

# Application of Environment-Friendly Processes Aimed at Bonding $\beta$ -Cyclodextrine onto Cellulosic Materials

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## Abstract

*The impact of the pre-treatment of cellulose material in NaOH solution and of microwave radiation on the mechanism of crosslinking  $\beta$ -cyclodextrin with cellulose was investigated in this paper. Citric acid and Na-hypophosphite as catalysts were applied for better crosslinking. Inclusion compounds with tea tree essential oil and cyclodextrines grafted onto cotton fabric were made in order to verify that cavities were available for inclusion compounds. The permanence of  $\beta$ -CD on the cellulose fabrics treated was tested by ISO 6330:2012. The properties and structure of the materials treated were characterised by Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM). The breaking force and mechanical damage of the materials were determined according to EN ISO 13934-1:1999. The results indicated better bonding between cellulose and  $\beta$ -cyclodextrin where microwave treatment was applied, the phenomenon of which is of great importance for long lasting wash durability. In order to quantify the odour releasing behaviour of  $\beta$ -CD treated cellulose fabrics, olfactometric determination of the intensity of odour was undertaken.*

**Key words:** cellulose, pre-treatment, alkaline medium,  $\beta$ -cyclodextrin, citric acid, microwave.

## Introduction

Cyclodextrines (CDs) are cyclic oligosaccharides which can, due to their specific shape, form inclusive complexes with hydrophobic substances. Together with biodegradability and lack of toxicity, this property offers a wide range of possible applications in textile finishing processes. CDs are used in dyeing processes, where they form complexes with dye molecules, and, with some restrictions, as retardants and equalising agents. Apart from this, they reduce the amount of dyestuff in dyeing effluents. Adding CDs in the rinsing phase of washing processes removes unwanted surfactants residual on fabric [1]. CDs can also be permanently bonded to textile surfaces with the aim of modifying the properties of textiles. This is the method to achieve, depending on the complexing molecules, UV protection, antimicrobial protection, insect protection, reduction of unpleasant odours, or gradual release of pleasant ones. It is also possible to complex active pharmaceuticals, making the process applicable for medical purposes as well [2-4].

Cyclodextrines cannot be directly bonded to textile materials as they are not reactive. Literature mentions various methods of bonding [5, 6]. One of the frequently implemented is modifying CDs with trichlorotriazine, which results in monochlorotriazinil- $\beta$ -CD (MCT- $\beta$ -CD). The compound obtained is then grafted onto the textile material employing well-known methods of bonding reactive dyes. The methods of grafting MCT- $\beta$ -CD onto cotton [7], polyamide [8] and polypropylene fibres [9] have been investigated. The method of cross-linking cyclodextrine with cotton [10, 11], wool [11], polyester [12] and polyamide fibre [13] is often used as well, and polycarboxylic acids are frequently used as cross-linking agents (1,2,3,4-butanetetracarboxylic acid (BTCA), citric acid (CA), etc.), or epichlorohydrin and reactive dyes.

The purpose of this investigation was to establish an optimal method of bonding  $\beta$ -cyclodextrine onto cotton using citric acid as a mild and non-aggressive cross-linking agent. The method of cross-linking cellulose with polycarboxylic acids (PCA) and cyclodextrines in the presence of a phosphoric catalyst was developed by Martel et al [11]. The reaction can be seen in *Figure 1*. PCA is dehydrated at a temperature below the cross-linking temperature, which creates a temporary cyclic anhydride which readily reacts with hydroxyl groups in cellulose, through the process of esterification. The two remaining hydroxyl groups of the PCA react with each other

and create another anhydride which can then react with the hydroxyl group in another cellulosic chain or in cyclodextrine. To make this reaction possible, the PCA must contain at least three carboxyl groups, separated by one or two linked atoms.

As the success of esterification depends, among other things, on the availability of cellulose hydroxyl groups, we investigated the possibility of increasing the number of available hydroxyl groups by alkalisating the cellulose. In this process, part of the hydroxyl groups in the fibre reacts with sodium hydroxide and is transformed into -ONa groups, and the fibre swells. This type of cellulose is called alkali cellulose and is more reactive than natural cellulose [15, 16].

In an attempt to reduce the amount of energy and time necessary for the treatment, we performed cross-linking processes employing microwaves [17-19], comparatively with the conventional method of treatment. After applying  $\beta$ -cyclodextrine onto the fabric, we performed simultaneous conventional and microwave treatments adding tea tree essential oil, known for its antimicrobial properties. In this type of treatment the essential oil creates an inclusive complex with the  $\beta$ -cyclodextrine bonded to cotton, to be released gradually, forming long-term antimicrobial protection for the textiles treated. The characteristics of this protection will be investigated in the course of the investigation.

