

Effect of atmospheric plasma process on the water-repellent finishing performance and permanence

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ABSTRACT – REZUMAT

Effect of atmospheric plasma process on the water-repellent finishing performance and permanence

This study aims to investigate the effect of atmospheric plasma treatment on the performance and permanence of water repellency finish. Air and nitrogen plasma treatments were applied to pre-treated polyester-based fabrics prepared for dyeing. Fluorocarbon solutions, with and without cross-linkers, were applied to the fabrics using the pad-dry-cure method. Contact angle measurements, FT-IR analysis, SEM analysis, and whiteness measurements were conducted. The samples underwent repeated washing 10 times, and measurements were retaken. The contact angle values of fabrics with plasma pre-treatment and water-repellent properties, using only fluorocarbon, were better than those of fabrics with water-repellent properties using cross-linker and fluorocarbon. The contact angle values obtained 20 seconds after the drop was placed on the fabric were very close for both samples. Similar permanence properties were obtained after repeated washings when atmospheric plasma pre-treatment was used instead of the cross-linking chemical used to ensure the permanence of the water-repellent finishing.

Keywords: atmospheric pressure plasma, water-repellent, finishing, contact angle, permanence

Influența procesului cu plasmă atmosferică asupra performanței și durabilității finisajului hidrofug

Scopul acestui studiu este de a investiga influența tratamentului cu plasmă atmosferică asupra performanței și durabilității finisajului hidrofug. Tratamentele cu plasmă atmosferică și cu azot au fost aplicate materialelor textile pe bază de poliester pretratate pregătite pentru vopsire. Soluțiile de fluorocarburi, cu și fără agenți de reticulare, au fost aplicate pe materialele textile folosind metoda de fulardare-uscare-condensare. Au fost efectuate măsurători ale unghiului de contact, analize FT-IR, analize SEM și măsurători ale gradului de alb. Probele au fost supuse spălării repetate de 10 ori, iar măsurătorile au fost efectuate din nou. Valorile unghiului de contact ale materialelor textile cu pretratament cu plasmă și proprietăți hidrofobe, folosind doar fluorocarbura, s-au dovedit a fi mai bune decât cele ale materialelor textile cu proprietăți hidrofobe folosind agent de reticulare și fluorocarbura. Valorile unghiului de contact obținute la 20 de secunde după ce picătura a fost plasată pe materialul textil au fost foarte apropiate pentru ambele probe. Proprietăți similare de durabilitate au fost obținute după spălări repetate atunci când s-a utilizat un pretratament cu plasmă atmosferică în locul agentului de reticulare chimic utilizat pentru a asigura durabilitatea finisajului hidrofug.

Cuvinte-cheie: plasmă la presiune atmosferică, hidrofug, finisare, unghi de contact, durabilitate

INTRODUCTION

Textiles are subjected to chemical (wet) processes in order to provide the desired functional properties [1]. The plasma process stands out as one of the most promising technologies, offering an alternative to various wet processes in textiles while reducing energy, water, and chemical usage [2].

Expectations for fabrics are increasing day by day, and fabrics are endowed with different usage properties according to these expectations. Water repellency is one such expectation. The chemicals utilized in the water-repellency process form a film layer on the product surface capable of retaining water for a certain period, thus enabling water repellency [3]. Fluorocarbons are extensively employed in water-repellent finishing processes owing to their oil and dirt-repellent properties [4]. There are various studies in the literature regarding the addition of water repellency to fabrics. Balcı et al. compared the foam

application method, which imparts water repellency to denim fabric using different chemicals, with impregnation and coating methods [5]. Akıncı et al. investigated the effects of weft density and filament fineness on the degree of water repellency in water-repellent fabrics. They observed that the filament fineness and density values did not consistently result in an increase or decrease in the water contact angles of the fabrics [6].

In order to impart water-repellent properties to fabrics, various methods are employed including the sol-gel method [7–11], nanocoating [12], chemical modification [13–16], etc. In recent years, research has been focused on surface activation of textiles using plasma and plasma graft polymerization [1, 17].

