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Plasma Modification of Filter Nonwovens Used for the Protection of Respiratory Tracts

Abstract

The filter materials commonly used in micro-filtration processes consist of nonwoven fabrics made by melt-blowing. In order to improve the filterability of nonwovens, they are subjected to various modifications. This paper proposes the treatment of nonwovens with low-pressure cold plasma (LPCP) using process gases such as air, methane and hexamethylidisiloxane (HMDSO). The effectiveness of this treatment was assessed on the basis of the results of the penetration of nonwovens by NaCl aero-dispersion in a flux of air and flow resistance data. The effects of plasma on filter nonwovens, especially on their chemical structure and surface topography were evaluated by means of ATR-IR spectroscopy and SEM microscopy. The nonwoven filters obtained show a high effectiveness of filtration and do not increase the resistance of air flow.

Key words: filtration, polypropylene nonwoven, filter, plasma treatment, low-pressure cold plasma.

Introduction

Polypropylene (PP) nonwovens produced by melt-blowing and spun-bonding have found common use in the filtration of various media, such as gases (gas separation), aero-dispersion (aerosols) and liquids [1-4]. The popularity of these materials in filtration processes is due to their specific, very porous structure conditioned by the multi-layer fiber system in the fabric. This structure provides the proper parameters required in filtration, such as a high effectiveness, low resistance to air flow and long operation life of filters. In the field of health protection, PP nonwovens are used to make respiratory protective devices that clean the air i.e. removing hazardous aerosol solid particles (dust and smoke) and liquid aerosol (mist and fog). These types of aerosol, occurring in air in the work and life environment, pose serious hazards to health, particularly causing chronic diseases of the respiratory system. Therefore preventive measures are taken consisting, among others, in using effective filtering personal protection. Examinations of the filtration properties of PP nonwovens designed for protection [5] have shown that the way of depositing particles of aerosol on filter fibers strongly depends on the filling degree of a nonwoven space with fibers, as well as on the fiber metrological parameters (surface and thickness of nonwoven-forming filaments) and metrological parameters of the nonwoven itself. These factors affect the resistance of air flow and the filtration efficiency of aerosol particles present in the flowing carrier gas. Thus far, the efficiency of nonwoven filters has been improved by modification of the factors mentioned

above or by incorporating a permanent electric charge in the process of corona discharge (the formation of fibrous electret structures) [5, 6]. Studies carried out by European research centers [7] have shown that electrete filters lose their protective properties when exposed to hazardous aerosol present in the atmosphere of a work environment. Thus there is a need for replacing electrostatic activation with another method for the modification of filter nonwovens. The present study proposes a modification of PP nonwovens with low -pressure cold plasma (LPCP). Such a process is dry, clean, environment-friendly and inexpensive. Plasma treatment has already been successfully used for the modification of filter membranes in medical and technical devices with permanent hydrophilicity [8, 9], separation [10] and permeable [11-14] properties. There a small number of literature reports on the plasma modification of filter materials designed for the protection of the respiratory system [15].

Using the hexamethylidisiloxane (HMDSO) plasma treatment of acetylocellulose membranes for ultra-filtration and polyamide membranes for micro-filtration, highly effective filters have been obtained for the selective filtration of gas mixtures (He/N₂) [10]. The plasma modification of polyethylene, polysulfone and polyethersulfone membranes has increased the filtration effectiveness of the aqueous dispersion of molecules and polymers [8]. The filtration cartridges of medical devices designed for the filtration and separation of organic media (bacteria and proteins) treated with plasma improve the process effectiveness and the quality of

sterile filters [16, 17]. When hydrophobic or hydrophilic properties are imparted to filter materials designed for specific applications, the process effectiveness is considerably increased [18].

Considering the complex character of the process and the dependence of treatment effects on many process parameters (type of process gas, discharge power, treatment duration, gas flow rate, etc.), it is vital to establish optimal conditions for the treatment. In this study, PP nonwovens made by the melt-blowing method were modified by means of non-polymerising gases (argon, air) and polymerising gases (methane and HMDSO) [19]. Plasma treatment was performed using different process parameters: pressure, power and duration of the plasma treatment. The effects of the modification were assessed on the basis of test results of the penetration of aerosol solid particles through nonwoven filters and the resistance to air flow.

