Extraction of Cellulose Nanocrystals via Sulfuric Acid Hydrolysis from Post-Consumer Cotton-Elastane Textile Blends with Elastane Recovery

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Table of Contents

Abstract of Research

An estimated 92 million tons of textiles are discarded yearly, contributing to global environmental crisis. Cotton upcycling presents a promising alternative to landfills or incinerators; cotton can be converted into high-value, biodegradable nanocellulose forms with applications across industries. However, the widespread blending of cotton with elastane (spandex) complicates any reuse, as elastane becomes entangled in machinery during traditional recycling. This research develops a scalable, sustainable upcycling pathway for extracting cellulose nanocrystals (CNCs) from post-consumer cotton-elastane textile blends. First, the sample fabric undergoes selective dissolution of elastane using N,N-dimethylacetamide (DMAc). Through filtration, cotton is separated, DMAc is recovered, and elastane fibers are isolated in a form suitable for further recycling. Sulfuric acid hydrolysis is applied to the separated cotton, producing CNC yields of approximately 55%. Dynamic Light Scattering particle size analysis of the CNCs in suspension showed an average hydrodynamic diameter of 60.1nm, which aligns with CNCs from USDA Forest Products Labs. This pathway outperforms an experimental comparison method, where direct sulfuric acid hydrolysis without prior fabric separation yielded fewer CNCs (50.3%), failed to recover elastane, and produced smaller CNCs with an average size of 51.1nm. Based on current commercial facilities, the developed method of pre-separation and subsequent acid hydrolysis can save 1880 kg (about 11,000 t-shirts) of cotton-elastane waste per day. These findings support a scalable and sustainable route for upcycling cotton-elastane blends, which are otherwise challenging to recycle, advancing toward a circular economy.

1. Introduction

The global fashion industry has expanded rapidly, with fiber production more than doubling from approximately 58 million tons in 2000 to 124 million tons in 2023 [1]. This surge is largely driven by unsustainable business models, fast fashion, and frequent inventory turnover, leading to a projected 160 million tons of fiber production by 2030 if trends persist [1]. These trends result in 92 million tons of textile waste annually [2], yet only a small fraction of textile waste is recycled – less than 1% of fiber production in 2023 was from recycled textiles [1]. In the European Union alone, over 70% of textile waste (8.5 million tons) was either incinerated or sent to landfills in 2019 [3]; incineration releases toxic fumes harmful to surrounding communities and environments, while landfills require valuable space and can take years or centuries for materials to degrade.

One significant recycling challenge involves textiles containing elastane (also known as spandex or LYCRA®), a synthetic fiber made from a polyurethane chain. Known for its elasticity to reversibly extend 400-800% of its length [4], elastane is often added to other fibers like cotton or polyester in small proportions (typically less than 10%) to enhance stretch, flexibility, and comfort. However, this same elasticity that makes elastane popular in apparel complicates conventional recycling processes: during mechanical shredding, elastane fibers can become entangled in machinery, causing jams or breakdowns [5]. As elastane becomes more prevalent in textiles, improving the efficiency and scalability of recycling elastane fiber blends will be an essential component for sustainable textile waste management.

Cotton, a major global fiber accounting for 10% of fiber production (24.7 million tons in 2023) [1], is particularly valued for its softness, breathability, and high absorbency. It is primarily composed of cellulose (>90%), a natural structural polymer existing in plant cell walls that consists of long chains of β-glycosidic linkages, giving it excellent strength and durability [6]. Cotton's high cellulose content compared to other natural sources such as wood (with a cellulose content of <50%) make it an excellent candidate for the upcycling process of nanocellulose extraction, where low-value textile waste is repurposed into a versatile, high-value material [6]. Furthermore, during the initial textile manufacturing process, cotton undergoes scouring and bleaching which removes non-cellulosic impurities such as waxes and pectins, thus simplifying nanocellulose extraction by eliminating the need for additional purification steps [6].

Nanocellulose, the nanoscale form of cellulose, has high mechanical strength, high surface area, and tunable surface chemistry; these properties enable its use in a wide range of industries, from biomedical applications to cosmetics, packaging, and electronics [7]. Nanocellulose is biodegradable, making it an eco-friendly alternative to materials such as microplastics, which are commonly used in personal care products. Among the different forms of nanocellulose, cellulose nanocrystals (CNCs) have enhanced mechanical strength due to their stiffness and thermal

