow going through the sample. This situation did not attract much attention in the past, as to our knowledge. The present model is relevant for the situation studied

experimentally in the present work, as well as is closely linked to the industrial context related to creation of load-bearing fibrous surfaces.

The present work aims at forced filtration of hot air through a compound porous matrix, with one material having a low melting temperature, making it a molten binder. In this process, it is important to understand the transient thermal history inside of a nonwoven mat due to heating with a constant inlet air temperature. Theoretical model of the thermal process is proposed and compared to the experimental data of the present work.

| Nomenclature | | | |
|---------------------|--|----------------------|---|
| $c_{p,g}$ | specific heat capacity of gas flowing through the nonwoven | <i>Greek symbols</i> | |
| | | α | effective thermal diffusivity of porous medium and gas, Eq. (3) |
| $c_{p,s}$ | specific heat capacity of nonwoven material | α_s | thermal diffusivity of porous medium |
| k_g | thermal conductivity of gas | λ_n | eigenvalues, Eq. (10) |
| k_s | thermal conductivity of porous medium | ρ_g | density of gas flowing through nonwoven |
| L | longitudinal thickness of the porous medium | ρ_s | density of nonwoven material |
| Pe_{eff} | effective Peclet number, Eq. (11) | | |
| t | time | σ | related to the specific heat capacities of porous medium and gas, Eq. (2) |
| $T(x,t)$ | temperature distribution in porous medium | | |
| U_g | inlet gas velocity | | |
| x | longitudinal coordinate | | |

2. Experimental

2.1 Materials

Two separate experiments were conducted to elucidate the effect of forced hot air convection on the internal structure of polymer nonwoven mats. In the first type of experiments,

model PET nonwoven mats of thickness 1.5 cm were heated by forced convection of hot air through them in a setup described in the following subsection. These samples were cut as cylinders of the 1.5 cm thickness and diameter 1.7 cm, i.e. these samples were thick, which was useful for measuring temperature inside them and distinguishing it from that of the oncoming hot air flux. The material properties of PET and air are available in [23] and [24], respectively, and the relevant values are listed in Table 1.

| Gas (air) at 350 °C | | Solid skeleton of the nonwoven (PET) | |
|--|--------|--|-------|
| $c_{p,g}$ [kJ kg ⁻¹ K ⁻¹] | 1.009 | $c_{p,s}$ [kJ kg ⁻¹ K ⁻¹] | 1.040 |
| k_g [W m ⁻¹ K ⁻¹] | 0.0314 | k_s [W m ⁻¹ K ⁻¹] | 0.375 |
| ρ_g [kg m ⁻³] | 1 | ρ_s [kg m ⁻³] | 1385 |

Table 1. Physical properties of the gas and nonwoven material used in the experiments.

The average porosity ϕ of the nonwoven mat was measured experimentally. A scanning electron microscope (SEM), Hitachi S-3000N, was used to image the upper layer on nonwoven facing the flow and then a MATLAB code was used to process the data from the SEM images and determine the porosity of nonwoven mats. Before imaging, samples were sputter coated with 7.5 nm of platinum-palladium and several SEM images of this kind and an intermediate stage of their processing are shown in Fig. 1.

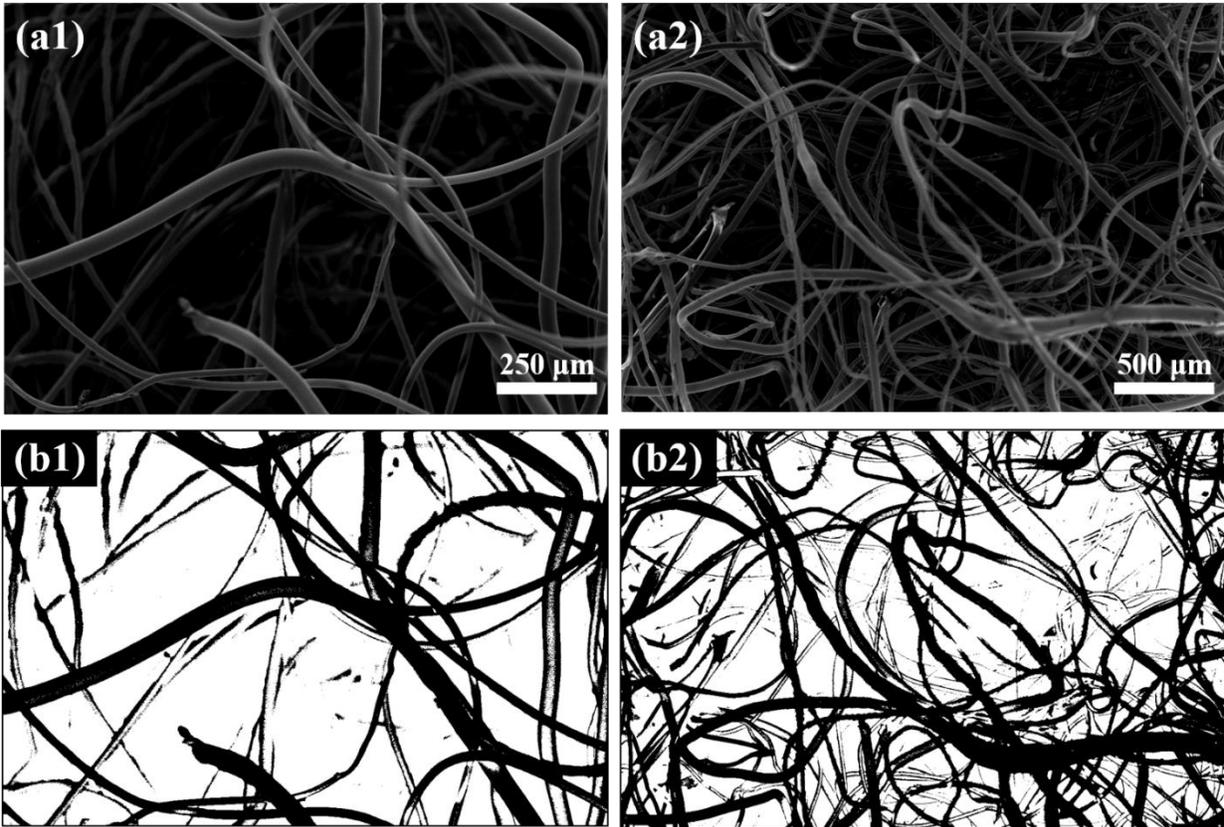


Figure 1. SEM images of PET nonwoven mats. Panels (a1) and (a2) show the original SEM images. Panels (b1) and (b2) depict the images processed in such a way that all fibers on the surface layer which will face the hot air flow are located in focus. Accordingly, the fibers at the surface are seen as black filaments on the white background.

