Preparation of β-Cyclodextrin/Latex Dispersion for Coating of Cellulose Fabric

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ABSTRACT: In recent years there has been an increasing demand for the application of natural products to address problems in the environment. The use of products derived from renewable resources can provide a sustainable strategy to replace totally or partially synthetic products. *Cymbopogon*, commonly known as lemongrass, is a tropical grass cultivated mostly for its essential oil, which has demonstrated several bioactivities, including antibacterial and antifungal properties. In the present work the formation of inclusion complex between β-cyclodextrin (β-CD) and lemongrass essential oil (LGEO) as well as its fixation onto cotton fabric were investigated. Inclusion complex was prepared through solution method followed by freeze-drying. The starting materials and inclusion complex were characterized by FTIR, TG/DTG and SEM. The cotton fabric incorporated with inclusion complex was investigated by FTIR and XRD. The results suggested the success of complexation in the molar ratio of 0.015:0.018 mol/L (LGEO:β-CD) and the viability of fixing the inclusion complex leading to a potential functional textile.

KEYWORDS: β-cyclodextrin, lemongrass, inclusion complex, functional textile

1 INTRODUCTION

Throughout the history of humanity textile materials have always played important roles. Firstly people used them as protection from cold and rain but afterwards the aesthetic aspect became a social symbol. Nowadays a new generation of textile materials emerges with improved functionalities [1]. Researchers from around the world have investigated textiles with novel functions such as anti-wrinkling properties [2], aroma sustained release [3], smart functionalities delivered to textiles by integrating smart stimuli-responsive polymers [4], antibacterial activity acquired with inorganic nanostructured materials [5], protective functions based on membrane science [6] and durable insect repellency [7], among others.

The new functionalities can be imparted into textiles by surface modification of fabric or fiber/yarn via chemical and/or physical processes. The insecticides cypermethrin and prallethrin were fixed permanently onto the surface of cotton fabrics producing an effective personal protection against mosquitoes [8].

Karthik, Rathinamoorthy & Murugan [9] pointed out that textile finishing based on natural materials, which are safe and biodegradable, is an environmentally friendly approach to manufacture high performance textiles. A complete review made by Shahid-ul-Islam and colleagues highlights the most important textile applications of environmental friendly plant-based products; especially plant derived bioactive agents with antimicrobial properties [10].

Essential oils (OEs), also called volatile oils, are aromatic oily liquids obtained from plant material, most commonly by the method of steam distillation. They consist of a complex mixture of organic, volatile and lipophilic compounds, which generally impart odor to plants [11].

Cymbopogon, commonly known as lemongrass, is a genus of an herbaceous plant from *Pocaceae* family

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and is widely used to obtain lemongrass essential oil (LGEO) [12].

The major phytoconstituent of lemongrass oil is citral, a mixture of two stereoisomeric aldehydes: the *trans* isomer geranial (40–62%) dominates over the *cis* isomer neral (25–38%). Other constituents are limonene, linalool, geranyl acetate and myrcene [13].

Biological research has shown that various biological activities are associated with LGEO [12]. The major activities associated with LGEO are insect repellency [14], fungicide and bactericide properties [12], mainly because the presence of citral.

Although LGEO, as well as other EOs, has significant potential applications, the use is limited due to its low solubility in water and poor stability in presence of light, heat and oxygen [15]. To overcome these drawbacks, microparticles containing LGOE have been prepared, for example, by complexation of LGEO or its isolated compounds with cyclodextrins [16–19].

Cyclodextrins (CDs) are cyclic oligosaccharides that present peculiar chemical conformation. It is a three dimensional structure which resembles to a truncated cone constituted by D-glucopyranose units linked by α-1,4 glycosidic bonds. Each glucopyranose unit has three free hydroxyl groups located in the upper and lower side of the exterior of the cavity. Inside the cavity has oxygen atoms of glycosidic bonds and this specific distribution of groups makes the exterior of the cavity hydrophilic in contrast to the hydrophobic interior of the cavity [20].

Typical native CDs are denoted α, β and γ-cyclodextrins and the most characteristic feature for all of them is their ability to form inclusion complexes with various molecules, ions and polymers, through host-guest interactions [20]. β-CD is the most frequently used owing to its greater complexation ability associated with a lower cost [20].

Thus, in an attempt to protect the LGEO from thermo chemical degradation and control its release, it is also an objective of this research to prepare and characterize inclusion complex between β-CD and LGEO (β-CD:LGEO).

