

Colour management in circular economy: decolourization of cotton waste

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Abstract

Purpose – While aiming to create methods for fibre recycling, the question of colours in waste textiles is also in focus; whether the colour should be kept or should be removed while recycling textile fibre. More knowledge is needed for colour management in a circular economy approach.

Design/methodology/approach – The research included the use of different dye types in a cotton dyeing process, the process for decolourizing and the results. Two reactive dyes, two direct dyes and one vat dye were used in the study. Four chemical treatment sequences were used to evaluate colour removal from the dyed cotton fabrics, namely, HCE-A, HCE-P-A, HCE-Z-P-A and HCE-Y-A.

Findings – The objective was to evaluate how different chemical refining sequences remove colour from direct, reactive and vat dyed cotton fabrics, and how they influence the specific cellulose properties. Dyeing methods and the used refining sequences influence the degree of colour removal. The highest achieved final brightness of refined cotton materials were between 71 and 91 per cent ISO brightness, depending on the dyeing method used.

Research limitations/implications – Only cotton fibre and three different colour types were tested.



Practical implications – With cotton waste, it appears to be easier to remove the colour than to retain it, especially if the textile contains polyester residues, which are desired to be removed in the textile refining stage.

Originality/value – Colour management in the CE context is an important new track to study in the context of the increasing amount of textile waste used as a raw material.

Keywords Circular economy, Colour management, Decolorization, Fibre recycling

Paper type Research paper

Introduction

The annual consumption of textile fibres has increased dramatically during the past decade; from 50 million tons to more than 70 million tons (Floer, 2011). The global production of man-made cellulosic fibres is predicted to increase from the present (5.7 million tons in 2013) (CIRFS, 2015) to 19.0 million tons by 2030, because of increases both in per capita consumption and in population (Sixta *et al.*, 2015). The human population is growing, and global economic development creates pressure towards western ways of consuming, meaning increasing the use of many products, including textiles and especially fashion items. Accordingly, the need for textile fibres is growing, although the possibility to increase the production of e.g. virgin cotton is limited. The production of cotton is limited by seasonal weather conditions and by the land area available for fibre cultivation. The land area is needed to cultivate food for the increasing population, and therefore, increasing cotton cultivation is not feasible. The cultivation of cotton also has a high environmental impact, which needs to be considered by looking for alternative and more environmentally friendly fibre sources for the textile industry.

These developments are the reason why the textile and fashion industry is looking for ways to move its activities into closed loop systems. Moreover, political pressure will change industrial practices. Since 2016, European Union (EU) legislation has forbidden the landfill disposal of organic materials, including textile wastes. Additionally, the member states of EU will be required to set up a separate collection for discarded textiles by 2025. This has created the need to recycle textile waste. These developments lay the ground for the importance of developing new methods to use cotton waste as a valuable source of raw material and emphasize the importance of the circular economy approach (CE) in the textile industry. In the CE approach and while aiming to close the loop in the industry, all materials and chemicals inside the materials should be recycled and used again in a second production cycle (Niinimäki, 2017; 2018).

One approach towards CE is the chemical recycling of discarded cotton textiles back to textile fibres, by a process in which cotton cellulose is dissolved and regenerated into man-made cellulose fibres. Recent studies have shown that recycled cotton can be processed to dissolving pulp suitable for man-made cellulose fibre production (von der Eltz, 1994; Lindström and Henriksson, 2013; Xianjun and Liangmei, 2013; Katajainen 2016; Asikainen *et al.*, 2018; Leaderlab, 2017; Wedin *et al.* 2018). The commercial production methods for man-made cellulose fibres able to use cotton as a raw material are the viscose, 2.15 million tons/year in 2016 (CCFGroup, 2017) and lyocell (Refibra, 2018) processes. Recently introduced emerging processes to produce man-made cellulose fibres are the cellulose carbamate (Katajainen, 2016) and ionic liquid-based Ioncell-F processes (Sixta *et al.*, 2015; Michud *et al.*, 2016). In both these processes, recycled cotton can be used as raw material to produce regenerated cellulose fibres (Katajainen, 2016; Teng *et al.*, 2018; Asaadi *et al.*, 2016).

When aiming to create methods for fibre recycling, the question of colours in recycled waste textiles is also in focus; should we try to keep the colour or should we try to remove it while recycling the fibres? There are several options for colour management with waste textiles: to remove colour; to collect; and sort waste cotton textiles based on their colour (Esteve-Turrillas and de la Guardia, 2016) and retain colour throughout the refining stages and regenerated fibre production (Smirnova *et al.*, 2016, Smirnova, 2017); or to blend waste textiles ignoring their colour and to produce staple fibres with greyish – brownish shades (Heikkilä *et al.*, 2018; VTT News, 2017a). The latter “mix” could possibly be bleached after the spinning or textile manufacturing process and then treated in the finishing phases by conventional techniques such as dyeing. The other option with these mixed shades is to end up by dyeing the material with a darker colour (IFC, 2018) or even with black colour (Heikkilä *et al.*, 2018; VTT News, 2017b), to result in an even colour shade. This option does not sound attractive from the commercial point of view. Colour is an important factor in the textile and fashion industry, and it is hard to imagine textile collections or new fashion items entering into markets only in black colour. All the previously mentioned methods for colour management do include challenges, and one way to manage the colour in textile waste is first to remove the colour of the waste and then to produce a new colour by dyeing the recycled fibre. This method would probably be the industry’s way of handling colour in the recycled fibre, although all processing would add to the environmental impact of the product.

