

Muslin Deweaving through Combined Mechanical, Thermal and Chemical Methods

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Abstract: Fabric waste has become an escalating problem that stems from the ever-shortening clothing lifecycle. Previous cotton recycling processes used mechanical methods to break the cotton down into fiber; this comes at the cost of compromised strength. Sodium hydroxide has long been used in the textile industry to increase dye absorption and luster through mercerization. In this paper, the deweaving of cotton muslin fabric was attempted using the chemical interactions of NaOH in combination with heat and mechanical forces through agitation. Different NaOH concentrations were tested to determine the optimum condition for fabric decomposition on a laboratory scale. Overall, the muslin fabric treatment with 0.5 M NaOH yielded the most promising results for fiber quality retention and chemical usage. The NaOH solution was shown to be feasible in effectively deweaving multiple muslin fabrics consecutively. While the deweaving process reduces the mechanical strength of the fabric, overall, the recycling method was successful in minimizing chemical waste and deweaving time.

Keywords: Fabric recycling, NaOH, Muslin deweaving, FTIR spectroscopy, Tensile strength

Introduction

Fabric waste production has been steadily growing and has increased by nearly 300 % from 1990 to 2018 [1,2]. This spike in textile waste has been spurred by trends in the garment sector, such as fast fashion, where consumers purchase and discard clothes at a higher rate than before [3]. The recycling of cellulose-based fibers is of particular interest as the cellulose derivative cotton accounts for a third of all textile fibers [4]. Some cellulose recycling processes use mechanical means to break down the fabric; this process causes compromised strength and quality and necessitates blending the recycled material with virgin fibers, thus limiting the number of times a fabric can be recycled [5,6]. Other cellulose recycling processes use chemical recycling in which the cotton fibers are dissolved with a concentrated alkaline solution and recovered to be used in new fabrics [5,7]. The solvent used in chemical recycling is often expensive and toxic, making this method undesirable for commercialization [8].

A new method for cellulose fabric recycling is to cause deformation and swelling using chemical interactions combined with mechanical processes to deweave the fabric mesh. This allows for a rapid breakdown of the fabric into reusable fibers. Mechanical forces such as heat and abrasion can cause the fiber meshes to break apart under the shear and tensile stresses associated with scraping against the stirrer and walls of the container. Additionally, cellulose expands

when heated. The expansion causes macroscopic swelling in the fabric mesh [9]. In addition to mechanical interactions, interactions with chemicals such as sodium hydroxide (NaOH) are of particular interest as past publications have shown its ability to swell cellulose samples; it is also a relatively common and inexpensive chemical, allowing for greater commercial viability of this process [2,3].

NaOH has been used in the textile industry since the 1800s in the mercerization process, where fabrics were soaked in a basic solution. The mechanism of mercerization is believed to be that sodium ions penetrate the cellulose structure through amorphous regions and diffuse into crystalline regions, disrupting the strong hydrogen bonds between the cellulose polymer strands [10,11]. The cellulose and sodium ions form a complex referred to as Na-Cellulose [10]. This complex is a state of compromised hydrogen bonds that allows for greater water penetration, causing the fibers to swell [12]. Mercerization is conducted by placing a fiber sample in a NaOH solution for a few minutes, followed by neutralization with either water or acid [13]. The solution used, temperature, and time reacted can affect the end product from the mercerization process [14]. During the process, the NaOH can modify the topology of the fibers by removing impurities, leading to a more adhesive and rough fiber surface. The resulting product has a higher absorption of dye and tensile strength per unit volume of fabric. Following the mercerization process, the fibers break down into smaller fibers in a process called fiber fibrillation; this causes the cellulose to have a larger reactive surface area and increases the fabric's luster [13]. The mercerization process

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has been primarily used to increase the strength and dye retention of cellulose-based fabrics; however, it can be used to assist in deweaving a sample. The mercerization process operates optimally between -10 and 4 °C and NaOH concentrations between 8-10 wt% (2.1-2.65 M) [15].