According to Simonsic & Tomsic [21], in view of the need for ecologically friendly antimicrobial finishing of textiles, much research has focused on the synthesis of antimicrobial agents where the leaching antimicrobials have been replaced with the bound antimicrobials. The latter can be prepared using complexes with CDs, and encapsulated nanoparticle agents embedded into polymer matrices.

In this context, the present work aimed to use β-CD:LGEO complex as bound antimicrobial textile agent by fixing it onto cotton fabric with the aid of an acrylic resin (latex).

2 EXPERIMENTAL

2.1 Materials

Lemongrass essential oil (LGEO) (*Cymbopogon flexuosus*) was purchased from Ferquima (Brazil). According to the manufacturer, it was obtained by distillation from the leaves and the main component citral (40% geranial and 30% neral). β-cyclodextrin (β-CD) (assay ≥97%) was purchased from Sigma Aldrich (USA). Both were used as received without further purification.

2.2 Methods

2.2.1 Gas Chromatography of LGEO

The chromatographic experiments were performed according to the procedure described by Aragão and collaborators [22]. The identification of volatile components in lemongrass oil was performed by gas chromatography-mass spectrometry (GC-MS) using a mass-selective detector (Shimadzu QP-2010 Plus). The used chromatographic column was a fused silica capillary column with stationary phase Rtx-5MS, with 30-m length and 0.25-mm internal diameter, using helium as carrier gas. The temperature of the injector and detector was 240 °C. The initial column temperature was 60 °C, programmed for an increase of 3 °C per min until reaching a maximum temperature of 240 °C. The identification of components was done by comparing the Kovats index for each peak obtained by injection of sample and of a series of n-alkanes standards (C9-C27). Quantification of chemical constituents in the essential oils was performed using a gas chromatograph (Shimadzu GC-2010 Plus) equipped with a flame ionization detector (GC-FID). Nitrogen was used as carrier gas in the Rtx-5MS capillary column with 30 m length and 0.25 mm internal diameter. Temperature of the injector and detector was fixed at 240 °C. The temperature program of the furnace was the same as that used in the GC-MS analyses. A 10-mg sample was diluted in dichloromethane (1 mL), followed by injection of a further 1 μL.

2.2.2 Preparation of β-cyclodextrin Inclusion Complex

β-CD:LGEO complex was prepared by complexation in solution and freeze-dried [23]. LGEO was dissolved in ethanol (concentration of 0.015 mol/L) and β-CD was dissolved in a mixture of water/ethanol (1:2) heated at 55 °C (concentration of 0.018 mol/L). LGEO solution was added slowly to the β-CD solution and the mixture was kept under constant stirring at 150 rpm

for 4 h, at room temperature, using a metabolic water bath incubator shaker (Marconi MA 093). Afterwards, the solution was freeze-dried (Liotop L101).

2.2.3 *Fourier Transform Infrared Spectroscopy*

Fourier transform infrared spectroscopy (FTIR) spectra of LGEO, β-CD, and β-CD:LGEO complex were recorded on a Nicolet spectrophotometer, model 6700 with an attenuated total reflection (ATR) device, over a range of 4000–650 cm^{-1} by averaging 32 scans at a resolution of 4 cm^{-1}.

2.2.4 *Thermogravimetric Analysis*

Thermogravimetric analysis (TGA) of β-CD:LGEO was performed using a Seiko-SII Nanotechnology Inc. model Exstar 7200 equipment. Samples weighting about 10 mg were heated from 25 to 600 °C at a heating rate of 10 °C min^{-1}, under nitrogen flow (flow rate of 20 $cm^3\,min^{-1}$).

2.2.5 *Scanning Electron Microscopy*

Morphology of complex particles were investigated by scanning electron microscopy (SEM) (Vega 3LM, Shimadzu), at accelerating voltage of 10 kV. Samples were previously vacuum-coated with gold to avoid electrostatic charging during examination.

2.3 Dispersion of β-CD:LGEO Complex in the Latex and Coating Application

The acrylic resin (latex) used to fix β-CD:LGEO complex onto cotton fabric was synthesized, via emulsion polymerization, in our laboratory according to the procedure previously described [24]. β-CD:LGEO aqueous dispersion (1% w/V) was mixed with the latex (1:1 V/V) forming β-CD:LGEO/latex dispersion. Pre-washed samples of cotton fabric (0.5 g) have been exhausted into the dispersion and dried for 24 hours at room temperature (25 °C). The modified textile was then characterized to evaluate the success of coating fixation.