The original SEM images were converted to black and white in such a way that fibers at the surface are seen alone on the white background as in Figs. 1 (b1) and 1 (b2). A MATLAB code was used to calculate the porosity of each sample as follows. The number of white pixels in each image was counted and divided by the total number of pixels in that image (using the processed SEM images). Ten SEM images were taken and processed using this method resulting in the average porosity of $\phi = 0.60$.

In the experiments of the second type, the effect of forced hot air convection on nonwoven mats containing a mixture of polycaprolactone (PCL) and polyacrylonitrile (PAN) electrospun nanofibers was elucidated. These porous nonwoven mats are denoted as PCL-PAN mats. The melting point of PCL is 56-65 °C [25] and that of PAN is 319 °C [23]. The reason for choosing nonwovens different from PET used in the first type of experiments was due to the need to reach the melting point of the binding polymer. Since electrical heating of air was used, it was impossible to reach the melting point of PET, which is 250-265 °C [23]. On the other hand, using PCL, it was possible to heat up the PCL-PAN nonwoven to temperatures high enough for melting PCL nanofibers and conglomerating by them the PAN nonwoven. Such conglomeration is of interest in aiming to improve the mechanical properties of nonwovens and is reminiscent of the processes used in the automotive industry.

2.2 Electrospinning Setup

Electrospinning used to form PCL-PAN nonwoven mats employed a modified setup ramifying from the one described in [26]. In brief, two needles were placed horizontally on the opposite sides of a 35 cm rotating drum (cf. Fig. 2). The needles were attached to a DC high voltage source, whereas the drum was grounded. A solution of 15 wt% of PCL ($M_w = 80$ kDa) dissolved in a 1:1 ratio (by weight) mixture of dimethylformamide (DMF) and tetrahydrofuran (THF) was fed through one needle to issue PCL nanofibers horizontally onto the rotating drum collector, whereas in the needle on the opposite side, 12 wt% solution of PAN ($M_w = 180$ kDa) in DMF was used to issue PAN nanofibers. All materials were obtained from Sigma-Aldrich.

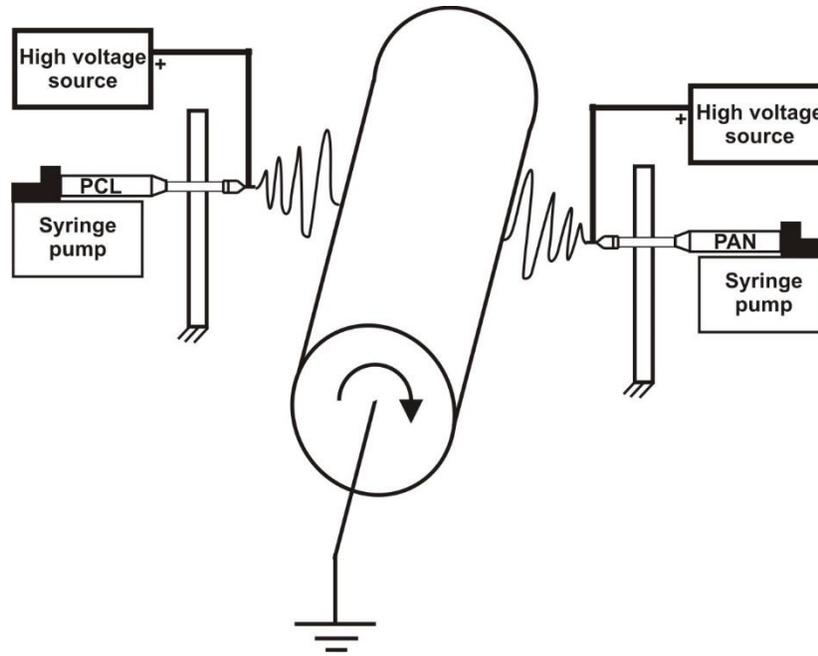


Figure 2. Schematic of the electrospinning setup. The PCL solution was issued toward one side of the rotating drum collector, whereas the PAN solution was issued toward the opposite side to form and interwoven nonwoven mat of PCL and PAN nanofibers.

Nanofibers were collected onto the rotating drum collector covered with aluminum foil for 90 min. Samples were then cut into 3 cm (in diameter) thin disks with the machine direction imposed by the drum marked on the sample edge. The sample thickness was 0.17 mm. These samples were installed in the convective heating setup described in the following section.

2.3 Apparatus for Convective Heating of Nonwovens

A convective heater was designed to generate filtration flow of hot air through porous nonwoven samples. In the experiments of the first type, porous PET nonwoven samples were

installed in the convective heater as shown in Fig. 3. High pressured air entering the convective heater was heated up to a constant inlet air temperature T_f by using a cartridge heater encased in a copper-finned shell. The temperature T_f was regulated using a power controller and temperature relay. The channel housing the porous nonwoven samples was constructed from Teflon, which provides an appropriate insulation to minimize heat losses. A thermocouple, Omega 5TC-TT-T-40-36, was inserted to the sample axis at a distance of 2 mm from the front surface of nonwoven. This thermocouple was used to measure transient temperature of the nonwoven mat.