Experimental – materials and methods

Materials

The materials used for the LPCP modification were PP nonwovens made by the melt-blowing method. These nonwovens had an average thickness of 1.5 mm and surface weight of 80 g/m². They were formed in two categories of average filament thickness: 2.12 μm and 5.8 μm.

Modification process

The PP nonwovens were treated with plasma in a laboratory system designed by us using a tube flow-through reactor equipped with two opposite copper electrodes positioned outside the reactor, sup-

plied with power from a RF 13.56 MHz generator through a resonance matching circuit. The RF generator used made it possible to control the power within the range 10 ÷ 300 W. The glass reactor tube (diameter: 9 cm, length: 50 cm) was evacuated using a two-stage rotary pump. The process gas flowing along the axis of the tube was controlled by a micrometer leak valve, whose pressure was monitored by a resistance gauge within the range 10^{-3} Tr ÷ 1Tr. Samples placed in glass holders were positioned in the inter-electrode zone with their surface being contiguous to the direction of the flowing gas. Prior to the plasma treatment, the samples were pretreated in the reactor with Ar plasma to clean their surface. Based on the preliminary tests, described later, the following optimal conditions were established for the pretreatment parameters: argon pressure 0.1 Tr, power 100 W, and duration 5 min.

Three types of plasma-forming gases were used: oxidative gas (air), hydrocarbon gas (methane) and the vapour of an organosilicone precursor: hexamethyldisiloxane (HMDSO).

The effect of various plasma treatments, including Ar plasma, on the filterability of nonwovens was analysed in terms of different treatment parameters: gas pressure (from 0.1Tr to 0.01Tr), discharge power (from 10W to 200W) and the duration of the plasma treatment (from 1 min. to 10 min).

Test methods

Examination of the chemical constitution of the PP surface with the use of ATR-IR spectroscopy

IR spectrophotometric examinations of the PP surface were performed by the IR-ATR

technique using an attachment with KRS-5 crystal and a FTIR-8300 spectrophotometer. IR spectra were recorded as a function of the transmittance within a wavelength range of 400 cm^{-1} to 4000 cm^{-1} .

Microscopic examination of the PP fiber surface

The PP fiber surface was observed by means of a JSM-5200LV electron microscope from JEOL (Japan) using a detector of secondary electrons. Prior to observations, the fiber surface was dusted with gold using a JFC-1200 magnetron duster from JEOL. The samples were examined under a high vacuum with the use of the detector of secondary electrons. The accelerating voltage was 15-25 kV and magnification – up to 35000x. Images were recorded using the SEMAFORJEOL System.

Laboratory filtration tests

The efficiency of the penetration of aerosol solid particles through non-woven filters was tested with the use of measuring apparatus that measures the penetration of materials by sodium chloride aerosol according to Standard EN 143:2000 [20]. The concentration of the aerosol was controlled directly before and behind the filter by means of a flame photometer, being an integral part of the apparatus. The operation principle of the flame photometer is the measurement of the intensity of a light beam generated by a hydrogen burner. The intensity of scattered light is a measure of the aerosol concentration. The measurement range of the photometer was from 0.001 to 100%. The polydisperse sodium chloride aerosol prepared for the tests had an average diameter of particles of about $0.6\text{ }\mu\text{m}$ and concentration of $(8 \pm 4)\text{ mg/m}^3$. The volumetric intensity of the aerosol flow was 95 l/min.

The measurement of the filtration effectiveness was made as an average within $(30 \pm 3)\text{ s}$ at the third minute of the test duration. The tests were carried out at room temperature and RH below 60%. Calculation of the penetration was made according to $P [\%] = C_2/C_1 \cdot 100\%$, where: P is the penetration, C_1 – the sodium chloride concentration before the filter, and C_2 is the sodium chloride concentration after the filter. The resistance to air flow through the filter material was also determined.

