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Cyclodextrin-coated denim fabrics as novel carriers for ingredient deliveries to the skin

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ABSTRACT

Garments with coverage of a large part of the body generate an exceptional opportunity for transferring active ingredient to the skin. A fabric finished with β-cyclodextrin derivatives can play a role as a host for a next active substance as a guest. In this paper, different methods for permanent fixation of Monochlorotriazinyl-β-Cyclodextrin (MCT)-β-CD, on inner side of a denim fabric were investigated as a carrier with the purpose of substance formation inclusion complex with (MCT)-CD hydrophobic cavity. In order to fix MCT-CD on the fabric surface, two methods were investigated: (1) simultaneously or one step- and (2) pre- or two step-carbonating with sodium carbonate. Varying chemical and physical analyses such as gravimetrical and elemental analysis, FTIR spectroscopy, SEM images and fastness properties measurements, were studied to evaluate the quality of the host molecule stabilization on the textiles substrates and distinction of better application method.

1. Introduction

The new development in textiles is focused on the health promoting aspects with delightful functional properties (Wang & Chen, 2005; Singh, Varun, & Behera, 2011) and assist skin protection (Cheng et al., 2009). Textiles have always been considered as a “second skin” for human due to its health care effect in addition to the protection effects (Rubio et al., 2010).

While a wide variety of promising textiles based drug delivery technologies have been developed, there are several methods for designed textiles based slow release system by textile finishing including: (1) microencapsulation, (2) the host–guest interaction of Cyclodextrin, and (3) the active softener (Cheng et al., 2009). In the second method, cyclodextrin (CD) can be obtained through the enzymatic degradation of starch. The most common and industrially available types of cyclodextrins are α, β and γ-cyclodextrin with 6, 7 and 8 glucose units, respectively (Ammayappan & Moses, 2009; Holme, 2007). Above all, β-CD is the most interesting to be used in textiles due to simple production, availability-commercially production, cavity diameter, price and ease of attachment to textile surfaces. Cyclodextrins are considered as very important environment friendly auxiliaries due to their biodegradability and no toxic symptoms (Lee, Yoon, & Ko, 2000; Wang & Chen, 2006). Cyclodextrins are capable of forming inclusion compounds with a large number of organic materials. Therefore, textiles with new functional properties can be achieved (Hebeish & El-Hilw, 2001; Voncina & Majcen Le Marechal, 2005) while their release rate greatly decreases. β-CD finishing offers the textiles new functionality such as deodorant, insect repellency, antibacterial agent, auxiliaries in washing and dyeing processes, hydrophilicity, UV protection, slow release fragrances or skin-care components and stabilization of active ingredients (medical, hygienic, cosmetic, aromatherapy and home textiles) (Abdel-Halim, Fouda, Hamdy, Abdel-Mohdy, & El-Sawy, 2010; Ammayappan & Moses, 2009; Cabrales, Abidi, Hammond, & Hamood, 2012; Hebeish, Fouda, Hamdy, El-Sawy, & Abdel-Mohdy, 2008; Voncina & Majcen Le Marechal, 2005; Wang & Chen, 2005).This potential of β-CD originated from its peculiar shape and the presence of a hydrophobic cavity that produce the extraordinary ability to form stable inclusion compounds (Kistamah, Carr, & Rosunee, 2006; Nostro, Fratoni, & Baglioni, 2002). Inclusion complex formation is mainly affected by the geometric shape of the molecule rather than chemical interactions. The inclusion occurs through the displacement of water molecules from the hydrophobic cavity by the hydrophobic guest molecules, thereby leading to a more stable lower energy state. Inclusion complexes are in fact energy favorable. No hydrogen bonds are formed or broken during the formation of such host–guest complexes (Bhaskara-Amrit, 2011; Buschmann, Knittel, & Schollmeyer, 2001; Lee et al., 2000). Generally, water is used as a solvent for complex formation reactions. Once a formed complex is

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placed in water, the complex dissolves and equilibrium is reached between the free and complex cyclodextrin. Small amounts of water as they are set free from the skin are essential for the release of the active molecules. Afterwards the reloading of the cyclodextrin cavities with other organic molecules is possible again (Bhaskara-Amrit, 2011; Buschmann et al., 2001).