When waste cotton textiles are used for man-made fibre production, the material should undergo refining stages before dissolution to fulfil the quality requirements and tolerance levels of the regeneration processes and to provide suitable fibre properties. The refining stages increase cellulose reactivity, remove unwanted impurities and adjust cellulose viscosity (Lindström and Henriksson, 2013; Katajainen, 2016; Asikainen *et al.*, 2018; Leaderlab, 2017; Wedin *et al.*, 2018). In addition, it is possible to decolorize the waste cotton textiles during refining if wanted (von der Eltz, 1994; Xianjun and Liangmei, 2013; Asikainen *et al.*, 2018; Bigambo *et al.*, 2018; Wedin *et al.*, 2018). Strategic decisions are needed when constructing a system for CE and when closing the loop with textiles. These decisions also include colour management. For this reason, more knowledge is needed about how to keep the colour in the recycling process or how to remove it while recycling the fibres. It is also important to recognize, which colour types to use if aiming to keep or to remove the colour from cotton waste. This information is intimately associated with the recycling technology in use and with the pretreatment of fibres. The decolorization, together with the refining process, ensures high-quality dyeing and finishing results in the final fabric from recycled material, as it prepares the material for these next steps. The decolorizing and refining restore the waste material to its original clean, white state (Roy Choudhury, 2011; Hauser, 2014). The textile waste may contain several unrecognizable impurities, originating from the cotton cultivation, fibre, yarn and textile production, as well as from product use stages. From the chemical recycling process point of view, the dye is also a certain type of impurity, particularly as the dye-type and entire chemical specification of the textile are unknown and even unpredictable.

The objective of this research was to evaluate how different chemical refining sequences remove the colour of reactive, direct and vat dyed cotton fabrics and how they influence the specific cellulose properties. The selection of dye types for the study covers the dye groups frequently used for the dyeing of cotton (Božič and Kokol, 2008). The reactive dyes are the most used dyes for cotton (Khtari *et al.*, 2015), because of their good fastness properties and bright colours. The use of direct dyes in the dyeing of cotton has been in decline due to their lower fastness properties compared to the reactive dyes, but the advantage of direct dyes is

their lower cost and simple dyeing process (Räisänen *et al.*, 2017). Vat dyes are traditionally used for the dyeing of cotton yarns to produce denim jeans (Biermann *et al.*, 2006). Due to the dyeing method, vat dyes leave the core of the yarns undyed, a phenomenon that is widely exploited in the finishing of jeans (e.g. stone washing) to give them a worn look (Asplan, 1992). Additionally, the selected dye types attach differently to cellulose includes: reactive dyes form covalent bonds between the dye molecule and the hydroxyl groups of cellulose (Lewis, 2007) and direct dyes attach through van der Waals forces and hydrogen bonding (Lim and Hudson, 2004). Vat dyes are trapped mechanically inside the cotton structure during the dyeing process, in which the water-soluble leuco form of the vat dye penetrates the fibre structure and becomes trapped inside when it is oxidized back to an insoluble form (Asplan, 1992).

According to Asplan (1997), complete chemical stripping, i.e. removing dyes from coloured fabrics, may be carried out either with reduction or oxidation or with a combination of these methods depending on the dyes. In the past, direct dyes were removed by boiling the fabric in alkaline sodium hydrosulfite or bleaching with sodium hypochlorite. Vat dyes were treated in high-temperature reduction bath containing caustic soda and sodium dithionite or sodium hydrosulfite and quaternary ammonium salt as stripping assistant or a substance that was responsible to combine with the dye molecule, so it does not re-attach with textile material once it has been stripped out (Chavan, 1969; Sapers, 1971). Nowadays, oxidative chemicals such as ozone, hydrogen peroxide and peracids have replaced chlorine and permanganate-based treatments (Winkler *et al.*, 1997; Walger *et al.*, 2015; Eren *et al.*, 2016). In total, 89-94 per cent stripping efficiency of bi-hetero type reactive dyes were achieved when reductive treatment with the combination of sodium hydroxide and sodium hydrosulphite was used at elevated temperatures 80°C and 100°C (Uddin *et al.*, 2015). In addition to effective dye removal, a sequential alkali-acid or acid – alkali stripping of reactive dyes from cotton fabrics has been reported to remove cross-linked textile finish (Haule *et al.*, 2014; Wedin *et al.*, 2018). If hydrogen peroxide treatment was added into the acid-alkali sequence, over 90 lightness values was got with Tencel fabric dyed with azo-based reactive colours, Black 5 and Red 228 (Bigambo *et al.*, 2018).

In this paper, the effects of different chemical refining sequences on dyed cotton are reported. The studies include the management of impurities and adjustment of cellulose viscosity. The effects of refining sequences on the decolorization are studied by determining ISO brightness and colourimetric data of the cotton samples after each refining step. The results are discussed in the context of CE and considering what these findings could mean from the colour management viewpoint when recycling fibres and closing the loop in the textile industry.

2. Experimental

2.1 Producing samples of dyed cotton

To be able to investigate how different chemical refining sequences remove the colour of direct, reactive and vat dyed cotton fabrics, such fabrics were prepared for this study. Virgin cotton fabric (woven fabric with plain weave, 100 per cent cotton, 148 gm⁻², prepared for printing) was dyed with the above-mentioned dye types to provide a selection of five samples, listed in Table I. Actual cotton waste was not used, as chemical product specifications (including the dyes used) of waste textiles are not available, and waste cotton from different pre- or post-consumer sources would have different properties depending on the manufacturing process, treatments applied and habits of product use. Having the same base fabric in all trials enabled comparison of the results of different refining sequences with each other, and with the dyes used.

