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EFFICIENCY OF ELECTRET POLYCARBONATE NONWOVENS IN RESPIRATORY PROTECTION AGAINST NANOPARTICLES

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Abstract:

Toxicological research on the influence of noxious nanoparticles on human health indicates the need to develop efficient protective devices. In particular, this concerns respiratory protective equipment employing filtration nonwovens. This paper presents a methodology for the improvement of the filtration efficiency of electret nonwovens against nanoparticles by enriching amorphous polycarbonate (PC) with additives of different electrostatic potentials. We introduced perlite granules (positive charge) and amber granules (negative charge) to the polymer stream in melt-blown technology. Filtration efficiency was assessed by a standard method using paraffin oil and sodium chloride aerosol, as well as by a non-standard method using NaCl nanoparticles. The experiments showed that strengthening the effects of electrostatic forces by the introduction of modifiers is a promising approach to improving the efficiency of electret nonwovens against nanoparticles.

Keywords:

filtration efficiency, polycarbonate nonwoven, melt-blown process, nanoparticles

1. Introduction

For the past few years, nanotechnology has been observed to rapidly develop worldwide, leading to mass production of a variety of nanomaterials and, by the same token, to increased exposure of workers and consumers to them. This development has also increased social and public concerns over the safety of nanomaterials and related technologies. The importance of research and innovation for society is emphasized by the European Commission in its recently published communication entitled "EUROPA HORIZON 2020," dealing with intelligent development—development of a knowledge- and innovation-based economy with a focus on strengthening the role of knowledge and innovation as the driving forces of the growth of the European Union.[1]

New technological applications should not only be safe in themselves but also offer significant improvements in terms of human health and environmental protection. Thus, because of the fast growth of nanomaterial production and the progress of nanomaterial engineering and nanotechnology, safety aspects must be fully explored.

Recent toxicological studies have shown that nanoparticles released by technological processes may be dangerous to humans [1-4] as a result of inhalation.[5] Consequently, of great importance in prophylaxis is the use of effective respiratory protective equipment.

Currently, researchers are working on the nonwoven structures application with different filtering layers geometrics. [6-8] Numerous efforts are also being made to modify polymer filtration materials to improve their efficiency in terms of preventing nanoaerosol penetration.[9,13] Another approach to improve the effectiveness of filtration materials involves strengthening of the electrostatic attraction between the fibers and noxious aerosol particles. Studies [14-18] show that the electrostatic activation of fibers significantly improves the efficiency of filtration without increasing air flow resistance. The most common materials used in respiratory protective devices are manufactured using the melt-blown technology.

One of the mechanisms to improve the particle capture capacity by filtration materials is their electrostatic activation [15,19] whereby the charged particles are attracted to or repelled from the fibers according to the direction of the electric field, while the particles are electrically neutral, they are polarized and move in accordance with the electric field gradient.[20] Today, we can distinguish multiple activation methods that are widely described in the literature, for example,[18] electrospinning, the production of ultra-thin fibers from the solution with the electric field[21]; charging using the triboelectric effect that transfers electric charges during manufacture of needle felt[14,22]; and the corona discharge using the interaction of ionized gas. Electrets are typically produced by corona discharge, fiber surface modification by low-temperature plasma [23,24] or by the introduction of additives, such as fluorine or natural resin, to the structure of polymer fibers.[25]

From the point of view of respiratory protection, it is essential that the electret effect be constant over time. Unfortunately, numerous reports have shown that the particles of liquid aerosols deposited within electrets lead to loss of their filtration effectiveness over time, which is particularly dangerous if such respiratory protective equipment is used for protection against nanoaerosols.[26,27]

The nonwovens used in respiratory protective equipment are made of polypropylene (PP), mainly due to its good processing properties and low price. However, PP has been found to perform poorly in terms of electrostatic properties and, in particular, the durability of electret effects, which undermines its usability for protection against harmful nanoparticles. A study performed by Brochocka et al.[28] has shown that electret melt-blown PP fibers may be modified to improve their filtration by the addition of modifiers with different electrostatic potentials at the fiber-forming step. Modification with perlite granules (positive potential) and positive corona discharge increased the filtration effectiveness of electrets to a greater degree than modification with amber granules (negative potential) combined with positive corona discharge.

Thus, it is necessary to seek new methods for the improvement of electret filtration effectiveness to ensure the safety of the users of protective equipment in case the assumed protection level of that equipment is compromised. One of the ways of attaining this goal is to use another thermoplastic polymer (with appropriate modifications) in place of the commonly used semicrystalline polypropylene.

From the point of view of materials used for respiratory protection, amorphous polycarbonate (PC) exhibits some valuable properties, such as considerable thermal resistance, resistance to sterilization conditions, good shape stability, good electroinsulating properties, biological inertness, ease of recycling, and ease of fiber formation.[29]

Currently, in the European market, there are not many filtering materials produced by the melt-blown technique that would be characterized by a good ability to maintain electrostatic charges in time.[30] It is very difficult to get a filtering material for manufacturing reusable filtering half masks for respiratory protection that would display both high filtration efficiency and low breathing resistance.

Thus, this work presents technological research aimed at developing appropriate modifications of electret melt-blown PC nonwovens. The objective was to produce filtering materials from PC and to improve the effectiveness of electret melt-blown PC nonwovens in respect of nanoparticles using modifiers with different electrostatic potentials.

2. Experimental

2.1 Materials

The raw material for the production of filtering nonwovens was PC LEXAN 144 R granulate with a density of 1.20 g/cm^3 (from General Electric Company, USA). The properties of this PC material obtained from the manufacturer are given in Table 1. Listed parameters such as melting point and melting flow index (MFI) are crucial for melt-blown process.

Modifiers

Two granulates were used in the process of modification:

- natural resin (amber) with negative potential (from EDAN, Poland),
- volcanic rock (perlite) with positive potential (from TERMOFOR-BEŁCHATÓW, Poland).

A Quanta F 200 scanning electron microscope (SEM) with 500× magnification was used to examine the granulates in terms of granule shape and size (see Figures 1 and 2).