NaOH is a widely used chemical in various aspects of industry, including the mercerization of cotton, and the production of rayon, paper, and soap [2]. However, the toxicity of NaOH has been a concern [2]. The toxicity of NaOH depends on its concentration of hydroxide ions, which increase alkalinity [16]. NaOH is usually used in solid or 50 wt% solutions in industry so its corrosivity and effect on human health should be considered [17]. NaOH does not contribute to hazardous environments for aquatic organisms due to the neutralization by other substances, such as carbon dioxide in the environment [2,18]. As such, the washing step and low NaOH concentration are important in chemical processes utilizing NaOH.

This paper focuses on the chemical and mechanical interactions between cellulose-based fabrics in NaOH solutions. Although previous papers have shown that NaOH has properties that assist in deweaving, its efficacy is still under debate [11,12]. Furthermore, the effects of chemical changes in the cellulose structure arising from the process is an important consideration that has not been well studied. Multiple experimental trials were performed with different concentrations of NaOH to measure its impact on the deweaving process compared to a control of water. In addition, fabric samples of different sizes were subjected to a deweaving process to determine the optimal fabric size for the vessel size utilized. It is noted that the experimental conditions (i.e., low NaOH concentration and high temperature) of this research were not relevant to the mercerization condition (i.e., high NaOH concentration and low temperature) used to optimize a deweaving process.

A NaOH solution was reused to deweave multiple muslin fabric samples consecutively to determine the feasibility of reusing the solution. Finally, all the samples were analyzed with Fourier-transform infrared spectroscopy (FTIR), tensile testing, and optical microscopy to detect any changes in the cellulose structure from the recycling process.

Experimental

Sample Preparation and Material Testing

Cotton muslin samples, produced by Arthur R. Johnson Co., Inc., Brooklyn, N.Y, were purchased from the Fashion Institute of Technology. Sodium hydroxide (NaOH, reagent grade) was obtained from Sigma-Aldrich and was used without further purification. The deweaving process was performed on a Benchmark scientific hotplate stirrer (H3760-HS) connected with a temperature probe (H3760-TP). A cotton muslin fabric sample of specified dimensions was weighed before being pre-soaked with 5 ml of a specified

concentration of NaOH solution in a Petri dish. The fabric was repeatedly pressed into the solution until there was an absence of any hydrophobic or beading effect on the fabric surface. A 50 ml NaOH solution of the same concentration was placed in a 250 ml beaker and pre-heated to 50 °C with a stirring slide round stirring bar at a speed of 300 rpm. Once the solution temperature reached 50 °C, the muslin fabric sample was placed into the beaker. Every ten minutes, the stirring was stopped, and the fibers entangled around the stirring bar were separated. After thirty minutes, the muslin fabric sample was removed from the solution and rinsed three times with deionized (DI) water using a Buchner funnel. The fabric was placed in a Petri dish and was first dried at room temperature overnight and then placed in an oven to further dry at 50 °C for 24 hours.

Fabric Size Variation

The size of the muslin fabric was varied to determine the optimal fabric size for these experiments. The fabric sizes tested were 2 cm×2 cm, 3 cm×3 cm, and 4 cm×4 cm squares. For each of these samples, 50 ml of 1 M NaOH was used. The methods described in the sample preparation and material testing section were utilized.

NaOH Concentration Variation

3 cm×3 cm muslin fabric samples were treated with 50 ml of NaOH at different concentrations. The concentrations of NaOH were 0.5 M (2.0 wt%), 1 M (3.8 wt%), 2 M (7.4 wt%), 3 M (10.7 wt%), 4 M (16.0 wt%) and 5 M (20 wt%). The methods described in the sample preparation and material testing section were utilized. An additional sample using only DI water was subjected to the treatment as described in the sample preparation and material testing section.

Continuous Reaction

A 0.5 M NaOH solution was reused four times to treat four different 3 cm×3 cm muslin fabric samples consecutively. The continuous reaction follows the same method stated in the sample preparation and material testing section. Approximately 5 ml of the NaOH solution evaporated during each treatment and was replenished using a pipet before the subsequent treatment. Contaminants such as microfibers from each treatment were allowed to accumulate in the solution.

