

# Coating of Cotton Yarn with Poly(vinyl alcohol) and Poly(*N*-vinyl-2-pyrrolidone) Crosslinked via Ultraviolet Radiation

Ana Paula Serafini Immich,<sup>1</sup> Pedro Henrique Hermes de Araújo,<sup>1</sup> Luiz Henrique Catalani,<sup>2</sup> Selene M. A. Guelli Ulson de Souza,<sup>1</sup> Antônio Augusto Ulson de Souza<sup>1</sup>

<sup>1</sup>Federal University of Santa Catarina, Department of Chemical Engineering, P.O. Box 476, Florianópolis 88040-900, SC, Brazil

<sup>2</sup>Institute of Chemistry, University of São Paulo, Avenida Lineu Prestes, 748 São Paulo 05508-900, SP, Brazil

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**ABSTRACT:** The coating of cotton fiber is used in the textile industry to increase the mechanical resistance of the yarn and their resistance to vibration, friction, impact, and elongation, which are some of the forces to which the yarn is subjected during the weaving process. The main objective of this study was to investigate the use of synthetic hydrophilic polymers, poly(vinyl alcohol) (PVA), and poly(*N*-vinyl-2-pyrrolidone) (PVP) to coat 100% cotton textile fiber, with the aim of giving the fiber temporary mechanical resistance. For the fixation of the polymer on the fiber, UV-C radiation was used as the crosslinking process. The influence of the crosslinking process was determined

through tensile testing of the coated fibers. The results indicated that UV-C radiation increased the mechanical resistance of the yarn coated with PVP by up to 44% and the yarn coated with PVA by up to 67% compared with the pure cotton yarn, that is, without polymeric coating and crosslinking. This study is of great relevance, and it is important to consider that UV-C radiation dispenses with the use of chemical substances and prevents the generation of toxic waste at the end of the process. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 119: 2560–2567, 2011

**Key words:** coatings; crosslinking; radiation

## INTRODUCTION

In the textile industry, the coating or starching of cotton fiber is carried out to increase the mechanical resistance of yarn, that is, the tensile strength, and the resistance to vibration, friction, impact, and elongation, which are some of the forces to which the yarn is subjected during the weaving process. Thus, the mechanical properties of the coating must be suitable in terms of guaranteeing the mechanical stability and integrity of the yarn during its use. Therefore, the main objectives of technological advancements are to increase the productivity of the weaving process and to reduce environmental pollution.<sup>1</sup>

Several materials, including the ceramic materials AlO<sub>2</sub>, ZnO, SnO<sub>2</sub>, TiO<sub>2</sub>, SiO<sub>2</sub>, SiC, and ZrC, are used in the textile coating industry to increase the thermal

insulation of fabric. However, the application of these materials is hindered by the difficulty associated with coating the ceramic material onto the fabric.<sup>2</sup> Polyaniline is also used for textile coating because of its antimicrobial activity.<sup>3</sup> The linear copolymer of *N*-*tert*-butylacrylamide and acrylamide is used in the coating of yarn to increase the impermeability of the fabric on contact with water.<sup>4</sup> The behavior of the coating will always be dependent on the mechanical compatibility between the fiber and the coating material.<sup>5</sup>

The reinforcement of fibers with polymeric compounds consists of coating the fiber by impregnating a polymer matrix. However, the coating materials used in engineering must be generally accepted by industry and, thus, must comply with environmental safety standards. Given the array of chemical products available for polymeric matrices, the use of aqueous solutions or suspensions is required to carry out the coating.<sup>6</sup>

An excellent candidate that complies with environmental standards is poly(vinyl alcohol) (PVA) because it is a synthetic polymer that is soluble in water and has a high hydrophilicity, biocompatibility, nontoxicity, and a low biochemical oxygen demand.<sup>7</sup> However, there are problems associated with its discharge in effluents. Another possible

Correspondence to: A. P. S. Immich (paulaimmich@yahoo.com.br).

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candidate is poly(*N*-vinyl-2-pyrrolidone) (PVP), a hygroscopic synthetic polymer, which is soluble in water. The commercial success of PVP is due to its nontoxicity, adhesive characteristics, film-forming capacity, facility to form complexes, relatively inert behavior in relation to salts and acids, and its resistance to thermal degradation in solutions.<sup>8</sup>

One of the advantages of the use of PVA and PVP is that crosslinking processes can be used to improve the polymer chain stability and to affect the polymer adherence to the fiber, torsion factor, fiber quality, and performance and operating conditions of the machines used to process the coated fiber.

One process available for the crosslinking of PVA and PVP in fibers is UV radiation with low-pressure mercury lamps. The study of UV application is of great relevance because UV dispenses with the use of chemical substances and prevents the generation of toxic wastes at the end of the process; this is of considerable interest to the industry because of increased environmental awareness and the strictness of environmental regulations.

