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CLOSED-LOOP RECYCLING OF IONCELL® FIBRES

Master's Programme in Chemical, Biochemical and Materials Engineering

Major in Fibre and Polymer Engineering

Master's thesis for the degree of Master of Science in Technology submitted
for inspection, Espoo, 18 September 2022.

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Title of thesis Closed-loop recycling of Ioncell® fibres

Degree programme Master's Programme in Chemical, Biochemical and Materials Engineering

Major Fibre and Polymer Engineering

Thesis Supervisor Professor Dr. Herbert Sixta

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Date 18.9.2022

Number of pages 59

Language English

Abstract

In the aspiration of building a circular economy the textile industry needs to be remodelled. The current system is predominantly linear, starting with extracting natural resources and ending with large amounts of incinerated and landfilled textile waste. Textile recycling is not yet widely available, because none of the few existing technologies have been successful on the market.

The emerging Ioncell® technology produces high-performing regenerated cellulose textile fibres, originally from wood. Later, it has been shown that the same technology is suitable for cotton recycling. However, it has not yet been investigated whether the fibres produced with the Ioncell® process are recyclable by the use of the same technology. If Ioncell® intends to become an actor in a circular textile economy, the recyclability of the material is crucial.

In the course of this study, standard Ioncell® fibres were produced from wood pulp. The fibres were spun into yarn, knitted into fabric, and washed in a laundry machine fifty times to mimic household usage. The fabric was then chemically recycled. By measuring the fibre and yarn strength, chemical composition, and polymer structure, it was possible to conclude that the recycled Ioncell® fibres exhibited a minor decrease in quality. Nevertheless, they proved to be as suitable for yarn and textile production as the virgin Ioncell® fibres.

Keywords Regenerated fibres, Ioncell®, textile fibres, textile recycling, closed-loop recycling, circular economy, lyocell

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Titel Slutet återvinningskrets av Ioncell®-fibrer

Utbildningsprogrammet Magisterprogram i kemi-, bio- och materialteknik

Huvudämne Fiber- och polymerteknik

Övervakare Professor Dr. Herbert Sixta

Handledare / Granskare Dr. Marja Rissanen, Dr. Inge Schlapp-Hackl

Datum 18.9.2022

Sidantal 59

Språk Engelska

Sammandrag

För att kunna övergå till en cirkulärekonomi måste textilindustrin förändras från grunden. Det nuvarande systemet präglas av en linjär värdekedja, där råmaterial utvinns från naturen, förädlas till textilprodukter och efter användningen kasseras som avfall som antingen förbränns eller samlas på avstjälningsplatser. Tills vidare är textilåtervinning ovanligt eftersom ingen av de fåtalet existerande teknologierna har nått framgång på marknaden.

Med den nya Ioncell®-teknologin går det att producera regenererade textilfibrer av hög kvalitet ur dissolvingmassa. Senare har det upptäckts att även bomullstextiler kan användas som råvara i processen. Det har dock ännu inte forskats i huruvida Ioncell®-fibrer kan återvinnas med samma teknologi. Om Ioncell® skall bli en aktör i en cirkulär textilekonomi bör produkten gå att återvinna.

Den här undersökningen började med att det producerades konventionella Ioncell®-fibrer som spanns till garn och stickades till textiler. Textilerna tvättades i maskin femtio gånger för att efterlikna hushållsbruk, för att sedan malas och regenereras till nya fibrer. De nya fibrernas mekaniska, kemiska och strukturella egenskaper analyserades. Resultaten visade att Ioncell®-fibrer går att återvinna utan att kvaliteten försämras märkbart och utan att fibrerna förlorar sin duglighet att användas i textilproduktion.

Nyckelord Regeneratfibrer, Ioncell®, textilfibrer, textilåtervinning, slutet återvinningskretslopp, cirkulärekonomi, lyocell

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1 Introduction

Textiles and clothes are an integral part of all human societies, not only as a protection from cold weather and as cultural expression, but also in household applications and industrial settings. The production of textiles is an ever-growing industry. In 2021, it created a worldwide revenue of 1 636 billion US\$, which is expected to rise to 2 074 billion US\$ by 2025 (Statista, 2021a). In Europe in 2020, the textile and clothing sector generated a turnover of 162 billion euros (Euratex, 2020). The worldwide production of textile fibres is today three times as large as it was in 1975 (European Environmental Agency, 2019).

The growing trends are apparent in most of the Western countries. Clothes are produced for a low price and of low quality, which decreases their lifetime and encourages the consumer to frequently buy new clothes (Niinimäki et al., 2020; Ellen MacArthur Foundation, 2017). The EU member states are responsible for a large amount of the textile consumption. In 2017, the EU consumed 26 kg of textiles per capita (European Environmental Agency, 2019).

According to a mass balance analysis of the Finnish textile market in 2019, the total supply of textiles was 179 137 tonnes (Dahlbo et al., 2021). Most of it, 167 413 tonnes, were new and the rest were reused textiles. Finnish households purchased a total of 73 433 tonnes of textiles. When including consumer-to-consumer reusing, the average household textile usage was 11.3 kg of textiles. At the end of life, most of the clothes were incinerated for energy production. Only 4% were reused and 18% were recycled.

Niinimäki et al. (2020) investigated the textile value chain. It is challenging for the consumer to understand where the clothes originate from because the value chain is long, complicated, and intercontinental. This makes the environmental impact widespread and complex, and mostly invisible for the consumer. The major consequences are large amounts of waste and underutilized material, as well as a heavy societal and environmental impact deriving from the production of virgin

materials (Ellen MacArthur Foundation, 2017; European Environmental Agency, 2019).

The European Environmental Agency (2019) investigated the sources of environmental impact of the textile industry. The production of polyester, which accounts for 60% of the textile fibres today, requires more than 70 million barrels of oil per year. The cultivation of cotton, which is the second most abundant textile fibre on the market, requires large amounts of land and water. Only food production requires more land than cotton production in regards of consumption in the EU. However, only 7% of the cotton is cultivated in the EU. The remaining share is cultivated in other parts of the world.

To reduce the overconsumption, the European Parliament (2008) has defined a strategy. As a part of this strategy, all EU member states are expected to collect their textile waste separately from other waste by 2025 (European Commission). In Finland, the goal is to start the collection in 2023 (Suomen Tekstiili & Muoti). This opens a market niche for textile recycling.

The Ioncell® process has been proven suitable for the chemical recycling of cotton (Ojha, 2021; Asaadi et al., 2016; Haslinger et al., 2019a). This gives it potential to contribute to a circular textile economy. However, up to now the recycling of textiles produced with the Ioncell® process has not been implemented. The goal of this work is to close this research gap by answering two questions. Firstly: is it possible to recycle Ioncell® textiles by the means of the Ioncell® technology? Secondly: how does the chemically recycled material perform regarding polymer quality and mechanical strength, when compared to virgin Ioncell® fibres? To answer these questions, the chemical recycling of Ioncell® fibres was attempted. The quality of the resulting material was determined by testing and analysing the mechanical properties, intermolecular structure, polymer properties, and chemical composition.

This project contains a review of the current methods in textile recycling, experimental laboratory work, and a data analysis of the results.

2 Background

To understand this research, it is essential to understand some details about textile recycling and the Ioncell® technology.

2.1 Textile recycling

In this work, textile recycling refers to material recovery and recycling, which does not include energy recovery through incineration. Besides, textile reuse, which includes hand-me-down clothes or the second-hand market, is not considered.

The Ellen MacArthur Foundation (2017) distinguishes between five main categories of textile recycling. If the fabric retains its weave or knit and is cut or sewn into new products, it is called fabric recycling, or remanufacturing. This is applicable for pre-consumer cutting waste from apparel manufacturing, or from larger pieces of post-consumer waste. If the fabric is knitted, it can be unravelled in long segments of yarn for yarn recycling. The fabric and yarn can also be mechanically deconstructed for fibre recycling, where the fibres retain their structure. When the structure of fibres is lost, but the molecular structure retained, it is called polymer recycling. This can be achieved mechanically when plastic fibres are melted and re-extruded, or chemically when the fibres are dissolved with a chemical and regenerated. Finally, the polymers can be depolymerized for monomer recycling.

Positive as well as negative aspects can be attributed to each aforementioned approach (Ellen MacArthur Foundation 2017). Textile, yarn, and fibre recycling is possible without highly sophisticated technologies or chemicals. The waste can be sorted by colour or bleached and dyed. These recycling methods cause a decrease in material quality and durability. This is called downcycling (Sandin, 2017).

In contrast, polymer and monomer recycling can yield products with a quality close to that of virgin materials (Ellen MacArthur Foundation 2017). In the process of mechanical polymer recycling, dyes and other chemicals cannot be removed from the material, so it needs to be sorted by colour or bleached and dyed. Chemically recycled polymers and monomers can be separated from some contaminants and unwanted

chemicals during the processing.

Furthermore, recycling can be categorized by the different types of waste that may appear. Bhatia et al. (2014) distinguished between pre-consumer versus post-consumer waste as well as trashy, clean, and hard waste. Pre-consumer waste is generated before the textile reaches the consumer. This might be fibre processing waste, yarn spinning waste, textile weaving or knitting waste, or cutting waste from the sewing process. Post-consumer waste consists of used and discarded products. Trashy waste belongs to the pre-consumer category and can be collected from blow rooms, carding machines, and filters. It needs to be cleaned before reprocessing. This is not necessary for clean waste, which can be comber waste, combed sliver waste, ring spinning waste, and other uncontaminated waste streams from the production. Hard waste can be either pre- or post-consumer waste. It consists of yarns and textiles that need to be opened and processed before being turned into feedstock again.

2.1.1 Advantages and challenges

Zamani et al. (2014) investigated the textile waste management in Sweden, assuming the waste consists of 50% synthetic fibres (mainly polyester) and 50% cellulosic fibres (mainly cotton). The difference between the environmental impact of textile recycling and incineration for energy recovery is described as follows: in the case of incineration, the net global warming potential (GWP) was estimated as 0.23 tonne of CO₂-equivalent per tonnes of textiles. The recovered energy allowed to save 23 gigajoules per tonne of textile waste.

In the cases of recycling, the environmental impact depends on factors such as what material is replaced by the recycled product and which recycling category is applied. When the material is reused, as in the case of fabric recycling, the GWP is negative, compensating approximately 8 tonnes of CO₂-eq per tonne of textiles, and saving 164 GJ/tonne of energy (Zamani et al. 2014). Here, the estimated yield was 50% and the residues would be incinerated for energy production. However, the true yield of the

process is hard to estimate.

Zamani et al. (2014) also investigated the case of chemical recycling of polymers. Two different methods were applied: one for recovering cellulose with NMMO and one for recovering polyester through depolymerization. The results show that for cellulose recovery, the GWP is compensated by 5.5 tonnes CO₂-eq for every tonne of textiles and energy was saved to an extent of 46.5 GJ/tonne. For polyester recovery, it reached 0.9 tonnes CO₂-eq/tonne and 26 GJ/tonne of energy. The biggest advantage of these methods was the reduction of the demand for virgin cellulose and polyester.

To calculate the environmental impact of the transportation of textile waste, the recycling was assumed to be in Sweden (Zamani et al. 2014). The conclusion was that the longest possible route of transport would release merely 23.4 tonnes of CO₂-eq/tonne and consume 361 MJ/tonne of energy, which is significantly less than the compensated GWP and energy usage.

The results of the study described above are supported by Woolridge et al. (2005). To recycle one tonne of textile waste, 1 697 kWh is needed, whereas the production of one tonne of garments requires 66 648 kWh if it is cotton and 91 508 kWh if it is polyester. However, if the recycled textiles are spun into new yarns, knitted, dyed, finished, and made into new garments, another 43 248 kWh for cotton and 48 397 kWh for polyester is required (Woolridge et al., 2005).

Aside from the environmental advantages, there are also societal and economic reasons to favour a circular textile industry (Leal Filho, 2019). Sectors like transportation, collection, and sorting would offer opportunities for new employments and small businesses. Furthermore, assuming that waste is inexpensive, textile waste could serve as a cost-effective raw material for a multitude of products, spanning everything from new textiles to insulation materials and carpets.

Leal Filho (2019) investigated the challenges that currently prevent efficient textile recycling on a commercial scale.

A great challenge is the heterogenous nature of textiles. Blended fibres are hard to separate, and metal zippers, buttons, and other additions need to be removed prior to further processing. In addition, dyes and other chemicals are difficult to wash out. This raises the need for technologies that can separate and sort the different materials present in the textile waste to increase the possible amount of textile waste suitable for recycling.

With the current technologies, the processing of virgin materials is more economically profitable than the recycling of old textiles. Besides, the market for recycled textiles is not big enough to be incorporated on a larger scale. Because of this, stakeholders are unwilling to invest in the logistical and technological methods needed for textile recycling. Furthermore, the public awareness of the matter is limited, which is reflected in the lack of policies and standards from governing organs, as well as the lack of business actors in the field of textile recycling.

2.1.2 Market, industry, and directives

In recent years, effort has been made to investigate how a circular textile industry could be achieved in practice. This involves the rethinking of existing business models and the drafting of new legislations.

The challenges of the current textile recycling industry are described by the Biomimicry Institute (2020), not only from an environmental point of view, but also from a business perspective. They particularly criticize the globalized approach where the focus is on streamlined efficiency. Large companies dominate the market, making it hard for small local companies to succeed. This makes the whole industry less diverse, less adaptable to changes, and less resilient to external shocks. Another challenge is the fact that the textile recycling industry relies on the sorting of waste, on creating order from chaos, or, thermodynamically speaking, decreasing entropy. This requires energy and labour and is the opposite of how matter flows in nature: from order to chaos.

Textile end-of-life (EOL) management in the European Union has followed the

directive of waste management, directive 2008/98/EC, which was released in 2008.

The European Parliament (2008) has a plan for how waste ought to be managed in the member states. The primary approach is prevention. This means that the goal is to avoid the generation of waste altogether by extending the lifetime of products by designing them to last longer.