2.3.1 *Evaluation of the Coating Fixation*

FTIR and X-ray diffraction (XRD) were carried out with the purpose of indirectly verify the success of the coating fixation onto cotton fabric. FTIR was carried out in the same conditions previously described in subsection 2.2.3. The diffractograms were obtained with the aid of an X-ray diffractometer (Shimadzu model 7000) equipped with a copper target (CuK, = 1.54 λ) operating at 40 kV and 30 mA. The samples were scanned at a rate of 2° min^{-1} between 2θ = 5 and 80°.

3 RESULTS AND DISCUSSION

3.1 Chemical Characterization of LGEO

According to chromatographic analysis, the major LGEO components were neral (34.04%), geraniol (8.81%) and geranial (37.45%). Other 12 compounds were also identified (Figure 1). The analysis confirmed the presence of citral, responsible for antimicrobial activity, and is in agreement with other works which reported citral as the major phytoconstituent of lemongrass essential oil [13, 25, 26].

The absorptions in the FTIR spectrum of *Cymbopogon citratus* were reported by Lee *et al*. [27] as follows: 3403.65 cm^{-1} was assigned to the stretching of hydroxyl (–OH) group, 2919.03 cm^{-1} was related to alkane (C–H) group stretching, 2371.88, 2345.87 and 2138.23 cm^{-1}, were attributed to the stretching of alkyne (C≡C), –OH of carboxylate (– COO–) and nitrile (C≡N) functional groups, respectively. Other peaks reported by such authors were: at 1648.61 cm^{-1}, associated with carbonyl (C=O) stretching of amide groups, at 1403.63 cm^{-1}, due to the stretching of –OH bond and the peak at 1253.54 cm^{-1} caused by the deformation of C=O of carboxylic acids. Lastly the peak at 1053.59 cm^{-1} was assigned to C–OH stretching of alcoholic group, and

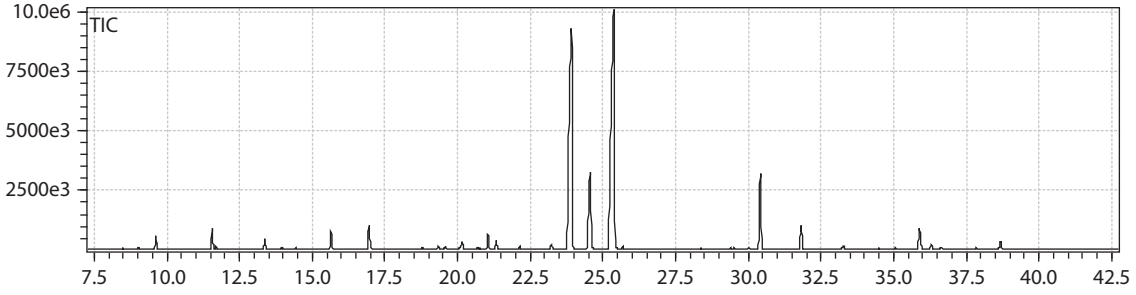

Figure 1 Gas chromatogram of lemongrass essential oil (LGEO).

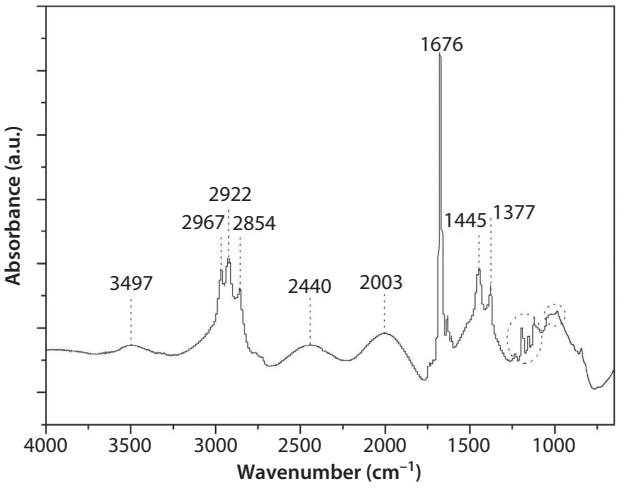

Figure 2 FTIR spectrum of lemongrass (*Cymbopogon flexuosus*).

the peak at 594.13 cm^{-1} was related to C–Cl stretching of alkyl halide group.

