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Evaluation of dyeing and finishing components in recycling and regeneration of coloured textiles

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Titel: Evaluation of dyeing and finishing components in recycling and regeneration of coloured textiles prototypes

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A Mistra Future Fashion Report

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preface

Thank you to DyStar, Lenzing AG, I:CO, H&M, Boob design, Wigéz, New Wave Group, Filippa K and Kappahl for providing data and valuable input.

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summary

The ability to create secondary raw materials with low impurity content is important from a fibre regeneration processing perspective. Cotton textiles contain colourants and textile finish, which can be considered as impurities in a chemical recycling process. Removal of such impurities in a decolourisation stage would enhance the fibre dissolving capacity and stabilise and simplify the fibre regeneration process.

Moreover, the removal of impurities from secondary raw materials is also important from an environmental and health perspective. Chemical recycling has the potential to deal with the risk of retaining or spreading hazardous substances further in re-circulated textile loops. Of specific concern are colourants or additives with carcinogenic or endocrine disruptive attributes as well as bioaccumulative substances such as PFAS (per- and polyfluoroalkyl substances), which are commonly used in textiles for dirt and water repellency.

This report describes an experimental evaluation of physical properties of decolourised cotton regenerated into viscose fibres and an analysis of potentially hazardous textile finish components throughout a decolourisation approach. The three main research questions in this report are:

1. Can we obtain viscose fibres from a decolourised black cotton fabric with similar physical properties as commercial viscose fibres by using the re:newcell decolourisation approach?
2. Can the impact from a DMeDHEU(1) anti-wrinkle and a fatty acid softener finish on the physical viscose fibre properties be eliminated using this decolourisation approach?
3. Could chemical recycling be a potential solution for recirculation of textiles which have a risk of containing hazardous substances of for example PFAS dirt and water repellent type?

The project has been performed within the Mistra Future Fashion research program by joint work from re:newcell, MoRe research, Swerea IVF, Swedish School of Textile and RISE, and with support from the companies DyStar®, Lenzing AG, H&M, I:CO, Boob design, Wiges, New Wave Group, Filippa K and Kappahl. The part of the study that concerned the dirt and water repellent was performed in cooperation with the research project SUPFES funded by the Swedish Research Council for Environment, Agricultural Sciences and Spatial Planning (FORMAS) under grant agreement No. 2012-2148.

The study showed that viscose fibres can be produced from decolourised cotton using the re:newcell decolourisation approach. However, the desired physical fibre properties were not obtained. More optimisation in the decolourisation stage is needed to adapt the cotton substrate for the viscose process.

A first indication of the softener to positively affect tenacity of the viscose fibre was observed. The anti-wrinkle treated fabric showed a tendency in higher reduced clogging number, but no influence on the physical fibre properties was seen. While caution should be taken in drawing conclusions from the few performed trials, it seems likely that the impact from the tested types of anti-wrinkle agent and softener on the viscose process could not be eliminated by the use of the decolourisation approach. This also raises the question of how persistent the softener is in a decolourisation process. The results from the analysis on dirt and water repellent substances (DWR/PFAS) throughout the decolourisation process showed that one third remained in the pulp. This suggests that the DWR binds to the fibres in a way which the decolourisation process is not able to reverse.

Taken together, these results are not sufficient to prove the hypothesis of whether chemical recycling could be a potential solution for recirculation of textiles with risk for containing hazardous substances of this sort.

The trials lead to the following conclusions: The used decolourisation approach has potential to remove the type of reactive dye, wrinkle-free agent and softener tested in this study. Adaption of the decolourisation process for the cotton substrate is needed in further trials. In the specific case of cotton flows with presence of dirt and water repellent finish we recommend designing of another decolourisation approach.

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1. introduction

1.1 background

There are several actors who seek to recycle cotton fabrics and regenerate the cotton cellulose into other types of textile fibres via chemical recycling. A few mentioned here are Lenzing AG who has introduced RefibraTM, Evrnu who has introduced the Evrnu fibre and re:newcell who is developing the re:newcell pulp. An important processing aspect in chemical recycling is the ability to create a pulp with a low amount of impurities. This may be crucial since it enhances the fibre solubility as well as simplifies and stabilizes the regeneration process. Therefore, the textile waste is preferably purified in a decolourisation step where colourants and textile finish are removed before it can be turned into a dissolving pulp and further regenerated into textile fibres. The need to create secondary raw materials with low impurity content is also crucial from an environmental and health point of view. Chemical recycling comes with the inherent risk of keeping or spreading hazardous substances further in a re-circulated textile loop. Of specific concern are colourants or additives with carcinogenic or endocrine disruptive attributes and substances which are bioaccumulative such as PFAS (per- and polyfluoroalkyl substances), commonly used in textiles for dirt and water repellency e.g. in outdoor wear, work clothes or as auxiliary chemicals for print dyeing.

The involved actors are engaged to find suitable decolourisation approaches to purify the textile waste. The removal of impurities from colourants and textile finishes has been investigated by several researchers [1-4]. Haule et al. [1] has shown a purification method to hydrolyse a DMDHEU anti-wrinkle finish on cotton and then successfully regenerate the pulp to Lyocell fibres. In the EU-funded Trash-2-Cash project cotton fabric printed with reactive dye was decolourised and regenerated into Lyocell-type fibres by the Ioncell-F process [3].

In a previous study conducted within the Mistra Future Fashion project [4] was a decolourisation approach by re:newcell tested to remove a combination of yellow, red, and blue reactive dye components and 1,3-dimethyl-4,5-dihydroxyethylene urea (DMDHEU) anti-wrinkle finish on a cotton fabric. The pulps were regenerated into textile fibres and the physical viscose fibre properties evaluated. Despite that the physical properties of the regenerated fibres were inferior than for commercial fibres, the results from the decolourisation approach indicated a positive effect on removal of the colourants and anti-wrinkle agent.

One of the main motivations for this study is the many different additives used for textiles finish, why more research is needed to understand the impact of each of them on the decolourisation of a fabric and a subsequent regeneration process into textile fibres. The research groups have so far focused on decolourisation of dyed and wrinkle-free treated textiles, but no study has to the authors' knowledge been performed on fabrics with softener and dirt and water repellent agents added in the production of the textile.

1.2 purpose of the study

The present experimental study continues the initial work by Wedin et al. [4], using the same textile decolourisation approach by re:newcell. The main purpose of this study is to evaluate regenerated viscose fibres obtained from decolourised cotton and the effects of dyeing and finishing components in a decolourisation process. In more detail, the objectives are:

Pilot viscose processability and regenerated fibre quality are evaluated on pulp obtained from decolourised cotton treated with a reactive black dye, a DMDHEU anti-wrinkle finish and a fatty acid softener. The additives are applied individually and in combination. The viscose fibre quality, i.e. physical properties, is compared with values of commercial dissolving pulps evaluated on the same viscose pilot. The research questions are:

1. Can we obtain similar physical properties of decolourised regenerated fibres from a black cotton fabric as commercial viscose fibres by using the re:newcell decolourisation approach?
2. Can impact from a DMDHEU anti-wrinkle and a fatty acid softener finish on the viscose process be eliminated using this decolourisation approach? Thus, a desired result is when the decolourised cotton pulps reaches the same values as commercial wood pulp and any impact from the textile finishing components have been eliminated during the decolourisation process.

Potentially hazardous components from dye and textile finishes (reactive black dye, anti-wrinkle finish, softener, PFAS dirt and water repellent agent) are chemically analysed throughout the textile decolourisation process. The leading research question on this part is:

3. Could chemical recycling be a potential solution for recirculation of textiles which have a risk of containing hazardous substances of for example PFAS dirt and water repellent type?

1.3 design of the study

The set-up of the experimental activities includes the steps: 1) application of dyes and textile finishes on a virgin cotton fabric, 2) decolourisation of dyes and textile finishes by bleaching and 3) viscose dope preparation and fibre regeneration. The test has been performed on a virgin untreated fabric withdrawn from the textile production in order to avoid influence of any other textile finishing substances.

Based on a survey sent to the companies H&M, Boob design, Wiges, New Wave Group, Filippa K and Kappahl, black dye was the most interesting colour to test because of the high volume of dark textiles in apparel. The selection of the textile finishes (anti-wrinkle and softener) was based on the most sold in volumes today by the company DyStar® since it was assumed that the frequency of these finishes would be higher in post-production and used cotton textile waste. Due to time constraints in the project no simulation of use phase (washing, wearing etc.) has been performed on the treated fabric and the results represent post-production waste rather than post-consumer waste.

