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Chemical Recycling of Cotton Textile Waste and Integration into Dissolving Pulp for Viscose Production

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By promoting increased circularity through reuse, repurposing, and recycling, we can significantly reduce textile product carbon dioxide emissions by 33 %, concurrently mitigating air, land, and soil pollution associated with textile production, with chemical recycling playing a crucial role due to its ability to produce materials with comparable quality to virgin raw materials, minimizing the loss of a textiles original properties. This research investigated the chemical depolymerisation of cotton textiles, aiming to transform them into a material that closely resembles commercial dissolving pulp. The investigation focuses on comparing two pre-treatment methods, each aiming to optimise the depolymerisation process. The treated cotton material was then blended with conventional dissolving pulp, enabling the production of man-made cellulosic fibres (MMCFs) through the well-established viscose process. The successful implementation of this novel approach paves the way for incorporating recycled cotton into the viscose process and reduce dependence on virgin pulp, minimise environmental impact and promoting a more sustainable textile industry.

1. Introduction

A significant surge in fibre production has been experienced by the textile industry over the past two decades, primarily driven by a combination of growing global middle class, rising living standards, and population growth. This linear trend is projected to continue, with an estimated 30 % increase in production by 2030 (Opperskalski et al., 2020). This surge has led to an issue of underutilisation of clothing, with damaged or unwanted clothing frequently ending up in landfills or incinerators, resulting in detrimental effects on individuals and the environment (Reichart, 2019). Urgent action is required to transition the textile industry from a linear to a circular production model, necessitating comprehensive efforts to recover and recycle post-industrial and post-consumer waste streams (Tonsi, 2023). A circular economy for textiles, apparel, and fashion offers a solution to address this issue and reduce textile waste. The waste hierarchy model prioritizes reuse as the preferred waste management option, but when goods cannot be repurposed recycling becomes the next best alternative. While recycling consumes energy and chemicals, it remains a better option than incineration (Zamani et al., 2015). The textile industry faces a significant challenge of finding economically viable methods to transform used textiles into raw materials for new fiber production. Most existing textile recycling approaches often fail to produce commercially viable products. There is currently a substantial gap in understanding the effectiveness and feasibility of chemical recycling technologies for cotton textiles. Several studies have explored various techniques for cellulosic fiber generation from waste textiles. Liu et al. (2019) highlighted the efficacy of alkaline treatments Alkaline processes have demonstrated relatively high recovery rates, but the quality of the resultant fibers can vary significantly based on the parameters employed. Ionic liquid (IL) technology has also emerged as a promising technology, studies conducted by Haslinger et al. (2019) and Asaadi et al. (2016), showed that certain ILs effectively dissolve cotton fabrics, making it possible to regenerate high-purity cellulose. However, similar to alkaline treatments, the use of ionic liquids raises questions about their economic viability and scalability for industrial applications. Research has explored various additional methods, notably in the work by Bagenholm-Ruuth et al. (2024) using hydrated zinc chloride as a solvent, Määttänen et al. (2021) investigated pretreatment methods, and Boondaeng et al. (2023) involving enzymatic hydrolysis, revealing numerous innovative approaches to chemical recycling. All of which have shown potential in enhancing fiber properties.

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Despite the promising results there is still limited research on the optimization of pre-treatment mechanisms, which could significantly improve the purity and quality of the recycled fibers. This study investigated the chemical depolymerisation of cotton textiles, focusing on the effectiveness of pre-treatments in removing non-cellulosic materials and impurities that hinder this process. The pre-treatments aimed to enhance the structural properties of cotton fibers for better suitability in producing Man-Made Cellulosic Fibers (MMCF). The improvements in depolymerisation efficiency promote circular economy principles by allowing cotton waste to be repurposed for viscose production.

2. Methodology

The viscose process was used as the foundation for the experimental plan. To prepare the cellulosic product firstly the cotton waste material had to be treated before preparing the viscose dope for further use.

2.1 Materials

Three Pre consumer cotton fabrics were obtained (Cot 1, Cot 7 and Cot 33). Fourier Transform infrared (FTIR) spectroscopy was used to analyse and confirmed that the fabrics were all composed of 100 % cotton.

