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Triclosan Encapsulated in Poli(L,L-lactide) as a Carrier of Antibacterial Properties of Textiles

Abstract

The subject of this paper covers research on the application of encapsulated biocides to achieve antibacterial textile products. Nonwoven with incorporated in its structure Triclosan, encapsulated in biodegradable polylactide, was the material of this research work. The influence of selected parameters of encapsulation and conditions of micro-sphere incorporation into nonwoven structure on the microbiological effect is also described as. Due to a inter-disciplinary approach, the research work was performed by Textile Research Institute in co-operation with the Centre of Molecular and Macromolecular Studies in Łódź the Adam Mickiewicz University in Poznań and the Technical University of Łódź.

Key words: encapsulated biocides, micro-spheres, triclosan, bio-active textiles, nonwovens.

Introduction

The development of research on materials based on biodegradable polymers allows to produce textiles of enhanced antibacterial properties. One of the possibilities is making use of controlled release of bioactive substances from polymer substrates. Controlled release of bioactive substances encapsulated in micro-capsules and in polymer micro-spheres introduced to textiles opens new directions of applications for textiles [1 - 4].

The antibacterial effect of textile fabric, containing micro-spheres with biocide, depends on the ability to release a defined amount of biocide in wearing conditions. This amount depends on micro-spheres parameters, biocide content as well as on the amount of micro-sphere dispersion and its concentration after having been introduced on a textile substrate [5 - 7]. The essential factor for the assessment of the effect obtained is the kinetics of

biocide release, which characterises the efficiency and durability of antibacterial properties.

The research work described in this paper is the continuation of studies on the application possibilities of the precise dosing of biocides encapsulated in polymer micro-spheres introduced onto a textile substrate [8].

The problem of durable incorporation of micro-spheres onto a textile substrate has not yet been solved on a technological scale.

Earlier laboratory tests [8] showed that the best results are obtained by the spraying technique. To continue this study, a laboratory stand for spraying the dispersions of polymer micro-spheres onto textiles under pressure was developed at the Textile Research Institute (IW) [9, 10].

The spraying device can automatically regulate:

- the amount of suspension introduced, and
- the speed of shifting the spraying jet and the substrate carrier.

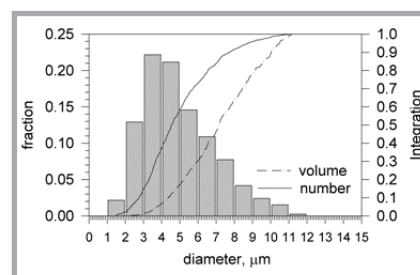


Figure 1. Exemplary histogram of micro-spheres size distribution (variant III'); $N = 789$, $D_{min} = 1.4482 \mu m$, $D_{max} = 11.1586 \mu m$, $D_n = 4.9373 \mu m$, $D_w = 7.1721 \mu m$, $D_w/D_n = 1.4526$.

The temperature and speed of sample drying can also be controlled. The chosen method of incorporating micro spheres into a textile substrate not only allows for surface deposition, but also suspension penetration into deeper levels of a nonwoven structure. Microscopic analysis proved that the energy of suspension droplets permits deep penetration into a substrate structure.

As in previous tests, cellulosic - viscose

Table 1. Micro-spheres parameters.

Sample	Number average diameter D_n , μm	Maximal diameter D_{max} , μm	Minimal diameter D_{min} , μm	Weight average diameter D_w , μm	Fraction of micro-spheres with diameter	Polydispersity D_w/D_n	Triclosan contents in dry particles, %
					$d < 2 [\mu m]$		
I	1.78	7.0	0.01	5.53	75	3.11	3.8
II	1.31	5.5	0.01	2.75	89	2.09	7.9
III	1.35	5.0	0.01	3.59	82	2.65	15.3
IV	1.14	6.5	0.01	3.68	91	3.24	28.5
					$d < 5 [\mu m]$		
III'	4.94	11.16	1.44	7.17	59	1.45	13
					$d < 25 [\mu m]$		
IV'	24.3	42.6	9.72	30.2	50	1.24	13

nonwoven, produced according to spun-laced technique, was the textile substrate in our study. Poly(L,L-lactide) micro-spheres containing triclosan were the source of rendering antibacterial properties.