The main objective of this study was to investigate the application of PVA and PVP polymers for the coating of cotton yarn and its subsequent crosslinking, with the aim of conferring temporary mechanical resistance to the yarn. This characteristic is required for the yarn to pass through the weaving process and, later, to have the temporary polymeric coating removed to ensure greater hydrophilicity, water absorption, touch, and softness.

## EXPERIMENTAL

### Materials

The main materials used in this study were combed 100% cotton yarns with 518 twists/m, PVPs with molecular weights (MWs) of 40,000 and 1,000,000 g/mol, and PVAs with MWs of 25,000 (98% hydrolyzed), 75,000 (99.7% hydrolyzed), and 133,000 g/mol (99% hydrolyzed) (Polysciences, Inc., Warrington, PA).

### Cotton fiber coating

Cotton fibers were coated with PVA and PVP with different MWs in aqueous solutions with concentrations of 2, 5, and 10 wt % of the solution, with the aid of a Foulard coating machine (Mathis, model Foulard FVH, Zürich, Switzerland) with a pressure of 1–2 bar between the rollers.

After the coating process, the samples were dried at different temperatures (25, 90, and 150°C) in an oven with air recirculation.

### Crosslinking of the fibers coated with PVA and PVP by UV-C radiation

The fibers coated with PVA and PVP, as described in the previous section, were crosslinked via 1 h of exposure to UV-C radiation in a LUZ CHEM ICH2 chamber (Cambridge, UK), with Sankyo Denki 68T5 UV-C lamps (São Paulo, Brazil) at a wave length of 254 nm.

### Tensile testing of the textile fiber

To verify the tensile strength of the fiber, tensile tests were carried out in an Emic DL-2000 machine (São José dos Pinhais, Brazil) with a cell load of 50 N. The standard methods used for the test were the international methods ASTM D 1445-95 "Standard Test Method for Breaking Strength and Elongation of Cotton Fibers (Flat Bundle Method)" and D 1294-95a "Standard Test Method for Tensile Strength and Breaking Tenacity of Wool Fiber Bundles 1-in."

Each sample was carefully laid on a paper sheet to be correctly placed in the machine and to avoid sliding. The paper sheet was then cut to prevent any influence in the tensile test. For each condition, the experiment was repeated 10 times.

### Calculation of the gel content (%)

The gel content of the polymers was measured by mass difference.

The cotton yarn was weighed at three different stages: (1) pure, before coating; (2) after coating and crosslinking; and (3) after solubilization of the polymer in heated distilled water at 90°C under magnetic agitation for 30 min.

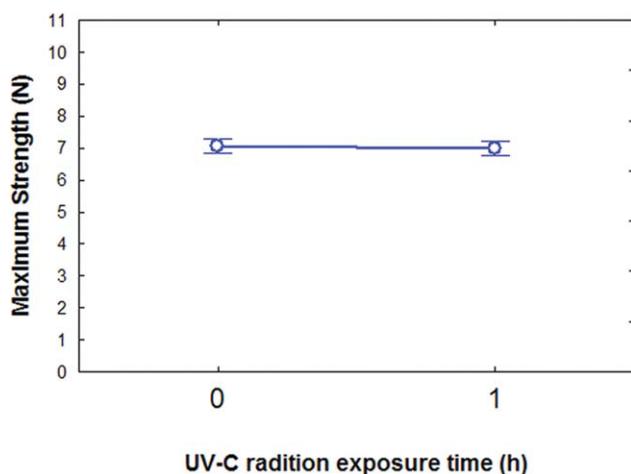
With eq. (1), the gel content, that is, the crosslinked part, of the polymers was determined.<sup>9</sup> The noncrosslinked part, that is, the sol fraction, was removed during the solubilization, which left only the crosslinked gel content in the yarn:

$$\text{Gel content} = \frac{m_3 - m_1}{m_2 - m_1} \times 100 \quad (1)$$

where  $m_1$  is the cotton yarn mass before the coating process (g),  $m_2$  is the cotton yarn mass after the coating and crosslinking processes (g), and  $m_3$  is the cotton yarn mass after coating solubilization (g).

### Scanning electron microscopy (SEM)

Analysis of the images of the yarn was carried out with SEM (Philips, type XL30, Eindhoven, Holand). The yarns were analyzed and identified as follows: 100% pure cotton yarn and 100% cotton coated yarn. The images were magnified up to 1000 times.



