Determination of the resistance of fabric printed with triclosan microcapsules to the action of soil micro-flora

B Golja and P Forte Tavčer
University of Ljubljana, Faculty of Natural Sciences and Engineering, Department of Textiles, Graphic Arts and Design, Aškerčeva 12, 1000 Ljubljana, Slovenia

Email: barbara.golja@ntf.uni-lj.si

Abstract. Microcapsules with a pressure-sensitive melamine-formaldehyde wall and triclosan core were printed to 100% cotton fabric with screen printing technique. Previous research showed excellent antibacterial activity (estimated for E. Coli and S. Aureus) of such fabric, so our aim in this research was to determine its resistance to the action of microorganisms present in the soil. The soil burial test was conducted. The breaking strength of the buried samples was measured and also the scanning electron microscope analysis was done. The results showed that none of the samples are resistant to decay. It is evident from SEM micrographs that on all of the buried samples greater morphological changes occur due to the functions of the soil microflora. It can be concluded that the samples printed with triclosan microcapsules are biodegradable which is environmentally preferable.

1. Introduction
In our previous research it was found out that pressure sensitive melamine-formaldehyde microcapsules (MCs) with antimicrobial agent triclosan (TCS) in the core, prepared by in situ polymerization method, can be successfully applied to cotton fabrics by screen printing [1]. Excellent antibacterial activity of the samples printed with the highest quantity of triclosan (100 g of MCs suspension per 1 kg of printing paste) microcapsules was estimated for E. coli and S. aureus, although the results were better for the latter. Even the washed samples showed good antimicrobial effect. Triclosan (2,4,4′-trichloro-2′-hydroxydiphenyl ether) is a member of the one of the major classes of antimicrobials for textiles – bisfenols [2, 3]. It exhibits a broad spectrum of antimicrobial activity (effective against many types of Gram-positive and Gram-negative bacteria), as well it has some antifungal and antiviral properties. The mechanism of triclosan blocks the active site of the enoyl-acyl carrier protein reductase enzyme (ENR), which is a significant enzyme in the fatty acid synthesis of bacteria. By blocking the active site, the inhibition of the enzyme and the prevention of the synthesis of fatty acids, which is necessary for building cell membrane and for reproducing, are achieved [4].

Our goal in this research was to determine the resistance of cotton fabric printed with triclosan microcapsules to the action of microorganisms present in the soil. The soil burial test was conducted. The breaking strength of the buried samples was measured and also the scanning electron microscope analysis was done.
2. Experimental part

2.1. Material used in the study
A microcapsule suspension with a pressure-sensitive melamine-formaldehyde wall and triclosan core (20% triclosan and 70% isopropyl myristate) prepared by in situ polymerization method was obtained from AERO, Chemical, Graphic and Paper Manufacturers d. d., Celje, Slovenia. A synthetic polyacrylate thickener (Tubivis DRL 300) and polyacrylate binder (Tubifast AS 30) were obtained from CHT (Germany). A pigment Bezaprint Gruen BT was obtained from Bezema (Switzerland) and was added to the printing paste only as an indicator of the print quality for the uniformity and wash fastness of prints to be observed.

2.2. Printing
The microcapsules were printed to 100% cotton fabric (Tekstina d. d. Slovenia) with screen printing technique using the printing paste with the concentration of 100 g microcapsules per 1000 g of the paste (4.8 g of triclosan per kg of fabric). For comparison also printing paste without microcapsules was used. Table 1 presents the recipe for printing pastes with and without microcapsules.

| Component      | Quantity (g/kg) |
|----------------|-----------------|
| Thickener      | 34              |
| Binder         | 150             |
| Microcapsules  | 100 0           |
| Pigment        | 2               |
| Distilled water| 714 814         |

Table 1. Printing paste recipe.

Printing was performed on the Mini MDF R-390 (Johannes Zimmer AG, Austria) laboratory magnetic printing machine. Samples were dried and cured in the Ernst Benz TKF 15-M500 drier. The printing conditions are presented in Table 2.

| Phase       | Conditions                         |
|-------------|------------------------------------|
| Printing    | Flat screen stencil: mesh 43 threads/cm  
|             | Printing speed: 80%                |
|             | Squeegee diameter: 8 mm            |
|             | Magnet pressure: level 5           |
|             | No. of passes: 2                   |

Table 2. Printing, drying and curing conditions for flat screen printing.