2.2 Processing equipment

Technological work was conducted at the experimental stand of the Central Institute for Labour Protection–National Research Institute (CIOP–PIB), which was described in detail by Brochocka and Majchrzycka (see Figure 3).[25]

Predried PC granulate was fed from the hopper to the heated extruder cylinder. It was brought to appropriate viscosity before extrusion from the fiber-forming die. Compressed air was passed from the regulator to the heat exchange unit, where it was dried and heated up to the desirable temperature. Subsequently, it was supplied to the fiber-forming die, and, upon exiting, it blew the polymer streams into elementary fibers, which were deposited on the collector to form a cohesive, porous nonwoven fabric. The nonwoven fabric production setup had certain control points to adjust the desired technological parameters. Table 2 gives the parameters used for the formation of PC nonwovens.

Given parameters were chosen as they have the most effect on the shape of the obtained fibers—diameter. There were no decomposition effects of PC observed during the process at given temperatures. The structural characteristics of the generated PC nonwovens before the modification process and after the process are presented in Table 3.

The modifiers were added in the form of granules at the fiber-forming step in the amount of 5% of PC weight, according to patent, [30] in a manner described at in detail by by Brochocka and Majchrzycka.[25] Figure 4 presents the PC nonwoven without modifiers, and Figures 5 and 6 show the distribution of modifiers in the filtering nonwoven.

The modifier located on a rotatable dispenser was supplied to a line connecting the injector fed with compressed air. Produced aerosol was administered directly into the stream of semiliquid polymer. The mixture of fibers and modifier particles were collected on the surface of the collecting device to form a dense nonwoven fleece with modifier uniformly distributed.

Table 1. Characteristics of the PC according to the manufacturer

Polymer type	Melting point (0C)	MFI (g/10 min)	Degree of crystallinity (%)
Polycarbonate LEXAN type 144 R	280–310	15.40	Amorphous

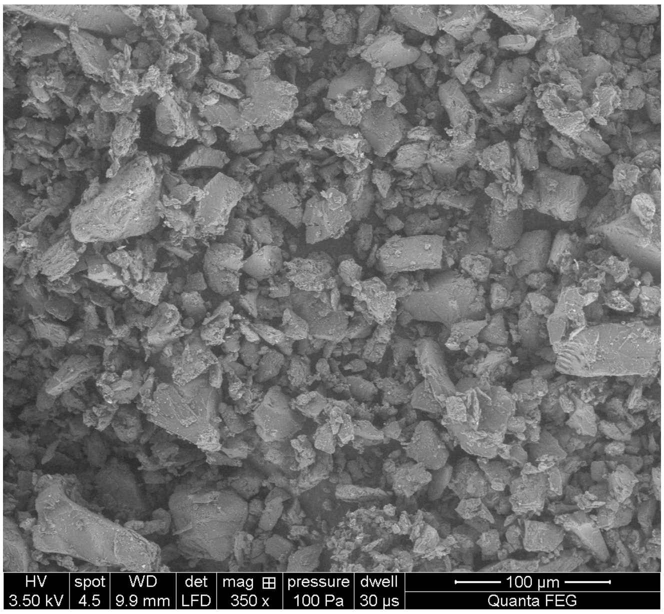

Figure 1. SEM image of amber granulate surface

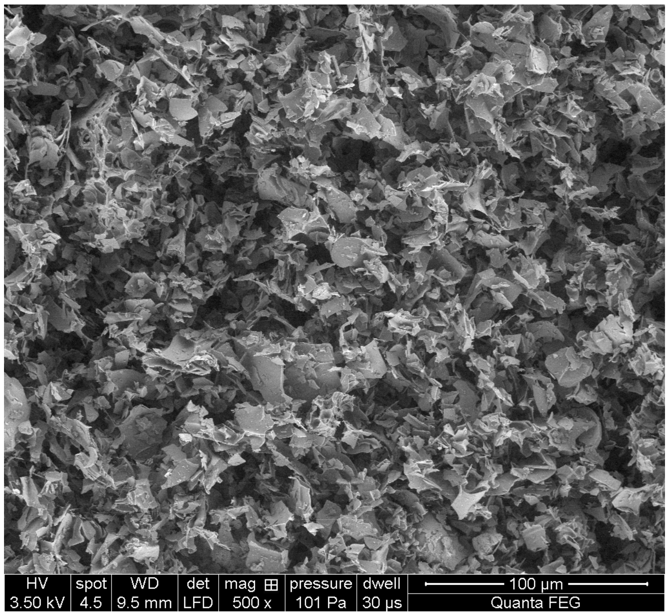

Figure 2. SEM image of perlite granulate surface

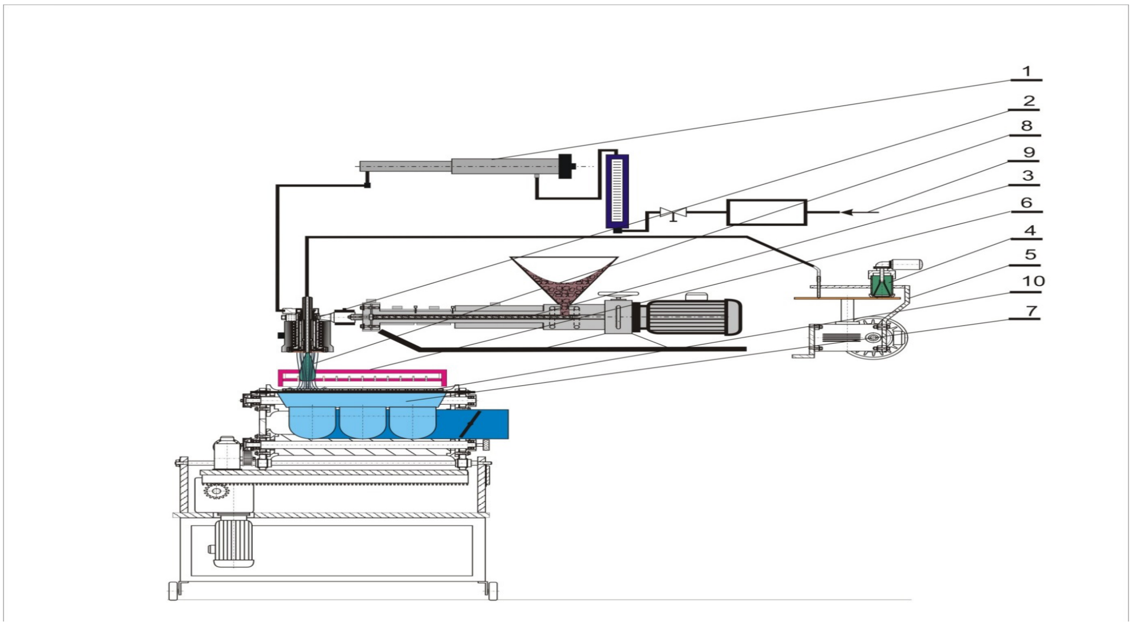

Figure 3. Setup for producing melt-blown filtering materials with modifiers in the form of powders: (1) air heater, (2) fiber-forming die, (3) extruder, (4) modifiers, (5)dispenser of modifiers, (6) electrostatic activator, (7) air suction, (8) fibers, (9) compressed air inlet, (10) collector