Contact angle measurement, Bundesman sprinkler test and spray test methods are used to determine the water repellency of fabrics. Contact angle measurements are the primary method used to determine

the wettability of various materials, including polymer materials. The main criterion for defining a surface as hydrophobic or hydrophilic is the size of the contact angle formed by a water droplet on the surface. In other words, the tendency of the liquid to spread on the solid surface indicates the water-repellent property of the solid. If the angle measures less than 90°, it indicates a hydrophilic surface; if it exceeds 90°, it suggests a hydrophobic surface, and if it surpasses 150°, it characterizes a superhydrophobic surface. When the surface is hydrophilic, the water droplet tends to spread across the solid surface, whereas, in the case of a hydrophobic surface, the water droplet is observed to remain stationary on the surface in a spherical shape [18].

In their study, Rajar et al. achieved water repellency on polyamide 6 (PA 6) fabrics by employing sol-gel finishing after oxygen plasma treatment. The findings revealed that both plasma treatment and finishing application influenced the morphological and chemical properties of the fibres. The researchers noted that the plasma treatment enhanced the adhesion of the water-repellent finish to the fabric, leading to a significant increase in washing durability on PA 6 fabric [8]. Dasdemir and Ibili 2017 developed superhydrophobic nonwoven surfaces via electro-spraying. After the successful application of fluorochemical solutions to nonwoven fabrics by filling and electro-spraying, they made contact angle measurements and obtained superhydrophobic surfaces [19]. Park et al. conducted a study where they generated a super-repellent fabric employing the plasma-enhanced chemical vapour deposition (PECVD) method, incorporating oxygen plasma etching and hexamethyldisiloxane (HMDSO). They successfully attained contact angles exceeding 160 degrees [20]. In their research, Furlan et al. administered a water repellent and flame-retardant finishing solution, formulated with fluoroalkyl-functional siloxane and organophosphonate, onto polyester samples after oxygen plasma treatment. They observed that oxygen plasma notably enhanced the wettability of polyester fibres, leading to increased absorption of the finishing solution. As a result, the treated samples exhibited superhydrophobic and flame-retardant properties [21]. Liu et al. utilized a solution composed of perfluoroalkyl acrylate, epoxide-containing silane, and silica nanoparticles to achieve amphiphobic fabric, followed by argon-plasma treatment. They introduced the argon-plasma-enriched coating method as a convenient approach for producing long-lasting superphobic fabrics [22]. In their study, Kale and Palaskar employed a blend of argon and hexamethyldisiloxane to impart water-repellent properties to 100% cotton fabrics using the plasma polymerization technique. They reported that the treated fabrics exhibited notable resistance to wetting with water [17]. Zanini et al. investigated the hydrophobic and oleophobic modification of pure cashmere and wool/nylon fabrics through plasma treatment at atmospheric pressure followed by impregnation with fluorocarbon resin. They determined that the fabric

surface treated with plasma before the finishing process exhibited a greater amount of fluorocarbon resin, resulting in a more uniform coating of the textile fibres [23]. In their study, Leroux et al. achieved hydrophobic fabric by directly injecting a fluoropolymer into the plasma dielectric barrier discharge. They concluded that air plasma treatment significantly improved the adhesion of the fluoropolymer to the polyester fabric [24].

Based on this information, the current study aimed to examine the impact of atmospheric pressure plasma treatment on the water-repellency performance and durability of fabrics. For this purpose, atmospheric plasma pretreatment was applied to polyester-based fabrics. The water-repellent finishing solution was applied to the fabrics using the pad-dry-cure pad-dry-cure method. Water repellent properties of the fabrics were evaluated by contact angle measurements, FT-IR and SEM analysis. Following 10 repeated wash cycles, contact angle measurements, FT-IR, and SEM analysis were conducted again to assess the permanence of the finish.

MATERIALS AND METHODS

Materials

The fabric utilized in this study is 100% polyester-based with a weight of 160 g/m². It has a weft density of 25 threads/cm and a warp density of 56 threads/cm. The weft linear density is 300 denier, while the warp linear density is 100 denier.