Results and discussion

Surface chemistry

Effect of Ar plasma on the properties of PP nonwovens

Argon plasma pre-treatment was used to clean the surface of fibers. The effects of Ar electrons and ions on the PP fiber surface were examined from the point of view of their influence on the chemical constitution of the fiber surface, microtopography and filtration properties. **Figure 1** shows the IR-ATR spectra of a PP nonwoven surface treated with argon plasma under conditions of 100 W power for 5 min. at a pressure of 0.1 Tr, together with those of an untreated nonwoven surface. Within the full range of weave numbers, no significant changes in the absorption properties of the fibers were observed, which indicates a lack of changes in the chemical structure of PP fibers due to Ar plasma. The outline of a new band observed at 1650 cm^{-1} , corresponding to C = O stretching vibration [21], may indicate a hydrophilic transformation of the fiber surface. The action of an inert gas on the polymer surface is assumed to result mainly in surface etching [22, 23], and the effect of surface functionalisation observed takes place during

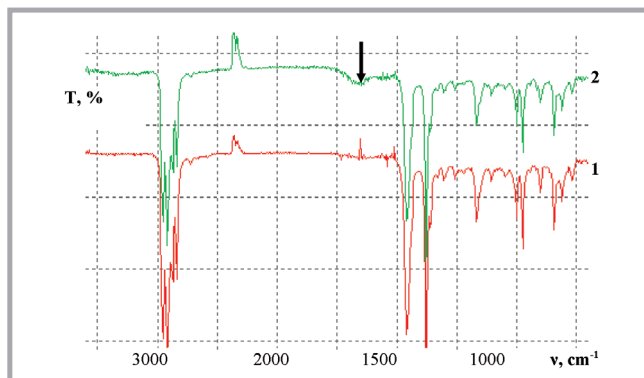


Figure 1. ATR-IR spectra of the PP nonwovens: 1 – untreated, 2 – ar plasma treated: power 100 W, pressure 0.1 Tr, time 5 min.

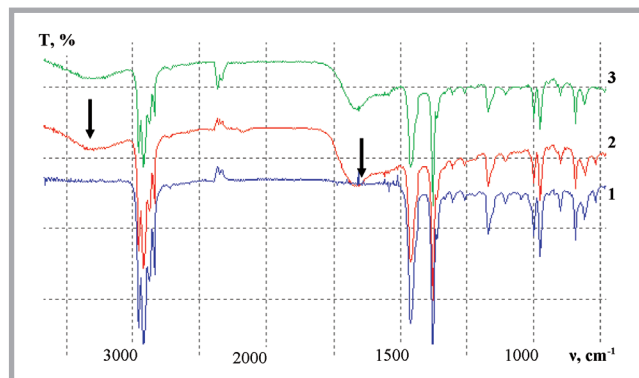


Figure 2. ATR-IR spectra of the PP nonwovens: 1 – untreated, 2 – air plasma treated: power 50 W, pressure 0.05 Tr; 3 – air plasma treated: power 100 W, pressure 0.05 Tr, time 10 min.

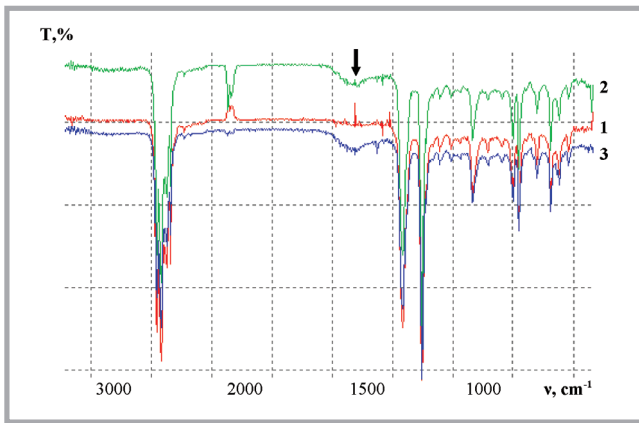


Figure 3. ATR-IR spectra of the PP nonwovens: 1 – untreated, 2 – methane plasma treated: power 50 W, pressure 0.05 Tr; 3 – methane plasma treated: power 100 W, pressure 0.05 Tr; time 10 min.