Table 2. Technological parameters of melt-blown production

Technological parameters of the process	**PC**
Temperature of the first zone of the extruder, ºC	315
Temperature of the second zone of the extruder, ºC	340
Air temperature, ºC	400
Nozzle temperature, ºC	370
Air flow rate, m^3/h	3.2
Polymer flow rate, g/min	12.0
Nozzles to collector distance, cm	13.0
Collector speed, m/s	0.3
Supply voltage heating elements nozzle, V	190

Table 3. Structural parameters nonwoven PC without and with modifier

Type of nonwoven	Basis weight (g/m²)	Thickness (mm)	Min fiber diameter (μm)	Max fiber diameter (μm)	Mean fiber diameter (μm)	Standard deviation (mm)
PC without modifier	90 ± 5	1.46	0.26	3.6	1.04	0.61
PC with modifier	95 ± 5	1.50	0.20	3.8	1.11	0.66

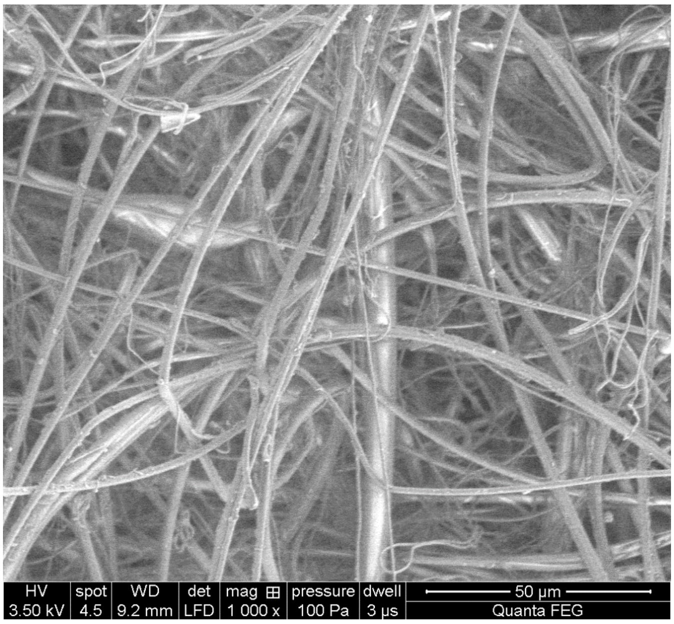

Figure 4. Nonwoven polycarbonate (PC) without the modifier

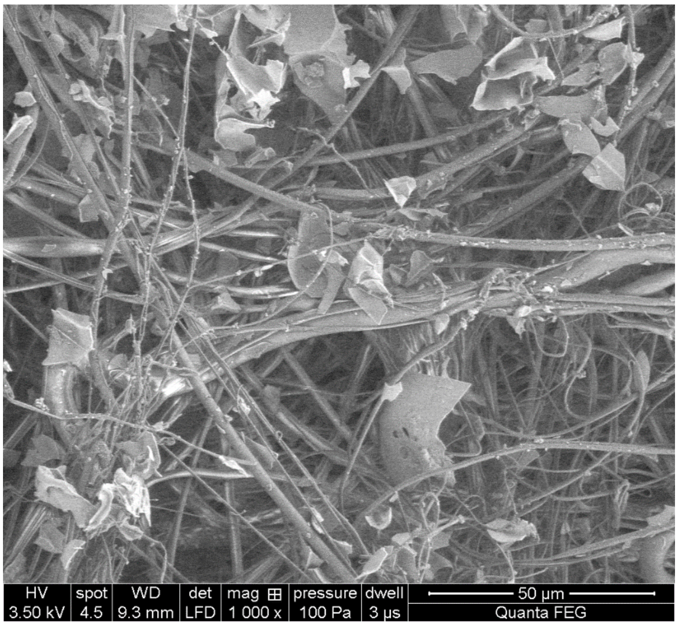

Figure 6. Nonwoven polycarbonate (PC) with perlite granules

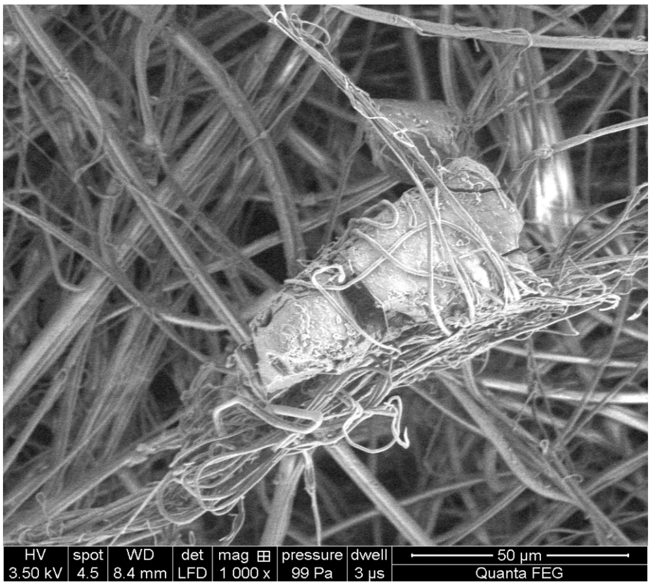

Figure 5. Nonwoven polycarbonate (PC) with amber granules

Figure 7 shows how the modifier was distributed using a device introducing modifier into a stream of the polymer. The basic idea of this method is to supply a fiber-channel head with a screw that allows mixing and pressing the polymer melt and modifiers that are inserted continuously to the head channel.