Denim goods could potentially present excellent effective base to be used in cosmetic textiles with a gradual release active materials. Since the denim cotton garments are extremely popular around the world and people put it on for long time during daily life, in this research, the 100% denim cotton fabric was selected as a natural plant fiber with its unique comfort properties as a carrier of active ingredients after finishing with CD. Therefore different methods were tested for the fixation of CD on the washed fabric and creation of a proper host–guest system. The finished fabrics were evaluated for their chemical grafting of CD and changes in physical properties with SEM images, weight gain (gravimetric), FTIR, elemental analysis, moisture regain, air permeability, wash and abrasion tests as well as bending properties in an attempt to find out the best procedure for fixation of CD derivatives.

2. Experimental

2.1. Materials

Indigo dip dyed denim fabric (100% cotton, 390 g/m^2, 3/1 serge) was selected as a textile substrate. Monochlorotriazinyl-β-Cyclodextrin (MCT-β-CD) was obtained from Wacker Chemical, Germany. Sodium carbonate and other chemicals were of analytic grade from Merck, Germany.

2.2. Fabric preparation

The denim fabric was firstly enzymatically desized and washed to remove any possible impurities which can adversely affect the surface treatments by 1 mL/L non-ionic detergent and 0.2 g/L sodium carbonate (pH 8–9) with L:R of 30:1 at 50–55 °C for 45 min. Then samples rinsed for 60 min and air-dried at ambient condition.

2.3. Fixation of MCT-β-CD onto denim fabric

The normal method for fixation of finishing agents on both sides of cotton fabrics is conventional pad-dry-cure method. Accordingly, in order to finish just inside the garment to have acceptable process cost, proper thickener agent was examined to control the finishing agent penetration to the other side of the fabric. Grafting of MCT-β-CD was carried out by coating with two different methods:

(1) Simultaneously treatment (ST) in which the MCT-β-CD (80 g/L) and required alkaline, sodium carbonate (50 g/L) were added to the thickener and coated on the fabric, (2) pre-carbonate treatment (PCT) that enhances reactivity of the fabric and minimize the reactive group hydrolysis due to simultaneous presence of MCT-β-CD and alkaline in aqueous solution, the cotton fabric was initially treated in an aqueous solution of Na_2CO_3 (20 g/L) for 5 min and air dried, after that the MCT-β-CD (80 g/L) was added to the thickener and coated on the fabric.

The impregnated samples were dried at 80 °C for 5 min, and then cured at 150 °C, for 5 min in the laboratory stenter ((Warner Mathis AG, Niederhasli/Zürich). Finally, the samples were rinsed thoroughly using hot tap water, followed by cold tap one for 10 min. The samples were given a final rinse with deionized water to remove all of the non-reacted MCT-β-CD and thickener from the samples surface, and again dried in air condition.

2.4. Characterization methods

2.4.1. Grafting evaluation

Total amount of fixed MCT-β-CD on the treated denim fabrics was evaluated gravimetrically from the weight difference before and after the above mentioned, ST and PCT, processes.

Attenuated total reflection infrared spectra (ATR-FTIR) of untreated denim, grafted denim and MCT-β-CD powder were taken on an FTIR spectrophotometer (Bomeme-MB 100) from their KBr disk. Each spectrum was scanned eight times with a resolution of 4 cm^{-1}.

Elemental analysis was performed by Elementar, Vario EL III to demonstrate the presence of nitrogen element on the treated fabric surface due to reactive group of MCT-β-CD. In this technique, a sample is burned in an excess of oxygen, and various traps collect the combustion products – carbon dioxide, water, and nitric oxide. The masses of these combustion products can be used to calculate the composition of the unknown sample.

Morphology of the samples surface was investigated by scanning electron microscope (SEM, XL30, Philips, Eindhoven, Netherlands) operated at an acceleration voltage of 20 kV. All of the specimens were sputter-coated with gold before microscopy, using an ion coater (SCDOOS, Bal-Tec, Balzers, Switzerland).

2.4.2. Physical evaluation

The effects of MCT-β-CD fixation on the fabric properties were evaluated by measurement of the wash fastness, moisture absorption, air permeability and bending properties of untreated, simultaneously treated (ST), and pre-carbonate treated (PCT) samples.