2.2 Pretreatments

Each sample was cut into 2-3 cm^2 pieces. These pieces were then shredded using a knife mill equipped with a 2 mm sieve. To ensure compatibility with dissolving pulp, two treatments were used to reduce the degree of polymerisation (DP) of the cotton textiles:

2.3 Sulphuric acid hydrolysis (treatment A)

Treatment A involved hydrolysis of 200 g sample in 5 % H_2SO_4 at 60 °C. A sample was taken at 10 min intervals between 10 and 120 min and viscosities measured. The viscosity-time curve was constructed using this data, allowing for the determination of the specific acid treatment time required to achieve the desired viscosity. An optimal viscosity for acid sulfite pulp was determined through aging kinetics, a process that assesses the rate of depolymerization. During this process, cellulose degrades due to oxidation from its reaction with atmospheric oxygen (Eriksson, 2015). This viscosity served as a reference point. The viscosity-time curves derived from the experimental data demonstrated the correlation between viscosity and the time necessary to reach that viscosity under particular treatment conditions.

2.4 Hypochlorite bleaching (treatment B)

Treatment B involved a bleaching, conducted at 60 °C. Samples were taken at 30 min intervals from 30 to 180 min, thereafter the fabrics were neutralised with demineralised water and dried at 60 °C. Similar to treatment A, viscosities were measured at each interval, and a viscosity-time curve was constructed.

2.5 Characterisation of treated and untreated samples

To understand the effects of the treatments on the cotton samples, the untreated and treated cotton samples were analysed and compared to dissolving pulp, using techniques or methods typically used to define pulp characteristics. These methods included viscosity measurement as per TAPPI test method T 230 om-08, determination of alkali solubility in accordance with TAPPI test method T235 cm-09, and evaluation of brightness and whiteness following ISO 2470-2 and ISO 11475:2004 methodologies, copper number was determined using TAPPI test method 430 cm:2009. Molecular weight distribution was analysed using size exclusion chromatography (SEC), and wide angle X-ray scattering and crystallinity index were assessed as per EN 13925-1…3, Non-destructive testing - X-ray diffraction of polycrystalline and amorphous materials. Finally metal content analysis was performed.

2.6 Viscose dope preparation

The treated samples were blended with dissolving wood pulp at 10:90 and 20:80 fabric to pulp ratios. The reactivity of the generated pulp from treated samples blended with dissolving pulp was evaluated using the Trieber test procedure (Treiber, 1962). The pulp reactivity is accessed using filtration value (Fw), which is the amount of viscose passing through a specific filter area at constant pressure until complete blockage, and the filter clogging value (Kw), which is the inverse of the filtration value (Treiber, 1962). This metric provides additional insight into the characteristics of the pulp, particularly in terms of its tendency to block or obstruct the flow of viscose through the filter system. By analysing both Fw and Kw, we can achieve a comprehensive understanding of the reactivity of the pulp derived from the treated samples and its interaction with the dissolving wood pulp, thus facilitating further developments in pulp processing and application in various industrial contexts.

3. Results and discussion

3.1 Pretreatments

Before reducing the degrees of polymerization (DPs) of the fabric samples align with the dissolving pulp, it was imperative to first establish the ageing times of both the untreated fabrics and their blend partner AS DWP. The ASDWP ageing time was 3 h and 30 min, at 240 mL/g. Utilizing this as a benchmark, the ideal viscosities for the two fabric samples were calculated with 410 mL/g for Cot 1 and 420 mL/g for Cot 7. The optimal treatment times were determined using DP reduction curve and found to be 53 min for Cot 1 and 50 min for Cot 7. A similar analysis was conducted for treatment B, resulting in an optimal time of 30 min for all fabrics.

3.2 Characterisation of Untreated and treated samples

Table 1 illustrates the differences in properties between treated and untreated fabrics, with an acid Sulphite dissolving wood pulp serving as a reference point.

Table 1: Characterisation of untreated and treated cotton fabrics

	ASDWP	UT Cot 1	TA Cot 1	TB Cot 1	UT Cot 7	TA Cot 7	TB Cot 7	UT Cot 33	TB Cot 33
Viscosity (mL/g)	502	1415	419	393	1313	442	389	1397	145
Brightness (%)	92.7	35.7	32.8	79.2	111.9	97.8	84.2	33.2	60.6
Berger whiteness (%)	83.6	14.7	9.3	72.6	CNBD	126	97.3	51.6	18.8
Yellowness (%)	3.4	26.2	25	3.2	CNBD	-16.1	-8.2	-31.3	24.9
Cu No.	1.27	0.17	1.00	1.36	0.21	2.10	9.70	0.162	4.95
S18 (%)	4.94	0.28	0.91	1.24	1.14	1.83	1.64	0.60	1.45
S10 (%)	10.50	1.02	2.06	2.95	0.8	4.78	3.18	1.24	18.21*
α-cellulose	92.3	99.4	98.5	97.9	99.0	96.7	97.6	99.1	90.2