Table I.
Dyes and parameters
of the dyeing process
used in this study

Dye type	Reactive		Vat	Direct
Dye name	Remazol Brilliant Orange 3R spec	Remazol Carbon RGB	Indanthren Blau BC 3 % (Ultramarine Blue)	Direct Blue 1 (Pontamine Blue)
EC number	243-653-9	241-164-5	204-980-2	220-028-9
Fabric weight (kg)	1, 25	1, 25	3	1
Dyeing method	Washing machine (Talpet HC75)		Batch dyeing process in a kettle	Washing machine (Wascator FOM71)
Dye concentration (% owf)	4		1, 5	1, 5
Liquor ratio	20:1		30:1	20:1
Dyeing temp. (°C)	60		30	90
Dyeing time (min)	60		20	45
Additives used	Sodium carbonate	<i>In dyeing</i> 10 g/L	NaOH 25% <i>In vat preparation</i> 3g/(g of dye)	<i>In pre-washing</i> Anionic surfactant, Levapon, Bayer Germany <i>In dyeing</i> Sodium carbonate Sodium sulphate
	Sodium sulphate	80 g/L	Sodium dithionite	0.5 g/L 5 g/L
			<i>In the dyeing liquid</i> NaOH 25% Sodium sulphate Sodium dithionite	
			Sodium carbonate <i>In boiling</i> 1 g/L	<i>In final washing</i> Nonionic surfactant, Lavoral, Bozzetto GmbH 1 g/L

Source: Of EC numbers: European chemicals agency (ECHA)

Two different types of reactive and direct dyes were tested in this procedure to determine whether the decolouring result is also different between these broad categories of dyes. Two different reactive dyes were chosen for the study: Remazol Brilliant Orange 3R and Carbon RGB, both commonly used azo dyes. According to the provider DyStar, Brilliant Orange 3R is dischargeable, whereas Carbon GB is not (Pellonpää-Forss, 2016). Comparing a dischargeable and a non-dischargeable dye was of interest because this factor can have an impact on the result of colour removal. The selected direct dyes were both anionic diazo dyes, but with different washing fastness: according to the Colour Index the washing fastness of Direct Red 81 is good (Rating 4) and that of Direct Blue 1 is poor (Rating 2). Blue vat dyes were used to imitate the most common category of vat dyed products, jeans.

Table I shows the main parameters of the dyeing processes applied in this study. The selected dyeing methods are similar to the processes used in industry, but done in a smaller scale in a textile dyeing workshop. Reactive and direct dyes were dyed with programmable washing machines, with programs including prewashing, dyeing and rinsing steps and spin-drying after each step. The dyes and the additives were weighed into a beaker, dissolved in hot water and poured by hand into the washing machine in the beginning of the dyeing process. In the case of the direct dyeing process, the fabric was also prewashed at 40°C for 20 min before dyeing and for 15 min after dyeing. Vat dyeing was performed as a batch dyeing process in a large kettle on a stove instead of a washing machine, to reduce the exposure of dyeing liquid to oxygen. The use of a higher liquor ratio compared to washing machine processes enabled moving the fabric in the kettle. The vat was prepared by using warm (50°C) water (50 mL/1 g of dye) and adding NaOH and the reducing agent sodium dithionite. The vat was kept in a 50°C water bath for 10 min while the dyeing liquid was prepared by mixing the remaining amount of water (30°C) and additives and allowing the liquid to reduce for 5 min. The vat was then mixed with the dyeing liquid, and wet fabric was added to the kettle and dye. Finally, the fabric was rinsed with running cold water, boiled in a kettle, and rinsed and spin-dried in a washing machine.

2.2 Colour removal treatments

Guidelines in the selection of chemical treatments including colour removal were that the treatments can be easily implemented in the process steps used in chemical pulping and one treatment have several functions at the same time if possible.

The dyed cotton fabrics were shredded with a hammer mill using 10 mm screen and refined with a Bauer disk refiner (wet milling) to make the cotton structure more open before chemical treatments. Four chemical treatment sequences were used in this study to evaluate the colour removal of the dyed cotton fabrics includes: HCE-A, HCE-P-A, HCE-Z-P-A and HCE-Y-A. The use of different chemicals was not only for decolouring but also to remove unwanted impurities, increase cellulose reactivity and adjust cellulose viscosity for enhanced dissolution in regenerated cellulose processes. Hot alkaline extraction (HCE) was used to remove silicate and polyester (PSE) residuals, which are present in recycled textiles. The viscosity of cotton materials was adjusted either with sulphuric acid (A), ozone (Z) or hydrogen peroxide (P). The target viscosity level was 400-500 mL/g, which is suitable for Ioncell-F (Sixta *et al.*, 2015) and cellulose carbamate dissolution and regeneration processes (Valta and Sivonen 2010 and 2011). The main metal management was achieved with acid washing (A) using sulphuric acid. Ozone, dithionite (Y) and peroxide stages together with a HCE stage were used also for decolouring.

