

## Incorporation of the sunscreen agent, octyl methoxycinnamate in a cellulosic fabric grafted with $\beta$ -cyclodextrin

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### Abstract

The aim of the study was to investigate the incorporation of the sunscreen agent, octyl methoxycinnamate into cyclodextrin cavities covalently bound to cloth fibres. Tencel, a cellulosic fabric, was grafted with  $\beta$ -cyclodextrin molecules through reaction with monochlorotriazinyl- $\beta$ -cyclodextrin ( $\beta$ -CDMCT). The finished and untreated textiles were soaked in water-methanol mixtures containing 2% (v/v) of sunscreen agent and subsequently subjected to several washing cycles. The unmodified and modified fabrics were characterized by UV spectrophotometry and thermogravimetric analysis. The level of octyl methoxycinnamate entrapped in the Tencel tissue was determined by high-performance liquid chromatography and was found to be much higher (0.0203%, w/w) for the textile functionalised with  $\beta$ -CDMCT compared to the unmodified fabric (0.0025%, w/w). In addition, spectrophotometric assessment of UV transmission through the fabric samples using the Transpore<sup>TM</sup> test showed that the *in vitro* sun protection factor of the textile support was markedly enhanced (3.2-fold increase) by impregnation with octyl methoxycinnamate of the  $\beta$ -CDMCT grafted textile. Hence, even after repeated washings, the  $\beta$ -CD finished fabric exhibits higher sunscreen agent retention and photoprotective properties than the unmodified textile material.

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### 1. Introduction

The harmful effects of the solar UV radiation (290–400 nm) on human skin (i.e., erythema, cutaneous photoageing, immune suppression and various forms of skin cancers) have been the object of several studies that led to improved approaches in photoprotection (National Institute of Health, 1989; Gasparro et al., 1998; Green et al., 1999). The strategies advocated by health care authorities to prevent the sunlight-induced damage include reduced sun exposure, topical application of suncreening preparations and the use of proper clothing (National Institute of Health, 1989; Gasparro et al., 1998; Diffey, 2001; Gambichler et al., 2001; Edlich et al., 2004).

To enhance the sun protection factor of textiles, clothes can be coated with sunscreen agents. This operation has been performed

by adding the UV filters directly to the rinsing liquid during the laundry cycles (Edlich et al., 2004). A different approach is described in the present study for the incorporation of sunscreens into fabrics. This innovative procedure is based on cotton tissues grafted with  $\beta$ -cyclodextrin derivatives.

Cyclodextrins are toroidal-shaped cyclic oligosaccharides with a hydrophilic outer surface and an internal hydrophobic hollow interior. Cyclodextrins can entrap a vast number of lipophilic compounds into their hydrophobic cavity, depending on their size and molecular structure. For this reason cyclodextrins behave as hosts and the hydrophobic species are the guests. The driving force for such inclusion process is the enthalpic contribution that arises from non-covalent hydrophobic interactions (Loftsson and Brewster, 1996; Rajewski and Stella, 1996). This complexation phenomenon can modify some physico-chemical and chemical properties of the guest, for example enhancing its stability to oxidant agents and light and increasing its apparent aqueous solubility (Loftsson and Brewster, 1996; Rajewski and Stella, 1996; Uekama et al., 1998).

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The remarkable ability of cyclodextrins to include hydrophobic compounds has been exploited in several fields, spanning from pharmaceuticals to cosmetics, from food manufacturing to chromatography and textile finishing (Loftsson and Masson, 2001; Del Valle, 2004; Szente and Szejtli, 2004; Wang and Chen, 2005). The incorporation of cyclodextrins onto fabrics can be carried out by impregnation or spraying of the tissue with a cyclodextrin solution, or through covalent binding (grafting) of the cyclodextrins to the cloth surface (Lo Nostro et al., 2002; Martel et al., 2002). The latter approach has the advantage that it lasts longer than the simple surface adsorption. Grafted specimens retain their complexing properties also after handling and repeated washing cycles (Lo Nostro et al., 2002; Martel et al., 2002).