When waste is unavoidable, the European Parliament (2008) has set a hierarchy for how the waste should be managed. The next alternative – after prevention – is re-use, where something that has been discarded by one is repaired or refurbished and then used by another. Third in the hierarchy comes recycling, when the material is recovered from the discarded items. If that is impossible, the next option is to gain something else from the material, for example energy through incineration. Finally, the last option is to disposal at a safe location. This waste management hierarchy needs to be encouraged by local legislations and policies agreed upon in a transparent process.

When developing the framework, the EU Parliament (2008) urges its member states to take aspects like sustainability, technical and economic feasibility, human and environmental health, and societal impact into consideration. The responsibility to manage the waste would lie on the producer of the product or material.

Since the release of the directive 2008/98/EC, the EU Parliament has published a new framework to specifically target the textile industry (European Commission). It urges each member state to collect textile waste separately by the year 2025, along with an array of recommendations. These recommendations include developing and designing products in a way that make them suitable for a minimal-waste circular economy, finding applications for secondary raw materials, and embrace business models that offer products as services.

In a more recent strategy released by the European Commission (2022), it is further explained how countries can transition into a more sustainable textile economy. For example, by 2039, the EU market should only provide recyclable textiles with a long lifetime. This requires innovative product design as well as efficient fibre-to-fibre

recycling.

The waste management hierarchy is supported by Levänen et al. (2021), who investigated the environmental impact of each potential end-of-life scenario. Waste prevention has the lowest environmental impact, as it decreases the need to extract new raw materials and manufacture new products. Reusing has a lower energy consumption than recycling – regardless on the type of recycling – and is therefore preferred. In cases where the product is used as a service, such as lending or renting a pair of jeans, it was found that the environmental impact can be higher than in the case of conventional disposal through incineration for heat and energy recovery. This depends on whether the lifetime of the rented garment is at least twice as long as a privately owned garment, and whether the consumer uses low-carbon transportation to the renter.

2.1.3 Open-loop and closed-loop recycling

Payne (2015) stated that recycling can occur in a closed-loop or an open-loop system. Open-loop recycling (OLR) takes a discarded product and converts it into a new product, which is vastly different from the original. This reduces the need to extract new virgin materials, however, each cycle tends to bring the material closer to being discarded as waste (down-cycling). In the textile industry, OLR can be the process of converting plastic (PET) bottles into textile fibres, fabrics into low-value textiles, such as insulation materials.

Ajawa and Pavel (2005) describe how PET bottles are remanufactured into textiles. Post-consumer PET bottles are collected and cleaned. Their labels are removed and they are sorted based on colour. The bottles are then mechanically processed into PET flakes, which are liquefied and extruded. Thereby, the liquid is turned into polyester fibres through a spinneret. The high temperature required leads to thermal degradation shortening the polymer chains in the material. To increase the quality, the chains can be extended again through chemical treatments (polymerization). Apart from textile fibres, PET bottles can also be turned into films, sheeting, and

pipes.

The recycling of PET is well-established in the textile industry, especially as a blend with virgin PET (Payne, 2015).

Another example of OLR in the textile industry is the down-cycling of textiles. This method is described by Gulich (2006). First, any non-textile parts such as zippers and buttons are removed. The textile waste, which can be either pre- or post-consumer, is then mechanically torn and shredded to pieces by high-speed rotating pins and blades. The resulting fibres are grey, unless the waste is sorted by colour. The fibres can be used to produce industrial yarns and textiles, which do not have high standards of quality. The material is cheap and the process of breaking down the fabric has a relatively low energy consumption, especially compared to fibre manufacturing and re-granulation of plastics. Properties such as non-flammability and fibre strength remain from the original material, but the fibre length is decreased during the treatment. Furthermore, the fibres contain unopened pieces of thread and fabric.

If the textiles or fibres are unsuitable for yarn production, they can be recycled into non-woven textiles, such as insulation and carpet underlay (Payne, 2015). These products are, however, in general grey to the colour as a result of the heterogenous feedstock.

Sharma and Goel (2017) investigated how recycled fibres could be remanufactured into a felt-like material. Blends made of different amounts of polyester and cotton waste fibres were prepared and carded into an even web. Several webs were layered on top of each other and needle-punched into a compact mat.

Payne (2015) studied various benefits with OLR in the textile industry. The energy use is lower compared to the production of virgin materials, and the waste streams are smaller, as well as the carbon footprint. However, there are great differences between the benefits of the recycling of different materials due to the high diversity of textiles.

In a closed loop recycling (CLR) system, the recycled material is used as originally intended (Payne, 2015). For example, discarded garments and household textiles are recycled into fibres, which in turn are manufactured into new garments and household textiles. This case is an example of cradle-to-cradle CLR. Other approaches include recycling of pre-consumer waste or upcycling of post-consumer waste.

The ideal cradle-to-cradle (C2C) system, as described by McDonough et al. (2003), is built on three tenets. The first one states that waste equals food, based on the fact that waste does not occur in nature. Dry leaves fall to the ground, decompose, and become nutrients for other organisms. Engineers are encouraged to prioritize materials that safely decompose in nature or can be remanufactured by humans after disposal. The second tenet urges to use current solar income, as solar energy powers photosynthesizing plants. This includes other energy sources such as wind power, which comes from thermal flows caused by the sun. Engineers are encouraged to harness energy from renewable sources rather than depleting ones. The last tenet is a prompt to celebrate diversity, similar to how healthy ecosystems have a diverse community of living organisms, each developed to survive its surroundings in a unique way. For engineers, this means that the most sustainable solutions are adapted to fit the needs and opportunities of the local community and can therefore vary from place to place.

In C2C recycling, the discarded material can be divided into a technical waste stream and a biological waste stream (Payne, 2015). In the biological waste stream the material is safely biologically decomposed into nutrients and is then returned back to the soil. Not all biodegradable fibres are suitable for this, as some textiles are treated with hazardous chemicals that harm the environment when released into the soil. In the technical waste stream, the materials are remanufactured by industrial means. Polymer-based synthetic fibres, such as PET or polyamide, belong to this stream. Issues arise when two different materials, which would go to separate waste streams, are combined. These hybrid materials are not suitable for either waste stream without being separated first. Such problematic blends are currently made from cotton and polyester, viscose and polyester, or cotton and elastane. Similarly,

buttons, linings, zippers, and labels are difficult to remove by other means than manually by hand, which is a labour-intensive process.

The Biomimicry Institute (2020) suggests that the biological and technological streams could and should be merged into a single system. In summary, the technical cycle for fibres is not closed-loop, since even the most durable plastics only last for a few cycles. A portion of the material is released into the environment in each stage of processing and the remaining material will eventually end up unusable and discarded. Any material produced will eventually end up in the biological waste stream, whether or not it is intended to be biologically decomposed. The only truly closed loop is the biosphere, where material endlessly circulates. In this model, petroleum-based materials cannot count as closed-loop materials, as the carbon extracted from crude oil or gas from the earth will stay and accumulate in the biosphere. In other words, only cellulose-based fibres and other fibres from natural origin can truly be recycled indefinitely.

2.1.4 Mechanical recycling

Gulich (2006) described a type of mechanical recycling, similar to the one mentioned above. The textile waste is mechanically cut and shredded into fibres that can be spun into new yarn. A similar method is called Recover cotton (Esteve-Turrillas and Guardia, 2017).

Recover cotton is a method developed by a family business called Hilaturas Ferre, which was described by Esteve-Turrillas and Guardia (2017). Pre-consumer waste from different textile plants and small amounts of post-consumer waste is collected in different countries. The waste is sorted based on colour and quality, cut, shredded, and gently opened into fibres. The recovered fibres have a length ranging from 10 to 15 millimetres. They are spun into new yarns and potentially blended with other fibres. The colour of the yarn can be adjusted according to one or more of the colours of the recycled feedstock. This replaces the step of dyeing in conventional textile production, which reduces the water and chemical usage.

Esteve-Turrillas and Guardia (2017) performed a life cycle analysis (LCA) of textiles produced with the Recover technology and compared it to the conventional and organic cotton-based textile production. They compared, among other things, the global warming potential (GWP), acidification potential (AP), eutrophication potential (EP), and water use (WU). The cotton cultivation, ginning, and dyeing processes were compared to the Recover technology of cutting and shredding. The latter exhibited a GWP less than a tenth of the CO₂ equivalence of virgin cotton. The AP decreased to less than one hundredth. The EP decreased even more and the water usage in these steps was nearly zero for the Recover technology.

Even waste that consists of pure cotton is far from homogenous, as different pieces of textiles are made from fibres of varying quality. Ütebay et al. (2019) explain how the quality of recycled cotton can vary. Every process of mechanical recycling results in fibres that are shorter than the initial fibres. They are also more compact and less opened, resulting in coarse yarns unless blended with virgin cotton. An increased number of shredding passages that the fabric goes through can counteract the coarseness, as the fibres are more thoroughly opened and contain less fabric and yarn pieces. It also results in a decreased amount of short fibres. In sum, the recycled cotton fibres are 25-35% shorter than the virgin cotton. Furthermore, they found that dyes decrease the mechanical strength of the fibres.

Halimi et al. (2006) investigated how the quality of yarn made from mechanically recycled cotton is affected by spinning parameters and blending. The quality can be improved if the recycled fibres are blended with virgin fibres. The material can contain 15-25% waste cotton without a decrease in yarn and fabric quality, in comparison with 100% virgin material.

2.1.5 Chemical recycling

Chemical recycling of textiles is slowly gaining visibility on the market. Although textiles can be made from various materials, this chapter will focus on the chemical recycling of cellulose-based textile fibres. Various companies have developed

methods to chemically recover the polymers from unusable fibres. The first step is to turn the textiles back into feedstock.

Re:newcell is a biomaterials company located in Sweden (Re:newcell AB, 2019a). The company focuses on turning used textiles into Circulose® pulp (Re:newcell AB, 2019b). First, large quantities of post-consumer textile waste are collected, mainly by collaborating with fashion companies. The textiles need to primarily consist of cotton or other cellulosic materials. Non-textile parts such as buttons and zippers are removed from the shredded textiles, which then are chemically de-coloured and turned into a slurry. Polyesters and other contaminants are separated, so the remaining material is only cellulose. The slurry is dried and turned into sheets of pulp. This pulp can be sold to manufacturers that turn them into man-made cellulosic fibres, yarns, fabrics, and new clothes.

Terho (2020) explains how Infinited Fiber Company utilizes the cellulose carbamate method. The textile waste needs to contain at least 80% cotton and be cleaned from non-textile parts such as zippers and buttons.

To remove silicates, the cotton waste is ground finely and washed in an alkaline bath (Määttä et al. 2021). Next, the intrinsic viscosity is adjusted with ozone and hydrogen peroxide, targeting on 400-500 ml/g for the cellulose carbamate process. These treatments remove colours from the material. To remove metals, the material is washed in sulfuric acid.

Next, an alkaline treatment is initiated to remove the acid and to depolymerize the polyesters Terho (2020). After being thoroughly washed, the waste mainly consists of cellulose, and is ready to be treated for solubilization. Here, the reagents are water and urea, which splits into carbamate and ammonia. The ammonia dissolves in the water. The resulting cellulose carbamate powder is chemically stable and can be stored or transported safely.

After this stage, Terho (2020) explains, the process is similar to the viscose process.

The cellulose carbamate is mixed with dilute sodium hydroxide and stirred until it dissolves. The mixture is then filtrated to remove undissolved matter. After that, the alkaline solution is extruded through a spinneret into an acidic bath in a wet spinning process. Via neutralization, the cellulose is regenerated and turned into new fibres. To align the polymers within the fibres and make them stronger, they are stretched by rotating rolls to obtain a linear density of approximately 1.3 dtex. This gives them a strength of 26 cN/tex. The fibres are finally washed and cut into desirable length.

The cellulose carbamate fibres spun by Määttänen et al. (2021) reached a tenacity of 0.7-1.0 cN/tex

Paunonen et al. (2019) investigated textile recycling by means of the cellulose carbamate technology from an environmental, societal, and economical perspective. It was found that urea as a processing chemical has several strengths. For one, it is easily available as it is widely used in synthetic fertilizers, which have a smaller environmental impact than natural fertilizer. During the process, ammonia is formed, which reacts with acid in the spinning bath. This results in ammonium sulfate, which also is a fertilizer. However, the process is strongly alkaline and consumes sodium hydroxide. Sodium sulfite is formed, which can be recovered and reused. Another strength is that the technology can be implemented in existing viscose factories, avoiding the costs and resources for constructing new factories. Nonetheless, the process is still in the development stage and not yet commercially viable.

The product has various advantages, according to Paunonen et al. (2019). The Fabrics produced with cellulose carbamate fibres feel more like cotton to the touch than viscose and lyocell fabrics do. Dyes are easily taken up by the material, reducing the need for colorants, and thus reducing the consumption of water and toxic chemicals.

Haule et al. (2015) explained how cotton can be recycled through the lyocell process. The lyocell process relies on the ability of N-methylmorpholine N-oxide (NMMO) to completely dissolve cellulose without degrading it. The solution is then extruded through a spinneret, into an airgap, and into a non-solvent (water), which dissolves the NMMO. The result is pure cellulose fibres with a high degree of orientation and

elasticity.

Instead of wood pulp, Haule et al. (2015) used cotton waste from different sources. The cotton was cut into pieces, which were mixed in water and roll-beaten to a pulp. The pulp was air-dried, dissolved in an NMMO-solution, and spun into new fibres.

Haule et al. (2015) investigated the fibre properties and found out that day-to-day use and the presence of finishing chemicals had a negative impact on fibre strength. Nonetheless, the fibres made of cotton waste were stronger than virgin wood-based lyocell fibres. The virgin fibres had a tenacity of 34.7 cN/tex, while the recycled cotton fibres had 48.7 cN/tex, 42.5 cN/tex, and 37.2 cN/tex.

2.2 Ioncell® in chemical recycling

In the course of this thesis, sample textiles were produced and chemically recycled by means of the Ioncell® process. In this process, cellulose is dissolved in an ionic liquid and regenerated in a water bath (Sixta et al., 2015). This chapter presents earlier research conducted on the Ioncell® technology.