Characterization and Mechanical Property Testing

Fourier-transform infrared (FTIR) spectroscopy was conducted using a NicoletTM iS50 FTIR spectrometer equipped with an attenuated total reflectance (ATR) accessory. The spectra (32 scans/sample, 4 cm⁻¹ resolution) were collected at room temperature in the range of 400-4000 cm⁻¹ wavenumbers. Each spectrum was corrected by a background spectrum using the OmnicTM software (Thermo Scientific). Optical microscopy images were captured at 4× magnification using an Olympus IX51 inverted light microscope (Olympus,

Japan). Tensile testing was conducted using an Instron 5542 Advanced Material Testing System in accordance with the Standard Test Method for Tensile Strength and Young's Modulus of Fibers (ASTM C1557-14). Five strands of fiber were separated from each of the treated muslin fabric samples. The specimen length and diameter were set to 15.00 mm and 0.25 mm, respectively. Each specimen was mounted and stretched with a crosshead displacement rate of 2.00 mm/min until breakage. Stress to strain curves were plotted and the data were validated only when the breakage did not occur around the gripping region. The Young's modulus was calculated by taking the slope of the linear region of the stress to strain curve. The ultimate tensile strength is the maximum stress value before breakage.

Results and Discussion

Various sample sizes (i.e., 2 cm×2 cm, 3 cm×3 cm, and 4 cm×4 cm) were tested to determine the fabric size that yields the optimum results for these experiments. The optimal fabric size of 3 cm×3 cm was utilized for these experiments as the initial size variation tests found that sample 2 (3 cm×3 cm) was able to deweave in the shortest time (~19 minutes) (as reported in Table 1) compared to sample 1 (~22 minutes) and sample 3 (>30 minutes). Figure 1 shows that the fibers after completing the deweaving process, with both samples 1 and 2, can still clearly be seen and show no signs of dissolution while sample 3 did not completely deweave within the specified time. Thus, for the purpose of these experiments, the 3 cm×3 cm sample size provides the best measure of change in deweaving efficiency while also utilizing a larger size.

Effect of NaOH Concentrations on Morphology and Mechanical Strength of Fabric

A broad range of NaOH concentrations (0.5-5 M) was utilized to study the effect of NaOH concentration on the deweaving process of the fabric. As shown in Figure 2(A), the 0.5-3 M NaOH treated samples were all deweaved and the cotton yarns were observed.

In the case of samples treated with NaOH with a molarity greater than 3 M, especially the 5 M NaOH treated sample, the cotton yarns further dissociated into fibers similar to a cotton ball, indicating that the deweaving and dissolving of cotton fabric occurred at higher NaOH concentrations. The deweaving time was continuously decreased with increasing NaOH concentrations (i.e., 0.5 M NaOH treated sample=26 min and 5.0 M NaOH treated sample=7 min), indicating that the number of sodium cations is directly related to the deweaving time (Figure 2(B)). This result suggests that the morphology of cotton fabric could be controlled by the concentration of NaOH. Although the high concentration allows for a short deweaving time and is thus kinetically favorable, the increased toxicity and impact on the final products' properties, such as the mechanical properties, should be considered. It can be hypothesized that the mechanical strength of cotton yarns is higher than that of cotton fibers. While there is a slight decrease in deweaving time with higher NaOH concentration, overall, 0.5 M NaOH is less toxic and more environmentally friendly, making it much more viable for large-scale use in fabric recycling.

In addition to visual inspection of the deweaving phenomena, which depended on NaOH concentrations, optical microscopy was utilized to investigate the physical structure of a single strand. As shown in Figure 3(a), the

Table 1. Deweaving results of varied fabric sizes treated with 1 M NaOH

Sample	Fabric size (cm×cm)	Fabric diagonal (cm)	Mass start (g)	Mass end (g)	Deweaving time (min)
1	2×2	2.83	0.09	0.08	22
2	3×3	4.24	0.18	0.16	19
3	4×4	5.66	0.35	0.31	Not fully deweaved

Reaction conditions: 50 ml 1 M NaOH, 300 rpm. Note: the diameter of the 250 ml beaker is 6.5 cm.