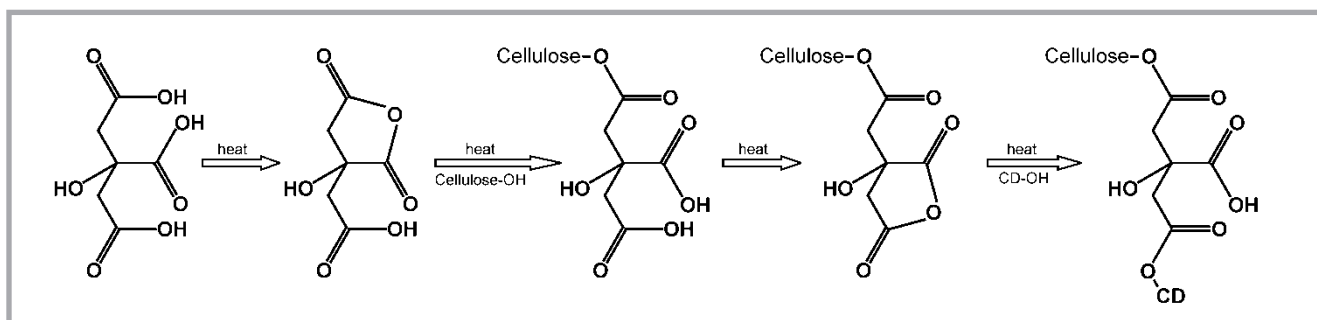


Figure 1. Cross-linking mechanism of  $\beta$ -CD and CA with cellulose [11, 14].

## Experimental

Optically brightened cotton fabric was used in the experiments, with a surface mass of 177 g/m<sup>2</sup>. Modifications to achieve target properties were performed using  $\beta$ -cyclodextrine, produced by *CycloLab R&D Ltd*. Tea tree essential oil (chemotype: terpineol 1,4, $\gamma$ -terpinen), produced by *Pranarom International*, was used to investigate the possibility of creating inclusive complexes.

A part of the samples was preacidicised for 60 seconds in 14% sodium hydroxide.  $\beta$ -cyclodextrine was bonded onto the fabric in a bath containing 25% cyclodextrine, 70 g/l of citric acid and 65 g/l of sodium hypophosphite hydrate as a catalyst, with the addition of 1 g/l of nonionic surfactant. The first group of samples was treated in a conventional manner. The samples were immersed in a bath at room temperature for 10 minutes, with no movement at all. The second group of the samples was placed in a Teflon vessel

of our own construction [20], with a bath in it, and treated in a microwave oven (*Tristar*, model MW-2896, 2450 MHz, nominal input power 1500 W) at 50% power, for 4 minutes, with a maximum temperature of 66 °C. After the treatment, all of the samples were squeezed on a padder, dried on a drying stenter at 80 °C for 4 minutes, and finally thermally cured at 170 °C for 2 minutes. Treatment formulations are shown in **Table 1**.

After treatment and drying, the fabric was washed in Mathis company equipment, in accordance with the standardised method ISO 6330:2000 [21], using a standard detergent, with no optical brightener. The procedure was used to investigate the permanence of  $\beta$ -cyclodextrine bonding to cellulose.

Essential oil complexing was carried out in an emulsion with 10% of oil to the mass of the fabric. Half of the samples were treated conventionally in Mathis equipment, at a temperature of 95 °C for 30

minutes. The other half was treated with microwaves for 15 minutes at the maximum measured temperature of 78 °C.

The samples treated were analysed using a spectroscope with a Fourier transformation of infrared spectrum (FTIR) (Perkin Elmer, software Spectrum 100). Four scans were made for each sample, at a resolution of 4 cm<sup>-1</sup> between 4000 cm<sup>-1</sup> and 380 cm<sup>-1</sup>. For better precision in detecting the links responsible for cross-linking  $\beta$ -cyclodextrine, the samples were treated with 0.1 M NaOH for 4 minutes prior to measuring, and dried in a drier at 100 °C [22]. The purpose of this treatment was to convert free carboxyl groups situated in the area of wave number 1580 cm<sup>-1</sup> into carboxylate anions. This left carboxylate esters in the area of 1730 cm<sup>-1</sup>, which were responsible for the bonds of cotton and CA, as well as of CA and  $\beta$ -cyclodextrine.