**Figure 1** Tensile test results for the pure cotton yarn exposed to different radiation times. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

### Toxicity test

Acute toxicity tests with *Daphnia magna* as the test organism were carried out according to regulation NBR 12.713. The exposure time of the test organism to the effluent was 48 h. The results from these tests are expressed as the dilution factor (DF). DF represents the first of a sequence of dilutions of one sample in which the acute toxic effect on the test organism is no longer observed.<sup>10</sup>

The sample analyzed was the polymeric coating layer removed from the yarn after a controlled washing in a Mathis Jigger instrument (model WJ, Zürich, Switzerland) with distilled water at 80°C. The following coating layers were removed for analysis:

1. Coating with PVA with an MW of 133,000 g/mol, with a 10% concentration, and crosslinked via UV-C radiation.
2. Coating with PVP with an MW of 1,000,000 g/mol, with a 10% concentration, and crosslinked via UV-C radiation.

According to Immich et al.<sup>10</sup> the maximum limit of DF for chemicals and textile effluents is 2.

## RESULTS AND DISCUSSION

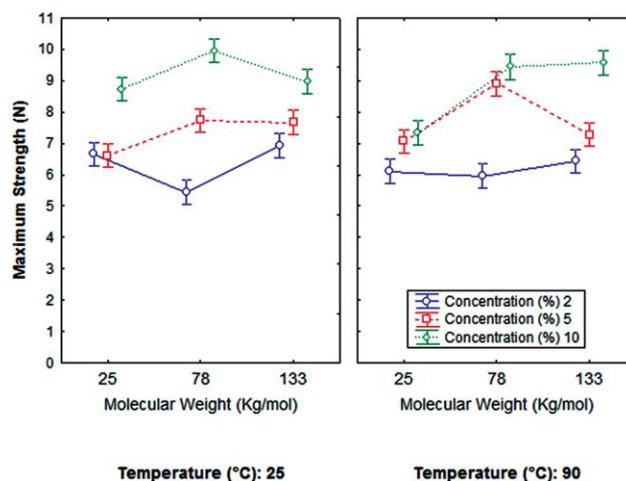
### Crosslinking of PVA and PVP via UV radiation

Initially, before the tensile test results for the cotton yarn coated with PVA and PVP and crosslinked were analyzed, it was necessary to ascertain the resistance of the pure cotton yarn, that is, without the coating and crosslinking process. Figure 1 shows the tensile test results for the pure cotton yarn.

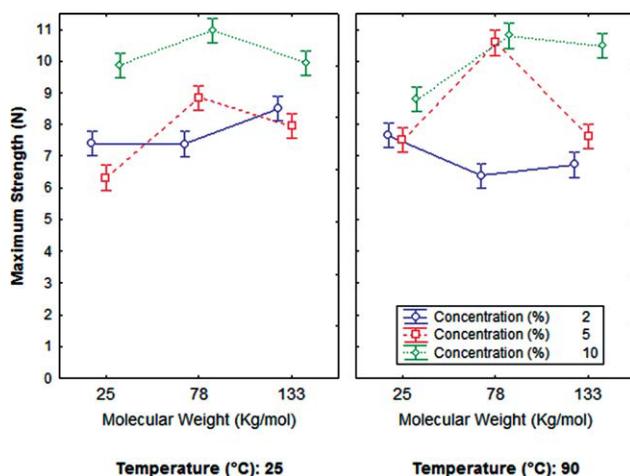
Figure 1 shows that the resistance of the pure cotton yarn, that is, without the polymeric coating, exposed to radiation decreased by approximately 1.16% compared to the yarn not exposed to radiation. Statistically, this change in resistance was insignificant. This behavior was expected because cotton fibers are composed mostly of  $\alpha$ -cellulose (88–96.5% w/w), and the cellulose had no group that absorbed UV radiation (254 nm).<sup>9</sup> Therefore, this insignificant photodegradation could have been induced by chromophore impurities, such as carbonyl, present in the fiber.<sup>11</sup> This change in resistance could also have been attributed to the lack of uniformity of the weaving process or to the simple experimental manipulation.

Before verifying the influence of the crosslinking process, we tested the fiber with only the polymeric coating of PVA, and the results of the tensile test are presented in Figure 2. An improvement in the yarn resistance with the polymer coating before crosslinking is shown in Figure 2. The yarn coating with PVA led to an increase in the resistance from 7.05 N (pure cotton yarn) to 9.95 N (yarn coated with PVA with an MW of 78,000 g/mol, with a solution concentration of 10%, and dried at room temperature), that is, an increase of approximately 41%.

The results of the tensile test obtained for the fiber coated with PVA and crosslinked by UV-C radiation are given in Figure 3. As shown in Figure 3, on exposure of the yarn (with the polymer coating with an MW of 78,000 g/mol, with a solution concentration of 10%, and dried at room temperature) to UV-C radiation, there was an increase in the cotton yarn resistance from 9.95 to 10.98 N, which represented improvements of approximately 10% in relation to the coated yarn without crosslinking and 56% in relation to the pure yarn.