The aim of this research was the determination of the influence of selected factors on biocide release kinetics and on antibacterial activity acquisition by textiles.

Former studies indicated that the antibacterial effect of textile substrate depends on:

- the parameters of micro-spheres with encapsulated biocide – micro-sphere size coefficients, unevenness coefficients and biocide amount enclosed in micro-spheres,
- the conditions of micro-sphere incorporation onto a textile substrate - micro-sphere concentration in the dispersion and incorporation of the technical parameters.

The tests performed and results of former studies were the base to analyse the effect obtained of:

- triclosan deposition and its release from a nonwoven substrate, and
 - microbiological effect
- on:
- micro-sphere size
 - triclosan concentration in dispersion
 - contribution of triclosan concentration in micro-spheres and that of micro-sphere concentration in dispersion, and
 - environmental humidity.

Experimental

Micro-sphere parameters

To produce active micro-spheres (as described in earlier work [10]), triclosan was encapsulated with poly(L,L-lactide).

One of the characteristic features of poly(L,L-lactide) is its ability to biodegrade – depolymerise to non-toxic products under the influence of factors included in living organisms. Hence, it is often used as a carrier of biologically active substances. During the biodegradation process – a biologically active substance is released from the carrier; the release rate depends on the biodegradation rate. The same scheme was applied in this work. The aim of this study was the attainment of nonwoven fabric with a prolonged antibacterial effect.

Microspheres loaded with triclosan were obtained by the emulsion solvent evaporation method. Both poly(L,L-lactide) and triclosan were dissolved in dichlo-

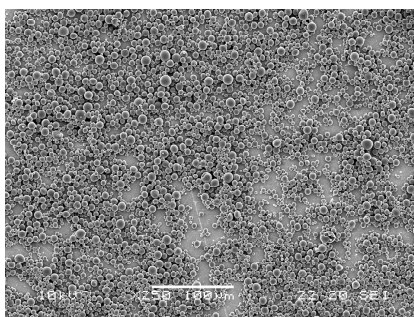


Figure 2. SEM image of micro-spheres – average diameter 4.9 μm .

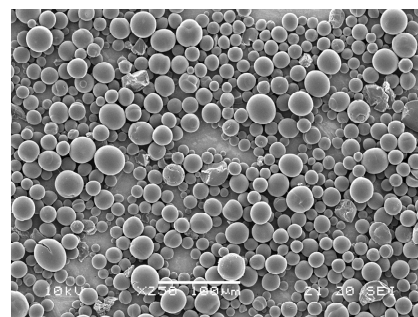


Figure 3. SEM image of micro-spheres – average diameter 24.00 μm .

romethane and instilled into poly(vinyl alcohol) aqueous solution under very strong mechanical stirring. Next, the emulsion formed was slowly stirred to evaporate the dichloromethane.

Histograms based on the images taken by electron scanning microscope – JEOL-JSM5500C allowed to define micro-sphere size [8]. **Figure 1** presents an exemplary histogram of micro-sphere size distribution.

Table 1 lists micro-spheres parameters of the variants of encapsulation analysed.

The data allow to differentiate 2 series of micro-spheres:

- of various sizes and constant triclosan content (variant III' and IV')

- of various triclosan content and uniform micro-sphere size.

Achieving uniformity in the size of micro-spheres and in doubled content of triclosan in consecutive variants were the stipulations of variants I – IV. The data presented in this table allow to state that, due to low and comparable polydispersity coefficients, we can assume that differences in the parameters of the variants analysed are very small and hence can be neglected. This is confirmed by the quantitative share – above 75% - of micro-spheres of a diameter smaller than 2 μm .