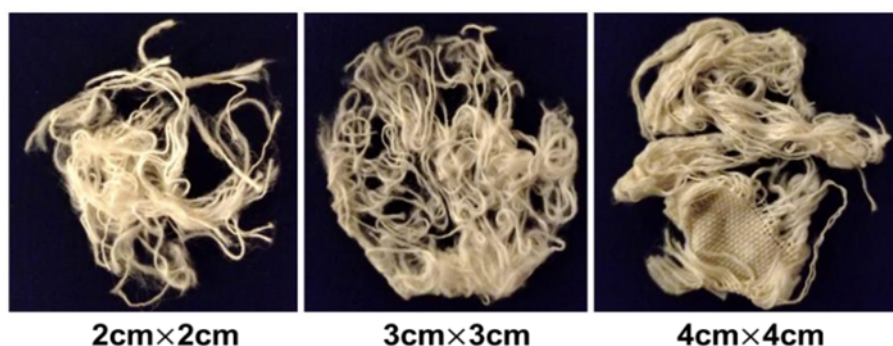


Figure 1. Deweaving results of the treatments of varying fabric sample sizes; 2 cm×2 cm, 3 cm×3 cm, and 4 cm×4 cm.

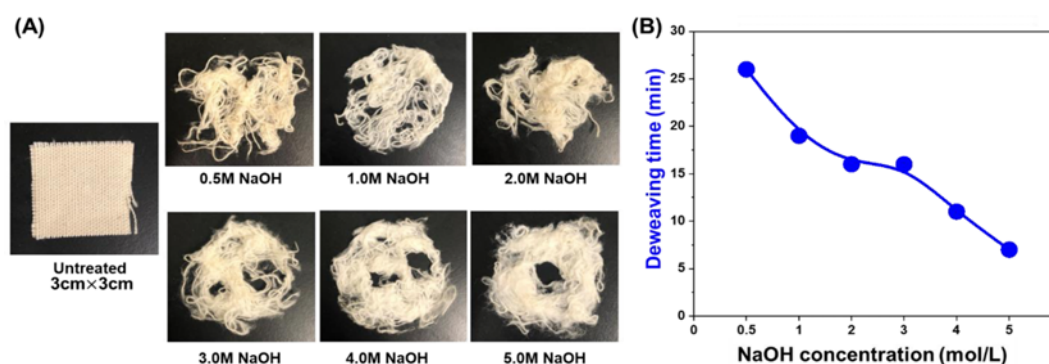


Figure 2. (A) Deweaving results from using various concentrations of NaOH on a 3 cm×3 cm muslin sample and (B) NaOH concentration effect on the deweaving time.