The processing conditions used are presented in Table II. HCE treatments were performed in a 40 L reactor equipped with a combined anchor blade mixer. The reactor was heated by pressurized water in a heating mantle. Extraction conditions (alkali concentration 80 g/L and

Table II.
Process conditions of
all colour removal
treatments

	NaOH, %	O ₃ , %	H ₂ O ₂ , %	Y, %	H ₂ SO ₄ , %	Consistency, %	Temperature, °C	Time, min
HCE @ all sequence	72					10	110	120
A @ HCE-A					1.2	10	90	120-180
P @ HCE-P-A	1.7		1.3			10	90	180
A @ HCE-P-A					2.55-3.14	2.5	60	60
Z @ HCE-Z-P-A		0.5			0.6-0.65	12	25	3:35
P @ HCE-Z-P-A	1.1		0.3-0.5			10	90	120
A @ HCE-Z-P-A					2.45	2.5	60	60
Y @ HCE-Y-A				0.5-1	0.01	4	80	60
A @ HCE-Y-A					1-1.2	10	90	120

temperature) were selected to be suitable for the dissolution of PES in recycled textiles (Negulescu *et al.*, 1998; Asikainen *et al.*, 2018), although it was known that there was no PES in the dyed cotton materials. Ozone treatment (Z) was performed in a 17 L plastic flow-through reactor in medium consistency (12 per cent). Initial pH 2 was adjusted with sulphuric acid before rinsing the cotton with oxygen. After that, ozone generation in the carrier oxygen gas was first stabilized and then an ozone flow was led to the potassium iodide solution for the determination of ozone content in the gas, and then into the reactor. The cotton was mixed with a blade mixer throughout the ozone charging. After the reaction time, the cotton was rinsed with oxygen again. The ozone content of the residual gas from the reactor was analyzed and the ozone generation was checked after the reaction time. Ozone formation and consumption were determined from the potassium iodide solution by titration with sodium thiosulphate. Acid (A), dithionite (Y) and hydrogen peroxide (P) treatments were performed in buckets heated in a water bath. Cotton material pre-heated in a microwave oven and most of the preheated dilution water were mixed and then pH was adjusted if necessary, before the chemical addition. In dithionite bleaching, pH was adjusted to pH 7 and in the acid stage to pH 2. During the reaction time, the buckets were mixed by hand.

After every stage there was always a standard laboratory washing: cotton was diluted to 5 per cent consistency with deionized water, the temperature of which was the same as in the preceding treatment stage, and dewatered. The materials were washed twice with cold deionized water with an amount equivalent to ten times the dry pulp absolute amount. After every stage, samples were taken for characterization.

2.3 Characterization

The dyed cotton fabrics and treated cotton materials were characterized by measuring their limiting viscosity, brightness, reflectance, metals contents and molar mass distribution. The characterization methods used were:

- ash content 525°C (ISO 1762:2001) from dyed starting materials and after the last treatment;
- metal content wet combustion (the samples are dissolved in nitric acid in a microwave oven before the analysis) + ICP-AES from dyed starting materials and after the last treatment;
- brightness ISO 2470-1:2009 (reflectance of blue light at 457 nm) from a split sheet after every treatment; and
- limiting viscosity (ISO 5351:2010) after almost every treatment.

For the molar mass measurements, the solid samples were dissolved in DMAc/8 per cent LiCl according to the solvent exchange method described by Berthold *et al.* 2004. The method includes activation of the sample with water, solvent exchange with methanol and DMAc. Two parallel samples of each cellulose sample were dissolved. After complete dissolution, the samples were diluted with DMAc providing final LiCl concentration of 0.8 per cent as in the eluent. The elution curves were detected using a Waters 2,414 Refractive index detector. The molar mass distributions (MMD) were calculated against 8 × pullulan (6,100-708,000 g/mol) standards, using Waters Empower 3 software.

The reflectance of the fabrics after dyeing and after each pre-treatment step was measured in the visible region of the spectrum from 400 to 700 nm with illuminant D65 and a standard observer at 10° with a Minolta CR-200 spectrophotometer (Minolta, Japan) The colourimetric data (L , a and b values) are given as averages of five measurements. The colour differences (ΔE) were calculated according to the equation

$$\Delta E = [(\Delta L)^2 + (\Delta a^2) + (\Delta b^2)]^{1/2}$$

3. Results and discussion

3.1 Dyed materials

The white cotton fabric was dyed with two reactive dyes, orange and black, two direct dyes, red and blue, as well as with blue vat dye [Figure 1](#). When comparing the dyed fabrics after dyeing and before any bleaching sequences, the colourimetric data of the fabrics were measured ([Table III](#)). The lightness (*L*-value) of the fabric dyed with reactive black was the lowest and the other colour components were not changed significantly, as expected. The fabric dyed with reactive orange was also dark and had a saturated hue as the values for the green-red (*a*-value) and blue-yellow (*b*-value) were high. The fabric dyed with direct

Figure 1. Original cotton and dyed cotton fabrics: top line: original cotton, direct blue, direct red; bottom line: reactive black, reactive orange and vat dyed blue



Table III. Colorimetric data of the undyed white cotton fabric and the dyed fabrics

Sample	L	a	b
Undyed cotton fabric	94.8	0.2	2.1
Reactive black (Remazol Carbon RGB), 4% owf	20.4	2.5	-2.0
Reactive orange (Remazol Brilliant Orange 3R), 4% owf	52.0	57.6	39.6
Direct Blue (Pontamine Blue), 1.5% owf	45.1	-3.5	-25.9
Direct Red (Direct Red 81), 1.5% owf	47.3	57.9	9.9
Vat blue (Indanthren Blau BC), 1.5% owf	49.0	2.8	-34.8