*Testing method was adjusted. CNBD – could not be determined
ASDWP – Acid sulphite dissolving wood pulp, UT – Untreated, TA – Acid treatment, TB – Bleaching treatment

Prior to treatment, Cot 1, Cot 7, and Cot 33 had high viscosities (1313-1415 mL/g) and varying optical properties; Cot 1 and Cot 33 were low due to colour, while Cot 7 was high due to optical brighteners. Treatments A and B successfully reduced viscosities to the desired range for viscose production (400-600 mL/g), aligning with Määttänen et al. (2021), who also utilised pre-treatments to achieved desired viscosities. Treatment A reduced the brightness of Cot 7 by 14.1 %, while treatment B reduced it by 27.7 %, indicating effective removal of optical brighteners, bringing it closer to the desired brightness level typical for dissolving pulp i.e. higher than 90 %, Treatment B improved brightness for Cot 1 by 43.5 % and Cot 33 by 27.4 %. Määttänen et al. (2021) also discovered that colour removal was effective with pre-treatments, which enhanced the brightness of the initially dark cotton fabric to approximately 80 %. Additionally, treatments increased copper content and solubility, indicating some deterioration, but ultimately aligned treated fabrics' properties with dissolving wood pulp recorded by Lawson (2023).

The presence of transition metals in dissolving pulp can interfere with the viscose production process, resulting in inconsistencies and inferior fiber quality. Contamination from inorganic chemicals may lead to spinning blockages and impact the uniformity of the fibers. Table 2 displays various metals that were tested.

The treatments resulted in significant reductions of the metal content, suggesting that these treatments have the potential of removing impurities. The treated samples had similar metal content to dissolving pulp as demonstrated by Maattanen et al. (2021) and Lawson et al. (2023).

Figure 1 shows the molecular weight distribution (MWD). Uniform MWD of dissolving pulp is vital for staple reactions during viscose production, influencing the final products quality. Untreated cotton fabrics show a narrow MWD similar to PHK dissolving pulp but contain higher molecular weight components that may compromised viscose quality. After treatment, the MWD curve shifted slightly towards the left, suggesting a reduction in high molecular weight material and an increased proportion of short-chain cellulose, potentially improving viscose production. These findings align with previous research by Strunk (2012) and Palme (2015) reported a similar shift following acid hydrolysis. Data in Table 3 highlights effective depolymerisation, both the weight average (Mw) and number average (Mn) molecular mass decrease with pretreatment processes. When considering the recycling of textiles into regenerated cellulosic fibers, such as viscose, this lower average molecular mass may be advantageous for textiles, as it falls within the preferred range for viscose producers (Palme, 2015).

Table 2: Heavy and transition metals

Determinant (mg/kg.dry sample)	AS DWP	UT Cot 7	TB Cot 7	TA Cot 7	UT Cot 1	TB Cot 1	TA Cot 1	UT Cot 33
Calcium as Ca	19	478	342	22	406	238	36	514
Potassium as K	3	20	14	5	19	3	7	23
Magnesium as Mg	4	235	148	8	135	74	6	90
Sodium as Na	354.1	336.4	378.9	4.890	224.8	662.7	26.29	140.9
Chromium as Cr	0.060	2.290	0.310	0.210	9.530	0.570	5.770	2.290
Copper as Cu	0.120	2.140	7.070	0.280	5.530	4.960	0.520	0.380
Manganese as Mn	0.100	2.690	2.800	0.210	1.040	1.200	0.380	0.800
Nickel as Ni	0.020	<0.010	3.130	0.300	0.210	1.990	0.230	<0.010
Zinc as Zn	0.130	1.590	2.130	0.380	3.050	3.580	2.250	0.850