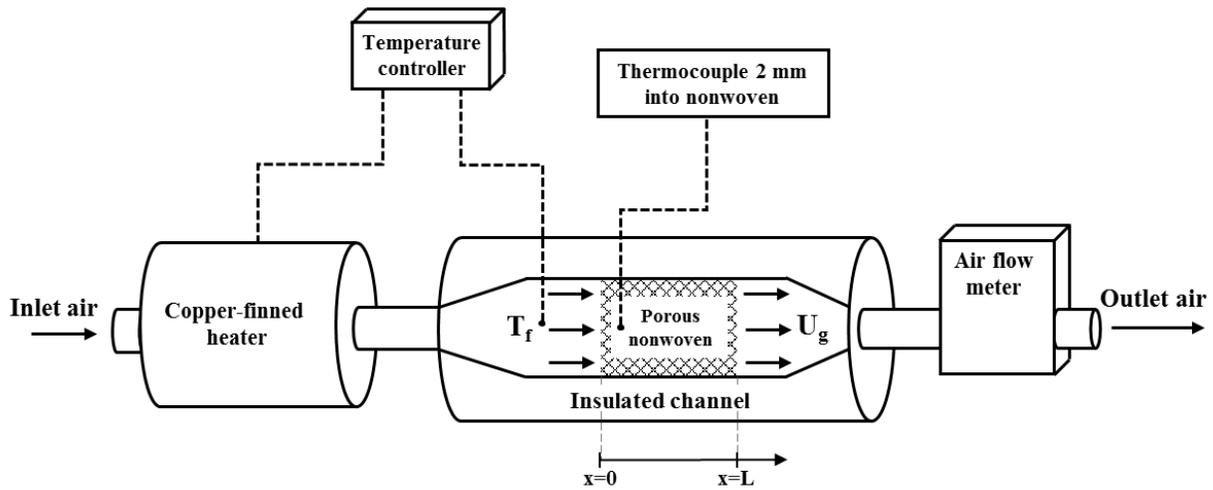


Figure 3. Sketch of the forced-convection setup used to heat nonwoven samples up. The inlet air is supplied at a fixed flow rate through a cartridge heater encased in a cylindrical copper-finned shell resulting in a controlled constant air temperature T_f , which affects the front side of porous nonwoven sample installed in the insulated channel. The average velocity of hot air exiting the sample U_g was regulated by the upstream pressure and measured by an air flow meter at the exit. Temperature at the distance of 2 mm inside the sample from its front side was measured by a thermocouple.

The average air velocity U_g was measured using a flow meter Omega FMA-1820A behind the sample. The average velocity through the sample was found as the ratio of the measured flow rate to the cross-sectional area of the sample. It should be emphasized that the pressure loss across the samples was negligibly small. The inlet air temperature T_f was set at approximately 50 °C. The velocity U_g was in the 0.5 – 1 m/s range. These values were dictated by limitations imposed by the setup.

In the experiments with PCL-PAN nanofiber samples the setup was modified as shown in Fig. 4. Namely, the air flow meter at the exit was disconnected and the thermocouple removed (cf. Figs. 3 and 4). This modification was made since the air flow meter used in the prior setup was limited to a maximum flow rate of 10 L/min and temperature of 50 °C.

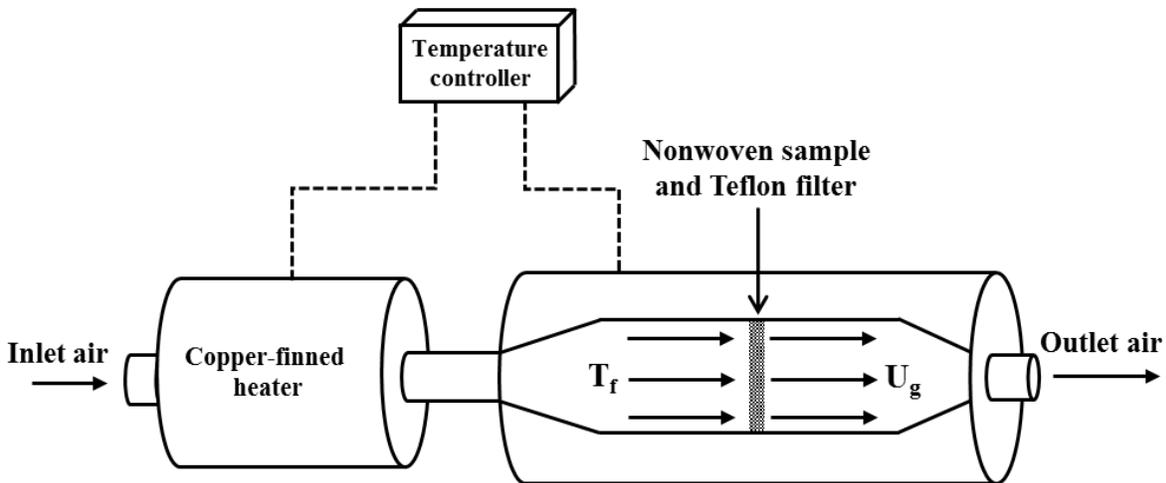


Figure 4. Sketch of the modified forced hot air convective heater used with PCL-PAN samples.

PCL-PAN nonwoven samples were placed onto a highly porous Teflon filter (Advantec WU-36240-06, of porosity 0.73 and pore size 5 μm) to facilitate mechanical integrity during

blowing of hot air through them, and installed into the convective heater, 1.7 cm deep in its exposed section that was heated by the hot air from the cartridge heater section. As before, this inlet air temperature was sustained constant. Such constant air flux temperatures chosen for these experiments were 55 °C, 70 °C, and 80 °C. The velocity of the oncoming air at the sample was in the 15-25 m/s range.

