

Desizing of starch sized cotton fabrics with atmospheric pressure plasma

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Abstract Atmospheric pressure plasma is examined as a desizing agent for starch-sized cotton samples. Plasma treatment is examined both for its physical etching properties and for its ability to induce desizing of the starch during a scouring bath. The effect of the plasma gas composition and plasma exposure duration are evaluated. A 2-min helium/oxygen plasma is found to be the only condition that appears to improve desizing of starch.

Keywords Cotton · Starch · Desizing · Plasma

Introduction

Water pollution by textile mills is a constant source of media coverage and public health concern, particularly in China and other developing nations (Yardley

2013; Wencong 2012; Hewson 2013). As a response to this mounting crisis, governments are increasing fines and global textile brands are pushing their mill suppliers to reduce water pollution (Chu 2015). Reduction of effluent for fabric and fiber processing is therefore of great interest to the textile industry.

Cotton is a natural fiber grown domestically and in many foreign markets with production levels exceeding 80 million bales per year (Johnson et al. 2014). Weaving of cotton yarn into fabric requires application of size to warp yarns prior to weaving, and subsequent removal of size prior to fabric finishing. Starch, the most commonly used size for cotton fabric, is a common natural resource produced from rice, potatoes, corn and or indigenous plants (Moreau 1981; Moreland 1980). Starch is water soluble when applied to cotton warp yarns but loses water solubility upon drying. Conventional removal of starch is performed in a solution of enzymes and other chemicals and consumes significant quantities of water.

Conventional starch desizing and scouring is a two-step process. First, cotton is desized using an enzymatic desizer to remove the starch size. After starch removal, samples are scoured at high temperatures in a caustic to remove oils and lubricants. These oils and lubricants can interfere in the dyeing process.

Plasma processing represents a green alternative to enzymatic desizing that uses electric fields and energized particle bombardment, rather than water and chemicals, to remove size. Plasma is composed of active particles including electrons, ions, excited

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species, and UV radiation that can react chemical and physical with the surface of an introduced substrate. Use of atmospheric pressure plasma to remove PVA size from cotton, rayon, and polyester/cotton blends has already been successfully investigated (Matthews et al. 2005; Cai et al. 2003a, b; Bae et al. 2006), but the application of atmospheric pressure plasma to remove starch size has not been explored.

Vacuum plasma treatment of granular starch and starch films has been shown to reduce molecular weight and induce depolymerization (Lii et al. 2002b), increase oxidation (Lii et al. 2002a), or alternately, under different plasma conditions, to increase cross-linking and reduce solubility (Zou et al. 2004). Vacuum conditions leads to large mean free paths and very energetic ions and electrons capable of rapid and extreme surface modification (Mori et al. 1994). Due to limitations in vacuum systems included expensive vacuum equipment, long pump-down and treatment times, and a restriction to batch processing, industrial application of vacuum plasma treatments of textiles has not been widely accepted (Hocher et al. 2003). However, the advent of atmospheric pressure plasma processing (Yokoyama et al. 1990) represented a new opportunity for industrial use of plasmas, and a significant array of commercial devices are now available. With significant modifications in gas and processing parameters, atmospheric plasma can realize nearly all of the kinetic processes and potential for surface activation of vacuum plasma (Shenton 2001).

Atmospheric pressure plasma processing offers the potential to eliminate or reduce water consumption in the desizing process. This research examines the effects of different gas mixtures and durations of plasma treatment on direct starch removal, and removal of starch during scouring.

Experimental

Materials

Samples

Fabric used in the experiments was commercially starch-sized cotton fabric woven in a 1 × 2 twill. One inch of fabric consisted of 105 and 52 filling and warp yarns at a mass of 0.1870 g (43% weft and 57% warp

by mass). Fabric was cut into 12.7 cm × 20.32 cm strips and labeled.

Plasma treatment

Plasma treatment was performed on the NCAPS (North Carolina Atmospheric Pressure System), built at North Carolina State University and located on Centennial Campus in the College of Textiles (Fig. 1).

NCAPS is a dielectric barrier discharge (DBD) plasma with two 60 × 60 cm copper electrodes imbedded in 2 cm thick polycarbonate Lexan™ dielectric. The electrodes are housed inside an inner and outer chamber that allow for venting of the plasma gas and provide shielding from the high voltage. Voltage is supplied to the device by eight Pyramid 15 V direct current power supplies coupled together in parallel and alternated by a 4011 5 MHz BK Precision Function Generator. The voltage passes through two step-up transformers 180° out of phase to both the upper and lower electrodes. The function generator is tunable, but most operations are performed at 1.3 kHz where power is maximized.

Voltage, current, and ambient gas temperature are all monitored by an oscilloscope and computer with solvers for power, electron number density, electron temperature, and electron-neutral collision frequency. A LabView® program monitors these parameters and the flow of gas out of the Mass Flow meters. Data can be viewed in real time on both the oscilloscope and in the LabView® system.