2.2.1 The Ioncell® process

The Ioncell® process was developed as a technology to produce cellulose-based man-made fibres. Michud et al. (2016) and Sixta et al. (2015) explained how it works. The raw material, which is cellulose pulp, is dissolved in an ionic liquid, for instance, 1,5-diazabicyclo[4.3.0]non-5-enium acetate ([DBNH][OAc]). Mixtures of IL and 13 w% cellulose were produced by kneading them into homogenous dopes. The dopes were then filtrated to remove the remaining undissolved particles. They were chemically stable and could be stored for a few days at refrigerator temperature before the dry-jet wet spinning.

By dry-jet wet spinning, the dopes were turned into fibres (Michud et al., 2016; Sixta et al., 2015). One dope at a time was loaded into an extrusion cylinder and extruded at an elevated temperature through a multi-hole spinneret, which turned the dope into filaments. The filaments were stretched in an air gap before being immersed a

coagulation bath where the cellulose was regenerated. The fibres were collected, cut into desired lengths, washed, and dried.

The fibres made by Michud et al. (2016) were then spun into yarns, doubled into two-ply yarns, twisted, and knitted or woven into different garments and accessories.

Michud et al. (2016) tested the tensile properties of the yarns and fibres, as well as the polymer orientation of the Ioncell® fibres and commercial viscose fibres for comparison. The Ioncell fibres outperformed the viscose fibres with dry tensile strengths between 45.6 cN/tex and 48.3 cN/tex, compared to the 20.7 cN/tex for viscose. Furthermore, their strengths decreased less than 5 cN/tex under wet conditions whereas viscose lost more than 10 cN/tex. Viscose had a higher elongation that decreased from over 19% to under 17% in wet conditions while Ioncell® fibres had an increase in elongation - from less than 10% to over 11%. Regarding polymer orientation, which was measured with birefringence, the Ioncell® fibres exhibited a total orientation of over 0.650, and the viscose fibres had a total of 0.425.

Sixta et al. (2015) reached tensile properties in the same range as Michud and found an increase in strength when the cellulose content was increased. The results also showed an increased orientation, which was assumed to be part of the reason behind the higher strength.

The tensile properties of Ioncell® fibres were investigated by Elsayed et al. (2021), too. The tenacity of the fibres was 49.5 cN/tex and the elongation was 12.2%. Apart from DBN, dopes were produced with ionic liquids based on other superbases and the tensile properties of the fibres were investigated. N-methylmorpholine N-oxide (NMMO), which is used in the commercialized lyocell process, gave fibres with a tenacity of 46.5 cN/tex and an elongation of 12.4% at break. Fibres made from a dope based on 7-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-enium acetate ([mTBDH][OAc]) reached tenacities of 49.9 cN/tex and elongations of approximately 9.8%. Finally, 1,8-diazabicyclo[5.4.0]undec-7-enium acetate ([DBU][OAc]) resulted in fibres with tenacity values around 56.3 cN/tex and elongations of 9.8%.

2.2.2 Ionic liquid

Ionic liquids (ILs) are a group of organic salts with melting points below 100 °C (Zhu et al. 2006). Some ILs have the property of dissolving cellulose without altering it chemically. They are chemically stable, non-flammable, and have an almost non-existent vapour pressure, which makes them compatible with work safety.

There are three ionic liquids used in the Ioncell[®] process (Elsayed et al., 2021). They are called 7-methyl-1,5,7-triazabicyclo[4.4.0]dec-5-enium acetate ([mTBDH][OAc]), 1,8-diazabicyclo[5.4.0]undec-7-enium acetate ([DBU][OAc]), and 1,5-diazabicyclo[4.3.0]non-5-enium acetate ([DBNH][OAc]), which is described in more detail below.

The ionic liquid [DBNH][OAc] is made by mixing equimolar amounts of acetic acid and 1,5-diazabicyclo[4.3.0]non-5-ene (DBN) at an elevated temperature (Michud et al., 2016; Sixta et al., 2015). This results in a neutral ionic liquid called [DBNH][OAc], as illustrated in figure 1.

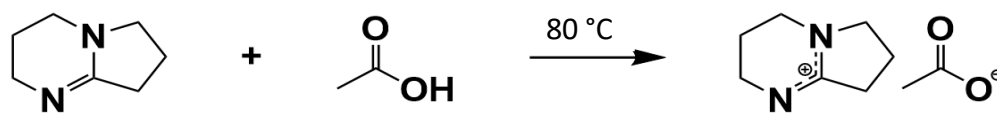


Fig. 1. An illustration of the reaction of DBN and acetic acid resulting in [DBNH][OAc].

Parvinen et al. (2015) studied the recyclability of [DBNH][OAc]. For the ionic liquid to be economically viable on the market, it needs to be recyclable. For the study, the pulp was dissolved in IL, and then regenerated in a water bath of similar temperature as the coagulation bath during dry-jet wet spinning. When the pulp was regenerated, it was removed by filtration and the water/IL mixture was recovered. The solution was concentrated by removing water with a rotary evaporator, then the remaining IL was dried in a high vacuum rotary evaporator. On average over all the cycles, 97.5% of the process solvent was recoverable, and most of the losses were claimed to be caused by the simple laboratory equipment. Therefore, the yield will be closer to 100% if the process is optimized. Moreover, while most of the process solvent was

from the desired ionic liquid, it also contained hydrolysis products, which were formed during the processing. In fact, the estimated hydrolysis rate was 6.0-13.6 mol% for each cycle. The increasing amount of hydrolysis products affected the dissolvability of pulp.

The quality of the pulp was also investigated (Parvinen et al., 2015). It was found that the molecular composition of the cellulose stayed roughly the same, meaning that the polymer did not degrade when pulp was dissolved in [DBNH][OAc] and regenerated. Furthermore, the molecular weight of the polymers did not decrease. However, the colour and the texture of the pulp were affected by each cycle of the IL. It went from white and cotton-like to yellowish and rougher.

For the Ioncell® process to be commercially viable, Elsayed et al. (2020) investigated the recyclability of [mTBDH][OAc] and [DBNH][OAc]. The [DBNH][OAc] was recycled once and the [mTBDH][OAc] four times. After one cycle, the [DBNH][OAc] had formed by-products via hydrolysis. The recycled IL could no longer produce any spinnable dope with the same cellulose content as before (13 w%) due to its poor quality. However, the [mTBDH][OAc] was still producing spinnable dope after five cycles. Since it was thermally more stable, it resulted in a lower degree of hydrolysis of the mTBD.

2.2.3 Cotton recycling

The Ioncell® technology was developed using pulp as a raw material, but Ojha (2021), Asaadi et al. (2016), and Haslinger et al. (2019a) showed successfully that it also is possible to use cotton as a raw material. Cotton was chosen to be investigated as a raw material because it is the most abundant natural textile fibre industry (Statista, 2021b). It also has a particularly high cellulose content. The environmental impact of the cultivation of cotton originates from the consumption of water, pesticides, and arable land that could be used for the cultivation of food crops (Mather, Wardman, 2015).

Asaadi et al. (2016) investigated the possibility to produce textile fibres from end-of-

life (post-consumer) cotton bedsheets from hospitals. The waste was divided into three batches, of which the intrinsic viscosity was measured. It ranged from 452 ml/g to 750 ml/g. Some of the cotton waste was degraded by acid and enzyme hydrolysis in order to investigate how the intrinsic viscosity (and thereby also the degree of polymerization) affects the spinnability of the material. The acid treatment was performed at 90 °C with 0.3 M sulfuric acid. The treatment duration varied, depending on the target intrinsic viscosity.

The treatment reduced the intrinsic viscosity of the cellulose. The lowest obtained viscosity was 315 ml/g. Besides, the intrinsic viscosity was adjusted by blending cotton with wood pulp. Some of the cotton was blended with softwood pulp with viscosities of 268 ml/g and 540 ml/g, resulting in blends of 393 ml/g, 450 ml/g, and 468 ml/g. The reference sample was virgin birch wood pulp with a viscosity of 476 ml/g.

During the spinning trials, Asaadi et al. (2016) found that the cotton samples with the highest viscosities – 587 ml/g and 750 ml/g – were not spinnable. The three samples with the lowest viscosities – below 365 ml/g – also proved unsuitable for spinning. The cotton samples with the mid-range viscosities of 438 ml/g and 452 ml/g were possible to spin into fibres, which showed tenacities of up to 58.4 cN/tex, compared to 48.1 cN/tex for the reference pulp-based fibres with similar DR. Two of the three blends – the ones with the highest and lowest viscosities – were spinnable, yielding fibres with tenacities of 50.7 cN/tex and 52.0 cN/tex for similar DR.

Ojha (2021) used industrial cotton waste as raw material. The waste consisted of 100% cotton towel rolls provided by Lindström Oy. Some of the towels were unused (pre-consumer), while others had been used (post-consumer). Some of the post-consumer towels had been dyed blue to conceal stains, which had been accumulated during their lifetime. The viscosity of the cotton samples was higher than of wood pulp: over 1000 ml/g for the pre-consumer white cotton, around 1000 ml/g for the white post-consumer cotton, and over 750 ml/g for the dyed postconsumer cotton. Each material underwent an acid washing and acid hydrolysis to remove inorganic

impurities and to decrease the viscosity to 400-500 ml/g.

In this study, Ojha (2021) compared the spinnability and quality of the different cotton samples to the standard pulp. The results showed that a too high intrinsic viscosity decreases the spinnability, while acid-treated cotton is spinnable even at high DRs (up to 18). The recycled cotton fibres exhibited tenacities as high as 60 cN/tex, while standard pulp-based fibres of the same DR 11-12 showed tenacities below 54 cN/tex.

Haslinger et al. (2019a) investigated the recycling of cotton/polyester blends. The starting material was shredded textile waste consisting of 50 w% cotton and 50 w% polyester. To remove colours and impurities, it was pre-treated with ozone, alkaline washed, and bleached. By this treatment the intrinsic viscosity of material was adjusted. It was then dissolved in [DBNH][OAc] with a slight surplus of acetic acid to obtain a dope of 6.5 w% cellulose. The cellulose dissolved and the solid polyester was removed by filtration. Then, more textile waste was added to the dope, resulting in a cellulose content of 10.5 w%, and filtrated again. The solid residue was purified from cellulose by dissolving it in ionic liquid and filtrating it once more. The cellulose-containing dope was turned into fibres through dry-jet wet spinning. The resulting fibres showed linear densities of 0.75-2.95 dtex, and tenacities of 27-48 cN/tex.

3 Experimental

3.1 Goal

The goal of this laboratory work was to illustrate the chemical recyclability of laboratory produced Ioncell® fibres. Fibres were created out of pulp, yarn was spun, fabrics were knitted, and a consumer lifetime was mimicked by laundering the fabric 50 times. The washed textiles were then ground into new feedstock and turned into new fibres with the Ioncell® technology. Throughout the process, several samples were collected to analyse the mechanical, chemical, and intermolecular properties, to create an overview of how the polymer quality changed throughout the process.

3.2 Materials

The virgin raw material used in this work was kraft-bleached pulp (birch, Enocell 3 and 4) provided by Stora Enso. The ILs were produced from 1,5-Diazobicyclo(4.3.0)non-5-ene (Fluorochem, DBN, CAS: 3001-72-7, $C_7H_{12}N_2$, $M = 124.18 \text{ g/mol}$) and acetic acid (Supelco, 100%, CAS: 64-19-7, $C_2H_4O_2$, $M = 60.052 \text{ g/mol}$).

For a blend, post-consumer 100% cotton waste (CO roll towels) was provided by Lindström Oy.

The fibres were spin finished with a mixture prepared from Archroma Leomin PN pa (20%) and Archroma Afilan CVS liq (80%). In accordance to EN ISO 6330:2012 (SFS), the laundry cycles were performed using the standard “detergent 1” (WOB AATCC), American Association of Textile Chemist and Colorist (AATCC) 1993 without optical brightener.

The intrinsic viscosity was determined by means of a cupriethylenediamine (CED, CAS: 13426-91-0, $C_2H_6CuN_2$, $M = 121.63 \text{ g/mol}$) solution purchased from OY FF-Chemicals AB, as directed by the standard SCAN-CM 15:88, revised 1988 by the Scandinavian Pulp and Board Testing Committee. For the GPC measurements, the

solvent N,N-Dimethylacetamide (DMAc, CAS: 127-19-5, C₄H₉NO, M=87.122g/mol) from VWR chemicals was used.

3.3 Sample production

The production of virgin Ioncell® textile “waste”, or cycle 0, was conducted in analogy to the methods described by Michud et al. (2016) and Sixta et al. (2015). Ground Enocell pulp was dissolved in ionic liquid and turned into fibres by dry-jet wet spinning. Then, the fibres were spun into yarn and further knitted into sample fabrics, which were washed in a laundry machine 50 times to mimic life a cycle. For cycle 1, the fabrics were ground into feedstock and turned into new fibres. In the course of his BSc studies, Twaroch (2022) continued with cycle 1, using all the leftover feedstock for fibre production, and then spun the cycle 1 fibres into yarns, knitted them into fabrics, and laundered the fabrics. Finally, a small sample of Cycle 2 fibres was produced using cycle 1 fabric as feedstock. A scheme of the research project is presented in figure 2.

