



# Influence of moisture on wettability and sizing properties of raw cotton yarns treated with He/O<sub>2</sub> atmospheric pressure plasma jet

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## ABSTRACT

The He/O<sub>2</sub> atmospheric pressure plasma treatment can effectively improve the wettability and sizing properties of raw cotton yarns. However, the treatment effects may be greatly influenced by the moisture regain (MR) of the cotton yarn. In this study, raw cotton fibers, yarns and rovings with three different MRs (0.5%, 9.3% and 26.4%) are treated by atmospheric pressure plasma jet (APPJ) in He/O<sub>2</sub> at a ratio of 30:0.3 L/min to study how MRs affect their wettability and sizing properties. SEM and XPS analyses show that cotton fibers with lower MRs are more efficiently etched and more polar bonds, such as C–OH/C–O–C, O–C–O/C=O and O–C=O, are imparted by the APPJ treatment. Consequently, the cotton yarn obtains higher wicking heights and smaller contact angles at lower MRs. The size adhesion analysis proves that the adhesion between the roving and the size becomes weaker as the MR increases. Observation of the cross sections shows that the roving with the lowest MR has a more uniform layer of size permeation than that for higher MR samples, while the side facing the plasma jet has a thicker size impregnation than the other side. The MR has negative effects on the surface etching, the improvement of hydrophilicity, sizing adhesion and size permeation uniformity of raw cotton yarns by APPJ.

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## 1. Introduction

Raw cotton fiber contains not only cellulose but also non-cellulosic constituents, such as pectins, waxes, proteins, sugars and some contaminations, in the cuticle layer which are responsible for poor hydrophilicity [1–3]. Because of the water repellency, traditional sizing techniques, such as high-pressure sizing and prewet sizing have been used to improve the size penetration of cotton yarns [4–11]. However, these conventional techniques are rather energy and water consuming and therefore may potentially be harmful to the environment. Plasma treatments, especially atmospheric pressure plasmas, as clean, dry and environmentally friendly physical technologies have been used to modify surface properties of polymers, including many textile materials [12–20]. As early as in 1974, Lawton studied low-temperature gas plasma treatment on poly (ethylene terephthalate) (PET) to improve its adhesion to rubber in tires [21,22]. More recently, some preliminary researches in sizing technology by means of plasma techniques have been reported [23–27]. Our previous research also

demonstrated that helium and oxygen (He/O<sub>2</sub>) gas atmospheric pressure plasma treatment can substantially promote the wettability and sizing properties of raw cotton yarns [28].

However, there is a potential problem for atmospheric pressure plasma treatment on hygroscopic materials especially those used in textiles. The moisture regain (MR) of these materials will change with the relative humidity (RH) of the environment and thereby may influence the effects of the atmospheric pressure plasma treatments as reported in our previous studies [29–32]. The aim of this study is to investigate the effects of MR on the wettability and sizing properties of raw cotton yarns using an atmospheric pressure plasma jet (APPJ) with He/O<sub>2</sub> gas. The physico-chemical changes that occurred during the treatments were studied using scanning electron microscopy (SEM), X-ray photoelectron spectroscopy (XPS), dynamic wicking test and water contact angle measurement. A roving impregnation method was adopted to test the adhesion between the yarn and the size. A stereo video microscope was used to observe the distribution of size impregnation into the roving.

## 2. Experimental

### 2.1. Materials and sample preparation

The cotton fibers were provided by Shandong Sanyang Textile Co., Ltd with diameters of 15–20 μm in the form of a 60 cotton count yarn

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with a twist factor of 342 and a cotton roving of 640 tex. In order to prepare samples with different MRs, the cotton fiber, yarn and roving were classified into three groups respectively. Sample 1 was dried in an oven for 24 h at 80 °C to get a 0.5% MR (RH 5%). Sample 2 together with the control group was stored in standard textile testing conditions (20 °C and 65% RH) for 24 h to get a 9.3% MR. Sample 3 was kept in a desiccator with a constant RH of close to 100% for 24 h to obtain a 26.4% MR. In order to precisely control the MR during plasma treatment, the chamber for the plasma treatment was sealed with a plastic film and preconditioned to obtain the corresponding environmental RH. The size employed in this study was phosphate modified starch with a viscosity of 10 mPa.s provided by Shandong Xiangyu Technology Group Co., Ltd.

## 2.2. Plasma treatment

The samples with different MRs were one-side treated on an APPJ machine (Atomflo-R, Surfex Company, USA) with He/O<sub>2</sub> gas at a ratio of 30:0.3 L/min. The APPJ employed a capacitively coupled electrode design and produced a stable glow discharge at atmospheric pressure with 13.56 MHz radio frequency. More detailed information about the plasma machine was given in the literature [33]. The sample was fixed on a steel frame and moved on a conveying belt perpendicular to the nozzle. The jet-to-sample distance was 1.5 mm and the stationary treatment time was equivalent to 20 s. The detailed processing parameters are listed in Table 1.

