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Polymer Microspheres as Carriers of Antibacterial Properties of Textiles: A Preliminary Study

Abstract
A significant part of medical, fibre-based materials are antibacterial textile fabrics which can be obtained by various advanced technologies. The application of new nano-technologies offers the possibilities of producing and implementing such products. In this work we present some preliminary tests concerning the irreversible immobilisation of poly(L,L-lactide) microspheres loaded with Triclosan onto viscose nonwoven structures. In the available publications concerning encapsulation techniques, there is no information on trials pertaining to the production of microspheres from poly(L,L-lactide) with Triclosan. The experiments described in this paper were divided into two parts: the synthesis of microspheres with Triclosan (carried out at the Polish Academy of Sciences, Łódź), and the attachment of microspheres to a textile fabric structure (carried out at the Textile Research Institute, Łódź). The results were quantified and the microbiological efficiency of the modified fabrics was examined.

Key words: *antibacterial textile, medical textile, microspheres, Triclosan.*

Introduction

Progress in the polymer sciences over recent decades has made it possible to produce advanced materials, especially for medical applications, pharmacy and diagnostics [1,2]. As a result of the significant share of textile fabrics in the medical area [3,4], the application of nanotechnologies is bound to increase in the near future [5]. The antibacterial property of textiles is an essential feature in many assortments of fabrics used in prophylaxis, medical treatment and hygiene. The possibility of the sensitive reaction of biocides closed in polymer microspheres incorporated in the textile base opens up the possibility of being applied for medical purposes.

In this work, we present some preliminary test results concerning the irreversible immobilisation of poly(L,L-lactide) (PLLA) microspheres loaded with Triclosan onto a viscose nonwoven structure. In the available publications concerning encapsulation techniques, no information is available on trials pertaining to producing microspheres from poly(L,L-lactide) with Triclosan. Independently of this, however, the aim of our investigation was to explore the possibility of obtaining nonwovens with antibacterial properties by incorporating microspheres including Triclosan. The experiments described in this paper were divided into two parts: the synthesis of microspheres with Triclosan (carried out at the Polish Academy of Sciences, Łódź), and attaching microspheres to a textile fabric structure (carried out at the Textile Research Institute, Łódź). The antibacterial agent Triclosan was chosen because it is widely used in cosmetics, soaps, detergents and finishing agents for textiles. Viscose nonwoven was used as the base textile material.

The scope of our research work covered:
- the production of a suspension containing microspheres loaded with Triclosan,
- tests on the bonding of microspheres with a base textile material,
- a quantitative assessment of the effect achieved (including microbiological tests).

Experimental

Synthesis of microspheres loaded with Triclosan

Poly(L,L-lactide) was chosen because of its ability to biodegrade to non-toxic products. This feature is very important for 'in vivo' applications, and poly(L,L-lactide) is widely used as a matrix for drug delivery systems. During the biodegradation process, a drug (or other biologically active substance) is delivered at a specific rate which is dependent on the biodegradation rate. This system was used in this work because of the prolonged period of Triclosan delivery.

Poly(L,L-lactide) with M_n=20,000 was synthesised by pseudoanionic ring-opening polymerisation [6-9] using aluminium tri-isopropoxide $Al(O\text{-}C_3H_7)_3$. As a solvent we used 1.4-dioxane dried over sodium-potassium alloy.

The microspheres were obtained by the solvent evaporation method described in [10]. The poly(L,L-lactide) and Triclosan were dissolved in methylene chloride, and were then slowly added to the sodium dodecylsulphate (SDS) aqueous solution. The mixture was sonicated to obtain smaller drops of polymer-triclosan solution, and dried over 48 hours. The microsphere suspension was centrifuged, and the particles were re-suspended into distilled water.

The amount of Triclosan in the microspheres was determined by UV spectrophotometry. The microspheres were dissolved in CH_2Cl_2, and the absorbance of Triclosan at 280 nm was measured. A calibration curve for Triclosan had been previously prepared using a few standard solutions of Triclosan in dichloromethane (Figure 1).

