

Introduction

Globally, 92 million tons of textile waste are generated annually, driven by fast fashion and unsustainable business practices. In the US, 85% of discarded textiles are landfilled or incinerated, costing \$4 billion per year. Landfills consume space, and textiles take centuries to decompose, leaching chemicals into soil and water. Incineration releases toxic fumes, posing health risks and contributing to greenhouse gas emissions. Cotton is 90% cellulose, making it viable for upcycling into cellulose nanocrystals (CNCs), a form of nanocellulose. CNCs are produced via sulfuric acid hydrolysis, a chemical process that selectively hydrolyzes the amorphous regions of cellulose and conserves the highly crystalline regions, yielding rod-like nanostructures. CNCs are biodegradable and have applications in biomedicine, personal care, packaging, electronics, and other diverse industries. The US Forest Service has a pilot plant for CNC production, and a commercial runs with a capacity of 1 metric ton per day. Both these facilities use sulfuric acid hydrolysis, demonstrating that CNC production is scalable; however, it requires adjustments to unlock cotton as a source for sustainable nanomaterials.

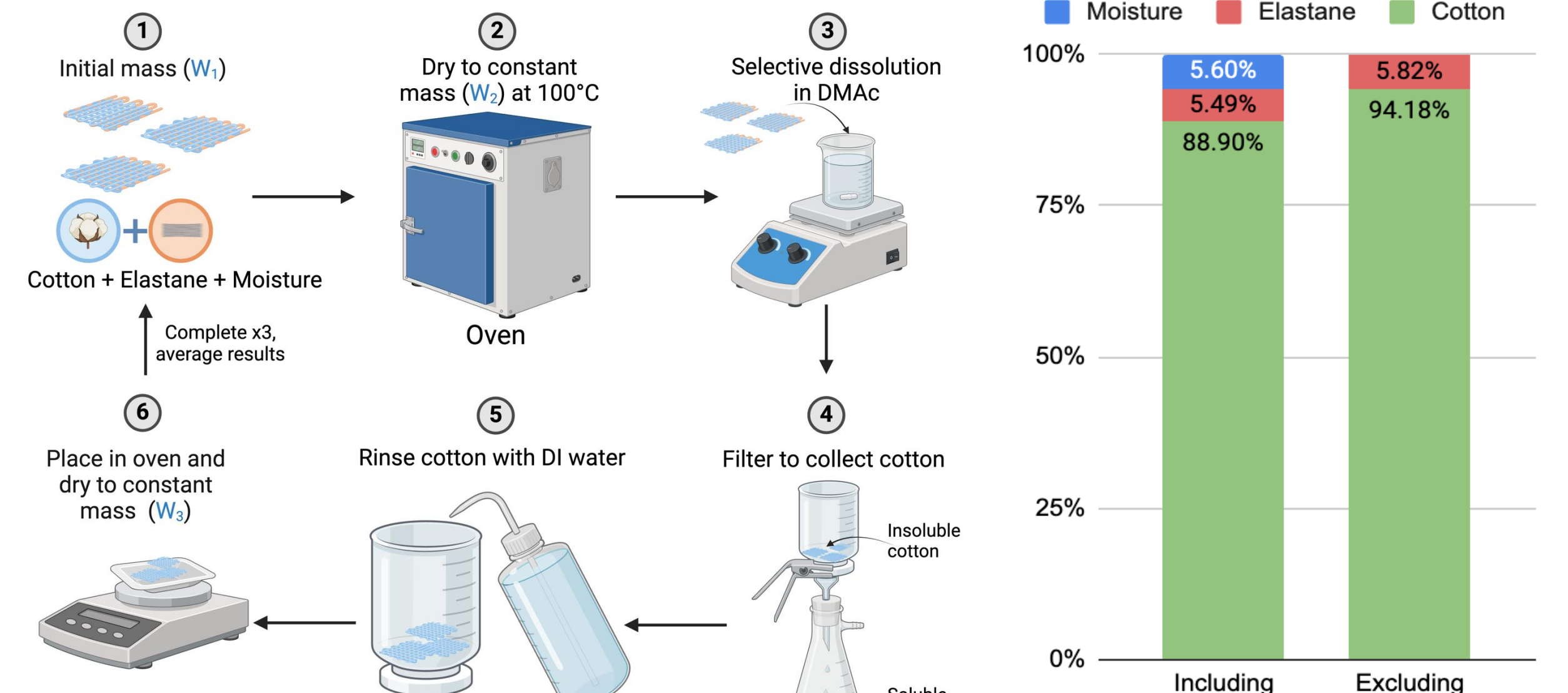
Previous research has examined CNC extraction from pure cotton and cotton blended with polyester or acrylic. My research takes this a step further by considering cotton-elastane blends. Elastane (also known as spandex or LYCRA®), is a synthetic fiber able to reversibly extend 400-800% of its length. It is often added to other fibers like cotton or polyester in small proportions (typically less than 10%) to enhance stretch, flexibility, and comfort. However, elastane tangles in machinery during the mechanical shredding stage of conventional recycling processes, causing jams or breakdowns. Thus, cotton-elastane is non-recyclable.