The degree of esterification (D.E.) was calculated for all of the samples, using the following formula [23, 24]:

$$D.E. [\%] = \frac{I_{1730}}{I_{1730} + I_{1570}} \cdot 100$$

Where:

D.E. – Degree of esterification

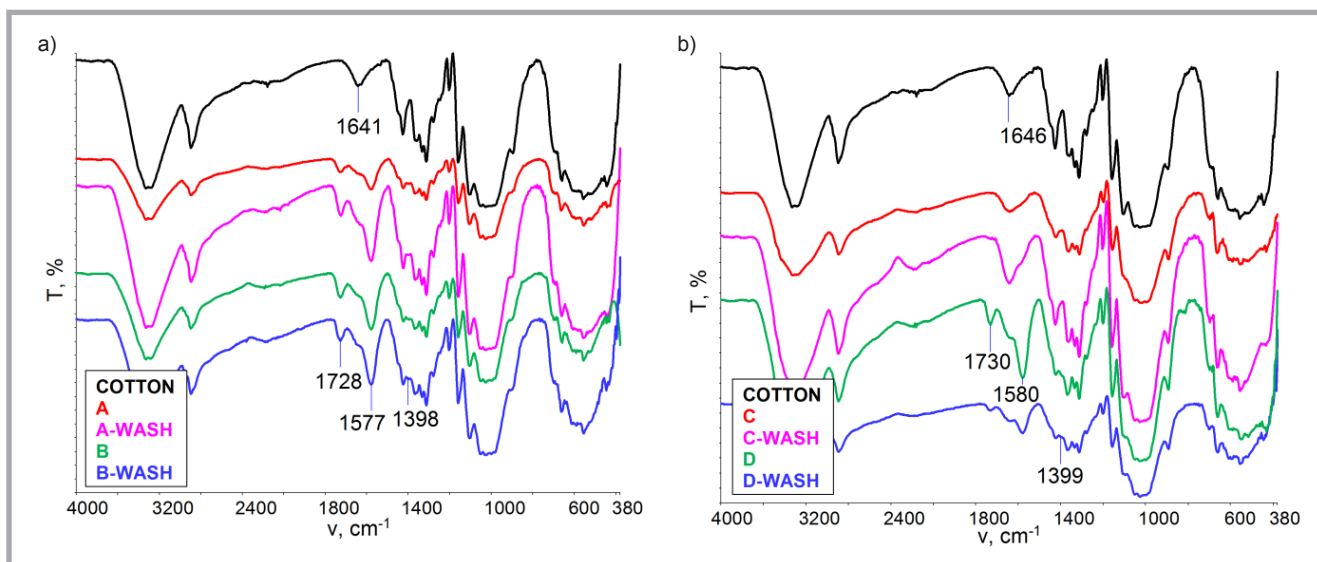
I – Intensity

The samples were scanned using a high-resolution scanning electron microscope (FE-SEM), Tescan, MIRA\LMU, Czech Republic, using different magnifications in order to investigate morphological surface characteristics and to confirm the bonding of  $\beta$ -cyclodextrine particles onto the cellulosic material. Prior to scanning, all samples were coated with a gold-palladium mixture in a SC7620 Sputter Coater Emitech steamer.

The breaking force ( $F_p$ ) and breaking elongation ( $\epsilon_p$ ) were measured for the untreated fabrics and treated samples ac-

Table 1. Samples are differentiated by the following treatments: 1. No pretreatment and pretreatment with NaOH, 2. Application of  $\beta$ -CD via cross-linking with citric acid by conventional and microwave heating, 3. No-wash and one washing cycle, 4. Essential oil application by conventional and microwave heating.

1. Pretreatment →	2. CD Application →	3. Wash&Dry →	4. Essential Oil
COTTON	A (conventional)	A	A-CON
		A-WASH	A-MW
		A-WASH	A-WASH-CON
	B (microwave)	B	A-WASH-MW
		B	B-CON
		B-WASH	B-MW
COTTON + NaOH	C (conventional)	C	B-WASH-CON
		C	B-WASH-MW
		C-WASH	C-CON
	D (microwave)	D	C-MW
		D	C-WASH-CON
		D-WASH	C-WASH-MW
			D-CON
			D-MW
			D-WASH-CON
			D-WASH-MW



**Figure 2.** FT-IR spectra of untreated and treated/washed samples.

according to the norm ENISO 13934-1:1999 [25] on a MESDAN-LAB Strength Tester dynamometer. All samples were conditioned prior to testing. Testing conditions were as follows: testing sample dimensions 100 mm x 50 mm, drawing speed 100 mm/min, and preloading 2N. Mechanical damage ( $U_m$ ) was calculated from the breaking force obtained experimentally using the following expression:

$$U_m = \frac{F_0 - F}{F_0} \cdot 100$$

Samples of 5 cm x 5 cm were prepared to determine the sample mass change compared with untreated samples. They were dried to an absolute dry condition and weighted on an analytical balance according to the norm ISO 3801:1977 [26].