The second part was designed to focus on analysing potentially hazardous components throughout the decolourisation approach. The pulp and waste water from the trials with reactive dye, anti-wrinkle and softener were evaluated. Additionally, as a worst-case scenario, a common dirt and water repellent PFAS was applied on the virgin fabric, which was decolourised and screened for hazardous fluorocarbon degradation products that would end up in waste water and/or the pulp by the selected decolourisation approach.

The treatments and process steps are outlined in figure 1.

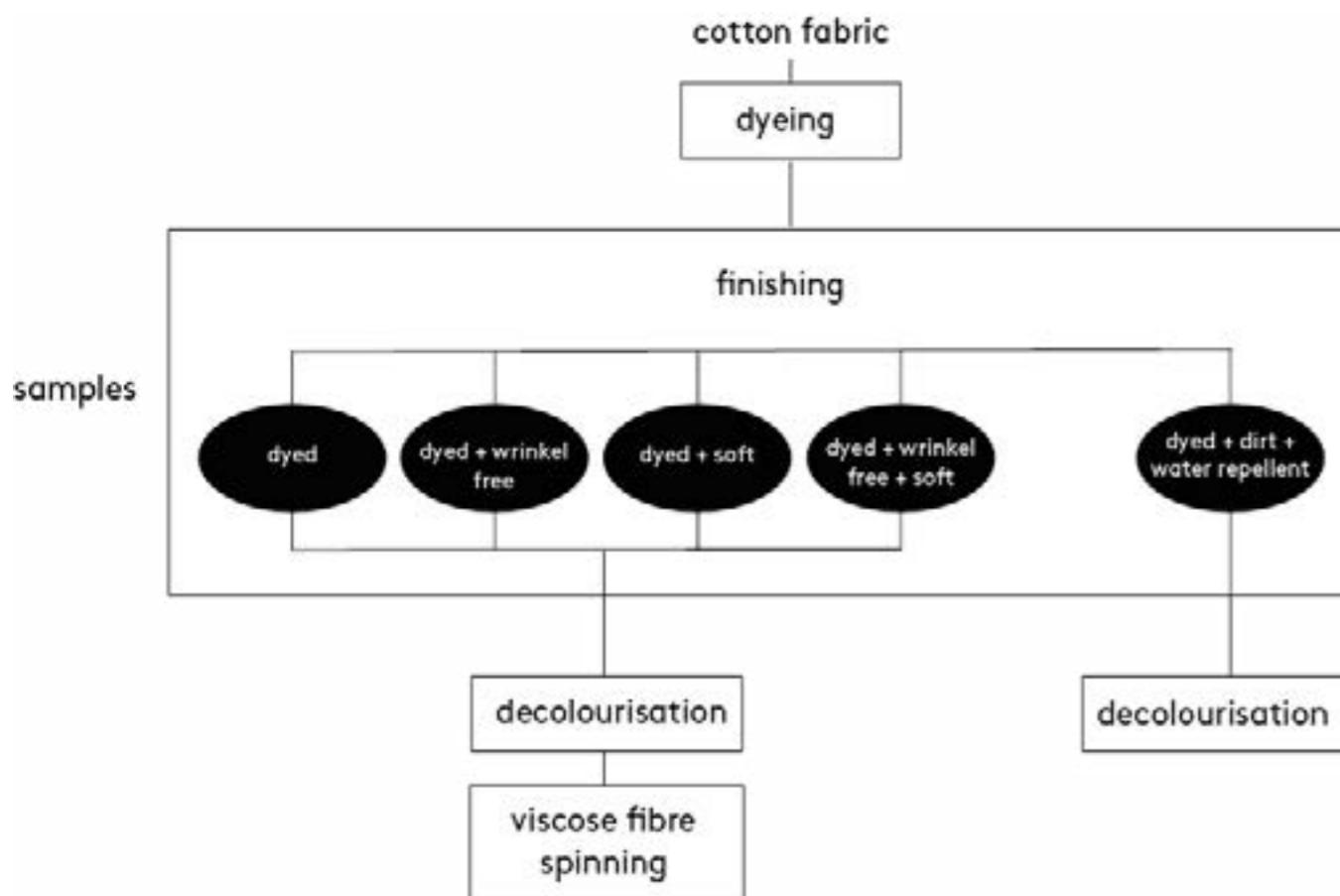


Figure 1: Outline of the experimental process steps.

'chemical recycling comes with the inherent risk of keeping or spreading hazardous substances further in a re-circulated textile loop.'

2. method

2.1 material and chemicals

Plain-woven mercerized cotton fabric prepared for dyeing with a grammage of 145 g/m² was supplied by H&M. The reactive dye Remazol® Deep Black RGB, wrinkle-free agent Evo Pret RFF and softener Evo Soft HWH were supplied by the DyStar group. Evo Pret RFF is a modified dimethyloldihydroxy-ethyleneurea (DMeDHEU) agent and Evo Soft HWH a hydrophilic fatty acid condensate. The dirt and water repellent (DWR) used for the worst-case scenario was Ruco-guard AFC6 (PFAS).

2.2 application of dyes and textile finishes

The application of dye was performed at the Swedish School of Textiles. The fabric was cut to a width of 50 cm and dyed in an overflow jet dyeing apparatus by Mathis AG. The maximum load capacity was 1 kg per batch. After initial testing, the adapter for drum dyeing was used due to the stiffness of the fabric. Overflow jet was no option. The following recipe was used:

NaCl, electrolyte	50 g/l
Arbyl SFR, wetting	1 g/l
Remazol® Deep Black RGB	4.5 % on fabric weight
Soda ash, pH	5 g/l
NaOH 50 %, pH	2 ml/l
Liquor ratio	1:15

The salt was dissolved together with the fabric directly into the machine at a starting temperature of 20 °C. After 15 min, the dye and wetting agent were added through the addition tank. The temperature was then increased from 20 to 60 degrees at 2 degrees/min. After 30 min at 60 °C the soda ash was added and after another 30 min the NaOH was added. Thereafter, the temperature was kept at 60 min. The total program was 120 min and shown in detail in Appendix. The dye bath sample was collected and the fabric was carefully rinsed and washed at 95 °C. The drying of the fabric was performed in a Mathis laboratory stenter frame at 120 °C. Because the fabric had to be dyed in batches, the fabrics were mixed before drying in order to eliminate possible process related differences.

The dyed fabric was divided into five batches and treated differently dependent on the textile finish conditions approach. Application of the anti-wrinkle and softener was made at the School of Textiles. The impregnation was performed in a foulard at 2 bar pressure and at approximately 75% wet pick-up followed by drying at 120 °C and curing at 150 °C for 60 s. The following impregnation conditions were used:

1. Impregnation in water
2. 1 g/l 60 % acetic acid + 120 g/l Evo Pret RFF
3. 1 g/l 60 % acetic acid + 20 g/l Evo Soft HWH
4. 1 g/l 60 % acetic acid + 20 g/l Evo Soft HWH and 120 g/l Evo Pret RFF

The dirt and water repellency (DWR) impregnation was performed at Swerea IVF. The dyed fabric was pressed and cleaned with pressurised air. The impregnation consisted of soaking samples piece by piece in the DWR solution for 2 min, pressing in Foulard, drying for 2 min in 120 °C and curing for 1 min in 160 °C.

2.3 decolourisation

The decolourisation was performed by re:newcell. A 3-step bleaching sequence was used to decolourise the dyes and hydrolyse the anti-wrinkle agent. The first step in this sequence was performed in alkaline conditions, while the second and third steps were performed in acidic conditions. The aim of the first two steps, where chemical charges and reaction times were same for all 4 samples, was to decolourise the dye whereas the third step was performed to hydrolyse the crosslinking agent as well as to achieve the intrinsic viscosity of the resulting pulp around 500 ml/g, which is the target intrinsic viscosity for viscose manufacturing process. In order to reach this target viscosity for all the pulps, the reaction time was different in each case.

Type of bleaching agent and exact conditions of all the steps cannot be disclosed for reasons of commercial confidentiality. From a sustainability perspective, the selected bleaching sequence could be integrated into the viscose process, leading to reduced costs and environmental impacts.

2.4 viscose dope preparation and fibre regeneration

Viscose dope preparation and fibre regeneration was performed by MoRe Research. The preparation of viscose solution comprises several steps, in the following order: steeping, pressing, shredding, pre-ageing, xanthation, dissolving, and ripening. The steeping was carried out for 30 min at 50 °C with 18 % sodium hydroxide.