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.

Fabric Characterization

A pristine post-consumer blue-dyed cotton-elastane garment, labeled as such on its tag, was donated for this research. To determine the fiber content, the components of the fabric were separated by treatment in 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 initial drying of the fabric enabled calculation of the moisture content, which corresponds to the amount of water absorbed from the air under normal conditions without the fabric appearing wet. The average moisture content aligns with the expectation that a predominantly cotton-based cotton-elastane blend would have a slightly lower overall moisture content than pure cotton (~7%). The composition of 94.18% cotton and 5.82% elastane (excluding moisture) closely mirrors product labelling. FTIR spectroscopy was employed to analyze the initial fabric blend and its separated components, and to confirm fiber identity. 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 evaporated, 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.

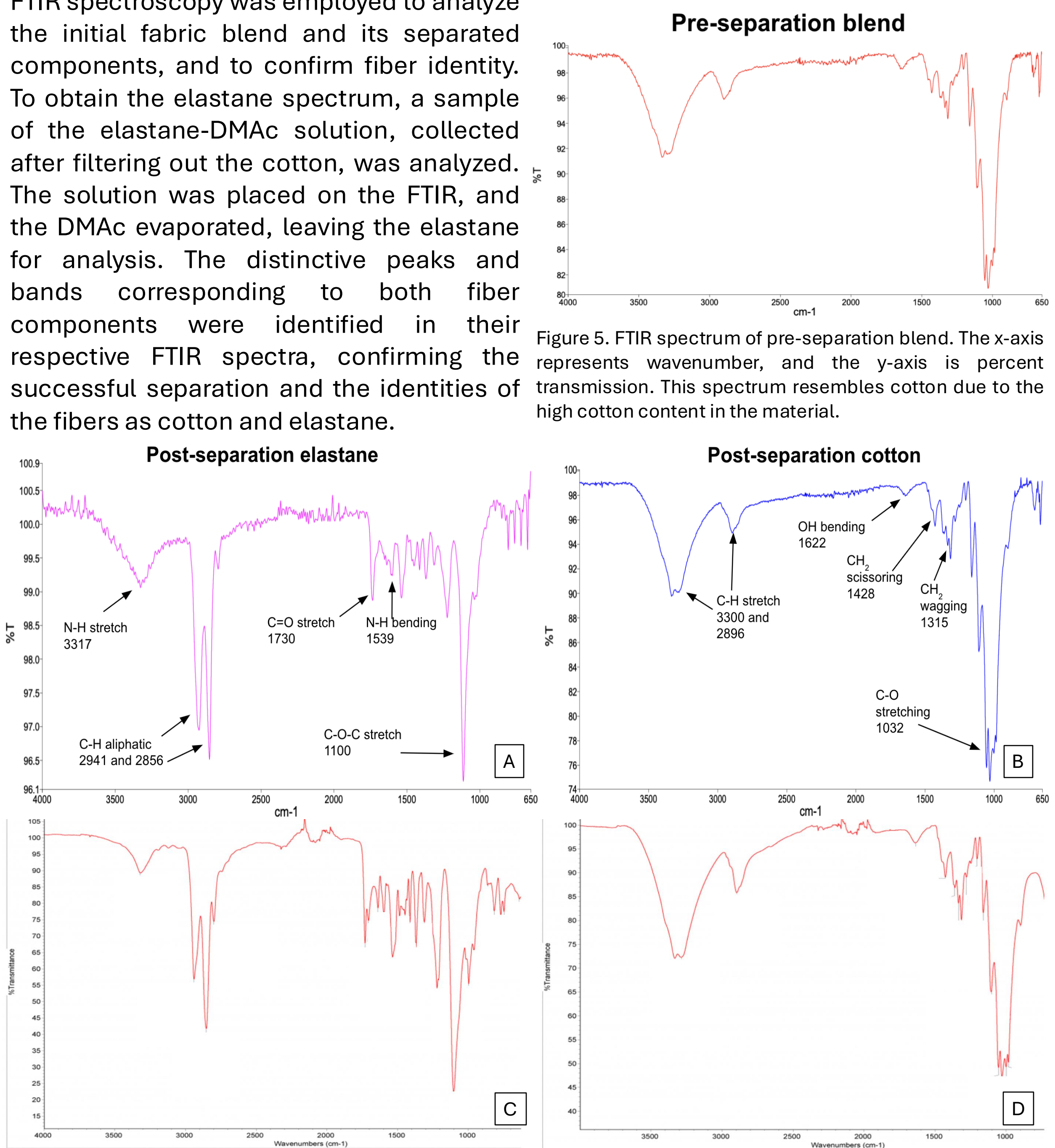
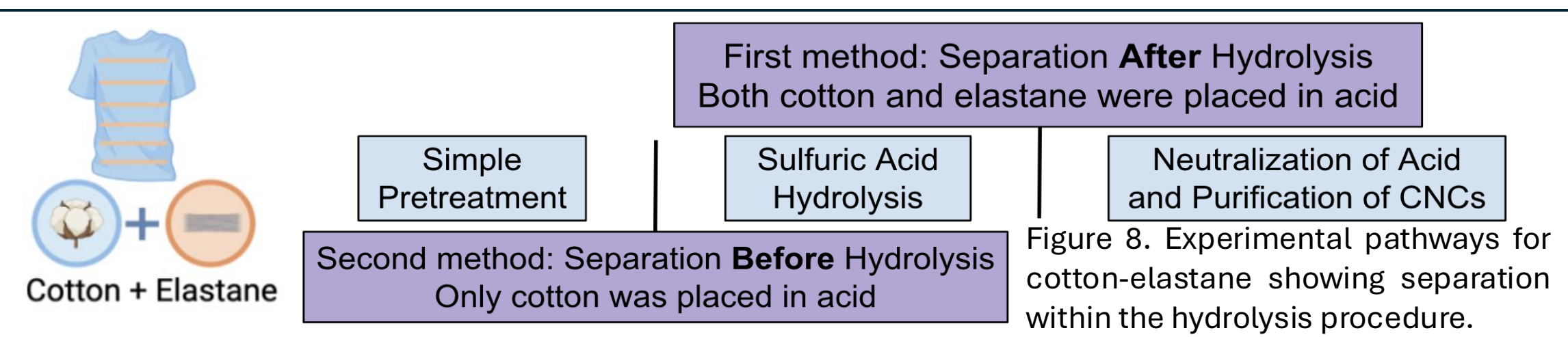
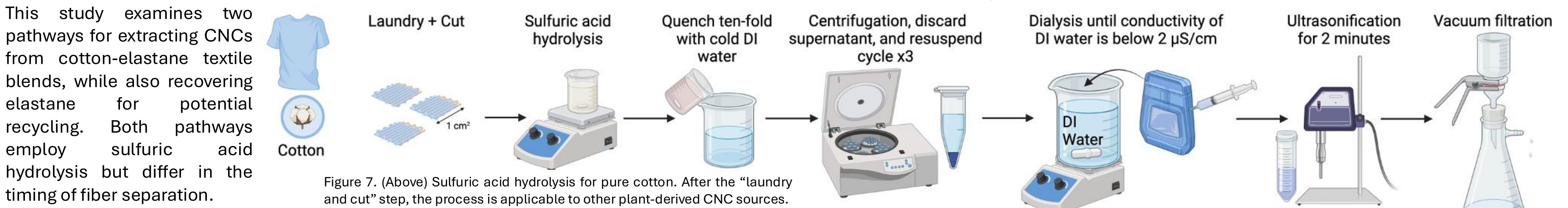


Figure 6. The FTIR spectra of the separated elastane (A) and cotton (B) components with important peaks labelled. Below (C & D) are the fiber's corresponding spectra from an FTIR library to confirm fiber identity. Notably, elastane contains nitrogen; the FTIR spectrum of elastane shows N-H stretching 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.