The rate of discharge from the inclusive complexes was measured subjectively by periodically testing the sample scent intensity. The samples were individually put into plastic sample bags and left there for 7 days. They were tested by a committee of 7 members, who attributed the grades of scent intensity to samples, grading them from 1 to 5 (1 – no scent; 5 – intensive scent). After the first day of testing, the samples were left in the open air, strictly separated, at room temperature. The testing was repeated after 5 and 10 days.

## Results and discussion

**Figure 2** shows the FT-IR spectra of untreated and treated samples. Considerable changes can be seen on the treated samples in the area of carboxyl groups

at 1730  $\text{cm}^{-1}$  and 1580  $\text{cm}^{-1}$ . The peak in the area of 1580  $\text{cm}^{-1}$  represents free carboxyl groups, which were, with the aid of 0.1 M NaOH, converted into carboxyl anions. Carboxyl esters, responsible for the bonds of cotton and CA as well as of CA and  $\beta$ -cyclodextrine, thus remain in the area of 1730  $\text{cm}^{-1}$ . The peak in this area confirms successful cross-linking of  $\beta$ -cyclodextrine and citric acid with the cellulose on the samples, designated A, B and D. The peak on these samples could be clearly seen even after washing, which indicated permanence of the treatment.

No cross-linking occurred with the sample designated C. The reason could be that the treatment with alkali changed the pH of the bath from acidic to alkali, which probably affected esterification. The samples with confirmed cross-linking also exhibit a change in the wave area of 1398  $\text{cm}^{-1}$ , which indicates a kind of bending within  $\text{CH}_2$  on the C-6 atom of the cellulose structure.

Results for the esterification degree are presented in **Table 2**. It can be seen that the treatment with essential oil had no significant impact on the esterification degree achieved. The samples with no pretreatment (A, B) exhibited a similar esterification degree, regardless of the method of treatment. The preautoclaved samples treated in a bath using microwave energy (D) show the presence of ester bonds, which indicates cross-linking of  $\beta$ -cyclodextrine, citric acid and cellulose. No esterification was recorded with the preautoclaved and conventionally treated sample (C).

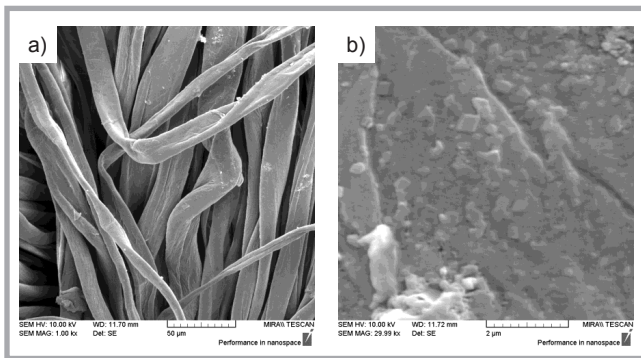
**Figure 3** shows a sample treated with  $\beta$ -cyclodextrine using a conventional method. Morphological characterisation of the sample fibre surface revealed inconsistent coverage of the particles, supposedly  $\beta$ -cyclodextrines.

**Figure 4** shows the same sample after washing, with the above particles also

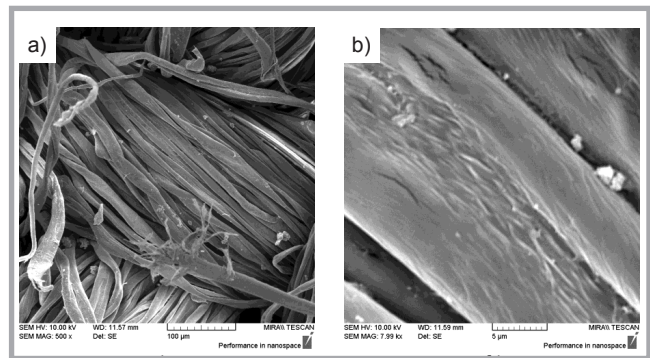
**Table 2.** Esterification degree for treated samples.

