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Antibacterial Air Filter Nonwovens Modified by Poly(Lactide) Microspheres Containing Triclosan

Abstract

The paper analyses the properties of filter nonwovens modified with triclosan-containing microspheres in order to obtain antibacterial effects. The carrier of triclosan consists of microspheres made of degraded polylactide (PLA) that gradually releases triclosan, providing a uniform dosing of the antibacterial effect. The incorporation of triclosan-containing microspheres into the structure of nonwovens was carried out by padding or spraying a bath containing 2.5% or 5% microspheres. The antibacterial effect and influence of the modification on the air permeability of the filter nonwovens were assessed. The stability of the antibacterial effect was assessed on the basis of simulation tests with the use of a test stand operating with an air conditioner. Analysis of the results obtained showed that the modification used allows one to attain very good antibacterial properties. Better effects can be obtained with the use of the padding technique than by spraying. The simulation tests of nonwoven padded with a bath containing 5% of microspheres and performed with the use of an air conditioner showed that the antibacterial effect was stable for 12 months.

Key words: microspheres, antibacterial, air filter nonwoven, triclosan.

Introduction

The possibility of changing air parameters in closed rooms by forced circulation under specific conditions of temperature and humidity has become the basis for the wide use of air-conditioning devices [1]. The area of air-conditioner application has also included car interiors [2 - 4], where it is essential that air-conditioners are equipped with air filters that filter out dust particles, smoke (also cigarette smoke) water vapour, soot etc. contained in atmospheric air supplied to the air-conditioner system [2]. On account of the air purity degree required obtainable from the air-conditioner system, filter systems containing coarse filters and/or fine ones are used [5]. Most frequently, the materials used to make a filter consist of filter nonwovens or their composites [5, 6]. The filtration efficiency of nonwovens considerably depends on the material structure resulting from the technology used. The diversity of the nonwoven structure is influenced by various parameters of fibres, their random arrangement and the manufacturing process parameters [7, 8].

Air also contains micro-organisms in the form of bacteria, viruses and fungi that constitute an additional hazard to human health. Air filters in air-conditioners can protect against these hazards by imparting antibacterial properties to the filter fabrics [8 - 11]. To impart a bioactive character to textile fabrics, several active substances such as biocides with an antimicrobe effect that differ in both their

chemical structure and action mechanism are used during the fabric's finishing processes [12, 13]. By incorporating a biocide into the structure of the fabric one can obtain bioactive properties in it. From among micro-biocides used in textiles, phenol compounds are of particular importance, especially a compound from the group of bisphenol micro-biocides: 2,2,4'-trichloro-2'-hydroxydiphenyl ether, and *triclosan*, which has been used for years in medicine and stomatology as a disinfecting and antiseptic agent, as well as in the production of cosmetics and sanitary agents [14]. This compound strongly binds to the surface of fibre, inhibiting the growth of microbes for a long time. The advantage of triclosan as a biocide is its low minimal inhibitory concentration (MIC), which reduces side-effects accompanying the use of each biocide [15].

One of the modern technologies of making products with new bioactive properties, uses the possibility of biocides reacting precisely as a result of their incorporation into polymeric microspheres [16 - 18]. The biocide carrier is a degradable polymer that gradually releases the biocide, providing its uniform dosage [18]. The incorporation of degradable microspheres containing a biocide into the structure of fabric allows one to obtain antibacterial effects with prolonged action.

The modification of nonwovens with triclosan in microspheres of polylactide has been reported in several previous papers

[18, 21, 22]. The subject of these studies was to establish optimal parameters of microspheres and conditions of their incorporation into a nonwoven structure [18 - 21].

The subject of the research presented in this paper are nonwovens used in air filters. The aim of our work is the analysis of antibacterial effects obtained by the modification of selected variants of filter nonwovens using microspheres with triclosan.

Experimental

Materials

Microspheres containing triclosan - spherical shells with a diameter of 3 μ m, made of degradable poly(L-lactid), filled with 5% triclosan to be released after the shell degradation were used [20]. Air filter nonwovens made by various techniques were selected for the study. Characteristics of the filter nonwovens selected for the modification are listed in *Table 1*.