Table IV. Metal and ash contents of original and dyed cotton fabrics

Raw material	Al, mg/kg	Ca, mg/kg	Co, mg/kg	Cu, mg/kg	Fe, mg/kg	Mg, mg/kg	Si, mg/kg	ash (550°C), %
Original cotton	9	150	5.4	1	41	58	55	0.08
Direct blue	22	230	6.0	0.6	34	79	89	0.09
Direct red	22	230	3.6	4	26	67	74	0.09
Reactive orange	8	295	7.5	5.4	36	61	20	0.10
Reactive black	12	743	3.8	10.6	26	126	46	0.25
Vat dyed blue	17	224	<0.5	5.1	18	54	71	0.02
Commercial dissolving pulp (Sixta 2006)		15-100	0.1	2.5-3.5	2-8	220	50-65	~0.1

red had saturated red hue. The fabrics dyed with vat blue and direct blue had had blue hues, but their saturation was not as high as in the case of red and orange dyed fabrics.

The initial limiting viscosity of the cotton material, determined after the wet milling, was over 1,700 mL/g. This is a typical level for bleached cotton fabrics and was at the same level as [Wedin et al. \(2018\)](#) has reported. The dyeing method did not influence the viscosity value, but its determination was challenging from the reactive black dyed material due to dissolution difficulties. Metal and ash contents of the original cotton and the dyed cotton materials are presented in [Table III](#). Dyeing appeared to increase the calcium, aluminium, copper and silicon contents of the fabrics. The used dyes did not contain metals, but the dyeing treatments were carried out with tap water and with technical grade chemicals, which appeared to be the reason for the increase in inorganic materials. These metal contents and iron were higher than in commercial dissolving pulp, which is the raw material used for commercial regenerated cellulose fibres ([Sixta, 2006](#)). The presence of certain inorganic compounds such as silicates, Ca salts, and catalytically active transition metal ions clearly impairs the filterability and spinnability of a cellulose spinning dope. In addition, cellulose contamination with inorganic compounds leads to a gradual clogging of the spinnerets, which alters the uniformity of the fibre titre ([Lenz, 1981](#)). Fe(II) ions also promote light-induced yellowing, and together with Cu(II) are involved in detrimental reactions in the presence of hydrogen peroxide ([Sixta, 2006](#)).

3.2 Adjustment of limiting viscosity and removal of impurities

The limiting viscosity levels of dyed cotton materials were adjusted to between 410 and 510 mL/g by the colour removal sequences. This is a typical viscosity level of commercial dissolving pulps, which are used in the production of man-made cellulose fibres. An example of the formation of cellulose MMD over the colour removal processing steps is presented in [Figure 2](#). It can be noticed that dyeing did not influence the molar mass, but HCE decreased the molar mass before the final adjustment with bleaching chemicals and acid. [Figure 3](#) presents an example of variation of molar mass distribution between different dyeing methods with the same colour removal treatment sequence, HCE-Z-P-A. No tendency was observed of the dyeing method or specific treatment sequence to influence the molar mass distribution in different ways.

During the refining sequences, the removal of impurities such as metals and ash was effective. A HCE stage decreased the silica content to below 30 mg/g, and the following stages slightly more in most cases. The residual calcium content was reduced to between 10 and 76 mg/g with acid treatment. Milder conditions in the acid stage resulted in higher contents. Only the iron content, 7-38 mg/g, was higher than recommended for dissolving pulp in most cases. In this study, chelation chemicals were not used to prevent side reactions in hydrogen peroxide bleaching caused by Fe(II) and Cu(II) because these reactions decrease cellulose viscosity, which was another reason to use peroxide treatment.

Sample	L	a	b
Undyed cotton fabric	94.8	0.2	2.1
Reactive black (Remazol Carbon RGB), 4% owf	86.3	4.1	10.0
Reactive orange (Remazol Brilliant Orange 3R), 4% owf	90.7	2.1	3.6
Direct Blue (Pontamine Blue), 1.5% owf	92.0	0.9	1.9
Direct Red (Direct Red 81), 1.5% owf	91.5	1.6	1.6
Vat blue (Indanthren Blau BC), 1.5% owf	72.1	-1.2	-20.0

Table V.
Colorimetric data
after HCE sequence
and undyed cotton
for the reference

Figure 2.
Formation of cellulose MMD over the colour removal processing steps of direct blue dyed cotton