Samples	Esterification degree D.E., %	Samples	Esterification degree D.E., %
A	40.39	C	–
A-WASH	37.88	C-WASH	–
A-CON	40.37	C-CON	–
A-WASH-CON	31.63	C-WASH-CON	–
A-MW	40.40	C-MW	–
A-WASH-MW	39.87	C-WASH-MW	–
B	38.88	D	35.83
B-WASH	37.67	D-WASH	34.29
B-CON	38.12	D-CON	27.50
B-WASH-CON	37.62	D-WASH-CON	26.39
B-MW	37.08	D-MW	34.86
B-WASH-MW	36.48	D-WASH-MW	29.59

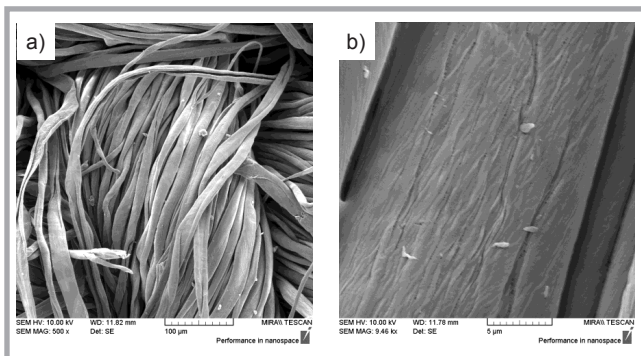




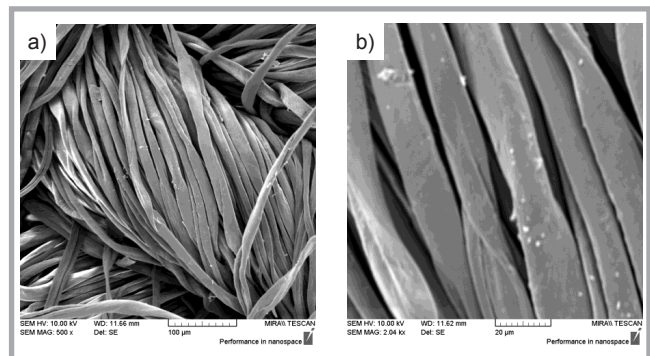
**Figure 3.** SEM images of the sample treated with  $\beta$ -cyclodextrine using a conventional method (A) at different magnifications: a) 1 kx, b) 30 kx.



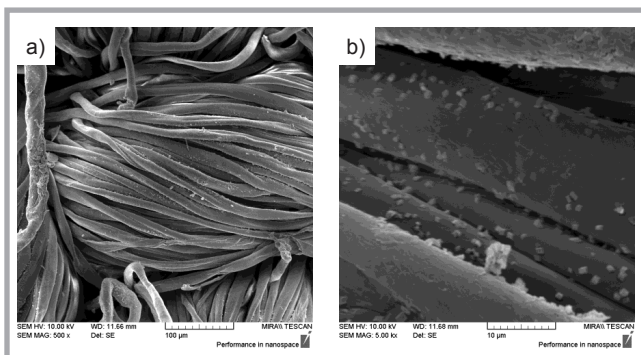
**Figure 4.** SEM images of the sample treated with  $\beta$ -cyclodextrine using a conventional method after one washing cycle (A-WASH) at different magnifications: a) 500x, b) 8 kx.



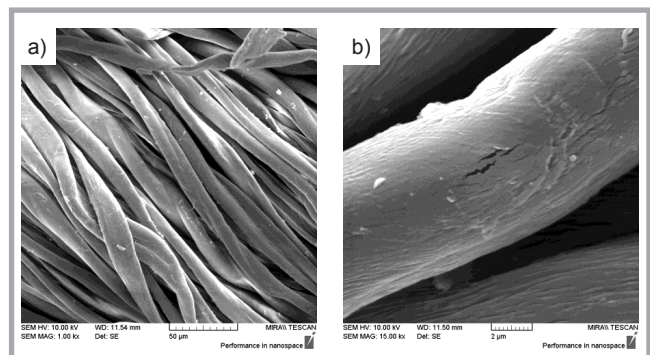
**Figure 5.** SEM images of the sample treated with  $\beta$ -cyclodextrine using a microwave oven (B) at different magnifications: a) 500x, b) 9.5kx.



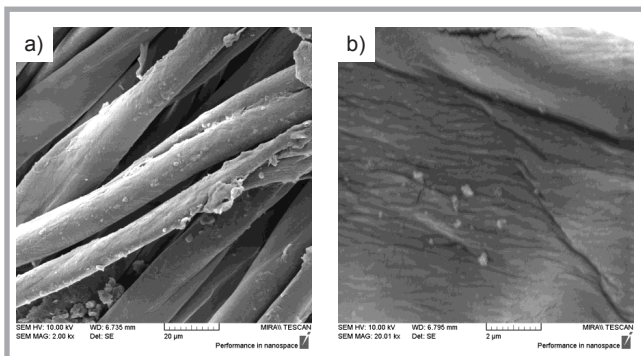
**Figure 6.** SEM images of the sample treated with  $\beta$ -cyclodextrine using a microwave oven after one washing cycle (B-WASH) at different magnifications: a) 500 x, b) 2 kx.