Methods

Atmospheric pressure plasma pretreatment was first applied to polyester-based fabrics using air and nitrogen gases. The water-repellent finishing process involved the application of liquors containing fluorocarbon + crosslinker, as well as liquors containing only fluorocarbon. After finishing the process, contact angle measurements, FT-IR analysis, SEM analysis and Berger whiteness measurements of the samples were performed. To investigate the permanence of the finish, the fabrics were subjected to a repeated home washing process 10 times and contact angle measurements, FT-IR analysis, SEM analysis and Berger whiteness measurements were made again (figure 1).

Atmospheric pressure plasma

Atmospheric pressure plasma pretreatment was applied to polyester-based fabrics, using two types of gases: air and nitrogen. The parameters of the atmospheric pressure plasma process are outlined in table 1.

Water repellent finishing

Water-repellent finishing was applied to polyester-based fabrics using a laboratory-type vertical padding machine, following the pad-dry-cure method. The uptake ratio was set at 50%. The water-repellent finishing recipe used for the samples is detailed in the provided table 2. The pH of the solution prepared as per the formulation was adjusted to 5. Following impregnation, the fabrics were dried at 120°C for

3 minutes. Subsequently, they were cured at 160°C for 2 minutes.

Contact angle measurements were performed before and after repeated washings according to the dynamic drop sessile method using an optical goniometer (Attension, Theta Lite 100, Sweden). FT-IR analysis of the fabrics was made using Perkin Elmer UATR Two before and after repeated washing, and

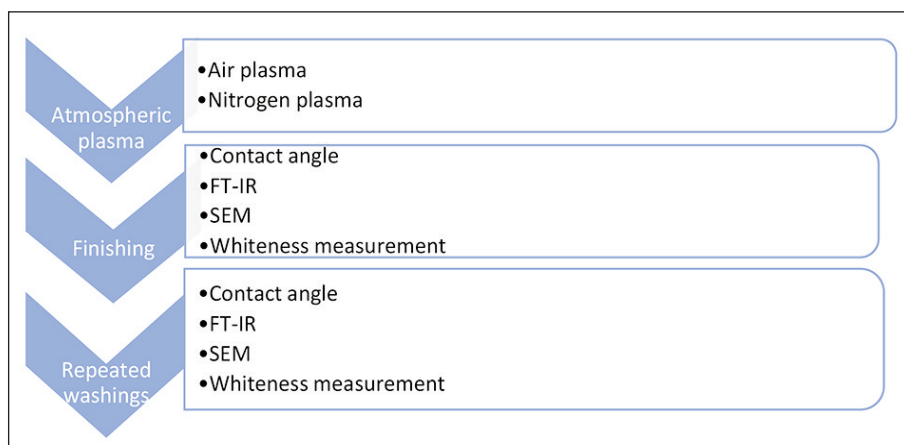


Fig. 1. Flow Chart

Table 1

| PARAMETERS OF THE ATMOSPHERIC PRESSURE PLASMA | | | | | | | |
|---|-------------|-----------------|--------------|---------------|----------------|----------------|---------------|
| Jet | Voltage (V) | Frequency (kHz) | Power (Watt) | Distance (mm) | Pressure (bar) | Ionisation gas | Speed (m/min) |
| RD1010 | 280 | 21 | 600 | 10 | 3 | Air | 5 |
| PFW70 | 280 | 21 | 600 | 3 | 3 | Nitrogen | 5 |

Table 2

| WATER REPELLENCY TEST PLAN | | | | | |
|----------------------------|-------------|--------------------------------|------------|----------------------------------|-----------------------------------|
| Sample number | Sample code | The raw material of the fabric | Plasma gas | Fluorocarbon (Mithril SFC) (g/l) | Cross-linking (Mithril EXT) (g/l) |
| 1 | PES-R-F+C | 100% polyester | Reference | 50 | 10 |
| 2 | PES-A-F+C | 100% polyester | Air | 50 | 10 |
| 3 | PES-N-F+C | 100% polyester | Nitrogen | 50 | 10 |
| 4 | PES-R-F | 100% polyester | Reference | 50 | - |
| 5 | PES-A-F | 100% polyester | Air | 50 | - |
| 6 | PES-N-F | 100% polyester | Nitrogen | 50 | - |

whiteness measurements were made using the Datacolor device.