stability [8], and this type will be the focus of this research. CNCs are traditionally produced via sulfuric acid hydrolysis, a chemical process that selectively hydrolyzes the amorphous regions of cellulose and conserves the highly ordered crystalline regions [9], yielding a rod-like nanostructure with diameters of 2-5 nanometers and lengths of approximately 100 nanometers [8].
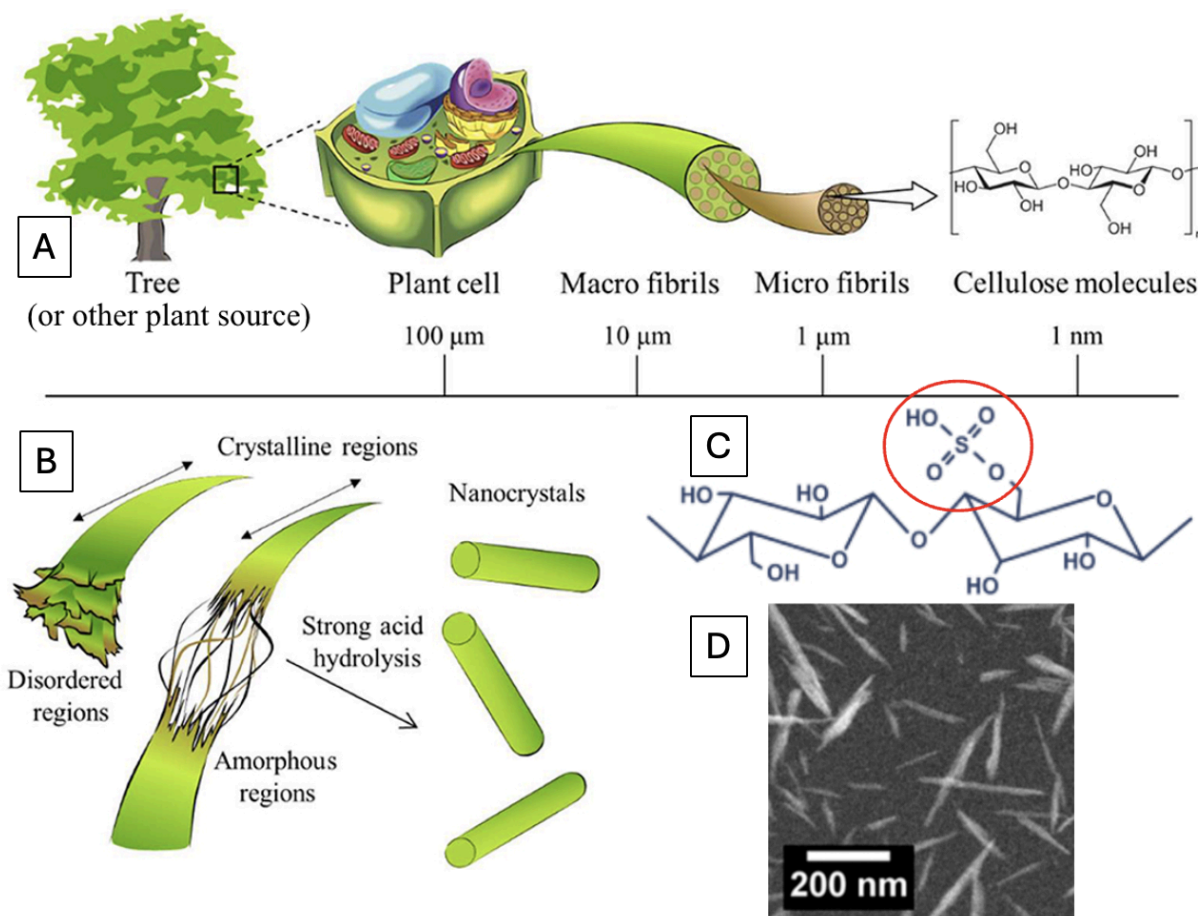

Figure 1. A) Hierarchical breakdown of cellulose from plant to molecule. B) Sulfuric acid hydrolysis of cellulose. C) Molecular structure of CNCs The sulfate half-ester group (circled in red) helps disperse the CNCs in water. D) Scanning Transmission Electron Microscopy of CNCs (credits: Miyashiro et al., CelluForce, Ruiz-Caldas et al.).

Research on nanocellulose extraction from post-consumer textiles is relatively new: a 2024 review identified only 12 studies since 2012, with few examining blended materials and none investigating cotton-elastane blends specifically [6]. To explore this gap, this research will simultaneously address two issues within management of waste textiles: the challenges of recycling elastane and the potential for upcycling cotton through nanocellulose extraction. Given elastane's growing presence in textiles and its impact on recyclability, research into processing cotton-elastane blends is crucial for expanding the potential of nanocellulose extraction from post-consumer textile waste.

2. Engineering Goal

The goal of this research is to develop a scalable, sustainable upcycling pathway for post-consumer cotton-elastane textile blends. Sulfuric acid hydrolysis will be utilized to extract quality, high-value cellulose nanocrystals (CNCs) from the waste cotton. Elastane fibers will be recovered in a form suitable for potential reuse.

3. Fabrics Characterization

A pristine post-consumer blue-dyed cotton-elastane garment, labeled as such on its tag, was donated for this research.

3.1. Separation of fibre types

The initial mass of the fabric was recorded as W_1. The fabric was cut into small pieces (~1 cm²), dried in an oven (100°C) to constant mass, and reweighed (W_2). The difference in mass represents the moisture content of the fabric, which corresponds to the amount of water absorbed from the air under normal conditions without the fabric appearing wet. To separate fiber types, the fabric was treated with N,N-dimethylacetamide (DMAc) at room temperature, using a fabric:acid ratio of 1:50 (g/mL) and a treatment time of 30 minutes, under constant stirring. The solution was filtered through a stainless steel mesh to collect cotton fibers, which are insoluble in DMAc. The cotton was rinsed thoroughly with deionized water and dried in an oven (100°C) to constant mass and weighed (W_3). The procedure was repeated for three batches and results were averaged.

3.2. Determination of fiber content

Fabric composition was calculated based on the following equations:
  Moisture content (wt%) = $(W_1 - W_2)/W_1 \times 100$
  Cotton content (wt%) = $(W_3/W_1) \times 100$
  Elastane content (wt%) = 100 - (cotton content) - (moisture content)
Fabric composition excluding moisture content was also calculated:
  Cotton content (wt%) = $(W_3/W_2) \times 100$
  Elastane content (wt%) = 100 - Cotton content

The average moisture content of the fabric sample was determined to be 5.6%. Cotton, known for its high absorbency, typically exhibits a moisture content of ~7%, while elastane has a much lower moisture content of <1%. This aligns with the expectation that a predominantly cotton-based cotton-elastane blend would have a slightly lower overall moisture content than

pure cotton. The average fiber composition of the fabric was calculated as 88.9% cotton and 5.5% elastane, with a moisture content of 5.6%. When moisture content was excluded, the fabric composition was determined to be 94.2% cotton and 5.8% elastane. These values closely mirror the proportions indicated on the product label.