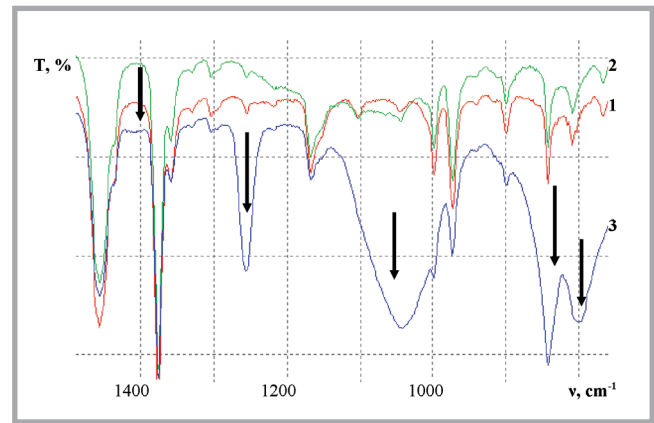


Figure 4. ATR-IR spectra of the PP nonwovens 1 – untreated, 2 – HMDSO plasma treated: power 50 W, pressure 0.05 Tr; 3 – HMDSO plasma treated: power 50 W, pressure 0.1 Tr; , time 10 min.

moisture absorption from the surroundings after removing the sample from the reactor (so-called post-treatment) [24].

Effect of air plasma on PP non-woven properties

Owing to its composition, air generates plasma that contains reactive oxygen, peroxide and nitrogen species which, together with electrons, interact with the polymer surface, causing not only the etching of the surface layer but also its functionalisation. The air plasma treatment of nonwovens resulted in the oxidation of the fiber surface, as confirmed by two broad bands: 1660 and 3320 cm^{-1} (Figure 2), indicating newly formed carbonyl C = O and hydroxyl – OH groups, respectively [21].

Effect of methane plasma

In the chemistry of hydrocarbon plasma modelling, methane is considered to be a precursor of hydrocarbon polymers. The structure of these polymers strongly depends on the parameters of methane plasma and, consequently, on the content of coordinated carbon atoms of the type sp^1 , sp^2 & sp^3 [25, 26]. Depending on the assumed parameters of methane plasma in the synthesis, one can obtain a polymer

with the physical properties and chemical structure required: from hydrogenated carbon amorphous layers to hard, chemically inert diamond-like a-C:H layers with a high content of sp^3 carbon [27]. The treatment of PP nonwoven with RF methane plasma and effective power from 25 W to 200 W under a pressure of 0.05 to 0.1 Tr results in the formation of plasma polymer layers. This polymer is formed in methane plasma that contains various free radicals (CH_3 , H, H_2 , CH, C_2H_4 , C_2H , C_2H_3 , C_2H_2) [25] as products of methane dissociation under the influence of collisions with plasma electrons. Plasma polymer, as shown by the ATR-IR spectrogram (Figure 3), has the character of a hydrocarbon polymer, similar to the substrate on which it was deposited. The accentuated broad band with maximum absorption at 1650 cm^{-1} seems to be the result of post-treatment [24].

Effect of HMDSO plasma

Plasma containing HMDSO as a precursor is used to obtain organo-silicone polymers of the SiOC:H type [30,31,32]. The synthesis of polymer performed in plasma under conditions of controlled RF discharge allows to obtain films with various thicknesses, compositions, and

cross-linking degrees, as well as the properties required, such as the wettability, elasticity, hardness abrasion resistance, chemical resistance, biocompatibility, permeability, etc.

In this study, PP non-wovens were treated with HMDSO plasma with an effective power ranging from 25 W to 200 W at a pressure of 0.05 to 0.1 Tr. In this process the fibers treated were coated with a plasma polymer film, whose chemical identification is shown in the ATR-IR spectrogram (Figure 4). Unlike the untreated sample, the plasma-treated nonwoven strongly absorbed IR radiation in four bands: 800 cm^{-1} , 841 cm^{-1} , 1040 cm^{-1} and 1256 cm^{-1} . The absorption within these bands identifies chemical groups containing Si [32, 33, 34, 35, 36]. The absorption in band 800 cm^{-1} is relative to the bending stretching Si-O-Si group and CH_3 rocking in $\text{Si}(\text{CH}_3)_2$. The absorption in the 840 cm^{-1} band is relative to the stretching bands of $\text{Si}(\text{CH}_3)_x$ and CH_3 rocking in $\text{Si}(\text{CH}_3)_3$. The 1040 cm^{-1} band shows the asymmetric stretching band of the Si-O-Si group. The 1260 cm^{-1} band is relative to the stretching of CHx and bending modes of the methyl groups in $\text{Si}(\text{CH}_3)_x$. In plasma excited with a high