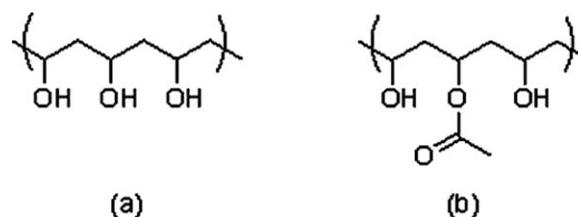
**Figure 2** Tensile test results for the cotton yarn coated with PVA. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 3** Tensile test results for the cotton yarn coated with PVA crosslinked via UV-C radiation. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

These results indicate that PVA responded to the UV-C radiation stimuli; this led to the crosslinking of the polymer and the consequent increase in the mechanical resistance of the yarn. According to Kaczmarek and Podgórski<sup>12</sup> and Paralikar,<sup>13</sup> the pure PVA (100% hydrolyzed) did not absorb the UV-C radiation at the wavelength of 254 nm. However, the exposure of the polymer to UV-C radiation led to an initial degradation of the polymer chains. This photodegradation of PVA could be considered a process initiated by the impurities, chromophore groups present in the polymer, such as carbonyl groups (responsible for the absorption in the near-UV region at wavelengths of 380–200 nm).

The presence of carboxylate groups (CO—O) in the PVA originated from the polymer production. PVA was obtained by the hydrolysis of poly(vinyl acetate); thus, it may have been contaminated with residual acetate groups derived from an incomplete hydrolysis process. Although the contamination of carboxylate groups in PVA was low (maximum = 2%), the effects of the photochemical process on the

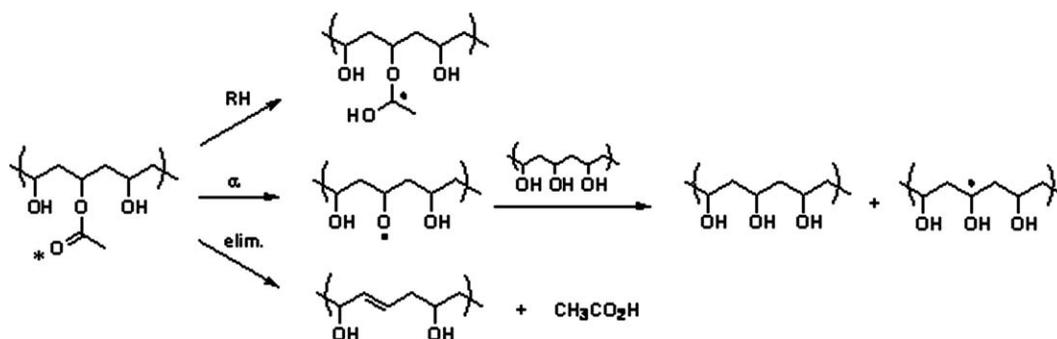


**Figure 4** Chemical structure of PVA: (a) 100% hydrolyzed and (b) containing residual groups.

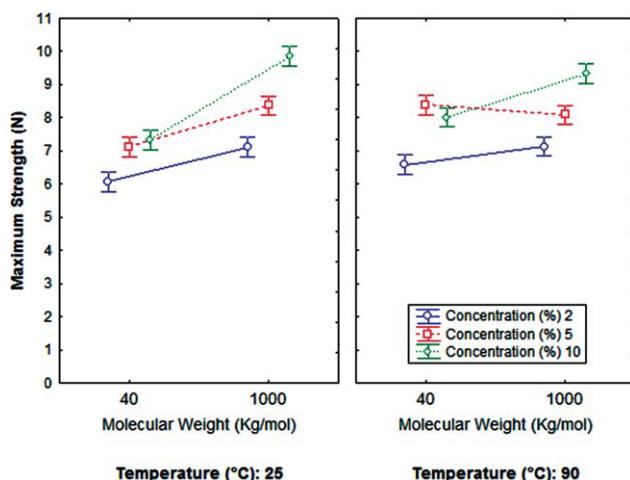
polymer may be decisive because the carboxylate groups absorb the UV radiation and can, thus, generate free radicals.<sup>14</sup> Therefore, the carboxylate groups initiate the degradation or sensitize the process in the PVA. These free radicals formed may enhance chain degradation or may recombine; this leads to the crosslinking process. Subramanian<sup>15</sup> studied polymer photodecomposition via UV radiation and verified that, in the presence of initiators, the decomposition of poly(styrene peroxide) is proportional to the absorption of UV radiation. The same thing could be happening with PVA using the carboxylate groups as initiators.