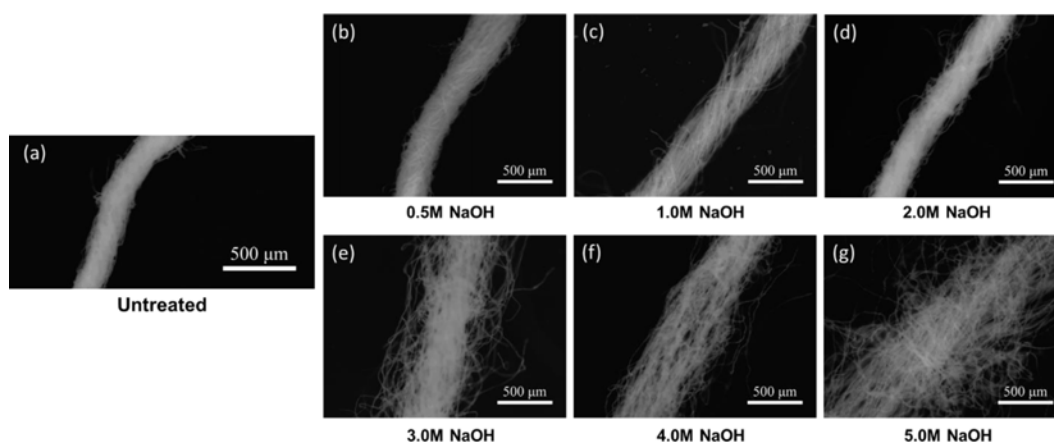


Figure 3. Cotton fiber treated with different NaOH concentrations and rinsed with DI water; (a) original cotton fiber, (b) 0.5 M NaOH treated cotton fiber, (c) 1 M NaOH treated cotton fiber, (d) 2 M NaOH treated cotton fiber, (e) 3 M NaOH treated cotton fiber, (f) 4 M NaOH treated cotton fiber, and (g) 5 M NaOH treated cotton fiber. Conditions: 300 RPM, 50 °C, 50 ml of 0.5-5 M NaOH, rinsed 3 times with DI water.

untreated sample's strand was composed of many microfibrils that were helically twisted into a bundle. This helical conformation is essential to the structural rigidity of the muslin fabric. After the cotton fabric was treated with low (0.5-2 M) concentrations of NaOH, the fiber strand remained intact as shown in Figure 3(b)-(d). However, the microfibrils became severely swollen and individualized when treated with NaOH concentrations of ≥ 3 M as shown in Figure 3(e)-(g). These swollen fibers also have a roughened surface with wrinkles [19]. It has been reported that highly swollen fibers are good resources for making cotton-reinforced composites: these composite materials are gaining popularity in several fields (i.e., construction, automotive, and aerospace) due to their lightweight, sound-absorbing, and high-strength properties [20,21]. It has also been reported that the increased interfacial area greatly improves fiber-matrix adhesion, granting the composite a higher Young's modulus and tensile strength compared to composites made with cellulose I fibers [22,23]. Thus, muslin fabric treated with higher

NaOH concentrations can be used to create cotton fiber-reinforced composites, while muslin fabric treated with lower NaOH concentrations can be directly spun into yarn that is readily able to be woven into fabric.

Tensile strength testing was performed to study the relationship between NaOH concentration and the mechanical properties of treated samples. As shown in Figure 4, both the Young's modulus and tensile strength of the muslin fabric decreased as a result of mechanical treatment and increasing NaOH concentrations. The significant drop in mechanical strength from the untreated fabric to the 0.5 M NaOH treated fabric indicates that mechanical treatments, such as direct contact between the fabric sample and stirring bar (and reactor wall), are the main contributors to the degradation of the mechanical strength of yarns (or strands); however, we could not entirely ignore the NaOH effect. Abrasion caused by the stirring bar may contribute to this interfibrillar swelling, thus enlarging the gap between the fiber's crystal structure and allowing hydroxide ions to penetrate [24,25].

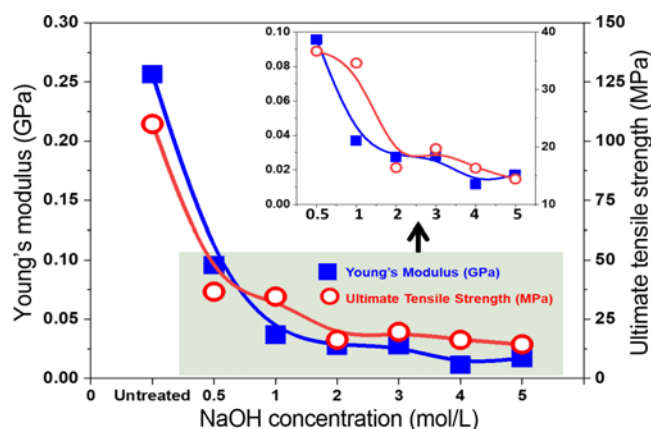


Figure 4. Young's modulus and ultimate tensile strength of untreated and NaOH treated (inset) samples.