To generate a plasma discharge, helium gas is flowed between the electrodes. Helium is always used as the primary discharge gas due to its low ionization potential. Other gases including oxygen, CF₄, methane, forming gas, and nitrogen can be introduced into the helium stream where they dissociate and ionize through collisional processes. These secondary gases are added in very small percentages, usually only 1–2% by mass. All gases are regulated by MKS Mass-Flo Controllers connected to a MKS 4-channel controller and readout unit.

Samples were treated with plasma using a batch treatment cell (Fig. 1). The cell is located between the upper and lower electrodes with inlet holes for gas flow. Use of the cell reduces gas consumption and improves the quality of the plasma. The cell was manufactured from 1.3 cm transparent, polycarbonate Lexan™ with an internal area of 43.2 cm by 43.2 cm.

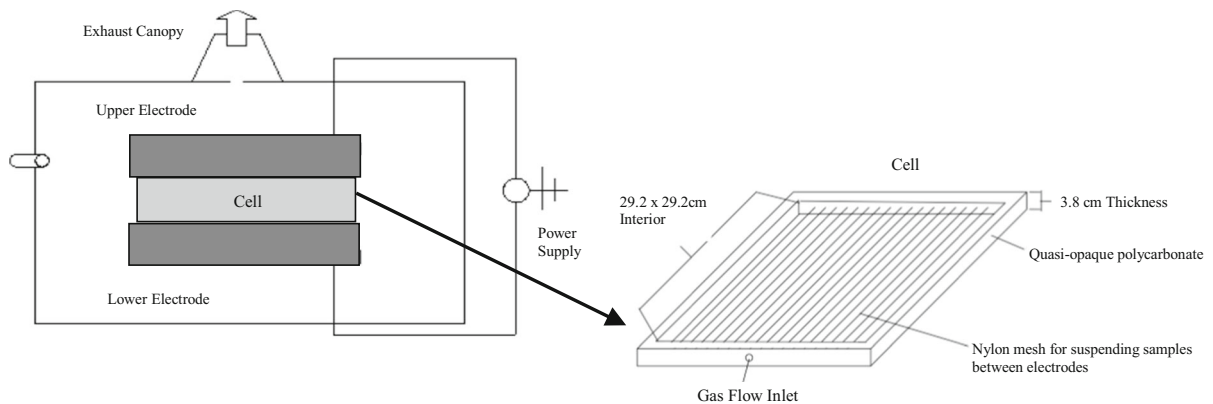


Fig. 1 NCAPS atmospheric pressure plasma device

A nylon mesh in the center of the cell allows samples to be suspended equidistant from both plasma electrodes and provides plasma treatment on top and bottom of the sample. The thickness of the cell and the electrode gap spacing are fixed at 3.8 in.

Procedure

Atmospheric pressure plasma was examined as both an etchant and a starch solubilizer for cotton fabric sized with starch. Plasma etching occurs as a result of high-energy plasma particles colliding with and volatilizing the starch coating. Increased solubility is a result of a reduction in molecular weight and increased surface polarity.

Starch-sized cotton samples were treated with atmospheric pressure plasmas of different gas composition for varying durations ranging from 30 s to 10 min. Samples were weighed before and after plasma exposure to determine the change in mass resulting from plasma etching. Immediately after the plasma exposure and weighing, samples were scoured in a conventional scouring process. Less than 60 s is allowed to elapse between plasma treatment and scouring, to minimize changes to the surface composition that may occur over time upon exposure to ambient environment (Pykonen et al. 2008). After scouring, samples were weighed to determine if additional size was removed. Four samples were treated for each experimental condition.

Conditioning and weighing

Plasma etching and solubilization of samples was determined by change in mass. Samples were weighed after 24 h of conditioning under controlled temperature and humidity conditions, prior to plasma treatment and after scouring, at 65% humidity, 20 ± 5 °C, using a O Haus' Explorer balance with four significant decimals. Weight measurements were also taken at ambient conditions in the laboratory immediately before and after plasma treatment using a Mettler Toledo AB 104-S/FACT balance with a sensitivity of ± 1000 g.

Conventional desizing

Total weight of starch on the greige cotton fabric samples was determined by enzymatic desizing with Rapidase™. Enzymatic desizing was accomplished using a 24:1 liquor ratio in a Gibbs Machine. Samples were placed on spiral holders and desized for 30 min at 80 °C. The desizing bath consisted of 2% Rapidase™, 4% wetting agent (Renex), and 7% of a 25% NaCl solution. Treatment was terminated by rinsing the fabric in warm water for 5 min. Samples were then air-dried and conditioned as described above to determine amount of desizing by change in mass.