3.3. Fourier transform infrared spectroscopy (FTIR) Analysis.

FTIR spectroscopy was employed to analyze the initial fabric blend, its separated components, and to confirm the fiber types as cotton and elastane. The spectrum of the pre-separation fabric blend closely resembles that of cotton due to the high cotton content in the material. To obtain the elastane spectrum, a sample of the elastane-DMAc solution, collected after filtering out the cotton, was analyzed. The solution was placed on the FTIR, and the DMAc was allowed to evaporate, leaving the elastane for analysis. The distinctive peaks and bands corresponding to both fiber components were identified in their respective FTIR spectra, confirming the successful separation and the identities of the fibers as cotton and elastane.
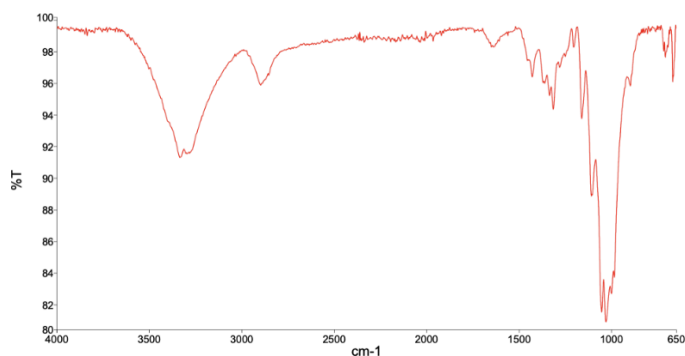

Figure 2. . FTIR spectrum of pre-separation blend. The x-axis represents wavenumber, and the y-axis is percent transmission. This spectrum resembles cotton due to the high cotton content in the material.
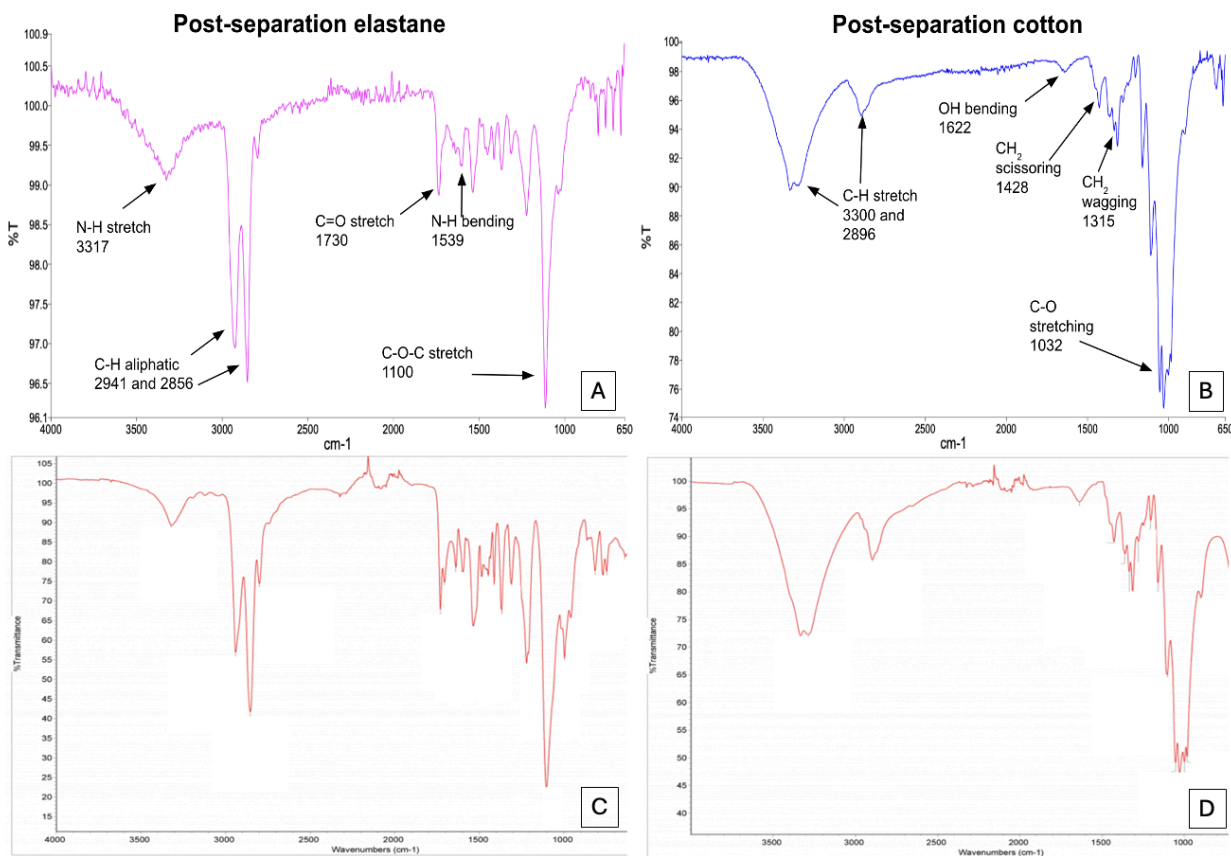
Figure 3. The FTIR spectra of the separated elastane (A) and cotton (B) components with important peaks labelled. Below (C & D) are the fiber's corresponding spectra from an FTIR library to confirm fiber identity. Notably, elastane contains nitrogen; the FTIR spectrum of elastane shows N-H stretching at 3317 and N-H bending at 1539. One of the most distinct peaks of cotton is the C-O stretching at 1032. FTIR library credit: Institute of Chemistry University of Tartu, Estonia

