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Thermochemical degradation of cotton fabric under mild conditions

Michael Cuiffo¹, Hye Jung Jung², Asta Skocir³, Theanne Schiros⁴, Emily Evans⁵, Elizabeth Orlando⁶, Yu-Chung Lin⁷, Yiwei Fang⁷, Miriam Rafailovich⁸, Taejin Kim^{9*}  and Gary Halada^{9*}

*Correspondence:
taejin.kim@stonybrook.edu;
gary.halada@stonybrook.edu
⁹ Associate Professor,
Department of Materials
Science and Chemical
Engineering, Stony Brook
University, Stony Brook, NY
11794, USA
Full list of author information
is available at the end of the
article

Abstract

Textile waste presents a major burden on the environment, contributing to climate change and chemical pollution as toxic dyes and finishing chemicals enter the environment through landfill leachate. Moreover, the majority of textile waste reaching landfills is discarded clothing, which could be reused or recycled. Here we investigate environmentally benign morphology changing of cotton textiles as a precursor for reintegration into a circular materials economy. At 50 °C using low concentrations of acids and bases, the interfiber structures of woven cotton were successfully degraded when treated with the following sequence of chemical treatment: citric acid, urea, sodium hydroxide, ammonium hydroxide, and sodium nitrate. Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, X-ray photoelectron spectroscopy (XPS) and scanning electron microscopy (SEM) reveal separation of the constituent fibers without depolymerization of the cellulose structure, and streaming potential measurements indicate that surface charge effects play a key role in facilitating degradation. The proposed reaction procedures show feasibility of effective waste-fabric recycling processes without chemically intensive processes, in which staple fibers are recovered and can be re-spun into new textiles.

Keywords: Cotton fabric degradation, Electron microscopy, Vibrational spectroscopy, X-ray photoelectron spectroscopy, Streaming potential, Surface charge transfer, Citric acid

Introduction

Global population growth, linear production models and the constant demand for new of “fast fashion” have resulted in increased apparel production and expenditures with a corresponding increase in the quantity of clothing and textile waste (Ma et al., 2019). The clothing industry is one of the most polluting industries in the world, producing ~ 92.3 million tons of textile in 2013 and anticipating ~ 148 million tons fashion waste in 2030 (Bird, 2018). According to the Environmental Protection Agency (EPA), textiles represented 8% of the 11.2 million tons of municipal solid waste (MSW) landfilled in 2017. Further, 84% of discarded clothes in 2012 went into either a landfill or an incinerator in the United States (Wicker, 2016; EPA report). When natural fibers (e.g., cotton, linen and silk) and semi-synthetic fibers (e.g., rayon, tencel and modal) are buried in landfills, they produce potent

greenhouse gases (e.g., methane) in addition to soil contamination by toxic chemicals from pesticides, dyes and finishing treatments (Allesch & Brunner, 2014; Turner et al., 2015). Cotton (non-organic) is considered to be one of the world's most environmentally harmful crops due to its unsustainable chemical (e.g., fertilizer, pesticide) demands and water consumption (Micklin, 2007; Sandn & Peters, 2018; Imtiazuddin et al., 2012). No less than 500 billion USD of value is lost annually due to underutilization of clothing and limited textile recycling (Ellen MacArthur Foundation, 2017). Diverting high volumes of cotton textile waste is a cost-effective route to minimize pollution, water, chemical, and energy-intensive production of new clothing.

Over the last few decades, degradation of cellulose-based materials using different methods (e.g., mechanical, thermal, chemical, and enzymatic) has been investigated to improve the fiber-waste recycling or regeneration efficiency (Sferrazza et al., 1996; Deschamps, 1998; Rowe, 2000). From an economic point of view, chemical treatment has some advantages compared to other methods, including high fossil energy expenses for mechanical and thermal treatment and high cost of enzyme sources (Mansfield et al., 1999). Furthermore, chemically treated cellulose waste can possibly provide high quality fibers (Notman, 2020). It has been reported that both inter- and intramolecular hydrogen bonding in cellulose (~95% composition of cotton fiber) can hinder the use of a weak acid solvent to regenerate cellulose-based waste (De Silva & Byrne, 2017). Celluloses with a higher degree of polymerization (DP) are correspondingly more difficult to degrade, a process found to be facilitated by use of agents to swell fibers from wood and cotton linter sources and degrade intramolecular bonds (Wang et al., 2008). The reported corresponding partial breakdown of intermolecular bonds and reduction in DP is of value for certain recycling processes, but would likely also degrade fiber microstructure which provides strength for reuse. Recently, Evrnu has produced isolated cellulose molecules from the post-consumer waste cellulose-containing materials using multi-state processes including supercritical CO₂ washing, enzyme treatment, and swelling agent application (Flynn & Stanev, 2016). While Evrnu's process is expected to produce regenerated fibers at scale (~1 ton/day), high temperature (>320 °C), high pressure (>2.5 Mps), and high production cost (ionic liquids and enzymes) limit the degree of reduction of environmental impact.