In the pressing step, a press factor of 2.8 was targeted, but the reached press factor varied between the samples (presented in the result section). The shredding was performed at 50 °C for 30 min followed by pre-ageing at 50 °C. A pre-ageing curve was established to determine the required pre-ageing time to reach a limiting viscosity of 250 ml/g before xanthation. This curve was employed to determine the pre-ageing time for all samples, since a controlled limiting viscosity number after pre-ageing is required for good processability [5, 6]. Pre-ageing times to reach 250 ml/g for the various samples are presented in the results section. Xanthation was performed for 2.5 h at 32 °C with the addition of 36 % carbon disulphide; the cellulose xanthate thus formed was subsequently dissolved for 3 h at 7 °C.

Finally, the viscose solution was ripened for 16 h at room temperature. Before wet spinning of the viscose solution, the solution was filtered (30 µm) and deaerated. The wet spinning was performed at 50 °C using a spin-bath composition of 130 g/L of sulphuric acid, 310 g/L of sodium sulphate, and 9.5 g/L of zinc sulphate. A pump flow of 5 ml/min was used, and a single spinneret with 90 holes, 80 µm in diameter, giving an extrusion speed of approximately 11 m/min. Godet speed 1 was set to 18.8 m/min and godet speed 2 was set to 29.9 m/min, giving an approximate draw ratio of 1.7 and an approximate stretch 59 %.

2.5 characterisation of fabrics, pulps, waste water and viscose fibres

Several methods have been used to characterise the fabrics, decolourised pulps and process waters for chemical residues, cellulose pulp properties, viscose solution for spinning and performance of the regenerated viscose fibres. The characterisation has been performed by RISE, More Research, re:newcell and Swerea IVF (RISE IVF since 01-10-2018). The included analyses were: intrinsic viscosity number, ISO-brightness, molecular mass distribution by gel permeation chromatography (GPC), Fourier transform infrared (FTIR) spectroscopy recorded in ATR mode, metal analysis, ash content, extractive content, FTIR on the extracts and quantification of extractives.

The viscose solution was characterised in terms of the cellulose content, alkaline content, ball fall, gamma number, and reduced filter-clogging number (K_r). The gamma number is defined as the number of xanthogenate groups per 100 anhydroglucose units and describes the degree of substitution, with the highest theoretical gamma number being 300. The reduced filter-clogging number (K_r) was determined according to Treiber [5], Treiber et al. [7], and the ZELLCHEMING III/6B/68 standard method [8]. K_r is obtained by filtering the viscose solution and measuring the filtered amount and ball fall time. The filtered amount is measured after 20 min and again after 1 h. Ripening index was measured prior to spinning according to the Hottenroth method [9]. The value is indicative of how fast the spin dope coagulates, and can be seen as measure of the quality of the viscose solution. A lower value means faster coagulation, making the material less suited for spinning. Detailed method descriptions are found in Appendix 1.

All fibres were tested for tenacity and elongation in both the conditioned and wet states. A Vibroskop 400 instrument (Lenzing Instruments, Gampern, Austria) was used according to SS-EN ISO 5079 to determine the fibre titre (i.e., linear density of the fibres), which is derived from the vibration frequency of the fibre. A Vibrodyn 400 instrument (Lenzing Instruments) was used according to SS-EN ISO 5079, ISO1130-1975, and ISO1973 to determine the fibre elongation (i.e., extension at break) and tenacity. Twenty fibres from each sample were tested. The standard deviation of the mechanical properties was adjusted for a 95 % confidence interval.

Screening for hazardous chemicals involved free formaldehyde, aniline, GC-MS screening and fluorine analysis. The first three analyses were conducted by Swerea IVF. Free formaldehyde (CAS no 50-00-0) was determined quantitatively according to SS-EN ISO 14184-1:2011. Aniline was determined by extracting the test material with 20 ml methanol for 1 hour in an ultrasonic bath. Internal standard was added to the extract which then was concentrated to about 2 ml using a rotary evaporator. The extract was diluted to 5 ml with methyl tert-butyl ether and analysed with a capillary gas chromatography coupled with a mass spectrophotometer (GC-MS). GC-MS screening of organic compounds on textile and pulp samples was performed by extracting the test material with methanol using a Dionex ASE 200, followed by detection with GC-MS (full scan mode). Tests on process/waste water was directly analysed on GC-MS (full scan mode). A NIST11 mass spectral database was used to, if possible, identify the detected compounds. The analysis is not quantitative.

The total fluorine content of the samples was determined by Combustion Ion Chromatography (CIC). The samples are pyrolysed in an oxidising atmosphere, the resultant vapours are absorbed in an aqueous solution, then introduced directly into the IC system for analysis of fluoride (F-) content. The analysis was performed by Steffen Schellenberger, PhD student at the Department of Environmental Science and Analytical Chemistry (ACES), in a cooperation with the research project SUPFES.

'removal of impurities in a decolourisation stage would enhance the fibre dissolving capacity and stabilise and simplify the fibre regeneration process.'

3 result and discussion

3.1 decolourisation

The target intrinsic viscosity of 500 ml/g after the decolourisation was successfully reached (Table 1). Furthermore, the bleaching response was similar for the decoloured pulps. The ISO brightness after decolourisation was in the range of 68-77 % and did not reach the desired commercial ISO brightness of dissolving pulps at approximately 85 % or higher (Table 1). Logically, a small change in the removal of such dark colourant has a large influence on the brightness. The bleaching response may be sensitive to inhomogeneities in the defibrated material which explain the broad brightness interval of the samples.

Table 1: Intrinsic viscosity and ISO brightness of pulps after decolourisation

Sample	Intrinsic viscosity (ml/g)	ISO brightness (%)
Dyed	490	68
Dyed and wrinkle free	470	73
Dyed and soft	510	68
Dyed, wrinkle free and soft	500	77

FTIR spectra confirmed that the decolourisation step removed the anti-wrinkle finish (Figure 2) since the peak at approximately 1680 cm⁻¹ (carbonyl stretch) disappeared. The FTIR could not distinguish the softener. After the decolourisation, the FTIR spectra for all the samples (Figure 2) looked very equal. As desired, the ash, metal and extractive content, FTIR spectra of the extractives and molecular mass distribution were similar for all the decoloured pulps (see Appendix). Since the target viscosity was reached and impurity contents considered as low, "standard" dissolving and spinning conditions could be used in the viscose dope preparation and regeneration.

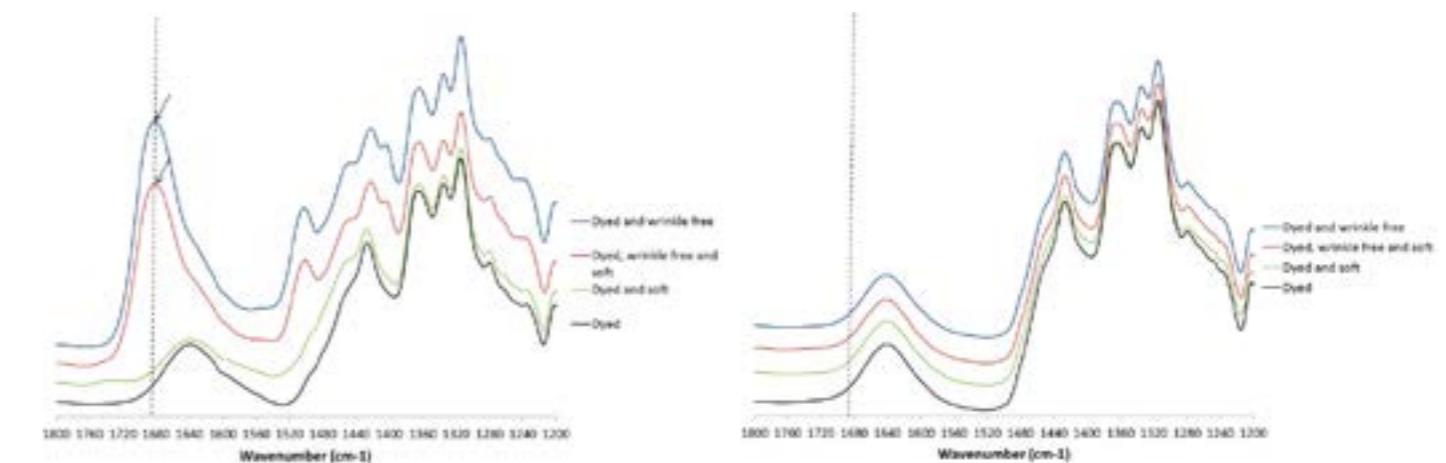


Figure 2: ATR-FTIR spectra of treated fabrics after extensive washing (left) and of corresponding pulps after decolourisation (right).