Modification of nonwovens

Nonwoven porosity was not tested but will be included in future studies.

Methods of incorporating triclosan-containing microspheres into the textile substrate: *Padding* - double immersion of the textile substrate was performed in a modifying bath with the use of a two-roller padding machine from BENZ-

Switzerland, followed by drying at 70 °C in a laboratory drier from Mathis.

Spraying of the nonwovens was carried out by means of laboratory spraying test stand equipment of own design. It consists of a guide to which is mounted a spraying pistol connected to the compressor. The operator panel software allows you to adjust the parameters of spraying. We used the following conditions of spraying:

- Spraying rate of the modifying bath: 1 ml/dm²,
- Nonwoven travel rate under the spraying jet: 100 mm/s,
- Reducer pressure: 0.3 MPa >
- Heating temperature: 60 - 70 °C.

The modifying bath contained the following agents:

- Bioactive agent – an aqueous suspension of poly(DL lactide) containing 5% of triclosan prepared for the experiments. The concentration of microspheres in the modifying bath was 2.5% or 5%.
- Binding agent - Helizarin Binder TX 4738 from BASF (Germany) - 10 g/100 ml of the modifying bath.
- Cross-linking agent - Helizarin Fixing Agent TX 4737 from BASF (Germany) – 2 g/100 ml of the modifying bath.
- Wetting agent: Fluowet - 0.35 g/100 ml of the modifying bath.

The optimal modifying bath composition was determined in early works [23]. According to this experiment, a modifying bath containing 2.5% of microspheres was chosen during preliminary tests. The padding of samples was selected as the preliminary method due to better stability of microspheres in the structure. A detailed explanation of the parameters of tests and methodology assumed is presented in the section “Results and discussion”.

Modified nonwovens

The following samples were prepared:

- Ap 2.5% - padded with a modifying bath containing 2.5% of microspheres
- Bp 2.5% - padded with a modifying bath containing 2.5% of microspheres
- Cp 2.5% - padded with a modifying bath containing 2.5% of microspheres
- Cp 5% - padded with a modifying bath containing 5% of microspheres
- Cs 2.5% - sprayed with a modifying bath containing 2.5% of microspheres

Table 1. Parameters of filter nonwovens; PP - Polypropylene, PET - Polyester, *Mean, **Standard deviation.

Non-woven	Material	Production technique	Application area	Linear density of fibre, dtex	Surface weight, g/m ²		Thickness, mm	
					M*	SD**	M*	SD**
A	PP	Needling	Air-conditioner	21.84	187	12.3	4.54	0.13
B	PET	Spunlace	Face mask filtration	1.78	189	5.22	1.07	0.40
C		Spunbonded	Cabin air filtration	7.84	101	6.10	0.80	0.06

Methods

Assessment of antibacterial effects

The effect of the triclosan-containing microspheres on the fabric antibacterial activity, depending on the type of textile raw material and modifier application technique, was assessed by means of microbiological tests according to Standard PN – EN ISO 20645 Textile fabrics. Determination of antibacterial activity was undertaken by the Agar diffusion plate test using the method of diffusion on a plate with agar and test bacteria: *Staphylococcus aureus* (ATCC 6538) and *Klebsiella pneumoniae* (ATCC 4352). For each bacterium, two fabric samples were prepared on both sides of the nonwoven (4 in total) along with two reference samples (a textile substrate with the same composition but without triclosan). The level of antibacterial activity in this method is assessed by determining the growth of bacteria or their lack within the contact zone between the agar nutrient and sample tested, and measuring the size of the retardation zone of bacterial growth around the sample.

Microscopic observations

Images of the nonwovens before modification are shown by means of photos in transmitting light, taken with the use of a stereoscopic microscope - SZ60 from the Olympus Company (Japan) at a 50× magnification.

Microscopic assessment of the modified nonwovens surface was made by means of a VEGA3 TESCAN (Czech Republic) scanning electron microscope (SEM) at a magnification of 500×. The surface of each specimen was subjected to gold vacuum sublimation.

