A Novel Non-Aqueous Fabric Finishing Process

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Abstract

A comparison of properties between fabrics treated in vacuum and atmospheric plasma chambers is underway. Both sized and desized denim fabrics treated in fluorocarbon plasmas under vacuum have shown a significant improvement in hydrophobicity. The effect was greater for desized than for sized fabric. Fabrics will be treated in atmospheric plasmas of air/He and air/O₂ as controls, then in air/fluorocarbon plasmas. These fabrics will be compared to those treated with fluorocarbon plasmas in vacuum. Plans have been developed for a new atmospheric plasma chamber to be housed at the College of Textiles at NCSU. This device will allow for controlled treatment variation throughout a fabric roll and continuous fabric processing.

Project Goals

The initial goals for this project were:
1. To investigate non-aqueous processes for finishing textile products.
2. To create a continuous non-aqueous fabric finishing system, encompassing desizing, scouring, dyeing, and finishing.

In response to the feedback received from the Technical Advisory Committee, the focus will be on investigation of non-dyeing finishing processes.

Introduction

Plasma treatments have been used to induce both surface modifications and bulk property enhancements of textile materials, resulting in improvements to textile products ranging from conventional fabrics to advanced composites. These treatments have been shown to enhance dyeing rates of polymers, improve colorfastness and wash resistance of fabrics, and change the surface energy of fibers and fabrics. Research has shown that improvements in both toughness, tenacity, and shrink resistance can be achieved by subjecting various thermoplastic fibers to a plasma atmosphere. Recently, plasma treatments have been investigated for producing hydroscopicity in fibers, altered degradation rates of biomedical materials (such as sutures), and for the deposition of antiwear coatings.

Plasma treatment may be performed either in vacuo or at atmospheric pressures. There are advantages and disadvantages to each process:

* Vacuum processes:
  * usually not viable for industries requiring large amounts of throughput
  * vacuum systems are expensive, and take up lots of space
* a wide variety of plasma chemistries can be used since it is a closed system.
* Sample size is limited by system configurations
* Vacuum processes and fundamental physics are well understood.

**Atmospheric processes:**
* great for industries requiring large amounts of throughput (such as the textile industry)
* Plasma chemistries are limited - must have environmental enclosure when using certain gases (those producing unfavorable by-products)
* Sample size is practically unlimited (depending on the configuration of the sample)
* Processes should be more suited to organic materials (due to lower temperatures, etc.)
* Technology is still relatively new, and not as much is understood concerning the plasma physics and chemistries.

**Experimental**

The primary goal of the preliminary experiments conducted for this project was to compare the vacuum plasma processes, inductive and capacitive, to the atmospheric plasma process. Currently we are processing denim fabric in order to make it hydrophobic. We have used both CF$_4$ and C$_3$F$_6$ treatments in the vacuum process and plan to run these gases in an atmospheric process. Treatments made in the atmospheric process so far have been used to establish a baseline for the effects of other elements (e.g. oxygen, helium, and nitrogen) in the system. Contact angle measurements on samples of the treated material have been made to compare the hydrophobicity of the treated materials.

**Materials:**

The material tested thus far is denim fabric, sized and desized. All of the denim was made by Mt. Vernon Mills. Samples tested on all apparatus were 6x6 inches. This is the size limit for treatment in the atmospheric chamber as well as the second vacuum chamber used. Desizing was performed by treatment with $\alpha$-amylase enzymes at around 120°F. AATCC standard methods were used.

**Plasma Devices:**

Vacuum treatments were initially performed in a RF Plasma Products AMN 3001 E plasma chamber, in both inductive and capacitive modes, on both sized and desized denim fabrics. Power varied between 100 and 300 watts at various durations. Pressure in the chamber was between 50 and 75 mTorr. Flow rate was kept consistent at 50 sccm. During testing, technical difficulties with the chamber resulted in the need to switch to a new but similar chamber (Figure 1). Variables were maintained with the exception of chamber pressure, which was allowed to go to 150 mTorr. Wattage in the second chamber ranged between 50 and 160. Treatments were performed for either 30 or 60 seconds.
Atmospheric treatments were performed in a custom chamber designed and fabricated by the Nuclear Engineering Department at NC State (Figure 2). This capacitively coupled device is operable at frequencies between 1 and 12 kHz. The voltage between the plates can be up to $7.8kV_{rms}$ and the electrode gap spacing is variable. A plasma is typically stabilized at frequencies between 6 and 9 kHz, depending upon the gas.

Treatments:

Thus far, we have treated denim fabric under vacuum plasma processes, capacitive and inductive, with both CF$_4$ and C$_3$F$_6$ gases. The experimental matrix arranged for these experiments varied the chamber pressure, power, and time. For the CF$_4$ gas treatments, sized and desized fabrics were treated with each possible combination of variable treatments. The pressure differed at 50mTorr and 75mTorr, the power at 100W and 300W, and the time at 30 and
60 seconds. The CF₄ treatments were also completed under capacitive and inductive mode. For the C₃F₆ treatments, a similar matrix was used but some of the treatment values changed. The pressure differed at 50, 100, and 150mTorr, the power at 50W and 160W, and the time again at 30s and 60s. These treatments, as of now, have only been conducted in capacitive mode.