Desizing by plasma treatment

Desizing was conducted on the NCAPS (North Carolina Atmospheric Plasma System) machine. The

device's electrodes were warmed for 15 min in pure helium discharge prior to sample treatment. Samples were then placed equidistant from the two electrodes on top of a nylon mesh and treated individually for the specified time. The times chosen for treatment were 0.5, 2, 5, and 10 min.

Four gas blends were selected for testing. All gas blends contain helium to sustain the plasma discharge with or without small fractions of other gases. Both oxygen (O₂) and carbontetrafluoride (CF₄) are 'reactive' plasma gases, capable of dissociating into various chemical compounds including O*, O₃*, F*, F₂, etc., whereas helium and argon are noble gases, capable only of excitation and ionization.

The four gas mixtures investigated were:

20.00 L/min Helium

20.00 L/min Helium/0.30 L/min CF₄

20.00 L/min Helium/0.30 L/min O₂

20.00 L/min Helium/0.30 L/min Argon

Scouring

After plasma treatment, samples were weighed and immediately scoured. Scouring consisted of 30 min at 156 rpm in a New Brunswick Scientific Model G76D Gyrotoray® Water Bath Shaker (Edison, NJ). Each sample was placed in an individual beaker with a scouring solution of 3.0 g/L 50% NaOH, 1.5 g/L Lufibrol KB (surfactant), and 1.5 g/L Kierton NB (lubricant) at a liquor ratio of 20:1.

Characterization techniques

Mass calculations

Each sample was weighed immediately before plasma treatment (W_0), immediately after plasma treatment (W_p), and after scouring and conditioning (W_s) using an Explorer microbalance with an accuracy of ± 1000 micrograms.

Quantity of size present was calculated based on the individual mass of each sample, for initial quantity of size (S_0), size remaining after plasma treatment (S_p), and size remaining after scouring (S_s). Several formulas were used to determine etching ability of the plasma, solubilization of the size in the scouring bath, and the overall desizing of samples treated by plasma and scoured.

Plasma etching :

$$\% \text{ Change in Mass} = 100 * \left(\frac{W_p - W_0}{W_0} \right) \quad (1)$$

Plasma desizing :

$$\% \text{ Removal} = 100 * \left(\frac{S_p - S_0}{S_0} \right), \quad S_p = \left(\frac{W_p - W_0}{W_0} \right) \quad (2)$$

Desizing and scouring :

$$\% \text{ Removal} = 100 * \left(\frac{S_s - S_0}{S_0} \right), \quad S_s = \left(\frac{W_s - W_0}{W_0} \right) \quad (3)$$

Scouring :

$$\% \text{ Removal} = 100 * \left(\frac{S_s - S_p}{S_p} \right) \quad (4)$$

For the purposes of this research, it will be assumed that all loss in weight can be attributed to size removal and that cotton remains unaffected by plasma treatment. Although there may be minor weight loss due to etching of the cotton fibers themselves, majority of weight loss is likely due to the preferential etching of the amorphous starch coating. In addition, generation of oxygen functionalities on cotton and remaining starch may also modify mass readings by increasing surface water adsorption.

Tensile testing

Tensile testing was conducted for all samples using a MTS Q-Test/5 with an Elite Controller attachment according to ASTM D 5305-95 Standard Test Method for Breaking Force & Elongation of Textile Fabrics (Strip Method). A 113.4 kg load cell was used with attached leather grips to prevent slippage. The system was connected to a computer running Testworks 4 software package, which analyzed and recorded the data. For each experimental condition, four samples were cut along the grain into 2.5 cm \times 15 cm strips. Each strip was then conditioned for 24 h in the testing facility at 65% humidity, 20 ± 5 °C, at ambient pressure. After conditioning, each strip was individually tested and averages were taken for each trial.

Scanning electron microscopy (SEM)

All samples were sputter-coated with gold on a SC 760 Mini Sputter Coater from Quorum Technologies for 45 s and examined in a Phenom Scanning Electron Microscope from FEI Company (Hillsboro, Oregon, US).

Iodine dyeing

The TEGEWA starch indicator test was performed according to the standard. Indicator was prepared by dissolving 10 g of potassium iodide in 100 mL of deionized water with 0.635 g iodine. Solution was then added to 700 mL of deionized water and 200 mL of ethanol for a total of 1 L solution.

Starch samples were cut using a template into 3.81 cm × 3.81 cm squares and the iodine solution was applied with a paintbrush. Samples were then rated according to the TEGEWA scale.

Dyeability

Starch-sized samples were dyed to examine the effect of plasma treatment on dyeability. Samples were dyed in a 2% Remazol Brilliant Blue R Special MIS by DyStar (Reactive Blue 19) with 60 g/L anhydrous sodium sulfate, 7 g/L Fumexol 100 (non-ionic defoamer) in a Werner Mathis AG Lab Jumbo Jet JFO with basket assembly. The samples were agitated at 12 m/min for 30 min at 32 °C, then 2.5 g/L NaOH was added and the temperature increased to 60 °C over 30 min. 2.5 g/L NaOH was then added and temperature increased for another 30 min to 140 °C. Samples were then rinsed sequentially in cold water for 20 min and in hot water for 10 min. Samples were then centrifuged for 30 s in a Bock Centrifugal Extractor to remove excess water, and pressed with a warm iron.