2.4 Temperature Measurement

The inlet air temperature was measured by thermocouple (Omega 5TC-TT-T-30-36) with the response time of 0.3 s (up to 67% of a real value), which means that it would take approximately 2.25 s (up to about 100% of a real value) for the thermocouple to respond to an abrupt change in temperature. The thermocouple was regulated by the temperature controller (Watlow Ez-Zone). The nonwoven temperature was measured using the thermocouple (Omega 5TC-TT-T-40-36) inserted to the sample axis at a distance of 2 mm from its front surface. This thermocouple had a response time of 0.2 s, which means that it would take approximately 1.2 s for the thermocouple to respond to an instantaneous change in the nonwoven temperature. The standard deviation of the thermocouple was ± 1 °C. The thermocouples which measured the inlet air temperature, as well as the nonwoven temperature, were connected to a thermocouple acquisition module, Omega TC-08, which allows, in principle, measurements 10 times per second. However, in the present experiments temperatures were measured only twice per second due to the thermocouple response time.

2.5 Nonwoven Heating: Experimental Procedure

The inlet air was pre-heated to a predetermined steady-state air temperature, T_f . This value was then recorded and the inlet air flow through the copper-finned heat exchanger was turned off. After this pre-heating stage, a nonwoven PET sample with the thermocouple embedded axially at $x=2$ mm from the front was loaded into the channel with the flow meter being attached to the exit. This allowed the initial temperature of the PET sample, T_i , to be the room temperature. Immediately after sealing the channel, the inlet air flow was turned on again, as well as the data acquisition device triggered to experimentally record the transient temperature for 15 s. The inlet air velocity was also immediately recorded using the flow meter. The copper-finned heat exchanger was turned off while loading the sample for the entire test, to sustain a steady-state inlet temperature T_f . This was done to prevent the inlet air from heating to higher temperatures. The latter could happen if the heat exchanger (including the temperature and power controllers) would be triggered on, which creates temperature fluctuations of ± 2 °C. While the sample was loaded, the stagnant air continued to be heated by the copper fins. This created an impulse of hot air at the beginning of the test, with the temperature higher than a desired inlet air temperature, leading to possible experimental inaccuracy. In addition to the difficulties of regulating the inlet air temperature, there were also inaccuracies associated with controlling the inlet air velocities. In fact, air entering the convective heater did not have a permanently constant velocity. However, the heating time of nonwoven PET samples in these experiments was approximately 3 s, and during this time span the inlet air velocity remained relatively constant, decreasing by only 7.4 %. After 15 s, the inlet velocity would diminish by 16.0 %.

2.6 Heating Effect on the Sample Stiffness

The effect of sample heating on the modulus of elasticity was explored as follows. First, the air channel was heated up to a desired constant air flux temperature, T_f . After that, the inlet air flow and copper-finned heat exchanger were turned off and a disk-like PCL-PAN sample (a nonwoven membrane) was installed into the insulated air channel. Next, the inlet air flow and the copper-finned heat exchanger were turned on and the samples were heated (i) for 5 min at 55 °C, or (ii) for 30 s at 70 °C, or (iii) for 5 min at 70 °C, or (iv) for 30 s at 80 °C. Then, such samples were subjected to tensile tests described in the following sub-section.

As in the case of heating PET samples described in sub-section 2.5, stagnant air also continued to be heated by the copper fins in the present experiments with PCL-PAN samples. In these cases the heat exchanger was turned on during the experiments to regulate the air temperature for the pre-determined periods of heating of PCL-PAN samples of 30 s and 5 min. This inlet air temperature fluctuated by ± 2 °C relative to the pre-determined constant inlet air temperatures. For the pre-determined inlet air temperature of 70 °C, the real values were fluctuating the 68-72 °C range, whereas for 80 °C, it was fluctuating in the 78-82 °C range.

2.7 Tensile Tests of PCL-PAN Nonwovens

A uniaxial mechanical testing machine, Instron 5942, with a 100 N load cell was used to obtain the entire stress-strain curve of the PCL-PAN nonwoven samples before and after heat treatment described in sub-section 2.6. The heat-treated samples were cut into rectangular strips of 0.5 cm by 3 cm with the longer side being in the machine direction. These samples were loaded in the testing machine with a 0.8 cm test length (in the machine direction) and clamped identically on both sides by pneumatic grips. The thickness of the unheated PCL-PAN samples

was 0.17 mm. After heat treatment, the measured thicknesses were in the 0.15-0.17 mm range for all heating scenarios (i)-(iv) described in sub-section 2.6. Tensile tests were conducted in triplicate at a constant rate of stretching of 0.25 mm/min with the lower clamp remaining at the initial position. The acquired data were converted into the stress-strain curves.

To determine modulus of elasticity and the yield stress using the data acquired in the tensile tests the following phenomenological Green's equation [27, 28] was employed,

$$\sigma_{xx} = Y \tanh\left(\frac{E}{Y} \varepsilon_{xx}\right) \quad (1)$$

Here σ_{xx} is the tensile stress, ε_{xx} is the tensile strain, E is Young's modulus, and Y is the yield stress.

It should be emphasized that Eq. (1) resembles not only the linear elastic behavior but also transition to perfect plasticity. Equation (1) was fitted to the experimental data so that the linear elastic region of the experimental data was approximated first with the coefficient of determination, R^2 of at least 0.99.

2.8 Microscopy

A scanning electron microscope (SEM), Jeol JSM-6320F, was used to prepare images of the nonwoven matrices of all unheated and heat-treated samples of PCL-PAN nonwoven samples. Before imaging, the samples were sputter-coated with 7.5 nm layer of platinum-palladium. The imaging was used to observe the effect of forced air convection on the sample micro-structure, and in particular, on the fiber conglutination, and to link these observations with the measured mechanical properties.