Functional tests

Simulation functional tests of the antibacterial effect stability of the modified nonwovens were carried out with a test stand on the horizontal discharge hood of an air conditioning unit - HIROSS HI-FRAME OVER (Italy), used to maintain a standard climate (20° C, 65% RH). The air stream blown out by the conditioner, at a rate ranging from 2.0 to 2.8 m/s,

passed through the surface of the sample fixed on its grids (*Figure 1*).

Microbiological tests with samples taken from the test stand device were performed according to the schedule of testing time established.

Results and discussion

The incorporation of triclosan-containing microspheres into the fabric structure during the modification processes causes a change in some properties of the nonwoven. Comparison of parameters listed in *Table 2* with those of the nonwovens before modification (*Table 1*) shows that regardless of the type of nonwoven defined by the technique of its making (Ap_{2.5%}, Bp_{2.5%}, Cp_{2.5%}) and the incorporation procedure (Cp_{2.5%}, Cs_{2.5%}),

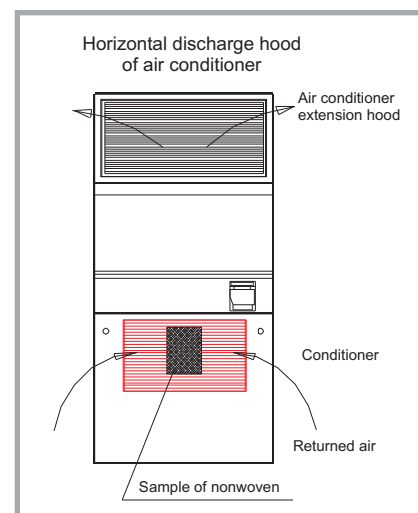


Figure 1. Test stand on the air conditioner.

Table 2. Parameters of nonwovens after modification; *Mean, **Standard deviation.

Samples	Surface mass, g/m ²		Thickness mm	
	M*	SD**	M*	SD**
Ap 2.5%	205	3.54	4.33	0.12
Bp 2.5%	212	10.03	1.08	0.05
Cp 2.5%	109	4.00	0.83	0.06
Cp 5.0%	123	1.78	0.77	0.06
Cs 2.5%	110	6.62	0.81	0.45

Table 3. Changes in the air permeability of modified nonwovens; *Mean,**Standard deviation. ^Air permeability – the velocity of air passing perpendicularly through a given area of fabric measured at a given pressure difference across the fabric test area (100 Pa, 200 Pa) over a given period of time. PN-EN ISO 9237 Textiles - Determination of permeability of fabrics to air. ^^Dap in % - Decrease in the nonwoven's air permeability after modification.

Samples	Air permeability- 100 Pa, mm/s [^]				Air permeability decrease, %
	Raw nonwovens		Modified nonwovens		
	M*	SD**	M*	SD**	Dap^^
Ap 2,5%	3546	148	3178	89.4	10.4
Bp 2,5%	369	13.8	304	12.1	21.4
Cp 2,5%	2518	406	2266	411	11.4
Cp 5,0%	2518	406	2235	303	13.9
Cs 2,5%	2518	406	2388	251	5.2

modification with microspheres does not change the thickness and surface mass of the nonwovens. An insignificant increase in the fabric surface mass can be observed in the case of increased microsphere concentration in the modifying bath (Cp_{2.5%}, Cp_{5%}).

One of the basic parameters for filtration process description is pressure loss in the air flow through the filter (flow resistance). A pure filter static pressure drop, at a nominal air flow intensity, is given in Pa [24]. In our work we used the method

of determining the air permeability described in PN-EN ISO 9237, which assumes that the pressure drop is fixed and the flow intensity is measured. In order to assess the effect of the modification of the nonwoven structure, measurements of air permeability were carried out before and after the deposition of microspheres on the nonwovens (Table 3). The results presented in Table 3 show that the difference between air permeability for the modified and raw nonwovens depends on the nonwoven structure (fibre thickness, modification technique).

Microscopic images of the structures of the filter nonwovens tested (Figure 2) clearly show these differences.

The considerable compacting of thin fibres in nonwoven B made by the spunlace technique causes a clearly lower air permeability. Nonwovens A and C, despite clear differences in fibre thickness and the technique of making, are characterised by similar air permeability values. The looser structure of these nonwovens and the clearances occurring allow considerably greater amounts of air to pass.