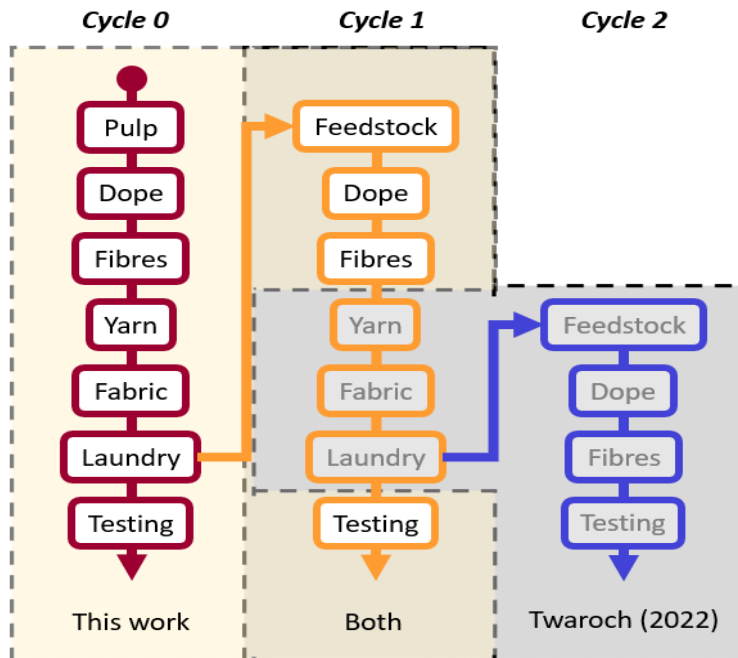


Figure 2. A scheme of the Ioncell® recycling project, where “this work” was performed by Sederholm, “Both” was performed by Sederholm and Twaroch, and “Twaroch (2022)” was performed by Twaroch.

3.3.1 Material preparation

The virgin Enocell pulp was ground to a powder (Sixta et al., 2015) with a Fritsch pulp mill with a 0.5 mm mesh and collected. Small fibre and fabric samples were ground with a Wiley Mini Mill 475-A and big fabric samples with a Wiley Mill M02 (Arthur H. Thomas Company).

The dry matter content was measured as described by the ISO 638:2008 standard. Three samples of roughly 100 mg were weighed on a RADWAG Wage Elektroniczne weighing balance, were dried in a Memmert oven overnight at 105 °C and cooled to room temperature in a desiccator. The dry samples were then weighed again, and the dry matter content (DMC) was calculated according to formula 1. This method was used throughout the project to determine the DMC and cellulose content.

$$\text{DMC} = \frac{m_{\text{dry}}}{m_{\text{wet}}} \cdot 100\% \quad (1)$$

DMC	dry matter content, in %
m_{wet}	mass of the sample before drying, in g
m_{dry}	mass of the sample after drying, in g

Additionally, to increase the intrinsic viscosity of the recycled Ioncell® fibres (cycle 1), a blend with post-consumer white cotton roll towels was produced.

Approximately 50 g of cotton towel rolls was ground into a fine powder. It was then added to 2.5 litres of 0.010 M sulfuric acid and heated to 40 °C. The mixture was kept at 40 °C and gently stirred for 40 minutes.

After that, it was rinsed over a filter and neutralized in a small bath with sodium hydroxide (1M). It was then filtrated again, centrifuged, and dried in an oven at 105 °C for four hours.

An overview of the materials prepared for analysis is provided in table 1.

Table 1. List of all samples ground as explained above.

Abbreviation	Description	Device
Pulp_c0	Enocell pulp, cycle 0	Fritsch pulp mill
Fib_c0	Standard Ioncell® fibres, cycle 0	Wiley Mini Mill
Fab_c0 – 0w	Standard Ioncell® fabric, cycle 0, before laundry	Wiley Mini Mill
Fab_c0 – 10/20/30/40w	Standard Ioncell® fabric, cycle 0, after 10/20/30/40 laundry cycles	Wiley Mini Mill
Fab_c0 – 50w	Standard Ioncell® fabric, cycle 0, after 50 laundry cycles (used as raw material for cycle 1)	Wiley Mini Mill, Wiley Mill
Fib_c1	Ioncell® fibres, cycle 1, made of Fab_c0 – 50w	Wiley Mini Mill
Fib_c1 ... _blend	Ioncell® fibres, cycle 1, made of Fab_c0 – 50w and cotton	Wiley Mini Mill
-	Cotton towels	Wiley Mini Mill

3.3.2 Ionic liquid

As described by Sixta et al. (2015), the solvent was prepared in a 6-litre reactor that was kept at 70 °C. At first, the DBN was added. Based on the amount of the DBN, an equal amount of acetic acid was added (formula 2) at a rate of around 6 ml/min. The amount of acetic acid slightly exceeded the amount of DBN to ensure that all DBN was consumed. When all the acetic acid had been added, the mixture was stirred for 30-60 minutes.

$$m_{\text{AcOH}} = \frac{M_{\text{AcOH}}}{M_{\text{DBN}}} \cdot m_{\text{DBN}} \quad (2)$$

m_{AcOH} and m_{DBN} mass of acetic acid and DBN, in g
 M_{AcOH} and M_{DBN} are the molar mass of acetic acid and DBN, in g/mol

The resulting ionic liquid (IL) was then collected in bottles and stored at room temperature with air-tight caps.

3.3.3 Dope

The dopes were prepared with a similar method as described by Michud et al. (2016) and Sixta et al. (2015). Prior to the dope production, the IL was melted at 80 °C. At first, IL was added to a kneading device. After that, pulp was added to reach a dry cellulose content of 13-15 w%. The amount of pulp to add was determined by formula 3.

$$m_{pulp} = \frac{m_{IL} \cdot c}{(100\% - c) \cdot DMC} \cdot 100\% \quad (3)$$

m_{pulp}	amount of pulp to add, in g
m_{IL}	amount of ionic liquid, in g
c	target cellulose content, in %
DMC	dry matter content as calculated above, in %

The pulp and IL were mixed at 80 °C using 30 rpm, and at reduced pressure (20-50 mbar). Every 30 min the kneader was opened to monitor the pulp dissolution under a Zeiss Microscope with a Linkam heating plate heated to 80 °C and a Moticam 5.0 MP camera. It took 1.5-3 hours for the pulp to dissolve. When the pulps had dissolved, the dopes were filtered with a hydraulic press through a multi-layered GDK Ymax2 metal filter at 80 °C. A small sample was taken for rheology measurement. The rest was stored in a refrigerated room until the fibre spinning.

Before preparing dopes for cycle 1, the dissolution was studied under the Zeiss Microscope. A few drops of IL were added to a microscopy slide and placed on the heating stage, then some pulp was added with a tweezer and the mixture was heated to 80 °C. When the dissolution proved successful, some small dopes were kneaded to investigate suitable kneading and spinning conditions.

The kneading parameters of all dopes for cycle 0 (using Enocell pulp) are presented in table 2 and those made for cycle 1 (using the fabrics from cycle 0) are presented in table 3. All cycle 0 dopes and the larger cycle 1 dopes were kneaded and filtered with large units. The smaller cycle 1 dopes were kneaded with a smaller kneader and filtered with a smaller Enerpac filter press.

Table 2. Kneading parameters of the cycle 0 dopes: kneading and filtration units used, dry amount of pulp in dope, pulp concentration in dope, temperature, pressure, and kneading time.

Dope	Units	m [g]	c [%]	T [°C]	p [mbar]	t [min]
D_c0_20210506a_HS	Large	228	13	80	20	120
D_c0_20210506b_HS	Large	228	13	80	20	120
D_c0_20210507_TA	Large	215	13	80	20	120
D_c0_20210512_TA	Large	214	13	80	20	120
D_c0_20210514a_TA	Large	233	13	80	20	120
D_c0_20210514b_TA	Large	201	13	80	20	120
D_c0_20210519a_TA	Large	231	13	80	20	120
D_c0_20210519b_TA	Large	217	13	80	20	120

Table 3. The kneading parameters of the cycle 1 dopes: kneading and filtration units used, dry amount of pulp in dope, cellulose (Fab_c0 – 50w) concentration in dope, temperature, pressure, and kneading time.

Dope	Units	m [g]	c [%]	T [°C]	p [mbar]	t [min]
D_c1_20210714_HS	Small	3.12	13	80	47	90
D_c1_20210715_HS	Small	3.14	16	85	41	120
D_c1_20210729_HS	Large	151	14	85-90	65	190
D_c1_20210805_HS	Small	5.67	13	85	41	120
D_c1_20210806_HS	Small	5.15	15	85	41	150
D_c1_20210810_HS	Large	177	13	85	55	90
D_c1_20210813_HS	Small	3.44	14	85	45	120
D_c1_20210818_NT_ISH	Large	171	13	85	50	180

Finally, two small dopes were made of a mixture of Ioncell® fabric (90%) and Lindström cotton waste (10%) to measure how blended fibres would perform compared to pure recycled Ioncell® fibres. The kneading parameters of these dopes are presented in table 4.

Table 4. The kneading parameters of the blended dopes: feedstock materials, kneading and filtration devices, cotton content of dry pulp, dry amount of cotton, Fab_c0-50w and pulp in total, pulp concentration in dope, temperature, pressure, and kneading time.

Parameter	D_c1_20210826_HS_blend	D_c1_20211119_HS_blend
Feedstock	Fab_c0-50w and cotton towel rolls	
Devices	Small units	
m _{Fab_c0} [g]	2.97	2.50
m _{co} [g]	0.32	0.27
m [g]	3.29	2.77
c [%], dope	13.98	14.76
T [°C]	85	85
p [mbar]	40	45
t [min]	240	150

3.3.4 Fibres

The larger dopes were spun into fibres as described by Michud et al. (2016) and Sixta et. al (2015). They were spun with a Fourné Maschinenbau spinning unit using a multifilament spinneret (ET 400 x 0.1 x 0.2 11/18 001). It was connected to a coagulation bath filled with tap water cooled to less than 14 °C, and to two godets with two rollers each to collect the fibres on. The dopes were pre-heated to 80 °C and added to the extrusion cylinder, which was heated to 65-70 °C, depending on the rheology of the dopes. The filaments were then extruded at pressures of 15-30 bar (also depending on the rheology) through the spinneret, stretched in an airgap, immersed into the water bath, and collected on the godets. The extrusion rate was 8.5 cm³/min. The air gap between the spinneret and the bath was about 0.5 cm. The godet speed was adjusted in relation to the extrusion speed to stretch the fibres to reach the desired draw ratio (DR). The DR was calculated with formula 4.

$$DR = \frac{V_{\text{godet}}}{V_{\text{extrusion}}} \quad (4)$$

V_{godet} collection speed of the godets, in m/min
 $V_{\text{extrusion}}$ extrusion speed, in m/min

The smaller dopes were spun with a monofilament system: (Fourné Maschinenbau, spinneret diameter: 0.1 x 0.2), which was connected to an approximately 5 °C water bath and two godets. This spinning unit had an extrusion velocity of 0.010 cm³/min.

The spinning parameters from the larger unit are presented in table 5 and the parameters from the smaller unit are summarized in table 6.

Table 5. An overview of the spinning trials of cycle 0, as well as their spinning parameters: cylinder temperature, extrusion pressure, extrusion rate, bath temperature. The extrusion velocity was 8.500 cm³/min during all trials. The fibres were collected at DR 11.

Dope	Unit	Fibres	T _{cyl} [°C]	p [bar]	T _{bath} [°C]
D_c0_20210506a_HS D_c0_20210506b_HS	Large	Fib_c0_20210507_HS_DR11	69.4-72.4	23.1-35.2	11.0-11.9
D_c0_20210506b_HS D_c0_20210507_TA D_c0_20210512_TA	Large	Fib_c0_20210514_HS_DR11	67.6-68.9	22.7-25.3	11.6-13.6
D_c0_20210514a_TA D_c0_20210514b_TA	Large	Fib_c0_20210521_HS_DR11	67.2-68.9	15.0-20.7	9.4-10.3
D_c0_20210519a_TA D_c0_20210519b_TA	Large	Fib_c0_20210524_HS_DR12	64.1-68.0	14.2-24.6	9.4-11.0

Table 6. An overview of the spinning trials of cycle 1, as well as their spinning parameters: cylinder temperature, extrusion rate, bath temperature, and DR. The extrusion velocity was 0.010 cm³/min (small unit) or 8.500 cm³/min (large unit). The pressure is missing because the sensor was broken.

Dope	Unit	Fibres	T _{cyl} [°C]	T _{bath} [°C]	DR
D_c1_20210714_HS	Small	Fib_c1_20210726_HS_DR11 -14	56.4-56.6	5.0	11-14
D_c1_20210715_HS	Small	No fibres collected	55.0-70.0	5.0	under 11
D_c1_20210805_HS	Small	No fibres collected	55.0-70.5	5.0	under 11
D_c1_20210813_HS	Small	Fib_c1_20210816_HS_DR11	59.3-67.0	5.0	10-11
D_c1_20210810_HS	Large	Fib_c1_20210812_HS_DR11	51.3-58.8	8.4-9.4	11
D_c1_20210818_NT_ISH	Large	Fib_c1_20210819_HS_DR11	56.6-62.3	7.6-8.1	11
D_c1_20210826_HS_ blend	Small	No fibres collected	57.2-69.1	5.0	4
D_c1_20211119_HS_ blend	Small	Fib_c1_20211119_HS_DR5- 7_blend	70.4-75.0	5.0	5-7

Two dopes (D_c1_20210729_HS, D_c1_20210806_HS) were never spun into fibres due to moisture damage, poor dissolution, or poor quality.

Some of the collected fibres were cut into 10 cm pieces for tensile testing, the rest were cut into 40 mm staple fibres. All fibres were washed thoroughly and air-dried. Before being spun to yarn, the staple fibres were treated with a spinline mixture containing Archroma Leomin PN pa (20 w%) and Archroma Afilan CVS liq (80 w%). The mixture was added to a bath containing water, 20 times the dry mass of fibres, and heated to 50 °C. Lastly, the fibres were once again air-dried.

3.3.5 Yarns

In preparation for the yarn spinning, the staple fibres were left overnight in a

conditioned room with an air humidity of 65% and a temperature of 20 °C. The conditioned fibres were fed through a Mesdan Lab Trash Analyzer 281C that opened the tight bundles and removed larger impurities, such as rough fibres and agglomerations from the fibre spinning. The carding was done by a Meslan Lab – Laboratory carding machine 337A. The opened fibres were fed into it in batches of 30 g. When the fibres had been carded, they were rolled into slivers and fed through a Meslin Lab – Stiro roving lab 3371 stretching and doubling device. At first, a thick sliver was drafted into a thinner sliver. Then, two thinner slivers were combined and drafted. Finally, two doubled slivers were combined into roving. The roving was fed into a Spinn tester - Ser.M.Tes Ring Lab spinning device with a traveller of 50 mg (Bräcker ISO 50, M1f). It was stretched to reach a linear density of 30 tex and twisted into primary yarn with a Z twist of 740.