## 2.3. SEM analysis

The surface topology of the fiber samples was observed using SEM (JSM-5600LV Model, Japan). The magnifications of the image were set at 1000× and 5000× and the specimens were coated with gold prior to the SEM analysis.

## 2.4. XPS analysis

The chemical compositions of both the control and the treated fiber surfaces were analyzed using an XPS model ESCALAB 250 (Thermo Electron VG Scientific, USA). The X-ray source was Al K $\alpha$  (1486.6 eV), operating at 150 W and 20 eV. The pressure within the XPS chamber was 10<sup>-7</sup> to 10<sup>-8</sup> Pa. Photo emitted electrons were collected at a take-off angle of 45° and the deconvolution analysis of C1s peaks was carried out using XPSPEAK software. The C1s core level peak was calibrated at 285 eV.

## 2.5. Wettability measurement

The yarn samples were subjected to both the dynamic wicking and the contact angle measurement. The wicking height was tested according to the standard capillary testing method of textiles ZBW 04019-90. The cotton yarn was carefully wound onto a steel frame to get a yarn sheet with a dimension of 16.5 cm×2.5 cm at about 80 yarns per centimeter. Potassium dichromate solution with a concentration of 0.5% was prepared and the temperature was kept at

27 °C. Sheet samples with different MRs were plasma treated and balanced in 20 °C and 65% RH for 24 h and then suspended vertically with its lower end dipped in the solution for 30 min. The capillary rise measurement was performed three times for each sample and the average value was recorded.

Water contact angles of the control and the APPJ treated cotton yarns were measured by an OCA15 EC type tester using the sessile drop method [34,35]. A 0.5  $\mu$ l drop of distilled water was put on the surface with a microliter syringe and observed through an optical microscope equipped with a camera and a computer with an image capturing and processing software. The mean angle value on both sides of the drop was adopted as the useful contact angle. An average of at least six measurements was taken for each sample.

## 2.6. Size adhesion test

The adhesion strength of the size to the roving is an important factor to evaluate sizing property. In this study, a roving impregnation method was used to evaluate the adhesion between the roving and the size. Roving samples with different MRs were plasma treated and then balanced in 20 °C and 65% RH for 24 h. In the roving impregnation, a homogeneous size liquid with a concentration of 1% was prepared and the temperature of the liquid was kept at 95 °C. Then, the cotton roving was carefully wound onto a steel frame with a length of 165 mm and a width of 50 mm. The frame with the roving was immersed in the size liquid for 5 min and then naturally dried at ambient temperature. Finally, the roving was cut off from the frame as a strip and then tested on an XL-1B sizing adhesion tester (Shanghai New Fiber Instrument Co., Ltd.) after stored in 20 °C and 65% RH for 24 h. The test was carried out at 100 mm gauge length and 100 mm/min crosshead speed. A total of 30 specimens were tested and the mean breaking strength of the sized roving was taken as an indication of the adhesion between the fiber and the size.

## 2.7. Analysis of cross sections of sized rovings

In order to assess the extent of impregnation of the size into the roving, a 0.1 mol/L iodine solution was used to stain the cross sections of the sized rovings which were then observed under a KH-1000 type stereo microscope.

## 3. Results and discussion

### 3.1. SEM results

The SEM results for the control and APPJ treated cotton fibers with different MRs are shown in Fig. 1. The control has a relatively smooth fiber surface besides the natural grooves and three APPJ treated samples have rougher surfaces indicating different extents of etching. It was obvious that the plasma treatment had a minor etching effect on the cotton fibers with 26.4% MR, while the most pronounced change was observed in the 0.5% MR group. For Sample 1, a large number of nearly round-shaped particles with diameters of around 0.5 to 1  $\mu$ m appeared on the fiber surface after the plasma treatment. A moderate etching effect was found on the fiber surface of Sample 2 with a higher MR of 9.3%. It can be concluded that cotton fibers with lower MRs can be more efficiently etched than those with higher MRs.

The plasma surface etching takes place by the electrons, ions, radical species and ultraviolet radiations generated by the plasma. The etching process involves three mechanisms, namely penetration of UV radiation, physical sputtering and chemical reaction [36]. The O<sup>+</sup> ion bombardment and UV radiation in the He/O<sub>2</sub> plasma played an important role in not only physical sputtering but also chemical reaction or oxidation. Our previous research found that the plasma etching can be greatly facilitated by high MR for hygroscopic

**Table 1**  
Processing parameters of APPJ treatments.

Parameter	Values
Helium flow rate (L/min)	30
Oxygen flow rate (L/min)	0.3
Jet-to-substrate distance (mm)	1.5
Treatment duration (s)	20
Output power (W)	40

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