Figure 4 shows the chemical structure of the PVAs that were 100% hydrolyzed [Fig. 4(a)] and containing residual acetate groups [Figs. 4(b)]. The photolysis of PVA containing residual acetate groups is illustrated in Figure 5.

The crosslinking reactions that occurred in PVA must also have been associated with the drying temperature used before irradiation with UV light. Studies carried out by Figueiredo et al.<sup>16</sup> showed that when the PVA was heated, the energy employed was used to modify the special organization of the chains and to establish hydrogen bonds between the hydroxyl groups; this led to an increase in the polymer crystallinity. However, the heating intensity and time led to chain degradation. Holland and Hay<sup>17</sup> showed that the thermodegradation of PVA led to an initial increase in the MW of the polymer followed by a reduction. These observations may be due to the competitive effects of the crosslinking



**Figure 5** Photolysis of PVA. (RH represents the hydrogen transference by a free radical and  $\alpha$  represents the chain scission caused by the free radical generated by the UV radiation. "Elim." represents the elimination of the ester molecule.)

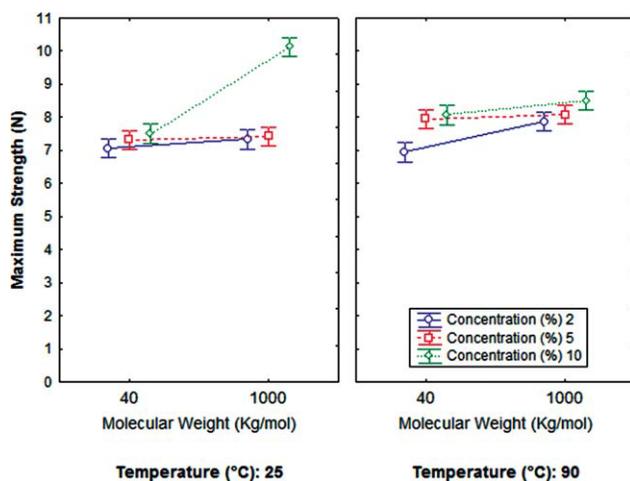


**Figure 6** Tensile test results for the cotton yarn coated with PVP. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

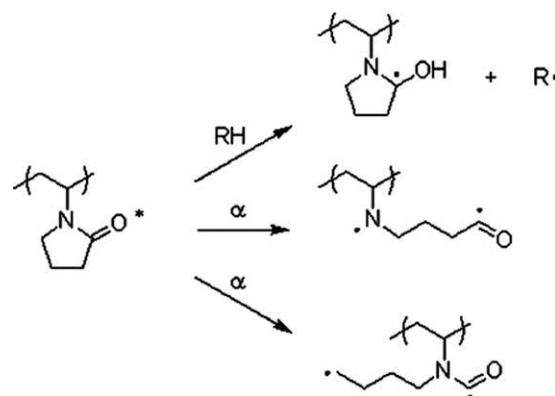
and degradation or chain scission. Thus, the investigation of the polymer drying time and radiation exposure time are fundamental for optimizing the crosslinking process.

To evaluate the effect of the type of hydrophilic synthetic polymer on the mechanical resistance of the coated cotton fiber, the same experiments applied to PVA were performed with PVP. The results obtained from the tensile testing of the fiber coated with PVP are shown in Figure 6.

Figure 6 shows an improvement in the mechanical resistance with the polymeric coating before crosslinking, which increased the resistance from 7.05 N (pure cotton yarn) to 9.85 N (the best result was obtained with the fiber coated with PVP with an MW of 1,000,000 g/mol, with a solution concentration of 10%, and dried at room temperature), that is,



**Figure 7** Tensile test results for the yarn coated with PVP and crosslinked via UV-C radiation. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 8** Photolysis of PVP.<sup>18</sup> (RH represents the hydrogen transference by a free radical and  $\alpha$  represents the chain scission caused by the free radical generated by the UV radiation.)

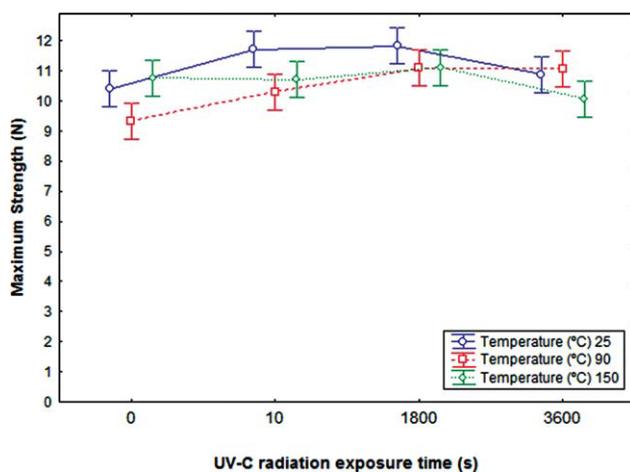
an increase of up to 40%. This coating layer forms a cylindrical polymeric capsule along the yarn and confers superficial protection to the fiber. The coating layer is shown later in Figures 14 and 15.