The influence of the nonwoven structure is also visible in the decreased air permeability (Dap %) after modification. The greatest decrease is shown by nonwoven B. Nonwovens A and C, padded with a bath containing 2.5% of microspheres (Ap_{2.5%} and Cp_{2.5%}) show the air permeability decreased by about 11-14%. An increase in the concentration of microspheres up to 5% in sample Cp_{5%} causes an insignificant decrease in air permeability. Comparison of the padded and sprayed samples (Cp_{2.5%} and Cs_{2.5%}) shows a clear effect of the technique of incorporating microspheres into the non-

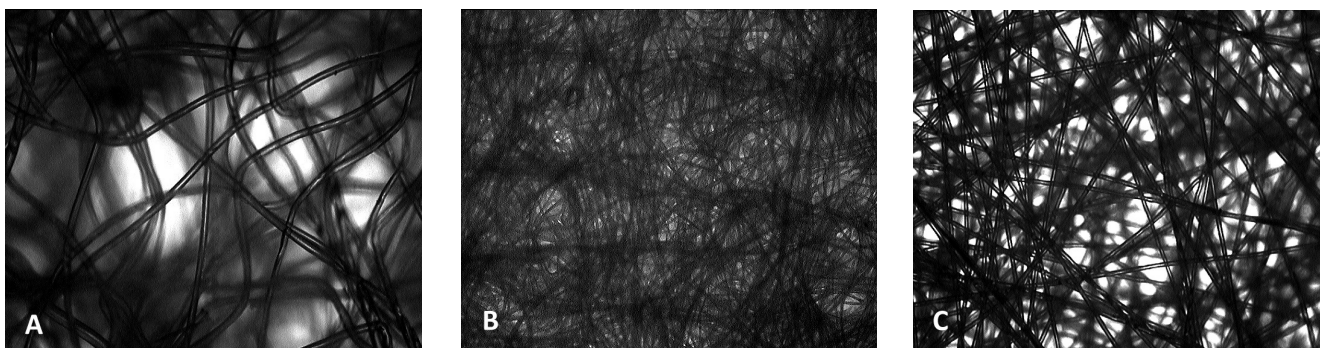


Figure 2. Microscopic images of the nonwoven structure in transmitted light (magnitude 50×) (A, B & C - samples described in Table 1).

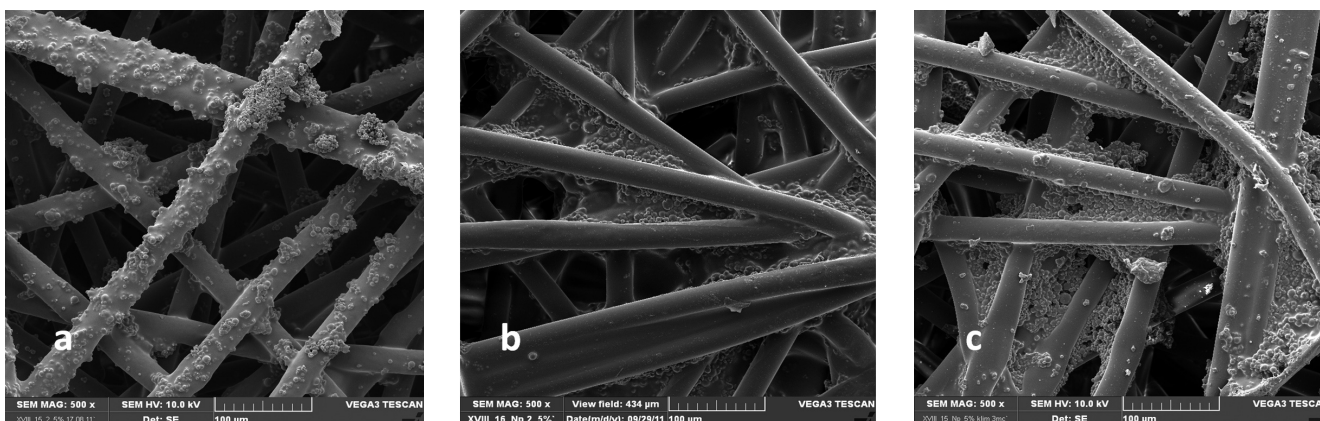


Figure 3. Comparison of the nonwoven structures after modification (SEM images: magnitude 500×); a) Cs_{2.5%}; b) Cp_{2.5%} & c) Cp_{5.0%}.

