Agnieszka Brochocka,
Katarzyna Majchrzycka

Technology for the Production of Bioactive Melt-Blown Filtration Materials Applied to Respiratory Protective Devices

Central Institute for Labour Protection – National Research Institute
ul. Czerniakowska 16, 00-701 Warszawa, Poland

Abstract
This paper presents the modification of a technology for the production of melt-blown nonwoven filtration materials. A method of introducing a bioactive agent during melt-blown production was developed. Bioactive properties were obtained by means of biologically active modifiers. As a result of microbiological tests, it was proven that the addition of modifiers at the initial technological stage does not give satisfactory results because of difficulties in combining the modifier particles with the polymer granulate, as well as the negative influence of the temperature of the molten polymer on biocidal properties. The technological research was concerned with establishing a method of introducing various biocidal agents at different points of melt-blown production. To this end, modification of a laboratory station head and take up device was performed, and then the process parameters were optimised with special consideration for polymer consumption and polymer blow temperature. The results of the microbiological tests confirmed the usefulness of nonwoven filtration materials for the construction of respiratory bioprotective devices due to their bioactive properties.

Key words: *melt blown filtration material, microorganisms, bioactive fibers, bacteria, bioactive agents.*

Introduction

Microorganisms, which are present everywhere at work in the human's environment, are of great significance. On the one hand, they play a fundamental role in element circulation, soil fertility, digestive processes, food and pharmaceutical production as well as waste biodegradation, but on the other hand, pathogenic flora is the cause of numerous serious illnesses, both in people and animals [1]. Therefore, from the point of view of health protection, it is most important to reduce the population of microorganisms to a non-threatening level [2 - 5]. There are no specially designed protective devices for respiratory system protection against microorganisms, which is why half masks and filters intended for protection against industrial aerosols are used at present. However, this type of respiratory protective equipment does not prevent the development of microorganisms deposited on the filtering material whilst the equipment is used. This phenomenon is of particular importance for respiratory protection as microclimate conditions under the facepiece are conducive to these unfavourable effects. With long-term use excessive microorganism colonisation may make the respiratory protective device dangerous. Intensive research was therefore undertaken aimed at developing a new type of biologically modified filtration nonwoven whose high filtration efficiency of fine dispersion particles, relatively low air flow resistance, and the ability to stunt microorganism development within at least 2 hours from the moment of deposition onto the filtration material are conditions for its application in the construction of respiratory protective devices.

Numerous research centres have been carrying out work aimed at developing bioactive fibres or textiles, such as dressings, surgical thread, vascular prostheses, and hospital linen. From the description of Polish patent PL 200059 B1 [10], it is known that synthetic fibres are provided with antibacterial properties by preliminarily swelling them with benzene or toluene, and after the solvent has been removed, imbuing them with modifying bath containing a biocide of the nitrofuran series, catalyst, imbuing activator and/or dispersing agent.

Also from the description of Polish patents PL 179483 and PL 187392 [11], the way of providing synthetic fibres with antibacterial properties is known, consisting in creating active centres on fibres in the form of peroxides and hydroperoxides. Later, during grafting, acid carboxylic groups are introduced into the fibres, after which they are imbued with an antibiotic aqueous solution.

In another patent, the way of providing polyamide, polynitroacrylic, polyester, and polypropylene fibres with antibacterial properties consists in treating fibres by preliminary swelling them with benzene or toluene, after which they undergo modifying bath treatment with a biocide of the_nitrofuran series, an acid dispersing agent and imbuing reaction activator or dispersing agent; however, the fibres that do not include functional groups in their particles undergo grafting with vinyl monomers prior to the swelling procedure [12].

It should be emphasised that all the solutions developed are not sufficient from the point of view of efficient respiratory protection requirements, primarily because of fibre thickness and the textile structure produced. Due to the fact that the high efficiency of filtration nonwovens in retaining particles is a necessary condition for their application in respiratory protection devices, only technologies providing a wide scope of nonwoven structure parameter control could be considered. Thus, it was decided to apply the melt-blown technology of fleece forming.

The main aim of the research was to develop a technology for manufacturing filtration nonwovens that can be used for designing bio-protection for the respiratory system as well as to develop, design and experimatelly check an appropriate equipment.

Experimental procedure

In order to obtain high efficiency filtration at a level guaranteeing compliance with the requirements of second and third protection classes [8, 9], attempts were made to produce filtration nonwovens by means of fiber-forming polymer blow with the addition of bioactive modifiers. The work was carried out with the use of experimental devices belonging to the Central Institute for Labour Protection – National Research Institute, Department of Personal Protective Equipment. Microbiological tests were carried out at the Technical University of Lodz, Institute of Technology Fermentation and Microbiology.