Spectrophotometry

Dyed samples were evaluated for depth of shade by evaluating the absorption coefficient (K/S) values for each sample and comparing with an undyed control sample using a X-Rite Spectrophotometer (McDonald 1987). GretagMacbeth Color iControl software was employed for the color analysis and the wavelength with the greatest absorbency was chosen (600 nm).

Results and discussion

Size removal

Enzymatic desizing and scouring

Weight measurements were evaluated to determine quantity of size removed during the plasma treatment process and the subsequent scouring.

A set of eleven samples were enzymatically desized with the RapidaseTM and exhibited an average size removal of 8.82% o.w.f. with a standard deviation of 0.33. Five samples were reserved for testing and the rest scoured conventionally (Table 1).

Average removal from the scour bath of the conventionally desized samples was 1.70% with a standard deviation of 0.17%. For conventional desizing with scouring, an average of 10.31% mass o.w.f. was removed from the cotton with a standard deviation of 0.50%.

Plasma etching

Four different plasma gases were used to treat the starch-sized cotton samples for durations of 0.5–10 min. The decrease in weight after plasma treatment is illustrated in Fig. 2. Clearly, atmospheric pressure plasma successfully etched a large quantity of starch from the cotton samples. Regardless of gas used, plasma exposure durations were directly related to reductions in mass.

Gas composition was also a significant factor in plasma etching. Reactive plasma gases (CF₄ and O₂) caused a decrease in mass for the entire 30 min, whereas for non-reactive gases, mass first decreased and then increased. This is indicative of a redeposition of etched material between 30 s to 2 min of exposure, followed by a steady decrease thereafter.

The etching behavior of all gases was modeled by two rates, an initial rate of etching (0–30 s) and a rate of etching from 30 s to 10 min. Rates of etching for the non-reactive plasma gases is significantly higher (Table 2).

The rate of etching for all gases decreased after 30 s, and the reactive gases, O₂ and CF₄ were observed to etch at a higher rate than He and Ar. Initial etching rates are attributed to pure etching, whereas chemical reactions may contribute to the later rates of etching for the reactive gases.

Table 1 Enzymatic desizing and scouring

| Enzymatic desizing size removed (% of mass) | Scouring mass removed (% of mass) |
|---|-----------------------------------|
| 9.27 | 1.55 |
| 8.78 | 1.73 |
| 8.44 | 1.45 |
| 8.74 | 1.90 |
| 8.63 | 1.70 |
| 8.21 | |
| 9.05 | |
| 8.95 | |
| 8.59 | |
| 9.04 | |
| 9.28 | |
| <i>Average</i> | |
| 8.82 | 1.70 |
| <i>Stdev</i> | |
| 0.3312 | 0.17 |

Fig. 2 Etching of starch sized cotton by plasma

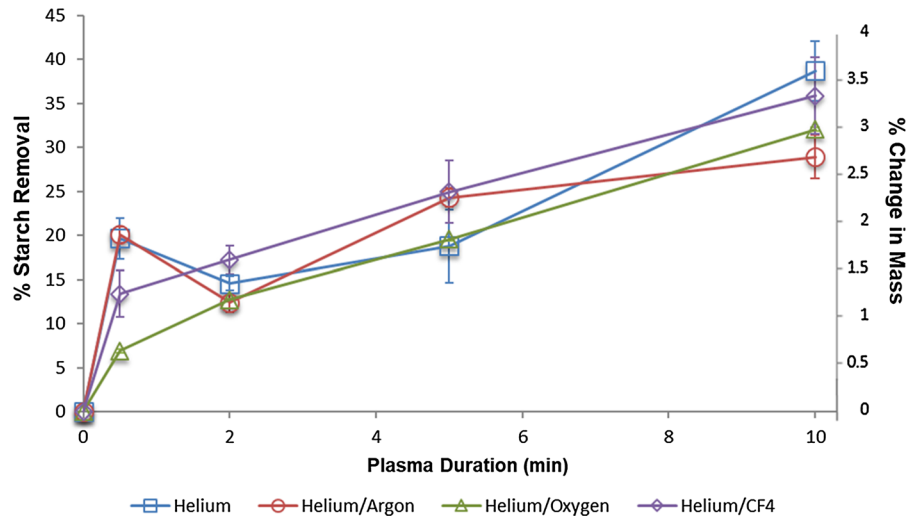


Table 2 Etching rates of plasma

| Gas blend | Etching rate (0–0.5 min) | Etching rate (0.5–10 min) |
|------------------------|--------------------------|--------------------------------------|
| Helium | $Y = 3.4619x$ | $Y = 0.1985x + 1.1511, R^2 = 0.772$ |
| Helium/Oxygen | $Y = 1.2132x$ | $Y = 0.2268x + 0.5764, R^2 = 0.9942$ |
| Helium/Argon | $Y = 3.5437x$ | $Y = 0.1178x + 1.3725, R^2 = 0.6369$ |
| Helium/CF ₄ | $Y = 2.3579x$ | $Y = 0.2082x + 1.1023, R^2 = 0.9981$ |