3.2 viscose dope preparation and fibre regeneration

Using the bench scale pilot equipment at MoRe Research, the bleached textile pulp was prepared into a viscose dope which in turn was regenerated into viscose fibres. The quality of the viscose dope was characterised by the cellulose concentration, sodium hydroxide concentration, gamma number, ball fall time and reduced filter clogging value (Table 2). A portion of the produced viscose dope was also filtered and de-aerated. This viscose dope was then used for the fibre regeneration and for the analysis of the ripening index (Hottenroth index). The regenerated fibres were analysed with respect to their titer, elongation and tenacity (Table 2).

Table 2: Analysis results for the four samples of the viscose trial as well as reference values. The values from the fibre analyses are presented with their standard deviation (parenthesised). The reference values correspond to alkali cellulose or viscose dope produced from "normal" commercial dissolving pulp, and fibres regenerated from commercial viscose.

Unit	Dyed	Dyed and wrinkle free	Dyed and soft	Dyed, wrinkle free and soft	Reference
Alkali cellulose					
NaOH	%	15.3	16.9	16.2	16.5
Cellulose	%	34.1	34.2	28.3	30.8
Pre-ageing					
Pre-ageing time	min	143	141	164	154
Viscose					
NaOH	%	4.2	4.1	4.6	4.1
Cellulose	%	9.2	9.7	9.5	9.3
Filtering (m20)	g	1.257	0.662	3.901	2.198
Filtering (m60)	g	1.374	0.740	4.191	2.305
Ball fall time	s	77.7	74.8	64.5	61.7
Kr		2434	4527	868	1627
y-number		40	34	38	34
Ripening index		15	14	9.5	9.5
Spinning					
Fibre yield	g	0.44	1.59	0.50	1.50
Fibre (dry)					
Titer	dtex	2.36 (0.41)	2.54 (0.39)	2.49 (0.23)	2.43 (0.19)
Elongation	%	17.32 (3.19)	17.35 (4.17)	15.80 (2.31)	15.28 (2.86)
Tenacity	cN/tex	11.87 (2.25)	11.47 (2.23)	15.64 (1.85)	15.97 (2.91)
Fibre (wet)					
Titer	dtex	2.36 (0.36)	2.41 (0.33)	2.44 (0.26)	2.48 (0.20)
Elongation	%	21.39 (4.27)	18.36 (2.95)	17.12 (2.44)	17.25 (3.29)
Tenacity	cN/tex	4.56 (1.03)	5.21 (1.02)	6.09 (1.26)	7.01 (1.29)

The pre-ageing curve for the four samples (Figure 3) showed that all samples behaved similarly and had quite short pre-ageing times. To reach an intrinsic viscosity of 250 ml/g, the pre-ageing time varied between about 140 and 165 min for the samples (Table 2). By experience, commercial dissolving pulps often have pre-ageing times around 180–360 min using this pilot with the same settings. As such, the pre-ageing time was quite short for all samples, which is generally preferred by the industry.

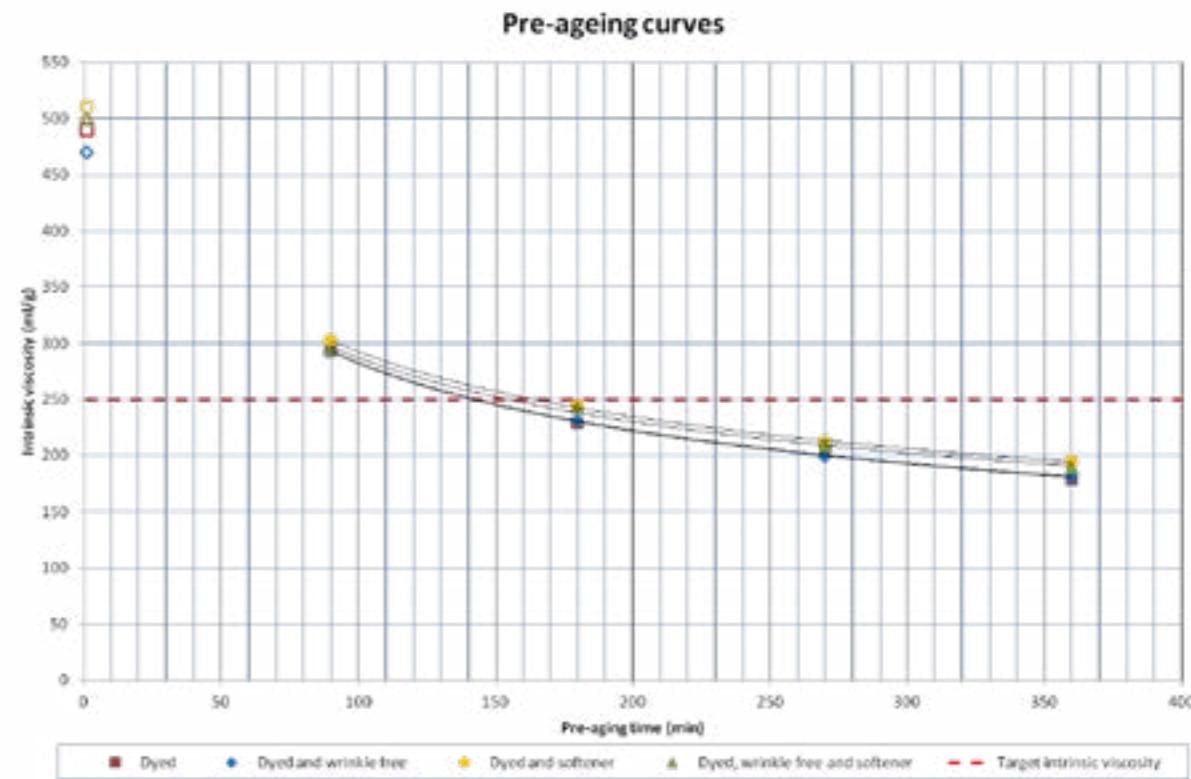


Figure 3: Pre-ageing curve for the tested bleached textile pulps (solid) and the initial limiting viscosity of the pulps (hollow). The target intrinsic viscosity of 250 ml/g is marked by the red dashed line.

The viscose dope preparation of the bleached textile pulps generally went well, but with one difference from processing of commercial dissolving pulps which was observed as that the bleached textile pulps were harder to dewater in the pressing stage after mercerization. This made the normal target press factor of 2.8 unattainable. In these trials, the press factor which was reached was 2.90, 3.10, 3.25 and 3.25 for the four samples, respectively. The cellulose concentration and sodium hydroxide concentration of both the alkali cellulose and the viscose dope is measured predominantly as a quality control of the viscose dope preparation.

Comparing the sodium hydroxide and cellulose content in the alkali cellulose with the reference values showed that for all four samples the cellulose content in the alkali cellulose was in the lower range or even below the reference. The sodium hydroxide content in the alkali cellulose was somewhat above the reference for three of the samples. This is expected for the elevated press factors of this trial. The cellulose and sodium hydroxide concentrations of the viscose dopes were all at reasonable levels, as were the ball fall viscosity for all four samples (Table 2).

The reduced filter-clogging number, Kr, (Table 2) ranged between 868 and 4527 in the order: dyed and soft < dyed, wrinkle-free and soft < dyed < dyed and wrinkle free. A high Kr number may indicate that the spinning could be negatively affected because the viscose solution contains differing amounts of micro- and macroscopic gel particles. Not only can macro gel particles influence the spinning process, but sub-microscopic particles and aggregates in the filtered spinnable solution can also have an impact [10].

Particles and aggregates, however, are inherent to the viscose dope, contributing to the properties of the dope and ultimately of the fibres. Typical Kr values of commercial pulps used in viscose production are 100–1200. While the results of this relatively small sample pool should be interpreted with caution, the general trend in the Kr values is that softener seems to have a positive effect on the Kr value (lowering it) while the anti-wrinkling agent seems to have a negative, albeit smaller, effect (increasing it). Normally, higher Kr values are correlated with faster pressure build-up in the spinning, due to clogging of the spinneret. For a trial where this pressure build-up is rapid enough that it limits the spin-trial (read: in most cases), the amount of fibres regenerated from one spinning trial can be seen as a rough measure of processability. Surprisingly, in this trial, there was no such correlation between Kr values and fibre yield.

The gamma number indicates the number of substituted groups of the cellulose polymer, and the ripening index describes how quickly the viscose dope coagulates. While the gamma number was within the reference range provided, there seems to be a trend where the wrinkle free samples obtained lower gamma numbers (t value of 0.04 with student t-test). With this in mind, it is possible that remnant of anti-wrinkling agent was interfering with the xanthation of the pulp, and that the tentative effect on the Kr values is an effect of this. For the ripening index, a general rule of thumb is that a value above 9 is considered good. While all samples fall above this threshold, the ripening index for the samples with softener were significantly lower (t value of 0.01 with student t-test).