For the atmospheric plasma device, no treatments have been conducted using etchant gases such as the CF₄ and C₃F₆. With the device operating at atmospheric pressure air will never be absent regardless of the type of gas treatment. Since any treatment in this device must consider a gas combination with air, experiments to evaluate background effects must be conducted. Therefore, treatments on denim fabric, sized and desized, have been conducted using an air-helium mixture. Air-oxygen mixture plasma treatments on the fabric will soon be evaluated as well. For the air-helium experiments, the plasma stabilized at 6 kHz with a 5 psi delivery pressure. These values remained constant with only the time in the chamber varying. The time values for this experiment were 0s (in the chamber), 30s, 60s, 120s, and 240s. Air-oxygen experiments will be conducted varying the delivery pressure (three different flow rates) and the time (0, 1, 2, 3, 4, 5, 10 minutes).

Fabric Analysis and Results:

The only characterization test done on the fabric so far has been the measurement of wettability. This has been done through the use of the goniometer in measuring the contact angle of the fabric after treatments. The contact angle was measured for each treated fabric at one minute intervals up to five minutes. The time of wet-out was also recorded. Figure 3 shows the device used (goniometer).

Figure 3: Contact angle measurements using the goniometer

The following charts (1-3) summarize the data collected thus far. It consists of average values of all the data collected. Most measurements were taken four times for the same sample. The blank chart cells are samples that have yet to be measured, however the treatment for these samples are complete. The cells containing N/A are measurements where all of the samples wet out too fast to measure an initial angle.
Table 1: Average Time (Seconds) to Wet Out Fabric

<table>
<thead>
<tr>
<th>Treatments</th>
<th>Desized Front</th>
<th>Desized Back</th>
<th>Sized Front</th>
<th>Sized Back</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air-He Atmospheric</td>
<td>1.6</td>
<td>1.6</td>
<td>6.7</td>
<td>6</td>
</tr>
<tr>
<td>C3F6, Capacitively Coupled</td>
<td>291</td>
<td>159</td>
<td>183</td>
<td>107</td>
</tr>
<tr>
<td>CF4, Capacitively Coupled</td>
<td>66</td>
<td>3</td>
<td>1.4</td>
<td>23</td>
</tr>
<tr>
<td>CF4, Inductively Coupled</td>
<td>214</td>
<td>154</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Table 2: Average Initial Contact Angle

<table>
<thead>
<tr>
<th>Treatments</th>
<th>Desized Front</th>
<th>Desized Back</th>
<th>Sized Front</th>
<th>Sized Back</th>
</tr>
</thead>
<tbody>
<tr>
<td>Air-He Atmospheric</td>
<td>N/A</td>
<td>N/A</td>
<td>26</td>
<td>8</td>
</tr>
<tr>
<td>C3F6, Capacitively Coupled</td>
<td>291</td>
<td>159</td>
<td>94</td>
<td>82</td>
</tr>
<tr>
<td>CF4, Capacitively Coupled</td>
<td>98</td>
<td>7</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CF4, Inductively Coupled</td>
<td>101</td>
<td>88</td>
<td>N/A</td>
<td>92</td>
</tr>
</tbody>
</table>

Table 3: Control Fabric Measurements

<table>
<thead>
<tr>
<th>Fabrics</th>
<th>Initial Contact Angle</th>
<th>Total Time to Wet out (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sized Front</td>
<td>N/A</td>
<td>5</td>
</tr>
<tr>
<td>Sized Back</td>
<td></td>
<td>7.8</td>
</tr>
<tr>
<td>Desized Front</td>
<td>N/A</td>
<td>1</td>
</tr>
<tr>
<td>Desized Back</td>
<td>N/A</td>
<td>1.5</td>
</tr>
</tbody>
</table>

The treatments with CF₄ plasma in both capacitive and inductive settings have yielded variable and inconsistent results. This could be due to fabric type, non-uniformity in the plasma, or simply the lack of a chemical reaction with this particular gas due to strong bond energies in the denim. Most treatments with the C₃F₆ plasma, done in capacitive mode only, have been successful in giving the fabric hydrophobicity consistently. We believe this is due to the tendency of C₃F₆ to form a type of film over the fabric. Sized and desized denim fabrics have also been compared in each of the experiments. Desized fabric appears to allow for better treatments when treated under the same conditions as the sized fabric. No tests have yet been done to analyze the durability of these treatments.

**Future Plans:**

In the near future, the background experiments in the atmospheric plasma device will be completed using the air-oxygen plasmas. From there we will begin using both CF₄ and C₃F₆ gases to treat the fabric. We will switch from denim fabric to nylon airbag fabric for a more
clear and simplistic analysis of the effect of the treatments. More treatments may be conducted under vacuum (and atmospheric) using a variety of gases so that the two can be more easily compared.

There has also been a prototype proposed from the Nuclear Engineering department here at NC State for a new atmospheric device for textiles. A diagram of this device is shown below in Figure 4. Designers include Dr. Orlando Hankins, Brian Bures, Tamer Lowry, and Dr. Mohamed Bourham as a consultant. This device will allow for controlled treatment variation throughout a fabric roll and continuous fabric processing. With the implementation of a device such as this, atmospheric plasma treatment for textiles can be further explored and optimized.

Figure 4: Proposed prototype of atmospheric plasma device for textiles.
Acknowledgements

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