3.3.6 Fabrics

Two skeins at a time were joined together with a Breveté S.G.D.G Desvignes spooling machine, and then S-twisted with an Agteks Direc Twist C6"-D6" twisting device with 300 twist per meter. This yarn was then fed through a Stoll CMS ADF 32W E7.3 multi gauge that knitted it into a total of thirteen roughly 50 cm x 50 cm pieces of rib-structured fabric.

3.3.7 Laundering

The laundry trial was conducted according to the standard procedure of EN ISO 6330 (SFS) for 50 laundry cycles. The fabric was washed inside laundry bags using an Electrolux Wascator FOM71MP laundry machine with a mild 37 min program and “detergent 1”, AATCC 1993 without optical brightener. It was then centrifuged in a separate device. Pure cotton ballast was added to the laundry machine to achieve a total load of two kilograms. Samples of the fabric were collected for analysis after 1, 3, 5, 10, 20, 30, 40, and 50 cycles.

3.4 Tests and analyses

The material characterization, tests and analyses were conducted as described by Michud et al. (2016), Sixta et al. (2015), and various standard procedures. The results will be presented in the “Results” section.

3.4.1 Dope rheology

The rheology of each dope was studied with an Anton Paar Physica MCR 302 rheometer. The samples were prepared and measured as described by Sixta et. al (2015). Thereby, the dope samples were cut into discs with a thickness of 1 mm and a diameter of 25 mm. Each sample was placed between a movable plate and a static plate, which was heated to different temperatures between 50 °C and 90 °C. The device performed dynamic frequency sweep test from 0.01 to 100 rad/s. The complex viscosities, storage and loss moduli, crossover moduli and angular frequencies were achieved by using the Cross model assuming the validity of the Cox-Merz rule.

3.4.2 Fibre testing

The tensile testing of the fibres was conducted in accordance with EN ISO 5079 and EN ISO 139 standard procedures. They were conditioned overnight at a relative air humidity of 65% at a temperature of 20 °C. Linear density, elongation at breakage, and tenacity were measured for twenty fibres of each sample. Then, the wet tensile tests were performed with the same device for 20 fibres after being soaked in water for 10 seconds.

Three fibres from each sample were selected for birefringence testing, based on how close they were to the average linear density of the sample: ± 0.1 dtex. The selected fibres were then attached to a microscope slide and investigated under a Zeiss Axio Scope.A1 microscope with a Zeiss Tilting Compensator B. The fibres were then analysed as instructed in the manual (Zeiss), which gave the polymer alignment in the fibres.

The structural morphology of some of the fibres was investigated by a scanning electron microscope (SEM, Zeiss Sigma VP), following the methods of Michud et al. (2016). To determine the cross-sections, a bundle of fibres was immersed into water and flash-frozen by liquid nitrogen. The ice was shattered, and the smashed pieces of ice containing fibres were collected and melted. The preserved fibres were dried and attached to tilted SEM sample holders. To get a view of the surface, some fibres were simply placed on regular SEM sample holders.

The scanning electron microscopy (SEM) samples were coated with 80 Au/20 Pd in a Quorum 150R S plus sputtering device.

3.4.3 Yarn testing

The linear density of the yarn was measured manually under the same humidity and temperature as it was spun. Ten meters of each skein was collected with a Mesdan LAB wrap reel and then weighed. Since the ends of the yarns tend to be irregular, this was repeated until the same skein gave three similar linear densities in a row. Then, the tenacity and elongation at breakage of the yarns were measured according to ISO 2062:2009(en). The testing device was a Tensile and compression testing machine MTS 400/M. The load cell was 50 N, the gauge length was 250 mm, and the testing speed was 250 mm/min.

3.4.4 Intrinsic viscosity

The intrinsic viscosity, which correlates with the degree of polymerization, was measured for each material throughout the work, from pulp to fabric to recycled fibres, following the SCAN-CM 15:88 standard procedure. The materials were ground and three samples of roughly 250 mg were moved to plastic bottles. Five copper rods were added to each bottle, which then was filled with 25 ml deionized water and shaken for 30 minutes in a Kauko Lehtinen shaking device. Then, 25 ml of cupriethylenediamine was added before shaking them for another 30 minutes, to dissolve the cellulose.

The bottles were then placed in a 25 °C water bath (Mistral Multi-stirrer) before they were run through a capillary to have the retention time measured. The relative viscosity was calculated according to formula 5.

$$\eta_{\text{rel}} = h \cdot t \quad (5)$$

η_{rel}	relative viscosity
h	calibration constant for a particular capillary, in s^{-1}
t	time it takes for a sample to flow through the capillary, in s

The relative viscosity was then converted to limiting viscosity according to the table provided in the standard. Then, the intrinsic viscosity was calculated with formula 6.

$$[\eta] = \frac{[\eta]_c}{c_{\text{pulp}}} \quad (6)$$

$[\eta]$	intrinsic viscosity, in ml/g
$[\eta]_c$	limiting viscosity as read from a table
c_{pulp}	concentration of cellulose in the sample in g/ml

3.4.5 Molecular weight distribution

The molecular weight distribution and averages were measured with gel permeation chromatography (GPC) following the methods of Pitkänen and Sixta (2020). Three samples of 50 mg were taken from pulp (P_c0), fibres (Fib_c0 and Fib_c1), pre-laundry fabric (Fab_c0–0w), and post-laundry fabric (Fab_c0–50w). They were placed in polypropylene tubes with 20 μm PE-frit filters. To each tube, 4 ml of water was added to activate the cellulose overnight. The next day, the water was removed through the filter using a vacuum manifold. To remove the last of the water, the tubes were filled with roughly 2 ml of acetone and emptied again. Then, they were filled with 4 ml of acetone and left again overnight to further activate the cellulose. The acetone was removed the same way as the water and was replaced with 4 ml of DMAc. On the following day, the DMAc was drained through the manifold and the cellulose samples were moved to 10 ml glass bottles. To start the dissolution, 5.0 ml of LiCl/DMAc (concentration 90 g/l) was added to each bottle. The samples were

stirred until the liquid inside was clear, which took approximately four hours. The samples were then diluted, 0.5 ml sample in 4.5 ml pure DMAc to reach a concentration of 9 g/l. The diluted samples were filtrated through 0.2 μ m filters, moved to plastic vials, and run through a Dionex UltiMate 3000 Column HPLC.

3.4.6 Monosaccharides and lignin content

The amounts of different monosaccharides were measured with high-performance anion-exchange chromatography (HPAEC) and the lignin content was determined gravimetrically and by UV spectroscopy. Sluiter et. al (2006) described how it works.

Three samples of roughly 300 mg were taken from each material. The samples were placed in DURAN glass bottles and 3 ml of 72% sulfuric acid was added. The bottles were put in a 30 °C water bath (Köttermann GWB) for 60 minutes, where they were stirred every 5 minutes. When the samples had dissolved, 84 ml of distilled water was added to achieve an acid concentration of 4%.

Parallel to the samples, a sugar recovery standard containing glucose (200 mg), mannose (50 mg), galactose (50 mg), and xylose (50 mg) was prepared. The sugars were dissolved in 100 ml of water. 84 ml of the solution was moved to a DURAN glass bottle for hydrolysis to which 3 ml of sulfuric acid was added. The rest of the sugar solution was not hydrolysed but saved for later analysis.

The samples and sugars were hydrolysed in a Systec DE-23 autoclave at 121 °C, after which the samples were filtered through VitraPOR Borosilicate 3.3 crucibles. The residue, suspected to be lignin, was dried and weighed.

Finally, the filtrate was diluted with milli-Q water by a factor of 100, added to plastic vials, and measured in a Dionex ICS-3000 HPAEC-PAD.

The amount of solubilized lignin was measured by UV-spectrometry at a wavelength of 205 nm. The samples were diluted by a factor of 5-10, then analysed in a Shimadzu UV-2550.

3.4.7 Chemical composition

The content of inorganic matter was measured gravimetrically by an ash content analysis and a thermal analysis was performed with an STA (simultaneous thermal analysis). For the ash content analysis, three samples of roughly 250 mg were taken from each pulp (Pulp_c0), fibre (Fib_c0 and Fib_c1), pre-laundry fabric (Fab_c0–0w), and post-laundry fabric (Fab_c0–50w). The samples were heated up to 575 °C for 5.5 h in a Naber Industrieofenbau oven. This temperature was high enough to cause the organic matter (cellulose, hemicellulose, lignin) to combust, while leaving only the inorganic matter in the crucibles (Mortensen and Wallin, 1989). Upon cooling down, the residue in each crucible was weighed and compared to the original mass of the samples as calculated with formula 7.

$$AC = \frac{m_f}{m_i} \cdot 100\% \quad (7)$$

AC	ash content, in %.
m_i	dry mass of the sample, in g.
m_f	mass of the sample after the treatment, in g.

Samples of about 15 mg were taken from each material for STA analysis. The samples were placed in small crucibles and analysed with a Netzsch STA 449 F3 Jupiter device. The degradation behaviour of the samples was monitored by increasing the temperatures from 40 °C up to 600 °C, in a helium atmosphere (gas flow: 70 ml/min) with a heating rate of 10 K/min, while measuring the mass of the sample. This data could then be plotted in different diagrams.

The presence of different (mostly metallic) impurities was measured with inductively coupled plasma optical emission spectrometry (ICP-OES), following the ISO 14869-3:2017 and SFS-EN 16170:2016 standard procedures. The samples were first digested in nitric acid, HCl, HF, and H₂O₂, then digested in H₃BO₃ for successful dissolution, and finally analysed with an Agilent 5900 SVDV ICP-OES – RAMI device.

The elemental composition of each material was analysed with a ThermoScientific

FlashSmart device, as instructed in the manual (ThermoScientific, 2017). Samples of about two milligrams were taken from each material. They were oxidized by oxygen gas and combusted in a tin container, then the combustion products were carried to a chromatographic column by helium gas. There the different compounds were separated and finally detected by a thermal conductivity detector.

The chemical composition of each material was analysed with a PerkinElmer FTIR spectrometer from wavenumber 500 cm^{-1} to 4000 cm^{-1} . By comparing the transmittance peaks at different wavenumbers, it was possible to determine if there had been any changes in the chemical composition of the materials (Nandiyanto et al., 2019).

3.4.8 Optical properties

The brightness and colour were measured with a SpectroScan (16130-818) and Spectrolino by GretagMacbeth, following the methods of the ISO 2470-1:2009 standard. Three samples of approximately 300 mg were suspended in 50 ml deionized water and moved to a Buchner funnel where the water was drained under vacuum. The resulting dense cakes were dried at $105\text{ }^{\circ}\text{C}$ in an oven overnight, then they were scanned three times on different locations at an observer angle of 10° , D65 illumination, polarized filter, abs white base, and a DIN density standard.

The crystalline properties of the fibres were measured by XRD with a Xenocs SAXS instrument in accordance with the methods of Moriam et al. (2021). Bundles of fibres were placed on sample plates, after which they were scanned for WAXS and SAXS analysis. The detector (Cu) did a vertical sweep to find the thickest part of the fibre, then measured the closest few millimetres of that. The acquisition time was 120 seconds.

4 Results and discussion

An overview of the laboratory work is presented in figure 3, which shows the material flow and the analyses that were made of each material.

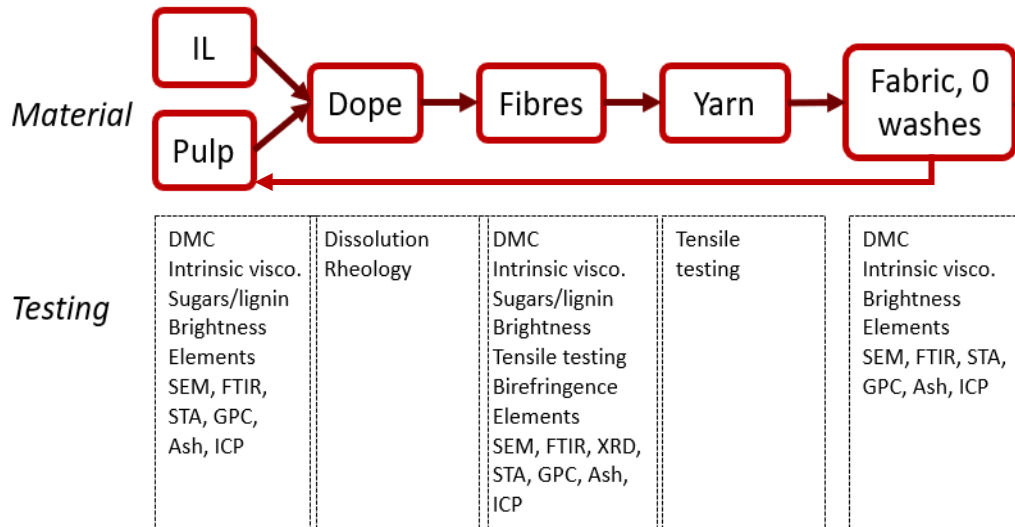


Figure 3. An overview of how the materials throughout the work were analysed.

4.1 Dope and spinnability

Microscopy pictures (figure 4) show that the pulp as well as the ground Ioncell® fabric rapidly dissolved in IL.