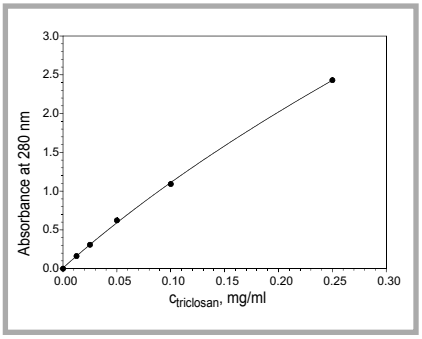

***Figure 1.** Calibration curve for Triclosan in CH_2Cl_2.*

Table 1. *Diameter of microspheres - number average and weight average.*

D_n	2.36 μm
D_w	8.69 μm
D_w/D_n	3.68
D_n(min)	0.54 μm
D_n(max)	16.60 μm

The amount of Triclosan in microspheres was calculated from the following formula:

$$\%Triclosan = \frac{C_{tricl} \cdot V}{m_m} 100\%$$

where:

C_{tricl} - the concentration of Triclosan determined from the calibration curve in %,

V - the sample volume in ml,

m_m - the mass of microspheres in the sample, in mg.

The Triclosan content was determined as 8.5% w/w. The particle size was measured using a Jeol JSM5500C scanning electron microscope and MultiScan image analysis software. Statistical analysis for more than 900 particles was applied. The results are collected in Table 1, where D_n - the numeric average diameter, D_n(min) - the numeric minimum diameter, D_n(max) - the numeric maximum diameter, D_w - the weight average diameter, and D_w/D_n - the ratio of the average diameter values. Figure 2 presents the particle size distribution, whereas Figure 3 shows an example picture of the microspheres distributed on a plate.

Considering that the assumed application of the product is for one-way use only, no fastness tests of the antibacterial effect were included in the investigation plan at this stage.

Immobilization of microspheres onto viscose nonwoven surface, and their biological activity

The viscose nonwoven was prepared using the spun-laced technique (mass per unit area of 65 g/m^2). The choice of this material at this stage of the research was based on its potential application areas (dentistry), and so on the specific requirements associated therewith. The material to be used in dentistry should be characterised by good liquid sorption capabilities, good softness and should not contain any bonding agents.

The microspheres with Triclosan were introduced into the viscose nonwoven

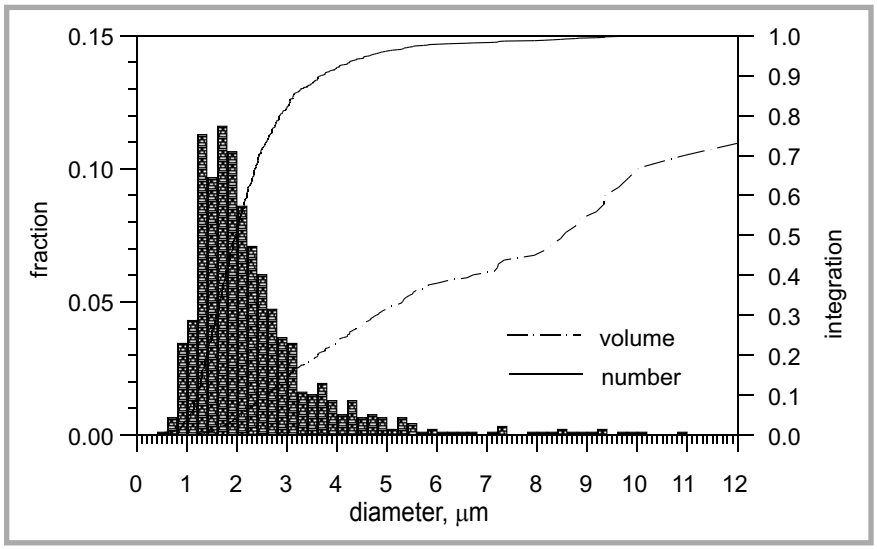

Figure 2. *Particle size distribution for PLLA microspheres loaded with Triclosan; the histogram presents the numeric diameter distribution.*