Materials:

Polymer used for melt-blown production – polypropylene Malen P F401 – density 0.905 - 0.017 g/cm^3

Bioactive agents:
- Microban® Additive IB12,
- Bronopol®,
- Silver (active compound – silver),
- Viroksan (active compound – magnesium monoperoxyphtalate),
- Magnesium monoperoxyphtalate (active compound – magnesium monoperoxyphtalate).

Technological methods:

I Addition of biocidal agent into the extruder's feeding zone (loading hopper in ***Figure 1***) (see page 94),

II Spraying biocidal agent onto the fibres blown (see ***Figure 1***),

III Deposition of biocidal agent onto the nonwovens (see page 94),

IV Inserting biocidal agent into the polymer streams in the fibren forming hand (see ***Figure 2***).

Test methods:
- Paraffin oil penetration test (see pages 93 and 94),
- scanning electron microscope were used for evaluation of fibre thickness and the regularity of the biocidal agent placed in the nonwoven structure(see page 95),
- Bacteriostatic and bactericidal activity (see page 96),
- Efficiency in filtering *E. coli* bacteria in the air flow of the nonwoven (see page 97),
- The total amount of biocidal bound to the fibres and absorbed on the surface was calculated on the basis of elementary analysis (see page 97).

The following technical-technological assumptions were accepted:
- Different thermoplastic polymers may be used for melt-blown nonwoven production. Based on polymer chemical composition analysis and the dielectric constant, the most commonly used polymer – polypropylene was selected for the melt-blown technology under discussion.
- The surface of the take-up device should be made of a net which allows the gathering of the finest polymer fibres and the biologically active substance during fleece forming by means of negative pressure created in a special sucker placed under the surface of the net. The structure of the take-up device should be made of dielectric material.
- The fibre-forming head should enable obtaining the necessary plasticisation of the polymer in the polymer nozzle capillary, as well as the insertion of the biologically active substance. This process is additionally facilitated by a very thin (0,15 mm) capillary wall, which should be used in the construction of the polymer nozzles. A construction of this kind enables heat absorption from the hot air used for blowing the polymer , causing its final heating. This kind of solution will considerably shorten the time the polymer stays in the hot temperature zone, which may cause its degradation.
- The polymer melt should be brought to a suitable plasticised form in the extruder cylinder with the following constant parameters: polymer yield and cylinder temperature, the maintaining of which will provide a constant amount of polymer flowing through the head.
- The control of a process with three technological parameters: head temperature, air temperature and the yield of air blowing the polymer with the head at a fixed distance from surface of the take-up device should lead to obtaining a melt-blown material with assumed parameters.

Table 1. *Test results of paraffin oil penetration for melt-blown nonwovens according to PN-EN 143:2000/A1:2006 [8].*

Penetration, % at:	Penetration, % at:
Flow rate 47.5 dm^3/min Concentration 20.08 mg/m^3 Test time: 3 min.	Flow rate 95.0 dm^3/min Concentration 20.27 mg/m^3 Test time: 3 min.
0.45	1.58

A block diagram of a laboratory stand for manufacturing biocidally modified melt-blown nonwovens according to the technological method I, II and III is presented in Figure 1. Granulated polymer is shifted from the loading hopper (1) to the heated extruder cylinder (W). It is melted to a suitable viscosity and brought to the fibre-forming head (G). Compressed air passes from the controller (R) to the heater (N), where it is dried and heated to the appropriate temperature. Next it is directed to the fibre-forming head (G), leaving which it blows the polymer streamlets extruded from the nozzles onto the elementary fibres (2), which settle on the take-up device (U), forming a dense porous fleece with dimensions of 100 x 30 cm.

The stand for forming melt-blown nonwovens has got adjustment points which enable the change of technological parameters, which allow the modelling of the morphological structure of nonwovens and, specifically, the suitable selection of fibre diameter. Depending on the nozzle temperature (T_d) and temperatures of extruder zones I and II (T_{W-I}, T_{W-II}), a varied polymer melt viscosity is obtained, which decides the conditions of its defibering, being also dependent on a suitable air temperature (T_p) and appropriate proportion of polymer consumption (P_p) and air consumption (W_p). A change in the linear velocity (V_l) or angular velocity (V_o) of the take-up device (U) allows the surface mass or the fibre packing density of the nonwoven unit to be adjusted.