woven structure on air permeability. Despite similar weight increases indicating a lack of significant differences in the quantitative deposition of microspheres (**Table 2**), the sprayed sample is characterised by a considerably lower decrease in air permeability. Changes in the structure of sample C, modified by spraying and padding (shown in **Figure 3**, explain the smaller drop in air permeability of the sprayed nonwoven, in which the deposition of microspheres occurs on the surface of fibres. The padding method allows the bath with microsphere to penetrate the structure space, forming a membrane that partially closes the spaces between fibres.

The main aim of the modification of filter nonwovens was to obtain antibacterial properties.

Primary evaluation of the effectiveness of the antibacterial modification was determined according to PN - EN ISO 20645 Textile fabrics: ‘Determination of antibacterial activity’. This is a qualitative evaluation method which defines the product as antibacterial when the inhibition zone is > 1 mm. In this work the durability of modification effects was assessed by an analysis of the size of the inhibition zone. Measurements were performed on two samples on both sides, hence we have only 4 test results, which is not sufficient to use statistical methods.

Taking into account the fact that according to the standard used (PN – EN ISO 20645), the assessment criteria recognise the inhibition zone at a level of 1 mm as good antibacterial properties, the results listed in **Table 4** confirm that the modification of nonwovens with the triclosan-containing microsphere allows one to ob-

Table 4. Antibacterial activity of filter nonwovens modified with triclosan-containing microspheres.

Sample	<i>Klebsiella pneumoniae</i> (ATCC 4352)	<i>Staphylococcus aureus</i> (ATCC 6538)
	Inhibition zone, mm	
Ap 2.5%	7	8
Bp 2.5%	6	7
Cp 2.5%	9	9
Cp 5.0%	9	12
Cs 2.5%	6	7

Table 5. Antibacterial activity of the filter nonwovens modified, assessed after the simulation test.

Sample	<i>Klebsiella pneumoniae</i> (ATCC 4352)			<i>Staphylococcus aureus</i> (ATCC 6538)		
	Time of exposure (months)					
	0	1	6	0	1	6
Inhibition zone, mm						
Cp 2,5%	9	4	1	9	5	2
Cp 5%	9	6	5	12	7	6
Cs 2,5%	6	5	0	7	6	1

tain very good antibacterial effects of all the nonwoven samples tested. The best results of antibacterial activity were obtained for the spunbonded polyester nonwoven (Cp 5.0%) modified by the padding method. Spraying incorporates relatively few microspheres located on the surface of fibres due to the one-sided and spot-deposition on nonwovens. The padding method causes the microsphere bath to penetrate the structure space, forming membranes that hold the microspheres with triclosan.

The gradual release of triclosan caused by the degradation of polylactide under special conditions of dynamic air flow through the filter nonwoven requires one to determine the antibacterial efficiency of the modified filter nonwovens in practical use. In order to do that, end simulation tests were carried out with the use

of the test stand on the air conditioner as described in the section “Methods”.

For these tests, nonwoven C modified with microspheres of different concentrations with the use of different techniques of incorporating the microspheres into the nonwoven structure was selected. The results of the tests performed after a period of six months are listed in **Table 5**. The results presented indicate that after a testing period of 6 months, the nonwoven modified with a bath containing 2.5% of microspheres with triclosan by both the padding and spraying methods show a decrease in the size of their inhibition zone to a level of (Cp 2.5%) or more clearly to (Cs 2.5%), i.e. total loss of antibacterial properties.