The real content of triclosan determined, presented in **Table 1**, indicates that the stipulations were obtained to produce

Table 2. Characteristics of variants of different triclosan concentration.

Variant	Micro-sphere diameter D_n , μm	Triclosan contents in micro-spheres dry matter C_{tm} , %	Micro-spheres concentration in dispersion C_{mz} , %	Triclosan contents in dispersion C_t , %
I	1.78	3.8	10	0.38
II	1.31	7.9	5	0.395
III	1.35	15.3	2.5	0.372
IV	1.14	28.5	1.25	0.356
III'1	4.9	13.5	2.5	0.337
III'2	4.9	13.5	5	0.675
IV'1	24.3	13.5	2.5	0.337
IV'2	24.3	13.5	5	0.675

Table 3. Nonwoven samples characteristics; * Real value determined by chemical extraction method and UV spectroscopy.

Sample	Micro-spheres diameter D_n , μm	Concentration of micro-spheres in dispersion C_{mz} , %	Triclosan contents in dispersion* C_{mz} , %	Total triclosan amount in nonwoven* W_{ct} , %
W / I	1.78	10	0.38	1.3
W / II	1.31	5	0.395	1.8
W / III	1.35	2.5	0.372	1.9
W / IV	1.14	1.25	0.356	1.8
W / III' A	4.9	2.5	0.337	2.0
W / III' B	4.9	5	0.675	2.4
W / IV' E	24.3	5	0.675	2.9

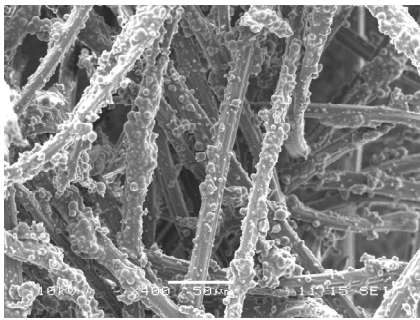


Figure 4. SEM image of nonwoven structure with introduced micro-spheres (variant W/I – 10% concentration of dispersion).

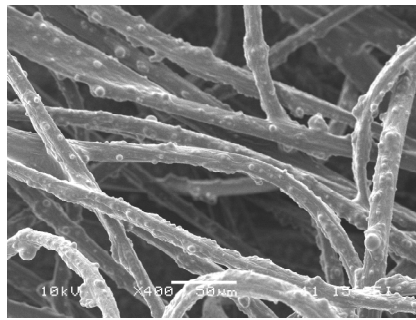


Figure 6. SEM image of nonwoven structure with introduced micro-spheres (variant W/III – 2.5% concentration of dispersion).

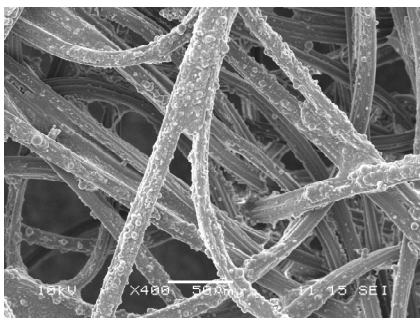


Figure 5. SEM image of nonwoven structure with introduced micro-spheres (variant W/II – 5% concentration of dispersion).

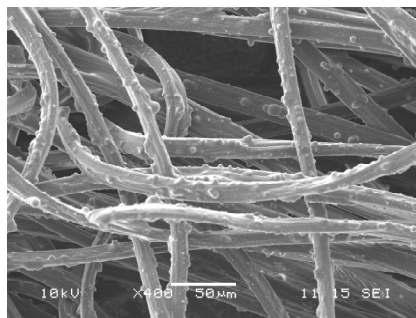


Figure 7. SEM image of nonwoven structure with introduced micro-spheres (variant W/IV – 1.25% concentration of dispersion).