In an effort to advance low energy, green chemistry textile recycling for a circular materials economy, we investigate the feasibility of using a weak acid, citric acid, in combination with agents such as sodium hydroxide to promote swelling and charge transfer and degrade cellulose-based textiles into precursors for recycled textile production. A suite of analytical techniques, including chemical and surface characterization via Fourier transform infrared (FTIR), Raman spectroscopy, and X-ray photoelectron spectroscopy (XPS), structural analysis via scanning electron microscopy (SEM) and streaming zeta potential analysis, were used to assess the viability of this approach for woven cotton recycling under mild conditions (e.g., Temp.: 50 °C, Conc.: 0.5 M, Reaction time/chemical: 30 min) and reduced environmental impact.

Method

Materials and sample preparation

Cotton muslin samples (tailoring muslin from Arthur R. Johnson Co., Inc., Brooklyn, NY, USA) were obtained from the Fashion Institute of Technology (FIT), typical of cloth

used by their students for clothing design. The specifications of the sample are 100% cotton, plain weave construction, 190 GSM, and a yarn count of 33 EPcm \times 9 PPcm. The following chemicals were used without purification procedures: citric acid (Sigma-Aldrich, SA.C.S. reagent grade), sodium hydroxide (Sigma-Aldrich reagent grade), ammonium hydroxide (J.T. Baker, 28–30% reagent grade), sodium nitrate (Fisher Scientific A.C.S. certified crystal), urea (Alfa Aesar, A.C.S. 98–100%). Rational for the overall selection of the chemistry chosen is explained below (Results and Discussion). All solutions were made using deionized water ($>18\text{m}\Omega/\text{cm}$). The experimental conditions (e.g., reaction temperatures, chemical concentrations, reaction times) are outlined below and the schematic diagram of reaction procedures and experimental conditions are shown in Scheme 1.

Once the interfiber structure of muslin fabric was degraded in the solution, the fluid was removed via vacuum filtration (Speedaire vacuum pump, Dayton) through a coarse or medium glass frit (Fisher Scientific). The fibers collected in the frit were then rinsed multiple times with deionized water ($>18\text{ m}\Omega/\text{cm}$) and dried at room temperature in air overnight.

Characterization

Scanning electron microscopy (SEM)

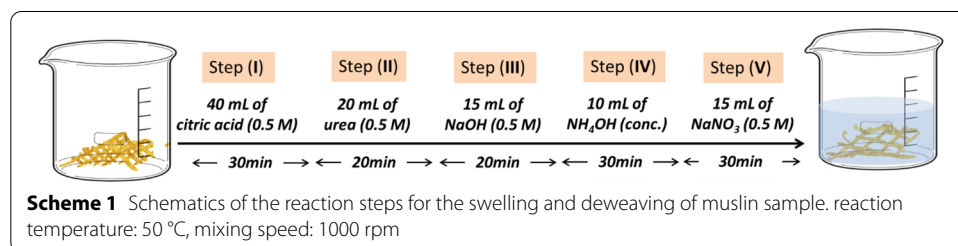
The SEM system used was a Leo 1550 with a Robinson back scatter Gemini detector with Energy dispersive x-ray analysis (EDAX), back scattering (BS), electron backscatter diffraction (EBSP), secondary electron (SE), and InLens detectors. SEM was used for determining surface morphology, as well as presence and dispersion of nanoparticles.

Streaming potential

Streaming potential (surface zeta potential) of as-received and treated textile was measured using a SurPASS 3 electrokinetic analyzer at Anton-Paar USA using a cylindrical cell and a 1 mM KCl solution as the electrolyte for the measurement. The pH (adjusted by the addition of 0.05 M HCl solution) was ramped beginning at $\sim\text{pH } 9$ and ending at $\sim\text{pH } 2.5$. During testing, at each pH condition, the sample was rinsed five times with the electrolyte solution before the streaming potential measurement was collected. The electrolyte solution was purged with nitrogen throughout the duration of the measurement to prevent the dissolution of carbon dioxide.

Raman spectroscopy

Raman spectra of the as-received and treated muslin samples were collected using a 514 nm He–Ne laser (Renishaw inVia confocal Raman microscope) under ambient



conditions. The scattered photons were directed into a single monochromator and focused onto an air-cooled charge-coupled device. Throughout the Raman spectroscopy studies, the laser powers were controlled in order to insure no thermal damage of sample. Samples were placed on the Raman-inactive quartz microscope slide (Ted Pella, Inc.) to avoid any spurious peaks in the analyzed samples. The Raman shift was calibrated with a built-in silicon standard sample. The spectral acquisition times were 10 scans accumulated with 10 s/scan.

Fourier transform infrared (FTIR) spectroscopy

The FTIR spectroscopy was conducted with a Nicolet iS50 FTIR spectrometer using a liquid nitrogen cooled MCT-A detector or a deuterated triglycine sulfate (DTGS) detector at room temperature. The FTIR spectra were collected at a resolution of 4 cm^{-1} with 256 accumulated scans for a better signal to noise ratio. The spectra were collected in $400\text{--}4000\text{ cm}^{-1}$ wavenumbers to investigate the chemical structure of samples. Sampling areas were continuously purged with dry air throughout the experiments in order to prevent the additional absorption of water vapor in the samples being analyzed.