The melt-blown technology developed by us allows the formation of nonwovens of surface mass 10 – 200 g/m^2, which is differentiated by layer-by-layer application of fibrous fleece. Due to the too low strength of small mass nonwovens, an optimum surface mass value of 90 g/m^2 was adopted for the experiments, achieved by putting 3 fleece layers on one another, each of 30 g/m^2 mass. It was assumed that for each variant deviation from the nominal, the surface mass should not exceed + 10%. The nonwoven variants formed were subjected to activation with a positive electric charge (+) of 25 kV. The tension growth leads to spark discharges which result in nonwoven

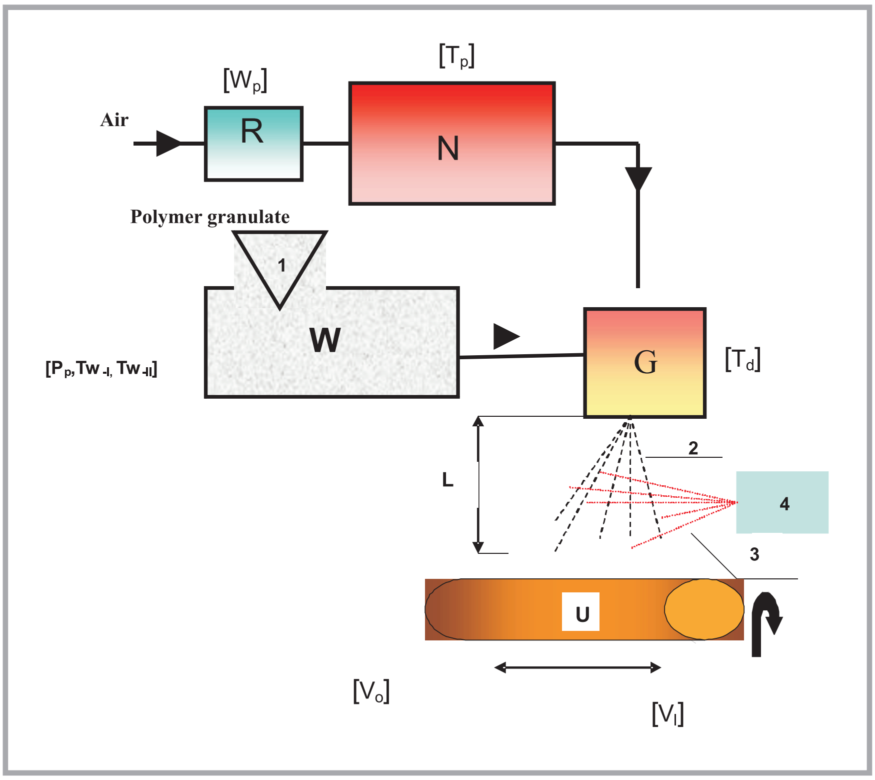

Figure 1. Block diagram of laboratory experimental stand for manufacturing melt-blown biological active nonwovens (in method I the bioactive particles are added to (1), in method II by device (4) and in method II onto the mat lying on take-up device U; 1 - loading hopper, 2 - elementary fibres, 3 - bioactive particle stream, 4 - the nozzle distributing bioactive particles, U - take-up, W - extruder, N - air heater, G - head, R - compressed air flow controller, L - head distance from the take-up device.

