



## THE MECHANICAL AND PHYSICAL PROPERTIES OF THE FABRICS TREATED BY $\beta$ -CYCLODEXTRIN

Silva **KREŠEVIČ VRAZ**, Jelka **GERŠAK** and Bojana **VONČINA**

University of Maribor, Faculty of Mechanical Engineering, Department of Textile Materials and Design

**Abstract:** Research work deals with the possibility to achieve the insecticide properties of the textile substrates treated with ecological friendly insecticide reagents. For this purpose, textile substrates made of wool and polyester and their blends were treated with the  $\beta$ -cyclodextrine and cedar oil which can be used as a moth oppression reagent. Cedar oil forms in combination with the  $\beta$ -cyclodextrine which is attached on textile substrate, a complex from which the oil can be released slowly.

$\beta$ -cyclodextrine ( $\beta$ -CD) can be attached on the textile substrate by the procedure of non-formaldehyde finishing with the use of the polyfunctional reagent BTCA (1, 2, 3, 4 buthanetetracarboxylic acid).

On the basis of the preliminary tests it was concluded that cyanamid (CA) as a catalyst significantly reduces the fixation temperature of  $\beta$ -CD/BTCA onto textile substrate. It was confirmed that at higher temperature hydroxyl groups of  $\beta$ -CD together with BTCA carboxyl groups form ester bonds; the net structure is formed which is at the same time with a help of physical forces permanently bonded to the textile substrate.

Different analyzing methods were employed -  $\beta$ -cyclodextrin/BTCA amount was evaluated by gravimetric method, morphological changes were assessed with the electronic spectroscopy and differences to touch before and after treatment with the KES-FB measuring system.

**Keywords:** cyclodextrin, microwave, mechanical and physical properties of textile fabrics

### 1 INTRODUCTION

In our research we have grafted beta-cyclodextrin ( $\beta$ -CD) onto wool, PET and PET/wool blend textile materials using a polyfunctional reagent 1,2,3,4-butanetertacarboxylic acid (BTCA). To reduce the curing temperature of grafting, catalysts such as cyanamide (CA) was used. Nanoencapsulated wool, PET and PET/wool blend fibres were further treated with cedar oil, which is known for being a natural insect repellent. Nanoencapsulated textile materials after-treated with cedar oil showed prolonged insect resistant activities compared to those textile materials treated with cedar oil only.

Cyclodextrine can tie up on the surface of textile substrate, therefore we could add certain functionality to textile substrate.

### 2 THEORETICAL PART

#### 2.1 $\beta$ -cyclodextrin

Textile materials treated with  $\beta$ -cyclodextrin ( $\beta$ -CD), which is the cheapest among all CDs can be important for medical and hygienic textiles and for the garments which can be used for the cosmetic purposes and for home textiles [1].



All hydroxyl groups in CD are orientated away from the centre of the molecule (Figure 1), so the cyclodextrin molecule has a hydrophobic cavity, but the molecule itself is water soluble because of the many outward-pointing hydroxyl groups. From the structure of  $\beta$ -CD it is evident that it is not reactive; several binders and polyfunctional reagents have been used to link  $\beta$ -CD on textile substrates, mainly on cotton or viscose fibres [2-5].

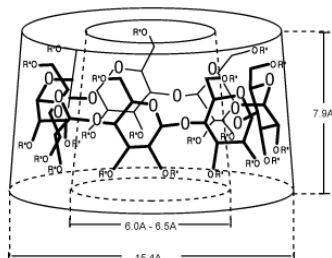


Figure 1: Structure of  $\beta$ -CD [6]

Cyclodextrins form inclusion compounds with various small molecules. Such complexes can be formed in solution, in the solid state, as well as when cyclodextrins are linked to a textile surface where they can act as permanent or temporary hosts to small molecules that provide certain desirable attributes such as fragrance and antimicrobial activity. Most frequently the host-guest ratio is 1:1. A basic criterion for the inclusion of guests into the cyclodextrin cavity is the size of the guests and the size of the cyclodextrin cavity.

## 2.2 KES-FB measuring system

The parameters of mechanical and physical properties were determined using the KES-FB measuring system (Figure 2). Each parameter plays an important role in the investigation of the interaction to achieve quality grade of the garment appearance [7].



Figure 2: KES-FB measuring system



### 3 EXPERIMENTAL

The influence of the fixation way of  $\beta$ -CD/BTCA on the change of mechanical and physical properties of different textile materials was investigated within the experimental part taking into account the goals of the research.