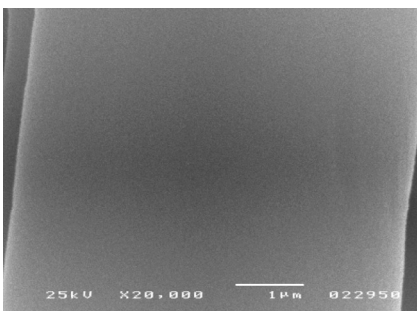


Figure 5. Untreated PP fibers

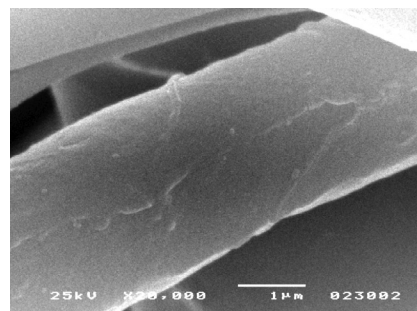


Figure 6. PP fibers treated with Ar plasma at 100W and 0.1Tr for 5min.

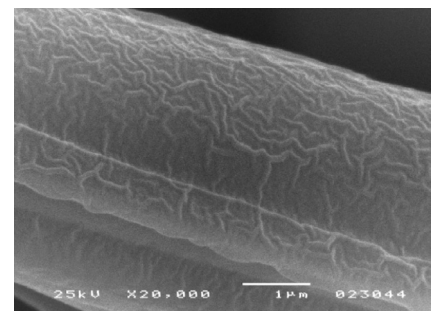


Figure 7. PP fibers treated with air plasma at 100 W and 0,05 Tr for 10 min.

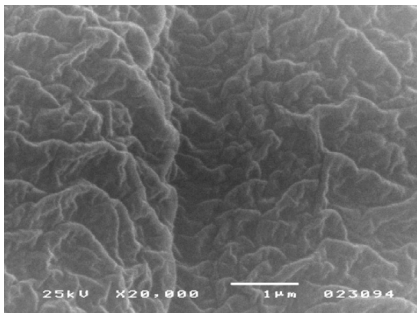


Figure 8. PP fibers treated with methane plasma at 100W and 0.05Tr for 10min.

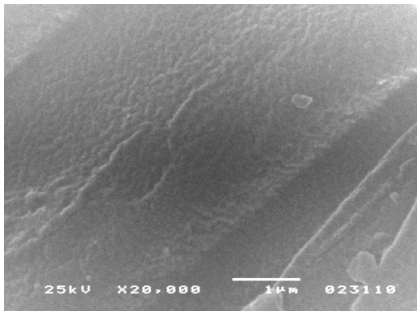


Figure 9. PP fibers treated with HMDSO plasma at 50W and 0.05Tr for 10min.

power (100 W), a new band – 1410 cm^{-1} indicates symmetric and asymmetric – CH_3 bending in $\text{Si}(\text{CH}_3)_x$. [31]

Surface topography

Figures 5-9 show examples of SEM images of elementary fibers in the surface layer of PP nonwoven modified with various types of plasma in comparison to untreated fibers. The SEM images of PP fibers shown in **Figure 7** confirm the occurrence of fiber surface etching by the ions of Ar plasma. The effects of etching increased with an increase in power, prolonging the Ar plasma action.

Figures 5-9. SEM images of PP fibers on nonwoven fabrics treated with plasma.

Figure 7 shows an image of PP fibers treated with air plasma. The functionalisation of PP fibers during plasma treatment is accompanied by the etching of the polymer surface layers. The SEM image of PP fibers treated with air plasma at a power of 100 W for 1-10 min shows a softly etched, mesh-like surface layer, from which the weakly bound polymer (amorphous PP) has been removed by plasma ions. The resulting effect of filament surface development can influence the filtration properties of the treated nonwovens.

Figure 8 shows the results of the action of methane plasma on PP fibers. This process results in the formation of a hy-

drocarbon polymer film on the PP fiber surface, and the surface developed indicates a gradual, local arrangement of the plasma polymer. The growing rate of the plasma polymer layers increased with increasing power. The facing of fibers with plasma polymer is not identical around the fiber perimeter, which indicates the limited range of plasma species penetrating the nonwoven structure [29].