Repeated washings

Fabric samples were subjected to a household washing procedure in a washing machine at 30°C for 30 minutes, with a total of 10 repetitions. The recommended amount of detergent, adjusted according to the weight of the fabric, was used for each wash cycle. Following the repeated washings, contact angle measurements, FT-IR analysis, SEM analysis, and whiteness measurements were conducted once more.

RESULTS AND DISCUSSION

Contact angle

Initially, contact angle measurements were conducted immediately after the droplet was applied to the finished fabric (Finished-0 sec) and then after 20 seconds. Subsequently, contact angle values were measured when the droplet was first applied to the fabric

after undergoing 10 wash cycles, and again after 20 seconds. All the results obtained are presented in the figure 2. When the test results were examined, as expected, the contact angle values of the fabrics decreased after repeated washings. When examining the contact angle values measured immediately after the droplet was first applied to the fabric (0 sec), in the case of utilizing fluorocarbon and crosslinker in the water-repellency solution, it was observed that the contact angle of the sample treated with nitrogen plasma was very close to that of the reference sample. However, the contact angle value of the sample treated with air plasma increased by approximately 4% compared to the reference sample. Upon examining the values measured at the end of 20 seconds, it was found that the value of the sample treated with nitrogen plasma was lower than that of the reference sample. Conversely, the value of the sample treated with air plasma increased by 1.7% compared to the reference sample. It can be concluded that air plasma application improves the contact angle values of