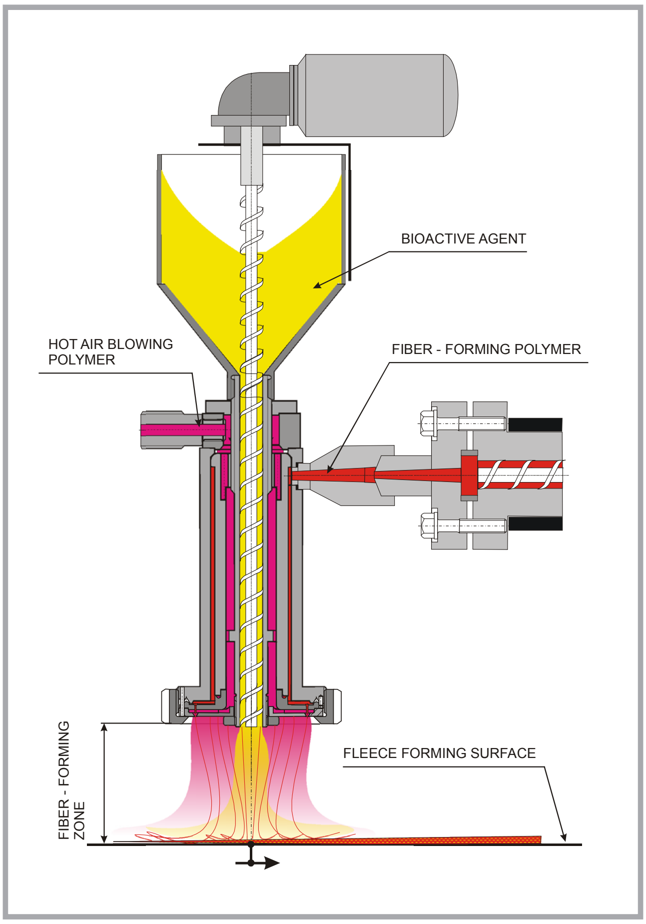

Figure 2. *Scheme of fibre-forming head according to the final method (IV).*

punctures, destroying its structure and disqualifying it as filtration material.

The characteristics of basic filtration parameters defined by a model aerosol –paraffin oil mist [8] are presented in ***Table 1***. Average values of penetration were calculated for five samples of filtration nonwovens with a surface mass of 90 g/m^2 and electrostatic charge.

The test results presented show that melt-blown non-woven with an electrostatic charge manufactured at an experimental stand according to method II (***Figure 1***) is characterised by an index of penetration against paraffin mist aerosol for the 2nd level of safety (a value of up to 6% required). Having the technological parameters for melt-blown filtration non-woven of the 2nd safety class set, further works directed at manufacturing bioactive filtration non-wovens were conducted.

Preliminary tests of melt-blown fleece forming technology involved the application of two bioactive agents:

- Microban® Additive IB12 in powder form (ready – made product), and
- Bronopol® in solution form (ready – made product).

Both substances conform to the 98/8/EU directive, which confirms their safety of use in contact with the user's skin. According to information by the producer, biocidal properties were verified for Gram-positive and Gram-negative bacteria.

Tests using Microban® Additive IB12 were carried out with the addition of powder at a concentration of 0.75%, and later at a 1.0% concentration, to the polymer (polypropylene) granulate in extruder zone I. (according to method I). Observations of the technological process highlighted the need to mix the powder particles of Microban® Additive IB12 with the polymer granulate prior to feeding the extruder. The necessity of this operation resulted from the settling of charged particles of Microban® Additive IB12 on the loading hopper walls and the consequent lack of possibility to mix with the polymer granulate inside the extruder. Bronopol® solution was sprayed on the melt-blown nonwovens formed. (according to method III) Tests were performed at a solution concentration of 0.1%.

Attempts were made by blowing the biological active agent directly into the

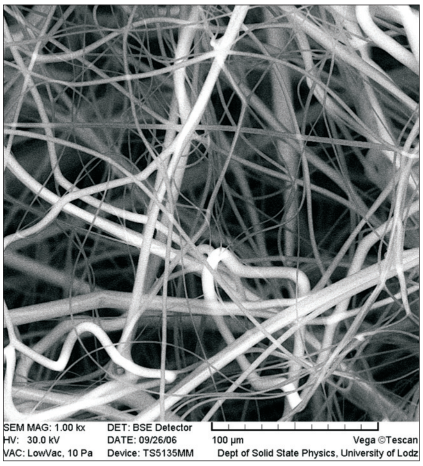

Figure 3. *View of filtration nonwoven surface without a biocidal agent.*

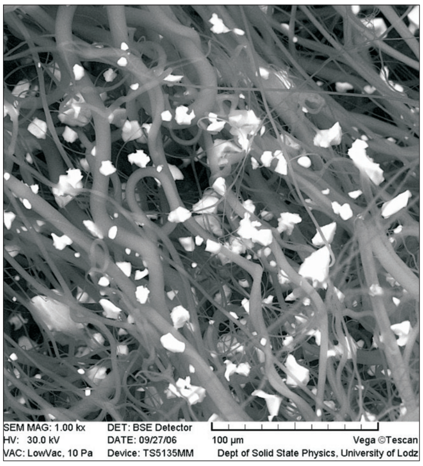

Figure 4. *View of filtration nonwoven surface with a biocidal agent.*

strem of fibres below the fibre-formin head also did not allow to obtain a positive result.