#### 3.1 Textile materials

Woven fabrics having three different material compositions were used for the research:

- 100 % wool fabric, plain weave, fabric weight of  $196.90 \text{ g/m}^2$ , warp 23 threads  $\text{cm}^{-1}$ , weft 31 threads  $\text{cm}^{-1}$ ;
- 100 % PET fabric, plain weave, fabric weight of  $70.20 \text{ g/m}^2$ , warp 32 threads  $\text{cm}^{-1}$ , weft 44 threads  $\text{cm}^{-1}$ ;
- Wool/PET blend (50:50) twill type of weave, fabric weight of  $262.20 \text{ g/m}^2$ , warp 25 threads  $\text{cm}^{-1}$ , weft 26 threads  $\text{cm}^{-1}$ .

#### 3.2 Fabric treatment

The textile materials were immersed in treating baths containing 8%  $\beta$ -CD, 6% BTCA, 1% CA, 1% ADHP; the wet pick-up was 100%; all the impregnated cloth was pre-dried at  $30^\circ \text{C}$  for 10 minutes. Two processes were employed to fix  $\beta$ -cyclodextrine onto the textile substrate surface: conventional thermo-fixation (TF- $115^\circ \text{C}$ , 3 min) and fixation with microwaves (MV, Energy –  $2 \times 500 \text{W}$ ).



Figure 3: Fixation with microwaves

The washing of textile materials was preceded at  $40^\circ \text{C}$  by standard the test method ISO 105-C01:1987(E). The weight gain of the finished fabrics was measured to yield the efficiency of the treatment according to standard test method DIN 53814.

#### 3.3 Mechanical and physical properties

With KES-FB measuring system the mechanical and physical characteristics of raw fabrics - wool, PET and wool/PET blend untreated and treated fabrics with  $\beta$ -CD/BTCA were determined.

Necessary for calculation of primary assessments feel H.V. mass of fabric is surface. Mass of fabric is being passed per unit of surface and is expressed in  $\text{gm}^{-2}$ , this chose for standard SIST ISO 3801.

Primary estimations of the fabric hand (H.V.) have been determined for all investigated fabrics in order to show the differences in applied materials.

### 4 RESULTS

Based on the research of the effect of fixation of  $\beta$ -CD/BTCA finishing on change of mechanical and physical properties of analysed fabrics, we present the results in the pt. 4.1. Table 1 presents the gain of mass using the gravimetrical method. Pt. 4.2. presents the results referring to the determination of



mechanical and physical properties and primary hand evaluations of fabrics, measured by KES-FB evaluation system, Tables 2, 3, and 4. Results gained by electronic microscope are presented in pt. 4.3, resp. in Figure 4.

#### 4.1 Gravimetric methods

Table 1: Gain of mass with  $\beta$ -CD/BTCA treated wool textile materials according to the treating methods, Gain of mass with  $\beta$ -CD/BTCA treated PET textile materials according to the treating methods, Gain of mass with  $\beta$ -CD/BTCA treated wool/PET blend according to the treating methods.

Samples	Fixation processes	pH of finishing baths	Gain on mass of $\beta$ -CD[%]
W – TF2	TF	2.3	3.10
W - TF4	TF	4.2	2.70
<b>W – MV2</b>	<b>MV</b>	2.3	<b>5.00</b>
<b>W - untreated</b>	-	-	-
P – TF2	TF	2.3	1.30
P - TF4	TF	4.2	1.60
P – MV2	MV	2.6	2.00
<b>P – MV4</b>	<b>MV</b>	4.0	<b>2.30</b>
<b>P - untreated</b>	-	-	-
M – TF2	TF	2.3	4.50
M - TF4	TF	4.2	4.00
<b>M – MV2</b>	MV	2.3	<b>5.60</b>
M – MV4	MV	4.0	5.20
<b>M - untreated</b>	-	-	-

The greatest gain of mass of  $\beta$ -CD/BTCA that was fixed on a textile material, was determined in samples W-MV2 (5%), P-MV4 (2.3%) and M-MV2 (5.6%), fixed in wet state with microwaves after the treatment.

#### 4.2 Results of measurements of mechanical and physical properties of analysed textile materials

Tables 2 and 3 present the results of those mechanical and physical properties of analysed fabrics that are most important for fabric hand.