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

Experimentation of Sulfuric Hydrolysis Pathways

Sulfuric acid hydrolysis has shown to be an effective method for producing quality CNCs. First, the fabric undergoes simple pretreatment to gently clean and increase surface area. Sulfuric acid hydrolysis is performed with optimized parameters (64% sulfuric acid, 50°C, fabric:acid ratio of 1g:10mL, 90 minutes) and quenching stops the reaction. The subsequent steps neutralize excess acid and purify CNCs. The final product is a suspension of CNCs with sulfate-half ester groups in acid form ($-\text{OSO}_3\text{H}$) on the CNC surface.



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.

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 (leftmost) 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. 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 (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.

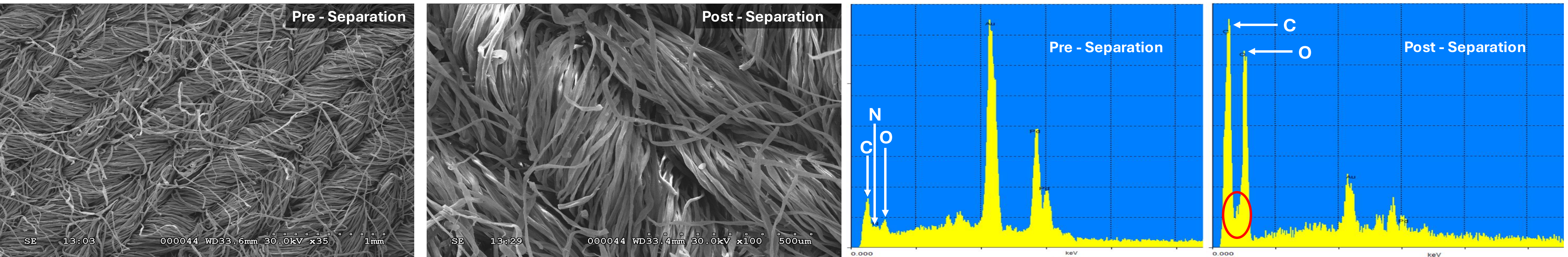
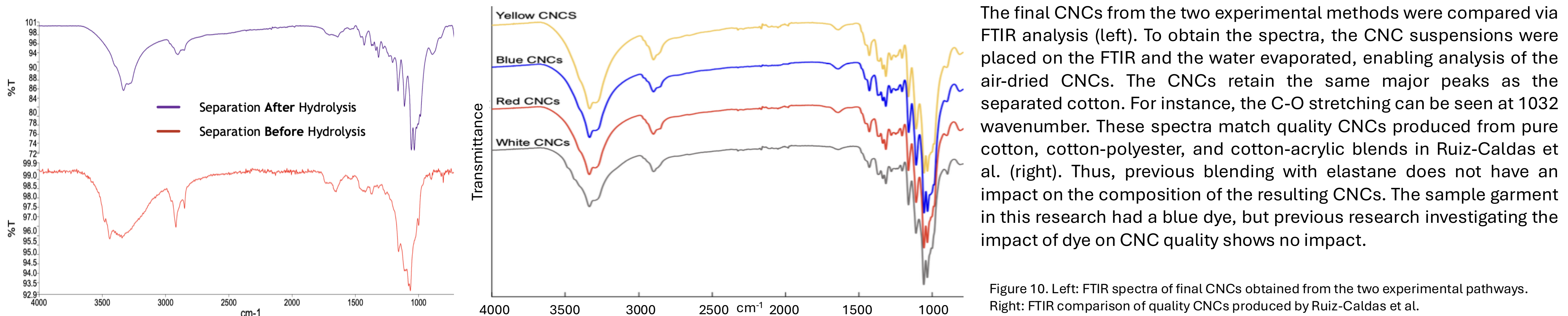


Figure 9. SEM images (left) of the cotton-elastane fabric before and after separation, revealing the transition from a web-like structure with elastane fibers to ribbon-like cotton fibers. EDS spectra (right), with peak heights representing relative amount, highlight the disappearance of nitrogen post-separation (circled in red), confirming successful removal of elastane from the blend.

Compositional Comparison



Morphological Comparison

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 can have industrial quality.