X-ray photoelectron spectroscopy (XPS)

XPS was conducted using a Physical Electronics Quantum 2000 microprobe with PHI Compass 6.4 software and a 16 channel photoelectron detector. The monochromatized Al X-ray source was operated at 25 W with a 100 micron beam size. A pass energy of 23.5 eV was used, and 8 to 20 scans were collected for each region shown. The work function for the XPS system is 4.55 eV. All XPS analysis was conducted at an ultrahigh vacuum of 1.0×10^{-10} to 1.0×10^{-9} torr. All XPS spectra were collected at a 60° take off angle with respect to the sample surface and all spectra were corrected for charging by using the C1s line of adventitious carbon at 284.6 eV as a reference point. All curve fitting followed the methods outlined by Savitsky and Golay (Savitsky & Golay, 1964), and Sherwood (Briggs & Seah, 1993) as well as procedures developed over the years by G. P. Halada and C.R. Clayton using PHI Multipak, version 9.2 software (Halada & Clayton, 1993).

Mechanical testing

The tensile strength, Young's Modulus, and toughness of woven cotton muslin samples were measured by Instron 5542 testing station. The diameter of the cotton yarn sample is $\sim 200\text{ }\mu\text{m}$ and the length of the sample is 150 mm. The extension rate is set at 2 mm/min.

Results and discussion

The chemicals we have chosen for the degradation process were carefully selected for their known inherent chemical reactivity with cotton fibers while not being used in conjunction with one another in the same solution until now. We hypothesize that each chemical we add to reaction over time causes a number of physical and/or chemical changes to the fibers, or more precisely the surface chemistry of the fibers, as well as some degree of interfibrillar swelling. We suspect the citric acid helps to remove impurities, such as metal ions, and hence preparing the fabric surface for further reactions

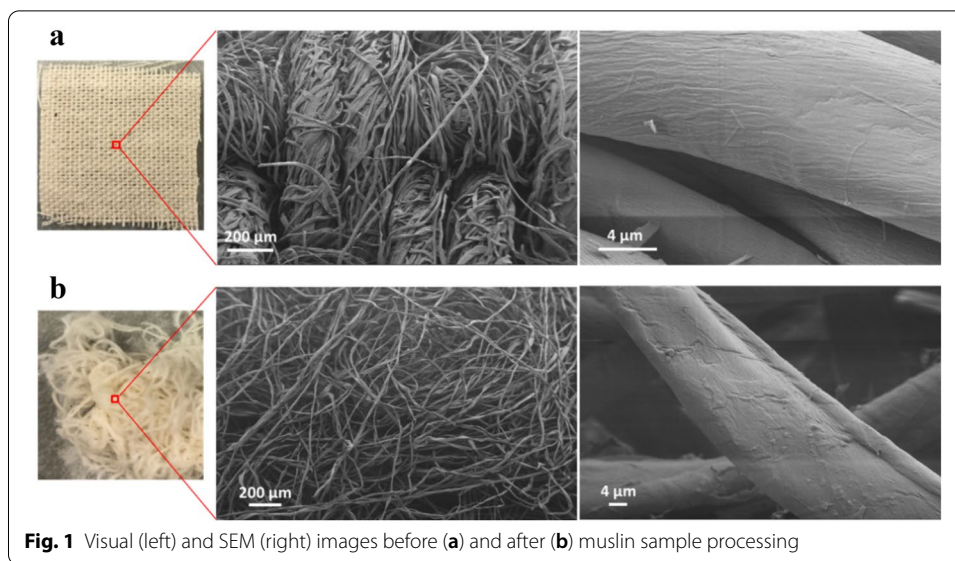
(McSweeney et al., 2006; Thanh & Nhung, 2009). It may also contribute to oxidation of fiber surfaces, resulting in additional carbonyl or carboxyl ligands (Ye et al., 2015). Initial tests using citric acid alone showed some limited separation of fibers, but only under less benign condition (high temperature close to 100 °C as well as with the addition of HCl; hence a chemical treatment was designed which would perform successfully at lower temperatures and with addition of harsh acids. Citric acid is also known to react with urea (though not necessary in the presence of cellulose) to form soluble urea citrate (Palciekienė, et. al., 2005)—this could help to limit the degree of degradation, as pure sodium hydroxide/urea solutions are potentially capable of degradation of cellulose to a greater degree than desired (Cai, et. al. 2005).