The FTIR spectrum of LGEO is shown in Figure 2. Although LGEO used in the present work is derived from the species *flexuosus* instead of *citratus*, both have almost identical chemical composition [28].

As can be observed in Figure 2 the spectrum shows absorption at 3497 cm^{-1} corresponding to –OH groups is in agreement with the results of GC-MS, where the presence of neral, geranial and other alcohols in small proportions were detected.

The intense band observed at 1676 cm^{-1} is due to vibrations of C=C (cis and trans), confirming the presence of conjugated double bonds (C=C-CHO) in citral which are common in acyclic monoterpenes [29]. The peak at 1632 cm^{-1}, reported as stretching of C=O of the aldehyde group [27, 29], can be considered as the small shoulder to the right of the main peak in the carbonyl region.

The peaks at 1445 cm^{-1} and 1377 cm^{-1} can be assigned to the bending of -CH_2 and -CH_3 groups respectively [29]. At lower wavenumbers appeared several peaks in accordance with those reported to LGEO [27, 29], although in this spectrum they are not perfectly distinguishable. Also the two large absorptions located at 2440 and 2003 cm^{-1} were not found in the literature as characteristic of citral. The overall FTIR analysis showed the key bands reported in the literature for LGEO [27].

3.2 Preparation and Evaluation of β-CD: LGEO complex

The presence of a hydrophobic cavity in CDs enables them encapsulate a variety of hydrophobic molecules

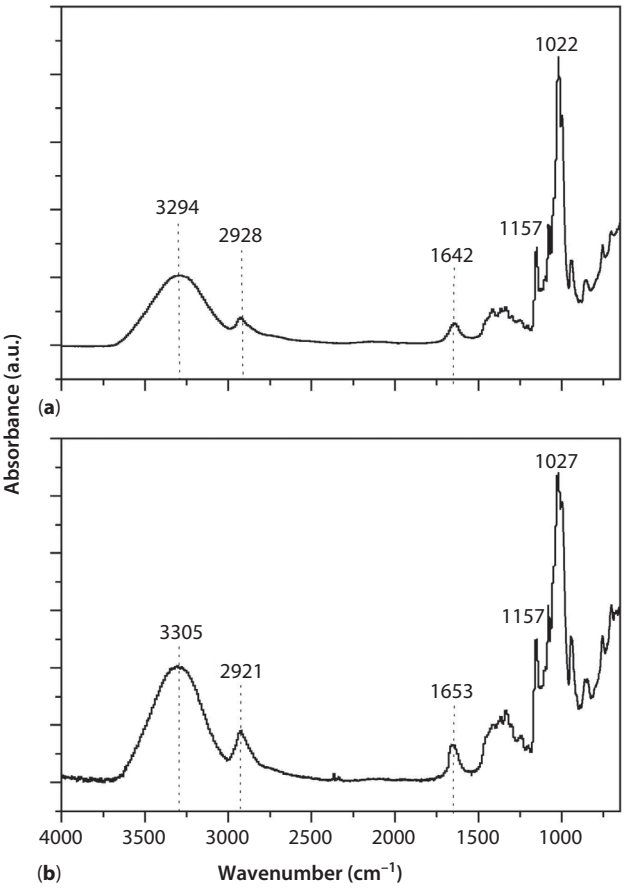

Figure 3 FTIR spectra of: (**a**) pure β-cyclodextrin (β-CD) and (**b**) complex obtained after freeze-drying.

or parts of molecules inside their cavity through non-covalent interactions to form inclusion complexes of the host-guest type [20]. β-CD is the most used owing to its cavity with an internal diameter of 6.5 Å and a depth of 8 Å [20].

Veiga, Pecorelli & Ribeiro [30] pointed out that usual stoichiometry ratios for complexes are 1:1 and 1:2. The majority of β-CD guest molecules should possess molecular weight between 100 and 400 daltons. Once citral (major component of LGEO) has molar mass of 152.24 g/mol and the complex was prepared with LGEO (not with isolated citral), the amounts used for complexing viz. 0.015 mol/L of LGEO and 0.018mol/L of β-CD seemed appropriate. Rungsardthong and his team [31] have prepared complexes of citral (isolated from lemongrass) with of β-CD. After complexation kinetic studies they concluded that the optimal ratio was 1:2 citral to β-CD.