Table 2: Tensile, shear and bending property with wool, PET and wool/PET blend textile materials

	WT [cN]		EMT [%]		G [cN]		2HG [cN]		2HG5 [cN]		B [cNcm <sup>2</sup> ]		2HB[cNcm]	
	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft	Warp	Weft
W-TF2	4.66	5.64	3.37	4.07	0.62	0.51	0.83	0.74	1.74	1.42	0.086	0.059	0.034	0.020
W-TF4	9.70	12.15	6.95	8.59	0.71	0.51	0.83	0.78	1.84	1.49	0.081	0.061	0.024	0.017
<b>W-MV2</b>	4.91	5.89	3.49	3.86	1.07	0.93	1.52	1.59	3.58	3.21	0.150	0.076	0.145	0.040
W - untreated	3.81	5.70	3.34	4.59	0.45	0.45	0.49	0.47	0.83	0.47	0.075	0.059	0.017	0.014
P-TF2	3.93	3.93	2.73	1.17	0.49	0.43	0.54	0.44	1.94	1.47	0.038	0.056	0.024	0.025
P-TF4	7.60	7.60	4.90	2.10	0.51	0.45	0.47	0.37	2.01	1.49	0.040	0.047	0.022	0.028
P-MV2	4.36	4.36	2.88	0.95	0.44	0.42	0.61	0.47	1.49	1.27	0.041	0.056	0.025	0.030
<b>P-MV4</b>	8.58	8.58	5.27	1.85	0.48	0.40	0.59	0.32	1.79	1.23	0.048	0.067	0.030	0.033
P - untreated	3.05	9.65	0.59	2.10	1.00	0.84	0.43	0.57	5.65	5.10	0.182	0.124	0.072	0.071
M-TF2	1.63	1.86	0.90	1.00	4.94	4.57	2.70	2.87	18.25	17.76	0.496	0.375	0.228	0.219
M-TF4	4.03	4.85	2.20	2.56	3.25	3.25	2.89	2.23	14.23	13.48	0.279	0.523	0.222	0.145
<b>M-MV2</b>	6.29	5.46	1.81	1.37	2.16	2.09	2.16	2.40	9.19	9.04	0.446	0.412	0.256	0.231
M-MV4	6.42	5.59	3.64	3.20	1.54	1.69	2.13	2.18	6.86	7.25	0.389	0.375	0.218	0.164
M - untreated	5.55	3.78	4.39	2.85	0.71	0.75	1.47	1.52	2.57	2.57	0.143	0.155	0.065	0.082

Table 3: Compression and surface property with wool, PET and wool/PET blend textile material

	WC [cNcm]	RC [%]	Tm [mm]	To [mm]	C [-]	MIU [-]		MMD [-]		SMD [μm]	
						Warp	Weft	Warp	Weft	Warp	Weft
W-TF2	0.22	55.46	0.31	0.66	0.53	0.168	0.173	0.010	0.015	3.92	5.72
W-TF4	0.20	58.37	0.30	0.60	0.50	0.173	0.182	0.011	0.020	3.59	6.06
<b>W-MV2</b>	0.27	61.01	0.46	0.73	0.37	0.174	0.191	0.010	0.022	3.73	6.11
W - untreated	0.32	55.88	0.37	0.82	0.55	0.165	0.184	0.011	0.023	4.42	6.82
P-TF2	0.08	75.74	0.13	0.34	0.62	0.192	0.238	0.016	0.010	4.34	1.95
P-TF4	0.06	80.63	0.15	0.22	0.32	0.159	0.214	0.009	0.010	3.50	2.15
P-MV2	0.07	49.22	0.22	0.29	0.24	0.165	0.225	0.016	0.009	4.79	2.15
<b>P-MV4</b>	0.08	66.88	0.16	0.22	0.27	0.160	0.218	0.018	0.012	4.54	2.34
P - untreated	0.05	56.00	0.19	0.23	0.17	0.173	0.143	0.010	0.021	1.71	6.02



	WC [cNcm]	RC [%]	Tm [mm]	To [mm]	C [-]	MIU [-]		MMD [-]		SMD [μm]	
						Warp	Weft	Warp	Weft	Warp	Weft
M-TF2	0.18	57.64	0.35	0.65	0.46	0.176	0.166	0.021	0.016	5.28	4.77
M-TF4	0.18	63.52	0.36	0.64	0.44	0.162	0.163	0.017	0.016	4.60	3.40
<b>M-MV2</b>	0.24	52.77	0.45	0.83	0.46	0.164	0.173	0.016	0.015	4.19	4.56
M-MV4	0.22	63.38	0.35	0.70	0.50	0.165	0.171	0.016	0.017	3.99	3.75
M - untreated	0.30	50.92	0.33	0.76	0.57	0.166	0.175	0.014	0.017	4.22	4.67

