

# Dyeing and stiffness characteristics of cellulose-coated cotton fabric

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**Abstract** In this study, the effect of cellulose coating on dyeing and other properties of cotton fabric was investigated. Three different reactive dyes were used for dyeing coated cotton fabric. The effect of cellulose coating on the dyeing properties of cotton fabric was studied by determining the *K/S* values of coated substrate at different concentrations of cellulose and dye. The *K/S* value decreased by 40–60 % with increasing coating concentration of cellulose from 0 to 5 %. The results show that the stiffness was increased from 0.16 to 2.50 N/m by coating of cellulose on the surface of cotton fabric. The stiffness was permanent as confirmed by ten multiple washings. Mechanical properties remained excellent. X-ray diffraction analysis showed that the amount of cellulose II increased slightly after solvent treatment. Fastness properties of cellulose-coated cotton fabrics against rubbing, washing, and perspiration were good.

**Keywords** Cellulose coating · Stiffness · Bending rigidity · Cellulose II · Dyeing

## Introduction

Cotton is a leading textile fiber due to its unique properties such as hydrophilicity, biodegradability, durability, good dyeability, and relatively low cost (Ali et al. 2015). Application of starch is widely used for increasing the bending rigidity of collars and sleeves of men's shirts and the ruffles of girl's petticoats. However, such stiffness is not permanent, because starch dissolves in water during washing and the fabric loses its stiffness, resulting in the need to reapply starch after each laundering. However, stiffness can be permanently increased by coating a fabric with cellulose, because cellulose (Yuan et al. 2015) is not soluble or dispersible in water like starch, meaning that the coating remains on the fabric.

In recent years, coating directly with cellulose, or its derivatives to achieve compatibility with the coated material, has been applied. Cellulose coating is used for different applications such as bioactive composite coating (Imran et al. 2010), wood coatings (Gruneberger et al. 2015), extending the shelf-life of rainbow trout fillets (Raeisi et al. 2015), high oxygen barrier and targeted release properties (Molgaard et al. 2014), active packaging (Lavoine et al. 2014), etc. Herein, we present a new method of coating as well as a new method to increase the stiffness of cotton fabric permanently by using cellulose solution. Cellulose solution means dissolution of cellulose in a suitable solvent. Cellulose is one of the most abundant biopolymers in Nature, consisting of repeated units of

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$\beta$ -(1→4)-linked D-glucose. Cellulose is not soluble or dispersible in water, which is why it will not dissolve in water during washing once coated onto cotton fabric and does not need to be reapplied like starch. Cellulose is not soluble in common solvents and does not melt before thermal degradation, because it has three alcoholic groups and forms complexes via inter- and intramolecular bonds (Klemm et al. 2005). Solvents such as NaOH–CS<sub>2</sub> (Shen et al. 2010; Wang et al. 2013), *N,N*-dimethylacetamide (DMAc)/LiCl (Dupont 2003), *N*-methylmorpholine *N*-oxide (NMMO) (Gao et al. 2011), ionic liquids (Gupta and Jiang 2015; Kimura et al. 2014), etc. can dissolve cellulose directly. For this study, the urea–thiourea–NaOH (Zhang et al. 2010) solvent system was chosen to prepare cellulose solution. This solvent system is powerful and can dissolve cellulose quickly. This solvent system can dissolve up to 6 % cellulose directly, and is inexpensive and less toxic (Zhou and Zhang 2000; Isobe et al. 2012; Renfrew and Taylor 1990; Jin et al. 2007). The degree of polymerization (DP) of cellulose decreases after dissolution in urea–thiourea–NaOH (Zhang et al. 2000), but this is not of great importance for coating. Cellulose solution can be coated onto the surface of cotton fabric by using roller padding to increase the stiffness of cotton fabric.

Herein, we discuss the effect of cellulose coating on dyeing of cotton fabric. Reactive dyes have the ability to form covalent bonds with the cotton substrate during their application process, providing a wide range of shades with outstanding washfastness and better lightfastness on cotton. These properties place this class of dyes at the quality end of the market (Renfrew and Taylor 1990). Reactive Red 240, Reactive Yellow 95, and Reactive Blue 49 dyes were used for dyeing cotton fabric in this work. Properties such as *K/S*, relative color strength, and *L* (lightness–darkness), *a* (negative—green to positive—red), *b* (negative—blue to positive—yellow) values (Leon et al. 2006) were studied to understand the effect of cellulose coating on dyeing.

## Materials and methods

### Materials

Vian Biocel V Mg-bisulfite softwood pulp was supplied by Lenzing Biocel Paskov A.S. Cotton fabric was obtained from Tepna Nachod, Czech Republic.

Urea was purchased from Pentachem. Thiourea, sulfuric acid, and NaOH were purchased from Lachner, Czech Republic. Reactive Red 240, Blue 49, and Yellow 95 reactive dyes were purchased from Synthesia a.s. All chemicals were of minimum assay of 99 % and were used without further purification.