Figure 9 shows the results of PP fiber treatment with HMDSO plasma. This process results in the formation of a new organo-silicone plasma polymer and its deposition on the fiber surface, which develops at a sub-micrometric scale. The growing rate of the plasma polymer layers increased with an increase in the power and process duration.

Filtration capabilities of PP nonwovens

The efficiency of filtration, i.e. the ability of the nonwoven to catch pollution, e.g. particles of dust from the stream of flowing air, was measured using leakage P, i.e. the penetration of the filter material by applying test aerosols (e.g. NaCl). The higher the value of NaCl test aerosol penetration, the lower the efficiency of filtering i.e. the filtering non-woven has lower protective features. The particle filters were classified according to their filtering efficiency [EN 143:2000/A1:2006 [20]. There are three classes of particle filters: P1, P2 and P3, in ascending order of filtering efficiency, measured using the penetration of the test aerosol. The requirements for the maximum filter

penetration of sodium chloride at 95 l/min were assumed as follows [20]: for filter class P1 – 20%, class P2 – 6%, and class P3 – 0.05%, being regarded as suitable. The requirements for the maximum breathing resistance at 95 l/min were assumed as follows [20]: for filter class P1 – 210Pa, class P2 – 240Pa, and class P3 – 420Pa.

Effect of Ar plasma

Ar plasma treatment was used to prepare PP nonwovens for modification. The test results (**Table 1**) show that in the nonwovens formed from thicker fibers with an average diameter of about 5.8 μm , the penetration of NaCl aerosol slightly increased in comparison to that of unmodified fibers, especially when the pressure of the plasma was low (0.05 Tr). The use of a higher Ar pressure results in a more beneficial effect: a decrease in aerosol penetration, especially in the case of nonwovens made from thinner fibers (an average thickness of 2.12 μm). At the same time, the PP non-wovens formed from thicker fibers and treated with Ar plasma at a lower pressure (0.05 Tr) showed an increase in resistance to air flow, from 51 Pa to about 70 Pa. The use of a higher Ar pressure allows for the better modification of filtration properties: the filtration efficiency increases in both types of nonwovens without any increase in air flow resistance. Based on the results obtained, the following parameters of the pretreatment process were established: power 100 W, treatment duration 5 min, and Ar pressure 0.1 Tr.

Table 1. Filtration measurement results for PP filter nonwovens before and after Ar plasma treatment.

Type of nonwovens (thickness of PP fibres, μm)	Plasma treatment conditions			Penetration, % / resistance, Pa	
	Pressure, Tr	Power, W	Time, min	Before treatment	After treatment
2.12	0.1	100	5	22.7/94	8.9/89
	0.05	25	10	35.6/51	39.0/70
5.8	0.05	100	10		36.7/66
	0.1	25	10		37.0/75
	0.1	100	5		25.2/56
	0.1	100	10	26.6/65	

Table 2. Filtration measurement results for PP filter nonwovens before and after air plasma treatment.

Type of nonwovens (thickness of PP fibres, μm)	Plasma treatment conditions			Penetration, % / resistance, Pa	
	Pressure, Tr	Power, W	Time, min	Before treatment	After treatment
2.12	0.05	100	1	18.5/100	1.9/100
	0.05		10	18.8/100	3.7/88
5.8	0.05	100	1	36.0/60	5.8/60
	0.05	100	10		9.9/60

Table 3. Filtration measurement results for PP filter nonwovens before and after methane plasma treatment.

Type of nonwovens (thickness of PP fibres, μm)	Plasma treatment conditions			Penetration, % / resistance, Pa	
	Pressure, Tr	Power, W	Time, min	Before treatment	After treatment
2.12	0.1	50	10	22.7/94	5.6/86
	0.05	50	10		3.0/92
5.8	0.1	25	10	35.6/51	11.3/44
		50			13.2/52
		100			11.6/46
	0.05	25		7.7/50	
		50		9.5/53	
		100		8.4/49	

Table 4. Filtration measurement results for PP filter nonwovens before and after HMDSO plasma treatment.