Sodium hydroxide increases pH to 3–3.5 and is well known to swell cotton fibers (Gavillon & Budtova, 2008; Isogai & Atalla, 1998; Jin et al., 2007; Moigne & Navard, 2010). Urea acts as a stabilization agent to prevent the agglomeration of deweaved fibers and swelled cellulose molecules (Ershova et al., 2012; Xiong et al., 2014). The addition of ammonium hydroxide and sodium nitrate could control the pH and neutralize the solution. In addition, both sodium hydroxide and ammonium hydroxide are used to remove some of the non-cellulosic surface species (waxes, pectin) found on cotton fibers, which would in turn facilitate surface reactions of cellulosic fibers with citric acid (hypothesized to further impact surface charge) (Chung et al., 2004). Treatment with ammonium hydroxide has also been noted to increase the degree of swelling by sodium hydroxide (Warwicker, 1969). It is likely that this is also due to removal of some surface waxes and pectin, a process which way be enhanced by the use of sodium nitrate. None the less it is worthwhile to note that the weight difference between as-received and treated sample was trivial; hence any loss of material is minimal, another critical condition if the fiber microstructure is to be maintained (to retain some mechanical properties and hence utility as a source material for re-weaving). Much stronger acids (phosphoric, sulfuric, perchloric) have also been found to swell cotton fibers, though as noted these would increase the environmental impact of the proposed process (and also likely introduce unwanted reactions) and hence are rejected for the chosen process.

Overall, therefore, to develop a process which can (a) operate at reasonably low temperatures, (b) avoid the use of harsh acids, (c) promote interfibrillar swelling, and (d) retain general fiber structure while providing extensive deweaving, the five part chemical process (combined with fairly mild thermomechanical agitation) was developed.

Scanning electron microscope (SEM): effect of treatment on the surface morphology

The surface morphology of as-received and treated cotton textile samples is revealed with optical and scanning electron microscopy (Fig. 1). Optical microscopy shows that the as-received muslin fabric is composed of a tightly-woven fabric structure. SEM imaging reveals that each fiber, which is ~ 200 µm in diameter, is composed of smaller strands, ~ 20–25 µm in diameter, which are tightly bundled together in the untreated sample. Higher magnification imaging reveals the surface of the strands to be composed of even smaller highly oriented fibrils, ~ 0.5 µm in diameter. As shown in Fig. 1b, the treated sample was deweaved completely. SEM imaging show complete decomposition of the 200 µm fiber bundles into the smaller strands, without any specific orientation. Higher resolution SEM imaging shows that the surfaces of

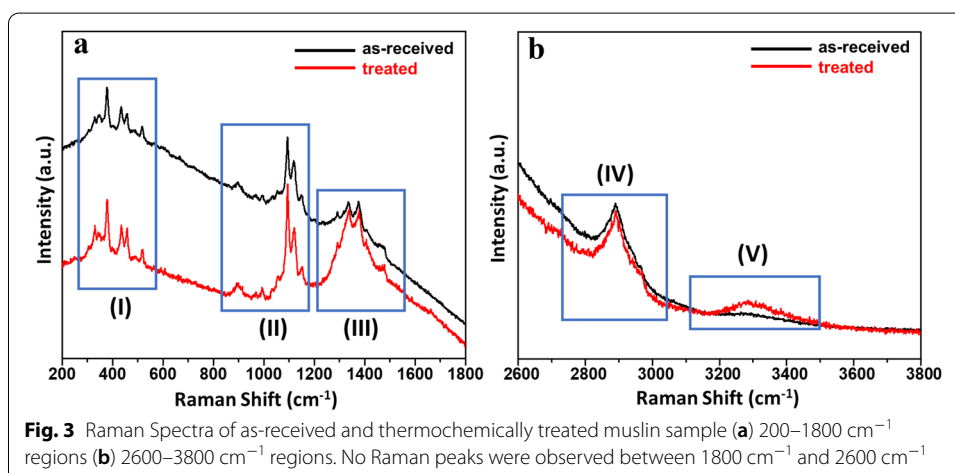
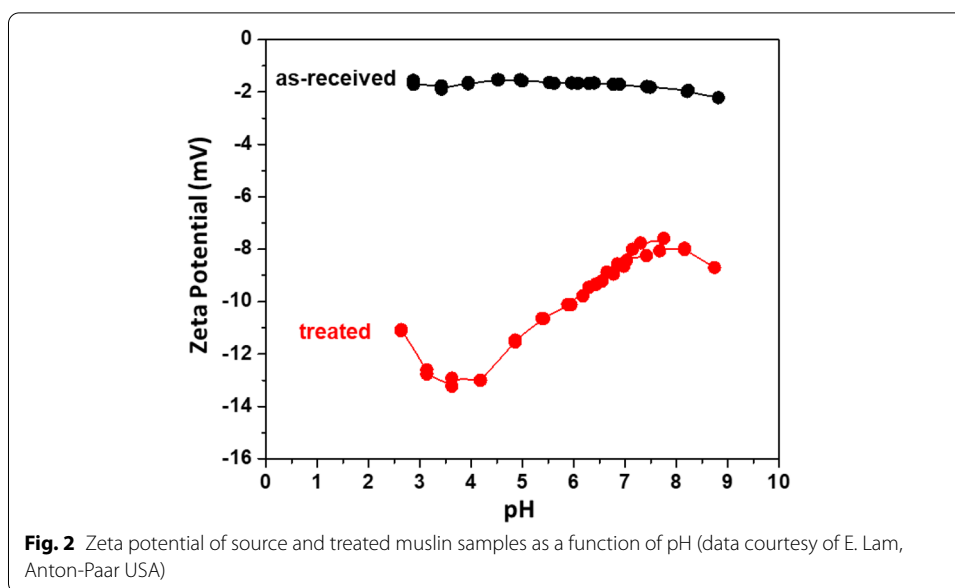