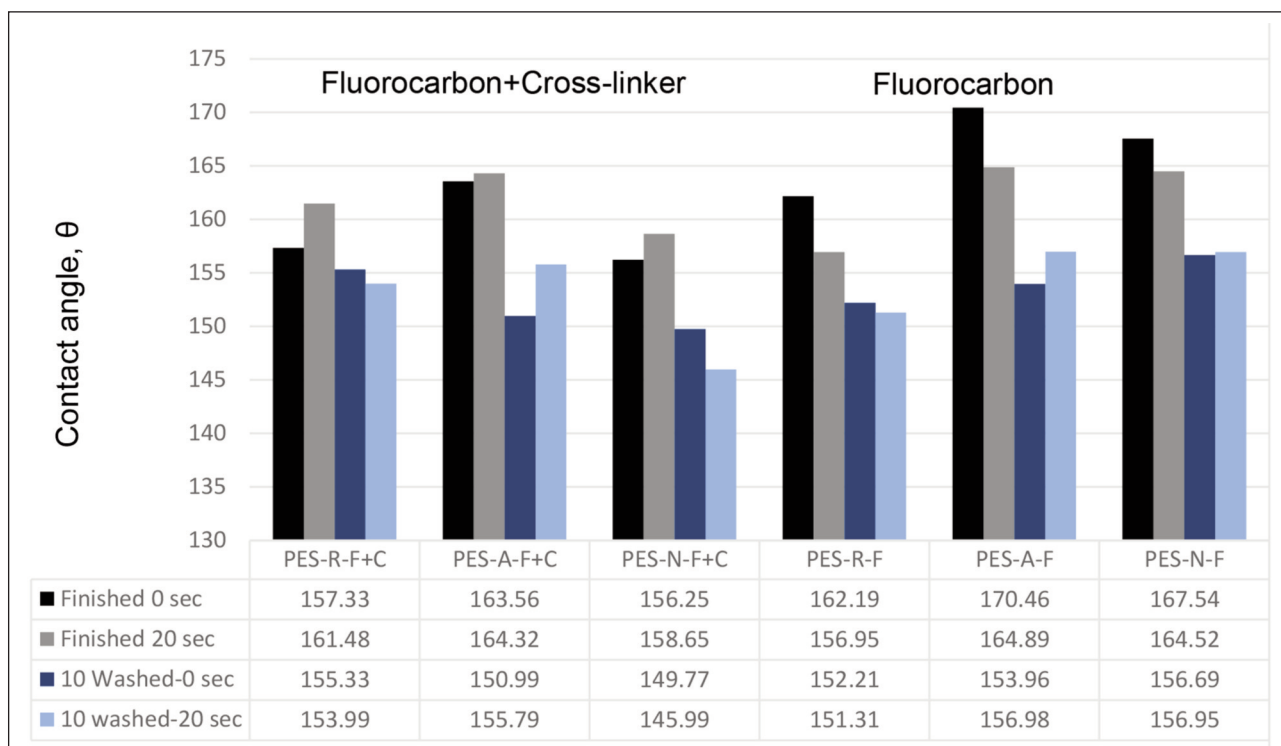


Fig. 2. Contact angles

fabrics after finishing. After repeated washings, the contact angle values of the air and nitrogen plasma applied samples were lower than the reference sample. The plasma treatment appears to have had an adverse impact on the contact angle values in the samples subjected to repeated washing (10 times) following the water-repellency finishing process.

In the finishing process using only fluorocarbon after the plasma treatment, when the drop was first dropped on the fabric, the contact angle value of the air plasma applied sample improved by 5% compared to the reference sample, and the nitrogen plasma applied sample improved by 3.2%. As a result, plasma pretreatment improved the initial contact angle values of the fabrics. 20 seconds after the drop was dropped on the fabric, the contact angle values of the samples showed similar behaviour and improved compared to the reference sample. When the contact angle values measured after 10 repeated washings were examined, the contact angle values of the plasma-treated samples were higher than the reference sample.

Contact angle values were found to be better in fabrics with plasma pre-treatment and water-repellent properties with only fluorocarbon than in fabrics with water-repellent properties using crosslinkers. The contact angle values obtained 20 seconds after the droplet was applied to the fabric were nearly identical to those of the reference sample when fluorocarbon and cross-linker were applied. Plasma treatment also provides the permanence value after repeated washings provided by the cross-linking chemical. As a result, plasma pretreatment of polyester fabrics eliminates the necessity of employing cross-linkers and yields superior water-repellency values.

The impact of plasma type and washing method on the contact angle values of fabrics was analyzed using analysis of variance, and the findings are outlined in table 3.

In the test results of between-subject effects, it can be concluded that plasma type and washing type factors have an effect on the general linear model in the case of using fluorocarbon and cross-linker in polyester fabrics, as well as in the case of using only fluorocarbon.

Table 3

| TESTS OF BETWEEN-SUBJECTS EFFECTS | | | | | | |
|-----------------------------------|-------------|-------------------------|----|-------------|--------|-------|
| Dependent variable: contact_angle | | | | | | |
| Substance | Source | Type III Sum of Squares | df | Mean Square | F | Sig. |
| F+C | Plasma type | 317.664 | 2 | 158.832 | 5.193 | 0.049 |
| | Wash type | 1390.855 | 3 | 463.618 | 15.159 | 0.003 |
| F | Plasma type | 90.797 | 2 | 45.399 | 16.961 | 0.030 |
| | Wash type | 317.522 | 3 | 105.841 | 39.541 | 0.000 |

