Preparation of Triclosan microcapsules and printing on cotton textiles

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INTRODUCTION

Microencapsulation applications in textile industries are diverse. Typical inventions include microencapsulated dyes, pigments, fire retardants, antistatic agents, enzymes, softeners, fragrances, thermochromic and photochromic agents, antimicrobial agents, insect repellents, cosmetic and medical components, and microencapsulated phase change materials for dynamic accumulation and release of heat.

This contribution describes (1) microencapsulation of Triclosan (Figure 1), a commercial antimicrobial agent, by the *in situ* polymerization method in O/W emulsion , (2) preparation of formulations and printing on woven cotton textiles by a flat screen printing technique, and (3) some preliminary results of testing, including thermal stability and release of Triclosan, and resistance of microcapsules on the textiles to washing.

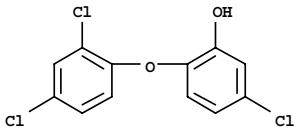

Figure1: Structure of Triclosan

Triclosan is a wide spectrum antimicrobial agent, effective against many common bacterial species. Its antimicrobial activity derives from the inhibition of bacterial growth by blocking lipid biosynthesis (Levy et. al., 1999).
Commercial applications are numerous, Triclosan is used as a bacteriostat and preservative for cosmetic and detergent preparations, as well as an antiseptic and disinfectant in medications (HSDB, 2009).

MATERIAL AND METHODS

Preparation of microcapsules by *in situ* polymerization

Triclosan (Sigma-Aldrich) was dissolved in isopropylmyristate and was used as a core material in *in situ* polymerization microencapsulation. Melamine-formaldehyde resinous precondensate (Melamin, Slovenia) was used as a water-soluble prepolymer for microcapsule wall formation. Styrene-maleic acid anhydride copolymer (Hercules) was used as a modifying agent/emulsifier for *in situ* polymerization process.

Laboratory scale microencapsulation was performed in a 1L stainless steel reactor, diameter 90 mm, equipped with exchangeable dissolver stirrers with impeller speed of 1200-6000 rpm, and a cooling/heating system. The microencapsulation process, based on Knez (1995) and Kukovic & Knez (1997) was used, consisting of the following steps: (1) preparation of an aqueous solution of a modifying agent and its partial neutralisation, (2) emulsification of a core material to produce an

O/W emulsion, (3) addition of an amino-aldehyde prepolymer for wall formation; (4) induction of polycondensation reaction by raising the temperature to 70 - 80^{o}C; (5) microcapsule wall formation by the polycondesation process at an elevated temperature for about 1 hour; (6) termination of polymerization by raising pH to 7.0 and cooling the system to room temperature; (7) removal of residual formaldehyde by ammonia as a scavenger.

Printing on Cotton Textiles

White woven 100% cotton fabric (Tekstilna, Slovenia) was used as a carrier for printing. Triclosan microcapsules were formulated into printing pastes with the following additives: synthetic polyacrylate thickener Tubivis DRL 300 (CHT, Germany), polyacrylate binder Tubifast AS 30 (CHT, Germany), crosslinking agent Tubifix ML 55 (CHT, Germany), and distilled water.
Patterns of a square shape were printed on the woven cotton fabric by the flat screen printing technique. Printing was performed on the magnetic printing machine Mini MDF R 390 (J. Zimmer, Austria) by the use of a flat screen and rod magnetic squeegee. After printing, the samples were air dried and thermally fixed at 150 °C for 5 minutes. Samples of printed cotton fabrics were washed at 40 °C according to the ISO 105-C01:1989 (E) standard, and air dried.

Analyses and tests

Microcapsule diameter and size distribution were measured by Alkatel Cilas Laser Granulometer 715. Olympus microscope BX60 with a Sony CEN50 camera was used for the characterization of visual appearance, individual microcapsule size and morphological characteristics of microcapsules. Scanning electron microscopy was performed by JEOL JSM-6060LV microscope, at accelerating voltage 10 kV, with sample coating C + Au/Pd.
Thermal stability and permeability of microcapsules was measured gravimetrically as a mass loss at an elevated temperature T=135°C after 30, 60, 120 and 180 minutes of heating.