the strands are not significantly changes, while the nanoscale fibrils are still visible and oriented along the fibril strand diameter. The surface of fibers appears smooth and round. (Fig. 1a) After treatment, the SEM revealed that the muslin samples were fully unwoven. (Fig. 1b) Compared to the as-received fibers, the surface of treated fibers appears flatter and rougher. In addition to the deweaving nature of the degradation phenomena, the treated fibers appear whiter in appearance.

Analysis of SEM images indicate that, on average, ~ 70% swelling of fibers is evident following treatment, though this varies significantly. A portion of fibers also appear flatter and more ribbon-like in aspect.

Streaming potential: effect of treatment on the surface charge

To investigate the effect of chemical treatment on the surface charge of textile fibers, and gain mechanistic insight on the degradation process, we use streaming (zeta) potential measurements to compare the voltage difference at a range of pH values for both untreated and processed muslin fabric. The zeta potential, ζ , is calculated according to the Fairbrother-Mastin equation for streaming potential measurements (Fairbrother & Harold, 1924). In contrast to the untreated cotton samples, the treated muslin shows a significant difference in both curve shape and magnitude (Fig. 2). Both samples display negative ζ , indicating a negative surface charge. The untreated muslin has ζ values close to -2 mV, suggesting minimal presence of charged species (or a relatively similar amount of positively and negatively charged species) on the surface. Upon treatment, however, the muslin sample ζ decreases sharply from -11 mV to -14 mV, and then increases to -8 mV at pH values greater than 4. The increase in zeta potential, primarily due to adsorbed water, at increasingly alkaline pH is indicative of sample swelling (Luxbacher et al., 2014).



Raman and FTIR spectroscopy: effect of treatment on the molecular structure

Molecular level insights on the bonding and vibrational structure of samples are obtained with Raman spectroscopy. Figure 3 presents the visible-Raman (514 nm) spectra of as-received and thermochemically treated muslin samples. It has been reported that the peak assignment of cellulosic material's Raman (and FTIR) spectra could be difficult due to the complexity of molecular structure of lignocellulose (Blackwell et al., 1970; Edwards et al., 1994). As expected, the muslin sample's Raman spectrum also contains several weak, medium, and strong intensity peaks. The Raman spectrum were divided into five zones over the range 200–3800 cm^{-1} (Fig. 3a: 200–1800 cm^{-1} Raman shift, Fig. 3b: 2600–3800 cm^{-1} Raman shift to facilitate analysis (note: no peaks were observed between 1800 cm^{-1} and 2600 cm^{-1}) and peak assignments are provided in Table 1. Compared to the as-received sample's Raman spectrum, no significant peak shift was observed in the Raman spectrum of the thermochemically treated samples,

Table 1 Raman and FTIR peak assignments

Raman shift (cm^{-1}) ^a	FTIR wavenumber (cm^{-1}) ^b	Peak assignment
328–454		C–C–C ring deformation
516		C–O–C glycosidic linkage vibration
563		In-plane vibration
	713	CH ₂ rocking vibration
891	895	C–O–C in-plane symmetric vibration (Raman) out of plane C–O–C, β -glycosidic bond (IR)
	1043	C–O stretching
1093		C–O–C glycosidic linkage vibration (Asym)
1117		C–O–C glycosidic linkage vibration (Sym)
	1127	C–O–C bridging vibration
1147		C–C ring vibration
1292		CH ₂ twisting vibration
1333		CH ₂ wagging vibration
	1371	O–H bending vibration or C–H deformation
1375		CH ₂ deformation vibration
1459		C–O–H vibration
	1429	–OH stretching or C–H wagging vibration
	1633	C–O stretching
	1734	C=O stretching
2891		CH ₂ stretching vibration
	2901	Aliphatic CH and CH ₂ stretching
3289		O–H and C–O–H stretching
	3413	–OH stretching

^a Raman peak assignment (Blackwell et al., 1970; Edwards et al., 1994; Flynn & Stanev, 2016)

^b FTIR peak assignment (Chan et al., 2015; Oh et al., 2005)