### Coating of cotton fabric with cellulose

Cellulose solution was prepared by dissolving cellulose pulp (0, 1, 3, and 5 %) in the urea–thiourea–NaOH–water solvent system at –12 °C. Cellulose solution was applied on the surface of cotton fabric by roller padding at room temperature (RT, 15–20 °C). The padding duration was 30 s, which is why the dissolved cellulose did not precipitate during padding at 15–20 °C. After padding, the fabric was washed in 100 g l<sup>-1</sup> HCl (aq), followed by water till neutralization. Then, the coated fabric was dried at 70 °C for 20 min and then pressed by ironing. The amount of cellulose added is presented in Table 1.

### Dyeing with reactive dyes

The dyeing bath was prepared by dissolving NaCl (50 g l<sup>-1</sup>), Na<sub>2</sub>CO<sub>3</sub> (20 g l<sup>-1</sup>), and reactive dye in distilled water. Dyeing was carried out at 70 °C for 60 min at fabric–liquor ratio of 1:50. Dyeing was carried out with 3, 9, and 15 % dye concentration (%w/w). Then, the dyed samples were washed with hot (80 °C) water and dried at 70 °C in a hot-air convection oven for 30 min.

### Surface morphology

The surface morphology of control and 5 % cellulose cotton fabric was observed by scanning electron microscope (TS5130-Tescan SEM) at accelerating voltage of 30 kV.

### Color strength and related parameters

The reflectance of coated and uncoated fabric samples was measured using a spectrophotometer (Datacolor 110<sup>TM</sup>, Switzerland) at  $\lambda_{\max}$ , and the Kubelka–Munk equation was used to determine the *K/S* value of both fabrics as

**Table 1** Cellulose coating

Cellulose solution (%)	Weight of fabric before coating (g)	Weight of fabric after coating (g)	Added cellulose/g of fabric	Added cellulose (%)
1	4	4.05	0.012	1.2
3	4	4.08	0.020	2
5	4	4.15	0.037	3.7

$$\frac{K}{S} = \frac{(1 - R_{\lambda_{\max}})^2}{2R_{\lambda_{\max}}}$$

where  $K$  is the coefficient of absorption,  $R_{\lambda_{\max}}$  is the reflectance of the fabric at peak wavelength, and  $S$  is the coefficient of scattering.

The relative color strength and color difference between cellulose-coated and uncoated dyed samples were calculated using the following formula:

$$\begin{aligned} \text{Relative color strength (\%)} \\ = \frac{K/S \text{ of coated sample}}{K/S \text{ of uncoated sample}} \times 100, \end{aligned}$$

$$\Delta E = \sqrt{(\Delta L)^2 + (\Delta a)^2 + (\Delta b)^2},$$

where  $\Delta L = L_{\text{coated}} - L_{\text{uncoated}}$ ,  $\Delta a = a_{\text{coated}} - a_{\text{uncoated}}$ ,  $\Delta b = b_{\text{coated}} - b_{\text{uncoated}}$ , where  $L$  is lightness, the  $a$  value quantifies the redness–greenness, and the  $b$  value quantifies the yellowness–blueness (Dev et al. 2009).

#### Fastness properties

The coated samples were washed according to the standard conditions given in the ISO 105-C06 test method to assess staining of adjacent fabrics and color change after washing. The rubbing fastness of both coated and uncoated samples was evaluated according to the ISO 105-X12 test method. The coated fabrics were evaluated for their perspiration fastness using the ISO 105-E04 test method.

#### X-ray diffraction analysis

The crystalline structure of cotton fabric was analyzed using an X'Pert<sup>3</sup> X-ray powder diffractometer (PANalytical, USA) at angles from 8° to 70° in steps of 0.017°. The amount of cellulose II was calculated using the simulation method (French 2014; Nam et al.

2016; Yue et al. 2015). The crystallographic information file containing the published coordinates of the asymmetric units of cellulose I $\beta$  and cellulose II was downloaded from the supplementary material of the paper by French (2014). The whole provided contents were copied into a notepad file. The I $\beta$  unit cell was adjusted from  $a = 7.784$  to  $7.906$  Å for cotton fabric cellulose and saved in .cif file format. The diffraction pattern was simulated using Mercury 3.5.1 software (Macrae et al. 2008). The full-width at half-maximum (FWHM) was set at  $2\theta$  of 1.5° with the Cu K $\alpha$  wavelength set at 1.54056 Å. A March–Dollase factor (Dollase 1986) of 2.0 was applied to the (0 0 1) plane to obtain the preferred orientation. For incorporation of the amorphous fraction into the simulation, the pattern of cellulose II with FWHM of 9° (Nam et al. 2016) was simulated using the Mercury software.

#### Tensile properties

The breaking force of control and coated fabrics was measured to determine the effect of cellulose coating on the tensile properties of the cotton fabric, measured using a TIRA test 2300 Labor Tech machine according to the ISO 1924-2 standard test method.