The regenerated viscose fibres were analysed with respect to their titer, tenacity and elongation in both dry (conditioned at 23°C and 50 % RH) and wet state (Table 2). Both the titer and elongation for all samples were a bit above the reference ranges, and is to be seen as normal. The tenacity was well below the reference range for the dyed and dyed+wrinkle free samples, while the tenacity of the dyed+soft and dyed+wrinkle free+soft samples were just below the reference range. The significance of differences in tenacity between the samples was tested with a student t-test, grouping the samples pair-wise and testing in presence or absence of softener and anti-wrinkling agent, respectively. It was found that presence of softener significantly affected the tenacity of the fibres (t-value 10-6 for samples without anti-wrinkling agent and t-value 3*10-6 for samples with anti-wrinkling agent). No significant difference was found between the elongations of the fibres from the different samples. The regenerated fibres are shown in Figure 4.



Figure 4: Materials after the process steps: 1) Original fabric 2) dyed fabric with or without textile finishes 3) decolourised pulp before and after grinding and 4) regenerated fibres.

Evo Soft HWH is a hydrophilic fatty acid condensate and applied as a polyethylene (PEG) dispersion. Due to its chemical nature, the softener is not resistant to washing and the decolourisation process. It would thus likely be removed in the various process steps and is not expected to affect the viscose production. However, a tendency for the softener to positively affect the viscose process was seen in this project. While caution should be taken in drawing conclusions from such a small trial, it does raise interesting questions about the persistence of this kind of softener.

PEG derivatives are sometimes used in the viscose process as performance additives [11]. As the softener used in this trial was applied in a PEG dispersion, it is possible that the positive effect on the Kr values and the tenacity is due to the same mechanism as for these performance additives, caused by traces of PEG. It should however be noted that a stable material feedstock is generally preferred in industrial processes. A process which fully removes any softener or effect thereof would thus likely be preferred, as it would stabilise and simplify the further processing of the material.

The viscose dopes for all four samples in this trial exhibited high to very high (read: poor) Kr values, indicating that all samples would have poor processability in the viscose process. For the samples where softener was absent, the dry tenacity of the regenerated fibres was also quite low. The bleached textile pulp was thus not suitable for viscose production with the settings used. It should however be noted that no process optimisation was performed for the material in this project, and that the settings used were chosen for benchmarking of wood-based dissolving pulps. It is thus possible that process optimisation for the material could drastically change the quality of the viscose dope and regenerated fibres produced from it. The generally preferred scenario is to adapt the pulp in the decolourisation stage rather than in the viscose process. Since the decolourisation approach in the present study succeeded to hydrolyse the wrinkle-free treatment the approach has potential for such substrates.

Moreover, a softener not resistant to washing would likely not be an issue for the decolourisation to handle. Thus, this decolourisation approach has potential, but more optimisation is required for dyed cotton substrates with or without wrinkle-free and softener treatments.

3.3 potential hazardous chemical substances throughout a chemical recycling

The study of hazardous chemicals throughout the chemical recycling was based on a theoretical model on transfer of substances through different recycling processes from Jönsson and Posner [12]. The base for selection of analysis stems from their indication of which hazardous chemicals are relevant for cotton textiles as well as testing standard for Öeko-Tex certification. The study included analysis of samples both from this task as well as the previous task 4.1.6 which carried out a similar study, although with a different cotton fabric. Colourants, auxiliaries and finishes were not identical but of similar common types. The previous task is described in further detail in [4].

formaldehyde

The sample with dye, wrinkle free and softener was tested for concentrations of formaldehyde both before and after decolourisation. Results showed no detectable concentrations (< 16 mg/kg). In the previous task of this project where a different type of wrinkle-free agent was used, the same analysis showed formaldehyde values of 110-124 mg/kg (which is above Öekotex standard 100 for clothes <75 mg/kg). Unfortunately, it was not possible to analyse the samples after decolourisation within the scope of the study but as formaldehyde is rather volatile it is likely that the concentrations were low and even below detectable concentrations in the pulp.

aniline

Analysing free aniline can give an indication of the presence of potentially hazardous (2) azo-substances (colourants) and their degradation products. A sample from the previous task was tested for free aniline but results showed no detectable concentrations. The colourants used in this task were considered to have even better environmental performance (marketed with ECO-passport claims from Öeko-Tex) and therefore not likely to give response on the analysis. None of the samples from task 4.1.7 was therefore tested. Results from a previous similar study by Jönsson et al. [13] showed presence of aniline in both pre- and post-consumer cotton but the concentrations were not detectable after decolourisation. If the sample tested had given a detectable concentration an analysis of the waste water had been performed here to see how the process affects the substances.

GC-MS screening of organic compounds

The textile sample with dye, wrinkle free and softener was analysed and compared with the pulp after decolourisation treatment. The textile showed presence of an organic compound, 1,3,4,6-tetramethyl-3a,6a-dihydroimidazo[4,5-d]imidazole-2,5-dione; CAS 10095-06-4, but it was not detected in the pulp after treatment indicating that it is either broken down or washed out. Analysis on the process water did not show detectable concentrations either, which on the other hand could be due to the high dilution of the process water.

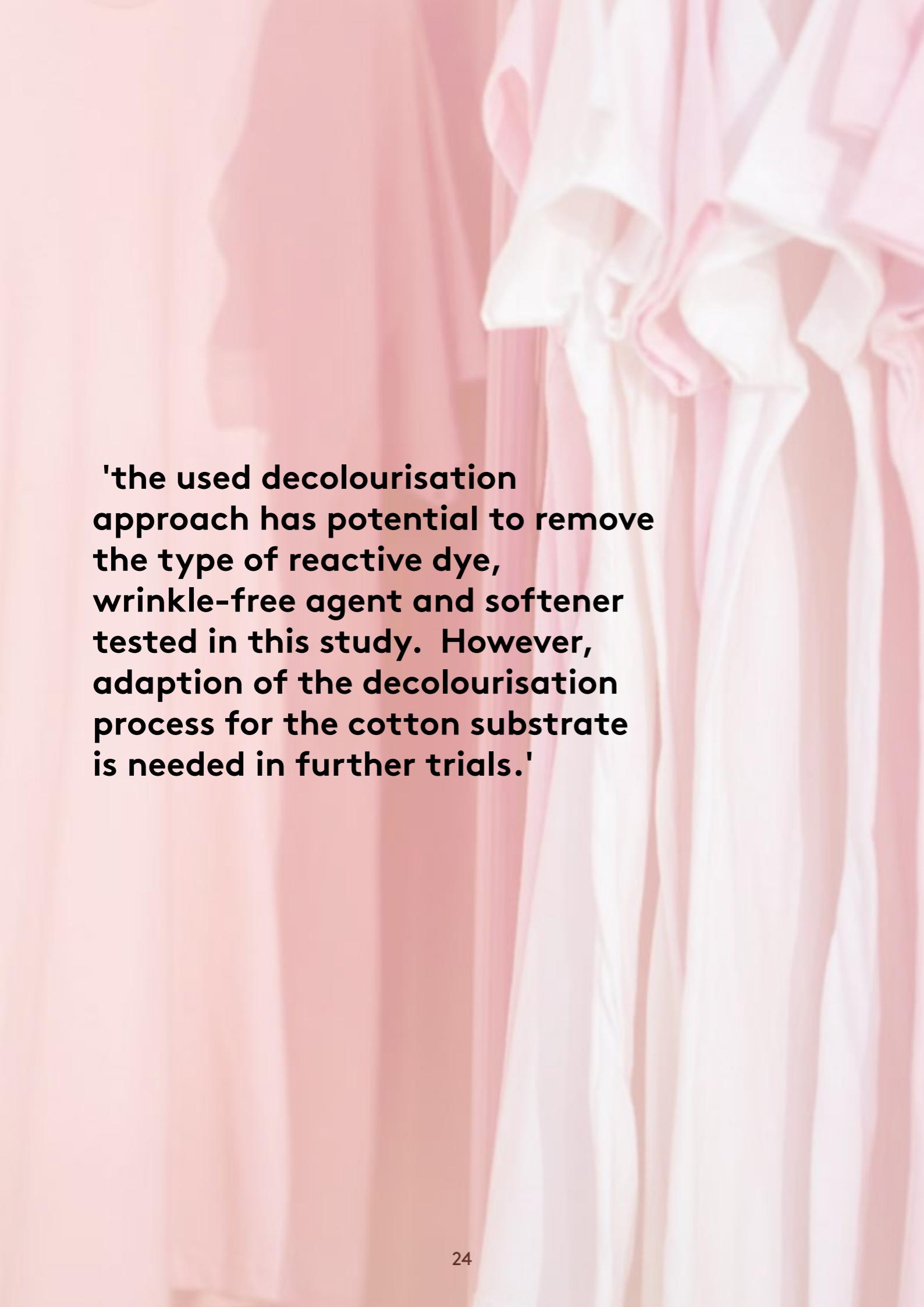
fluorine analysis

The sample impregnated with DWR was analysed for total fluorine concentrations. Results showed that approximately one third of the applied fluorine (weight per weight sample) remained in the pulp through the decolourisation treatment. This indicates that the DWR forms a bond with the fibre that does not break in the process. Note that the decolourisation process has not been designed to target the chemicals used in the DWR. The DWR consists of large molecules that are very hydrophobic so it can also be that the bond has been broken but the substance is not washed out due to its characteristics. To differentiate between which of the reasons for the high F presence in the pulp another washing method could be tried (washing detergent and dilution in something else than water). As this sample has not been washed or worn, the results also show that about two thirds of the fluorine are indeed washed out in the decolourisation treatment and ends up in the process water. The concentration of fluorine in the waste water from decolourisation was low (due to high dilution) and much lower than levels detected in the dyed cotton that had not been treated with DWR. The raw cotton was not analysed in this study.