This study describes the chemical grafting of monochlorotriazinyl- $\beta$ -cyclodextrin ( $\beta$ -CDMCT) onto Tencel, a cellulosic fabric obtained from wood pulp.  $\beta$ -CDMCT was selected for this investigation since it is commercially available, has no irritating or sensitizing effects (Reuscher and Hinsenkorn, 1996) and represents an efficient tool for surface modification of textiles (Lo Nostro et al., 2003). In addition, the  $\beta$ -cyclodextrin macrocycle has been shown to be the most suitable host for the inclusion of sunscreen agents (Scalia et al., 2002). Tencel was chosen as the clothing material since, while retaining some structural properties that are typical of natural cotton fibres, it exhibits reduced variability in texture, specific weight and specific area (Lo Nostro et al., 2001, 2002, 2003). The present work also reports on the inclusion of octyl methoxycinnamate (OMC), the most widely used sunscreen agent (Scalia et al., 2002), in the  $\beta$ -CD cavities grafted on the textile surface. The influence of the complexed UV filter present on the fabric surface on the sun-protective capacity of the finished textile was then evaluated.

## 2. Materials and methods

### 2.1. Materials

Monochlorotriazinyl- $\beta$ -cyclodextrin ( $\beta$ -CDMCT) was obtained from Wacker-Chemie Italia (Milan, Italy) and used as received. Tencel was kindly provided by Tecnotessile Srl (Prato, Italy). The fabric was carefully rinsed in boiling aqueous  $\text{Na}_2\text{CO}_3$  for 3 h, and then dried at room temperature before any chemical treatment or test. Octyl methoxycinnamate (OMC; Fig. 1) was supplied by Roche Ltd. (Geneva, Switzerland). Methanol and acetonitrile were high-performance liquid chromatography (HPLC)-grade from Sigma–Aldrich (Steinheim,

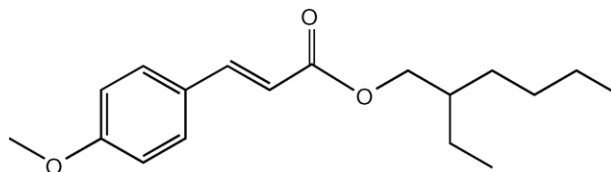


Fig. 1. Chemical structure of octyl methoxycinnamate.

Germany). All other chemicals were of analytical-reagent grade (Fluka, Milan, Italy).

### 2.2. Grafting of Tencel with $\beta$ -CDMCT

Permanent grafting of  $\beta$ -CDMCT was carried out according to a previously reported method (Lo Nostro et al., 2003), with minor modifications. The procedure (see Fig. 2) consists in soaking for 5 min the fabric samples (typically 4 cm  $\times$  4 cm) at room temperature in an aqueous solution of  $\beta$ -CDMCT (15%, w/v) and  $\text{Na}_2\text{CO}_3$  (15%, w/v), under magnetic stirring. The samples were then squeezed to remove the excess solution. To minimize the reaction of  $\beta$ -CDMCT with air moisture, the impregnated samples were cured in an oven at 130 °C for 15 min at atmospheric pressure (dry heat), and then carefully rinsed with demineralized water to remove any unreacted  $\beta$ -CDMCT. The tissue was then conditioned in a dry box at constant relative humidity (56%) and room temperature.  $\beta$ -CDMCT was detected on the treated textile surface through UV spectrophotometry. The grafting yield was evaluated by weighing the sample before and after the treatment, with a weight increment of about 5%.

### 2.3. Impregnation of grafted Tencel with OMC

Modified and unmodified fabric samples were treated with OMC by soaking the textile material for 2 h under stirring in a water-methanol mixture (30:70, v/v) containing 2% (v/v) of the UV filter. The samples were then roll-squeezed, washed several times at room temperature with running tub water, deionized water and 30% (v/v) methanol in water. The latter mixture was found to be more efficient than soapy water for the removal of adsorbed material from the fabric surface.

The OMC uptake of the untreated and grafted Tencel fabrics was evaluated by HPLC, UV and thermal analyses, as described below.

### 2.4. High-performance liquid chromatography

The HPLC apparatus consisted in a Model LabFlow 3000 pump (LabService Analytica, Bologna, Italy), a Model 7125

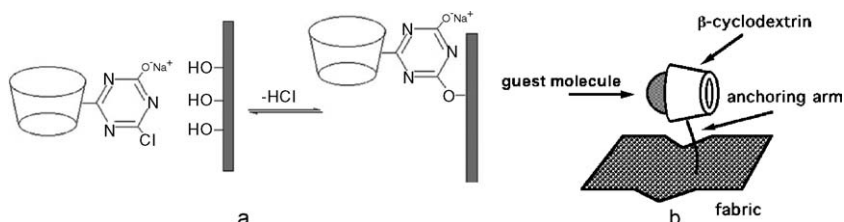


Fig. 2. Chemical grafting of monochlorotriazinyl- $\beta$ -cyclodextrin onto a cellulosic fibre (a). Scheme of a host-guest inclusion complex grafted on the textile surface (b).

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