A corona discharge device was used to impart electret properties to the PC nonwovens with and without modifiers. It was equipped with positive multipoint electrodes placed near the nonwoven collector and a negative counter electrode placed under the take-up screen. The total charging voltage was 30 kV, which resulted in a current of 300 ± 50 μA. Such a setup makes it possible to control the flow of charges, which do not become dispersed.

2.3 Testing methods

The efficiency of the filtering materials was established by measuring particle penetration, that is, the number of particles that were not retained by these materials. Particle penetration and air flow (breathing) resistance were examined using the methods of respiratory protective equipment evaluation specified in the relevant standards [29,30] as well as using the non-standard method described further on. The standard measurements involved two model aerosols: sodium chloride and paraffin oil mist.

In the designed (not standard) test method, sodium chloride (NaCl) was used in the form of a suspension generated from a 0.1% water solution by means of a Collison atomizer. The nanoaerosol was passed through a desiccator and ion neutralizer and fed into the chamber holding a tested nonwoven sample. Figure 8 presents the size distribution of the nanoaerosol generated by the Collison atomizer.

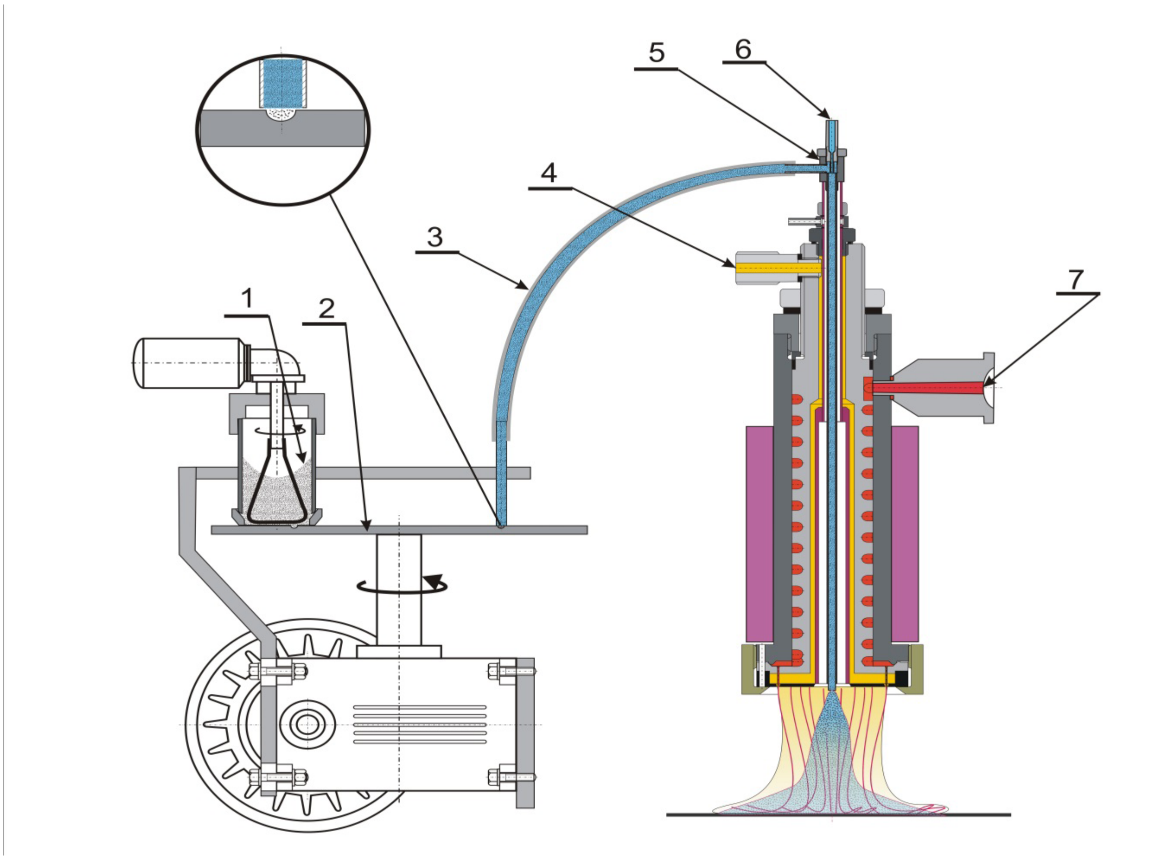

Figure 7. Head unit and fiber-insertion device of powdery modifier. (1) Modifier, (2) dispenser, (3) connecting tube, (4) hot air, (5) injector, (6) compressed air, and (7) melted polymer.