#### Air and water vapor permeability

The air permeability of control and coated cotton fabrics was analyzed using a Textest FX instrument according to the ASTM D2986 standard test method. The air permeability was measured at pressure of 200 Pa and range of 3. The measuring principle depends on measurement of the air flow through the fabric under a certain pressure gradient  $\Delta p$ .

The water vapor permeability was analyzed using a Permetest device (Sensora Instruments) (Hes and Loghin 2009) with the fast skin model according to the ISO 11092 standard.

## Stiffness of treated fabric

A TH-7 instrument (Fridrichova 2013) was used to measure the bending force of the fabric. The stiffness was calculated from the value of the bending force. The TH-7 is a new instrument for measurement of textile bending rigidity and is comparable to the standard device Kawabata KES-FB 2.

## Results and discussion

### Color strength and related parameters

The *K/S* value of the dyed fabric was directly proportional to the amount of dye present in the fabric. The relative color strength (%) and *K/S* values of the cellulose-coated dyed samples are presented in Table 2, from which it is clear that the *K/S* values of the cellulose-coated dyed fabrics were lower than for control dyed samples. Dye uptake decreased as the cellulose concentration was increased, as reflected in the observed values. Reactive Blue 49 dye showed better dyeability than Reactive Red 240 or Reactive Yellow 95. The lower *K/S* values for the cellulose-coated cotton fabrics are related to the applied cellulose. The possible cause of this decrease is that the coated pulp cellulose was mercerized after dissolution, reducing dye uptake by the fabric because it was on the surface. To investigate the change in the surface morphology of the coated cotton fabric, scanning electron microscopy (SEM) was used to estimate the influence of the modification process on the fabric. SEM images of cellulose-coated and uncoated cotton fabrics are presented in Fig. 1. These micrographs reveal that the applied cellulose formed a film on the surface of the cotton fabric, covering the spaces between yarns. Table 2 shows that the relative color strength (%) of the dyed samples decreased with increasing cellulose concentration for each shade % of the dye. These results further indicate that the cellulose coating decreased the dye uptake by the coated cotton fabric.

The Commission Internationale de l'Eclairage (CIE) *Lab* system was used to determine the color parameters and color variation, where  $L^*$  indicates the darkness–lightness value with 0 to 100 representing black to white, the  $a^*$  value goes from

negative (green) to positive (red), and the  $b^*$  value goes from negative (blue) to positive (yellow);  $\Delta E$  gives the total color difference. High  $L^*$  values indicate that the dyed coated sample was lighter than the uncoated sample. Reactive Blue 49 dye showed lower  $L^*$  values (Table 2) than Reactive Red 240 or Reactive Yellow 95. However, the  $L^*$  value increased with increasing cellulose concentration, meaning that all the coated samples were brighter compared with uncoated cotton fabric. The color difference values ( $\Delta E$ ) are presented in Table 2, showing that there was a significant color difference between the uncoated and coated cotton fabric samples with different cellulose concentrations, even though both samples were dyed with the same dye concentration. The  $\Delta E$  values for the samples dyed with Reactive Blue 49 were better than for the other two dyes used for dyeing. Hence, Reactive Blue 49 showed better dyeability of coated cotton fabric compared with Reactive Red 240 or Reactive Yellow 95.

### Fastness properties

During use, textiles are frequently subjected to washing, rubbing, and perspiration, so the durability of coated cotton fabrics under these conditions is very important. These effects were therefore evaluated, as presented in Table 3. Washing and rubbing fastness were evaluated for all three types of dyed fabric. The washing fastness of the cellulose-coated cotton fabrics against staining of adjacent fabrics (wool and cotton) was excellent (4–5) for all dyed fabrics, and the color change was also acceptable (2–3). The rubbing fastness ratings for the cellulose-coated cotton fabrics were good, namely 4 for dry rubbing and 3 for wet rubbing conditions. Thus, there was no significant impact of the cellulose coating on the washing or rubbing fastness properties of the cotton fabric.

The perspiration fastness results under acidic and alkaline conditions in terms of the grayscale ratings for cellulose-coated and uncoated samples are presented in Table 4. The grayscale ratings in the case of the cellulose-coated dyed samples for the color change were 2–3 under both acidic and alkaline conditions, meaning that the dyed samples were sensitive to pH. The ratings (4–5) for staining with wool as adjacent fabric were excellent, and those with cotton as adjacent fabric were also good. The perspiration