The main absorptions observed in the FTIR spectrum of β-CD (Figure 3a) correspond to those reported by Sambasevam *et al*. [32]. These are: 3294 cm^{-1}, 2928 cm^{-1}, 1642 cm^{-1}, 1157 cm^{-1} and 1022 cm^{-1} which corresponds to the symmetric and antisymmetric

stretching of O-H, CH_2, C-C and bending vibration of O-H respectively.

On the other hand, Figure 3b shows the difference in wave numbers between β-CD and the inclusion complex. It can be noticed that after the contact with the essential oil, most of the bands shifted to lower or higher wave numbers. Such shifts after formation of inclusion complex have already been observed by other authors [32].

Weishemeir and colleagues [16] found similar results when prepared micro particles containing β-cyclodextrin and *Cymbopogon citratus* essential oil. They observed that β-CD peak at 1.642 cm^{-1} shifted towards a higher wavenumber at 1653 cm^{-1}. Such shift was ascribed to the strong absorption of citral at 1.670 cm^{-1}.

Through the comparison between FTIR spectra of LGEO (Figure 2) and β-CD:LGEO complex (figure 3b), it is possible to perceive that the main differences are the shift of the bands at 3497 cm^{-1} and 1676 cm^{-1} to lower wavenumbers along with the suppression of the triplet in the C-H absorption region.

The results suggest the occurrence of intermolecular interactions between β-cyclodextrin and lemongrass essential oil, such as the formation of hydrogen bonding and the presence of van der Waals forces during their interaction to form the inclusion complex. The findings are in agreement with Restrepo *et al*. [19], who prepared inclusion complexes of lemongrass essential oil with β-cyclodextrin by supercritical CO_2 technique.

The thermal stability of LGEO in β-CD:LGEO was investigated by TGA. The TGA thermograms of pure β-CD (Figure 4a) and inclusion complex (Figure 4b) were given for comparison. In TGA thermogram of β-CD, initial weight loss at below 100 °C was due to water loss, and the major weight loss at around 300 °C corresponded to degradation of β-CD [33]. The cavities of cyclodextrins crystallised from water are not empty, but filled with water molecules. Some are included into the cyclodextrin cavity, others are integral parts of the crystal structure (crystal water). The cyclodextrin inclusion complexes are formed by substitution of included water by the guest molecule. The temperature reported for evaporation of such molecules was between 30 °C and 130 °C [34]. After loss of water the temperature kept stable presenting a plateau until to reach the temperature of β-CD degradation.

On the other hand, in the TGA thermogram of inclusion complex there was not observed loss of water in this range of temperature nor the plateau of thermal stability. Instead, it was perceived a slight constant inflection from the temperature of 25 °C until reaching the maximum degradation, which was attributed to gradual release of essential oil from the complex.

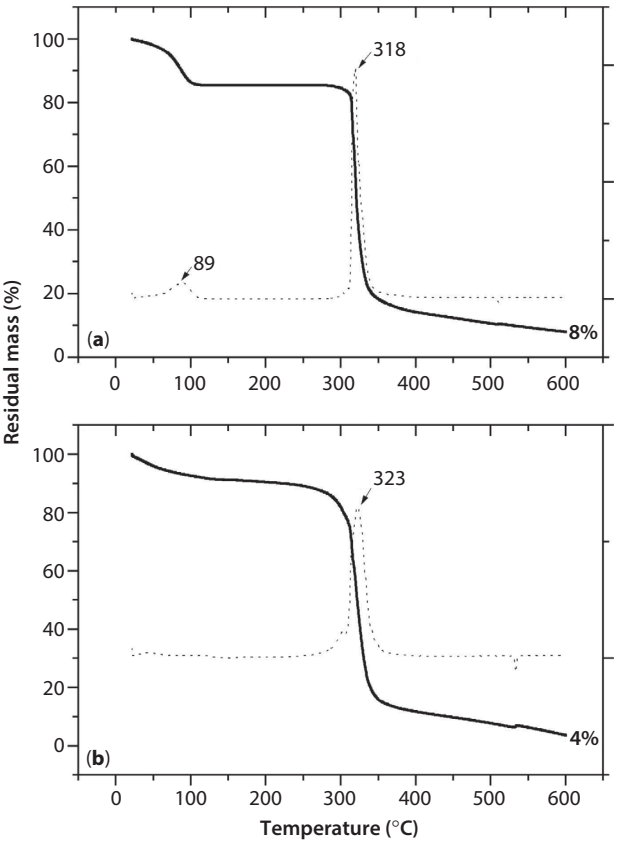

Figure 4 TG and DTG curves of: (**a**) pure β-cyclodextrin (β-CD) and (**b**) complex from 25–600 °C.