Wash fastness of samples were determined in accordance with AATCC Test Method 61-1994 tests No. 2A using a Wascator FOM after three wash cycles.

Abrasion resistance of samples was determined in according to BS 12947-2:1999, under pressure of 9 kPa s, by rubbing the circular samples against a standard wool fabric and the weight difference was evaluated after 3000 cycles.

As the cyclodextrin molecule contains many hydroxyl groups, after stabilizing of this molecule on the fabric surface, the ability to moisture absorption of fabric can be increase. Therefore, moisture regain, MR, of samples were measured by conditioning the samples at standard room by temperature of 25 °C and relative humidity of 65% for 24 h. Eq. (1) was used to calculate MR (Semnani, Mazrouei-Sebdani, & Abedinzadeh, 2013).

$$\mathrm{MR} = \frac{W_{\mathrm{water}}}{W_{\mathrm{dry\ fiber}}} \times 100 \tag{1}$$

where W_{water} is the amount of water absorbed by dry fabric from the air at the standard condition (ASTM D1576) and $W_{\mathrm{dry\ fiber}}$ is the weight of dry fabric.

The air permeability tests were performed in accordance with the BS 9237-1995 test method; after the samples had been conditioned for 24 h at 65% RH and 20 °C. Test surface area was 5.07 cm^2; pressure drop 100 Pa; and 10 tests for each sample. In this experiment the air permeability, R, in millimeters per second was calculated using Eq. (2):

$$R = \frac{qv}{A} \times 167 \tag{2}$$

where qv is the arithmetic mean flow rate of air, in cubic decimeters per minute (liters per min); 167 is the conversion factor from cubic decimeters (or liters) per minute per square centimeter to millimeters per second; and A is the area of fabric under test (in cm^2) (Mazrouei-Sebdani & Khoddami, 2011).

In this project, for evaluating hand properties, bending length (cm), bending stiffness (mg cm) and bending module (kg cm^2) were

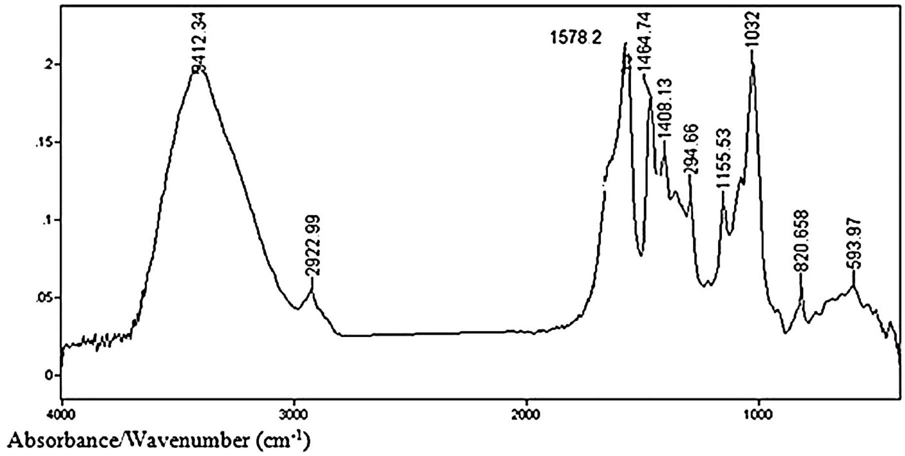

Fig. 1. FTIR spectrum of pure MCT-β-CD powder.

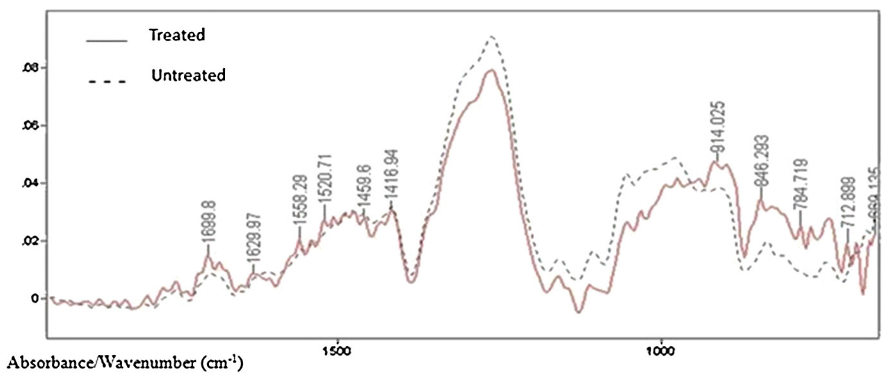

Fig. 2. ATR-FTIR spectrums of the untreated and treated denim fabrics with MCT-β-CD.

measured. Bending length of samples was determined according to BS3356 standard test method. To calculate the bending stiffness and module, bending length, weight per square meter and thickness of samples were evaluated.