The degree of degradation appears to be worsened as the NaOH concentration becomes higher. This relationship may be explained by the correlation between the NaOH concentration and interfibrillar swelling displayed in Figure 3, where the diameter of the strand expands with increasing NaOH concentration. It has been reported in the literature

that the penetrating hydroxide ions convert the structure from cellulose I to cellulose II and the fabric's lattice orientation from parallel to antiparallel [26]. The change in lattice orientation is closely related to the mechanical properties of the fabric, as it has been reported that cellulose II (antiparallel configuration) shows poor mechanical properties compared to cellulose I (parallel) [27]. As the amount of cellulose II present in the sample increases with increasing NaOH concentration, the decreasing Young's Modulus and ultimate tensile strength may be partially attributed to the conversion of cellulose I to cellulose II [27]. Based on the tensile strength test, it could be expected that the composition ratio of cellulose I to II follows the order: 0.5 M > 1 M > 2-5 M. Based on the visual/optical images and mechanical strength results, it could be concluded that surface morphology and physical properties of the fabric (and yarn) are changed with varied NaOH concentrations.

FTIR spectroscopy was performed to investigate the effect of NaOH concentration on the molecular structure of the cotton fabric. It is expected that the residues of NaOH, even after rinsing, on the cotton fabric surface could be observed using the ATR accessory because the penetration depth into the sample is a few micrometers (< 2 μm) [28,29]. The FTIR spectra of the untreated cotton fabric and 0.5 M-5 M NaOH