Analysis of those mechanical and physical properties of analysed fabrics, that are most important for fabric hand, shows in woollen wool/PET fabrics the increase of shear properties (G, 2HG, 2HG5) of treated fabrics (W-MV2, M-MV2), which is reflected with increase of primary hand values and significantly influences the fabric draping, production process, as well as appearance of finished garments. High values of bending rigidity influence favourably the formability of analysed fabrics. Deformation work WT, which influences the softness, flexibility and smoothness was increased in treated fabrics W-MV2, M-MV2, both in warp and weft directions. Deformation work WC was lower in treated samples W-MV2, M-MV2 comparing to untreated fabrics. Minor differences between the treated (W-MV2, M-MV2 and P-MV4) and untreated samples have been determined regarding the parameters of surface properties MIU, MMD and SMD – these properties influence the hand and appearance of the fabric. In samples of treated PES fabric P-MV4 we detected an increase of the deformational work WT and elongation EMT. The values of shear properties (G, 2HG, 2HG5) were lower in treated PES samples P-MV4 than in untreated ones, which is also reflected with lower values of primary hand values comparing with woollen fabrics W-MV2 and blend M-MV2. Lower values of bending properties (B, 2HB) of treated samples P-MV4 comparing to untreated PET fabrics show favourable effect of treatment on fabric hand and flexibility. Higher values of deformational work WC and compressibility C of treated samples P-MV4 comparing to untreated samples assure higher primary hand values.

Table 4: Results of primary hand values H.V. of analysed wool, PET and wool/PET blend textile materials

Samples	Koshi	Numeri	Fukurami	Sofutosa
W – TF2	5.36	4.95	4.50	4.02
W - TF4	5.34	4.93	4.48	4.00
<b>W – MV2</b>	<b>6.25</b>	<b>4.81</b>	<b>4.78</b>	<b>4.48</b>
<b>W - untreated</b>	<b>6.08</b>	<b>4.57</b>	<b>4.38</b>	<b>3.66</b>
P – TF2	4.39	4.90	2.31	2.41
P - TF4	4.37	4.89	2.29	2.39
P – MV2	4.33	4.93	2.42	2.62
<b>P – MV4</b>	<b>4.44</b>	<b>3.10</b>	<b>2.43</b>	<b>2.69</b>
<b>P - untreated</b>	<b>4.30</b>	<b>3.21</b>	<b>2.39</b>	<b>2.65</b>
M – TF2	9.18	3.28	3.78	0.89
M - TF4	9.17	3.26	3.75	0.86

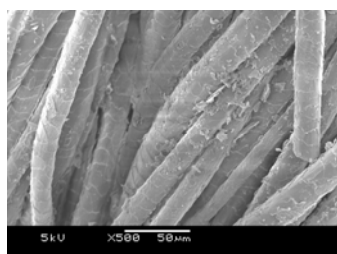


Samples	Koshi	Numeri	Fukurami	Sofutosa
<b>M – MV2</b>	<b>9.01</b>	<b>4.73</b>	<b>4.24</b>	<b>3.13</b>
M – MV4	8.79	4.78	4.29	2.08
<b>M - untreated</b>	<b>8.75</b>	<b>4.39</b>	<b>3.99</b>	<b>2.99</b>

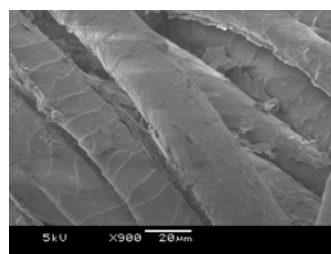
It can be seen from the results that fixation with microwaves of  $\beta$ -CD/BTCA treated textile materials proved to be more appropriate as conventional thermo fixation. Primary estimation KOSHI, which expresses rigidity, resp. elasticity, increases after treatment regardless used materials, same the primary value NUMERI. The value NUMERI was a bit lower in PES fabric, which reflects the elastic and soft character, characteristic for woollen materials. Primary estimation FUKURAMI reflects the fullness and softness, which was lower in PES fabric compared to woollen and blended fabric. FUKURAMI value was higher in all treated samples regardless the material composition. SOFUTOSA is a primary estimation, a combination of all three mentioned estimations and is important for a general evaluation of a fabric – it expresses the softness. It can be seen from the results that fixation with microwaves favourably influenced the improvement of the estimation of primary hand SOFUTOSA.

#### 4.3 Results of microscopy

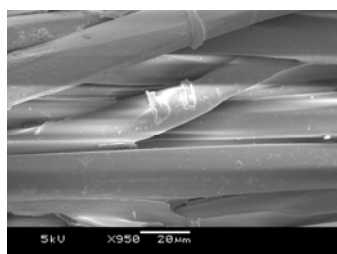
With electronic microscopes analysed of change surface of fibres before and for treatment with  $\beta$ -CD/BTCA. On figure 4 are showed analysed fibre before and after treatment with  $\beta$ -CD/BTCA.