Weight per square meter and thickness of samples were determined in accordance with ASTM: D3776-96 and ASTM: D1777-64, respectively. Therefore, bending stiffness and bending modules can be obtained from Eqs. (3) and (4):

$$A = WC^3 \times 10^3 \tag{3}$$

where W is the weight per square of samples (g/cm^2), C is the bending length (cm) and A is bending stiffness (mg cm).

$$q = \frac{12G \times 10^{-6}}{g^3} \tag{4}$$

where G is the bending stiffness (mg cm), g is the thickness of samples (cm) and q is the bending modules (kg/cm^2).

3. Results and discussion

3.1. Gravimetrical measurement

Gravimetrical measurement can estimate the efficiency of MCT-β-CD fixation on the substrate. Studying the dry add on percentage due to MCT-β-CD fixation on denim fabric was carried out by weighing samples finished with methods of ST and PCT as well as those just coated with thickener (alginate). This procedure enabled us to find out the weight gain due to the thickener, alginate residue on the treated samples. The results were shown in Table 1.

Table 1
Results of gravimetrically measurement.[a]

Treatment	Weight before treatment (g)	Weight after treatment (g)	% Add-on
One-step or Simultaneously treatment (ST)	6.45 ± 0.42	6.96 ± 0.45	7.81 ± 0.15
Two-step or per-carbonation treatment (PCT)	6.39 ± 0.36	6.86 ± 0.37	7.51 ± 0.30
Alginate only	6.01 ± 0.69	6.03 ± 0.69	0.27 ± 0.14

[a] Treatment denim fabric in one step, two step and thickener only.

The results indicated that the effect of the thickener on the weight increase is not considerable. Also, comparison of two impregnation methods showed that differences between two methods are not statistically significant. Thus two fixation methods used for fixation of MCT-β-CD on the denim fabric do not have any prominence than the others.

3.2. ATR-FTIR spectroscopy

Figs. 1 and 2 show representative FTIR spectra of MCT-β-CD pure powder, ATR-FTIR spectrum of the cotton denim fabrics untreated and treated with MCT-β-CD.

Since MCT-β-CD molecule has a triazinyl group, a mean absorption band related to triazinyl nucleus without any substitute can be observed at about 1350–1587 cm^{-1}. With inter chlorine and cyclodextrin as side chain of triazinyl ring, characteristic bands for the triazinyl nucleus was shifted to wave numbers of

Table 2
Percentage of four elements (N, C, S and H) in different CD treated fabrics.[a]

Elemental content (%)	Treatment		
	Simultaneously carbonation	Pre-carbonation	After one cycle washing
N	0.64	0.49	0.35
C	44.49	44.8	44.27
S	0.73	0.96	0.619
H	2.02	2.14	3.71

[a] Elementary analysis performed for three samples: 1 – one step, 2 – two step, and 3 – one step treatment sample after one cycle washing.

1400–1650 cm^{-1}. Therefore, Fig. 1 appeared the peaks at around 1464–1578 cm^{-1} wave numbers which are assigned for stretching vibrations of C=N.

Accounted by comparable chemical structure of cellulose and cyclodextrin, ATR_FTIR spectra of treated and untreated fabrics showed so much similarity. However, the treated fabric revealed the sharper absorbance bands at 1450–1550 cm^{-1}, compared to the untreated fabric, possibly due to C=N nucleus. In addition, in Fig. 2, mean difference between treated and untreated ATR spectra are in 700–1100 cm^{-1} related to breath move of aromatic rings with specified band in 820 cm^{-1} consigned stretching vibrations of C—H that are next to an unsaturated aromatic ring. All these features could be the characterization of triazinyl nucleus in the MCT-β-CD molecule. Accordingly, absorbance changes occurred in the treated fabric spectra (especially absorbance bands in the 846 and 914 cm^{-1}) indicated successful fixation of MCT-β-CD on the denim fabric. It is noteworthy that the samples finished with both methods of ST and PCT shoed similar spectra.