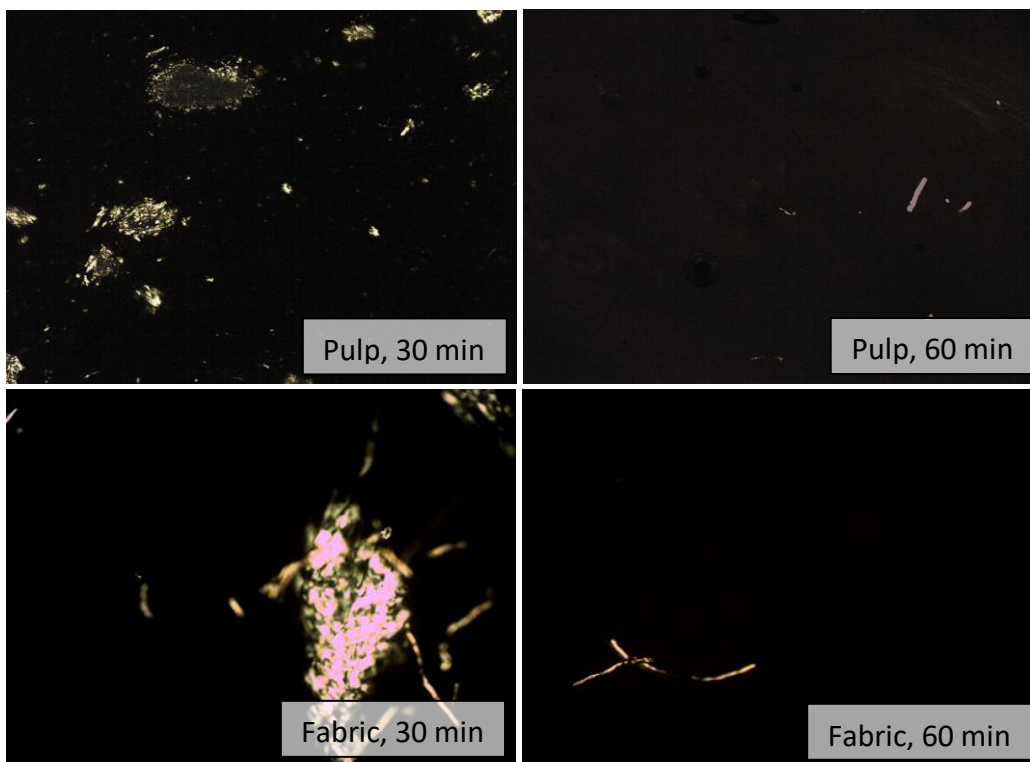


Figure 4. First row, c0 dope: Enocell pulp in IL after 30 min (left) and 60 min (right) of kneading. Second row, c1 dope: ground Fab_c0 - 50w in IL after 30 min (left) and 60 min (right) of kneading. The pictures are taken at 5x magnification under polarized light, where the solid matter appears white.

Cycle 1 dopes required slightly more time to dissolve than the cycle 0 dopes, as the kneading times in tables 2 and 3 showed earlier. The reason might be that the ground Fab_c0-50w formed compact agglomerations that were harder for the IL to penetrate. The ground Enocell pulp formed no such agglomerations but remained a fine powder.

The rheological properties of the dopes are summarized in figures 5-8. Some dopes did not get analysed because the rheology samples were ruined by air humidity while in storage.

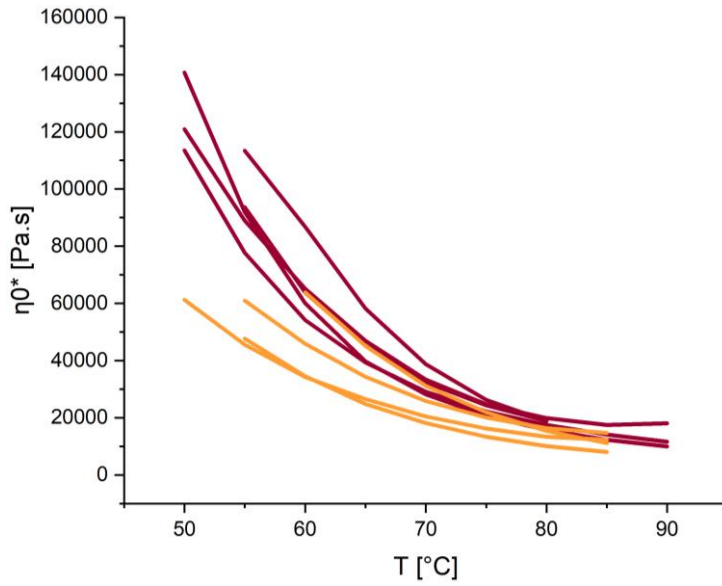


Figure 5. The temperature in °C versus the complex viscosity in Pas of c0 dopes(red) and c1 dopes (yellow).

Optimal spinning is achieved at zero-shear viscosities ranging 25 000-35 000 Pa*s (Sixta et. al, 2015). As seen in figure 5, this value was found at 65-75 °C for cycle 0 and closer to 60 °C for cycle 1, so the spinning conditions needed to be adjusted accordingly.

The blended c1 dopes are not included in figure 5 since the Cross model was not applicable due to the trends of the complex viscosities (see figure 6).

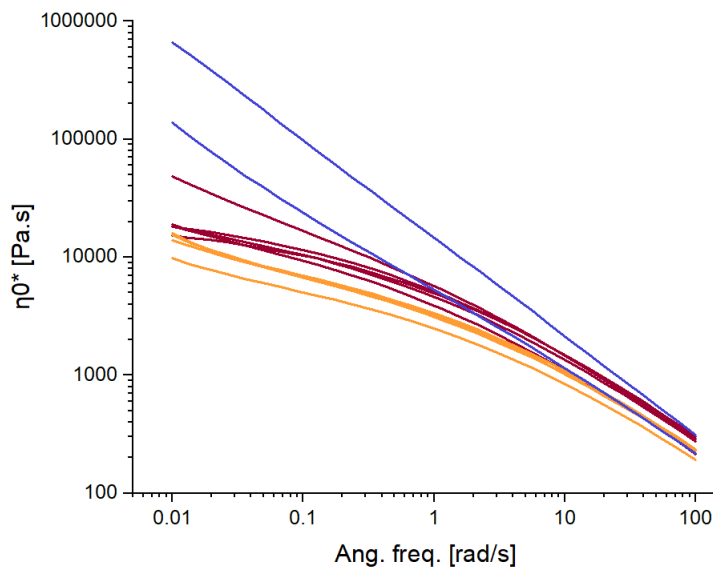


Figure 6. The angular frequency in rad/s versus the complex viscosity in Pa*s at 80 °C of c0 dopes(red), c1 dopes (yellow), and blended c1 dopes (blue).

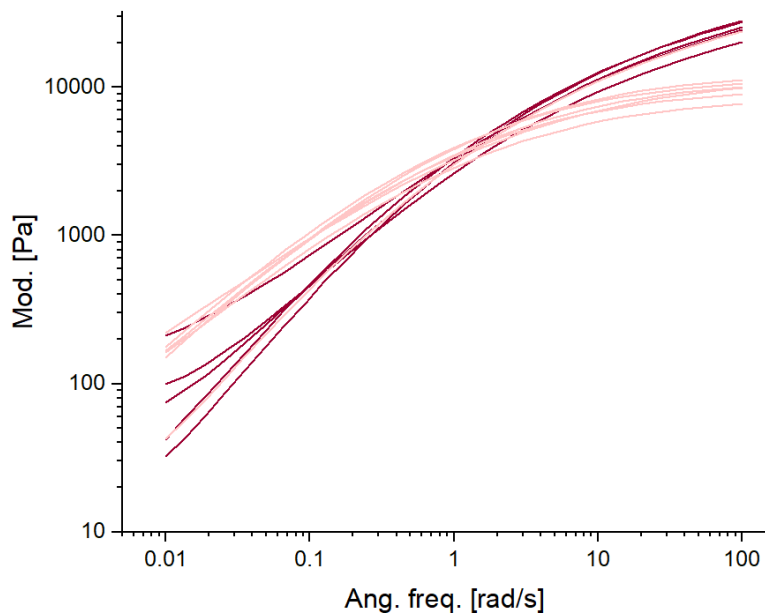


Figure 7. The storage modulus (dark) and loss modulus (light) versus angular frequency of c0 dopes at 80 °C.

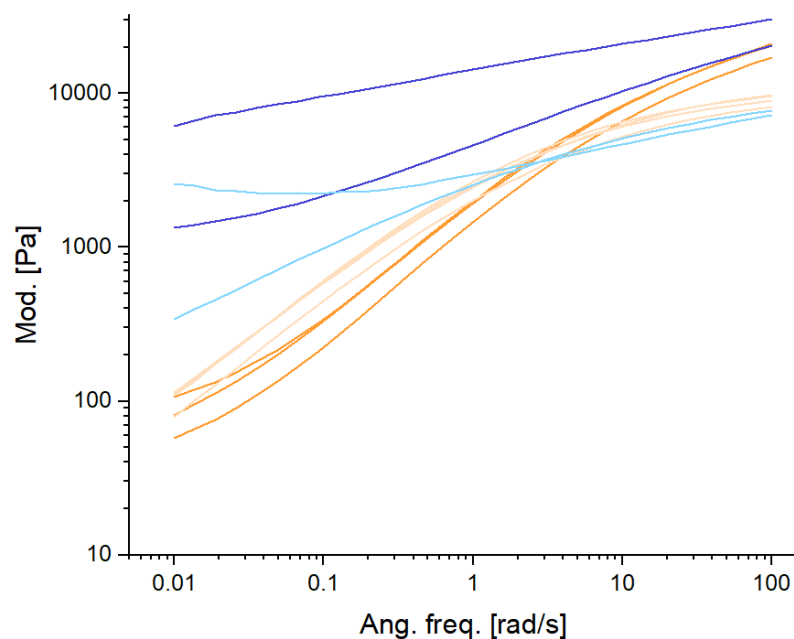


Figure 8. The storage modulus (dark) and loss modulus (light) versus angular frequency of c1 dopes (yellow) and c1 blended dopes (blue) at 80 °C.

The results show that cycle 1 dopes had a lower viscosity than cycle 0 dopes. This decrease was caused by the reduction of the DP of the cellulose chains in the course of the recycling procedure. Dopes produced with cellulose of elevated DP (cycle 0) show increased complex viscosities compared to dopes produced with cellulose of

lower DP (cycle 1) (Asaadi et al., 2016).

The viscosity increased when the cellulose consistence was increased, or the dope was produced with Ioncell® fabric blended with cotton, which also was observed by Michud et al. (2015). The crossover points between storage and loss moduli (figure 7) were measured to obtain information about the spinnability of the dopes. The most stable spinning is achievable when the crossover is at 1 rad/s or slightly higher (Sixta et al. 2015). Figure 7 illustrates the crossover points at approximately 1 rad/s for the cycle 0 dopes. The crossover points for the cycle 1 dopes were at approximately 1 rad/s, but the blended dopes had no crossover point. This explains the poor spinnability of the blended dopes.

4.2 Fibre and yarn properties

The results from the fibre testing are summarized in figure 9 and table 7. Figure 9 shows the elongations and tenacities of all fibre samples in comparison to other cellulosic textile fibres. Table 7 shows the average tensile properties from each cycle.

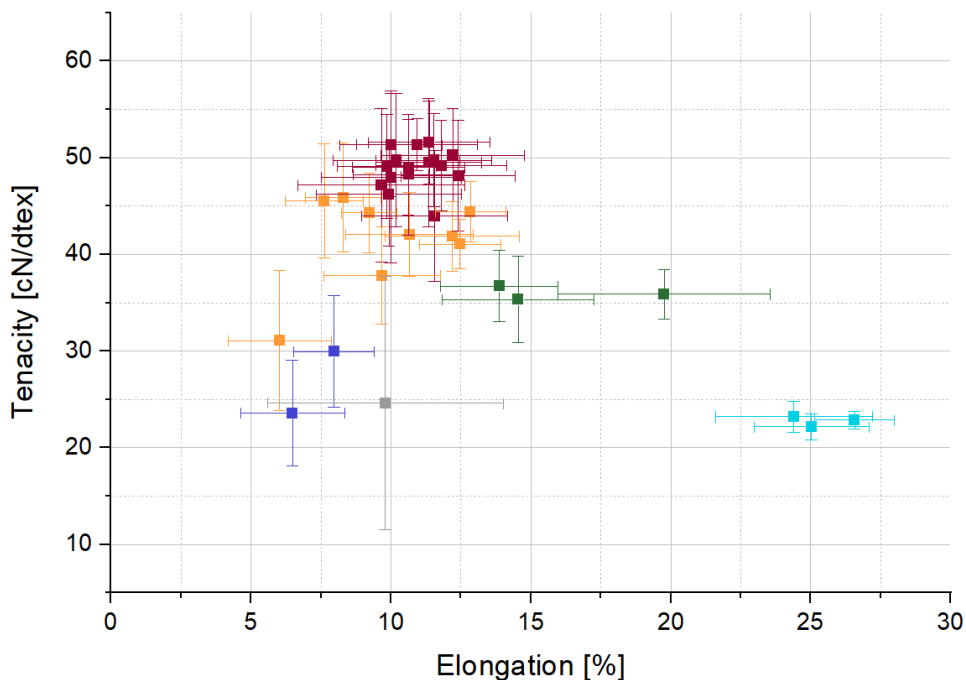


Figure 9. Elongation at break vs tenacity of each material in dry conditions: cycle 0 DR 11 fibres (red), cycle 1 DR 11 fibres (yellow), and cycle 1 blended (blue), with DR 5.1 and 7.1. For comparison, see the elongation vs tenacity of cotton (grey), lyocell (green), and viscose (cyan) fibres, as measured by Sederholm (2019).

Table 7. The average linear density, elongation at break, and tenacity of each material in conditioned ("dry") and wet conditions.

Material		Linear density [dtex]	Tenacity [cN/tex]	Elongation [%]
Cycle 0 fibres DR 11	dry	1.27 ± 0.12	48.90 ± 4.37	10.88 ± 1.92
	wet	1.27 ± 0.14	43.57 ± 4.76	13.25 ± 2.14
Cycle 1 fibres DR 11	dry	1.43 ± 0.17	41.54 ± 4.62	9.89 ± 1.67
	wet	1.38 ± 0.22	34.16 ± 4.50	11.51 ± 2.09
Cycle 1 blend DR 7.1	dry	2.24 ± 0.31	29.94 ± 5.79	7.97 ± 1.43
	wet	2.28 ± 0.22	22.53 ± 4.53	9.28 ± 1.98
Cycle 1 blend DR 5.1	Dry	3.11 ± 0.31	23.57 ± 5.47	6.48 ± 1.86
	wet	3.12 ± 0.31	20.83 ± 3.26	9.35 ± 1.61

The results show a slight decrease in tenacity when the material was recycled, while the elongation stayed roughly the same. The fibres from cycle 1 fibres had a higher linear density than those from cycle 0 despite having the same DR. Since tenacity is given in cN/tex, the higher linear density affects it negatively. Furthermore, blending the fabric with cotton waste did not seem to improve the mechanical properties of the recycled fibres.