3.4. Confirmation of Successful Separation of Fiber Types.

Confirming successful removal of elastane from the cotton-elastane blend was important for the accuracy of the fiber content determination in addition to the effectiveness of the experimental method of separation before acid hydrolysis. Scanning electron microscopy (SEM) was used to visually compare the fabric before and after the separation process, as well as examine the surface details of elastane. The initial cotton-elastane blend (upper left) shows the elastane fibers crossing and weaving between the cotton fibers to provide stretch to the garment. There is a layering effect where the elastane creates a web-like network across the cotton base. Once the elastane is removed via selective dissolution in DMAc, the ribbon-like structure of the parallel cotton fibers is more clearly illustrated (upper right). Additionally, the close-up image of elastane reveals its rough surface texture. Energy dispersive spectroscopy (EDS) was conducted on the cotton-elastane blend pre-separation and the fabric post-separation. The elastane consists of a polyurethane synthetic polymer containing carbon, nitrogen, and oxygen. Cotton, on the other hand, contains carbon and oxygen and lacks nitrogen. EDS analysis of the pre-separation

fabric shows that before the separation, there is carbon, nitrogen, and oxygen, with the nitrogen indicating the presence of elastane. After separation, the sample contains carbon and oxygen. The absence of nitrogen (Figure 9, circled in red) supports the conclusion that the elastane was successfully separated from cotton. The combination of visual (SEM) and chemical (EDS) analysis provides evidence that the separation process effectively removes elastane from cotton-elastane blended fabrics, resulting in pure cotton fibers that maintain their characteristic ribbon-like structure.
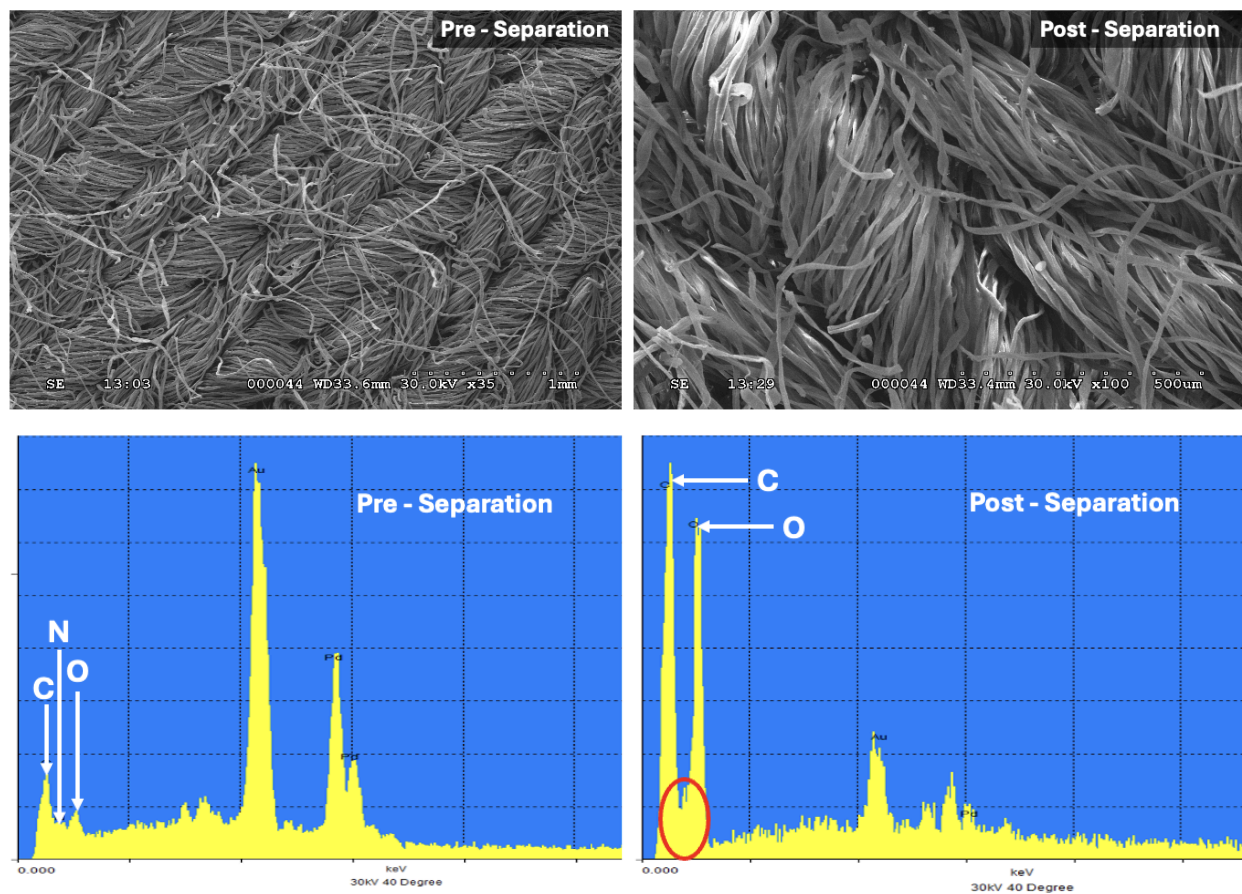

Figure 4. SEM images (upper images) of the cotton‑elastane fabric before and after separation, revealing the transition from a web‑like structure with elastane fibers to ribbon‑like cotton fibers. EDS spectra (lower images), with peak heights representing relative amount, highlight the disappearance of nitrogen post‑separation (circled in red), confirming successful removal of elastane from the blend.

4. Experimentation of Sulfuric Hydrolysis Pathways

4.1. Baseline Sulfuric Acid Hydrolysis Procedure

Sulfuric acid hydrolysis has shown to be an effective method for producing quality CNCs from cotton, certain cotton blends, and other cellulose sources. The fabric first underwent simple pretreatment to gently clean and increase surface area. The sample was washed with laundry soap, rinsed thoroughly with deionized water, and air-dried completely. The textile fragments were cut into small square pieces with a side length < 1 cm.