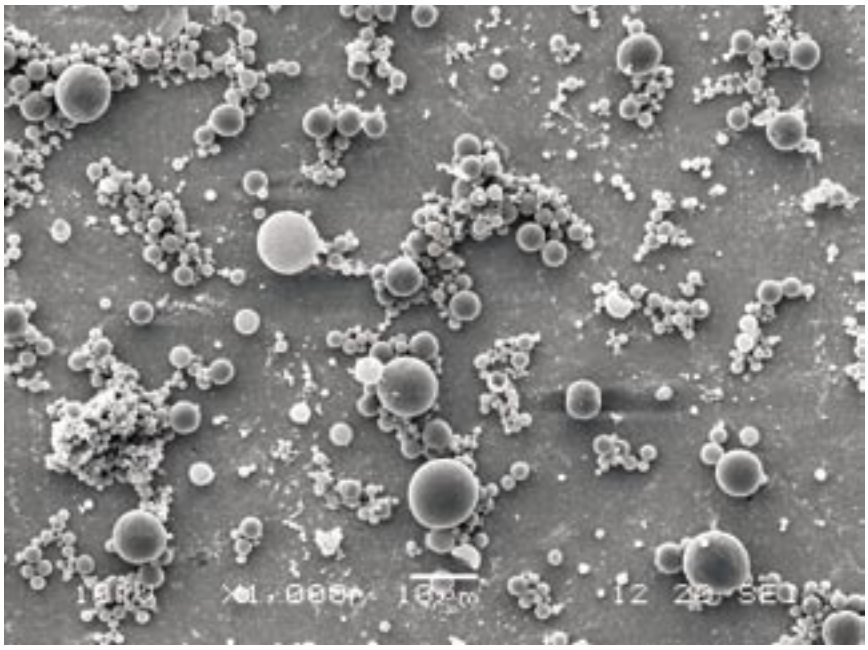

Figure 3. *Image of poly(L,L-lactide) microspheres loaded with the content of 8.5% of Triclosan distributed on a plate, obtained with the use of a Jeol JSM5500C scanning electron microscope.*

Figure 4. *Image of the viscose nonwoven with microspheres incorporated by the spraying method, obtained with the use of a Jeol JSM5500C scanning electron microscope.*

Figure 5. *Image of the viscose nonwoven with microspheres incorporated by the pouring method, obtained with the use of a Jeol JSM5500C scanning electron microscope.*

according to two methods:

- spraying the viscose nonwoven with the suspension of microspheres in distilled water with a spraying-gun (sample C),
- directly pouring the particle suspension onto the nonwoven surface (sample D).

The effectiveness of both methods is shown in Figures 4 and 5.

The comparison of results on the basis of these photographs shows that spraying is more efficient, as it allows larger amounts of microspheres to be introduced. The size of microspheres is differentiated. It also appears that the contribution of smaller particles is larger than is the case in the second method.

The comparison of the methods of incorporation was also made by determining the share (as % w/w) of microspheres in the sample mass after its drying. The amount of microspheres incorporated into each viscose nonwoven sample is shown in Table 2. The results presented in Table 2 confirm that the spraying method is more efficient; the microsphere contents in the nonwoven structure is greater.

We have also found that the microbiological activity is higher for the sprayed sample. The test results presented in Table 3 confirm the action of the introduced Triclosan. Microbiological activity was determined by using a standard procedure for medical textile fabrics with the use of an agar test [11]. Two samples prepared according to each method (spraying and pouring) were produced and subjected three times to the measurement of the inhibition zone. Microbiological tests indicate the high antibacterial efficacy of the Triclosan-loaded microspheres.

Table 2. *The content of microspheres in viscose nonwoven sample.*

Method	% w/w of microspheres particles
Spraying	18.6
Pouring	6.3

Table 3. *Microbiological activity of the samples with microspheres.*

Method	Inhibition zone diameter, mm	
	S. aureus	*E. coli*
Spraying	15.7	9.8
Pouring	12.9	8.5

Conclusion

The results described in this work show that a viscose nonwoven obtains microbiological activity caused by incorporation of immobilised particles loaded with Triclosan. These are only initial studies, but they open up the possibility of developing new technologies fpr medical textiles. However, further investigations are required, such as:

- techniques for incorporating microspheres other than those used in this investigation,
- optimising material characteristics - the percentage of Triclosan-loaded microspheres in a suspension, optimal from the point of view of the method's efficiency, and
- tests of other active substances and other base materials, depending on the application requirements.

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