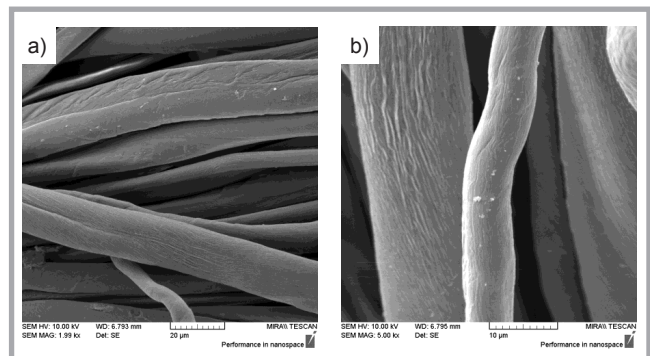
**Figure 7.** SEM images of the sample treated with  $\beta$ -cyclodextrine in a conventional manner; with pretreatment of 14% sodium hydroxide (C) at different magnifications: a) 500 x, b) 5 kx.



**Figure 8.** SEM images of the sample treated with  $\beta$ -cyclodextrine in a conventional manner; with pretreatment of 14% sodium hydroxide after one washing cycle (C-WASH) at different magnifications: a) 1kx, b) 15 kx.



**Figure 9.** SEM images of the sample treated with  $\beta$ -cyclodextrine in a microwave oven, with pretreatment of 14 % sodium hydroxide (D) at different magnifications: a) 2kx, b) 20 kx.



**Figure 10.** SEM images of the sample treated with  $\beta$ -cyclodextrine in a microwave oven, with pretreatment of 14 % sodium hydroxide after one washing cycle (D-WASH) at different magnifications: a) 2kx, b) 5kx.

present, indicating their bonding to the surface. However, their number was lower compared to the unwashed sample.

Figures 5 and 6 show the morphology of the B and B-WASH samples. Morphological characterisation by FE-SEM clearly exhibited the presence of  $\beta$ -cyclodextrine on the samples more equally distributed across the surface and in fibre pores. The  $\beta$ -cyclodextrine particles present were of spherical shape and considerably smaller in size, compared to the sample prepared by a conventional treatment.

Figure 7 shows fibre morphology for the sample treated with  $\beta$ -cyclodextrine in a conventional manner, with pretreatment of 14% sodium hydroxide. The fibre surface was covered to a significant degree by  $\beta$ -cyclodextrine particles and particle agglomerations.

Figure 8 shows a sample treated with  $\beta$ -cyclodextrine in a conventional manner, with pretreatment of sodium hydroxide, after a washing cycle. The quantity of particles was considerably reduced, as compared to the unwashed sample. However,  $\beta$ -cyclodextrine particles could clearly be seen in the structure of the fibres themselves.

Figure 9 shows the surface morphology of a sample fibre treated with  $\beta$ -cyclodextrine in a microwave oven, with pretreatment in sodium hydroxide. The fibre surface was unevenly covered with  $\beta$ -cyclodextrine particles of various dimensions.

Figure 10 shows the surface of a sample treated with  $\beta$ -cyclodextrine in a microwave oven, with pretreatment in sodium hydroxide, after a washing cycle. The reduced number of  $\beta$ -cyclodextrine particles was recorded on the surfaces of sample fibres.

Table 3 shows mechanical damage after treatment with  $\beta$ -cyclodextrine. Sample A exhibited strength loss weftwise and warpwise due to the acid treatment. Sample B exhibited more significant strength loss than the previous one, which can most probably be attributed to heating during the treatment applied. Samples C and D exhibited increased strength both in the warp and weft directions. Higher strength is a result of caustic treatment, which resulted in shrinkage and increased yarn density of the fabric.