differentiated variants I – IV. Apart from analytical assessment, microscopic images were taken of micro-sphere variants I – IV. These images showed that mi-

cro-spheres of these variants have similar character and do not differ substantially. **Figures 2 and 3** (see page 103) present microscopic images of micro-sphere var-

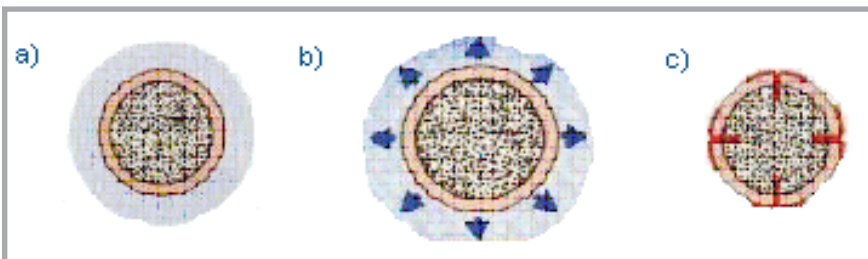


Figure 8. The procedure of triclosan extraction from modified nonwoven; a) total amount of triclosan in nonwoven – extracted by (CH_2Cl_2) [W_{ct} %]; b) triclosan released from micro-spheres onto nonwoven surface – extracted by (C_6H_{14}) [$W_{ct} \cdot W_{tm}$ %]; c) the rest of triclosan in micro-spheres – extracted by CH_2Cl_2 (after extraction by C_6H_{14}) [W_{tm} %].

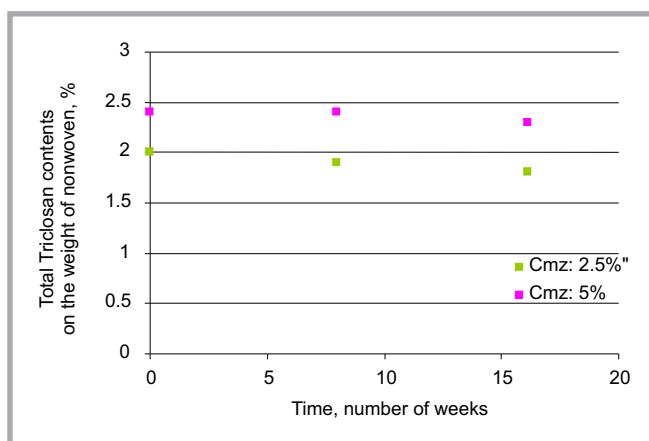


Figure 9. Diagram of triclosan release ($RH > 80\%$).

iants of different average values (variant III' and IV').

Preparing a dispersion with micro-spheres

The dispersion of micro-spheres in water with emulsating additives is introduced onto the textile substrate.

The total amount of Triclosan in this suspension C_t equals the product of Triclosan concentration in the micro-spheres C_{tm} and microsphere concentration in the suspension C_{mz} :

$$C_t = C_{mz} \cdot C_{tm}$$

The aqueous emulsion of poly(L,L-lactide) and the triclosan solution of defined micro-sphere concentration allowed to prepare different dispersion variants of the defined – assumed concentrations.

Table 2 (see page 103) lists concentration variants of microsphere dispersions and triclosan concentrations in microspheres.

In variants I – IV micro-sphere concentrations in dispersion were prepared in such a way to have triclosan contents in dispersion at the same level.

Micro-spheres incorporation onto nonwoven fabric

Viscose nonwoven of mass per unit area = 30 g/m², produced according to a spun-laced technique (water jet) called 'Hydronina 30' was used as the textile substrate for biocide in micro-sphere form.

The spraying method and lab spraying stand was used to introduce the dispersion under pressure onto the nonwoven structure. The parameters of spraying applied were the ones settled for a nonwoven of 30 g/m² processed on a lab spraying stand. Table 3 presents the characteristics of nonwoven samples with triclosan microspheres introduced.