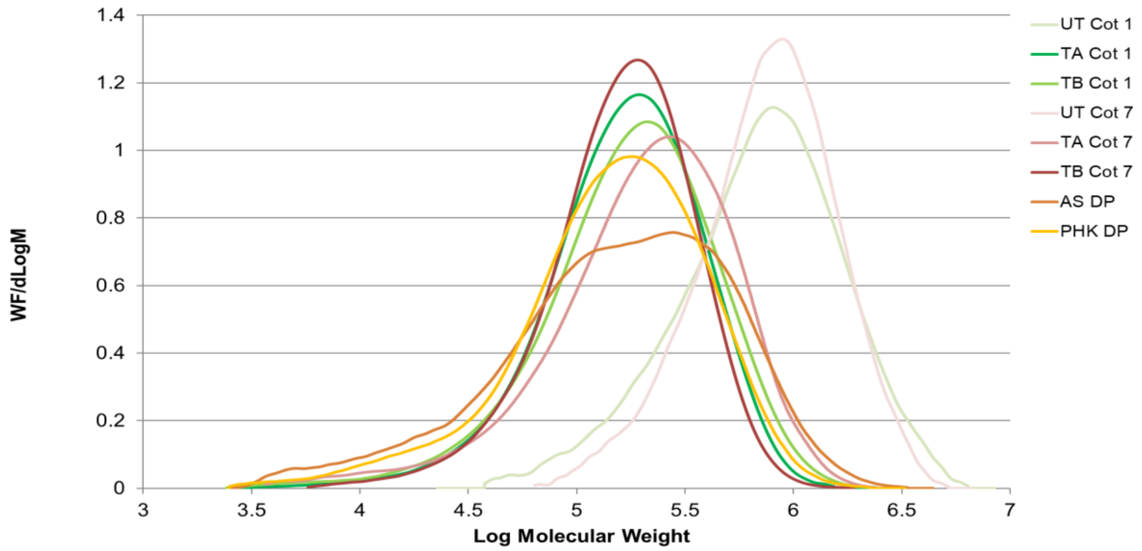

Figure 1: Molecular weight distribution

Table 3: Size exclusion chromatography (SEC) characterisation

	AS DWP	PHK pulp	UT Cot 1	UT Cot 7	TB Cot 1	TB Cot 7	TA Cot 1	TA Cot 7
Mn (g/mol)	113,041	219,498	448,006	566,816	98,290	111,574	111,574	102,622
Mw (g/mol)	204,865	412,185	975,078	961,810	250,971	207,952	222,112	296,521
PD (Mw/Mn)	2.8	3.0	2.2	1.7	2.6	1.9	2.1	2.9

3.3 Viscose dope Characterisation

Viscose dope can be evaluated through various techniques, with ball-fall and filterability (filter clogging number, Kw) being two of the most prevalent. These methods allow for the assessment and comparison of reactivity and quality (Karlsson, 2022). A lower Kw value reflects better filterability in viscose dope, indicating fewer unreacted alkali cellulose particles. Ball fall measurements assess viscosity by timing a ball's descent. Both Kw and ball fall measurements reveal the solution state influenced by alkali cellulose degree of polymerization (DP). The quality of 10 % hypo-treated viscose dope was good and similar to dissolving pulp, with variable but comparable ball fall times to Wedin, 2018 and Seppälä , 2023 also had variable ball falls ranging from 24-78s, indicating a potential variation in dope viscosity. DP values aligned with findings from Eriksson (2015), showing typical viscose fibers have a DP between 250 and 350. Additionally, 10 % acid-treated samples show higher Kw values than bleached samples, but perform better in ball fall and had better DPs.