3. Theoretical Model of Transient Heat Transfer in Nonwoven Samples

Consider forced convection of hot gas through an infinitely wide plug of porous medium of length L sketched in Fig. 5. Similarly to [13], the transient one-dimensional approximation of the thermal balance equation in the present case reads

$$\sigma \frac{\partial T}{\partial t} + \frac{\phi \rho_g c_{p,g} U_g}{(1-\phi) \rho_s c_{p,s}} \frac{\partial T}{\partial x} = \alpha \frac{\partial^2 T}{\partial x^2} \quad (2)$$

where $T(x,t)$ is the temperature distribution in the porous medium, t is time, x is the longitudinal coordinate, U_g is the gas velocity in front of the the nonwoven, and σ is related to the specific heat capacities of the porous medium and gas as

$$\sigma = \frac{\phi \rho_g c_{p,g} + (1-\phi) \rho_s c_{p,s}}{(1-\phi) \rho_s c_{p,s}} \approx 1 \quad (3)$$

Here $c_{p,g}$ is the specific heat capacity of the gas flowing through the nonwoven, $c_{p,s}$ is the specific heat capacity of the porous medium, ϕ is the porosity, ρ_g is the gas density, and ρ_s is the porous medium density.

Equation (2) implies that locally the porous medium and gas possess the same temperature T . This assumption is justified by the high porosity of the samples used and the fiber thinness. In Eq. (2) α is the effective thermal diffusivity of the porous medium and gas

$$\alpha = \frac{\phi k_g + (1-\phi) k_s}{(1-\phi) \rho_s c_{p,s}} \approx \alpha_s \quad (4)$$

Here k_g is the thermal conductivity of gas, k_s is the thermal conductivity of the porous medium, and α_s is the thermal diffusivity of the porous medium.

According to the values of the physical parameters involved, Eq. (4) yields the effective thermal diffusivity approximately equal to the thermal diffusivity of the fibrous samples. It should be emphasized that the estimates using Eqs. (3) and (4) are based on the fact that the gas density is much lower than that of the material which comprises the porous medium skeleton (either PET, or PCL-PAN).

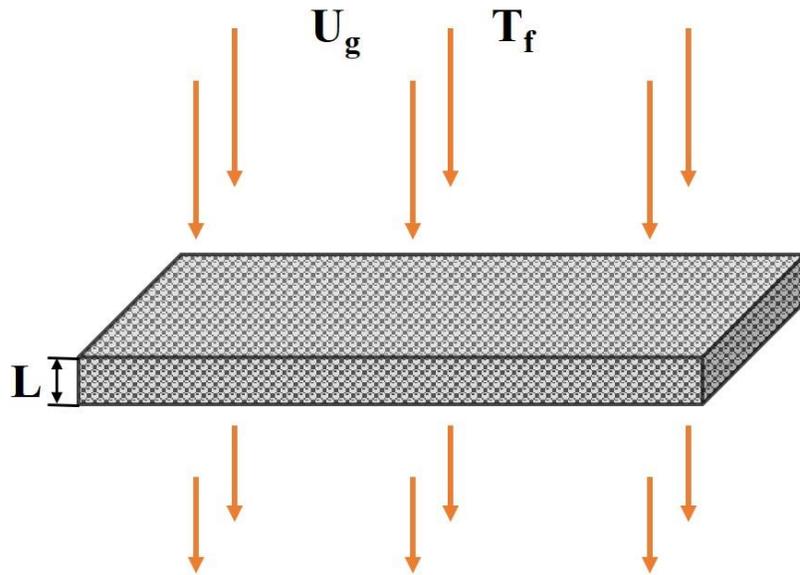


Figure 5. Forced convection of hot air through a porous nonwoven sample.

The porous medium is subjected to the inlet gas temperature T_f at the front side, and practically loses no heat by conduction to air behind its rear side. Accordingly, the boundary conditions for Eq. (2) read

$$T(0, t) = T_f \quad (t > 0) \quad (5)$$

$$\left. \frac{\partial T}{\partial x} \right|_{x=L} = 0 \quad (t > 0) \quad (6)$$

The initial temperature of the porous medium T_i is known, which results in the following initial condition for Eq. (2)

$$T(x, 0) = T_i \quad (t = 0) \quad (7)$$

The problem (2) with the boundary and initial conditions (5)-(7) is solved by the standard methods of the mathematical physics [29, 30]. In particular, using the substitution [29]

$$\theta(\bar{x}, \bar{t}) = \varphi(\bar{x}, \bar{t}) \exp\left(\frac{Pe_{eff}}{2} \bar{x} - \frac{Pe_{eff}^2}{4\sigma} \bar{t}\right) \quad (8)$$

and then the variables separation method [29, 30], one arrives at the following solution

$$\theta(\bar{x}, \bar{t}) = \frac{T_f - T(\bar{x}, \bar{t})}{T_f - T_i} = \varphi(\bar{x}, \bar{t}) \exp\left[\frac{Pe_{eff}}{2} \bar{x} - \frac{Pe_{eff}^2}{4\sigma} \bar{t}\right] \quad (9)$$

where

$$\varphi(\bar{x}, \bar{t}) = \sum_{n=1}^{\infty} A_n \exp\left[-\frac{\lambda_n^2}{\sigma} \bar{t}\right] \sin[\lambda_n \bar{x}] \quad (10)$$

In Eqs. (9) and (10) the factors in the series are given by the following expression

$$A_n = \frac{4\lambda_n}{\left(\frac{1}{2} - \frac{\sin[2\lambda_n]}{4\lambda_n}\right)(4\lambda_n^2 + Pe_{eff}^2)} \quad (11)$$

where the spectrum is found from the equation

$$\lambda_n = -\frac{\text{Pe}_{\text{eff}}}{2} \tan[\lambda_n] \quad (12)$$

and the effective Peclet number is defined as

$$\text{Pe}_{\text{eff}} = \frac{U_g L}{\alpha_s} \frac{\phi \rho_g c_{p,g}}{(1-\phi) \rho_s c_{p,s}} \quad (13)$$

In Eqs. (9) and (10) the dimensionless coordinate and time are defined, respectively, as

$$\bar{x} = \frac{x}{L}, \quad \bar{t} = \frac{t\alpha}{L^2} \quad (14)$$

4. Results and Discussion

4.1 Transient Temperature of Nonwoven Samples

Transient heating tests were conducted with PET nonwoven mats in four trials with the inlet air velocity, U_g , ranging from 0.69 m/s to 1.01 m/s, and the inlet air temperature of approximately 50 °C. In each case the initial temperature of the sample, T_i , and the measured inlet temperature, T_f , were recorded.