Table 3: Carbon fluorine content in dirt- and water repellent (DWR) treated fabric, in subsequent decoloured pulp and in the decoloured waste water.

Sample	C F [ng/mg] (average)	Standard deviation
Treated fabric		
Dyed, dirt and water repellent	3179.2	47.3
Dyed reference (without DWR)	73.3	-
Decolourized pulp		
Dyed, dirt and water repellent	1355.3	67.7
Waste water from decolourisation		
After first bleaching step	19.5	0.8
After second bleaching step	2.21	0.2

(2) A large group of substances of which some are connected to varying hazard categories; give allergic reactions, eye irritants, toxic for humans and ecosystems as well as carcinogenic effect.



'the used decolourisation approach has potential to remove the type of reactive dye, wrinkle-free agent and softener tested in this study. However, adaption of the decolourisation process for the cotton substrate is needed in further trials.'

4 conclusions

The aim of this report was to investigate decolourisation of cotton and regeneration into viscose fibres and an analyse the potentially hazardous textile finish components throughout the decolourisation approach. From the three main research questions the results showed that:

1. Viscose fibres can be produced from a decolourised cotton fabric but the physical properties suitable for a viscose pulp were not obtained.
2. The decolourisation approach was proven with spectroscopy to successfully remove the anti-wrinkle agent, but the removal of the softener was not possible to analyse. The impact from both the anti-wrinkle agent and softener on the physical properties of the produced viscose fibres was though proved to not be fully eliminated by the decolourisation approach.
3. The investigation was not able to neither prove nor disprove the hypothesis of chemical recycling be a potential solution for recirculation of textiles with a risk of containing hazardous substances (e.g. PFAS dirt and water repellent type)

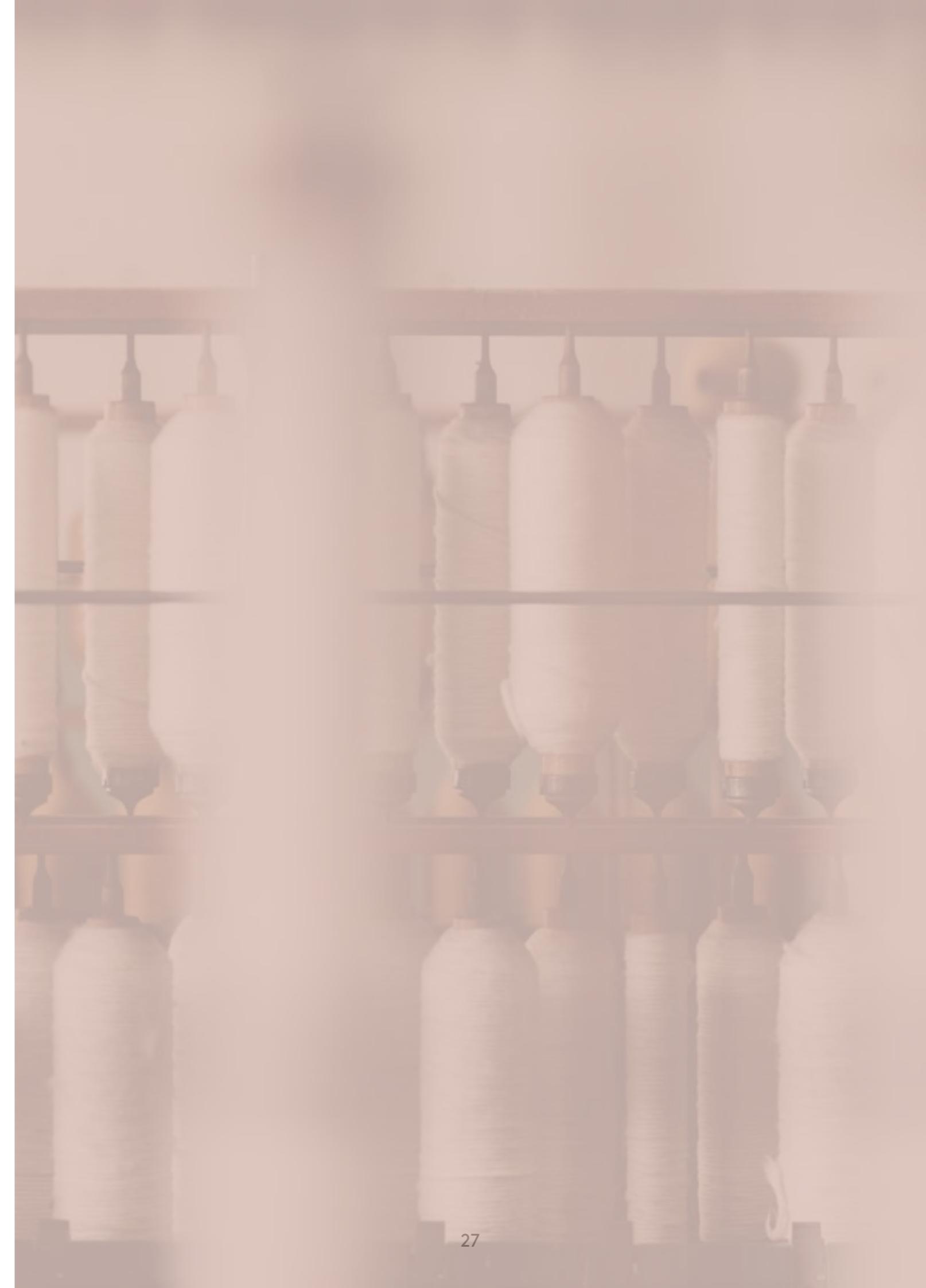
The results from the fibre regeneration trials showed that viscose fibres could be spun after decolourisation of reactive dyed cotton fabrics treated with wrinkle-free and fatty acid softener textile finish. However, the preferred scenario of pulps suitable for the viscose process was not obtained. The reduced filter clogging numbers (K_r) and the mechanical fibre properties of the decolourised black cotton fabric was worse than that of commercial wood pulps. Indications were also noticed that the softener had a positive effect on the K_r value while the anti-wrinkling agent seemed to have a negative, albeit smaller effect. A tendency for the softener to positively affect the viscose process was seen on the mechanical fibre properties. No tendency was seen for the anti-wrinkle agent on the mechanical fibre properties. While caution should be taken in drawing conclusions from such a small trial, it does raise some interesting questions about the persistence of the softener.

The results from the analysis on dirt and water repellent substance (DWR/PFAS) throughout the decolourisation process showed that two thirds of the applied fluorine were washed out in the process (it would probably also wash out with normal wear and washing), while one third remained in the pulp. This indicates that the DWR binds to the fibre in a way which the decolourisation approach is not able to break down. Altogether, the study does neither prove nor disprove the hypothesis that chemical recycling could be a potential solution for recirculation of textiles with risk of containing hazardous substances of this sort.