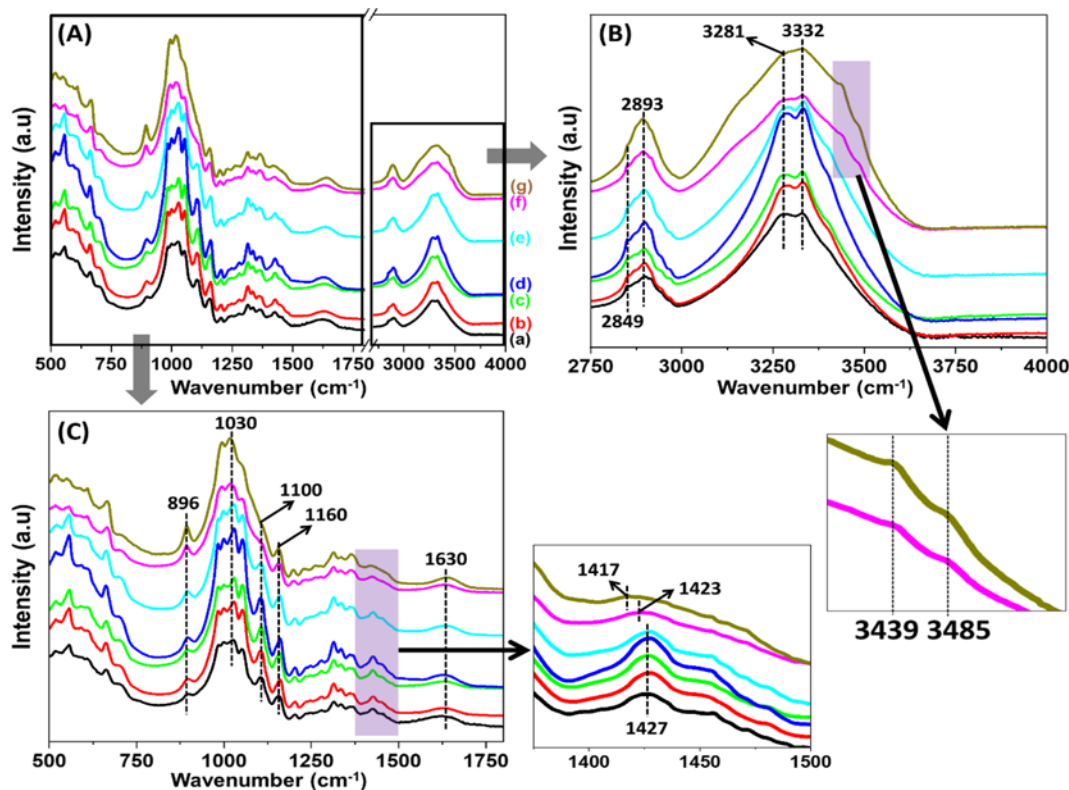


Figure 5. FTIR spectra of untreated cotton fabric and 0.5-5 M NaOH treated cotton fabric; (A) 500-4000 cm^{-1} wavenumber ranges, (B) 2750-4000 cm^{-1} wavenumber ranges, and (C) 500-1750 cm^{-1} wavenumber ranges. (a) untreated cotton fabric, (b) 0.5 M NaOH, (c) 1 M NaOH, (d) 2 M NaOH, (e) 3 M NaOH, (f) 4 M NaOH, and (g) 5 M NaOH.

Table 2. FTIR peak assignments of the NaOH treated samples' spectra

Wavenumber (cm ⁻¹)	Peak assignments	References
~896	COC symmetrical stretching mode (β -glycosidic linkage)	10,13
~1030	CO stretching mode	10,12
~1100	COC asymmetrical stretching mode, Asymmetric in-plane stretching band	32,33
~1160	COC asymmetric stretching mode	10,13,33
~1427	O-H bending mode, CH ₂ scissoring	31,33,34
~1630	O-H bending mode (adsorbed water molecules)	32-34
~2849	C-H stretching mode (symmetric)	29,30,32,33
~2893	C-H stretching mode (asymmetric)	10,12,29,33
3270~3330	O-H stretching mode	29,31,33,35

treated samples are shown in Figure 5((A)=500-4000 cm⁻¹, (B)=2700-4000 cm⁻¹, (C)=500-1800 cm⁻¹) and peak assignments are reported in Table 2. Most samples showed similar spectra (i.e., similar peak shapes and IR bands) in the 500-4000 cm⁻¹ regions, indicating that the NaOH treatment did not significantly change the molecular structure of the cotton fabric. Although we cannot exclude the presence of NaOH after rinsing, the quantity of NaOH could be traceable. Please note that according to literature NaOH IR bands are usually detected at ~880 cm⁻¹ and ~1435 cm⁻¹ which are not seen in the treated samples, thus suggesting no traceable amounts of NaOH remaining after rinsing [30]. It has been reported that cellulose I (parallel packing of polyglucosan chains) converts to cellulose II (anti-parallel packing) after NaOH treatment and rinsing procedures, and the structural differences could be distinguished by means of FTIR, especially based on the O-H stretching mode peaks [30]. Some slight peak shape changes and new peaks are observed in the 3000-3750 cm⁻¹ region of the spectra of samples treated with NaOH > 3 M (Figure 5(B)). The new peaks at 3439 cm⁻¹ and 3489 cm⁻¹ are assigned to the O-H intermolecular hydrogen bond in

cellulose II structure, while 3281 cm⁻¹ and 3332 cm⁻¹ are O-H stretching mode in cellulose I structure. In addition to the O-H stretching mode band, the shape of -CH₂- stretching mode bands at 2750-3000 cm⁻¹ is continuously changed with increasing NaOH concentration. Transformation of cellulose I to II can be confirmed in < 1750 cm⁻¹ wavenumber ranges. As shown in Figure 5(C), the ~1427 cm⁻¹ peak, which is assigned to the in-plane O(6)-H bending mode, was shifted to lower wavenumbers and its intensity was decreased with increasing NaOH concentration. This result supports that cellulose I transformed to cellulose II and is well matched to the literature results [31]. Since the time to deweave continuously decreased with increasing NaOH concentration (except between the 2 M and 3 M NaOH samples) while the emergence of cellulose II was only shown to occur above 3 M NaOH concentrations, no clear correlation between the transformation of cellulose I to cellulose II and the deweaving efficiency can be found from these results.