In addition the evaporation temperature of pure lemongrass essential oil is reported to be around 180 °C [15]. The absence of loss of mass in this range of temperature might be considered an indicative of the success of complexation. The enhanced thermal stability of the guest molecules included in the CD cavities has been reported for other CD complexes [35–39].

Scanning electron microscopy analysis allows qualitatively confirm the formation of inclusion complexes through direct assessment of morphology and particle size. The analysis consists in comparing the inclusion complex micrograph with those of the starting materials, for example. In this way marked differences between the micrographs usually are indicative of complex formation [34].

In the micrographs images showed in Figure 5, β-cyclodextrin presented a typical structure of parallelograms in the monoclinic form, with well defined surface boundaries and higher dimensions. The micrographs of the complex (Figure 6) exhibited the formation of aggregates with reduced particle size and modified morphology compared with pure β-cyclodextrin micrograph. The differences between the images were taken as evidence of the successful of complexation between β-CD and LGEO.

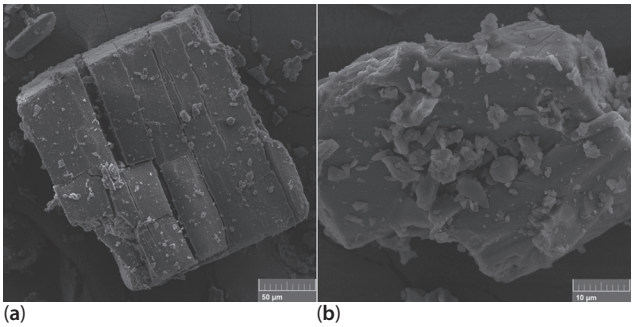

Figure 5 SEM micrographs of pure β-cyclodextrin: (**a**) at 1000 times of magnification and (**b**) at 5000 times of magnification.

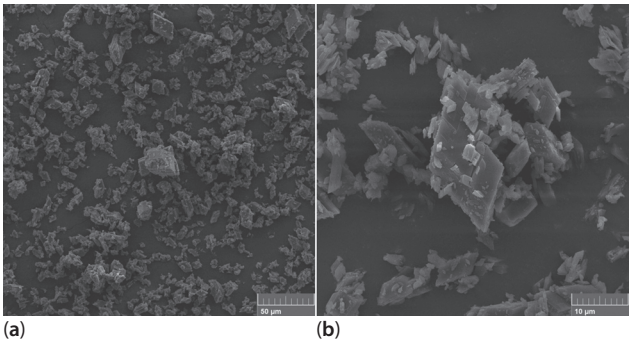

Figure 6 SEM micrographs of β-CD:EO: (**a**) at 1000 times of magnification and (**b**) at 5000 times of magnification.

3.3 Assessment of Active Coating Fixation onto Cotton Fabric

Figure 7 shows the subtraction of latex coated fabric spectrum from latex + β-CD coated fabric spectrum. The resulted spectrum still presented the main bands of pure β-CD (Figure 3a). This fact suggests that the synthesized latex is suitable for fixing β-CD on the fabric.

Moreover it was observed that the spectrum corresponding to the latex + β-CD:LGEO coated fabric overlapped with the spectrum of latex + β-CD coated fabric (Figure 8), suggesting that lemongrass essential oil (LGEO) had been protected inside cavity. The results found by Abdel Halim *et al*. [40] corroborate with this hypothesis. According to these authors, the superposition of the two spectra: modified textile with unload β-CD and modified textile with chlorhexidine diacetate loaded β-CD, was considered indicative of the presence of the complex in the textile.