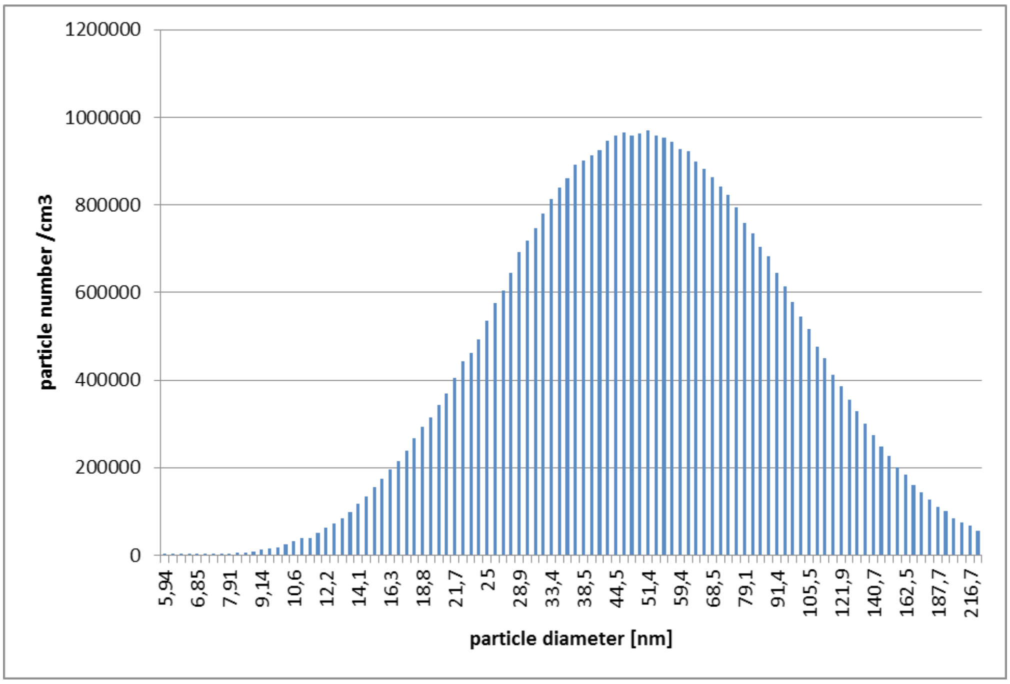

Figure 8. Dimensional distribution of NaCl nanoaerosol

The standard method of NaCl penetration test is based on the flame photometry, and the non-standard method is based on condensation particle counter with electrostatic classifier. The first method is a mass method and gives information only on total penetration value, and the second one gives the information on penetration of each particle size class but do not give a comprehensive value.

Figure 9 a and b shows an atomic force microscopic image of the studied nanoaerosol, which enables the determination of the particle shape and size. It is an example of the series of the pictures that were taken to assess the physical properties of the nanoaerosols. The sample surfaces were examined by the NanoScope 3D (veeco) equipped with extremely sharp hydrophobic probe using tapping mode.