Next, sulfuric acid hydrolysis was performed. A 64 wt% sulfuric acid solution was prepared at 50°C. Fabric pieces were added to the acid solution at a ratio of 1:10 (g fabric/mL acid), which corresponds to a textile mass concentration of 6.1 wt%. The reaction mixture was stirred at 300 rpm with a mechanical stirrer for 90 minutes. The reaction was quenched by 10-fold dilution with cold deionized water.

Subsequent steps were completed to neutralize excess acid and purify the CNCs. The suspension was centrifuged at 14000 rpm for 10 minutes. Supernatant was discarded and the precipitate was resuspended in deionized water. The centrifugation and washing cycle was repeated three times in order to remove most of the excess of acid. The resultant precipitate in gel form containing the CNCs and residual acid was suspended and transferred to a Thermo Scientific Slide-A-Lyzer Dialysis Cassette with a Molecular Weight Cut Off (MWCO) of 3.5k Da. The dialysis membrane was placed in a large container of deionized water, which was changed daily. The suspension was dialyzed against DI water until the conductivity of the water from successive washes was below 2 μS/cm. To further disperse the CNCs, the suspension was sonicated using an ultrasonic homogenizer (35 W, Qsonica) at 50% of amplitude for 2 minutes. The suspension was vacuum filtered with a glass microfiber filter (particle retention 0.5 μm). The final product is a suspension of CNCs with sulfate-half ester groups in acid form ($-OSO_3H$) on the CNC surface.

4.2. Cotton-Elastane Experimentation

This study examines two pathways for extracting cellulose nanocrystals (CNCs) from cotton-elastane textile blends, while also recovering elastane for potential recycling. Both pathways employ sulfuric acid hydrolysis, following methods established in previous studies, but differ in the timing of fiber separation. The methods were compared based on elastane recovery and CNC composition, morphology, and yield.

In the first method, hydrolysis is performed on the cotton-elastane blend, and elastane is recovered afterward via filtration. This has been demonstrated previously with cotton-polyester and cotton-acrylic blends. In the second method, cotton and elastane are separated prior to hydrolysis, and only the cotton is subjected to the sulfuric acid treatment. Separation is accomplished through selective dissolution of elastane in DMAc at room temperature for 30 minutes. Using fabric from the same garment controls for variables such as dye, weave, and processing history.
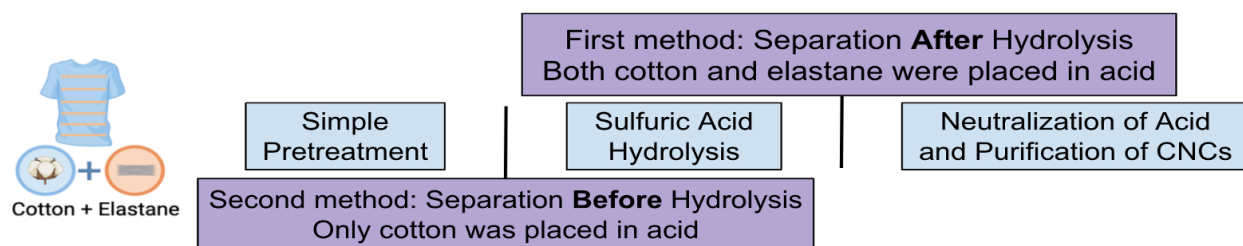

Figure 5. Experimental pathways for cotton-elastane showing separation within the hydrolysis procedure.

4.3. Elastane and DMAc Recovery Comparison

In the method of separation after hydrolysis, elastane was not recoverable upon filtration of the post-hydrolysis solution. EDS of the final CNC suspension detected small amounts of nitrogen, indicating contamination by elastane. Interestingly, previous studies have demonstrated high recovery rates for synthetic fibers such as polyester or acrylic this method. However, these synthetics are present in larger quantities in textiles and are more resistant to degradation under highly acidic conditions. Elastane, by contrast, has a distinct chemical composition and properties compared to other synthetics. It is more susceptible to degradation under sulfuric acid, making it less recoverable through filtration. As a result, this method is not suitable for effective elastane recovery.

In contrast, the method of separation before hydrolysis performed significantly better for elastane recovery. All elastane was successfully dissolved in DMAc and subsequently recovered after the cotton was separated. The recovered elastane was in a form suitable for further recycling. For example, the polyurethane chains in elastane could be depolymerized into poly(tetramethylene ether) glycol (PTMG). Additionally, the DMAc solvent was recovered in a pure condition. After evaporating DMAc on the FTIR, no elastane residue was detected, confirming that the recovered DMAc was pure and could be reused for cotton-elastane separation. This reduces the cost of CNC production using the method of separation before hydrolysis, particularly on a large scale, while significantly minimizing the ecological toxicity and overall environmental impact of CNC production. Separation before hydrolysis was concluded to be a superior method for elastane recovery compared to separation after hydrolysis.