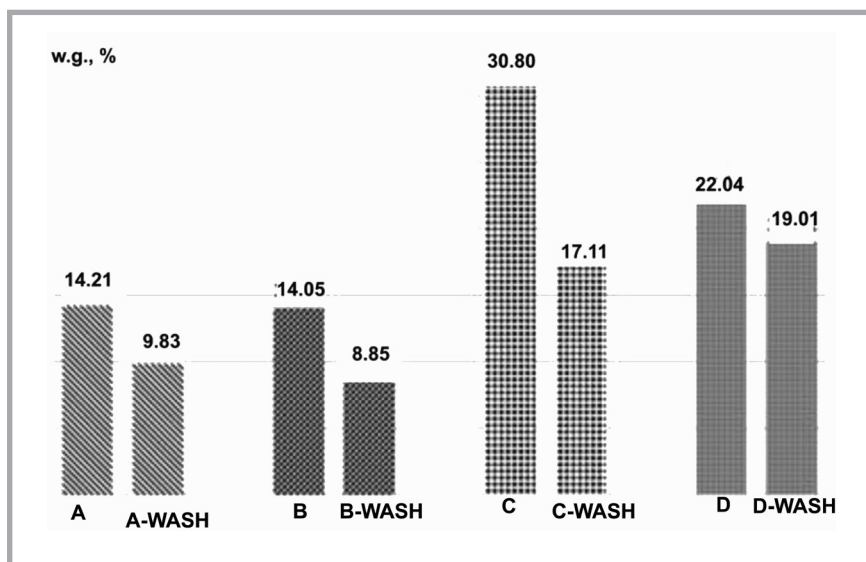


Figure 11. Weight gain (w.g.) [%] of the cotton fabrics treated with  $\beta$ -cyclodextrine.

Mass gain was tested for the samples treated with  $\beta$ -cyclodextrine, and their masses were compared to an untreated sample. The results obtained can be seen as a graph in Figure 11. Samples A and B, which were not previously caustically treated, exhibited approximately the same mass gain, which was reduced after a washing cycle, most probably due to the loss of  $\beta$ -CD that was not bonded. Samples C and D exhibited considerably higher mass gain. However, this was the result of higher fabric density due to shrinkage in caustic treatment, as well as the inclusion of  $\beta$ -cyclodextrine molecules in the cellulose chain structure. The reduced mass gain after washing for the conventionally treated sample C-WASH is much more pronounced than with other samples, which indicated weaker bonds of  $\beta$ -cyclodextrine with the fibre. Sample D exhibited the least difference in mass before and after washing, which points at proper bonding with  $\beta$ -cyclodextrine.

The results of periodic testing of the sample scent can be seen in the graphs in Figures 12 to 13.

The samples treated with essential oil in a conventional manner generally exhibited a considerably stronger odour than those in which essential oil was added in microwave treatment. This is probably due to the longer period of treatment.

A comparison of samples which had essential oil applied in the conventional manner and those pretreated with caustic revealed that the samples with no pretreatment had a stronger initial scent than

those pretreated. The intensity of the scent remained unchanged with the unwashed samples while it is moderately reduced with the washed ones. Sample C-CON, which was precausticised and treated with  $\beta$ -cyclodextrine in a conventional manner, was the only sample from the group of those treated in a conventional manner to lose the scent completely and not regain it, which led to the conclusion that  $\beta$ -cyclodextrine did not complex the essential oil, and that the concentration of  $\beta$ -cyclodextrine on that sample was nonexistent or much lower than on other samples. Sample D, precausticised and treated with microwaves, lost the scent completely, but only temporarily. After a time the scent returned, which indicated that  $\beta$ -cyclodextrine bonded to the sample and complexed the essential oil.

The ratings are considerably lower for the samples where essential oil was applied in a microwave oven, which makes the results less significant. The samples that were not precausticated exhibited loss of scent, to be regained after some time, the phenomenon of which being characteristic for the bonded  $\beta$ -cyclodex-

Table 3. Mechanical damage after treatment with  $\beta$ -cyclodextrine ( $Um_w$  – mechanical damage in warp direction,  $Um_p$  – mechanical damage in weft direction).

Sample	$Um_w$ , %	$Um_p$ , %
COTTON	0	0
A	17.90	17.62
B	38.09	42.25
C	-6.69	-5.24
D	-4.44	3.75