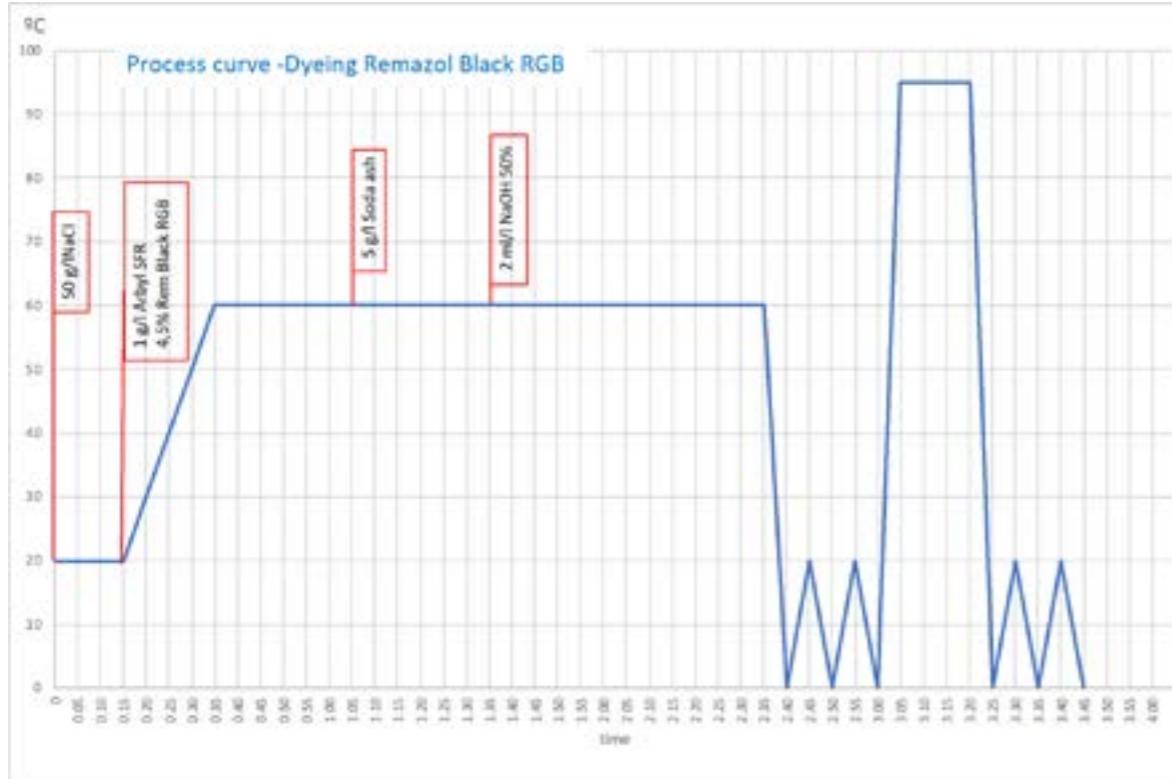
The recommendations from this study is further optimisation of the decolourisation approach for different dyed cotton substrates with anti-wrinkle finish and softener since although the decolourisation approach was not able to produce pulps suitable for the viscose process, the approach succeeded to hydrolyse the anti-wrinkle-free agent. In addition to this the decolourisation approach should also be designed to actively target the removal of DWR agents and thereby contribute to the knowledge on whether chemical recycling could be used for textiles treated with these kinds of substances.

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6 appendix



intrinsic viscosity

The limiting viscosity number was determined in cupriethylenediamine (CED) solution using a capillary viscometer following the ISO 5351:2010 standard method.

fourier transform infrared (FTIR) spectroscopy

FTIR spectra on fabrics and pulps were recorded in ATR mode using a Varian 680-IR FTIR spectrometer. Base line correction was made and 32 scans collected. The spectra were normalized at 2900 cm⁻¹ (C-O-C stretching vibration).

ash content and metal analysis

The samples were converted to ash at 525°C; the residues were subsequently dissolved in hydrochloric acid and analysed by ICP-OES after dissolution. Ash content determined according to ISO 1762. The ash and metal contents are shown below.

Ash content in % DS.

Sample	Ash content, at 525°C (%)
Fabric	
Untreated	<0.2
Dyed	0.5
Dyed and wrinkle free	0.5
Dyed and soft	0.5
Dyed, wrinkle free and soft	0.4
Dyed, dirt and water repellent	0.3
Pulp	
Dyed	<0.2
Dyed and wrinkle free	<0.2
Dyed and soft	<0.2
Dyed, wrinkle free and soft	<0.2
Dyed, dirt and water repellent	<0.3

Metal content. Amounts in mg/kg DS.

Sample	Ca	Cu	Fe	K	Mg	Na	P	Si
Fabric								
Untreated fabric	218	0.4	7.3	26	117	155	17	19
Dyed	1372	30	12	8.6	105	97	9.8	13
Dyed and wrinkle free	1206	30	16	13	171	157	48	15
Dyed and soft	1457	32	19	8.7	126	101	9.9	16
Dyed, wrinkle free and soft	1071	28	11	13	116	166	42	19
Dyed, dirt and water repellent	758	12	13	13	77	117	<1	16
Pulp								
Dyed	484	4.6	11	4.0	31	15	3.9	4.0
Dyed and wrinkle free	460	3.4	4.2	3.9	28	13	3.4	3.2
Dyed and soft	495	4.3	7.3	5.0	31	14	3.5	3.8
Dyed, wrinkle free and soft	249	5.2	6.4	4.8	19	16	3.4	3.8
Dyed, dirt and water repellent	543	20	7.2	12	36	54	<1	16

Metal content. Amounts in g/kg DS.

Sample	Al	As	Ba	Cd	Co	Cr	Mo	Mn	Ni	Pb	Tl	V	Zn
Fabric													
Untreated fabric	5461	<600	459	64	<125	473	<125	455	<125	<600	330	160	687
Dyed	3772	<600	1534	<60	<125	708	137	323	602	<600	<125	130	2007
Dyed and wrinkle free	3966	<600	1550	<60	<125	1225	146	397	770	<600	275	174	1748
Dyed and soft	4279	<600	1639	<60	<125	1138	<125	522	786	<600	256	<125	2569
Dyed, wrinkle free and soft	4032	<600	1360	<60	<125	676	<125	344	671	<600	325	<125	1758
Dyed, dirt and water repellent	4828	<600	1276	<60	<125	1571	<125	678	279	<600	945	<125	1736
Pulp													
Dyed	2058	<600	1011	<60	<125	468	<125	797	432	<600	<125	<125	2605
Dyed and wrinkle free	1615	<600	861	<60	<125	371	<125	198	225	<600	<125	<125	554
Dyed and soft	1828	<600	947	<60	<125	429	<125	199	237	<600	<125	<125	710
Dyed, wrinkle free and soft	1603	<600	483	<60	<125	398	<125	249	242	<600	132	<125	605
Dyed, dirt and water repellent	3131	<600	1066	<60	<125	805	<125	379	709	<600	842	<125	1755

Molecular mass distribution of cellulose

The cellulose was derivatised by tricarbanilation using phenyl isocyanate. The molar mass distribution of the cellulose tricarbanilates were determined by GPC using tetrahydrofuran as the mobile phase. The molar mass data from the refractive index (RI) detector and UV detector (at 280 nm) are presented below for the decolourised pulps.

Sample	M _p (kDa)	M _n (kDa)	M _w (kDa)	PD (M _w /M _n)
RI				
Dissolving sulfite pulp ref	725	133	6612	42.4
Untreated fabric	1471	635	1669	2.6
Pulp				
Dyed	251	143	5370	37.4
Dyed and wrinkle free	257	138	3992	29.1
Dyed and soft	264	153	5796	37.7
Dyed, wrinkle free and soft	292	145	3677	25.3
UV				
Dissolving sulfite pulp ref	958	163	5004	31.4
Untreated fabric	1452	618	1767	2.9
Pulp				
Dyed	294	170	5166	30.4
Dyed and wrinkle free	279	165	3878	23.5
Dyed and soft	311	185	5120	27.7
Dyed, wrinkle free and soft	352	171	3595	21.1

acetone extractive content and FTIR on the extracts

The samples were extracted with acetone in a Soxtec apparatus according to SCAN-CM 49:03. The extract was evaporated (105°C) and measured gravimetrically.