Type of nonwovens (thickness of PP fibres, μm)	Plasma treatment conditions			Penetration, % / resistance, Pa		
	Pressure, Tr	Power, W	Time, min	Before treatment	After treatment	
2.12	0.1	50	10	18.5/100	2.1/96	
			1		2.3/97	
	0.05		5	22.7/94	4.0/88	
			10	18.5/100	3.7/93	
5.8	0.1	25	10	35.6/52	16.2/50	
		50		36.0/60	7.5/59	
		100		35.6/52	1.4/49	
	0.05	25		31.5/55	5.6/56	
		50		36.0/60	12.0/56	
				5	31.5/55	8.9/54
				1	36.0/60	5.6/59

Effect of air plasma

Table 2 shows the values of parameters characterising the filtration properties of PP nonwovens treated with air plasma: the penetration of aerosol and flow resistance. PP nonwovens modified with air plasma can filter NaCl aerosol more effectively under standardised flow conditions. The penetration of aerosol in the nonwoven filter treated with air plasma decreased 4 ÷ 7 times depending on the conditions of the plasma treatment. The test results concerning the effect of the duration of air plasma treatment and the power supplied to the reactor indicate that the optimisation of the plasma treatment of filter materials should tend towards relatively short treatment durations and average power values. The use of power not higher than 50 W and an air pressure of 0.05 Tr for 1 min. already results in a high filtration efficiency. This observation concerns non-wovens formed from PP fibers with various thicknesses. The non-wovens made from thinner fibers with a diameter of 2.1 showed better filtration effectiveness than those formed from fibers with an average diameter of 5.8 μm .

It is also important that in the filtration process observed, the resistance to air flow in the PP nonwovens modified does not change significantly. This fact can be justified by the limited penetration of plasma species inside the material, which causes that the range of changes made is limited to the surface layer of the non-woven. However, the existence of this layer in the filter material plays an important role in terms of the efficiency of aerosol filtration.

Table 3 presents the results of testing the filtration properties of PP nonwovens treated with methane plasma. It is observed that aerosol penetration into PP nonwoven treated with methane plasma is already considerably decreased (~5 times) when the power supplied to the reactor is as low as 25 W. These nonwovens show more beneficial filtration properties when they are coated with thin layers of hydrocarbon polymer at a methane pressure of 0.05 Tr. The advantage of PP filter nonwovens modified with methane plasma is the fact that they do not affect the resistance to air flow.

Table 4 shows results of the modification of PP nonwovens with HMDSO plasma

using different plasma parameters (power and HMDSO pressure) and treatment durations. The penetration of NaCl into the plasma-treated filter was already considerably decreased (~7 times) when the plasma treatment had been performed for 1 min. at a power of 50W and pressure of the process gas of 0.05 Tr. One can assume that PP nonwovens have sufficiently good filtration capabilities when they are coated with thin layers of organo-silicone polymer. More effective filtration is shown by PP non-wovens made from fibers with a lower diameter. The air flow resistance in the HMDSO plasma-treated filter materials did not increase after treatment under accepted discharge conditions.

Conclusions

The medium of low-temperature RF plasma was used to modify the filtration properties of polypropylene nonwovens made by the melt-blowing method for the production of filtering respiratory protective devices designed to protect against solid particles. The process gases used in the plasma treatment included air, methane and HMDSO.

1. The examinations of the influence of process gas plasma on PP nonwovens prove the appearance of various effects resulting from this treatment. Air plasma brings about the functionalisation of the PP fiber surface and its etching, which results in the development of a specific surface of fibers that form the filtration layer.

The plasmas of gases that polymerise on nonwovens (methane, HMDSO) form films with a developed surface and thickness controlled by process conditions (power supplied to the reactor, discharge duration), as confirmed by the ATR-IR and SEM results. SEM images of the plasma-treated fiber surface indicate that plasma molecules penetrate only the top layer of nonwovens.

2. The plasma treatment of PP nonwovens considerably changes their filtration capabilities in relation to the penetration of NaCl aerosol or aerosol solid particles. In all the cases of process gases, similar effects were obtained: the efficiency of aerosol filtration in the plasma-treated non-wovens was increased. These properties can be imparted to PP non-wovens through treat-

ment with process gas plasma induced with a low gas pressure, e.g. 0.05 Tr, and relatively low power acting for a short time (1 min.) The optimisation of the conditions of modifying PP nonwovens with gas plasma allows to raise the protective class of a filter, e.g. from P1, according to EN 143:2000/A1:2006 [20], to protective class P2 if the construction layer of the filter is made from the modified nonwoven.

- Filters based on plasma-treated PP nonwovens cause no increase in air flow resistance.



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