The results obtained from the tensile testing of the fiber coated with PVP and crosslinked by UV-C radiation are given in Figure 7. According to Figure 7, the exposure of cotton fiber coated with PVP to UV-C radiation increased its resistance from 9.85 N (coated yarn) to 10.23 N (the best result was obtained with fiber coated with PVP with an MW of 1,000,000 g/mol, with a solution concentration of 10%, and dried at room temperature). This represented increases in the resistance of approximately 44% compared to the pure cotton yarn and 3% compared to the coated yarn.

The irradiation of PVP with UV-C light caused a very slight improvement in the results (only 3%). It was possible that the long irradiation period (1 h) involved in the UV-C crosslinking was responsible for this insignificant change. According to Fehine et al.,<sup>18</sup> these PVP crosslinking reactions result from the recombination of macroradicals produced by the direct photolysis of PVP with UV radiation. However, the products of this photolysis may affect the final results, either by hindering the crosslinking or by accelerating the polymer chain breaking process. The photolysis of PVP is illustrated in Figure 8.

Kaczmarek et al.<sup>19</sup> also verified that the exposure of polymers to UV radiation can generate the simultaneous and competitive reactions of crosslinking and degradation. Thus, the determination of the radiation exposure time is fundamental for achieving benefits in relation to the reaction of interest.

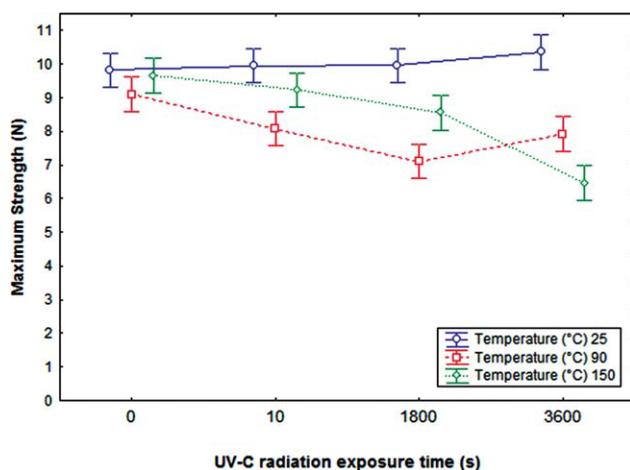
Thus, new polymer crosslinking tests were carried out with the best operating conditions, that is, with PVA with an MW of 78,000 g/mol and PVP with an MW of 1,000,000 g/mol, with a polymer solution



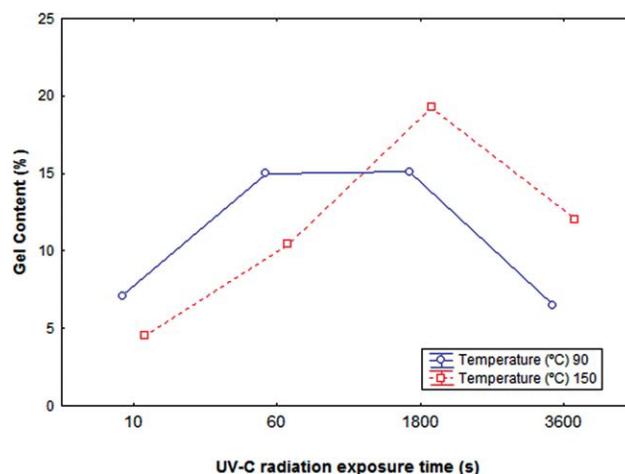
**Figure 9** Tensile test results for the yarn coated with PVA, dried at 25, 90, and 150°C and exposed to different UV-C radiation times. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

concentration of 10%, and with the following variations: polymer drying temperatures of 25, 90, and 150°C and UV-C radiation exposure times of 0 s, 10 s, 30 min, and 1 h. The results obtained for the yarn coated with PVA are given in Figure 9.