The experimentally measured transient temperature profiles acquired at $x = 2$ mm are compared to the theoretical solution given by Eqs. (9)-(14) in Fig. 6. The initial temperature of the samples, T_i , was taken as the temperature corresponding to the first data point recorded by the data logging device. The values of porosity ϕ were established using the method exposed in Fig. 1.

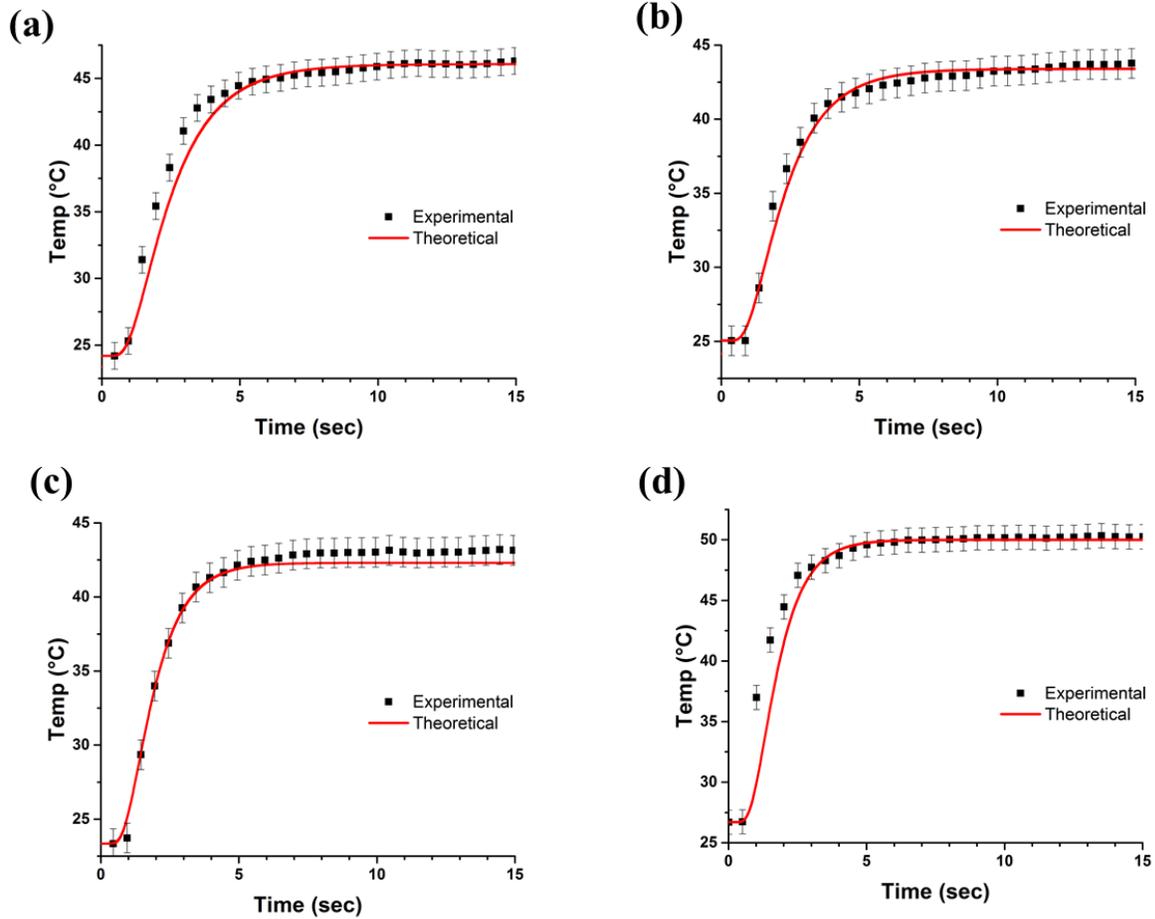


Figure 6. Experimentally measured transient temperature profiles in forced air convective heating of PET nonwoven mats for different values of the inlet velocity, U_g , the inlet air temperature, T_f , and the initial temperature T_i . Experimental data are shown by symbols, the theoretical predictions of Eqs. (9)-(14) are shown by red lines. Here (a) corresponds to heating a nonwoven mat with $U_g= 0.70$ m/s, $T_f= 46.1$ °C, and $T_i= 24.2$ °C, (b) corresponds to heating a nonwoven mat with $U_g= 0.76$ m/s, $T_f= 43.4$ °C, and $T_i= 25.1$ °C, (c) corresponds to heating a nonwoven mat with $U_g= 0.90$ m/s, $T_f= 46.6$ °C, and $T_i= 27.1$ °C, and (d) corresponds to $U_g= 1.01$ m/s, $T_f= 50.0$ °C, and $T_i= 26.7$ °C.

Figure 6 reveals a remarkable agreement of the theory with the experimental data. Some small discrepancy in Fig. 6c can be attributed to the above-mentioned experimental inaccuracy in the value of the inlet air temperature, T_f (while air was overheated). In addition to this discrepancy, the experimental data points shown in Figs. 6a-6c involve a slight time offset due to the delay in recording of the air temperature introduced by the thermocouple.

4.2 Effect of Forced Convective Heating on Mechanical Properties of Nonwovens

Tensile tests were conducted on the unheated and heat-treated PCL-PAN nonwoven samples. Fibrous materials can be significantly non-uniform, even if samples are taken from the same batch. Therefore, repeatable trials with similar samples are required to assess repeatability of the results and to determine the mean value of Young's modulus and deviations from the mean. The resulting stress-strain curves for all cases are shown in Fig. 7 with all separate trials listed in the insets. This figure also includes the results for the average modulus of elasticity, E , with the standard deviation found using fitting of the phenomenological Eq. (1).