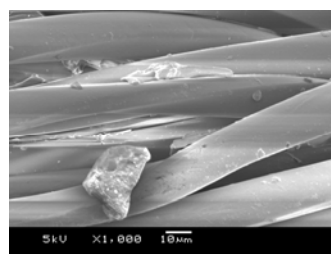
(a)



(b)

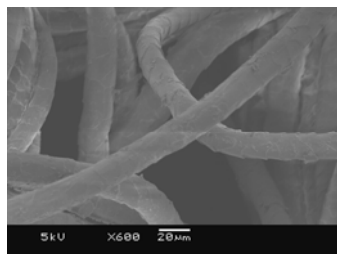


(c)

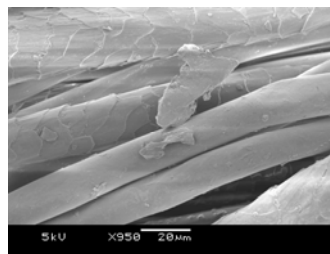


(d)





(e)



(f)

Figure 4 : Analysed fibre of woollen fabric W – MV2 with electronic microscope before (a) and after microwave fixing  $\beta$ -CD (b) Analysed fibre PET fabric P – MV4 with electronic microscope before (c) and after microwave fixing  $\beta$ -CD (d) Fibres of fabric analysed from mix of wool and PET fibres M – MV2 with electronic microscope before (e) and aftermicrowave fixing  $\beta$ -CD (f)

## 5 CONCLUSIONS

In our research we prepared wool, PET and wool/PET blend containing nanocapsules. BTCA molecules crosslink hydroxyl groups of  $\beta$ -cyclodextrins forming a network which is simultaneously physically anchored onto the textile substrate's surface.

$\beta$ -cyclodextrin fixed on textile materials with classical conventional thermo – fixation ( 115<sup>0</sup>C, 3 minutes) and as new manner of fixing did microwave fixing. We found, that use of microwaves improves efficiency of fixing  $\beta$ -CD/BTCA, above all at wool and wool/PET blend textile materials.

From KES-FB measuring system are on base sixteen parameters of mechanical and physical characteristics of textile materials found change of feel of untreated and of treated textile materials.

Results of the research showed the increase of primary estimation of hand value H.V. after the treatment with  $\beta$ -CD and fixation with microwaves in all treated fabrics, which is also reflected in improved hand of fabrics, treated with  $\beta$ -CD/BTCA.

## REFERENCES

- [1] Buschmann,H.-J., Knittel,D. and. Schollmeyer, E., (Inv.), Textile Material-Cyclodextrin, DP 40 35 378.8, 1990
- [2] Martel B, et al, *J. of Appl. Polym. Sci.*, 83, 1449, 2002.
- [3] Lee MH, *J Appl Polym Sci* 78: 1986-1991, 2000.
- [4] Lo Nostro P, *J. of Inclu. Phen. And Macro. Chem* 44, 423-427, 2002.
- [5] Vončina B., Majcen Le Marechal A., *Journal of Applied Polymer Science*, 96(4), 1323-1328, 2005.
- [6] Vončina B.: Cyclodextrins, TTRI, Avgust 2006
- [7] Geršak J.: Mehanske in fizikalne lastnosti tekstilnih materialov, Univerzitetni učbenik, UM, Fakulteta za Strojništvo, Tiskarna Tehniških fakultet Maribor, 2006





Author(s):

Silva KREŠEVIČ VRAZ M.Sc.

University of Maribor, Faculty of Mechanical Engineering, Department of Textile Materials and Design

Smetanova 17, SLO-2000 Maribor, Slovenia

Phone: +(386) (2) 220 7969

Fax: +(386) (2) 220 7996

E-mail: [silva.vraz@uni-mb.si](mailto:silva.vraz@uni-mb.si)

Prof. Jelka Geršak, Ph.D.

University of Maribor, Faculty of Mechanical Engineering, Department of Textile Materials and Design

Smetanova 17, SLO-2000 Maribor, Slovenia

Phone: +(386) (2) 220 7960

Fax: +(386) (2) 220 7996

E-mail: [jelka.gersak@uni-mb.si](mailto:jelka.gersak@uni-mb.si)

Assoc. Prof. Bojana VONČINA Ph.D.

University of Maribor, Faculty of Mechanical Engineering, Department of Textile Materials and Design

Smetanova 17, SLO-2000 Maribor, Slovenia

Phone: +(386) (2) 220 7911

Fax: +(386) (2) 220 7996

E-mail: [bojana.voncina@uni-mb.si](mailto:bojana.voncina@uni-mb.si)