Figure 9 shows that the increase in drying temperature did not lead to an improved mechanical resistance of the yarn. As previously explained herein, the heating of the PVA may have led to the thermodegradation of the polymer with the decrease in MW. This means that a breaking of the polymer chains may have occurred, and therefore, the structure of the coating layer may have been adversely affected. The increase in the UV-C radiation exposure time had a positive effect up to 30 min and increased the yarn resistance to 11.83 N, that is, increases of approxi-



**Figure 10** Tensile test results for the yarn coated with PVP, dried at 25, 90, and 150°C and with different UV-C radiation exposure times. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]



**Figure 11** PVA gel fraction, dried at 90 and 150°C and with different UV-C radiation exposure times. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://wileyonlinelibrary.com).]

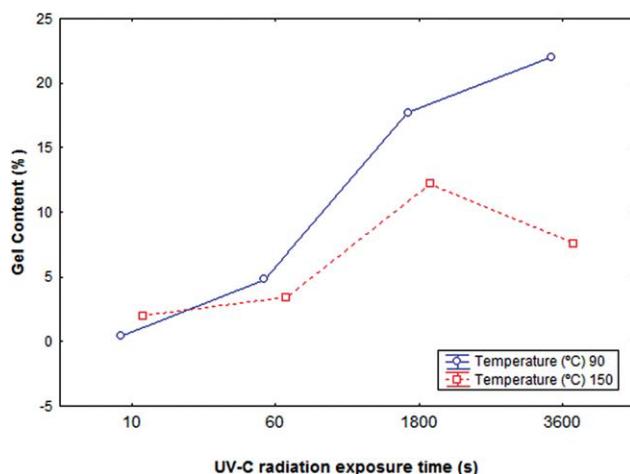
mately 67% compared to the pure cotton yarn and 19% compared to the yarn coated and without crosslinking. After this exposure time, the fiber tended to weaken, which reduced the resistance of the yarn.

Figure 10 shows the results for the yarn coated with PVP under the new operating conditions. According to Figure 10, we verified that the increase in temperature significantly decreased the mechanical resistance of the yarn and an increase in UV-C radiation exposure time tended to weaken it. It was possible that during the drying of the polymer solution on the fiber at a very high temperature (150°C), the crosslinking begins, which was finished with UV-C radiation. Thus, an excess of UV-C radiation would have led to a weaker fiber because it could initiate the degradation of the fiber or the polymer. Figure 10 does not show a single tendency in the behavior of the yarn resistance with the variation in the exposure time for the different drying temperatures. This lack of a tendency may be explained by the simultaneous and competing reactions of crosslinking and the breaking of the polymer chain.

### Gel fraction

The gel fraction of the PVA exposed to UV-C radiation in the cotton fiber is given in Figure 11. The gel fraction was calculated by the mass difference before and after the solubilization of the polymer in the yarn.

As shown in Figure 11, the behavior of the degree of PVA crosslinking in relation to the UV-C radiation exposure time followed the same tendency for both yarn drying temperatures of 90 and 150°C. As also shown in Figure 11, the exposure of the yarn to UV-C radiation for up to 30 min increased



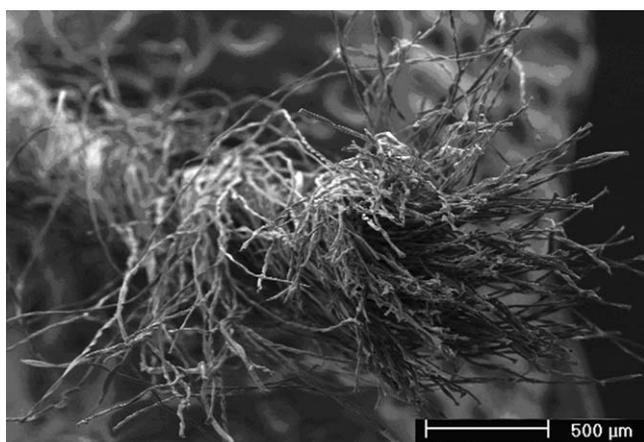
**Figure 12** Gel fraction of PVP, dried at 90 and 150°C with different UV-C radiation exposure times. [Color figure can be viewed in the online issue, which is available at [wileyonlinelibrary.com](http://www.interscience.wiley.com).]

the degree of crosslinking. However, after 30 min, the UV-C radiation had a negative effect on the polymer crosslinking, degrading it and facilitating its removal.

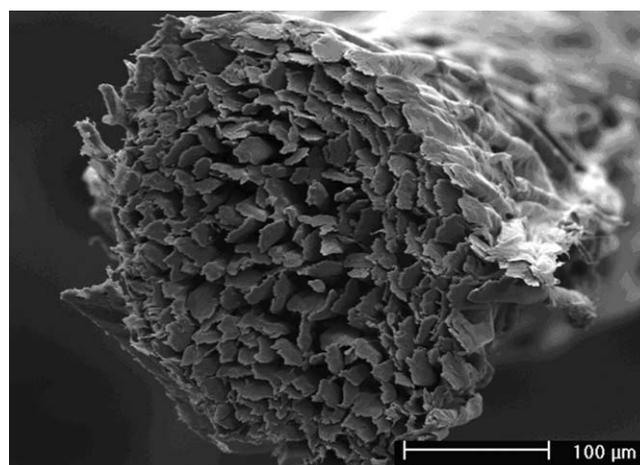
The gel fraction calculated for PVP and shown in Figure 12 indicated that for the yarn coated with PVP and dried at 90°C, the longer the UV-C radiation exposure time was the greater the gel fraction of the polymer formed in the fiber was.