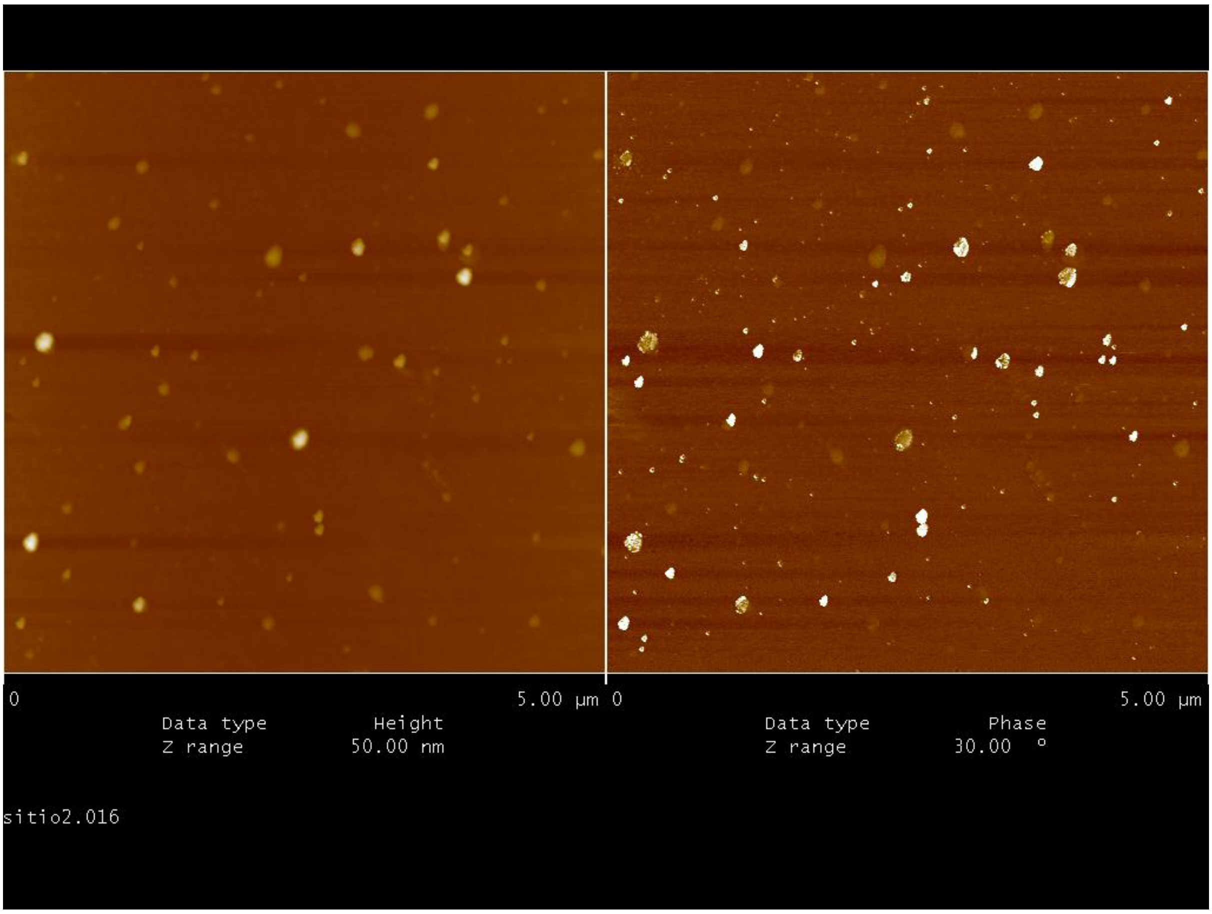

Figure 9. (a) NaCl nanoaerosols: 2D view

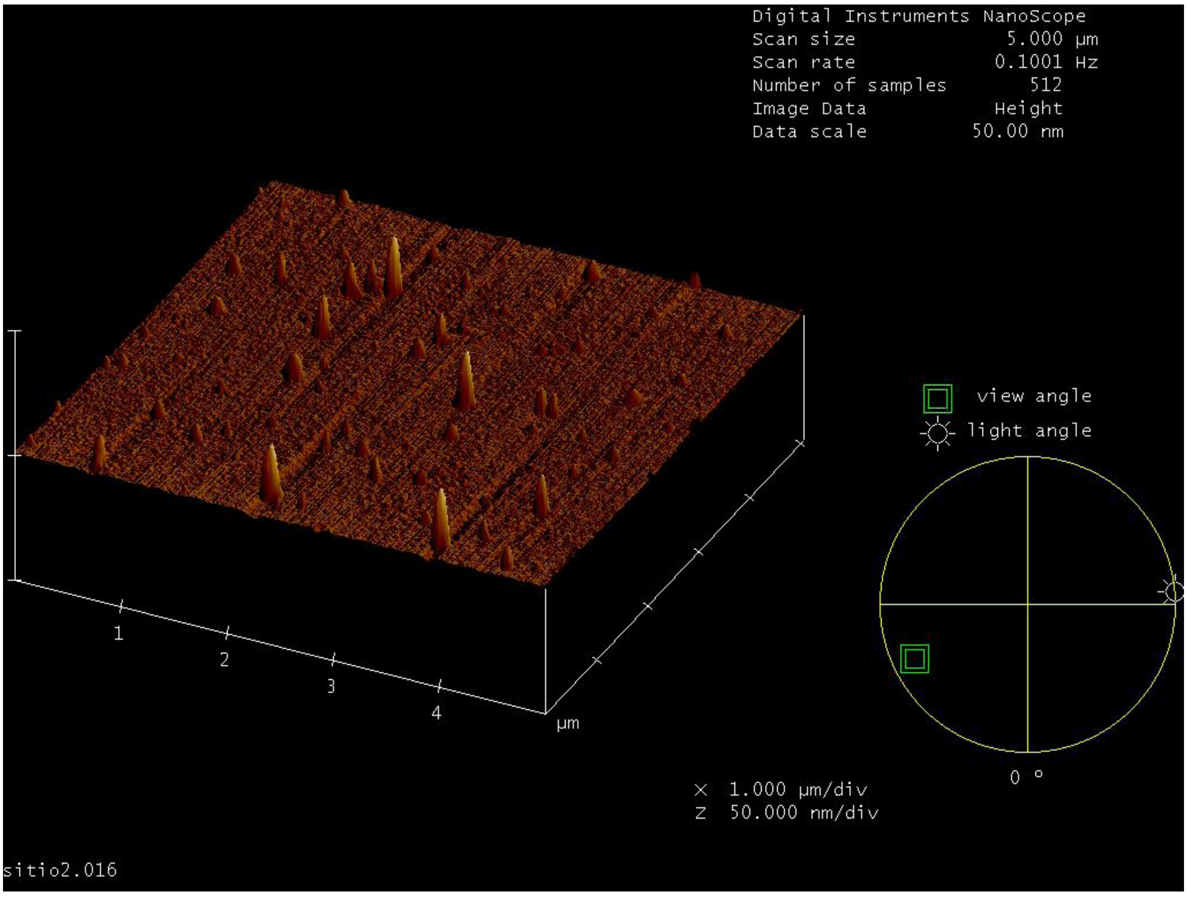

Figure 9. (b) NaCl nanoaerosols: 3D view

The NaCl nanoparticles used in the study were cubical with a similar fractal dimension (3). The NaCl aerosol was used for the tests as nonhazardous. In accordance with previous tests, the shape of particles with nanometric dimensions is not significant determinant for filter penetration in accordance with classical filtration theory.[10] They were neutralized by means of an ion neutralizer and exhibited a dielectric constant of 5.9.

The penetration of nanoparticles through the filtering nonwovens was examined using a 3800 electrostatic classifier and a 3775 condensation nanoparticle counter, both from TSI. The measurement range of the setup was from 7 to 270 nm, with 90 size classes. The test time was set to 7 min so as to enable the calculation of the average penetration value for three cycles of 126 s each, including 15 s breaks between them for the purpose of resetting the electrostatic particle classifier. The tests were carried out at an aerosol flow of 5,400 L/h. The area of the tested samples was 0.01 m^2 and constant for all measurements. The climatic conditions during the tests were 20 ± 5°C air temperature and 50 ± 20% relative air humidity.

A diagram of the experimental stand is presented in Figure 10.

The measurement results were statistically analyzed to confirm the effect of the introduced modifiers to the nonwoven fabrics on penetration of aerosols. This statistical analysis included

- calculation of mean values, variance, as well as standard deviations for 50–275 nm NaCl nanoparticle penetration;
- the Shapiro–Wilk test for normality of distribution;
- the Fisher–Snedecor test for comparison of variances and either the Student t-test (in the case of equal variances) or the Cochran–Cox test (in the case of different variances) for pairwise comparison of means.

3. Results and analysis

Table 4 shows the results for standard aerosol particle penetration, that is, for sodium chloride (mean particle diameter of 0.6 μm) and paraffin oil mist (mean particle diameter of 0.3 μm) determined in accordance with the methods commonly used for the evaluation and classification of equipment for respiratory protection against harmful aerosols such as dust, smoke, and mist. Figures 11 and 12 present the results for NaCl nanoparticle penetration through the modified and unmodified nonwovens, including classification into nanoparticle size classes, while Table 5 gives mean results for particles ranging from 50 to 275 nm.

The results presented in Table 4 confirm that the use of corona discharge during melt-blown fiber formation significantly increases the efficiency of removing pollutant particles from the stream of flowing air. The number of particles penetrating