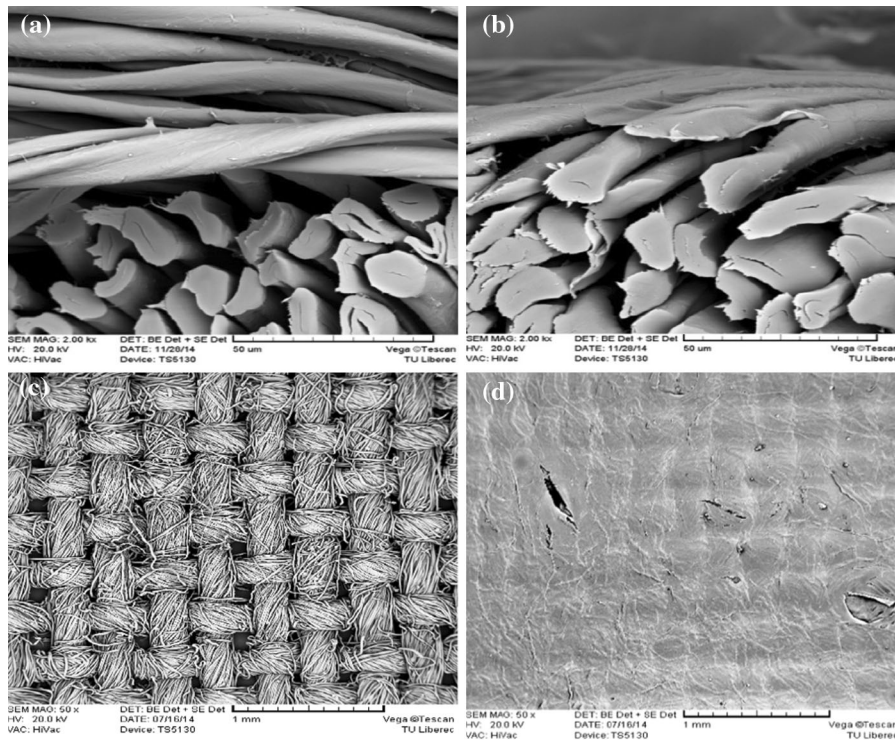
**Table 2** Spectrophotometric analysis of dyed samples

Dye content (% owf)	Dye	Cellulose concentration (%)	$L^*$	$a^*$	$b^*$	$\Delta E$	$K/S$	Standard deviation ( $\sigma$ ) of $K/S$	Relative color strength (%)	
3	Reactive Red 240	Control	53.22	61.15	2.25	0	7.29	0.38	100	
		1	59.33	62.21	2.75	6.22	2.54	0.95	34.87	
		3	61.34	62.98	3.23	8.38	2.36	1.23	32.45	
		5	63.75	63.26	3.56	10.81	2.25	1.54	30.97	
		Control	81.51	16.63	66.91	0	8.15	0.45	100	
	Reactive Yellow 95	1	82.59	17.26	67.49	1.37	3.74	1.12	45.98	
		3	82.87	15.14	61.93	5.37	3.07	1.45	37.72	
		5	84.32	21.28	73.46	8.51	3.21	1.62	39.45	
		Control	24.91	-3.89	-18.1	0	20.47	0.25	100	
		1	25.12	-3.45	-17.18	1.04	13.72	1.32	67.03	
	Reactive Blue 49	3	27.25	-2.77	-15.99	3.34	13.3	1.25	64.98	
		5	28.32	-2.45	-15.58	4.47	12.49	1.71	61.04	
		Control	42.17	59.55	13.72	0	20.75	0.61	100	
		1	47.26	61.1	5.66	9.65	12.14	1.98	58.51	
		3	48.37	61.85	5.72	10.37	8.79	1.59	42.36	
9	Reactive Red 240	5	49.98	62.12	5.85	11.38	7.7	1.92	37.15	
		Control	73.32	15.53	69.91	0	21.73	0.31	100	
		1	75.59	16.26	67.49	3.39	13.67	0.92	62.92	
		3	80.87	16.64	66.93	8.19	12.1	1.45	55.69	
		5	82.32	17.12	62.46	11.79	8.71	2.10	40.09	
	Reactive Yellow 95	Control	16.11	-4.22	-15.1	0	26.63	1.21	100	
		1	16.92	-3.35	-14.68	1.26	23.28	1.82	87.41	
		3	17.25	-3.67	-14.09	1.61	22.59	1.61	84.83	
		5	18.32	-3.75	-13.78	2.61	17.12	1.77	64.31	
		Control	37.94	57.92	14.72	0	24.91	1.10	100	
	15	Reactive Red 240	1	46.1	58.1	5.56	12.26	13.46	1.55	54.05
			3	47.21	58.98	5.61	13.04	12.4	1.20	48.32
			5	47.42	59.23	5.67	13.17	9.14	1.65	36.7
			Control	69.32	14.63	66.91	0	25.74	0.85	100
			1	72.52	17.26	59.49	8.49	16.13	1.62	62.67
Reactive Yellow 95		3	74.38	15.14	58.93	9.46	15.46	1.23	60.05	
		5	75.13	21.28	59.46	11.55	11.71	1.10	45.48	
		Control	12.11	-5.45	-13.01	0	29.68	0.52	100	
		1	15.02	-4.55	-12.88	3.04	25.6	0.95	86.26	
		3	15.95	-4.87	-12.19	3.96	24.78	1.09	83.49	
Reactive Blue 49		5	16.41	-3.95	-11.78	4.71	18.69	1.32	62.99	

fastness was observed for all three kinds of dyed sample, and the ratings were quite similar for all dyed samples. Hence, the perspiration fastness after coating cellulose on the surface of cotton fabric remained good.