4.4. FTIR Analysis of Final CNCs

The final CNCs from the two experimental methods were compared via FTIR analysis (left). To obtain the spectra, the CNC suspensions were placed on the FTIR and the water evaporated, enabling analysis of the air-dried CNCs. The CNCs retain the same major peaks as the separated cotton. For instance, the C-O stretching can be seen at 1032 wavenumber. These spectra match quality CNCs produced from pure cotton, cotton-polyester, and cotton-acrylic blends in Ruiz-Caldas et al. (right). Thus, previous blending with elastane does not have an impact on the composition of the resulting CNCs. The sample garment in this research had a blue dye, but previous research investigating the impact of dye on CNC quality shows no impact.
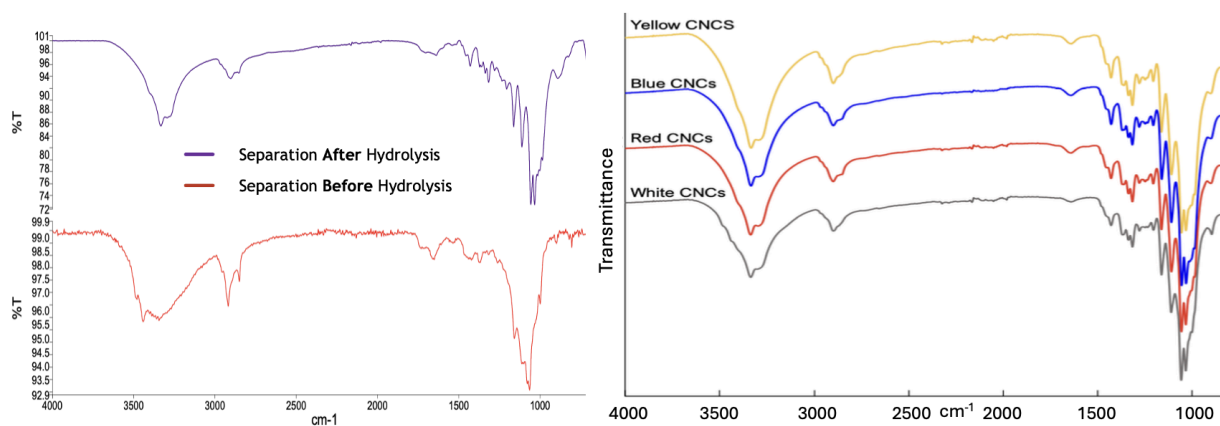

Figure 6. Left: FTIR spectra of final CNCs obtained from the two experimental pathways. Right: FTIR comparison of quality CNCs produced by Ruiz-Caldas et al [10].

4.5. Comparison of CNC Morphology

This research utilized Dynamic Light Scattering (DLS) to estimate the hydrodynamic diameter of CNCs. The hydrodynamic diameter represents the size of a sphere that moves at the same rate as the CNCs under Brownian motion. While there is no universally agreed-upon hydrodynamic diameter for CNCs, DLS provides a reliable method for comparing particle sizes between samples. The CNC sizes from the two methods were similar, measuring 51.1 nm and 60.1 nm. Both methods resulted in CNCs with slightly smaller diameters compared to those reported in previous studies. The observed size differences between studies may stem from variations in sulfuric acid hydrolysis duration, as shorter reaction times can yield larger CNCs. Notably, the CNCs from the method involving separation before hydrolysis fell within the size range of high-quality CNCs produced by the USDA Forest Products Laboratory pilot plant. This highlights that CNCs derived from cotton-elastane blends have the potential industrial production quality.
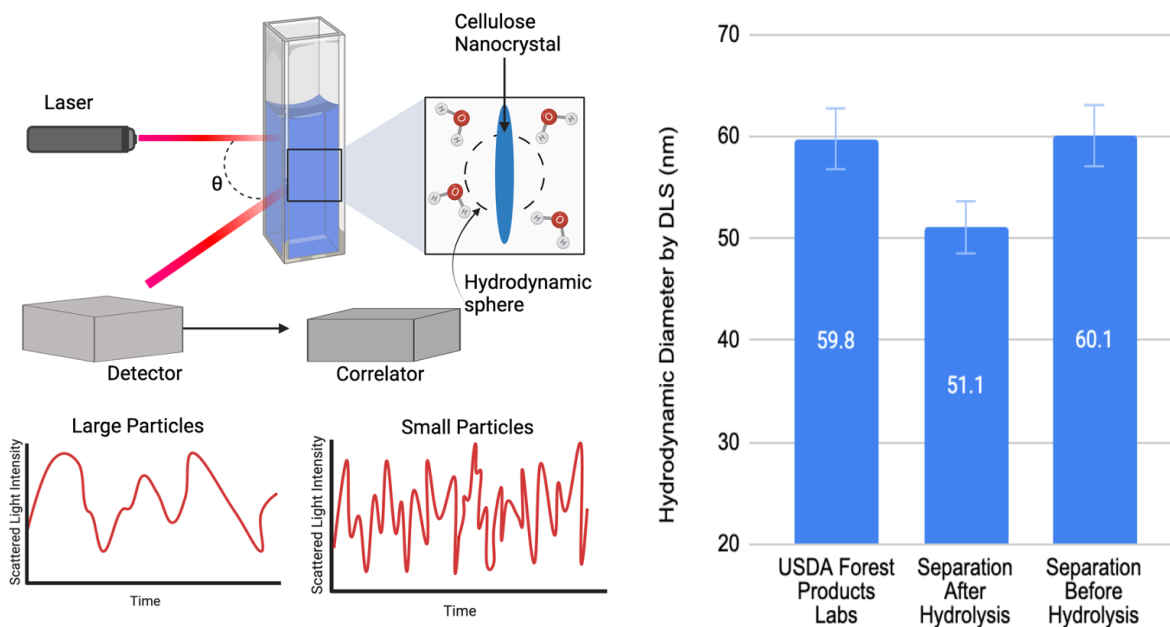
Figure 7. (Left) Diagram illustrating the working principle of DLS particle sizing. **Figure 8**. (Right) Comparison of average hydrodynamic diameter of CNC from this study compared to quality CNCs produced by the USDA Forest Service's pilot plant.
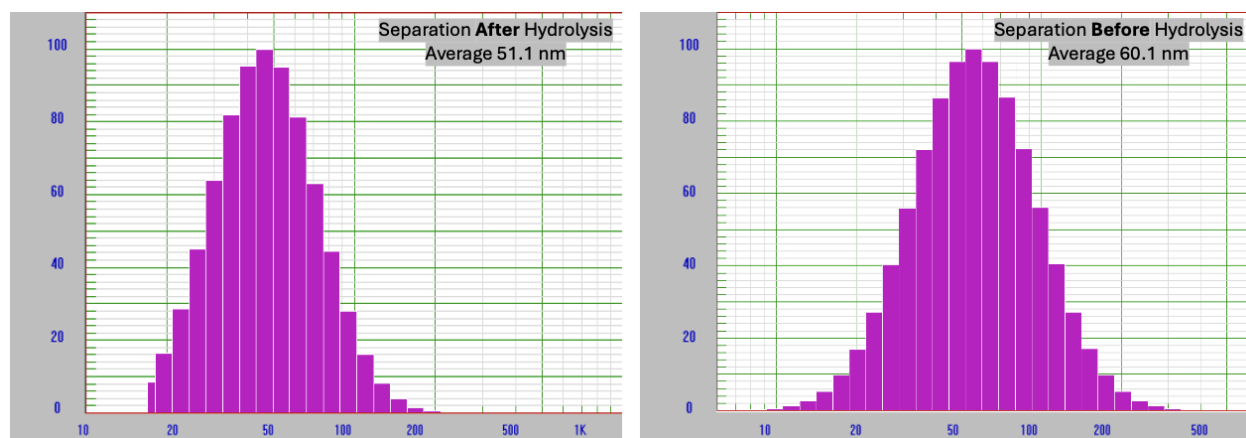