However, compared to the studies of Sederholm (2019), the cycle 1 fibres still showed a higher elongation or tenacity than other cellulosic fibres on the market. Cotton, for instance, had a tenacity of 25 cN/tex and an elongation of 9.8%. Viscose had tenacities of 22-23 cN/tex and elongations of 24-27%. For commercial lyocell fibres, the values were 35-37 cN/tex and 14-20%.

The fibres from cycle 0 are comparable with Ioncell® fibres from earlier studies. Michud et al. (2016) produced fibres with dry tenacities of 46-48 cN/tex and wet tenacities of 42-44 cN/tex. The dry elongations were 8.8-9.2% and the wet elongations were 11.3-11.7%. Sixta et al. (2015) produced Ioncell® fibres from dopes with different pulp concentration and achieved tenacities of 46.8 cN/tex (conditioned) and 42.6 cN/tex (wet), and elongations of 9.3% (conditioned) and 10.9 (wet).

The decrease in tensile properties can be explained by the physical structure of the

fibres. Figures 10 and 11 show SEM pictures of cycle 0 and cycle 1 fibres.

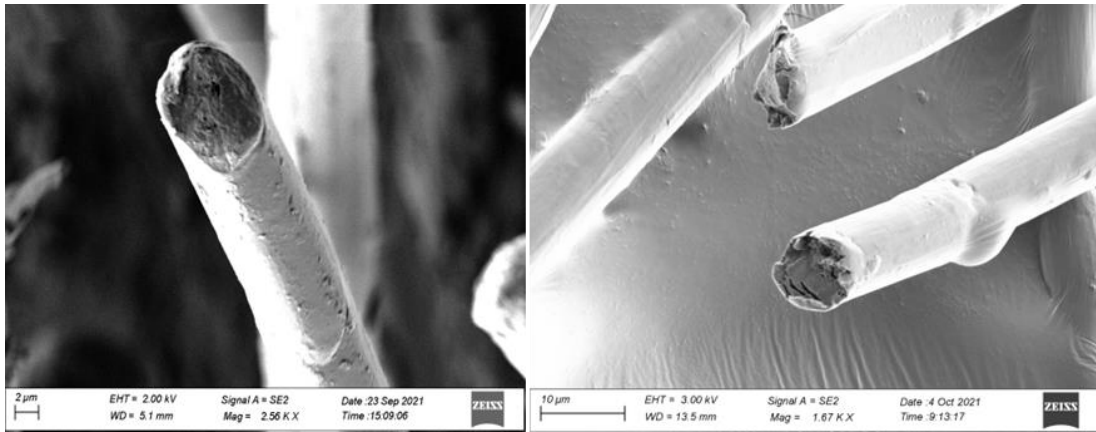


Figure 10. The cross-section of fibres from cycle 0 (left) and cycle 1 (right).

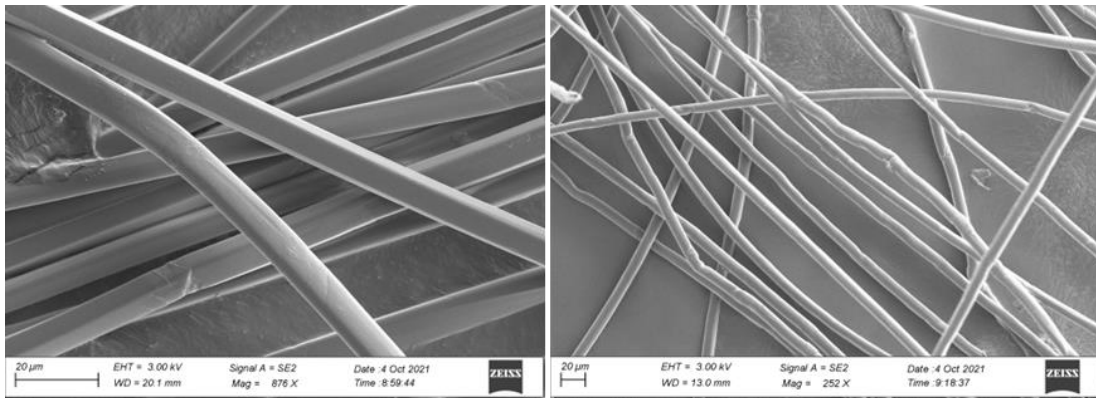


Figure 11. The surface of fibres from cycle 0 (left) and cycle 1 (right).

The cross-section pictures (figure 10) show no major defects or pits that would cause weakness. The surface pictures (figure 11) show that the recycled fibres show more irregularities: kinks and thicker sections. These irregularities cause weak regions, which rupture when the fibre is strained.

Elsayed et al. (2021) and Ojha (2021) achieved results similar to those of cycle 0 in a SEM analysis of DR 12 fibres. The cross-sections were even and regular. The fibres had a smooth surface, identical to that of cycle 0 fibres.

Table 8 compares the mechanical properties of the yarns produced in cycle 0 and cycle 1.

Table 8. The average linear density, elongation at break, and tenacity of the yarns in dry conditions.

Material		Linear density [tex]	Tenacity [cN/tex]	Elongation [%]
Cycle 0 yarn	dry	3.14 ± 0.19	28.04 ± 3.77	7.83 ± 0.81
Cycle 1 yarn	dry	3.01 ± 0.15	22.68 ± 3.10	7.71 ± 0.95

The linear density, tenacity, or elongation of the yarn decreased after being recycled. Since the fibres underwent a similar decrease in tenacity and elongation, the same was to be expected for the yarn. For both fibres and yarn the decrease was within the margin of error.

The cycle 0 yarns are comparable to the yarns produced and tested by Haslinger et al. (2019b). The uncoated single-ply yarn samples had a linear density of 22.9 dtex, a tenacity of 30.9 cN/tex, and an elongation of 8.1%.

4.3 Polymer properties

The GPC results, showing the molecular weight distributions of virgin pulp, cycle 0 fibres, and cycle 1 fibres are presented in figure 12.

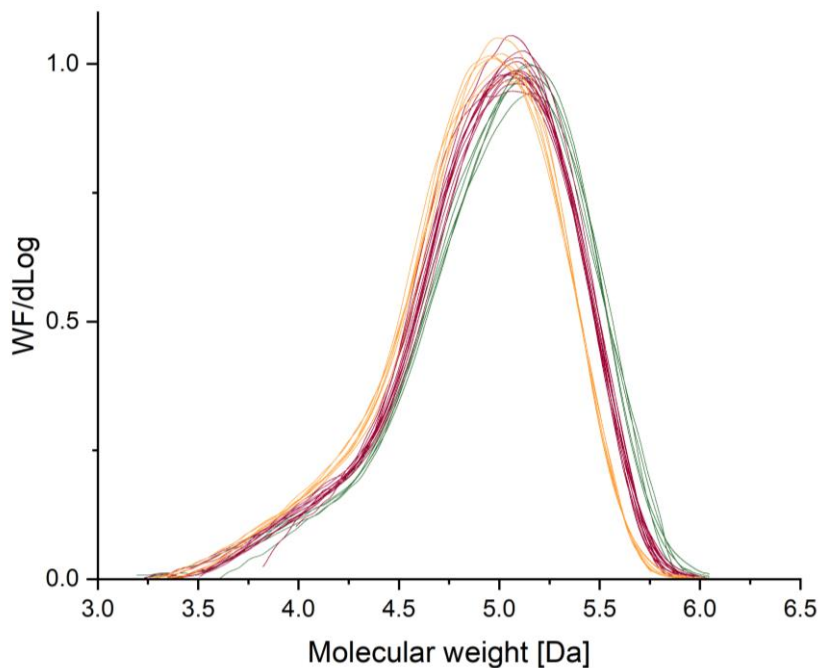


Figure 12. The molecular weight distribution of virgin pulp (green), c0 fibres (red), and c1 fibres (yellow).

Figure 12 shows that the distribution curves shifted towards a lower molecular weight when pulp became fibres and when the fibres were recycled. However, the curves retained their shape and did not become wider and flatter. This means that the material still was uniform despite the polymer degradation.

Ojha (2021) found a similar trend. The molecular weight distribution of pulp was compared to that of fibres. The curve retained its narrow shape but shifted towards a slightly lower molecular weight.

Table 9 shows the numerical data of the measurements.

Table 9. The weight average molecular weight (M_w), number average molecular weight (M_n), low molecular weight fractions of cellulose ($DP < 100$) and high molecular weight fractions of cellulose ($DP > 2000$) of the material: virgin pulp, cycle 0 fibres, cycle 0 fabric pre- and post-laundering, and cycle 1 fibres.

Material	M_w [kDa]	M_n [kDa]	DP < 100 [%]	DP > 2000 [%]
Pulp_c0	153.5 ± 5.6	54.7 ± 6.1	5.99 ± 1.17	11.13 ± 1.24
Fib_c0	127.6 ± 3.0	49.5 ± 5.8	6.46 ± 1.32	6.21 ± 0.47
Fab_c0_0w	122.2 ± 6.2	46.2 ± 9.2	7.56 ± 3.21	5.27 ± 0.64
Fab_c0_50w	121.5 ± 1.9	46.5 ± 2.9	7.08 ± 0.71	5.21 ± 0.28
Fib_c1	107.2 ± 1.2	41.1 ± 1.6	8.35 ± 0.42	3.26 ± 0.08

There was a notable decrease of all values, except the DP > 100, which increased. Elsayed (2021) obtained comparable results. Enocell pulp had an average molecular weight of 160.5 kDa, whereas the fibres reached 158.4 kDa. The number average molecular weight was 44.5 kDa for pulp and 49.3 for fibres. The DP beneath 100 decreased from 4.8% to 4.4%, and above 2000 it increased from 12.2 5 to 12.6%. Note, that Elsayed used [mTBDH][OAc] and this work was performed with [DBN][OAc].

The trend observed in the molecular weight distribution was also reflected in the intrinsic viscosity measurements, the results of which are presented in figure 13.

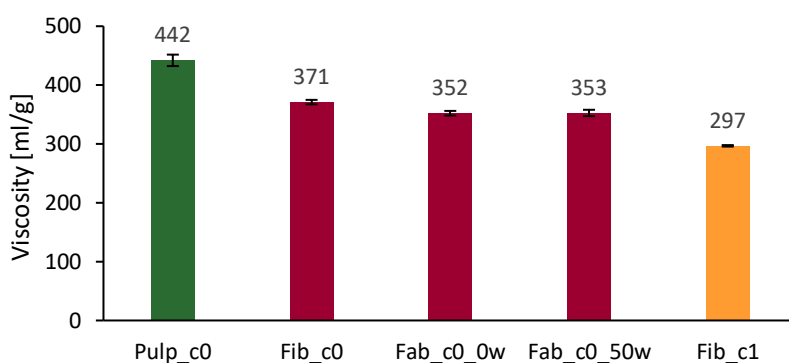


Figure 13. The intrinsic viscosity of the materials, with error bars: virgin pulp (green), cycle 0 fibres and fabric pre- as well as post-laundering (red), and cycle 1 fibres (yellow).

The decrease in intrinsic viscosity and consequently in degree of polymerisation offers an explanation to the decrease of the mechanical properties. De Silva and Byrne (2017) investigated how the degree of polymerisation affects the mechanical properties of regenerated cellulose fibres. Cotton waste of different degrees of polymerisation was dissolved in an ionic liquid and spun into fibres. The fibres with a low degree of polymerisation had a lower tensile strength than the ones with a higher.

Wedin et al. (2019) measured the intrinsic viscosity of different cellulosic textile fibres. The lowest viscosity, measured from viscose, was 153 ml/g. It decreased to 133 ml/g after 50 laundry cycles. Lyocell fibres decreased from 338 ml/g to 333 ml/g after 50 laundry cycles. Untreated cotton underwent a viscosity decrease from 1980 ml/g to 1819 ml/g, whereas treated cotton showed a decrease from 996 ml/g to 812 ml/g after the laundering. In comparison, the cycle 0 Ioncell® fibres in this work did not decrease in intrinsic viscosity during the laundry cycles. The resistance to damage in the laundry machine was also visible on the fabric. No tearing, pilling or deformation occurred during the washing.

According to Asaadi et al. (2016), the optimal intrinsic viscosity for lyocell-type dry-jet wet spinning is 400-500 ml/g. Consequently, due to the reduction of the viscosity this optimum was not achieved anymore. Nevertheless, it was possible to spin the produced dopes.

By the birefringence measurements the degree of orientation of the cellulose fibres was determined. Moreover, the degree of crystallinity was analysed with XRD. The results are presented in table 10.

Table 10. The total polymer orientation and crystallinity of the virgin fibres and recycled fibres.

Material	Orientation [%]	Crystallinity [%]
Cycle 0 fibres, DR 11	71.8 ± 1.5	52.6 ± 1.9
Cycle 1 fibres, DR 11	70.4 ± 1.9	45.2 ± 2.1

The results show that the polymer orientation was not significantly influenced by the recycling procedure, but the crystallinity decreased.

Sixta et al. (2015) obtained a higher orientation and a lower crystallinity for standard Ioncell® fibres. Fibres of DR 12.4 had a total orientation of 81.1% and a crystallinity of 35%. Ojha (2021), however, measured standard Ioncell® DR 11 fibres and determined an orientation of 75.6%. Crystallinity was not measured of the standard fibres, but DR 11 fibres made of chemically recycled cotton had crystallinities ranging from 38.3% to 39.7% and orientations of 72.3%-76.8%.

The XRD scattering of the fibre samples are shown in figure 14.