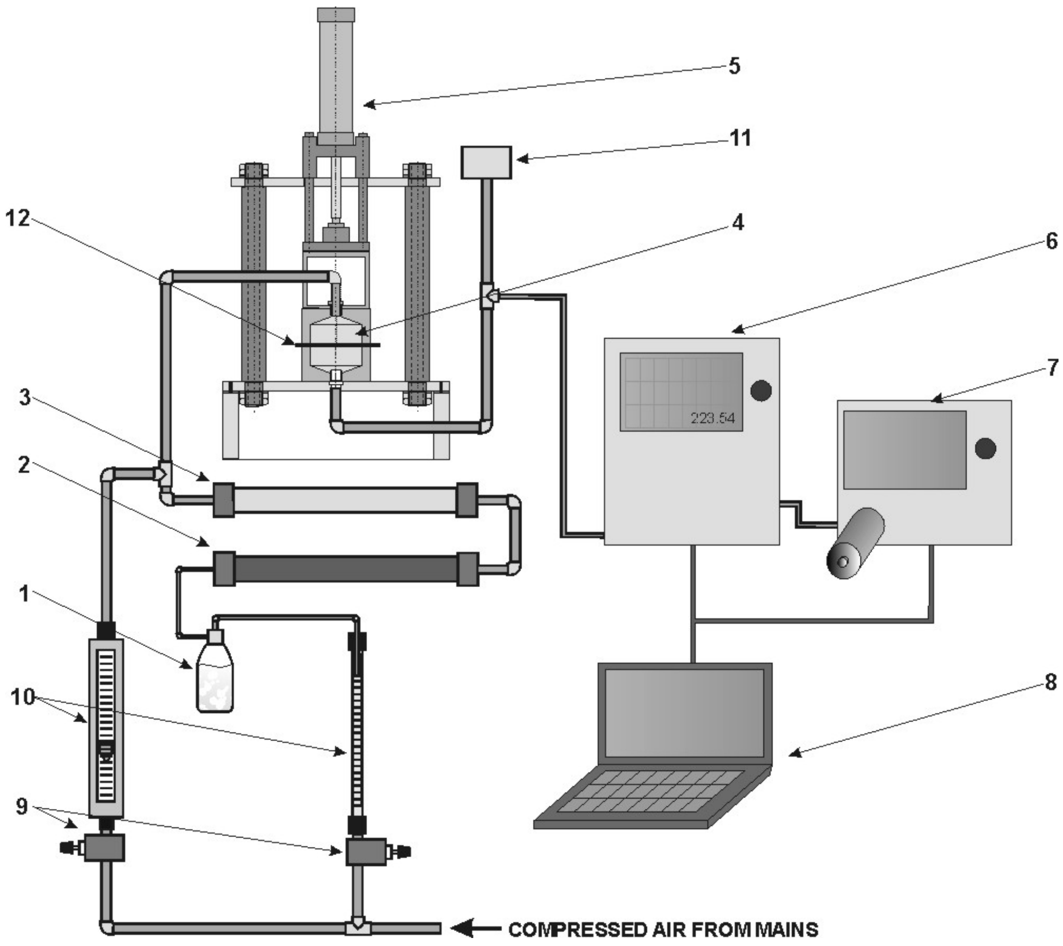

Figure 10. Setup for testing nanoparticle penetration through the filtering materials and elements: (1) nanoaerosol generator, (2) desiccant, (3) electrostatic charge neutralizer, (4) testing chamber, (5) pneumatic actuator, (6) electrostatic particle classifier, (7) condensation nanoparticle counter, (8) computer, (9) compressed air valve, (10) flow meter, (11) high-performance industrial filter, (12) tested sample

Table 4. Filtering parameters of nonwoven fabrics determined by standard methods [29,30].

Nonwoven type	Mean sodium chloride aerosol penetration (%)	Mean paraffin oil mist aerosol penetration (%)	Mean air resistance (Pa)
Polycarbonate nonwoven, charged	0.686	2.624	238.9
Polycarbonate nonwoven, uncharged	7.601	11.475	197.5
Perlite-modified polypropylene nonwoven, charged	0.299	1.148	268.2
Perlite-modified polypropylene nonwoven, uncharged	3.628	6.188	235.7
Amber-modified polycarbonate nonwoven, charged	0.539	3.025	215.1
Amber-modified polycarbonate nonwoven, uncharged	4.585	10.825	207.3

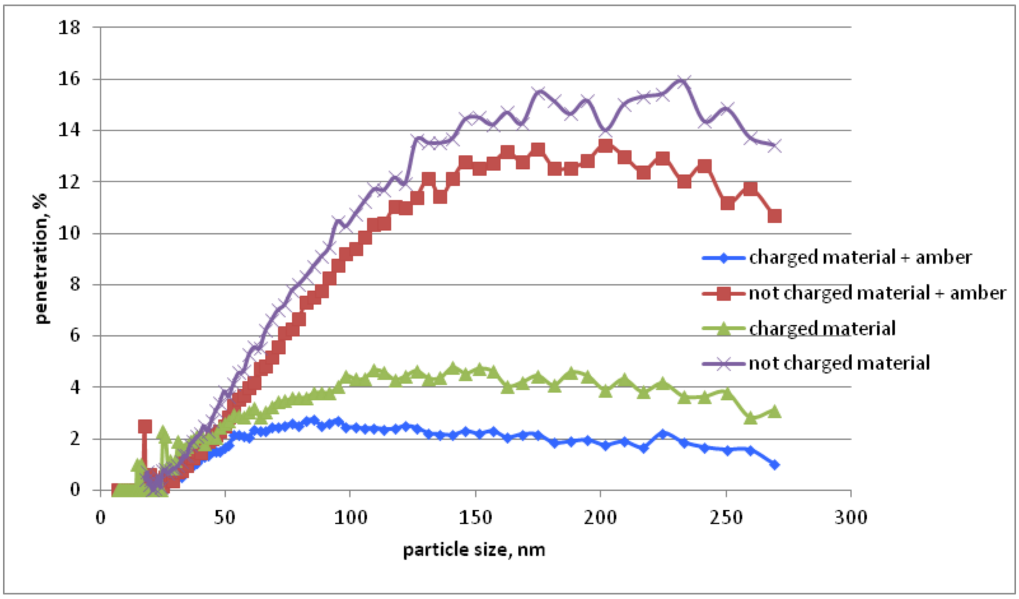

Figure 11. NaCl nanoparticles penetration results by electret nonwovens PC with and without modifier in the form of amber granules

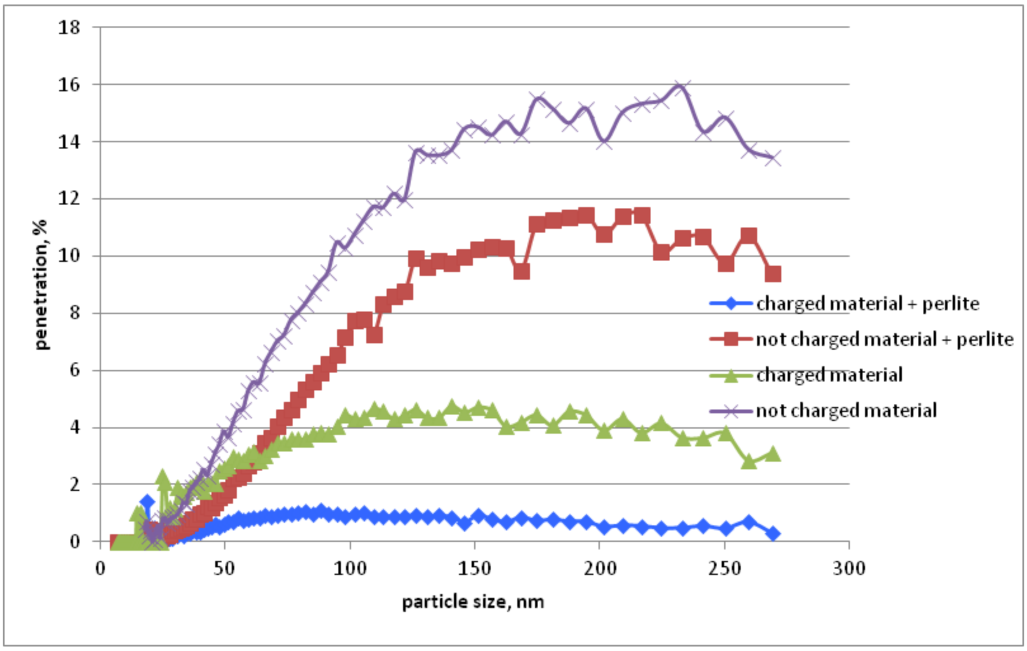

Figure 12. NaCl nanoparticles penetration results by electret nonwovens PC with and without modifier in the form of perlite granules