#### Effect of solvent system on cotton fabric

The effect of the urea–thiourea–NaOH–water solvent system on the cotton fabric was analyzed using X-ray diffraction analysis. Figure 2 shows an overlay of the



**Fig. 1** SEM images of (a, c) uncoated cotton fabric and (b, d) cellulose-coated cotton fabric

X-ray diffraction spectra of the original (black) and regenerated (red) cellulose pulp, while Fig. 3 shows an overlay of the X-ray diffraction spectra of control (black) and urea–thiourea–NaOH–water-treated (red) cotton fabric. In Fig. 2, the characteristic peak of cellulose I at  $16.20^\circ$  is missing for the regenerated pulp cellulose while the peaks at  $12.3^\circ$  and  $21.20^\circ$  show that the structure of the pulp cellulose has been converted from cellulose I to II. The X-ray diffraction spectra and overlapping peak resolution of the control and solvent-treated cotton fabric show main characteristic diffraction peaks at  $14.7^\circ$ ,  $16.3^\circ$ , and  $22.4^\circ$  for cellulose I. The small peak at  $12.3^\circ$  and larger shoulder at  $20^\circ$  show the presence of cellulose II in both samples (Fig. 3) with slightly more in the solvent-treated (red curve) sample.

The control cotton fabric showed peak of cellulose II because of the chemical treatments used during fabric processing to improve properties of cotton such as its dimensional stability, reactivity, luster, etc. (Cook 1984). The amount of cellulose II was esti-

mated by the simulation method (French 2014; Nam et al. 2016; Yue et al. 2015). The  $a$  value was adjusted to  $7.906 \text{ \AA}$  because of the less perfectly ordered cotton cellulose. Amorphous cellulose significantly influenced the diffraction pattern and was therefore included in the simulation. The diffraction pattern of cellulose II with FWHM of  $9^\circ$  was used for amorphous cellulose (Nam et al. 2016) in the simulation. Figures 4 and 5 show the diffraction pattern of the control and solvent-treated cotton fabrics, respectively, fit with the corresponding simulated pattern. Table 5 presents the composition of cellulose in the control and solvent-treated cotton fabric. Solvent-treated cotton fabric showed a slightly greater amount of cellulose II (21.5 %) compared with the control sample (17.2 %), indicating a solvent effect on the cotton fabric during coating. The effect of the urea–thiourea–NaOH–water solvent system on the structure of the cotton fabric was small because the treatment time was just 20 s at room temperature, with the same conditions used for cellulose coating.

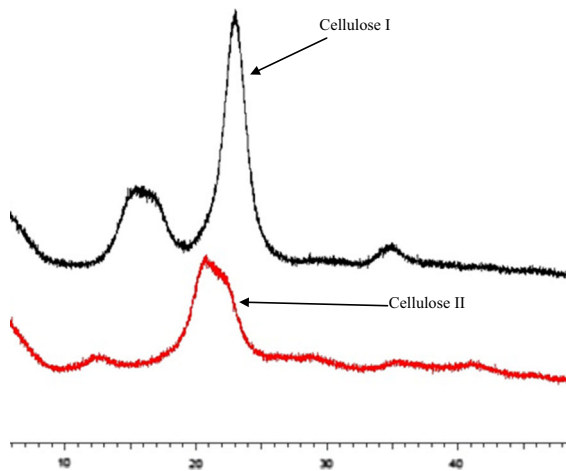
**Table 3** Washing and rubbing fastness properties of dyed samples