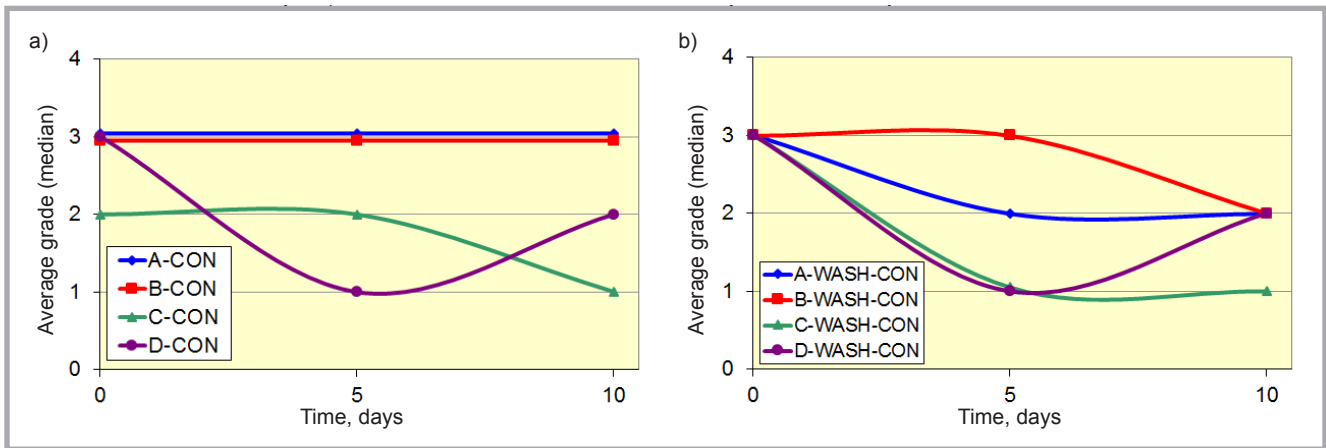


Figure 12. Odour release for conventional treatment with essential oil on a) unwashed samples and b) washed samples.

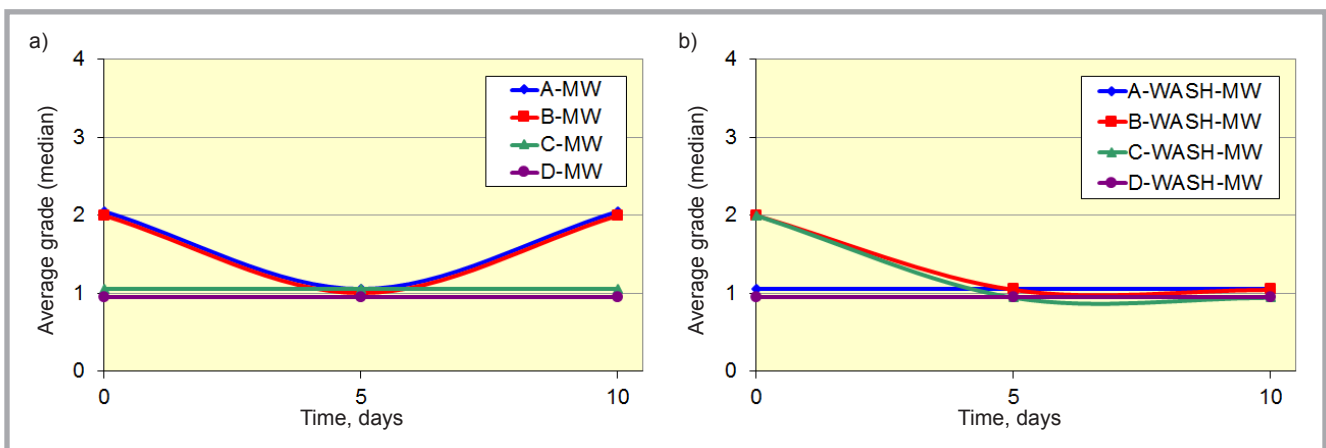


Figure 13. Odour release for microwave treatment with essential oil on a) unwashed samples and b) washed samples.

trine. To obtain a more significant result it would be necessary to use a longer time of microwave oven treatment.

## Conclusions

The impact of microwaves on  $\beta$ -cyclodextrin bonding on cotton was investigated, with citric acid as a cross-linking agent and sodium hypophosphite as a catalyst. The impact of pretreatment in sodium hydroxide was also investigated. Apart from microwave treatments, conventional treatments were also performed and results compared.

FE-SEM pictures confirmed successful bonding of  $\beta$ -cyclodextrin on cotton during microwave treatment, regardless of caustic pretreatment, which was also confirmed by the FTIR-ATR analysis. The samples treated in the conventional way exhibited successful cross-linking of  $\beta$ -cyclodextrin on the samples that were not pretreated, while no bonding occurred on those that were precausticated.

Sample B exhibited the highest mechanical damage, while sample D showed lesser damage than sample A. Overall sample D exhibited the best results in this aspect.

A subjective olfactometric method of determining the intensity of releasing scent in a time period was used to approximately define whether inclusive complexes could be formed of  $\beta$ -cyclodextrin bonded to cellulose and essential oil as a source of the scent. Based on the intensity of releasing essential oil from the inclusive complex, the best samples proved to be those designated A-CON, B-CON and D-CON. Treatment with essential oil in a microwave oven gave somewhat lower results, which was attributed to the shorter period of treatment. The results obtained require further investigations with the aim of achieving a stronger bonding of  $\beta$ -cyclodextrin with cellulose, as well as inclusive complexes with various substances, so as to be able to produce multifunctional textile fabrics.

## Acknowledgements

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