through the filtering electret nonwovens is much lower than that in the case of the non-electret variants, for both modified and unmodified nonwovens. The standard measurements involve two types of particles, that is, solid NaCl particles and liquid paraffin particles, and their penetration rates differ for the same nonwoven variants. The different particle sizes in these aerosols and their electrostatic properties lead to differences between liquid particles (mists) and solid particles (dusts) in the mechanism of their deposition on fibers. At the same time, it should be emphasized that the use of modifiers with different electric potentials (perlite and amber) increased the efficiency of the electret nonwovens as compared to the unmodified nonwovens. The results obtained for NaCl nanoparticle penetration were quite different. Figures 11 and 12 present the relationship between penetration rate and nanoparticle size for the nonwovens modified with amber and perlite granules, respectively. In the case of the non-electret nonwovens, the introduction of modifiers did not lead to significant changes in the efficiency of nanoparticle deposition on PC fibers. Here, the prevalent mechanism of nanoparticle deposition involved forces of mechanical attraction, the effectiveness of which largely depends on pore size. One of the effects of the modification of nonwoven fabric is the reduction of pore sizes, which also has the effect on improving the filtration efficiency.[28] However, the use of modifiers in the electret nonwovens triggered the expected increase in their efficiency as compared to unmodified electret nonwovens. This effect depends on nanoparticle size and is most pronounced in the range of 50–200 nm. Owing to the fact that, according to standard,[32] protective respiratory filtration equipment is evaluated in respect of a certain mean particle size characteristic of a given aerosol type, mean penetration rates were also calculated for the nanoparticles (Tables 5 and 6). The results show that the best variant in terms of the lowest nanoparticle penetration rate is the electret nonwoven modified with perlite (positive potential). This is also confirmed by the plot of penetration rates for nanoparticles of different size (Figure 12).

The differences in nanoparticle penetration through particular nonwoven variants were analyzed to determine the statistically significant differences. The results of statistical analysis are presented in the form of a matrix in Table 6. The statistical tests were conducted at a statistical significance of $\alpha = 0.05$. The α level is the probability of rejecting the null hypothesis: modification of nonwoven fabrics improves filtration efficiency. If the α level is 0.05, then the conditional probability of a type I error, rejection of given hypothesis, is 5%.

Statistical analysis confirmed our expectations and the above-mentioned hypothesis that modification of nonwoven fabrics improves filtration efficiency. Irrespective of the type of modifier used, all the variants of melt-blown nonwovens displayed an air flow resistance (respiratory resistance) of 197–270 Pa (Table 4). This confirms that the studied melt-blown fiber modification method improves the efficiency of the filter material against nanoparticles, while only slightly affecting the respiratory discomfort of the user. At the same time, it should be stressed that the resistance levels reported in this study are commonly found in respiratory protective equipment of comparable efficiency, which have breathing resistance on the some level.[34,35]

4. Conclusions

Presented method for the production of electret melt-blown PC nonwoven with modification by the addition of modifiers with different electrostatic potentials to amorphous PC improves filtration efficiency (fewer particles penetrate through the filtration nonwoven). As a result of modification with perlite granules (positive potential) combined with positive corona discharge, the electret exhibited better filtration properties than in the case of amber granulate (negative potential) with positive corona discharge. A similar effect was obtained by introducing these modifiers to PP electret melt-blown nonwovens.[29] The use of modifiers with the same charge sign as corona discharge led to greater electrostatic potential. This effect does not depend on whether the polymer is of crystalline nature or not.

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Table 5. Mean NaCl nanoparticle penetration through the nonwoven fabrics with and without modifier

	Amber-modified PC nonwoven with electrostatic charges	Amber-modified PC nonwoven without electrostatic charges	Perlite-modified PC nonwoven with electrostatic charges	Perlite-modified PC nonwoven without electrostatic charges	PC nonwoven with electrostatic charges	PC nonwoven without electrostatic charges
Mean	2.19	10.88	0.80	7.60	3.88	11.09
Standard deviation	0.41	2.13	0.20	2.76	0.56	3.32
Variance	0.17	4.55	0.04	7.62	0.32	11.02

Table 6. List of statistical test results

B \ A	Amber-modified PC nonwoven with electrostatic charges	Amber-modified PC nonwoven without electrostatic charges	Perlite-modified PC nonwoven with electrostatic charges	Perlite-modified PC nonwoven without electrostatic charges	PC nonwoven with electrostatic charges	PC nonwoven without electrostatic charges
Amber-modified PC nonwoven with electrostatic charges						
Amber-modified PC nonwoven without electrostatic charges	Statistically significant differences mean A < mean B					
Perlite-modified PC nonwoven with electrostatic charges	Statistically significant differences mean A > mean B	Statistically significant differences mean A > mean B				
Perlite-modified PC nonwoven without electrostatic charges	Statistically significant differences mean A < mean B	No statistically significant differences mean A = mean B	Statistically significant differences mean A < mean B			
PC nonwoven with electrostatic charges	Statistically significant differences mean A < mean B	Statistically significant differences mean A > mean B	Statistically significant differences mean A < mean B	Statistically significant differences mean A > mean B		
PC nonwoven without electrostatic charges	Statistically significant differences mean A < mean B	No statistically significant differences mean A = mean B	Statistically significant differences mean A < mean B	No statistically significant differences mean A = mean B	Statistically significant differences mean A < mean B	

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