Figures 4, 5, 6 and 7 present images of nonwovens with triclosan microspheres introduced.

The effect of changing the concentration of micro-spheres in dispersion can easily be observed. Moreover, these images indicate that the higher the concentration, the worse the uniformity of micro-spheres incorporation is. At concentrations above 5% the phenomenon of micro-sphere aggregation occurs, which causes weaker bonds amongst micro-spheres with fibres.

Assessment of triclosan contents in nonwoven

The aim of the physical-chemical tests was the determination of dispersion parameters (triclosan contents in micro-spheres, micro-sphere size and dispersion concentration) onto the amount of “free” triclosan which is released from micro-spheres. The relationship between “free” and encapsulated triclosan in textiles allows to predict the real amount of active triclosan and hence the degree of protection against microorganisms. The method of quantitative analysis of triclosan was elaborated for the purposes of this project by research workers of Adam Mickiewicz University in Poznań.

Quantitative analysis of triclosan contents in modified nonwoven covered the following: determination of the amount of triclosan released from polylactide and micro-spheres onto a nonwoven surface and the amount of triclosan in micro-spheres (**Figure 8**).

This analysis was based on UV-Vis spectroscopy.

To determine the amount of triclosan in polylactide micro-spheres and the amount of triclosan released onto a nonwoven surface, modified nonwoven was subjected to extraction in two solvents: hexane and dichloromethane. Both solvents are chemically neutral and do not absorb infrared radiation in the range in which maximum absorption of triclosan occurs, i.e. at $\lambda_{max}=278 - 280$ nm.

Hexane only dissolves “free” triclosan, whereas dichloromethane dissolves both triclosan and polylactide. Hexane extraction allows to estimate the amount of triclosan released, and dichloromethane extraction allows to determine the total triclosan content introduced onto a nonwoven surface. Dichloromethane extract contains triclosan released from micro-spheres as well as triclosan which remained in the micro-spheres (**Figure 8**).

Making quantitative analysis of the bio-active effect of viscose with encapsulated triclosan introduced, and of the amount of triclosan released, it must be noted that both depend on the parameters of the dispersion introduced, the polylactide biodegradation process and the conditions of the dispersion incorporation process onto a nonwoven structure.

The results obtained were used in an analysis of the influence of selected param-



Figure 10. SEM image of nonwoven with micro-spheres (variant I – half a year storing at RH 33%).

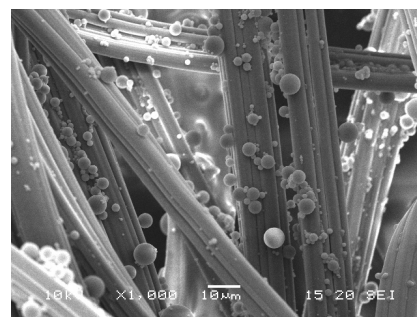


Figure 11. SEM image of nonwoven with micro-spheres (variant I – half a year storing at RH 98%).

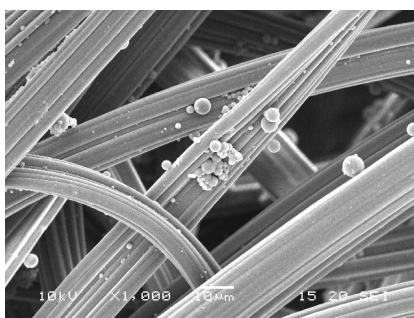


Figure 12. SEM image of nonwoven with micro-spheres (variant IV – half a year storing at RH 33%).

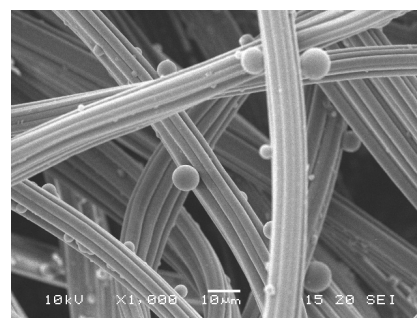


Figure 13. SEM image of nonwoven with micro-spheres (variant IV – half a year storing at RH 98%).