After two minutes of plasma treatment, the etching behavior of the reactive gases becomes linear, and can be fit to linear curve with R^2 values greater than 0.99.

If the linear depletion of starch were to continue indefinitely, complete plasma etching would be accomplished after about 40 min of plasma exposure.

Complete desizing was not expected because some starch is blocked from plasma exposure by filling yarns, redeposition of removed starch is likely, and rate of removal will diminish when a uniform starch coating is no longer present.

Plasma etching was also examined as the amount of size removed in relation to the quantity remaining (Fig. 2, left y axis). After 10 min in the plasma, between 30 and 40% of the starch has been removed by the plasma treatment irrespective of plasma gas mixture. Size removal is affected by the nature of the gas, and $He > He/CF_4 > He/O_2 > He/Ar$ for desizing of starch from cotton.

Plasma solubilization

After treatment by the atmospheric pressure plasma, samples were immediately scoured. Changes in mass due to scouring are shown below, both for the combined plasma/scouring process and for only the scouring bath. Percentage removal of remaining starch by scouring (rather than percentage removal of the overall initial quantity) is shown in Fig. 3.

The greatest quantity of size was removed with a helium/oxygen treatment of 2 min. All other plasma durations and gases do not remove significantly more size than the control. Treatment of samples with helium/CF₄ gas even caused a reduction in overall desizing and scouring. It is also interesting to note that 84.78% of starch is removed from the control during scouring. This is quite a large quantity of starch that was removed with only hot water.

To determine the effect of plasma-treatment on solubility of the starch, the quantity of starch etched

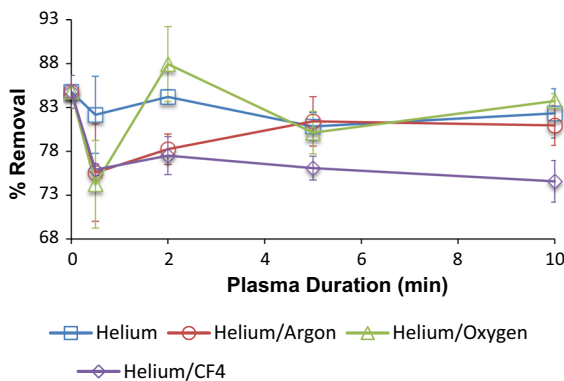


Fig. 3 Desizing of starch-sized cotton by plasma and scouring

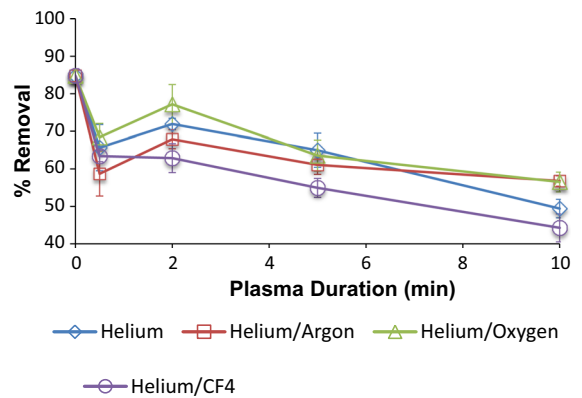


Fig. 4 % Removal of weight of scoured samples, excluding weight loss due to plasma treatment

off by plasma was excluded (Fig. 4). The percentage of starch and impurities removed is significantly reduced for the samples treated in plasma when compared to the control. As plasma duration increased, the mass removed during the scouring bath decreased. However, as plasma duration increased, less starch remains on the samples to be removed during scouring.

To address the differing quantities of remaining starch and determine if plasma increases the water solubility of the remaining starch, the scouring was replotted based on the ‘remaining’ quantity of starch and residues (Fig. 5). For all samples, excluding the helium/oxygen at 2 min, the removal of starch and residues was distinctly less than for the control. As treatment duration increases after 2 min, the rate of % removal also decreases, suggesting cross-linking of the starch by the plasma. Cross-linking reduces the

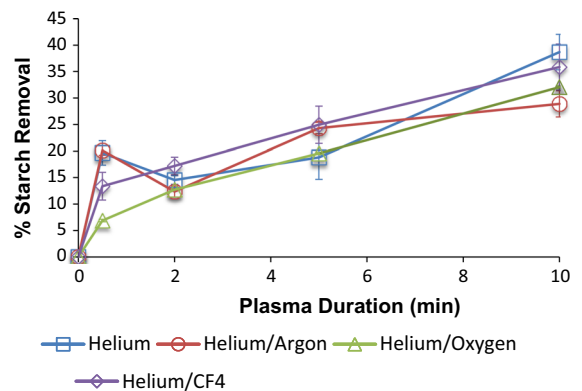


Fig. 5 % Removal of remaining starch after plasma desizing

solubility of the starch, decreasing removal by the scouring bath.