A little higher decrease in the inhibition zone size of the sprayed nonwoven can be justified by the greater flow of air blowing out single microspheres from

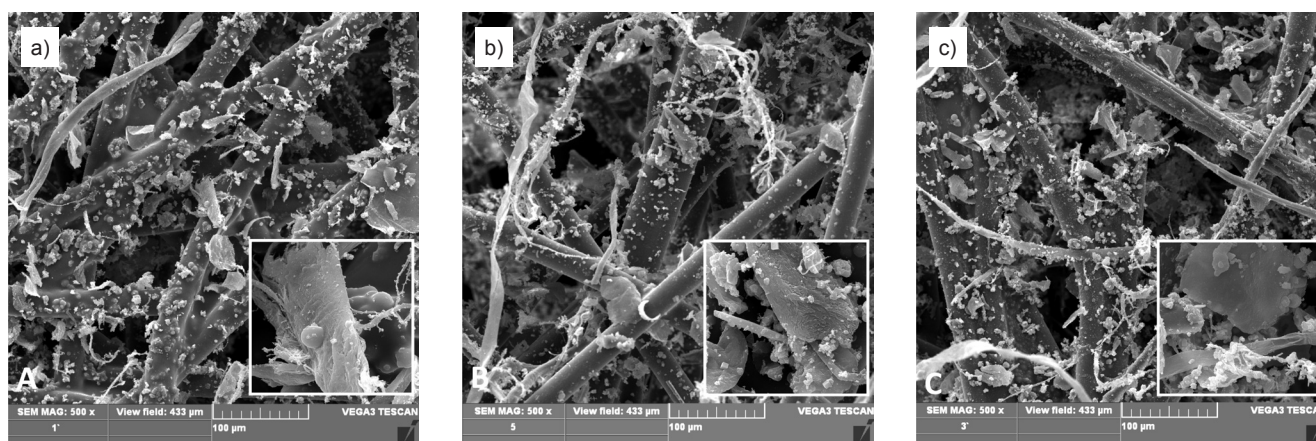


Figure 4. SEM images of nonwoven after 3 months of exposure (magnitude 500×, selected area 5000×): a) Cs 2.5%, b) Cp 2.5%, c) Cp 5.0%

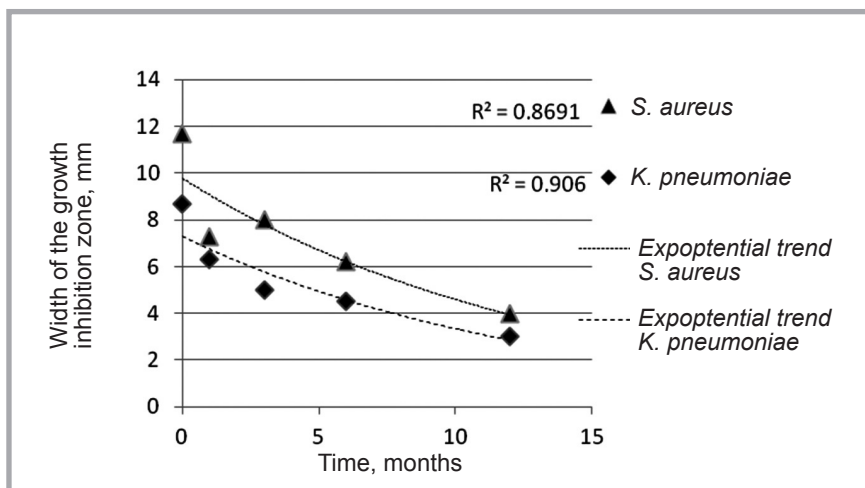


Figure 5. Change in the antibacterial effect as a function of time.

the nonwoven surface. The membranes formed between fibres in the padded nonwovens inhibit the flow, which is confirmed by the comparison of the decrease in air permeability Dap (Table 3).

SEM images of nonwovens after 3 months of exposure on the test stand are shown in Figure 4.

Photos of these nonwovens after functional tests by means of the air conditioner show a considerable quantity of impurities that obscure the microspheres with triclosan deposited. An increase in the concentration of microspheres up to 5% in the padding bath considerably increases the stability of the antibacterial effect in time. The microbiological test results of sample Cp_{5%} for which the simulation tests were continued for 12 months are shown in Figure 5.