3.3. Elemental analysis and SEM images

The elemental analysis was carried out to observe nitrogen element to prove permanent grafting of MCT-β-CD to the fabric surface with proper fastness properties. The results of elemental analysis of the treated fabrics at different conditions, before and after washing, were indicated in Table 2.

The presence of nitrogen element on the treated fabric surface is obvious with acceptable wash fastness due to the detected nitrogen content after washing.

SEM was conducted to examine the effect of grafting on the cotton fabrics morphology. Fig. 3 shows SEM images of the untreated fabric and fabric grafted by MCT-β-CD. Fig. 3(A) shows the SEM micrograph of the untreated cotton fabric where the surface is smooth while Fig. 3(B) shows the grafted sample, in which knob and speckled on the fiber surface are revealed that confirmed the grafting of MCT-β-CD molecules.

Table 3
Weight reduction percentage of the CD treated samples after washing.[a]

Number of washing cycle	1 step	2 step	3 step
One-step (ST)	0.35 ± 0.08	0.29 ± 0.04	0.05 ± 0.03
Per-carbonation (PCT)	0.40 ± 0.08	0.11 ± 0.11	0.03 ± 0.03
Only alginate	0.19 ± 0.04	0.06 ± 0.05	0.01 ± 0.03

[a] Stability of β-cyclodextrin binding measure by washing three samples ST–PCT and thickener only in three times.

3.4. Wash fastness

In every treatment, wash fastness is an important parameter that must be assessed considering end use of the fabrics. Results of three cycles washing were summarized in Table 3. The washing durability was expressed in terms of weight loss percentage after washing.

According to the results illustrated in Table 3, the highest weight loss was occurred during first washing cycles with a decrease trend after that. While a part of the weight loss is due to the removing of the thickener, alginate, its amount is practically insignificant. Furthermore, after three washing cycles, ST samples or one-step process, PCT samples or two-step process and control sample treated with only with alginate, lost 0.69%, 0.55% and 0.26% of their total weights, respectively. Subsequently, these results indicate the suitable fixation of MCT-β-CD with good wash fastness while statistically studying did not distinguish two grafting method.

3.5. Physical evaluation

As already mentioned, two fixation methods so far showed more or less similar properties. Therefore, a variety of physical tests were carried out to compare untreated sample with grafted denim fabrics in addition to the comparison of two finishing procedures. The results are shown in Table 4.

3.5.1. Abrasion resistance

Since there is a friction between fabric and the body, thus evaluation of the finishing treatment durability against rubbing is vital. Abrasion resistance was performed according to BS 12947-2:1999 standard test method. The ST sample showed the highest weight reduction with statistically significant difference with the other comparable samples indicating lower abrasion resistance due to the simultaneous application of alkaline and MCT-β-CD leading to higher hydrolysis rate of the finishing agent.

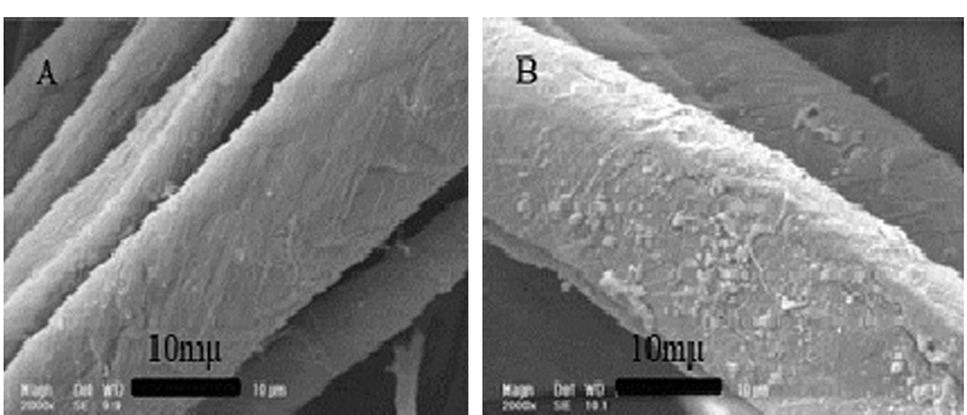

Fig. 3. (A) Untreated denim fabric and (B) treated denim fabric with MCT.