Dye content (% owf)	Dye	Cellulose content (%)	Washfastness			Rubbing fastness	
			Evaluation of color change	Evaluation of staining		Evaluation of staining	
				Cotton	Wool	Dry	Wet
3	Reactive Red 240	Control	2–3	4–5	4–5	4–5	3–4
		1	2–3	4–5	4–5	4–5	3–4
		3	2–3	4–5	4–5	4–5	3–4
		5	2–3	4–5	4–5	4–5	3
	Reactive Yellow 95	Control	2–3	4–5	4–5	4–5	3–4
		1	2–3	4–5	4–5	4–5	3–4
		3	2–3	4–5	4–5	4–5	3–4
		5	2–3	4–5	4–5	4–5	3
	Reactive Blue 49	Control	2–3	4–5	4–5	4–5	3–4
		1	2–3	4–5	4–5	4–5	3–4
		3	2–3	4–5	4–5	4–5	3–4
		5	2–3	4–5	4–5	4–5	3
9	Reactive Red 240	Control	2–3	4–5	4–5	4–5	3–4
		1	2–3	4–5	4–5	4–5	3–4
		3	2–3	4–5	4–5	4–5	3–4
		5	2	4–5	4–5	4–5	3
	Reactive Yellow 95	Control	2–3	4–5	4–5	4–5	3–4
		1	2–3	4–5	4–5	4–5	3–4
		3	2–3	4–5	4–5	4–5	3–4
		5	2	4–5	4–5	4–5	3
	Reactive Blue 49	Control	2–3	4–5	4–5	4–5	3–4
		1	2–3	4–5	4–5	4–5	3–4
		3	2–3	4–5	4–5	4–5	3–4
		5	2	4–5	4–5	4–5	3
15	Reactive Red 240	Control	2–3	4–5	4–5	4	3–4
		1	2–3	4–5	4–5	4	3–4
		3	2	4–5	4–5	4	3
		5	2	4–5	4–5	3–4	3
	Reactive Yellow 95	Control	2–3	4–5	4–5	4	3–4
		1	2–3	4–5	4–5	4	3–4
		3	2–3	4–5	4–5	4	3
		5	2	4–5	4–5	3–4	3
	Reactive Blue 49	Control	2–3	4–5	4–5	4	3–4
		1	2–3	4–5	4–5	4	3–4
		3	2–3	4–5	4–5	4	3
		5	2	4–5	4–5	3–4	3

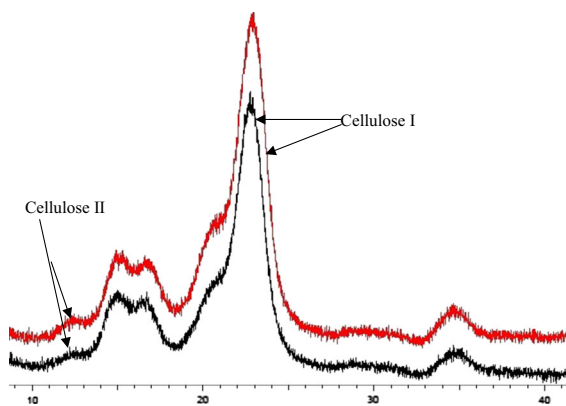
**Table 4** Perspiration fastness of dyed samples

Dye content (% owf)	Dye	Cellulose content (%)	Perspiration fastness					
			Evaluation of color change		Evaluation of staining			
			Acidic	Alkaline	Acidic		Alkaline	
					With wool	With cotton	With wool	With cotton
3	Reactive Red 240	Control	2–3	2–3	4–5	4–5	4–5	3–4
		1	2–3	2–3	4–5	4–5	4–5	3–4
		3	2–3	2–3	4–5	4–5	4–5	3–4
		5	2–3	2–3	4–5	4–5	4–5	3–4
	Reactive Yellow 95	Control	2–3	2–3	4–5	4–5	4–5	3–4
		1	2–3	2–3	4–5	4–5	4–5	3–4
		3	2–3	2–3	4–5	4–5	4–5	3–4
		5	2–3	2–3	4–5	4–5	4–5	3–4
	Reactive Blue 49	Control	2–3	2–3	4–5	4–5	4–5	3–4
		1	2–3	2–3	4–5	4–5	4–5	3–4
		3	2–3	2–3	4–5	4–5	4–5	3–4
		5	2–3	2–3	4–5	4–5	4–5	3–4
9	Reactive Red 240	Control	2–3	2–3	4–5	3–4	4–5	3–4
		1	2–3	2–3	4–5	3–4	4–5	3–4
		3	2–3	2–3	4–5	3–4	4–5	3–4
		5	2–3	2–3	4–5	3–4	4–5	3–4
	Reactive Yellow 95	Control	2–3	2–3	4–5	3–4	4–5	3–4
		1	2–3	2–3	4–5	3–4	4–5	3–4
		3	2–3	2–3	4–5	3–4	4–5	3–4
		5	2–3	2–3	4–5	3–4	4–5	3–4
	Reactive Blue 49	Control	2–3	2–3	4–5	3–4	4–5	3–4
		1	2–3	2–3	4–5	3–4	4–5	3–4
		3	2–3	2–3	4–5	3–4	4–5	3–4
		5	2–3	2–3	4–5	3–4	4–5	3
15	Reactive Red 240	Control	2–3	2–3	4–5	3–4	4–5	3–4
		1	2–3	2–3	4–5	3–4	4–5	3–4
		3	2–3	2–3	4–5	3–4	4–5	3–4
		5	2–3	2–3	4–5	3–4	4–5	3
	Reactive Yellow 95	Control	2–3	2–3	4–5	3–4	4–5	3–4
		1	2–3	2–3	4–5	3–4	4–5	3–4
		3	2–3	2–3	4–5	3–4	4–5	3
		5	2–3	2–3	4–5	3–4	4–5	3
	Reactive Blue 49	Control	2–3	2–3	4–5	3–4	4–5	3–4
		1	2–3	2–3	4–5	3–4	4–5	3–4
		3	2–3	2–3	4–5	3–4	4–5	3
		5	2–3	2–3	4–5	3–4	4	3