Due to the fact that it was impossible to introduce the biocidal agent into the polymer stream and its irregular placing in the nonwoven structure, attempts were made to produce filtration nonwovens using the fibre-forming polymer blow method with biocidal modifiers added directly to the fibre-forming head. The solution was formed by introducing a bioactive agent into the polymer stream and combining it permanently with the polypropylene polymer by means of an originally designed fibre-forming head structure, presented in ***Figure 2***. The biocidal agent is dosed to the head in a controlled way and then centrally and symmetrically transferred to the fibre forming zone. This way of feeding the agent enables the good mixing of fibres with the biocidal agent, which is sucked into the stream of fibres produced and evenly deposited on the fleece surface formed, resulting in smaller losses of the biocidal agent. The device for producing electret melt-blown nonwovens consists of an extruder, a heating connector placed between the extruder's cylinder and fibre-forming head, the latter being connected to the air heater and take-up device in the form of a net with a fan producing negative pressure. Its characteristic feature is the loading hopper placed over the head, which has a power engine of adjusted speed of rotation attached, connected to a screw placed centrally across the cylinder in the head axis, and runs along its length. The head core is in the shape of a cylinder, on the outer surface of which there is a spiral groove of the same diameter. The screw's cylinder is attached to the head with a cap. In its upper part the head is equipped with an extra stub pipe for connecting the hot air inflow supplied to the head through air openings. The head core is connected to the complex of polymer nozzles by means of a screwed sleeve., the construction developed by us enables the direct and effective providing of fibres with a biocidal agent directly in the course of fibre forming. Moreover, the agent may be added in a fully controlled way with the possibility of dosing according to requirements. Replacing the cone shape of the head core by a cylinder, and at the same time changing in the groove diameter from a decreasing to a fixed one, enabled the elimination of the unfavourable effect of channel clogging, which meant the need to dismantle the head for cleaning (***Figure 2***).

An experimental device for producing melt-blown nonwovens modernised in this way was used for making filtration nonwovens of polypropylene with the following technological parameters:
- temperature of extruding zone I – 280 °C
- temperature of extruding zone II – 274 °C
- air temperature – 295 °C
- head temperature – 280 °C
- air consumption – 8.8 m^3/h
- polymer consumption – 4.0 g/min.
- distance of take-up device from the head – 165 mm.

The surface mass of the nonwovens obtained was 90 g/m^2 , and the parameters of which were defined as follows [8]:
- penetration with paraffin oil mist – 2.50%
- air flow resistance - 106 Pa
- average fibre diameter – 2.43 µm (minimum value – 0.3 µm, maximum value – 10. 0 µm.

Next, the production of melt-blown nonwovens with the technological parameters thus defined and the nonwoven structure obtained was started with biocidal agents added in the amount of 60 g per square meter of nonwoven.

Nonwovens with the following biocidal agents were produced:
- Microban® Additive IB12 (active compound – silver)
- Silver (active compound –silver)
- Viroksan (active compound – magnesium monoperoxyphtalate)
- Magnesium monoperoxyphtalate (active compound – magnesium monoperoxyphtalate).

To illustrate the fibre thickness and the regularity of the biocidal agent placed in the nonwoven structure, photographs were taken using a VEGA 5135 MM Tescan scanning electron microscope with a 1000 times enlargement (***Figures 3, 4***).

Test results

The scope of the research involved the evaluation of the inhibition or biocidal efficiency of *Escherichia coli* growth after filtering the bacteria in the air stream through biologically active filtration material and then exposing the bacteria on the material for up to 8 hours.

E. coli was selected as the test strain in view of the criterion of high resistance to chemical disinfection. Tests were conducted in a stream of flowing air. Having filtered the air, the bacteria were exposed on melt-blown nonwovens in order to test the dynamics of microorganism dying under the influence of bioactive substances included in the materials tested.

The test nonwovens were cut out in the shape of rings 8 cm in diameter in aseptic conditions, on which the microorganisms were sprayed.

Assessment of *E. coli* bacilli growth inhibition efficiency was carried out for the following experiment variables:

a) Filtration material – melt-blown nonwoven made of polypropylene (PP) with the addition of bioactive modifiers:
  - Microban® Additive IB12 0.75%

- Microban® Additive IB12 1.0%
- Bronopol® 0.1%

without the addition of bioactive modifiers:
- Melt-blown nonwoven made of polypropylene (PP- control).

b) ***Incubation time*** of filtration materials inoculated with 24-hour *E. coli* bacilli culture:
- 0 hours (directly after inoculation),
- 2 hours
- 4 hours
- 8 hours.