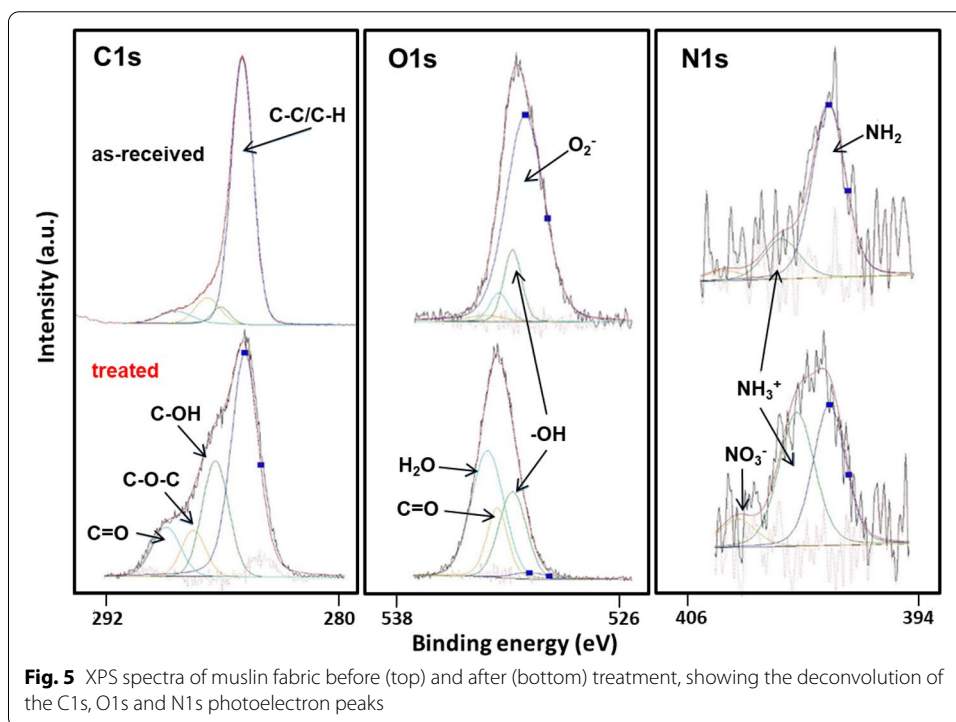
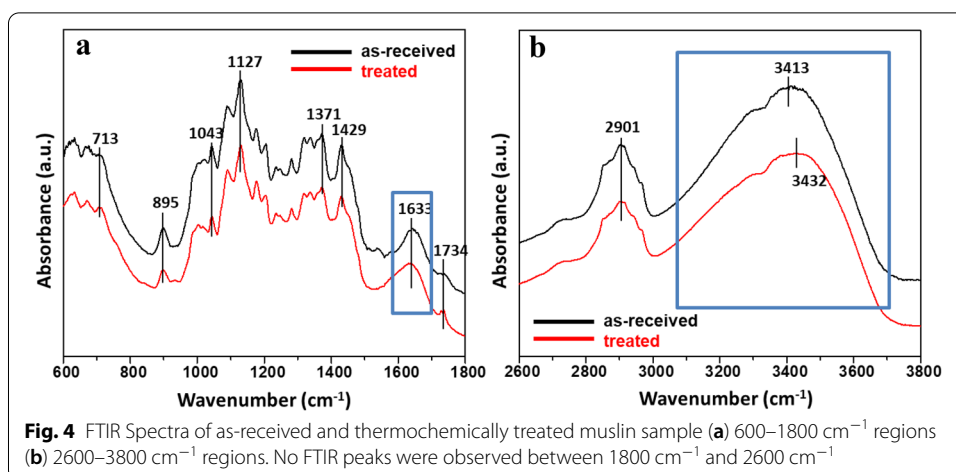
while intensities of peaks were changed, showing and increase in COC vibrations (zone II) and methyl groups (zone V). The data indicates that the cellulosic structure and polymer chain ligands remain intact, while the woven interfiber structure of muslin fabric was degraded, as observed in the SEM micrograph (Fig. 1).

Further insights on the detailed molecular structure of muslin are obtained with FTIR spectroscopy. As shown in Fig. 4, aside from a minimal change in the position of the main line and broadness of OH bending ($\sim 1633 \text{ cm}^{-1}$)/stretching ($\sim 3413 \text{ cm}^{-1}$) mode band peaks, no obvious differences were observed with chemical treatment. This result could indicate that the strength of inter/intra-hydrogen bonding were affected by thermochemical treatment (Oh et al., 2005).

The observed Raman and FTIR spectroscopy results confirmed that the polymeric cellulose structure was retained during the degradation process. In particular, the ring vibrational frequencies and glycosidic linkage vibrational frequency were little impacted by the thermochemical processing of the fabric. The detailed Raman and FTIR peaks in the fingerprint and functional group region are shown in Table 1.

X-Ray photoelectron spectroscopy (XPS): effect of treatment on the surface species

In order to understand surface species of as-received and chemically treated cotton fabrics, the XPS analysis was performed. Figure 5 shows the deconvolution of the high



resolution XPS photoelectron spectra in the C1s, O1s and N1s photoelectron regions for muslin fabric as received and following the treatment described. As clearly observable, the greatly enhanced surface sensitivity of XPS (< 10 nm for polymeric materials) allows us to detect changes which are very difficult to observe in vibrational spectra (which is significantly less surface sensitive—having a depth of analysis approximately several hundred times greater).