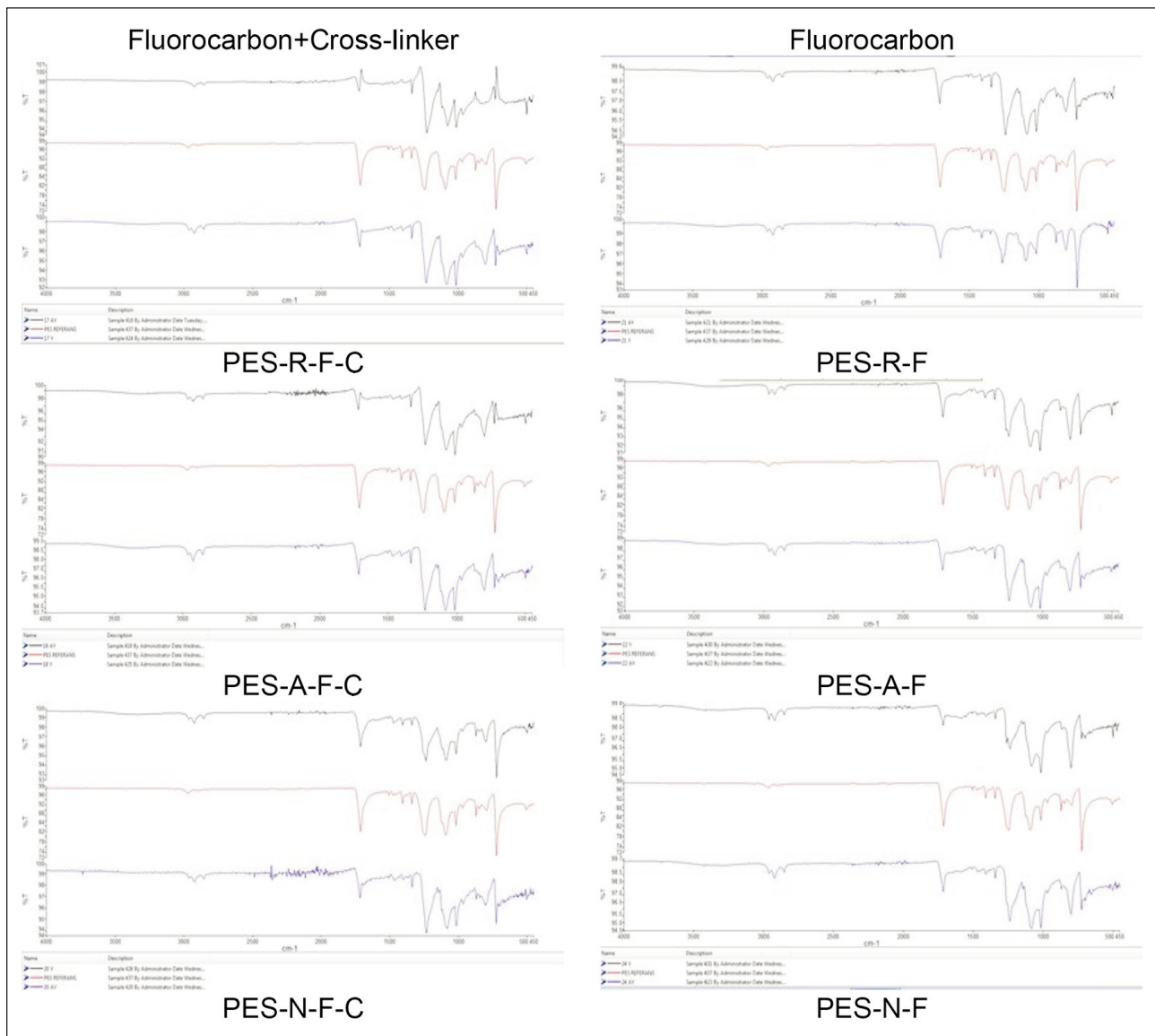


Fig. 3. FT-IR analysis

FT-IR Analysis

Measurements of the finished and washed fabrics were conducted, with the untreated fabric serving as the baseline in the FT-IR spectrum. When the infrared spectrum is examined; -NH stretching vibration was observed at 3000 cm^{-1} , the presence of C=O groups in the structure of urethane was observed in the $1700\text{--}1740\text{ cm}^{-1}$ band, and C-F stretching vibrations were observed at $1000\text{--}1400\text{ cm}^{-1}$. Additionally, it has been observed that finished fabrics and washed fabric peaks appear the same way. It has been noted that the washing process applied to the fabric does not effectively remove the finishing chemicals (figure 3).