Table 4. Effect of time and storing conditions [humidity] of modified nonwovens on total triclosan contents and the amount of released triclosan.

Sample	Time, weeks	Total triclosan contents in nonwoven W_{ct} , %		Contents of released triclosan on nonwoven surface $W_{ct} - W_{tm}$, %	
		RH < 5 %	RH > 80 %	RH < 5 %	RH > 80 %
W/III'A $C_{mz, 2.5\%}$	0	2.0	-	0.1	-
	8	2.0	1.9	0.1	0.2
	16	2.0	1.8	0.1	0.4
W/III'B $C_{mz, 5\%}$	0	2.4	-	0.2	-
	8	2.4	2.4	0.2	0.2
	16	2.3	2.3	0.2	0.6

Table 5. Triclosan contents in nonwoven after 8-month-period; RH = 33%.

Sample	Total triclosan contents in nonwoven W_{ct} , %	Contents of released triclosan on nonwoven surface W_{uw} , %	Amount of triclosan which remained in micro-spheres W_{tm} , %
W I	1.3	0.5	0.8
W II	1.8	0.6	1.2
W III	1.9	0.7	1.2
W IV	1.8	0.6	1.2

eters of micro-sphere incorporated into nonwoven on the triclosan release effect.

Tests on triclosan contents at various time intervals and in different conditions of environmental humidity were performed to determine the kinetics of biocide release. **Table 4** and the diagram in **Figure 9** present the results of this assessment.

The results presented in **Table 4** and in **Figure 9** allow to state that:

- triclosan content does not change in dry environmental conditions (RH < 5%); this means that in the time interval, no visible degradation of polylactide microcapsules occurs
- in conditions of relative humidity > 80%, there occurs an increase in

the amount of triclosan released in the time function; this means that after ca. 4 months about 20% of microcapsules degraded

- changes in triclosan content in nonwoven as a function of time are proportional to the amount of encapsulated triclosan in dispersion.

Table 5 (see page 105) presents results of the chemical assessment of four sample variants which differ in triclosan content in their micro-spheres and in dispersion. The amount of triclosan, according to selected indexes, does not differ considerably in tested samples after a 8-month-period. The differences between samples are within the error limit, resulting from the accuracy of the triclosan determination method and the unevenness of micro-sphere dispersion introduced onto nonwoven.

It turns out that the amount of triclosan released does not depend on individual concentration parameters but on the total amount of triclosan introduced. The results indicate that after a 8-month-period since nonwoven modification, triclosan release from micro-spheres reaches 36%.

Qualitative assessment of antibacterial effect

Microbiological tests were performed according to AATCC test method 147-1998 – Antibacterial Activity Assessment of Textile Material: The parallel streak method,

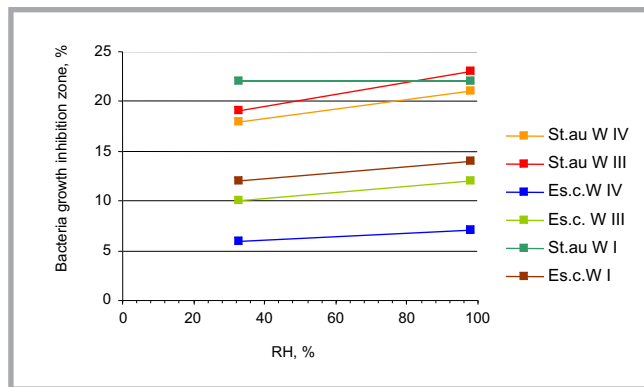
Table 6. Antibacterial effect – variants with different micro-spheres sizes.