The deconvolution of the C1s peak prior to treatment shows singlet peaks typical of cellulose, including C–C (which overlaps the large adventitious hydrocarbon at 284.6 eV which is seen on the surface of the fibers and always present on any materials). The other chemical bonding is associated with the cellulose structure of muslin

fabric are the singlets associated with C–OH (286.2 eV) and C–O/C–O–C at 287 eV (Kamlangkla et al., 2010). In addition to the typical cellulose structure related peaks, a photoelectron peak attributed to C=O or possibly a carboxylate group (COO[−]) was observed at 288.3 eV (Awada et al., 2012; Matuana et al., 2001). Although the C=O is not a part of the cellulose, it was observed in the FTIR ($\sim 1734\text{ cm}^{-1}$ in Fig. 4a) and is often present in cotton fibers due to processing. It should be noted that C=O/carboxylate ligands and the hydroxyl groups attached to the cellulose structure are very active functional groups which may aid in the degradation methodology. Likewise, the O1s photoelectron peak primarily indicates oxide (O₂[−], possibly from small oxide particles found in most as received fabric, a result of processing and cutting), hydroxide ligands (–OH, consistent with the C–OH peak detected in the C1s spectra), a small amount of sorbed water (H₂O), and a weak peak associated with carbonyl (C=O) ligands, which are consistent with the C1s photoelectron peak deconvolution. The deconvolution of the N1s peak from the as-received sample, while somewhat weak, includes the amine (NH₂) at 398.9 eV and a potential protonated amine NH₃⁺ at 401.7 eV, which is actually R–NH₃⁺ where R is likely a CH₃ group related to the aliphatic hydrocarbons observed in the FTIR spectra (Awada et al., 2012).

The surface chemistry of the degraded muslin fabric shows significant differences associated with the chemistry of the degradation process. As shown in the C1s spectrum, the intensity and concentrations of signals associated with carbon species are quite different from that of the as-received fabric. In particular, the percentage of the C1s signal from C=O/carboxylate and C–OH (or intensity ratio of C=O (or C–OH)/C–C) was much greater, indicative of the reactions with carboxylic acid as well as consistent with bleaching of the appearance of the fibers noted during the degradation process. The C1s peak positions (C–C at 284.6 eV, C–OH at 286.2 eV, C–O/C–O–C at 286.8 eV, C=O at 288.1 eV) of treated sample, however, were similar to the muslin fabric before degradation. This result provides no evidence of breakdown of the cellulosic structure, including glycosidic linkages, in agreement with vibrational spectroscopy as shown in Figs. 3, 4.

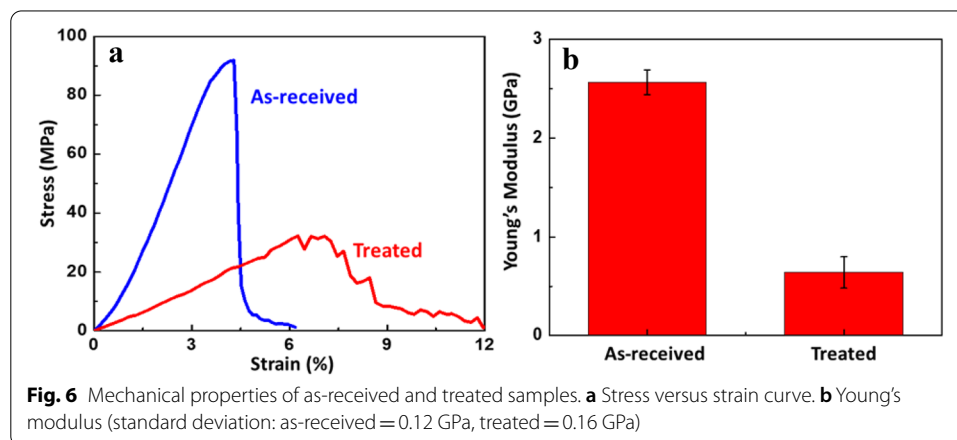
The O1s photoelectron spectra similarly shows significant surface chemical changes, including a loss of the oxide (O₂[−]) peak, consistent with observations from microscopy (not shown for brevity) that the degradation process removes any oxide particular contaminants, which may also benefits recycling and reuse of the fibers. Photoelectron peaks associated with both bound water (–OH and H₂O) and carbonyl (C=O) groups increase significantly, consistent with the FTIR vibrational spectroscopy, the C1s photoelectron data, and the observed negative zeta potential (and indications of fiber swelling). The deconvolution of the N1s peak in Fig. 5 also shows that chemical changes took place in the nitrogen bonding, although peak intensity was weak. A significant increase in the percentage of the N1s photoelectron intensity due to the protonated amine NH₃⁺ is noted (likely due to the use of ammonium hydroxide in the degradation process) and an increase in intensity associated with NO₃[−] (located at 403.4 eV) (likely due to the use of sodium nitrate in the degradation process). The presence of nitrate groups on the surface may also contribute to the negative zeta potential noted in the streaming potential data. However, the overall weakness in the N1s signal may indicate that it is not a major contributor.