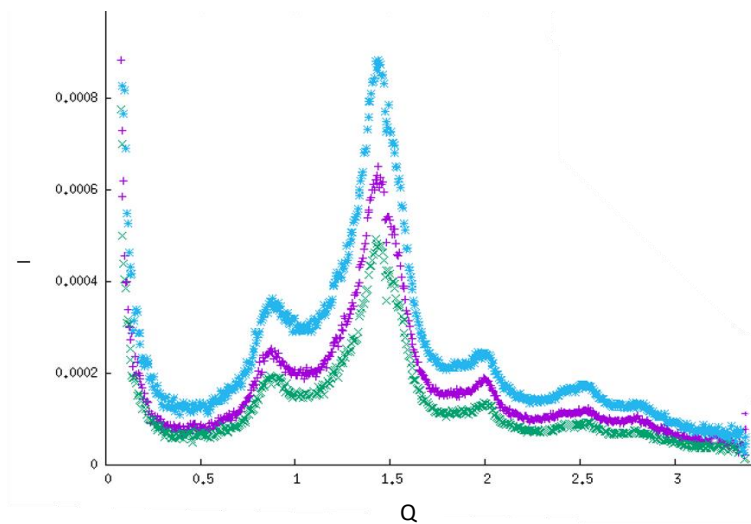


Figure 14. The scattering vector (Q) versus the diffracted X-ray intensity (I) of cycle 0 fibres (purple and green) and cycle 1 fibres (blue).

The results show similar reflexes for the different fibres, indicating that the crystalline structure was similar (Cellulose II). Only the intensity of the reflexes differs in

accordance with the crystallinity. Around $Q=1.5$ there are the reflexes 110 & 020 located and at around $Q=0.8$ the 1-10 reflex and at around $Q=2.5$ the 004 reflex. This is typical for the cellulose II structure, which is common in regenerated cellulose (Sébe et al. 2012).

4.4 Chemical composition

The results from the FTIR analysis are presented in figure 15.

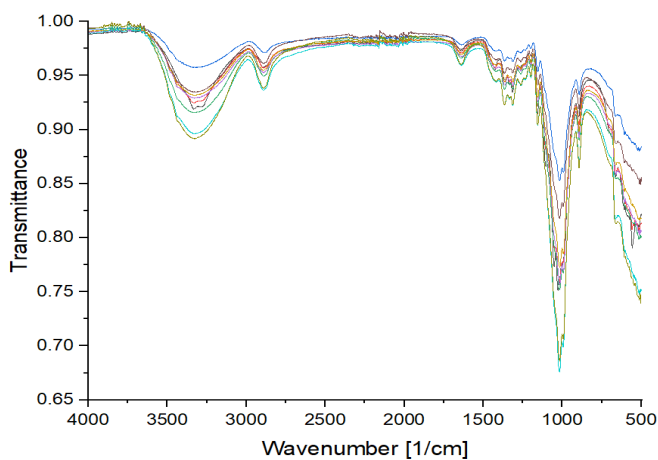


Figure 15. The FTIR results of each material: pulp (black and red), cycle 0 fibres (blue and green), cycle 0 fabric before (purple and gold) and after (cyan and brown) laundering, and cycle 1 fibres (grey).

The FTIR results show that the material did not undergo a significant change in chemical composition throughout the study. The trends of the infrared spectra remain the same. Chemically recycled cotton fibres by De Silva et al. (2017) produced a similar spectrogram, and the peaks at $3\,300\text{ cm}^{-1}$, $2\,900\text{ cm}^{-1}$, $1\,640\text{ cm}^{-1}$, $1\,375$, $1\,420\text{ cm}^{-1}$, and 898 cm^{-1} were defined as characteristic peaks for cellulose, which present O-H stretching, C-H bending, bound water, and C-H stretching modes respectively.

The thermodynamic behaviour was investigated with STA and the amount of inorganic matter was determined by ash content analysis. The results are shown in table 11.

Table 11. The inorganic matter (ash) content of each material according to manual gravimetry.

Material	Ash content (%)
c0_Pulp	0.09 ± 0.03
c0_Fib	0.11 ± 0.60
c0_Fab – 0w	0.34 ± 0.47
c0_Fab – 50w	0.11 ± 0.06
c1_Fib	0.19 ± 0.16

The results in table 11 show that only trace amounts of inorganic matter were found in the materials. The fibre spinning, spin finishing, and yarn spinning increased the amounts of impurities from 0.09% to 0.34%. However, most of these impurities disappeared in the laundering, bringing the inorganic matter content down to 0.11%. Once again, the amount increased when new (cycle 1) fibres were made.

The cotton waste that Ojha (2021) used as feedstock had equally varying amounts of inorganic matter, ranging from 0.12% to 0.56%.

The STA results presented in figure 16 show that all samples degraded at the same temperature and at the same rate.

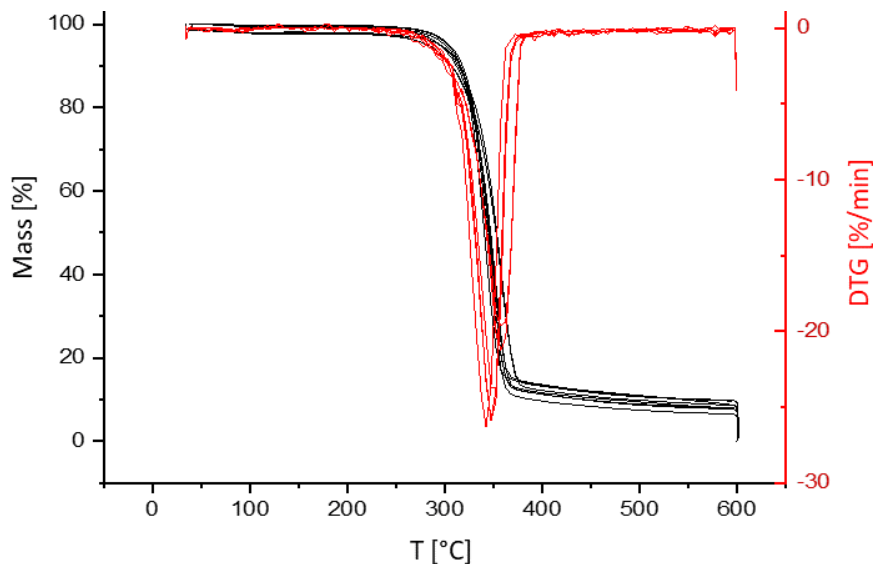


Figure 16. The temperature versus sample mass (black) and sample degradation rate (red) of all samples: pulp, fibres, and fabric.

The onset temperature of the degradation was 312-322 °C for all measurements. Ojha (2021) found similar thermal degradation trends for Ioncell® fibres made from pulp as well as cotton waste. The highest rate of degradation occurred at 300-400 °C.

The inorganic components were analysed by ICP-OES (inductively coupled plasma - optical emission spectrometry). Table 12 shows the contents of the most abundant metals in the samples.

Table 12. The contents of different elements in each sample.

Material	Al [ppm]	Ca [ppm]	Cu [ppm]	Fe [ppm]	Mg [ppm]	Si [ppm]	K [ppm]	Na [ppm]
c0 pulp	2.4	68.5	1.3	2.6	9.9	N/A	13.0	330.0
c0 fib	3.2	100.0	2.5	4.3	8.0	120.0	28.0	20.0
c0 fab 0w	32.0	195.0	20.9	38.0	19.0	210.0	127.5	100.0
c0 fab 50w	38.5	400.0	61.5	80.0	24.0	64.0	31.5	68.0
c1 fib	38.0	68.0	8.7	26.0	4.5	330.0	37.0	79.0

Table 12 shows that the most abundant metal was calcium. The amount was particularly high after the laundry trials, presumably because the laundry was performed with tap water rather than deionized water. This would also explain the increase of iron and possibly copper. Sodium was abundant in the pulp, but later decreased with over 50%. The sodium is therefore assumed to originate from pulping processes. Potassium is present in the spin finish solution (Archroma, 2013). This gives an explanation to its abundance in the unwashed fabric, and to its decrease during the laundry cycles.

Another source of metals is the wood itself. Wood naturally contains trace amounts of metals, such as potassium, calcium, manganese, magnesium, and iron (Saltberg et al., 2006). In particular, birch contains 67 ppm calcium and 51 ppm potassium. In comparison, an ICP analysis of cotton showed much higher contents of metals (Brushwood and Perkins, 1994). There, the samples had an average potassium content of 4650 ppm, calcium content of 740 ppm, and magnesium content of 600 ppm. Iron, copper, manganese, and zinc was also found in smaller amounts.

None of the detected elements exceeds the maximum amount stated in the REACH list of restricted chemicals (europa.eu).

Table 13 shows the results from the non-metal elemental analysis conducted with chromatography.

Table 13. The amounts of nitrogen, carbon, and hydrogen in each material.

Material	N [w%]	C [w%]	H [w%]
c0 pulp	0.01	42.39	6.07
c0 fibres	0.06	41.42	6.17
c0 fabric, 0w	0.08	41.57	6.17
c0 fabric, 50w	0.02	41.36	6.19
c1 fibres	0.05	40.81	6.16

The amounts of carbon and hydrogen remained stable throughout the process. Nitrogen is present in the IL, and its low concentration shows that the fibres were thoroughly washed after the dry-jet wet spinning. In comparison, a study by Fagernäs et al. (2012) showed that birch contains 49.7w% carbon, 6.1w% hydrogen, and 0.1w% nitrogen.

The lignocellulosic composition as measured with HPLAC and gravimetry is summarized in table 14

Table 14. The lignocellulosic composition of the pulp, fibres, fabrics, and recycled fibres.

Material	Cellulose [%]	Hemicellulose [%]	Lignin [%]
c0 pulp	93.9	5.6	0.5
c0 fibres	93.4	5.7	0.9
c0 fabric, 0w	94.1	5.0	0.9
c0 fabric, 50w	93.6	5.7	0.7
c1 fibres	93.2	6.0	0.8

The cellulose and hemicellulose did apparently not decrease when the material was processed. The amount of lignin was small from the beginning and therefore seemed to fluctuate slightly. However, the gravimetric analysis was not selective for lignin. It measured the amount of undissolved matter in the sample when the polysaccharides had been hydrolysed into monosaccharides and dissolved in diluted sulfuric acid.

Thus, any residual impurities in this measurement were classified as lignin. In comparison, Lê et al. (2016) studied the lignocellulosic composition of pulp used in fibre production with the Ioncell® process. The oven-dry pulp contained 94.9w% cellulose, 4.2w% hemicellulose, and 0.9w% lignin.

Finally, the optical quality of the material was determined by measuring the CIELab values. Table 15 shows the results.

Table 15. The CIELab values of pulp, c0 fibres, c0 fabrics (0w and 15w), and c1 fibres. L stands for black-white balance (or ISO brightness), A stands for green-red balance, and B stands for blue-yellow balance.

Material	L	A	B
c0 pulp	85.8 ± 1.5	-0.8 ± 0.1	2.6 ± 0.1
c0 fibres	86.0 ± 1.5	-0.9 ± 0.2	3.6 ± 0.1
c0 fabric, 0w	76.6 ± 3.1	-0.4 ± 0.2	4.7 ± 0.5
c0 fabric, 50w	79.9 ± 1.4	-0.6 ± 0.1	5.2 ± 0.2
c1 fibres	71.2 ± 2.0	-0.1 ± 0.1	8.3 ± 0.1

The brightness decreased steadily throughout the process. The decreased brightness was notable visually when comparing cycle 0 fibres to cycle 1 fibres. The sooner were pure white and the latter were natural white with a brownish yellow tint. Another study where Ioncell® fibres were produced from pulp showed an ISO brightness of 89% (Lê et al., 2016).

5 Conclusions

This study was conducted to illustrate whether fibres produced with the Ioncell® technology can be chemically recycled, and whether the recycled fibres are of a high enough quality for textile production. During the study, standard Ioncell® fibres were produced by dry-jet wet spinning of a dope made of birch pulp. The fibres were spun into yarn, and knitted into fabric samples, which underwent 50 washing cycles in a laundry machine to mimic a life cycle of a garment. Then, the fabric was ground, dissolved, and regenerated into new fibres. Throughout the process, material samples were frequently taken for quality analysis. The tensile properties and the molecular structure of the fibres were monitored, as well as the polymer degradation and change in chemical composition due to the accumulation of impurities and degradation products.

The results showed a slight deterioration of fibre quality. There was a detectable decrease in tenacity and elongation of the fibres, presumably originating from polymer degradation, and a decrease in crystallinity. Measurements of intrinsic viscosity and molecular weight distribution showed that dissolution and regeneration caused a significant polymer degradation. X-ray diffraction showed a decrease in crystallinity and SEM pictures showed that the recycled fibres were less even than the virgin fibres. A CIELab colour measurement showed that the recycled material acquired a yellow-tinted off-white colour. This means that the Ioncell® fibres are not completely closed-loop recyclable, since the recycling does impact the material quality. However, it is worth mentioning that the recycled fibres still had a higher tenacity than cotton, lyocell, and viscose fibres, as well as a higher elongation than cotton. Hence, it is safe to say the recycled fibres are suitable for textile production.

An unexpected finding was that Ioncell® fabric, besides being recyclable, exhibited a high resilience during the laundry cycles. There was no decrease in quality during the laundering, implying that garments made of Ioncell® fabric can stay in use for long periods of time before being discarded due to breakage, which is the most preferred approach to a sustainable textile economy (European Parliament, 2008).

These findings show that the Ioncell® technology is compatible with a circular textile economy. It enables fibre manufacturers to produce highly durable textile fibres out of waste materials, and even recycle their own fibres. This saves significant amounts of energy (Zamani et al., 2014; Levänen et al., 2021), reduces the amount of waste, and reduces the need to extract virgin natural resources.

This study was conducted on a laboratory scale, so material was lost during the processing and the numerous tests and analyses. Therefore, no reliable information about the yield of the recycling was obtained. Future research may illustrate the material balance and yield optimisation. Moreover, the ionic liquid used in this study was [DBNH][OAc]. Future research may show whether the recyclability is affected by using a different IL.

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