2.3. Analysis

2.3.1. Soil burial test. Determination of the resistance of fabrics printed with TCS MCs (100 g/kg) to the action of soil micro-flora was conducted using the soil burial test, according to the ISO 11721-1:2001 [5] and ISO 11721:2003 [6] standards. A container was filled with soil (pH 4.0-7.5 and 60% ± 5 of maximum moisture capacity). Cotton fabric samples (untreated – CO, printed without microcapsules – CO0 and printed with microcapsules – TCS100) were buried in the soil for a period of 5 days. After the defined incubation time, the samples were removed from the test soil, lightly rinsed with running tap water and immersed in 70% ethanol for 30 min before air-drying. Afterwards the
breakdown strength of the samples was measured using an Instron 5567 dynamometer in accordance with SIST ISO 5081:1996 [7]. Before the testing the samples were conditioned at temperature 20 ± 1 °C and relative humidity 65 ± 2%. The relative reduction in breaking strength, \( q_{\text{red}} \), of the buried cotton samples compared with the unburied samples was calculated from the average value of breaking strengths of 10 samples by using the following equation:

\[
q_{\text{red}}, t = \frac{F_t}{F_0}
\]

where \( q_{\text{red}} \) is the loss of breaking strength of buried cotton sample after the burying time \( t \), \( F_t \) is the breaking strength of buried cotton samples after the burying time and \( F_0 \) is the breaking strength of unburied cotton sample. If the reduction of breaking strength of buried printed sample is lower than 25% (\( q_{\text{red}} \) is higher than 0.75), it can be confirmed that the coating on the fibres is resistant to decay.

2.3.2. SEM observation. The morphology of the buried samples and the condition of the microcapsules was investigated using Scanning electron microscope Jeol JSM 6060. Before the observation the samples were coated with gold.

3. Results and discussion

3.1. Soil burial testing

The breaking strengths of the samples are shown in Table 3. None of the samples are resistant to decay, not even the sample containing TCS MCs. Indeed, the result is the opposite; the samples printed with (TCS100) and without MCs (CO0) had worse breaking strengths than the untreated buried sample (CO) because they both exceeded 80% reduction. The buried samples with TCS MCs had the worst breaking strength.

| Sample | \( F_t \) unburied sample [N] | \( F_t \) 5 days buried sample [N] | \( q_{\text{red}} \) buried sample [%] |
|--------|-----------------------------|---------------------------------|---------------------------------|
| CO     | 307.59                      | 69.14                           | 77.52                           |
| CO0    | 303.98                      | 40.96                           | 86.52                           |
| TCS100 | 307.61                      | 17.29                           | 94.38                           |

3.2. SEM micrographs

Figure 1 shows that on all of the buried samples both untreated and printed, greater morphological changes occur due to the functions of the soil microflora. On the fibres of the untreated sample surface cracks and holes formed after 5 days buried (Figure 1a). After five days buried, the samples without printed MCs was different, because it is obviously infected with fungi (Figure 1b). Weak mycelium growth is observed in the micrograph along with aggregates of spores and hyphae. The fibres do not seem to be damaged. Intensive growth of mycelium was present on the buried sample printed with TCS MCs (Figure 1c). The fibres and MCs beneath were not damaged. Some of the MCs remained undamaged and round in shape after the burial, which indicates that triclosan was still safely encapsulated and was not released to the surface.
Figure 1. SEM micrographs of unburied and 5 days buried cotton fabric a) untreated; b) printed without MCs; c) printed with TCS MCs (100 g/kg).

4. Conclusion
The results show that none of the samples are resistant to decay, because the reduction of their breaking strength after the burial is high. It is evident from SEM micrographs that on all of the buried samples greater morphological changes occur due to the functions of the soil microflora. It can be concluded that the samples printed with triclosan microcapsules are biodegradable which is environmentally preferable.

References
[1] Ocepek B, Boh B, Šumiga B and Forte Tavčer P 2012 Color. Technol. 128 95
[2] Bhargava H N and Leonard P A 1996 Am. J. Infect. Control 24 209
[3] Orhan M and Gunesoglu C 2007 Indian J. Fibre Text. Res. 32 114
[4] Glaser A 2004 Pestic. You 24 12
[5] ISO 11721-1:2001 Textiles - Determination of resistance of cellulose-containing textiles to micro-organisms - Soil burial test - Part 1: Assessment of rot-retardant finishing
[6] ISO 11721:2003 Textiles - Determination of the resistance of cellulose-containing textiles to micro-organisms - Soil burial test - Part 2: Identification of long-term resistance of a rot retardant finish
[7] Textiles - Woven fabrics - Determination of breaking strength and elongation (Strip method)