The growth inhibition efficiency (% reduction R) of *E.coli* bacilli for the given filtration material after a given incubation time was assessed in proportion to the control, which was the average bacilli number (cfu/test) obtained in time 0.

$$R = \frac{\overline{N}_0 - N_t}{\overline{N}_0} \cdot 100 ,\% \qquad (1)$$

where:
N_0 – average number of *E. coli* bacilli washed out of the test filter in time 0
N_t – *E. coli* bacilli number washed out of the test filter after 2, 4 or 8 hours of incubation time in 37 °C.

For the needs of the experiment, a concept of microorganism number index Z_t was used:

$$Z_t = \frac{N_t}{N_0} \qquad (2)$$

where:
N_0 – number of microorganisms with which the textile material was inoculated (cfu/test)
N_t – number of microorganisms in the given time (cfu/test)

A comparison of results obtained is presented in ***Tables 2*** & ***3***.

Based on the test results obtained, it was found that filtration nonwoven with active substances Microban® Additive IB12 and Bronopol® added does not reveal antimicrobial activity in any statistically relevant way. For statistical analysis, ANOVA 2-factor variation analysis was used in repeated measurements with an error probability of 1st type > 0.05. Lengthening the incubation time at a temperature of 37 °C was accompanied by a bacilli population (not, as expected, a reduction). The application of Microban® Additive IB12 and Bronopol® particles during the process of forming melt-blown nonwoven had no influence on the microorganism population tested (there being no statistically important differences), nor did spraying at the final stage of forming the non-woven.

__Table 2.__ Test results of a number of microorganisms after specific incubation times for melt-blown nonwovens with a biocidal agent; SD- standard deviation.

Bioactive modification type	Number of microorganisms ± (SD) × 10^{-7} in cfu/test after specific incubation times in hours			
	0	2	4	8
Microban® Additive IB12 0.75% added to PP granulate	4.72 ± 2.21	4.90 ± 3.48	8.90 ± 1.95	11.7 ± 5.41
Microban Microban® Additive IB12 1.0% added to PP granulate	4.72 ± 2.21	4.91 ± 1.14	10.9 ± 4.68	8.76 ± 2.68
Bronopol® 0.1% sprayed on melt-blown nonwoven PP	4.72 ± 2.21	12.0 ± 8.42	8.63 ± 3.16	6.93 ± 2.90
PP – control	4.72 ± 2.21	6.37 ± 0.64	9.97 ± 4.59	11.6 ± 2.92

__Table 3.__ Test results of a number of microorganisms index Z_t after selected incubation times for biocidal melt-blown nonwovens.

Type of bioactive modification	Microorganisms number index Z_t after selected incubation times in hours			
	0	2	4	8
Microban® Additive IB12 0,75% added to PP granulate	1	1.04	1.89	2.49
Microban® Additive IB12 1,0% added to PP granulate	1	1.04	2.31	1.86
Bronopol® 0,1% sprayed on melt-blown nonwoven PP	1	2.54	1.83	1.47
PP – control	1	1.35	2.11	2.47

In view of the test results above, further technological work was undertaken aimed at changing the construction of the fibre-forming head (see ***Figure 2***) and the choice of biocidal agents.

For further analysis of the efficiency of bioactive agents used after modification of the fibre-forming head (***Figure 2***), microbiological tests of the bacteriostatic and bacteriocidal activity were conducted, which are expressed by formulas 3 & 4. Bacteriostatic activity means microorganism growth inhibition, but not its extermination, whereas bactericidal activity means a destructive effect.

Criteria of evaluation of non-woven activity were established using normative regulations for marking the biostatic and biocidal effect of disinfectants for bacteria, on the basis of PN-EN 1276:2000/Ap1:2001.

A value under 0.5 was considered low, which meant a drop in the number of microorganisms by three times, whereas activity at the third level was considered high, meaning a drop in the number of microorganisms by 1000 times.

$$\textit{Bacteriostatic activity} = \log A_{tn}/B_{tn} \qquad (3)$$

where:

A_{tn} – number of microorganisms on the control material after a given time of exposure (without biocidal agent)
B_{tn} – number of microorganisms on a bioactive material after a given hour of exposure (with biocidal agent added).

$$\textit{Bactericidal activity} = \log C/B_{tn} \qquad (4)$$

where:
C – number of microorganisms on control material after time 0 (without biocidal agent)
B_{tn} – number of microorganisms on a bioactive material after a given time of exposure (with biocidal agent added).

The number of *E. coli* bacteria and, in particular, incubation hours after spraying the microorganism on bioactive and control nonwovens without biocide are presented in ***Tables 4*** & ***5***.

