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ORAL SESSION 5. INNOVATION IN MATERIALS FOR ENCAPSULATION

Chairperson : Christine Wandrey, Bruno Sarmento, Corinne Hoesli

AROMA ENCAPSULATION FOR ANTIBACTERIAL AND ECO-FRIENDLY TEXTILE FINISHING

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INTRODUCTION & OBJECTIVES

Encapsulation imparts new properties and added value to conventional fabrics (Nelson, 2002). Most of the commercially available microcapsules for textile applications are made of melamine-formaldehyde, urea-formaldehyde or phenol-formaldehyde resins, which have significant negative health and environmental effects. Recently, there has been a growing interest in the replacement of these resins with safe and environmentally benign materials.

The process of fixing the microcapsules onto textile substrates is critical in ensuring their durability and effectiveness. The commonly known industrial methods used for this involve the use of two main groups of binders; polymeric resins, and polyfunctional crosslinking agents. Polymeric resins are reported to partially inhibit the release of fragrance from the microcapsules. The chemical cross-linkers are subdivided into formaldehyde based, e.g., formaldehyde and glutaraldehyde, and non-formaldehyde based, such as polycarboxylic acids.

This work aimed at conferring fragrant and antibacterial properties to cotton fabrics employing new methodologies utilizing non-toxic and environmentally friendly materials.

MATERIALS & METHODS

Microcapsules were prepared by the complex coacervation method (Butstraen, 2014) but with some modifications. D-Limonene was used as the core material. Size distribution and mean particle size were determined by laser diffraction. Encapsulation efficiencies were measured by GC-FID. Fixation of the microcapsules onto fabrics was accomplished by using citric acid as a non-toxic cross-linker (See Figure 1). The fixation of the microcapsules onto fabrics was examined using FTIR-ATR. The percent of bacterial inhibition of the impregnated cotton fabrics and of the control fabric samples was assessed by the Standard Test Method under Dynamic Contact Conditions (ASTM Standard E 2149-01); with a modification that involved the renewal of the bacterial inoculum after each washing cycle.

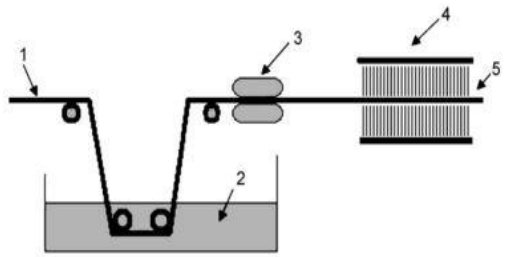

Figure 1: Fixation steps of the microcapsules onto cotton fabrics. Scheme adapted from (Rodrigues, 2009).

1.Untreated fabric 2. Impregnation bath 50 ºC (contains microcapsules, citric acid and catalyst) 3. Foulard (0.1 MPa) 4. Thermofixation (drying at 90ºC and curing at 150ºC) 5. Treated fabric

RESULTS & DISCUSSION

The produced microcapsules showed 94% encapsulation efficiency and a mean diameter of 39 µm. The particle size distribution is shown in Figure 2.

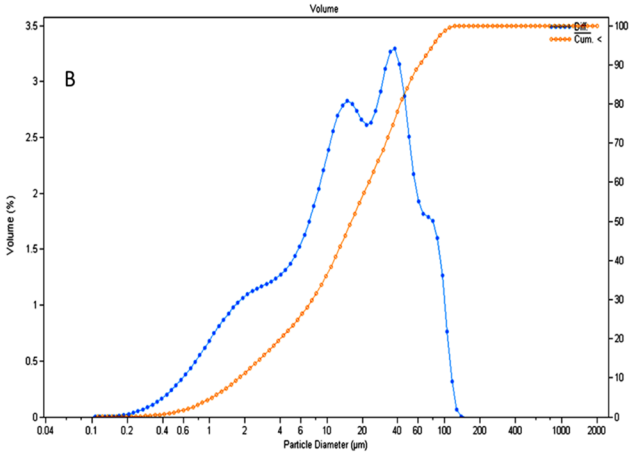

Figure 2: The particle size distribution in volume of the produced limonene microcapsules.

SEM was used to examine the cotton fabrics impregnated with microcapsules (Figure 3). Impregnated fabrics were also examined by SEM after being washed with 2% commercial soap followed by 0.1N acetic acid and deionized water to investigate the effect of washing on the adhesion of the microcapsules to the fabrics. The process was repeated three

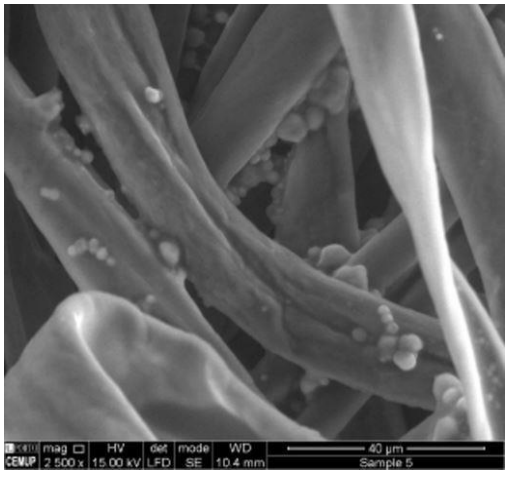

Figure 3: SEM image of cotton fabric impregnated with limonene microcapsules.

times. SEM images showed the microcapsules being still attached to the fabric (Figure 4).