Figure 9. Gaussian distributions of hydrodynamic diameter, with the x-axis representing hydrodynamic diameter and the y-axis indicating relative number of nanoparticles.

5. Innovative Determination of CNC Yield

5.1. Freeze-drying

Freeze-drying removes water from the CNC suspension, enabling the measurement of the CNC in powder form. However, the vacuum pump on the high school research lab's freeze dryer

broke, so freeze-drying was not accessible. To address this limitation, the first attempt of a workaround was a "two-step freeze-drying method." A small volume of the CNC suspension was frozen at -80°C and then placed in a vacuum chamber to sublimate the water. However, if the water began to melt during the process, CNCs could be inadvertently removed by the vacuum, leading to inaccurate measurements of CNC mass and thus yield.

5.2. Innovative CNC Quantification using FTIR Analysis

Due to inaccessibility to freeze-drying and inaccuracies in the two-step freeze-drying method, a creative quantification approach using FTIR spectroscopy was developed.

Purified quality CNCs were purchased to create calibration standards. A 10 g/L solution was prepared and serially diluted. For each standard, 5 µL of sample was analyzed in triplicate on the FTIR. After water evaporation, the FTIR spectrum of the remaining CNCs was analyzed, with the characteristic peak area integrated from 850 to 1500 cm^{-1}.
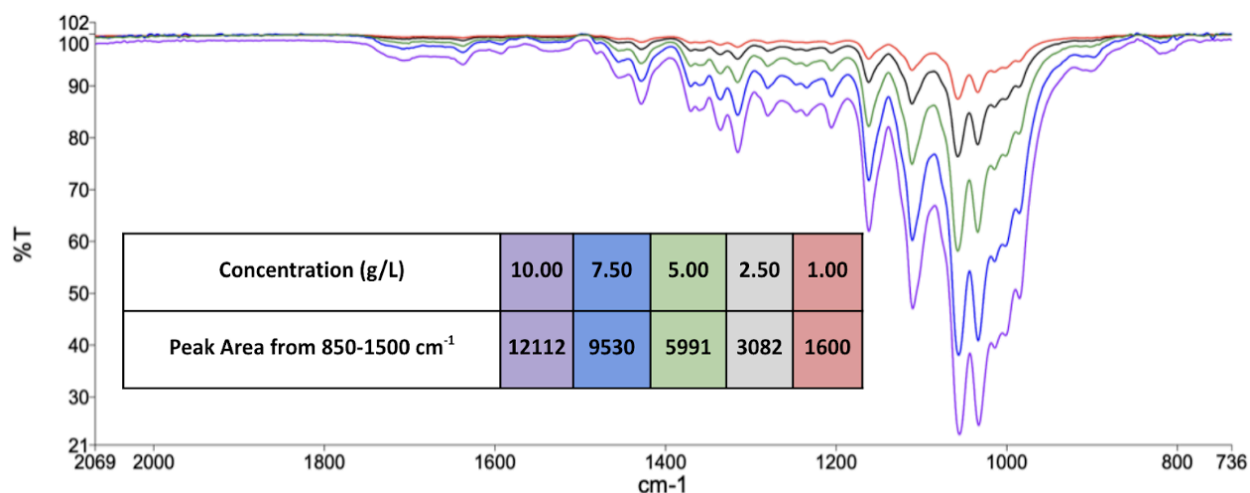

Concentration (g/L)	10.00	7.50	5.00	2.50	1.00
Peak Area from 850-1500 cm^{-1}	12112	9530	5991	3082	1600

Figure 10. FTIR of calibration standards, along with a table of concentrations and peak areas. Color coded columns correspond to respective spectra.