Tensile testing

Starch size is applied to cotton warp yarns to increase strength and reduce breakage during the weaving process; Fabric strength is therefore expected to decrease after desizing. Plasma treatment of fabric has been shown to alter fabric tensile properties as well, increasing or decreasing strength depending on device type and gas parameters (Cioffi et al. 2002).

The peak load of the plasma treated samples, shown in Fig. 6, did decrease slightly over time for all but the 99% helium/1% argon plasma, which steadily increased in tensile strength after an initial drop at 0.5 min. After scouring, all samples treated with plasma had a significantly higher peak load than the control sample desized with Rapidase™ and scoured. Samples exposed to the 99% helium/1% oxygen plasma, which was most thorough at desizing (93%),

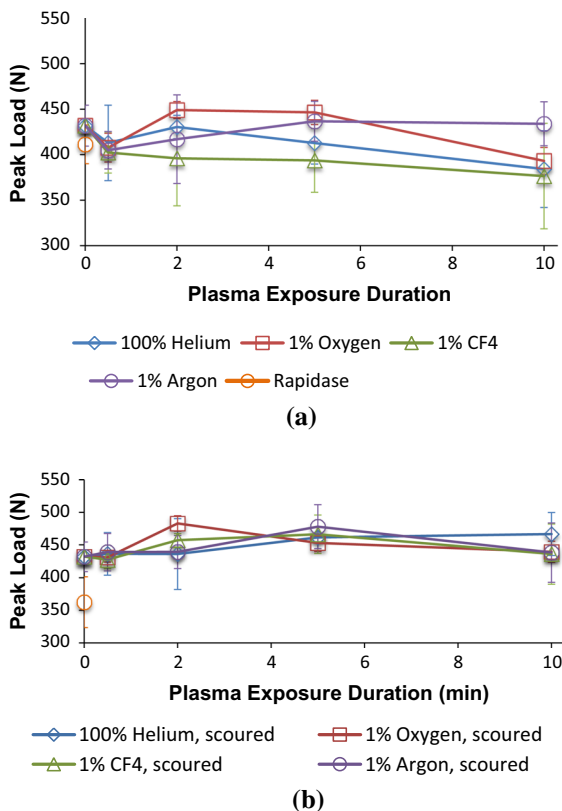


Fig. 6 Peak load for **a** plasma-treated, and **b** plasma-treated and scoured cotton

had the highest peak load and a peak load significantly greater than the 100% desized Rapidase™ sample.

Modulus for the cotton samples is expected to remain unchanged. Since strength is increased due to cross-linking of the polymer chains, a reduction in elongation is expected.

After scouring, the plasma-treated and Rapidase™ treated samples are all similar in elongation (Fig. 7), regardless of duration of plasma exposure. Only the sample set scoured, but not desized by plasma or Rapidase™ was not affected.

Iodine test: TEGEWA

Samples were dyed with a starch indicator to examine quantity of starch remaining and the samples were rated according to the TEGEWA scale (Figs. 8, 9). Dark blue-purple coloration indicated presence of starch, and samples with little to no color change were rated a 9. Variation in color was supposed to be

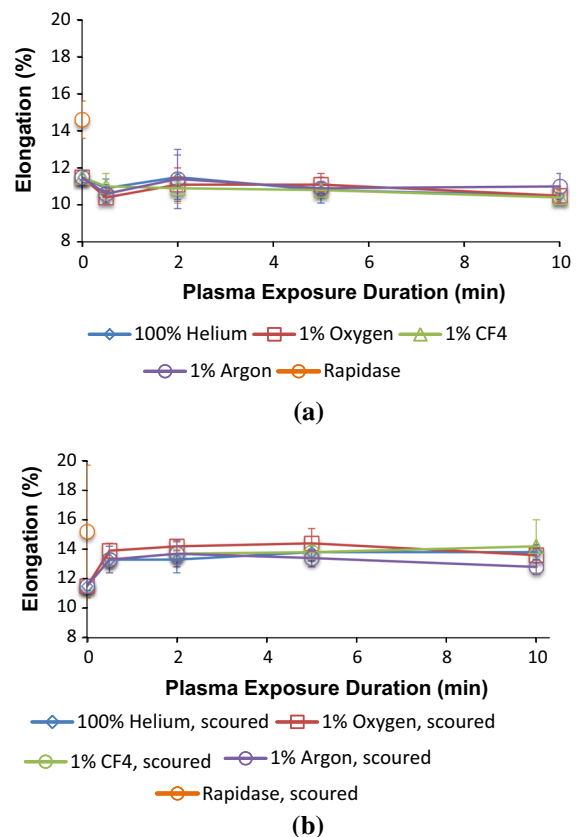


Fig. 7 Elongation for **a** plasma-treated, and **b** plasma-treated and scoured cotton