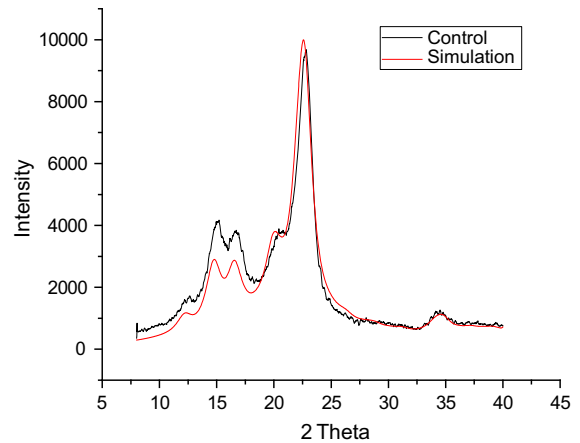
**Fig. 2** X-ray diffraction pattern and analysis of original (*black*) and regenerated (*red*) cellulose pulp. (Color figure online)



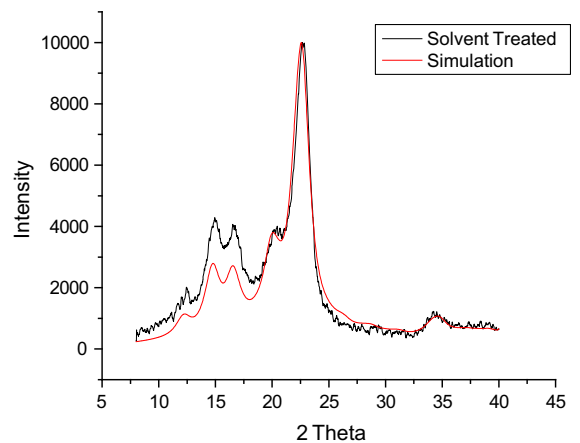
**Fig. 3** X-ray diffraction pattern and analysis of control (*black*) and solvent-treated (*red*) cotton fabric. (Color figure online)

#### Effect of cellulose coating on air and water vapor permeability of cotton fabric

Air permeability and water vapor permeability are important comfort properties of textile material, making it important to evaluate these properties after coating. It is clear from Table 6 that both the air permeability and water vapor permeability decreased after cellulose coating. The air permeability decreased by up to  $18 \text{ l m}^{-2} \text{ s}^{-1}$  and the water vapor permeability by up to 21.4 % after cellulose coating. The permeability of the coated fabric decreased because the applied cellulose forms a film on the surface, covering the spaces between yarns. Moreover, both the air and water vapor permeability decreased with



**Fig. 4** Diffraction pattern of control cotton fabric fit with simulated pattern. (Color figure online)



**Fig. 5** Diffraction pattern of Solvent Treated cotton fabric fit with simulated pattern. (Color figure online)

increasing cellulose concentration and can thus be controlled according to application requirements by altering the cellulose concentration.

#### Effect of cellulose coating on breaking strength of cotton fabric

The breaking force of cotton and coated fabric was measured using a Tira test 2300 instrument. Table 7 presents the breaking strength of the control and cellulose-coated cotton fabric. The tenacity of the cellulose-coated cotton fabric increased with increasing cellulose concentration. The tenacity of 5 % cellulose-coated cotton fabric was increased by

**Table 5** Estimation of cellulose II

Sample description	Cellulose I	Cellulose II	Amorphous content
Control cotton	47.4	17.2	35.4
Solvent-treated cotton fabric	45.2	21.5	33.3

**Table 6** Air and water vapor permeability

Coated cellulose (%)	Air permeability ( $1 \text{ m}^{-2} \text{ s}^{-1}$ )	Standard deviation, $\sigma$ ( $1 \text{ m}^{-2} \text{ s}^{-1}$ )	Water vapor permeability (%)	Standard deviation, $\sigma$ (%)
0	56.3	0.03	89.6	0.13
1	56.2	0.12	81.6	0.21
3	55.5	0.29	76.3	0.41
5	38.4	0.56	68.2	0.78

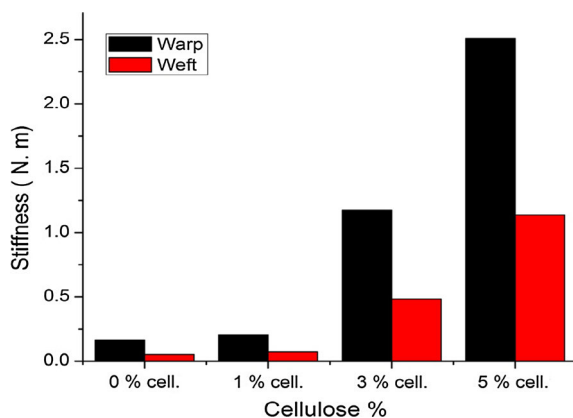
**Table 7** Breaking strength of control and coated cotton fabrics