Figure 5 shows a systematic decrease in the inhibition zone of bacterial growth within the testing time, maintaining a very good level of 3 mm and 4 mm for bacteria *Klebsiella pneumoniae* and *Staphylococcus aureus*, respectively. This indicates that this modified nonwoven, designed for car filters, will maintain its antibacterial properties for a period of 1 year. Thus to obtain the stability of these properties under use conditions in air conditioners and ventilation devices, one should use an increased concentration of triclosan-containing microspheres of up to 5.0%.

Conclusion

The examinations and tests performed showed that the modification of filter nonwovens by incorporating polylactide

microspheres containing triclosan into the fabric structure makes it possible to obtain the antibacterial effects expected. The measurement results of bacterial growth inhibition zones ranging from 6 to 9 mm obtained in microbiological tests indicate very good antibacterial properties of all the nonwovens modified. It was shown that the technique of incorporating the microspheres into the nonwoven structure has an influence on the effect obtained. Better results of the bacteria growth inhibition zones and the stability of the antibacterial effect can be obtained by the padding method. The spraying method deposits relatively few microspheres on the nonwoven surface due to the one-sided and spot-deposition. The padding method causes the microsphere bath to penetrate the structure space, forming membranes holding the microspheres with triclosan. The uniformity of the distribution of microspheres of triclosan in the structure of the nonwoven is a very important problem and is planned as the subject of further research. Similarly one can explain the smaller decrease in the air permeability of nonwovens after modification in Dap %. Microscopic images show a distinct influence of the structure of both the raw and modified nonwovens. Simulation tests of the antibacterial effect's stability on the measurement stand showed that after a testing period of 6 months, the nonwovens modified with a bath containing 2.5% of microspheres with triclosan by both the padding and spraying methods show a decrease in the size of their inhibition zone to a level close to the loss of antibacterial properties. An increase in the concentration of microspheres with triclosan to 5% during the padding pro-

cess increases the durability of the antibacterial effect to more than 12 months.

Taking into account the fact that the exchange period for a car filter is one year, the practical functional effect of the modification of filter nonwoven can be fully recognised.

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INSTITUTE OF BIOPOLYMERS AND CHEMICAL FIBRES

LABORATORY OF BIODEGRADATION

The Laboratory of Biodegradation operates within the structure of the Institute of Biopolymers and Chemical Fibres. It is a modern laboratory with a certificate of accreditation according to Standard PN-EN/ISO/IEC-17025: 2005 (a quality system) bestowed by the Polish Accreditation Centre (PCA). The laboratory works at a global level and can cooperate with many institutions that produce, process and investigate polymeric materials. Thanks to its modern equipment, the Laboratory of Biodegradation can maintain cooperation with Polish and foreign research centers as well as manufacturers and be helpful in assessing the biodegradability of polymeric materials and textiles.

The Laboratory of Biodegradation assesses the susceptibility of polymeric and textile materials to biological degradation caused by microorganisms occurring in the natural environment (soil, compost and water medium). The testing of biodegradation is carried out in oxygen using innovative methods like respirometric testing with the continuous reading of the CO₂ delivered. The laboratory's modern MICRO-OXYMAX RESPIROMETER is used for carrying out tests in accordance with International Standards.



The methodology of biodegradability testing has been prepared on the basis of the following standards:

- **testing in aqueous medium:** 'Determination of the ultimate aerobic biodegradability of plastic materials and textiles in an aqueous medium. A method of analysing the carbon dioxide evolved' (PN-EN ISO 14 852: 2007, and PN-EN ISO 8192: 2007)
- **testing in compost medium:** 'Determination of the degree of disintegration of plastic materials and textiles under simulated composting conditions in a laboratory-scale test. A method of determining the weight loss' (PN-EN ISO 20 200: 2007, PN-EN ISO 14 045: 2005, and PN-EN ISO 14 806: 2010)
- **testing in soil medium:** 'Determination of the degree of disintegration of plastic materials and textiles under simulated soil conditions in a laboratory-scale test. A method of determining the weight loss' (PN-EN ISO 11 266: 1997, PN-EN ISO 11 721-1: 2002, and PN-EN ISO 11 721-2: 2002).



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The following methods are applied in the assessment of biodegradation: gel chromatography (GPC), infrared spectroscopy (IR), thermogravimetric analysis (TGA) and scanning electron microscopy (SEM).

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