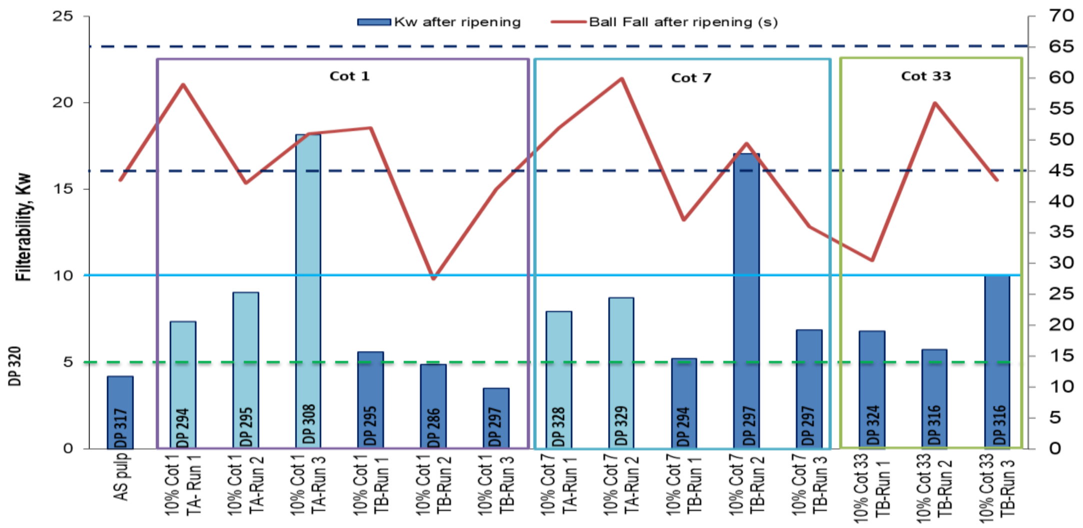

Figure 2: Viscose quality of the 10 % Hypo treated and Bleached blends

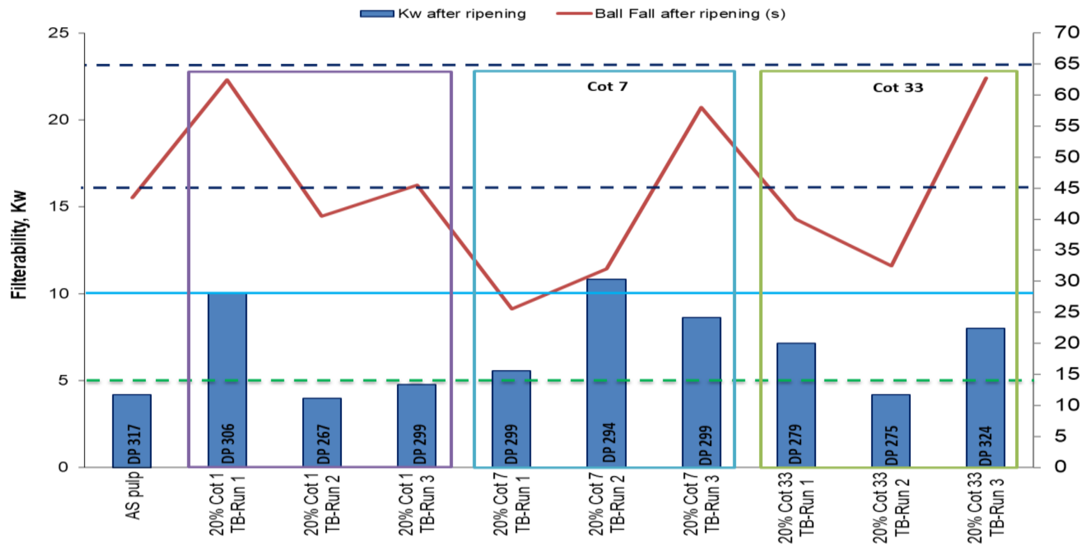

Figure 3: Viscose quality of the 20 % Bleached blends

Encouraged by the promising results from the 10% treatment B samples, the 20% treatment B blends were analysed to evaluated their performance. Figure 3 represents the 20 % fabric to pulp blends. There was a significant improvement in the viscose dope quality when the fabric content was increased to 20 %. Dope stability and filterability are increased by the greater intermolecular connections between the cellulose molecules in the pulp and fabric components. As a result, the viscose dope that is produced is more homogeneous and has improved spinnability properties, producing fibres that are stronger, longer-lasting, and of higher overall quality. Wedin (2018) concluded that treated cotton pulps exhibited improved filterability, characterized by a lower filter clogging value compared to untreated cotton pulp. This enhancement contributed to better viscose dope quality and improved spinnability of the resulting fibers.

4. Conclusions

The Untreated textiles failed to meet the specifications for dissolving pulp, exhibiting high viscosity levels (over 1000 mL/g), inconsistent brightness, either excessively high or low, and various impurities. These impurities include residual contaminants from dyes, as well as remnants of chemical finishes. The findings demonstrated that pre-treatment enhanced the characteristics of the cotton fibers, achieving a brightness increase of up to 43.5 %. The viscosity measured between 389 and 442 mL/g, aligning with the production standards for viscose. Additionally, they successfully depolymerized the cotton textiles, resulting in a degree of polymerization (DP) ranging from 267 to 324,that renders them suitable as a raw material for producing high-quality man-made cellulosic fibers. The favorable quality of the viscose dope, assessed through measures such as Kw, ball fall, and DP, highlighted the potential benefits of the pre-treatments in optimising the properties of the fabrics. This research significantly contributes to the development of sustainable and environmentally friendly methods for producing MMCFs, highlighting the potential of cotton textiles as a valuable resource in the textile industry.

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