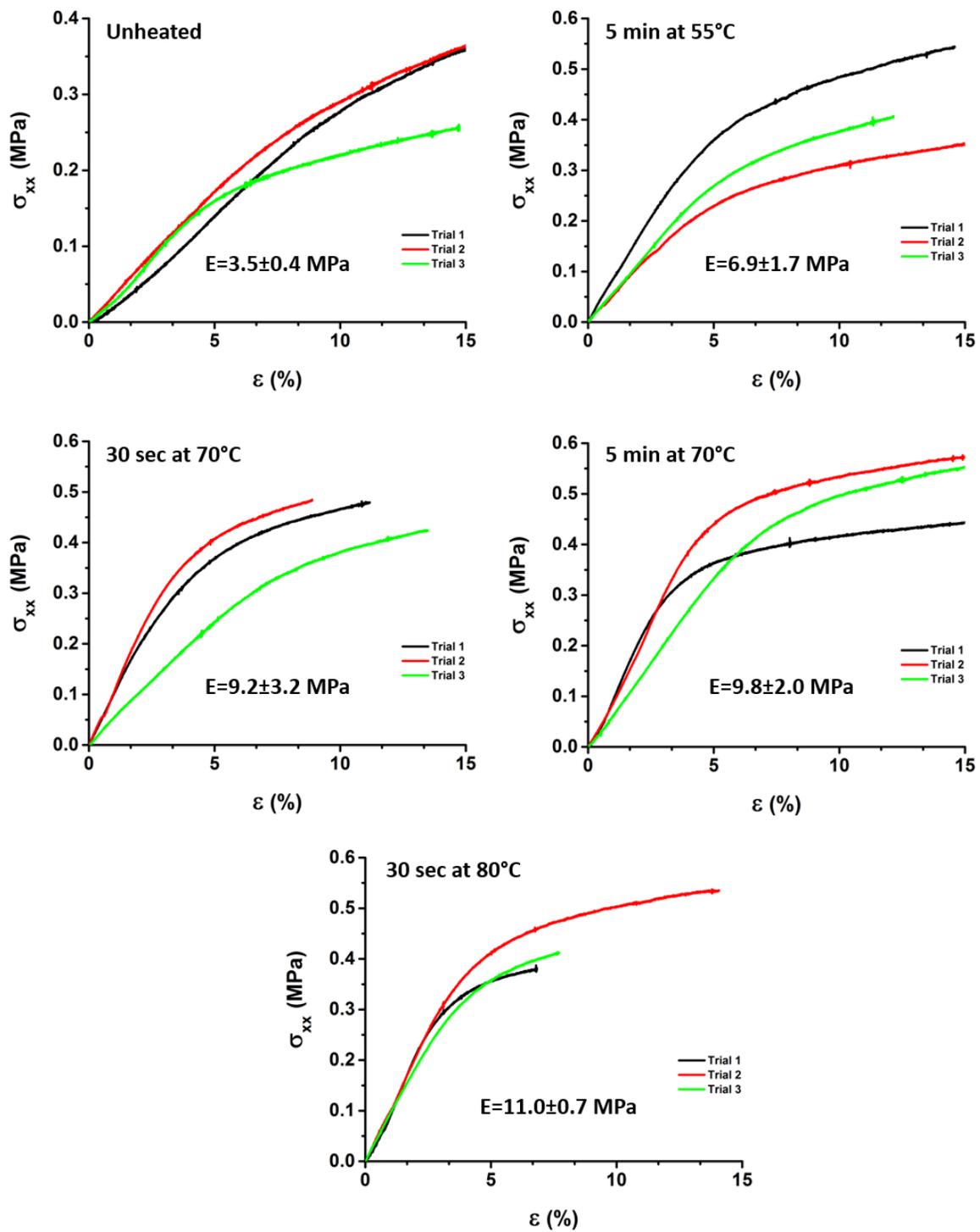


Figure 7. Stress-strain curves for the unheated and heat-treated PCL-PAN nonwoven samples.

The most noticeable change in the modulus of elasticity was due to heating at a temperature of 70 °C for 30 s or 5 min and 80 °C for 30 s. Both temperatures are above the melting point of PCL of 56-65 °C. This shows that heating up to temperatures past the melting point of PCL nanofibers would increase the modulus of elasticity of the PCL-PAN nonwoven up to a maximum value close in both cases of 70 °C or 80 °C (taking into account the standard deviation for the case of heating at 70 °C). Heating below the melting point of PCL, as for example, in the case of 55 °C for 5 min in Fig. 7, did not increase the modulus of elasticity to that extent. This was probably caused by an incomplete melting of PCL fibers in the nonwoven matrix. As mentioned previously, the melting point of PCL is around 56-65 °C. At 55 °C PCL melting only begins and thus PCL fibers do not conglutinate the PAM matrix sufficiently. Furthermore, the comparison of the results for heating PCL-PAN samples up to 70 °C for 30 s or 5 min, reveals no significant difference in the modulus of elasticity achieved. This shows that the most effective practical strategy for increasing sample stiffness would be the shortest forced air heating up to a temperature sufficient for melting of the binding PCL fibers, while heating for a longer period of time, or to higher temperatures would not necessarily increase the modulus of elasticity. Moreover, it can be detrimental due to the thermal degradation of polymers.

To further elucidate the effect of forced convective heating on the modulus of elasticity on the micro-mechanical level, SEM images of all unheated and heat-treated PCL-PAN nonwoven mats were obtained (cf. Fig. 8).