Before the assessment of coating fixation by DRX, the crystalline structures of β-CD (control) and β-CD:LGEO complex were investigated. The analyses were performed taking into account some considerations done in the literature. As stated by

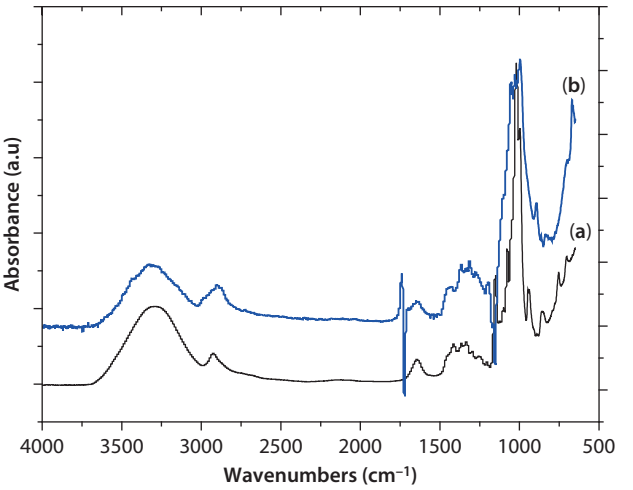

Figure 7 FTIR of: (**a**) pure β-CD and (**b**) subtracted spectrum.

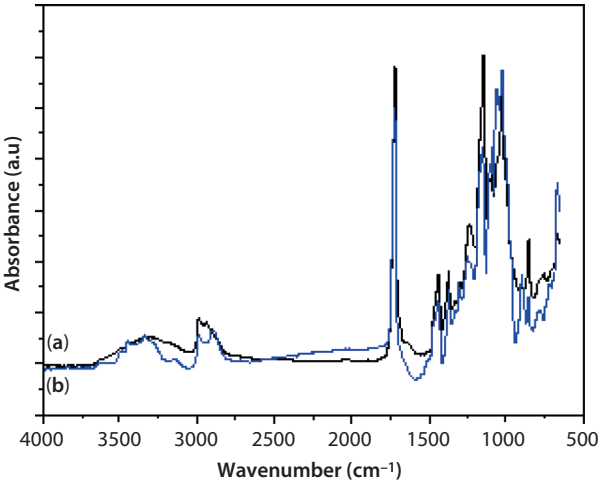

Figure 8 FTIR spectra of: (**a**) latex + pure β-CD coated fabric and (**b**) latex + complex coated fabric.

Rusa *et al*. [38], packing of the CD molecules within the crystal lattice occurs in one of two principal modes, described as cage and channel structures (Figure 9). According to them, all CDs in crystal hydrates usually adopt a cage structure.

Saenger *et al*. [41] reported that for β-CD the cage-type packing is observed with small alcohols, whereas complexes with larger alcohols and other guest molecules prefer the channel structure. However, these same authors pointed out that, only for α-CD the type of crystal packing is possible to be predicted with some degree of certainty. According to them, with small molecular guests α-CD forms cages, whereas with long molecular and ionic guests α-CD prefers channels.

The channel structure of α-CD has already been characterized by a strong reflection around 2θ = 20°

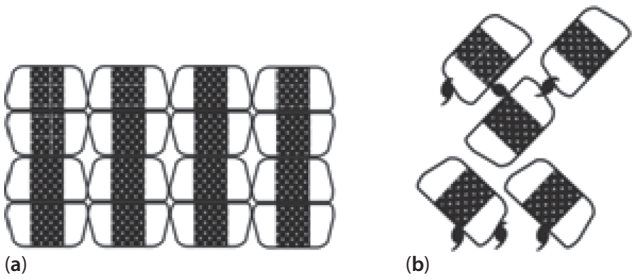

Figure 9 Packing of the CD molecules: (**a**) channel type and (**b**) cage type.

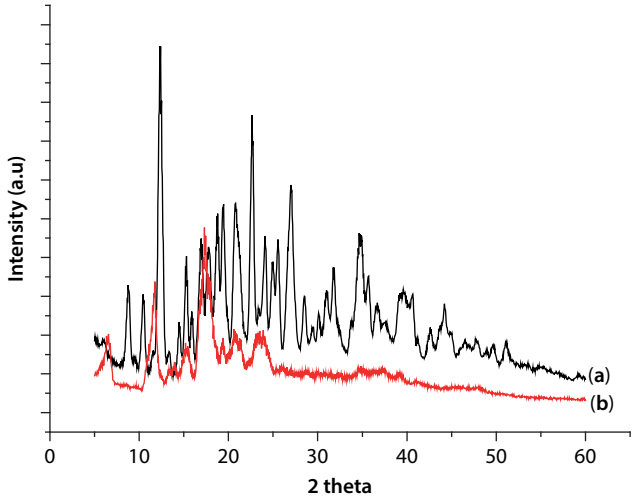

Figure 10 XRD patterns of: (**a**) pure β-CD and (**b**) β-CD:LGEO complex.

after poly(ethylene glycol) with molecular weights of 200 and 400 g/mol having been included in the CD cavity. Whereas the cage structure for α-CD was characterized by a strong reflection around 2θ = 12° [42].