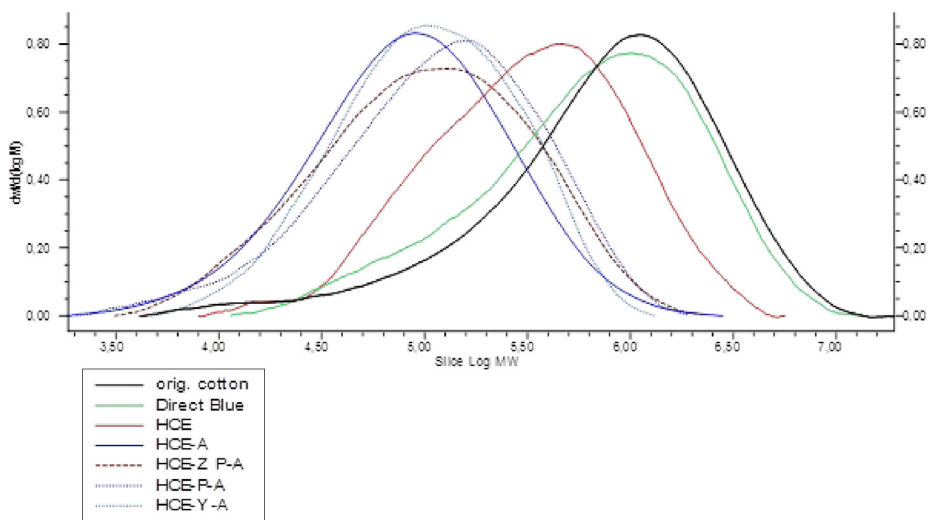
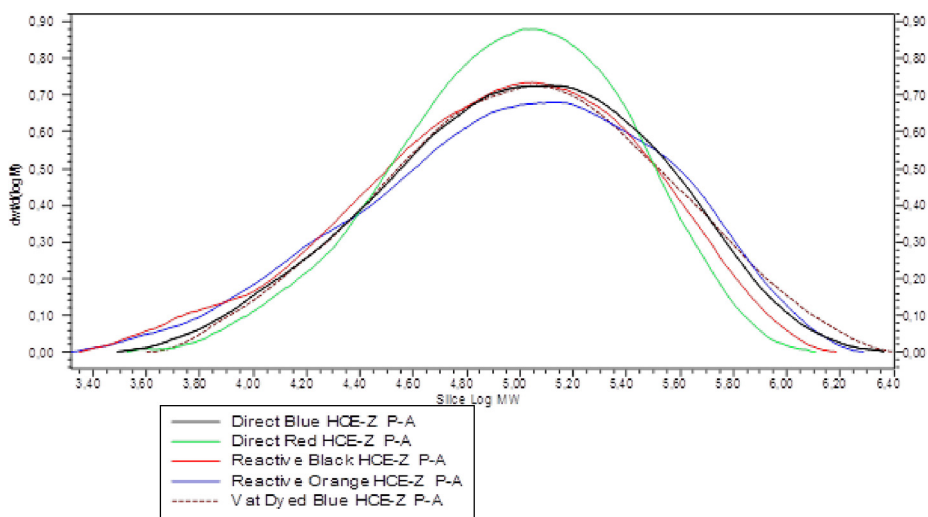


Figure 3.
Molar mass distribution of colour removed cotton materials after HCE-Z-P-A treatment



3.3 Colour after colour removal treatments

Before the chemical colour removal sequences, the dyed fabrics were shredded and refined with the wet milling treatment. Wet milling decreased the ISO brightness of the undyed cotton fabric (from 93.0 to 87.3) and produced colour difference (ΔE) of 2.3, indicating that the fabric was not bleached throughout, and that after the wet milling unbleached fibres, from inside the yarns, were present on the surface of the milled sample. Similarly, regardless of the dye type, the wet milling had an effect on the colour of the dyed fabrics; they were not dyed throughout and undyed fibres from inside the yarns were present on the surface of the

milled sample. The colour difference of the dyed and wet milled fabrics was calculated and the reactive orange dyed fabric had the lowest colour difference (ΔE 8.4). The reactive black and direct dyed fabrics had the colour differences between 10.6 and 13.5. Especially, in the case of vat dyed fabric, the wet milling clearly increased the colour difference (Figure 4) and ΔE -value was 24.6. It is well-known that the vat dyes attach to the surface of dyed material, leaving the core undyed (Hmida and Ladhari, 2015; Moustafa *et al.*, 2011).

The effect of chemical refining sequences after wet milling on colourimetric colour components, a- and b-values, of each sample, were determined, where the a-value stands for the green–red and the b-value for the blue–yellow colour. After wet milling, HCE was the first chemical treatment in each of the chemical colour removal sequences. HCE changed the positions of the a and b values closer to the neutral, for example, a and b values of reactive orange dyed fabric were 57.6 and 38.9 after dyeing, and after HCE treatment they were 2.1 and 3.6, respectively. The trend was the same for the fabrics dyed with direct red and direct blue. The fabric dyed with reactive black turned slightly yellow during the HCE, and the b component moved from -2 to 10 due to the HCE treatment. Vat dyed fabric was the exception: its blue colour component was rather persistent: the b-value of the dyed fabric was -34.8 and after HCE treatment -20.0 (Table V).

After the HCE, all the fabrics were clearly bleached and the effect of the following chemical sequences on the brightness was determined by measuring the ISO brightness. ISO brightness is a standard method when comparing white and near-white cellulose-based materials and thus, it was applicable for the cotton materials after the HCE treatment (Carvalho *et al.*, 2008). HCE produced fabrics with quite high ISO brightness, but it was not alone efficient enough to decolourize the fabrics. The ISO brightness values after this treatment were with reactive black 57.3 ISO per cent, reactive orange 73.2 ISO per cent, direct red 79.9 ISO per cent, direct blue 80.8 ISO per cent and vat dyed blue 65.7 ISO per cent. Further treatments were necessary to reach the acceptable ISO brightness values. Usually, acceptable ISO brightness of the bleached cellulose materials is above 90 per cent when targeting white cellulose materials and it was the target here (Carvalho *et al.*, 2008). The used sequences were: HCE-A, HCE-P-A, HCE-Z-P-A and HCE-Y-A. As can be seen from the second column of the HCE-Z-P-A sequences presented in Figure 5, the influence of ozone treatment (Z) on brightness increase was 1-17 ISO per cent, the lowest effect being with vat