A calibration curve was generated by plotting CNC concentration (x-axis) versus peak area (y-axis). The resulting well-fit linear regression allowed determination of unknown sample concentrations. The total extracted CNC mass was calculated by multiplying the determined concentrations by the total sample volume. CNC yield was calculated by dividing this mass by the weight of cotton in the original sample, which was determined using the proportions established in the fiber content study. The method involving fiber separation prior to sulfuric acid hydrolysis achieved a 55.3% yield, compared to only 50.7% for post-hydrolysis separation. These findings align with previous studies reporting reduced CNC yields when cotton is hydrolyzed in the presence of other fibers. The superior CNC yield (cotton basis) demonstrates

that separating cotton and elastane before sulfuric acid hydrolysis is the more effective approach for maximizing CNC production.
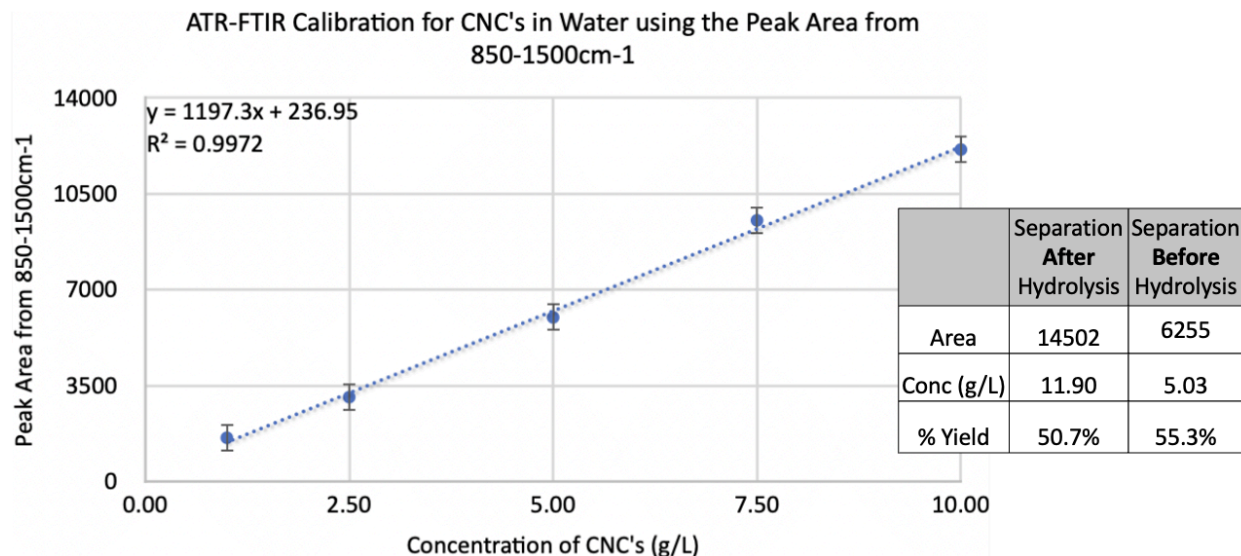

Figure 11. Left: Calibration curve with linear regression for CNC concentration determination. Right: FTIR peak areas for unknown samples with corresponding calculated CNC concentrations based on the calibration curve. These concentrations enabled quantification of CNC yield from experimental pathways.

6. Discussion and Conclusions

6.1. Proposed Upcycling Pathway
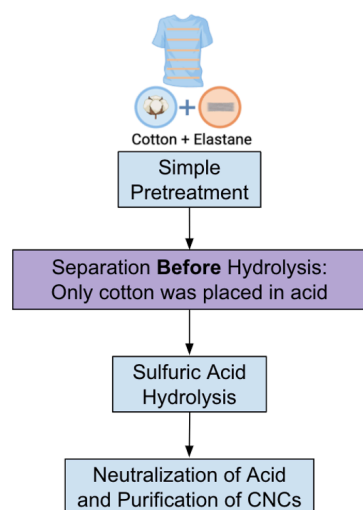
This pathway for cotton-elastane is designed for integration with existing industrial processes. The simple pretreatment eliminates the risk of elastane entanglement in recycling machinery because it cuts into small pieces instead of mechanical shredding.

Cotton is separated from elastane before hydrolysis via DMAc. Elastane is recovered in a form suitable for further recycling. DMAc solvent is recovered and reused for cotton elastane separation.

Pre-separation enables higher CNC yields (55.3%) compared to post-hydrolysis separation (50.7%). The final CNCs exhibit desirable nanoscale properties.

6.2. Two-fold Impact

The impact of this pathway is two-fold because it presents a textile waste solution that is both sustainable and scalable.

Sustainability: This research expands the potential for cellulose nanocrystal extraction upcycling to previously problematic cotton-elastane blends. Nearly complete recovery of elastane is achieved in a form suitable for recycling. Ecological toxicity is minimized due to DMAc solvent recovery and reuse. Environmental impact can be further reduced through efficient sulfuric acid recovery systems. Improved yield of the cellulose nanocrystals indicates the pathway's efficiency to convert cotton waste into cellulose nanocrystals. Cellulose nanocrystals are biodegradable and can help reduce reliance on plastics. The upcycling process avoids environmental harm and health risks associated with textile waste disposal. This process could prevent approximately 1,880 kg of cotton-elastane waste per day (equivalent 11,000 t-shirts) from entering landfills or incinerators, based on current commercial capacity.

Scalability and Economic Opportunity: There is a growing interest in cellulose nanocrystals, as evidenced by the USDA Forest Service's pilot plant and emerging companies. Using post-consumer cotton and cotton blends as an incredibly cheap source for cellulose nanocrystals avoids high disposal and land use fees associated with textile waste management. Little heat is needed for sulfuric acid hydrolysis procedure, lowering cost. Recovery of reagents (DMAc for cotton-elastane separation, sulfuric acid) drastically reduces cost. High-quality cellulose nanocrystal production paired with elastane recovery creates dual value streams. Recovered synthetics (elastane, polyester, or acrylic) remain suitable for further recycling applications. Cellulose nanocrystals are high-value with applications across diverse industries: packaging, electronics, personal care products, paints, paper, agriculture, biomedical, and more. This research contributes to circular economy goals in textile waste management.

7. Future Work

Future research will investigate CNC production from cotton-polyester-elastane blends, conduct a life-cycle assessment of the cotton-elastane upcycling pathway, and study efficient ways to further recycle elastane.

8. Acknowledgements

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