Fig. 8 Iodine stain test for indication of starch presence on unscoured, plasma treated samples immediately and after 24 h

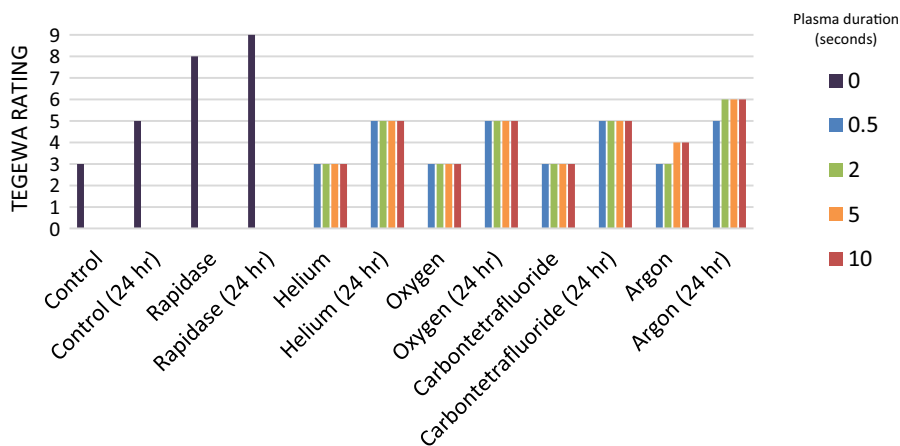
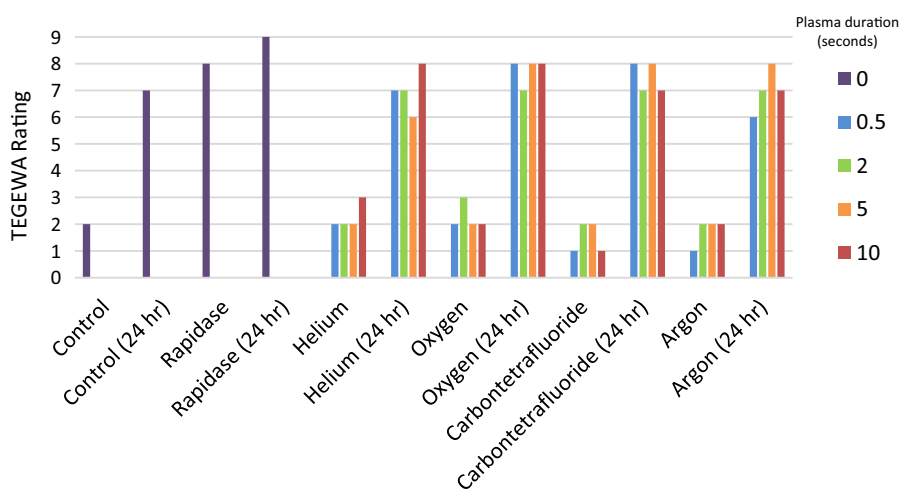


Fig. 9 Iodine stain test for indication of starch presence on scoured, plasma treated samples immediately and after 24 h



indicative of concentration of starch, but very little variation in color was noted. After 24 h, reduction in surface iodine revealed some reduction in starch presence. The conclusions to be drawn from the iodine staining test are that (1) enzymatic desizing does remove all starch and (2) plasma-treatment with subsequent scouring removes some additional starch, but that some starch still remains. These assertions are supported by the desizing data obtained from changes in mass (Figs. 2, 3).

Reactive dye test: spectrophotometry

The effect of plasma treatment on dyeability of the cotton fabric was assessed by spectrophotometry on samples dyed with reactive blue 19 (Fig. 10). Samples enzymatically desized, enzymatically desized and

scoured, or scoured control samples all have similar K/S values. Scouring of the control samples increases K/S.

Plasma treated scoured samples behave interestingly. Long durations of plasma treatment significantly increase the K/S values of unscoured samples. Alternately, K/S values decrease as plasma exposure increases for scoured samples. The highest K/S values are obtained at 2 min for the 1% oxygen plasma, unscoured.

Sized cotton is typically scoured to improve dyeability. It is notable that dyeability of the unscoured samples can be increased by oxygen plasma treatment to a comparable level to the conventionally desized and scoured cotton sample.