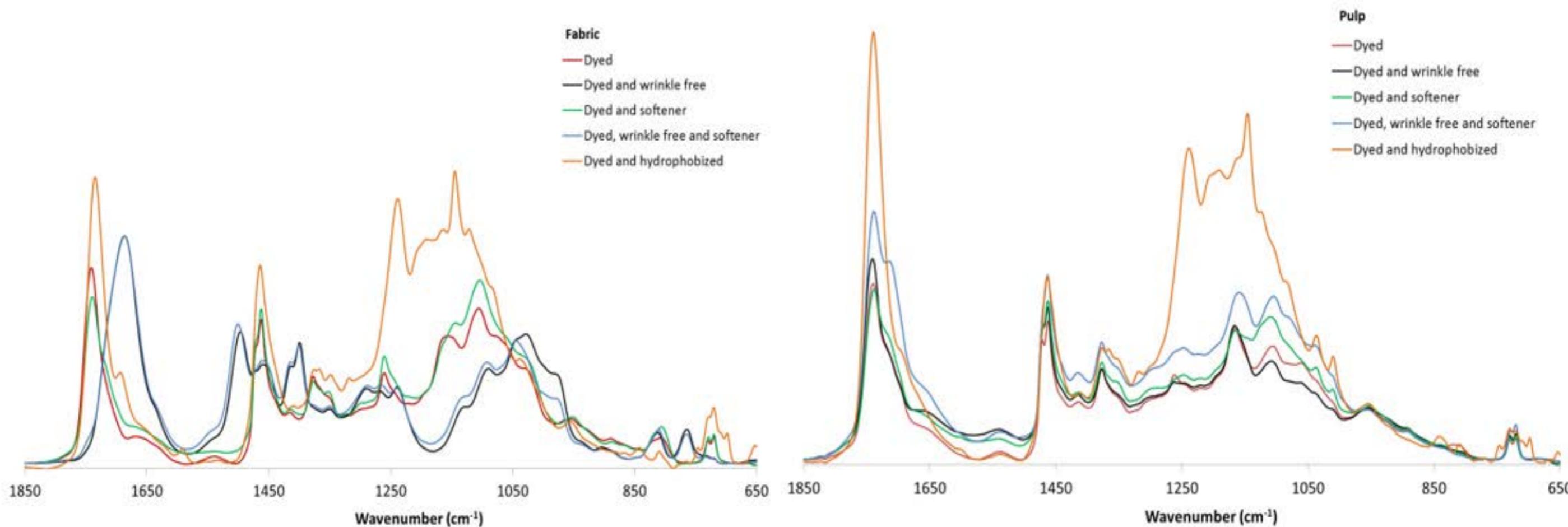
Sample	extractive amount (m/g DS)
Fabric	
Untreated	4.0
Dyed	2.2
Dyed and wrinkle free	19.0
Dyed and soft	3.8
Dyed, wrinkle free and soft	22.0
Dyed, dirt and water repellent	4.6
Pulp	
Dyed	<1.2
Dyed and wrinkle free	1.4
Dyed and soft	1.6
Dyed, wrinkle free and soft	1.4
Dyed, dirt and water repellent	<3.0

Quantification of extractives by GC/FID was made for the untreated fabric to investigate

occurrence of natural impurities. The extract was derivatised and quantified as specified groups using a short column gas chromatograph with flame ionisation detector (GC/FID Agilent HP6890). The substances separated as groups and reported as amounts in mg/g DS and relative distribution in % of extracted sample.

Sample	Fatty acids 11	Fatty acids 22	Sterols	Unknown	Steryl esters	Triglycerides	Total
Untreated fabric	0.3/8.9 %	0.3/8.4 %	1.2/34.4 %	0.9/25.2 %	0.4/11.5 %	0.4/11.5 %	3.5/100 %

FTIR spectra on evaporated extracts from the acetone extractive content analysis were recorded in ATR mode using a Varian 680-IR FTIR spectrometer. Base line correction was made and 32 scans collected. The spectra to the left are on fabric and to the right on pulp.



cellulose content of the viscose

The cellulose content of the viscose was determined by pressing a small amount of viscose solution between two glass plates. The plates were separated by sliding and then placed in a sulphuric acid bath until the viscose film which formed had loosened from the plates. The cellulose film was washed thoroughly with water and dried by removing excessive moisture with a filter cloth. The film was then placed in an oven at 105°C for 4 h. The cellulose content was calculated as the weight of the sample after drying divided by the weight of the viscose solution.

alkali content of the viscose

The alkali content of the viscose was determined by first weighing about 2.5 g of viscose and then dissolving it in 100 mL of deionised water with a magnetic stirrer. The solution was boiled after which 10 mL of 0.5 mol/dm³ sulphuric acid was added and it was set aside to cool. When the solution reached room temperature, several drops of phenolphthalein were added and then the solution was titrated with 1.0 mol/dm³ sodium hydroxide until the solution changed colour. The following equation was used to calculate the alkali content:

$$\text{Alkali content (\%)} = \frac{(V_{\text{H}_2\text{SO}_4} \cdot 2 \cdot c_{\text{H}_2\text{SO}_4} - V_{\text{NaOH}} \cdot c_{\text{NaOH}}) \cdot M_{\text{NaOH}} \cdot 100}{m \cdot 1000}$$

where

V = volume in mL,

c = concentration in mol/dm³, and

m = mass of the viscose solution in grams.

gamma number of the viscose

A viscose sample of about one gram was dissolved in 50 mL of 1 % NaOH by stirring for 30 min. The solution was then transferred into a 100-mL volumetric flask and diluted with 1 % NaOH. The flask was shaken and 5 mL of solution were transferred to a bottle containing 1.8 g of Amberlite IRA 402 ion-exchange resin (Sigma-Aldrich, St. Louis, MO, USA). The solution was left to react with the resin for 10 min, after which it was filtered using Munktell 5 filter paper (Fisher Scientific, Waltham, MA, USA) into a 250-mL volumetric flask and diluted with deionized water. The absorbance at 303 nm was measured with a CARY 100 Scan UV-visible spectrophotometer (Agilent, Santa Clara, CA, USA). The gamma number was calculated according to the following equation:

$$\text{Gamma number} = \frac{\text{Abs}_{303 \text{ nm}} \cdot 546.48}{m_{\text{viscose}} \cdot c_{\text{cellulose}}}$$

where

Abs = absorbance at 303 nm,

m = mass of the viscose solution in grams, and

c = cellulose concentration of the viscose dope in %.

reduced filter-clogging number

The reduced filter-clogging number (Kr) is obtained by filtering the viscose solution and measuring the filtered amount and ball fall time. The filtered amount is measured after 20 min and again after 1 h. The falling ball time was measured using a 1/8-inch steel ball allowed to fall in a 200-mm-deep column filled with the viscose sample solution.

$$K_r = \frac{\sqrt{m_2/m_1}}{(t_2-t_1) \cdot n^{0.4}}$$

where

t₁ = 1200 s (20 min),

t₂ = 3600 s (60 min),

m₁ = total amount of viscose in the beaker after 20 min,

m₂ = total amount of viscose in the beaker after 60 min, and

n₀₄ = ball falling time in seconds.

ripening index

Ripening index was measured prior to spinning according to the Hottenroth method, where 20 g of viscose solution is diluted with 30 g of water, and the volume of 10 % ammonium chloride needed to cause gelling is noted.

GC-MS screening of organic compounds on textiles and pulp was performed by extracting the test material with methanol using a Dionex ASE 200, followed by detection with GC-MS (full scan mode). Process water from decolourisation was directly analysed on GC-MS (full scan mode). A NIST11 mass spectral database was used to, if possible, identify the detected compounds. The analysis is not quantitative.

Sample	Substance	CAS no	Regulated in REACH	Harmonized EU classification	Hazard statement ¹	Comment
Textile sample 4.1.7 with dye, wrinkle free and soft	1,3,4,6-tetramethyl-3a,6a-dihydroimidazo[4,5-d]imidazole-2,5-dione	10095-00-4	No	No	n.o. ²	-
Pulp from sample 4.1.7 with dye, wrinkle free and soft	No compounds detected					
Textile -samples with wrinkle free in task 4.1.6	2,2-oxybisethanol (Diethylene glycol)	111-46-6	No		Dominating compound	
Textile - all samples in task 4.1.6	n-1 hexadecanoic acid	57-10-3	No			
Textile - all samples in task 4.1.6	Octadecanoic acid	57-11-4	No			
Textile - all samples in task 4.1.6	0-Octadecenamide	301-02-0	No			
Textile - all samples in task 4.1.6	Gamma-Sitosterol	83-47-6	No			
Pulp after decolourisation from all samples in task 4.1.6	Hexadecanoic acid	57-10-3	No	No	n.o. ²	Used to make metallic palmitates and esters; in soaps and cosmetics; in lube oils; for waterproofing; in food-grade additives; as a non-drying oil
Pulp after decolourisation from all samples in task 4.1.6	13-Docosenamide, (Z)-5	112-84-	No	No	n.o. ²	Used as foam stabilizer, antiblock agent for polyethylenes and solvent for waxes and resins.



Mistra Future Fashion is a research program that focuses on how to turn today's fashion industry and consumer habits toward sustainable fashion and behavior. Guided by the principles of the circular economy model, the program operates cross disciplinary and involves 60+ partners from the fashion ecosystem. Its unique system perspective combines new methods for design, production, use and recycling with relevant aspects such as new business models, policies, consumer science, life-cycle-assessments, system analysis, chemistry, engineering etc.

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