Mechanical properties: effect of treatment on the mechanical property

To verify the mechanical properties, tensile testing was performed on as-received and thermochemically treated five cotton yarn samples. As shown in Fig. 6a, the larger strain at the yield point of the treated sample indicates that an increased ductile compared to the untreated cotton. The Young's Modulus was calculated from the stress–strain curve as shown in Fig. 6b. While the elongation rate of the treated sample improves, yarns from the degraded sample have a decreased Young's Modulus and ultimate tensile strength compared to as-received sample. The tensile data is consistent with the internal structure observed with the SEM images. Please note that the tensile data was obtained from single fibers removed from the weave. The untreated fibers were observed to be composed of tightly bound partially oriented bundles of smaller strands. This highly organized structure was destroyed in the treated fibers, where were composed of loosely packed, randomly oriented strands. While the loose internal structure of the treated fibers decreased their overall tensile strength and modulus, it increased their elasticity and maintained the overall toughness of the fibers.

Conclusions

We have studied the deweaving phenomena of cotton fabric (e.g. muslin) under mild thermochemical reaction conditions (e.g. 50 °C) as a means of recovering staple fibers for recycled fabric. Cotton yarn was swelled and separated effectively without cellulose depolymerization using the sequence of chemical treatment: citric acid urea NaOH NH_4OH NaNO_3 . The Raman and FTIR spectroscopic techniques proved that the molecular structure of cellulose was not changed after thermochemical treatment, while the –OH stretching band was slightly shifted due to reductions in strength of inter/intra-hydrogen bonding. Although the molecular structure of treated muslin fabric was retained, the surface charge (zeta potential) of samples was significantly affected by thermochemical treatment, from – 2 mV (as-received) to – 8 to 14 mV (treated). It is proposed that the larger negative surface potential of the fibers, combined with mechanical agitation, facilitates deweaving of the cotton fabric. XPS results provided that while the polymeric structure of the cellulosic fibers is retained, the surface of degraded fabric is clearly altered by the treatment. In particular, photoelectron spectra indicated a significant increase in carbonyl/carboxylate ligands formed via reaction with carboxylic



acid and increases in sorbed water (related to swelling) and hydroxyl groups, as well as loss of any oxide present (likely related to contaminants from manufacturing processes). These findings prove the feasibility of the NaOH/urea/citric acid/ammonium hydroxide/sodium nitrate treatment at mild temperature ($\sim 50^{\circ}\text{C}$) for deweaving of cotton fabric for future recycling, via swelling and surface ligand binding to enhance the electrostatic repulsion. The current study also shows that woven interfiber structure of cotton muslin fabric can be degraded for subsequent reuse without full decomposition into constituent monomers or oligomers.

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Authors' information

Michael Cuiffo is a Research Assistant Professor at the Stony Brook University. Hye Jung Jung is a Professor at the Da Vinci College of General Education. Asta Skocir is a Professor at the Fashion Institute of Technology. Theanne Schiros is an Assistant Professor at the Fashion Institute of Technology. Emily Evans is a Junior student at the SUNY Polytechnic Institute. Elizabeth Orlando is a Junior student at the Ward Melville High School. Yu-Chung Lin is a Graduate student at the Stony Brook University. Yiwei Fang is a Graduate student at the Stony Brook University. Miriam Rafailovich is a Professor at the Stony Brook University. Taejin Kim is an Associate Professor at the Stony Brook University. Gary Halada is an Associate Professor at the Stony Brook University.

Authors' contributions

MC collected and analyzed the data. HJ participated in the manuscript preparation. AS participated in the manuscript preparation. TS participated in the manuscript preparation. EE collected the data. EO collected the data. TK analyzed and organized the data and prepared the manuscript. GH collected and analyzed the data and prepared the manuscript. YL collected and analyzed the data. YF collected and analyzed the data. MR analyzed data. All authors read and approved the final manuscript.

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Availability of data and materials

The data sets used and analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Competing interests

The authors declare that they have no competing interests.

Author details

¹Research Assistant Professor, Department of Materials Science and Chemical Engineering, Stony Brook University, Stony Brook, NY 11794, USA. ²Professor, Da Vinci College of General Education, Chung-Ang University, Seoul, Republic of Korea. ³Professor, Fashion Design Department, Fashion Institute of Technology, New York City, NY 10001, USA. ⁴Assistant Professor, Department of Math and Science, Fashion Institute of Technology, New York City, NY 10001, USA. ⁵Junior student, College of Nanoscale Science and Engineering, SUNY Polytechnic Institute, Albany, NY 12203, USA. ⁶Junior student, Ward Melville High School, East Setauket, NY 11733, USA. ⁷Graduate student, Department of Materials Science and Chemical Engineering, Stony Brook University, Stony Brook, NY 11794, USA. ⁸Professor, Department of Materials Science and Chemical Engineering, Stony Brook University, Stony Brook, NY 11794, USA. ⁹Associate Professor, Department of Materials Science and Chemical Engineering, Stony Brook University, Stony Brook, NY 11794, USA.

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