Sample	Bacteria growth inhibition zone, mm	
	<i>s. aureus</i>	<i>e. coli</i>
[W III'B] Dn = 4.9 µm	77	40
[W IV'E] Dn = 24.3 µm	67	35

Table 7. Results of microbiological tests; **Explanation:** *W It*, *W IIIIt*, *W IVIt* – samples after half a year storing in exicator at RH 33%; *W It'*, *W IIIIt'*, *W IVIt'* – samples after half a year storing in exicator at RH 98%; *Wo* – nonwoven sample without triclosan.

Sample	Bacteria growth inhibition zone, mm	
	<i>s. aureus</i>	<i>e. coli</i>
W It	18	6
W It'	21	7
W IIIIt	19	10
W IIIIt'	23	12
W IVIt	22	12
W IVIt'	22	14
W 0	0	0

Figure 14. Influence of humidity onto microbiological activity of non-woven fabric.



od, using the following standard strains:

- *Staphylococcus aureus* - representing gram positive bacteria, and
- *Escherichia coli* – from the gram negative bacteria group.

Table 6 presents results of the antibacterial effect of samples of similar triclosan content, differing only in micro-sphere size.

It can be observed that a better antibacterial effect – for samples with similar triclosan content – was obtained for micro-spheres of smaller size.

To obtain information on the kinetics triclosan release, samples stored in different conditions (RH) and in different time intervals were subjected to microbiological tests. The results are presented in **Table 7**.

Figures 10, 11, 12 and 13 (see page 105) present SEM images of selected nonwoven variants (with micro-spheres) stored in different conditions (RH).

Comparison of images clearly indicates increased micro-sphere degradation in conditions of higher air relative humidity. It is especially evident in samples of high micro-sphere concentration in dispersion (W I) – **Figures 10 and 11**.

The humidity effect is proportionally weaker in sample W IV – images of samples of the lowest micro-sphere concentration in dispersion **Figures 12 and 13**. At the same time it can be clearly observed that higher humidity results in degradation of micro-spheres of smaller sizes – substantially fewer in **Figure 13** when compared to **Figure 12**.

This is also confirmed by the analysis of inhibition zones presented in diagram **Figure 14**.

This diagram presents a clear tendency of inhibition zone increase with the in-

crease in micro-sphere concentration in dispersion (comparison of samples W It and W IVt). Moreover, it must be emphasised that the level of antibacterial activity of all sample variants with triclosan substantially exceeds the requirements of the AATCC standard. According to this standard, the antibacterial effect is considered satisfactory when the inhibition zone is above 1 mm.

Summary

The results obtained indicate that the amount of triclosan introduced onto/into nonwoven depends on the product of triclosan concentrations in micro-spheres and micro-sphere concentration in dispersion.

- The amount of triclosan released does not depend on the parameters of individual concentration but on the total amount of triclosan introduced.
- The best effects of introducing dispersions with triclosan were achieved for micro-spheres of uniform size distribution and low concentration in dispersion.
- Higher concentration of micro-spheres in dispersion generates aggregates of micro-spheres and hence non-uniform micro-sphere distribution in nonwoven structure.
- A better microbiological effect for nonwovens containing the same amounts of Triclosan was achieved for micro-spheres of smaller size.
- Sample storage time in dry conditions (RH 33%) did not effect nonwoven bioactivity. In conditions of increased humidity (RH 98%) there occurred a visible increase in the antibacterial activity of the sample variants tested in the case of both bacteria strains. The higher the humidity, the higher the rate of polylactide degradation is.
- The high microbiological effect achieved – substantially above the requirements – indicates the possibility of reducing the amount of triclosan in

a nonwoven structure; this should be the subject of further studies.

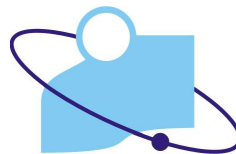
Acknowledgment

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The fee covers: participation in sessions, CD with abstracts and presentations, boarding, and social event. Hotel costs are not included.

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Additional information of the EL-TEX Symposium you will also find on pages 113 and 116.

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