Fig. 10 K/S values of starch-sized cotton dyed with reactive blue 19

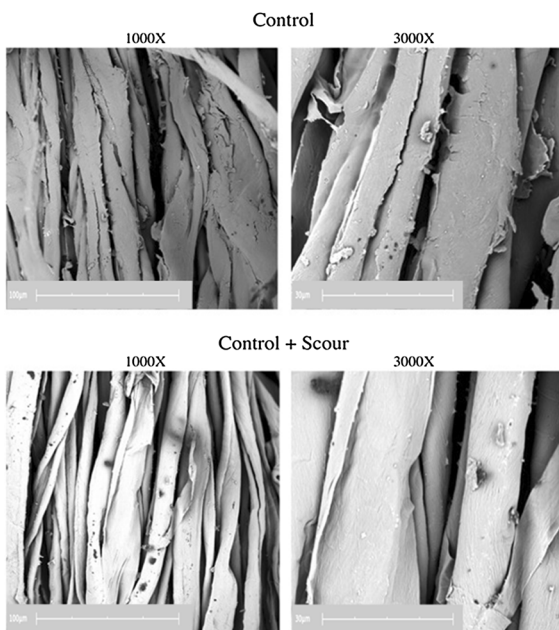
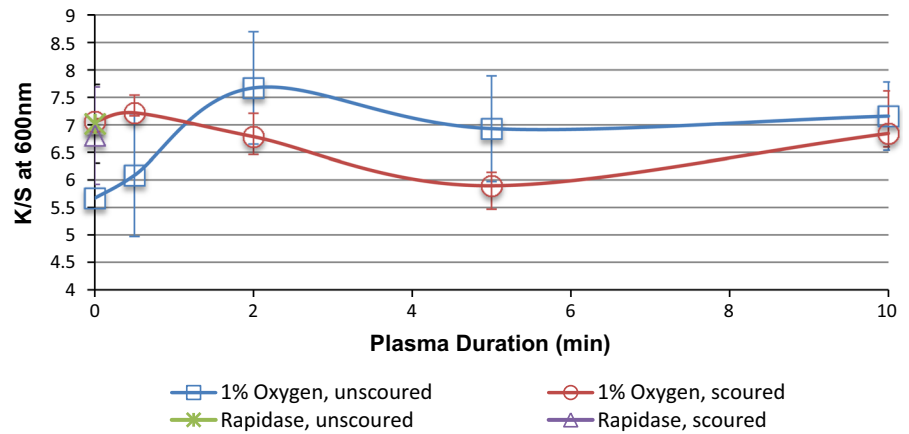


Fig. 11 SEM of starch-sized control and scoured samples

SEM

An electron microscope was used to examine the surface of the starch-sized fabrics (Figs. 11 and 12). Presence of size is clearly visible on the control sample as a thick, flat coating around the yarns. Treatment of the starch-sized samples by Helium/Oxygen plasma visibly removes some of the starch size. As seen at the 3000 × magnification, remaining size is located between fibers, where plasma penetration is limited. After washing, plasma-treated fibers are visibly cleaner than the controls, as suggested by the mass data.

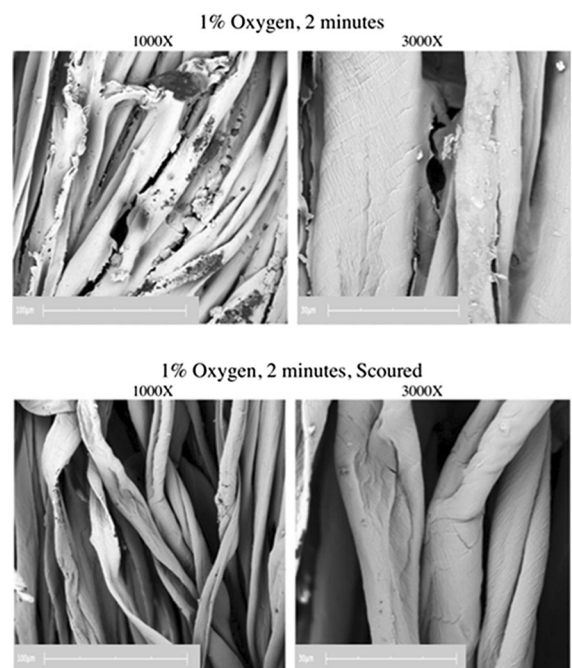


Fig. 12 SEM of starch-sized plasma treated and scoured samples

Conclusions

Atmospheric pressure plasma is identified as a method of starch desizing of cotton samples. Nearly 40% of starch can be removed after 10 min of plasma exposure with a linear rate of depletion. Scouring of the plasma treated samples does not remove significantly more size than removed for a scoured control, with the exception of one experimental condition: 2 min of helium/oxygen plasma. For most plasma

treatments and durations, remaining size is more resistant to removal during scouring, indicating that starch not etched by the plasma has been cross-linked.

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