Figure 4.
Vat dyed blue cotton
after wet milling

dyed cotton and highest with reactive black. A hydrogen peroxide stage (P) after ozone treatment (third column in the same sequence) improved brightness maximum by 7 ISO per cent and after the HCE (second column in HCE-P-A sequences) stage by 6-14 ISO per cent. The influence was highest with reactive black. Reductive dithionite (Y) bleaching (second column in HCE-Y-A sequences) increased brightness by 3-16 ISO per cent. The effect was strongest with reactive black and lowest with vat dyed cotton. The effect of an acid stage (A) (the last column in every sequence) on brightness increase was the lowest, 0-7 ISO per cent depending on the dyeing method and processing conditions.

For all the dyed fabrics the highest brightness was achieved with the same treatment sequence, HCE-Z-P-A. The highest brightness was achieved with direct dyes: direct red 91.2 ISO per cent and direct blue 90.5 ISO per cent. The brightness of reactive red was 86.5 ISO per cent, 82.7 ISO per cent with reactive black and 71.5 ISO per cent with vat dyed blue. The lowest brightness of all dyed fabrics was obtained when HCE-A treatment was used. With HCE-A treatment the brightness values were: direct blue 84.3 ISO per cent, direct red 85.5 ISO per cent, reactive red 75.7 ISO per cent, reactive black 64.2 ISO per cent and vat dyed blue 67.8 ISO per cent. As can be seen from Figure 5, the highest brightness i.e. the most effective colour removal was obtained with direct dyed materials and the lowest with vat dyed blue materials almost with all sequences except for the reactive black treated by HCE-A sequence. The development of dye removal during the most effective (HCE-Z-P-A) and ineffective (HCE-A) pretreatments with direct blue and reactive black is shown in Figure 6.

The additional chemical treatments after the HCE treatment did not significantly change the colour components a and b when the fabrics were dyed with reactive orange, direct red and direct blue. The fabric dyed with reactive black remained slightly yellow after the additional treatments; the best response was obtained with HCE-Z-P-A sequence, which produced a value of 4.1 for the component a. The vat dyed fabric retained the most colour compared to the other dyeing methods and dyes. The lowest value for the component b was -13.5, and it was obtained after the HCE followed by ozone treatment (HCE-Z).

When analyzing how close to the original was the obtained colour after different colour removal sequences, the colourimetric data were collected, the colour difference was calculated between the original colour (meaning undyed white fabric) and the colour of near-white fabrics (Table V) after chemical colour removal sequence (Figure 7). The fabrics dyed with the direct dyes, red and blue, reached the lowest colour difference, i.e. they were closest to the original colour. The fabric dyed with direct blue was quite light after the dyeing and it

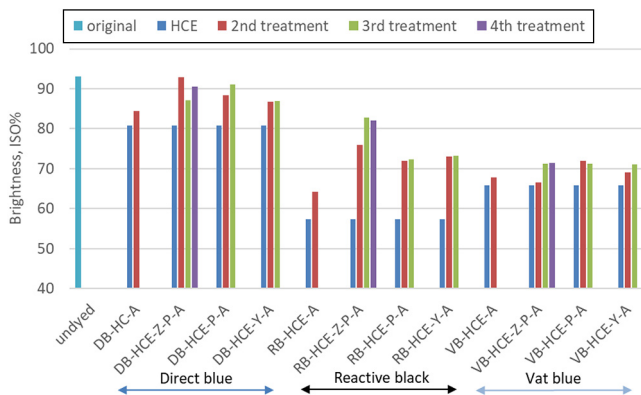
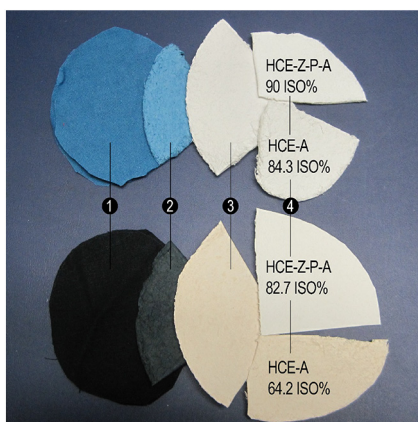
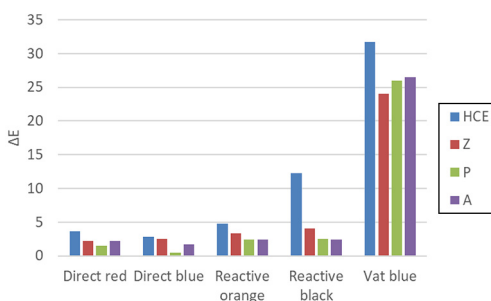


Figure 5.
Brightness development of direct blue (DB), reactive black (RB) and vat dyed blue (VB) cotton materials through different treatment sequences



Notes: Order: dyed fabric (1), after wet milling (2), after HCE (3), the highest final brightness with sequence HCE-Z-P-A (4, upper) and the lowest final brightness with sequence HCE-A (4, lower)

Figure 6. The removal of dye during pretreatment sequences of direct blue (upper) and reactive black (lower)



Note: Colour removal treatments: wet milling, hot alkaline extraction (HCE), ozone (Z), hydrogen peroxide (P), and sulphuric acid (A)