Feasibility of Reusing the NaOH Solution

Chemical waste is a global issue, and the amount of NaOH

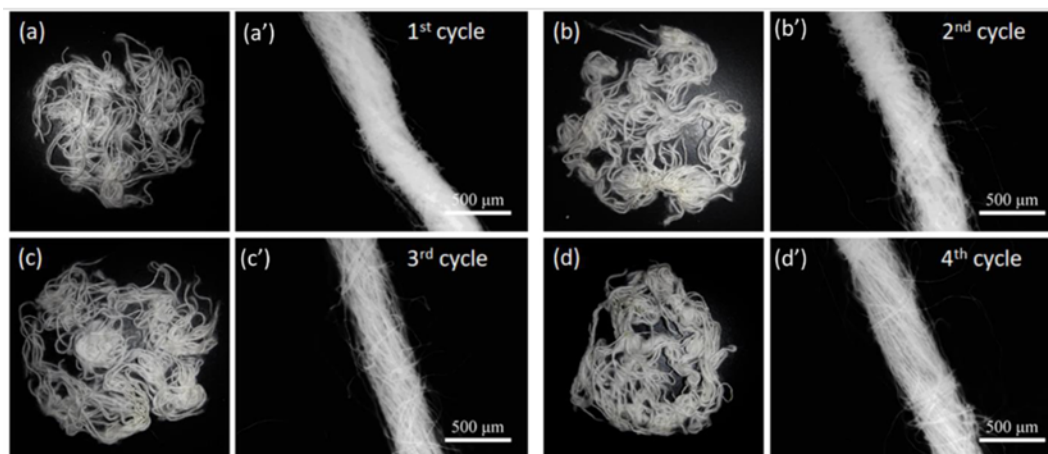


Figure 6. Cotton fabric treated with the 0.5 M NaOH solution for 4 cycles. (a, b, c, and d) Digital camera images (a', b', c', and d') Optical microscopy images; (a, a') 1st reaction, (b, b') 2nd reaction, (c, c') 3rd reaction, (d, d') 4th reaction. Reaction conditions: NaOH concentration (0.5 M, 50 ml), Reaction time (30 mins), Temperature (50 °C), mixing speed (300 rpm).

waste in the current experimental method can be drastically reduced if the aqueous NaOH solution is reused for multiple reactions. The feasibility of reusing the NaOH was investigated by deweaving four muslin fabric samples in the same aqueous NaOH solution consecutively. Figure 6 shows the digital camera (a, b, c, d) and optical microscopy (a', b', c', d') images of the muslin fabric samples treated with the same 0.5 M NaOH solution. All the muslin fabric samples deweaved completely after < 30 minutes of treatment. The optical microscopy images show that the strand is held together in a helical conformation of microfibrils. Upon closer inspection, the strands' opacity diminishes slightly with each cycle. This result indicates an increase in the microfibril swelling with each cycle. However, the strands remain intact even after several cycles without replacing the NaOH solutions, suggesting the treated cotton fabric and deweaved strands could be used to regenerate a cotton fabric without compromising the fiber quality.

Conclusion

In this work, we investigated the cotton fabric deweaving phenomena with varied parameters, such as NaOH concentration and the number of cycles the chemicals are reused for. The concentration of NaOH is an important factor in both the deweaving time and the final properties of the fabric strands. Although higher NaOH concentrations resulted in decreased deweaving time, they had negative effects on the tensile properties of the fibers, which increase the toxicity and material consumption. The increased number of sodium ions led to increased fiber breakage and decreased tensile strength and Young's modulus. The FTIR spectroscopic techniques provided that the cellulose chain orientation changes from parallel (cellulose I) to antiparallel (cellulose II) when the fabric is treated with > 3 M NaOH concentration. The optical microscopy, FTIR spectroscopy, and mechanical testing results indicated that high concentrations of NaOH led to cellulose II formation and decreased mechanical properties. It was also found that the NaOH solutions can be reused to treat new samples without increasing the deweaving time. From an environmental point of view, utilizing a 0.5 M NaOH solution should be a consideration for the deweaving of cotton fabric. To further investigate this process, it would be important to determine the scaling up of this process to larger volumes and fabric sizes.

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Conflicts of Interest

The authors declare that they have no conflict of interest.

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