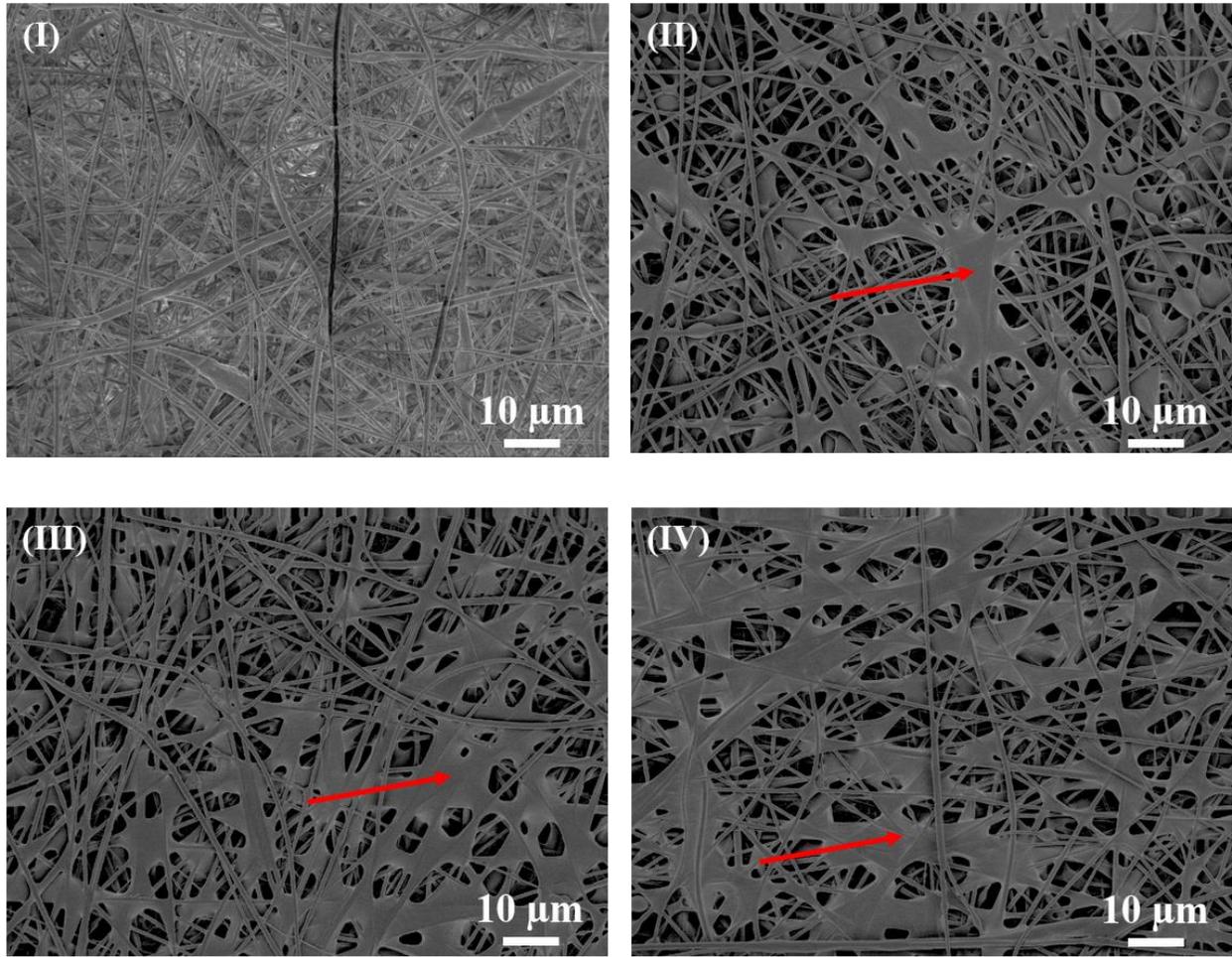


Figure 8. SEM images of the PCL-PAN nonwovens contrasting the unheated and heat-treated samples. Image (I) shows the unheated sample, while image (II) shows the samples heat-treated at 55 °C for 5 min; image (III) shows the samples heat-treated at 70 °C for 30 s, and image (IV) shows the samples heat-treated at 80 °C for 30 s. Red arrows point at conglutination locations in the heated-treated samples due to PCL melting. It should be emphasized that it is impossible to distinguish PAN and PCL fibers in the unheated nonwoven samples. Note that the dark line in the middle of panel (I) appeared due to charging of the sample during imaging and is an artifact.

In particular, these SEM images show an unheated PCL-PAN nonwoven matrix with no molten fibers. When samples were heated up, the PCL fibers were molten and bound the adjacent PAN fibers (which were not molten, since the melting temperature of PAN is 369 °C). This binding increased the modulus of elasticity and was revealed by the results in Fig. 7. It should be emphasized that PCL fibers were fully molten and conglutinated the nonwoven matrix when heated up to 70 °C or 80 °C. As the results for 55 °C show, the PCL fibers were not fully molten, while the modulus of elasticity increased. This increase was not large, since the melting temperature range of PCL was not fully covered in distinction from the 70 °C or 80 °C range. This confirms the link of the heating history with the achieved nonwoven stiffness established using the tensile test in Fig. 7.

5. Conclusion

Experiments with convective heating of fibrous porous media by forced filtration of hot air were conducted with Poly(ethylene terephthalate) (PET) nonwovens. They revealed the transient thermal history which was used to validate the transient one-dimensional single-temperature model also developed in the present work. The agreement of the experimental data with the theoretical predictions was very good, which allows using the solution obtained to predict different heating scenarios theoretically. These scenarios are of significant interest in relation to the stiffening of compound nonwovens by means of the forced filtration of hot air.

To explore experimentally the link of the thermal history to the achieved stiffening, model nonwoven mats comprised of electrospun Polycaprolactone (PCL) and Polyacrylonitrile (PAN) were heated up by means of forced air convection following different scenarios. It was observed

that heating up to higher temperatures above the melting point of the binding fibers (PCL), significantly increases the modulus of elasticity of the compound nonwovens. It was shown that heating up to different temperatures above the PCL melting point would make no difference in terms of increasing of the modulus of elasticity, and neither would heating for a longer period of time. SEM observations revealed micromechanical details of compound nonwoven conglutination by molten binder fibers.

The results show that the most effective practical strategy for stiffening compound nonwovens would be the shortest forced air heating up to the lowest temperature sufficient for melting of the binding fibers. Thermal scenarios for finding this strategy in any particular case can be provided using the theoretical model developed in the present work, which was fully validated by the present experimental data.

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