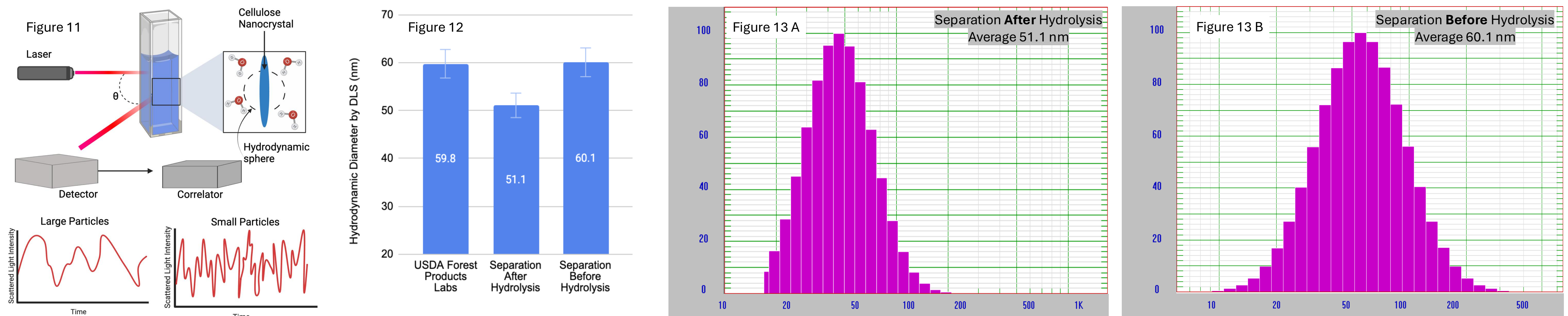


Figure 11. Diagram illustrating the working principle of DLS particle sizing. Figure 12. Comparison of average hydrodynamic diameter of CNC from this study compared to quality CNCs produced by the USDA Forest Service's pilot plant. Figure 13 A&B. Gaussian distributions of hydrodynamic diameter, with the x-axis representing hydrodynamic diameter and the y-axis indicating relative number of nanoparticles.

Elastane and DMAc Recovery

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.

Innovative Determination of CNC Yield

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

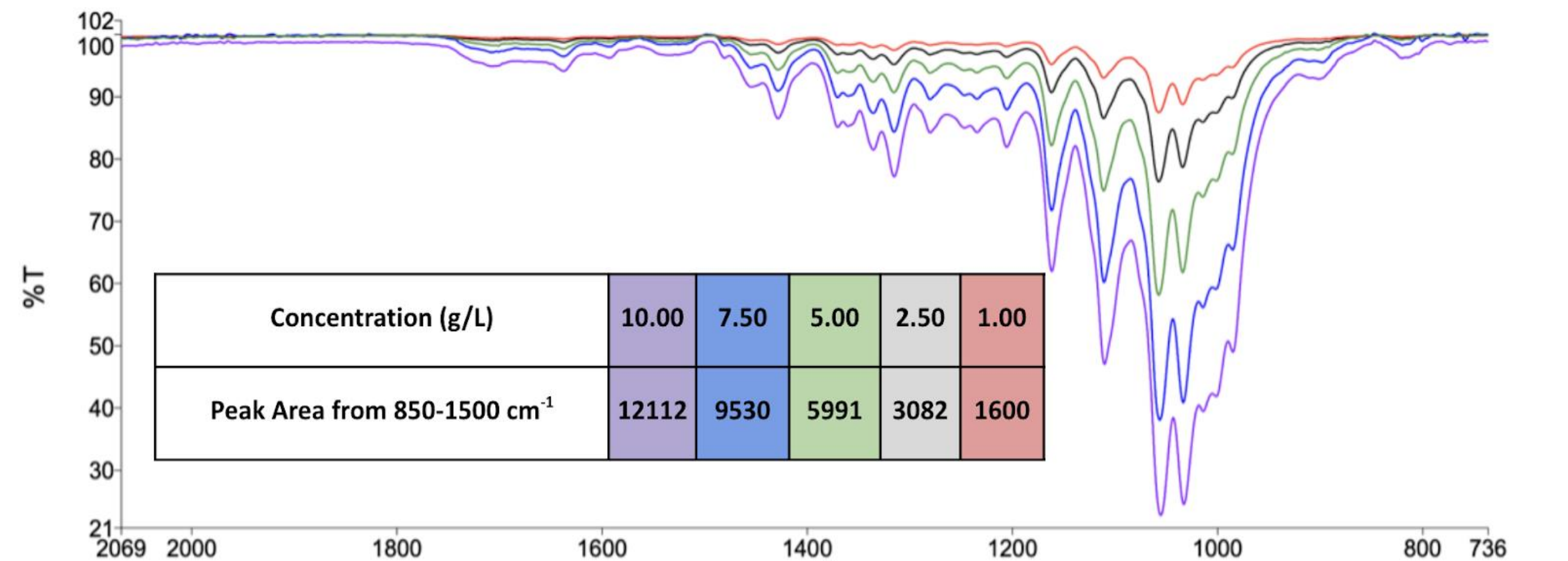


Figure 15. FTIR of calibration standards, along with table of concentrations and peak areas. Color coded columns correspond to respective spectra.