Sample description	Breaking force (N)	Standard deviation, $\sigma$ (N)
Cotton fabric	504.27	1.32
Solvent-treated cotton fabric	503.51	1.98
1 % cellulose-coated cotton fabric	545.86	2.14
3 % cellulose-coated cotton fabric	576.66	1.56
5 % cellulose-coated cotton fabric	600.40	2.46

96 N. The tenacity increased with increasing cellulose concentration, which may be because the coated cellulose attached to the surface of the cotton fabric, providing it with additional strength. The tenacity was not decreased because there was no significant change in the structure of the cotton fabric after coating, as proved by the X-ray diffraction patterns.

#### Stiffness of cellulose-coated fabric

Different concentrations of cellulose solution were prepared by dissolving cellulose in the urea–thiourea–NaOH–water solvent system. The cellulose concentration was increased up to 5 % by decreasing the water concentration. This solvent can clearly dissolve a maximum of 6 % cellulose, but such 6 % solution has very high viscosity and is difficult to apply to fabric. The prepared cellulose solution was applied on cotton fabric by using roller padding to increase its stiffness permanently. The stiffness of the control as well as coated cotton fabrics was measured using a TH-7 instrument (Fridrichova 2013). Figure 6 shows

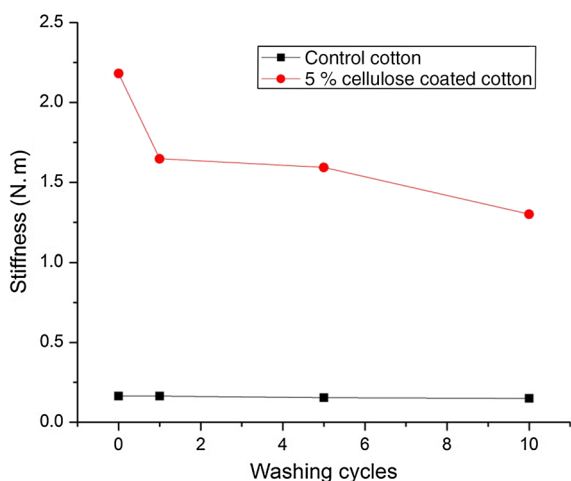
**Fig. 6** Stiffness of uncoated and coated samples

the effect of cellulose coating on the stiffness of the cotton fabric.

The cotton fabric with 5 % cellulose coating showed very high stiffness compared with the other samples. From Fig. 6 it is also clear that the stiffness increased with increasing cellulose concentration. The

SEM micrographs in Fig. 1 reveal that the coated cellulose was attached homogeneously to the fabric surface, which is why the stiffness was high and permanent. The coated cellulose was homogeneously distributed over the fabric surface because, during coating, the solvent molecules try to dissolve the fabric cellulose and link cellulose chains together. It is not easy to prove such linking by spectroscopic methods, because both molecules are the same. However, from the results of SEM imaging and the washing study, it is clear that there is a link between both kinds of cellulose chains, which is why the coated cellulose was not removed by the water during washing.

Figure 7 shows the effect of washing on the stiffness of the cotton fabric. This study was carried out with the 5 % cellulose-coated sample. Samples were washed with 4-gpl soap detergent at 40 °C. Figure 7 shows that the stiffness decreased slightly after the first washing, but thereafter there was no drastic change, and the stiffness remained high even after 10 washing cycles. There was no significant drop in stiffness after 5 washing cycles, because the stiffness decreased by only 0.29 N m between 5 and 10 washing cycles. This indicates that there is no need to reapply cellulose solution like starch. This study confirms that the stiffness obtained after applying cellulose to the surface of cotton fabric is permanent.



**Fig. 7** Effect of washing on stiffness

## Conclusions

Dissolved cellulose can be coated on cotton fabric to improve its stiffness permanently. The crystal structure of the pulp cellulose changed from cellulose I to II after dissolution. The cellulose II content increased slightly after solvent treatment of the cotton fabric, but the breaking strength and stiffness increased with increasing cellulose concentration. The decrease in the  $K/S$  values indicates that cellulose coating decreased the dye uptake by the fabric. The  $L^*$  value increased in the case of cellulose-coated samples, meaning that cellulose coating increased the lightness of the cotton fabric. Reactive Blue 49 dye showed better dyeability towards the coated cotton fabric compared with Reactive Red 240 or Reactive Yellow 95 dye. The fastness properties of both control and coated cotton fabrics against washing, rubbing, and perspiration were similar and good. The washing study confirmed that the stiffness of the cellulose-coated cotton fabric was permanent. The air and water vapor permeability decreased after cellulose coating but can be controlled by changing the cellulose concentration according to application requirements. Therefore, cellulose coating increased the stiffness permanently but decreased the dye uptake by the cotton fabric.

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