For the yarn coated with PVP and dried at 150°C, an increase in the radiation exposure time up to 30 min increased the gel fraction of the polymer in the fiber. However, UV-C radiation exposure times longer than 30 min led to polymer chain scission.

We, therefore, concluded that the best operating conditions for increasing the tensile strength of the 100% cotton textile yarn were PVA with an MW of 78,000 g/mol, with a polymer bath concentration of



**Figure 13** SEM of the pure cotton yarn (30× magnification).



**Figure 14** SEM of the yarn coated with PVA (175× magnification).

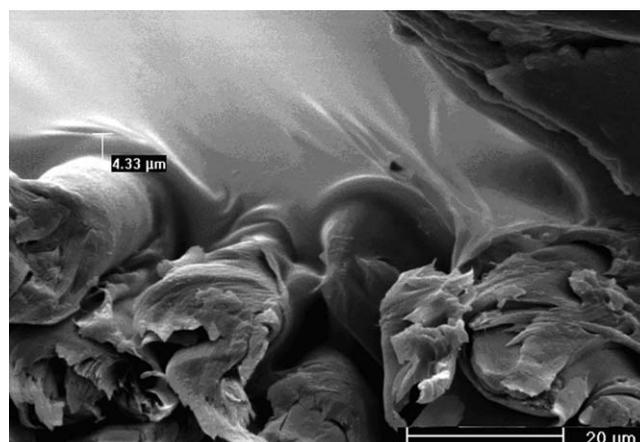
10%, dried at room temperature, and crosslinked via UV-C radiation with a bulb of wavelength 254 nm and a UV-C radiation exposure time of 30 min.

### SEM

The yarns analyzed by SEM were identified as follows: 100% pure cotton yarn (without a polymeric coating layer) and 100% cotton yarn with a polymeric coating layer.

The image in Figure 13 is a 30× magnification of the 100% pure cotton yarn, and it shows the distances between or dispersion of fibers in the absence of a coating layer. As shown, the fibers were free and isolated, which reduced the tensile strength of the yarn.

Pure natural fibers without any external coating are more susceptible to the attack and growth of microorganisms because of the large surface area and the capacity to retain moisture. To avoid this contamination, antimicrobial agents can be applied to the textile fibers through various methods, including the coating



**Figure 15** SEM of the yarn coated with PVA (1000× magnification).

of the fiber surface and the encapsulation of chemical agents within the fiber in a polymeric matrix.<sup>20,21</sup>

Figure 14 shows the organization of the fibers after the coating with PVA. In Figure 14, the compaction and joining of the fibers can be observed after the coating of the yarn with PVA, and the diameter appears to be more uniform. The yarn coated with PVA showed a reduction in diameter from approximately 603 to 343  $\mu\text{m}$ .

Figure 15 shows the 1000 $\times$  magnification of the yarn coated with PVA. At this magnification, it is possible to verify the polymer layer coating the cotton yarn. Figure 15 shows a polymer coating layer with a thickness of 4.33  $\mu\text{m}$ . Despite the polymer solution being impregnated into the yarn by Foulard compression rollers, the image analysis shows that the yarn coating was only superficial, without transference of the polymer solution to the fibers within the yarn.

#### Toxicity tests with *D. magna*

The results obtained in the toxicity tests showed that PVA and PVP had DF values that were less than or equal to the maximum allowed for industrial textile effluent, that is, 2. Therefore, the crosslinked and solubilized polymers were considered nontoxic toward the aqueous environment and could be discharged without the need for treatment.

### CONCLUSIONS

In this study, we examined the crosslinking process via UV-C radiation used on 100% cotton textile fibers coated with polymeric compounds and aimed to increase the mechanical resistance to traction.

The tensile tests carried out on the yarn indicated that the crosslinking via UV-C radiation with low-pressure mercury lamps at a wavelength of 254 nm increased the mechanical resistance of the yarn by

67% compared with the pure yarn, that is, without polymer coating and without crosslinking. The polymer that gave the best results in the tensile tests after the crosslinking process was PVA. The time of yarn exposure to UV-C radiation was shown to be fundamental for the final optimization of the process.

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