Purified quality CNCs were prepared 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} . 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.

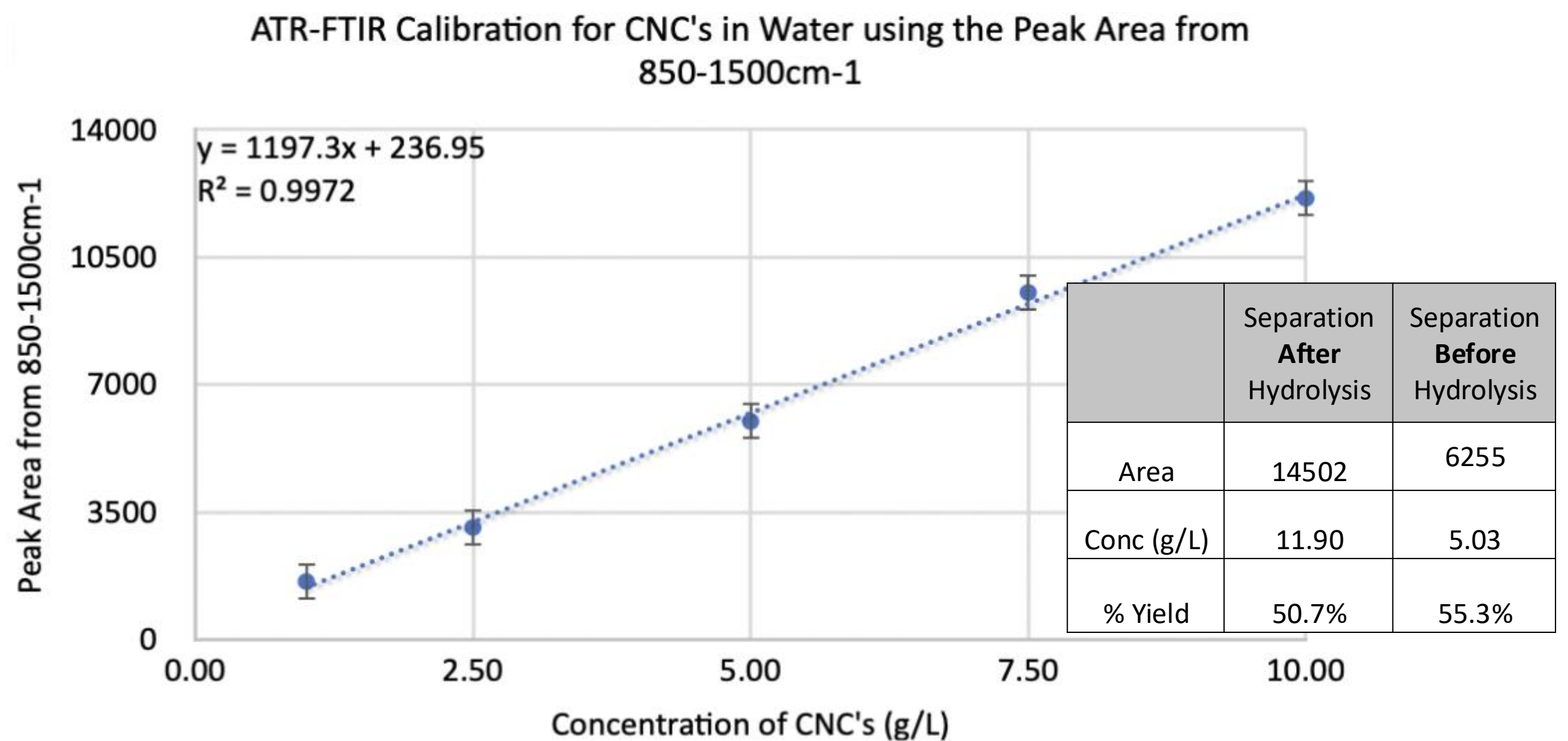
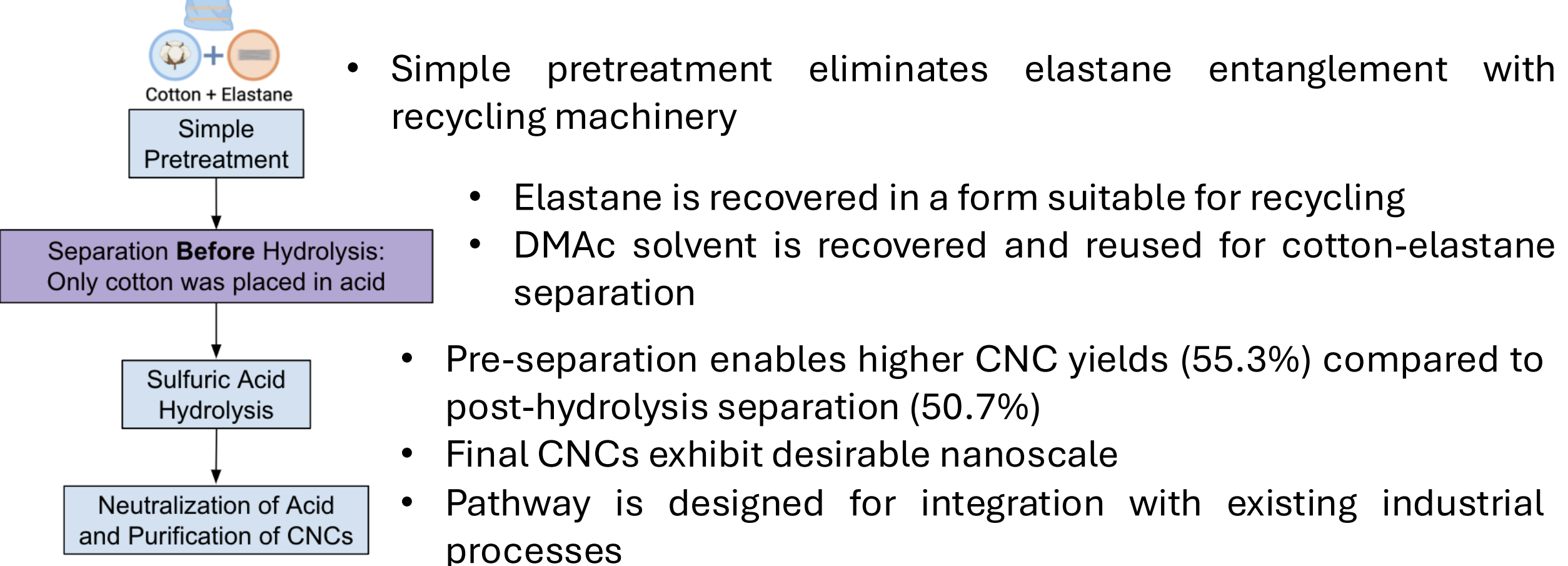