Table 4
Results of physical tests on the untreated and different CD treated fabrics.[a]

Tests	Treatment		
	Untreated	ST	PCT
-%Weight reduction (abrasion resistance)	0.39 ± 0.15	0.70 ± 0.21	0.45 ± 0.19
Moisture regain %	7.05 ± 0.32	7.28 ± 0.07	7.34 ± 0.18
Air permeability (mL(cm^2 × s))	11.02 ± 0.59	10.43 ± 0.49	9.93 ± 0.31
Bending length (cm)	4.96	5.92	5.98
Bending stiffness (mg cm)	6.47×10^{-4}	0.001	0.001
Bending module (kg cm^2)	1.76×10^{-5}	2.8×10^{-5}	2.6×10^{-5}

[a] Physical evaluation comparison between ST–PCT and untreated sample.

3.5.2. Moisture absorption

It is expected to observe better moisture absorption and water retention after grafting MCT-β-CD on the fabric surface because MCT-β-CD molecule has a large number of hydroxyl groups. Therefore, the effect of the grafting on the fabrics moisture absorption was studied. As expected, fabric moisture regain increased due to the hydrophilic nature of the CD molecules. But this amount is not statistically significant. However, the treatments did not show any adverse effect on the moisture regain.

3.5.3. Air permeability

The air permeability of a fabric is a measure of how well it allows the passage of air through the structure. The ease of air passage is of importance for a number of fabric end uses. According to Table 3, a decreasing trend was observed in air permeability of fabrics after MCT-β-CD treatment. As expected, the air permeability on the treated fabric surface was decreased when grafted with MCT-β-CD due to the decrease of the ways for passing air trough the fabric. Also, two treatment methods showed different permeability to air. This effect could possibly be due to the changes in fabric structure as a subsequence of alkaline behavior on the cotton fabric rather than the amount of fixed MCT-β-CD on the fabric surface.

3.5.4. Bending stiffness and bending modulus

The bending properties of all samples were increased after finishing with MCT-β-CD, Table 3, because of the coating of the fabric surface with the thickener and specially CD. This effect resulted from the increase in inter-fiber friction and the forces between yarns at cross over points which are reflected in the enhancement in stiffness. The adverse effects of finishing on bending properties of PCT and ST samples are not so significant while the differences between the untreated samples with finished ones according to the performed ANOVA test were statistically meaningful.

4. Conclusions

Denim fabrics are capable of being designed to fit the shape of body and contact directly with the skin providing qualifications required for gradual release of the active ingredients into different parts of body like legs and thighs. Therefore, it is crucial to create suitable system with proper release rate to use this kind of garment in cosmetic textiles. Accordingly, monochlorotriazinyl beta-cyclodextrin was applied to the denim fabrics as a carrier for the storage and control released of active ingredient while different possible methods were investigated to present appropriate fixation condition. Two methods were studied including: simultaneously application of MCT-β-CD with required amount of alkaline at the same step, and pre-carbonate in which the cotton fabric was initially treated in an aqueous solution of alkaline and then finished with MCT-β-CD.

Different physical and chemical techniques were examined to evaluate the rate of MCT-β-CD fixation, its fastness properties as well as changes in fabric properties due to the finishing. Present of MCT molecules on the grafted denim fabric surface was confirmed by gravimetrical, FTIR-ATR, elemental analysis in addition to SEM analyses. Quantitative tests and fastness analysis showed no significant differences between the two impregnation methods used in grafting steps. Coating fabric with cyclodextrin led to reduction of air penetration and hardening of the fabric hand. Also moisture absorption of grafted fabric was slightly increased due to available hydroxyl groups in the CD.

Overall, statistical analysis comparisons of the results of two grafting method by these parameters indicated that they have more or less similar characteristics with no superiority for any method. However, one-step, simultaneous method could be preferred for industrial applications due to its ease of application.

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