Based on the number of bacteria and, in particular, the time of exposure, as well as on the experiments on bioactive materials and control nonwoven (two experiments, hence control nonwoven was used twice), the bacteriostatic and bactericidal activity was calculated, the results of which are presented in ***Tables 6*** & ***7***.

On the basis of the results obtained, it was found that the most active nonwovens against *E. coli* were melt-blown nonwoven with viroksan and melt-blown nonwoven with magnesium monoperoxyphtalate (IMPULS). Bacteria coming into contact with these nonwovens had a killing effect immediately after depositing the bacteria on the samples tested.

Comparing the efficiency of the remaining materials with Microban® Additive IB12 and silver, it was found that high inhibition efficiency and a biocidal effect were observed after 4 hours of exposure with bacteria. Bacteria growth inhibition and biocidal activity grew with the exposure time. The nonwoven with silver demonstrated a 100% biocidal effect already in the 4th hour of exposure. The lowest antimicrobial activity of the filtration materials tested (also at a satisfactory level) was demonstrated by melt-blown nonwoven with Microban® Additive IB12. The chemical agent applied to this nonwoven (microban) has a satisfactory efficiency already in the 4th hour of incubation with bacteria but demonstrates higher *E. coli* growth inhibition abilities than biocidal properties.

The degree of preventing *E. coli* microorganisms on nonwovens with viroksan, magnesium monoperoxyphtalate and on control nonwoven in the air flow (at a speed of 30 l/min.) was additionally tested, the results of which are presented in ***Table 8***.

The tests demonstrated that all filtration materials with biocidal agents hold bacteria in the same satisfactory degree - 98% of all bacteria transported as bioaerosol in the air is stopped by the nonwovens tested.

In order to confirm the permanent ***deposition of the biocidal agent*** (magnesium monoperoxiftalene) in the structure of melt-blown non-woven, tests of the microorganism content in such material were carried out, as well as the ***durability of its bonds***.

Analysis of biocidal particles in polypropylene fibres modified with magnesium monoperoxide, which were the basis of the melt-blown non-woven, was carried out using ***elementary analysis and spectrophotometric analysis in ultraviolet.***

After they are introduced into polypropylene fibres, biocidal particles may be either permanently bound to the fibre or adsorbed on the surface. A biocidal substance adsorbed on the surface shows greater activity in comparison with a biocidal substance permanently bound to the fibre, although it may be desorpted due to physical or chemical factors.

The total number of biocidal particles introduced into polypropylene fibres was calculated on the basis of ***elementary analysis***. The analysis was carried out for four samples of bioactive melt-blown non-wovens with magnesium monoperoxiftalane. On the basis of the

Table 4. *Number of E. coli bacteria on nonwovens tested and, in particular, incubation hours.*

Time, hrs	*E. coli* bacteria number × 10^{-2} / nonwoven tested					
	melt-blown nonwoven with Microban® additive IB12		melt-blown nonwoven with silver		control nonwoven without biocide	
	repetitions	average	repetitions	average	repetitions	average
0	100 100	100	100 200	190	170 200	185
4	3.0 4.0	0 0	0 0	0	400 500	450
8	1.0 2.0	0 0	0 0	0	420 300	360

Table 5. *Number of E. coli bacteria on nonwovens tested and, in particular, incubation hours.*

Time, hrs	E.coli bacteria number × 10^{-5} / nonwoven test					
	melt-blown nonwoven with viroksan		melt-blown nonwoven with magnesium mono-peroxyphtalate		control nonwoven without biocide	
	repetitions	average	repetitions	average	repetitions	average
0	0 0	0	0 0	0	610 600	605
4	0 0	0	0 0	0	15.0 15.0	15.0
8	0 0	0	0 0	0	1.17 1.20	1.19

Table 6. *Bacteriostatic activity of nonwovens tested for E. coli bacteria; * bacteriostatic activity = 100% (no bacteria on nonwoven after exposure), "-" no bacteriostatic effect.*

Time, hrs	Bacteriostatic activity			
	Melt-blown nonwoven with:			
	Microban® Additive IB12	silver	viroksan	magnesium monoperoxyphtalate
0	0.27	-	*	*
4	2.11	*	*	*
8	2.38	*	*	*

Table 7. *Bactericidal activity of nonwovens tested for E. coli bacteria; * bactericidal activity = 100% (no bacteria on nonwoven after exposure), "-" no activity.*

Time, hrs	Bacteriostatic activity			
	Melt-blown nonwoven with:			
	Microban® Additive IB12	silver	viroksan	magnesium monoperoxyphtalate
0	**0.26**	-	*	*
4	**1.72**	*	*	*
8	**2.09**	*	*	*

Table 8. *Efficiency of nonwovens tested with respect to filtering E. coli bacteria in the air flow.*

Efficiency of nonwovens in filtering *E. coli*, %		
Melt-blown nonwoven with viroksan	Melt-blown nonwoven with magnesium monoperoxyphtalate	Control nonwoven without biocidal agent
98%	98%	98%

nitrate content in_polypropylene fibres with a biocidal agent, it was noted that the average content of biocidal particles in the four samples tested was 0.86 wt %, adjusted to active substances. The value above includes the total number of microbiocides, both permanently bound to the fibre and adsorbed on the surface.