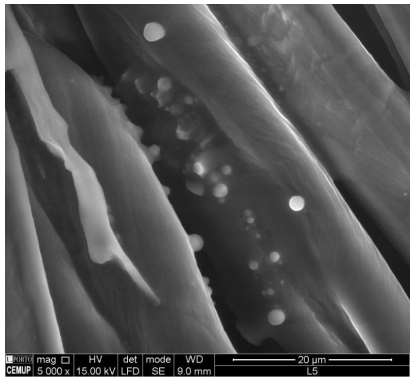

Figure 4: SEM image of impregnated cotton fabric after washing for three cycles.

The peak at 2855 cm^{-1} in the FTIR spectrum of the microcapsules (Figure 5) indicates the successful complex coacervation between chitosan and gum Arabic, as reported in the literature (Butstraen, 2014). The spectrum of cotton fabric impregnated with limonene microcapsules did not show the presence of the sharp peaks at 1742 cm^{-1} and 1693 cm^{-1} characteristic of citric acid, which is compatible with its effective bonding with the –OH groups of the cotton cellulose. The spectrum of the grafted cotton fabric also revealed the appearance of a new peak corresponding to the C=O ester stretching at 1729 cm^{-1}, which was absent in the control cotton fabric sample confirming the covalent attachment between the polymeric shell of the microcapsules with cotton cellulose via citric acid reaction.

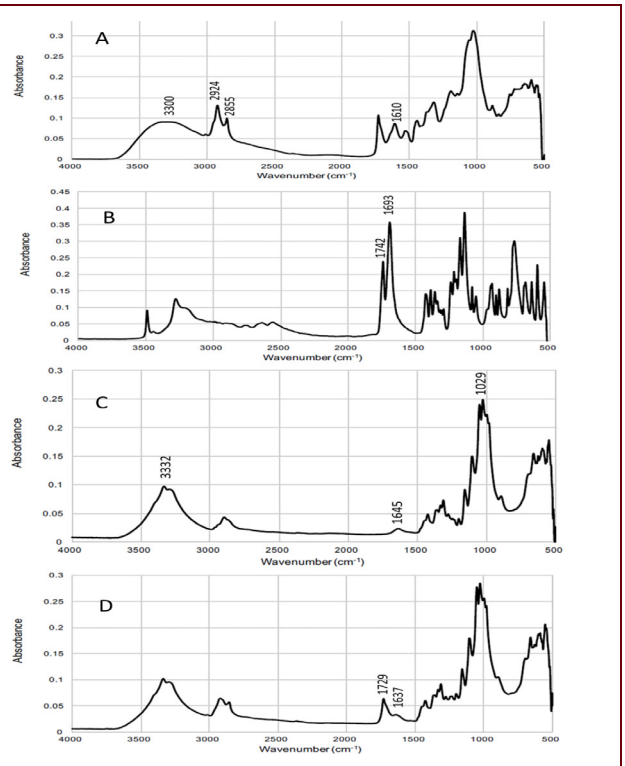

Figure 5: FTIR spectra of A) microcapsules B) citric acid C) control fabric D) treated fabric

Bacterial inhibition tests were conducted against *E. coli* and were calculated according to the following formula:

$$Reduction\ (\%) = \frac{(A-B)}{A} \times 100$$

where B is the CFU/ml for the treated fabric sample after the specified contact time and A is the CFU/ml for the inoculum before the addition of the treated fabric. The results of the assays are shown in Figure 6. The impregnated fabric showed 95.9% bacterial reduction after 15 minutes of dynamic contact. Every 15 minutes the fabric sample was washed with sterilized water and placed in contact with a new bacterial inoculum in order to take samples for colony counting. The percent bacterial reduction decreased with time, it was maintained throughout the 8 renewal cycles with values higher than 25% reduction.

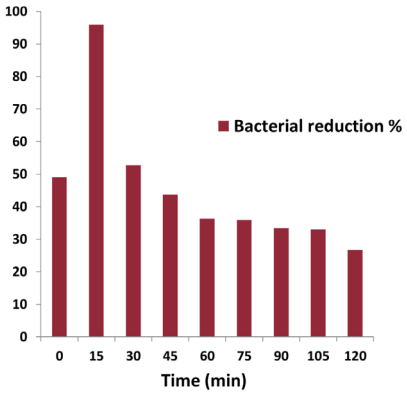

Figure 6: Results of the bacterial reduction % of cotton fabric impregnated with limonene microcapsules.

CONCLUSIONS & PERSPECTIVES

Imparting a durable antimicrobial finish to cotton fabrics by using microcapsules was successfully achieved using green and non-toxic materials. Future work will focus on maintaining the aroma durability of the treated fabrics according to end-use application (e.g., washing and abrasion test cycles).

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