Figure 16. Left: Calibration curve with linear regression for CNC concentration determination. Right: FTIR peak areas for unknown samples with corresponding calculated CNC concentrations based on the calibration curve. These concentrations enabled quantification of CNC yield from experimental pathways.

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.

Discussion and Conclusions

Proposed Upcycling Pathway



- Simple pretreatment eliminates elastane entanglement with recycling machinery
 - Elastane is recovered in a form suitable for 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%)
 - Final CNCs exhibit desirable nanoscale
 - Pathway is designed for integration with existing industrial processes
- ### Two-fold Impact
- #### Sustainability

 - Expands the potential for cellulose nanocrystal extraction upcycling to previously problematic cotton-elastane blends
 - Cellulose nanocrystals are biodegradable and can help reduce reliance on plastics
 - Improved yield indicates the pathway's efficiency to convert cotton waste into cellulose nanocrystals
 - Nearly complete recovery of elastane is achieved in a form suitable for recycling
 - Minimized ecological toxicity due to DMAc solvent recovery and reuse
 - Environmental impact can be further reduced through efficient sulfuric acid recovery systems
 - Avoids environmental harm and health risks associated with textile waste disposal
 - 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 & Economic Opportunity

 - Growing interest in cellulose nanocrystals, as evidenced by the USDA Forest Service's pilot plant and emerging companies
 - Avoids high disposal and land use fees
 - Post-consumer cotton and cotton blends are incredibly cheap source for cellulose nanocrystals
 - 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
 - Contributes to circular economy goals in textile waste management

Future Research

Future research should 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