The number of biocidal particles adsorbed on the surface that could be desorpted was calculated using the ***spectrophotometric method in ultraviolet.*** Four samples of melt-blown non-woven with a biocidal agent of around 20g in mass were weighed with an accuracy of up to 0.0001 g and extracted in 750 cm^3 of demineralised water of conductivity below 10 µS at a temperature of 35 ± 2 °C for 24 hours. Following extraction, the aqueous solution was transported quantitatively to the measurement flask, filled with water up to 1000 cm^3, the adsorption with methyl orange in ultraviolet was then measured, and on the basis of the model curve established, the concentration of microorganisms in the extract was evaluated.

It was noted that in the extraction conditions, 46% of biocidal particles included in polypropylene melt-blown non-woven with a biocidal agent undergo desorption. On the basis of the analyses carried out, it could be assumed that 54% of biocidal partiecles are permanently bound to the fibre, while the rest may undergo desorption. This, however, does not influence the efficacy of melt-blown activity, which was confirmed by the microbiological tests.

Conclusions

A technology and appropriate manufacturing equipment was developed for the production of bioactive polipropylene fibres devoted to melt-blown nonwovens used as air filtration.

From the point of view of requirements necessary to assure a high degree of individual respiratory system protection, a satisfactory result was obtained with respect to a new head construction which enabled a central dosage of bioactive agent to be applied in a symmetrical and controlled way to the fibre production zone.

Such a way of dosing the agent enables a proper mixture of fibres and bioactive agent, which is sucked into the stream of the fibres created and then evenly plotted onto the surface of the fleece formed.

As a result of microbiological tests, it was demonstrated that neither the addition of modifiers at the initial stage of forming melt-blown non-woven, nor spraying ready-made non-woven with a solution of bioactive agent produces satisfactory effects, because of difficulties in combining the modifier particles with the polymer granulate, in which the polymer flow temperature also has a negative influence on biocidal properties.

Acknowledgments

Publication prepared as part of project WKP 1/1.4.4/1/2005/11/11/576/2006/U, Entitled: 'New generation of filtering materials with nanofibres and modifiers' realised with the use of EU European Regional Fund as part of action 1.4 SPO-WKP.

References

1. *Wągrowska-Koski E.; The analysis of occupational diseases found in health protection workers between 1999-2001, Proceedings of Conference Safety of hospital work", Warszawa; 6-7 marca 2003 r.*
2. *Tadeusiak B., Bryska E.; The role of disinfection in workers' protection (in Polish), Proceedings of the 2nd Polish Scientific Educational Conference „ Work safety in a hospital; Warszawa 6-7.03.2003.*
3. *Dzierżanowska D., Jeliaszewicz J.; Hospital infections; Ed.: a-medica Press, 1999.*
4. *Bulanda M., Heczko P.; Hospital infections control; Rep. of International Federation of Infection Control & Polskie Towarzystwo Zakażeń Szpitalnych. Ed.: WMP, Kraków 1996.*
5. *Wenzel R., Edmund M., Pittet D.et al.; Hospital infections control ; Ed.: a-medica Press, 1999.*
6. *The 89/686/EU directive of Dec.21 1989 on uniformization of member states legislation concerning individual protective devices, Official Journal of the European Communities, L399, 30.12. 1989.*
7. *Ddirectiveof the Ministry of Economy of Poland concerning essential requirements for individual protective devices – Dec.21. 2005. Ed.: (Dz.U. Nr 259. Pos. 2173).*
8. *Standard EN-143:2000/A1:2006.*
9. *Standard EN-149:2001.*
10. *Technical University of Łódź, Patent PL 200059 B1, 2008.*
11. *Technical University of Łódź: Patents PL 179483 and PL 187392, 2006.*
12. *Technical Univesity of Łódź: Patent PL 174680, 2000.*

Received 11.10.2008 Reviewed 13.05.2009