RESULTS AND DISCUSSION

Characteristics of Triclosan microcapsules, produced by *in situ* polymerization

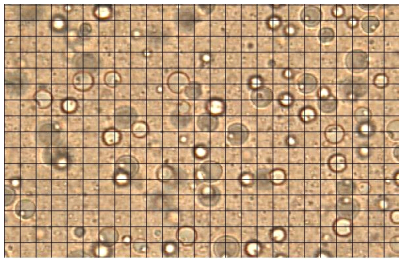

Figure 2: Triclosan microcapsules (1000X; net size 5 µm x 5 µm)

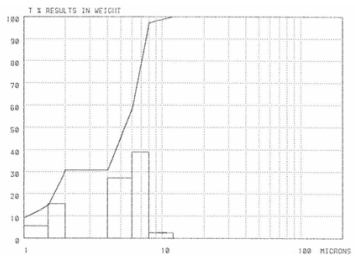

Figure 3: Triclosan microcapsules size distribution

The main properties of aminoaldehyde polymer wall microcapsules with Triclosan as a core material are described in Table 1. Microcapsule size distribution (Figure 3) is narrow, with average

diameters 4 - 8 µm. A fraction of small particles (1 - 2 µm) is probably a residue of redunadant wall material from the microencapsulation process.

Table 1: Characteristics of Triclosan microcapsules, prepared by *in situ* polymerization

Parameter	Value
Form and appearance	Aqueous suspension, white
Average microcapsule size	5.4 µm
Suspension viscosity	330 mPas
Solid content	31 %
pH value	8,0
Free formaldehyde	< 0,5 %
Thermal stability/permeability	Less than 2% leakage after 1h at 135°C

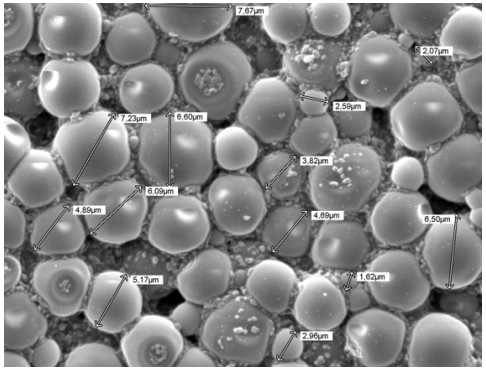

Figure 4: SEM of Triclosan microcapsules with size measurements (7000 x)

Printing Triclosan microcapsule formulations on a woven cotton textile

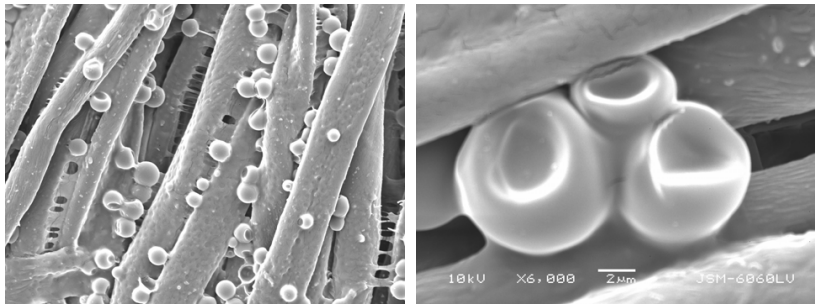

Figure 5: SEM of Triclosan microcapsules on a woven cotton textile before washing (left 950 x, right 6000 x)

The formulation and procedure for printing antimicrobial Triclosan microcapsules on cotton textiles was appropriate and successful, as observed from the SEM analysis (Figure 5). Microcapsules adhered evenly to the cotton fibres. The standardised test of textile washing at 40°C in a washing machine confirmed that microcapsules were washing resistant and remained on the fabric practicaly undamaged (Figure 6).

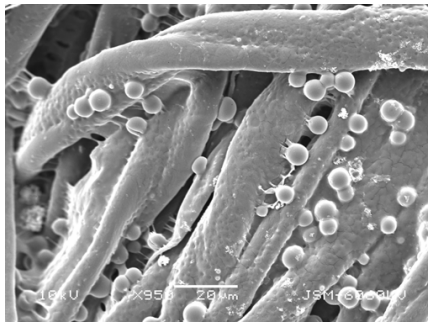

Figure 6: SEM of Triclosan microcapsules on a woven cotton textile after standardised washing at 40°C

CONCLUSIONS

The antimicrobial agent Triclosan was microencapsulated by *in situ* polymerization of aminoaldehyde resins. Microcapsules were formulated into a printing paste and applied to cotton textiles by the flat screen printing technique. The results of preliminary testing confirmed that microcapsules with the antimicrobial agent can be applied and adhered onto a woven cotton fabric, and that they remain undamaged on cotton fabrics during the standardized washing in a washing machine.

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