Figure 7. Effect of dye type on final colour after colour removal treatments (sequence HCE-Z-P-A) measured as the colour difference between the undyed cotton material and near-white samples after chemical treatments

reached a colour difference below one (not perceptible by the human eye) with HCE-Z-P, HCE-P and HCE-Y treatments. The fabric dyed with direct red was clearly darker after dyeing, but still reached a colour difference below one with HCE-Y treatment. The lowest colour differences of fabrics dyed with reactive dyes, black and orange, were below three, which means that the colour difference was detectable only by close visual observation. To remove the reactive dyes, HCE-Z-P-A sequence was necessary, especially for the black dye. For the reactive red HCE-Z-P, HCE-P and HCE-Y also produced a colour difference below three. Again, the vat dyed fabric did not have as good a response to the colour removal sequences as the other dye types. The lowest colour difference (24.0) was obtained after the HCE-Z sequence.

The dyes selected for the study attached to cotton differently: with covalent bonds (reactive dyes), with van der Waals forces (direct dyes) and with mechanical trapping (vat dye). Due to the different attachment mechanisms, the dyed cotton fabrics had different responses to the refining sequences. The HCE effectively removed reactive and direct dyes. It is probable that the reactive dyes were degraded so that the sodium hydroxide in high temperature was able to break the dye molecule, causing the loss of colour (Dockery *et al.*, 2009; Uddin *et al.*, 2015; Bigambo *et al.*, 2018). van der Waals forces between the direct dye molecule and cellulose are comparatively weak (Dockery *et al.*, 2009), and in an alkaline environment, the interaction was disturbed enough to strip the dye from cotton. Vat dyed cotton fabric was quite resistant towards the HCE sequence. Vat dyes have excellent fastness in alkaline conditions (Chattopadhyay, 2011) because they are not soluble and do not degrade. Vat dyes have been stripped by reducing them (Dockery *et al.*, 2009), but it is possible that the dye molecule is trapped inside the cotton again if the dye molecule is not degraded and re-attachment is not inhibited (Chatha *et al.*, 2012). The re-attachment of vat dyes probably occurred here because dithionite, refining sequence Y, is known to reduce vat dyes effectively (Dockery *et al.*, 2009). On the other hand, ozone sequence (Z) decreased the colour of reactive black and vat dyed fabrics more effectively. This was probably because of the ability of ozone, a powerful oxidant, to degrade dye molecules, the azo chromophore group, $-N=N-$, connecting aromatic rings, and thus, removing the chromophore (Turhan and Ozturkcan, 2013).

Post-consumer cotton waste includes some amount of PES material, for example, origin from sewing threads. This PES content should be removed in the pretreatment process to better manage of downstream processes of recycled fibre. If this PES removing happens with HCE, the colour of dyed material is also lost, regardless of the dyeing method used. This means that, in practice, colour retention of post-consumer cotton is difficult to realize.

Purifying the waste raw material by refining and decolourization also ensures the material quality in subsequent process steps. Transparency in the supply chain and life cycle of a product is also an important element for CE. The waste textile might originally have been produced 20 years ago or even earlier, which might indicate an unknown history of the material. However, it can be converted to a known history in the next cycle. If the chemically unknown material waste comes to recycling, it could be first decoloured and refined to reset its history. This prerequisite process would make the unknown material appropriate for CE. The creation of a transparent life cycle by using available technologies to tag products enables effective and high-quality recycling of material in subsequent cycles.

4. Conclusions

The colour removal of the dyed cotton materials succeeded to a certain extent with all the dyed materials. Most of the colour was removed in HCE, except in the case of vat dyed material in wet milling. The most effective colour removal sequence was HCE-Z-P-A with all dyeing methods. The removal was easier if dyeing was performed with direct colours, and almost as high final brightness was achieved as that of the original cotton, over 90 ISO per cent. The lowest brightness was obtained with reactive black and vat dyed blue.

In practice, most cotton waste includes some PES remains (e.g. from the sewing thread), and HCE is used for separating PES from cotton. This process destroys all colours to some extent, especially, in the case of direct and reactive dyed materials, and therefore, it might be easier to aim to remove the colour rather than trying to retain it when aiming for a chemical fibre regeneration process. The other impurities could also be removed, and fibre "history" could in this way be reset. The alkaline pretreatment process in decolourization removes

additive chemicals such as easy-care cross-linking agent from the fiber (Haule, L.V., *et al.*, 2014). These additives are impurities in the regeneration process, which might disturb the dissolution of cellulose and the influence on next phases, fiber and yarn generation performance are still unknown (Wedin *et al.*, 2018). Thus, the purifying of material guarantees the success in the next process phases. Additionally, the decolorization with alkaline improves the absorption of water and dyestuffs, as it releases one carbon molecule of the chain (Uddin *et al.*, 2015) and for example, the reactive dye stuff can again form a covalent bond with the fiber. The strong bond is needed for producing textile with high colour fastness in use. However, the application where high water absorption is required the alkaline treatment is beneficial.

In addition to direct, reactive and vat dyeing, pigment printing, by either traditional screen printing or digital printing, is a very common way to colour cellulose textiles, and therefore, the decolorization and refining ability of such dyes should be further studied. The colour management in CE point of view requires transparency, identification system, for the lifecycle of textile product. The dye class and decolorization method has a strong impact on results. Those two aspects are essential information and to be adjusted according to end-use application requirements to implement CE for textiles.

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