In spite of the constraints to characterize β-CD complexes by XRD, Kayaci *et al*. [33], reported that peaks at 2θ = 12° and 2θ = 17.7° were owing to channel-type packing of β-CD in the case of geraniol/β-CD inclusion complex.

The β-CD diffractogram presented in Figure 10a shows the narrow and intense peaks which, according to Kayaci *et al*., correspond to cage-type packing [33].

On the other hand, the XRD pattern of β-CD:LGEO complex doesn't match with those reported by the authors. One hypothesis for change in the diffraction pattern is that the lyophilization process used for complex production has modified the crystallinity of the material, since according to the literature the type of process used in the production of complexes with CDs can contribute to reducing crystallinity both of the host and guest. This was observed by Zarif *et al*. [43] whom have investigated the production of inclusion complexes of β-CD/vancomycin by freeze drying

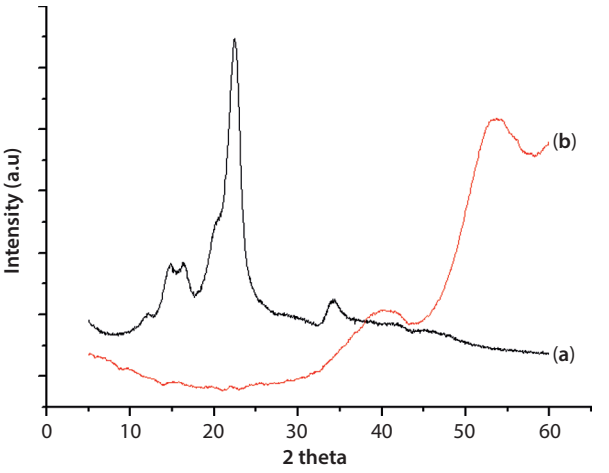

Figure 11 XRD patterns of: (**a**) fabric coated with pure β-CD and (**b**) fabric coated with β-CD:LGEO complex.

and kneading. Through characterization by XRD they concluded that in both methods, a significant reduction in crystallinity occurs mainly in the lyophilized complex.

Hashem *et al*. [44] investigated the production of inclusion complex of β-CD/oxatomide by the methods of mixing, coacervation, spray drying and freeze drying. By analyzing the XRD data it was also verified a reduction of crystallinity in the complexes relative to β-CD. Among of the methods, lyophilization and spray-drying produced the less crystalline complexes.

In view of the above discussion, it is reasonable to think that the transition between the cage and channel structures of β-CD was not clearly detected due to changes in crystallinity caused by the process for producing the complex.

Figure 11 shows XRD patterns of fabric coated with pure β-CD and fabric coated with β-CD:LGEO complex. As can be observed, the fabric coated with pure β-CD still remains crystalline. This fact is indicative of the presence of fixed β-CD, since the difratogram of the fabric coated with pure latex (not shown) corresponds to that of an amorphous material. On the other hand, the fabric coated with β-CD:LGEO complex presents a completely amorphous pattern. This result corroborates with the hypothesis that lyophilization changed the crystallinity of β-CD. However, it suggests that the use of latex is a potential approach for fixation the complex onto the fabric.

4 Conclusion

Lemongrass essential oil (LGEO) is bioactive, biodegradable, biocompatible and safe additive already used in food, drugs, cosmetics, and

sanitizing products. It can be considered as a promising candidate for textile functionalization, due to its effective antimicrobial action and pleasant fragrance. Its insolubility in water and poor thermal stability can be overcome by the formation of inclusion complexes between LGEO and β-cyclodextrin (β-CD). In the present work we investigated the formation of inclusion complex between β-cyclodextrin and lemongrass essential oil (β-CD:LGEO) and its fixation onto cotton fabric. LGEO was chemically characterized by gas chromatography-mass spectrometry (GC-MS) and confirmed citral as the major component of lemongrass, responsible for its antimicrobial activity. The experiments indicated successful complexation between LGEO oil and β-CD in molar ratio of 0.015:0.018 mol/L. The permanent fixation of pure β-CD and β-CD:LGEO onto cotton fabric was indicated by FTIR and XRD analyses. The results were promising regarding the use of β-CD:LGEO produce high performance textile, safe for the user and for the environment.

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