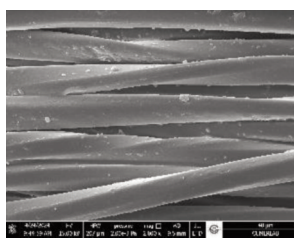
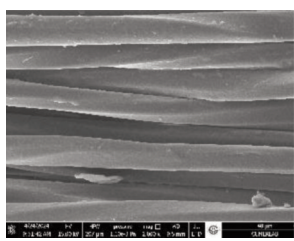
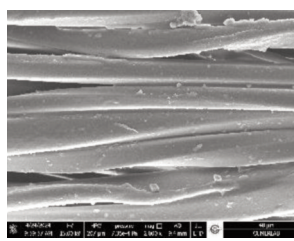
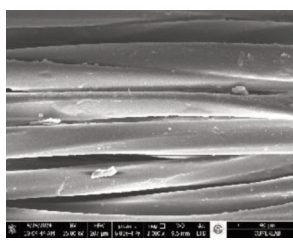
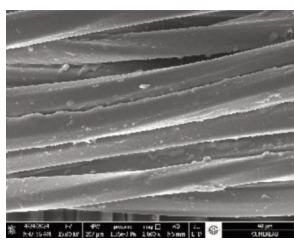
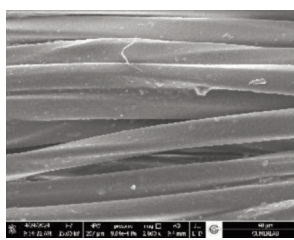
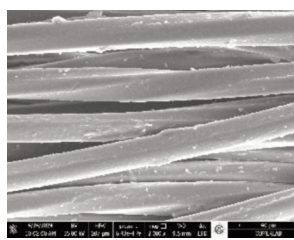
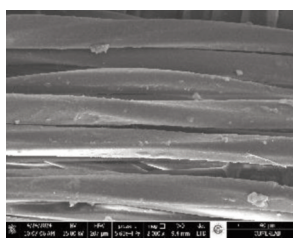
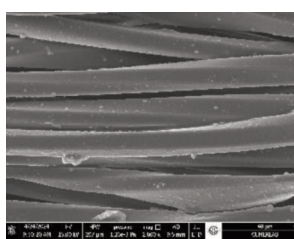
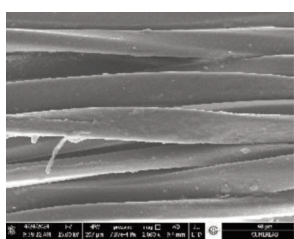
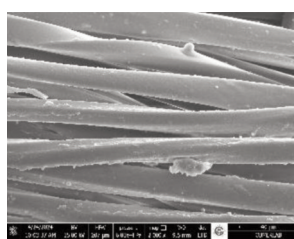
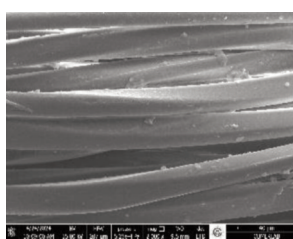
SEM Analysis

SEM analysis of the fabrics was conducted after finished and after 10 times repeated washings (table 4). According to the contact angle measurement results, the highest values were observed in PES-A-F and PES-N-F samples after finishing. When all sample

surface morphologies were examined after repeated washings, the contact angle of the PES-N-F sample was higher than the other samples after repeated washings, although no cross-linker was used.

Whiteness measurement

The water repellency finishing of polyester-based fabrics and the whiteness degrees after repeated washings were measured and the results are shown in figure 4. When the whiteness measurement results taken immediately after the water-repellent finishing process was examined, it was found that air plasma pretreatment applied to fabrics treated with both fluorocarbon-containing and fluorocarbon-crosslinker-containing finishing liquors reduced the whiteness values of the fabrics. It is thought that air plasma causes oxidation on the fabric surface [25] and the decrease in whiteness values is due to this reason. Nitrogen plasma pretreatment applied after the water-repellent finishing increased the whiteness values of the fabrics only slightly. The whiteness values

| SEM ANALYSIS | | | |
|--|--|---|--|
| Fluorocarbon+Cross-linker | | Fluorocarbon | |
| After finish | After 10 washings | After finish | After 10 washings |
|  |  |  |  |
| PES-R-F-C | PES-R-F-C | PES-R-F | PES-R-F |
|  |  |  |  |
| PES-A-F-C | PES-A-F-C | PES-A-F | PES-A-F |
|  |  |  |  |
| PES-N-F-C | PES-N-F-C | PES-N-F | PES-N-F |

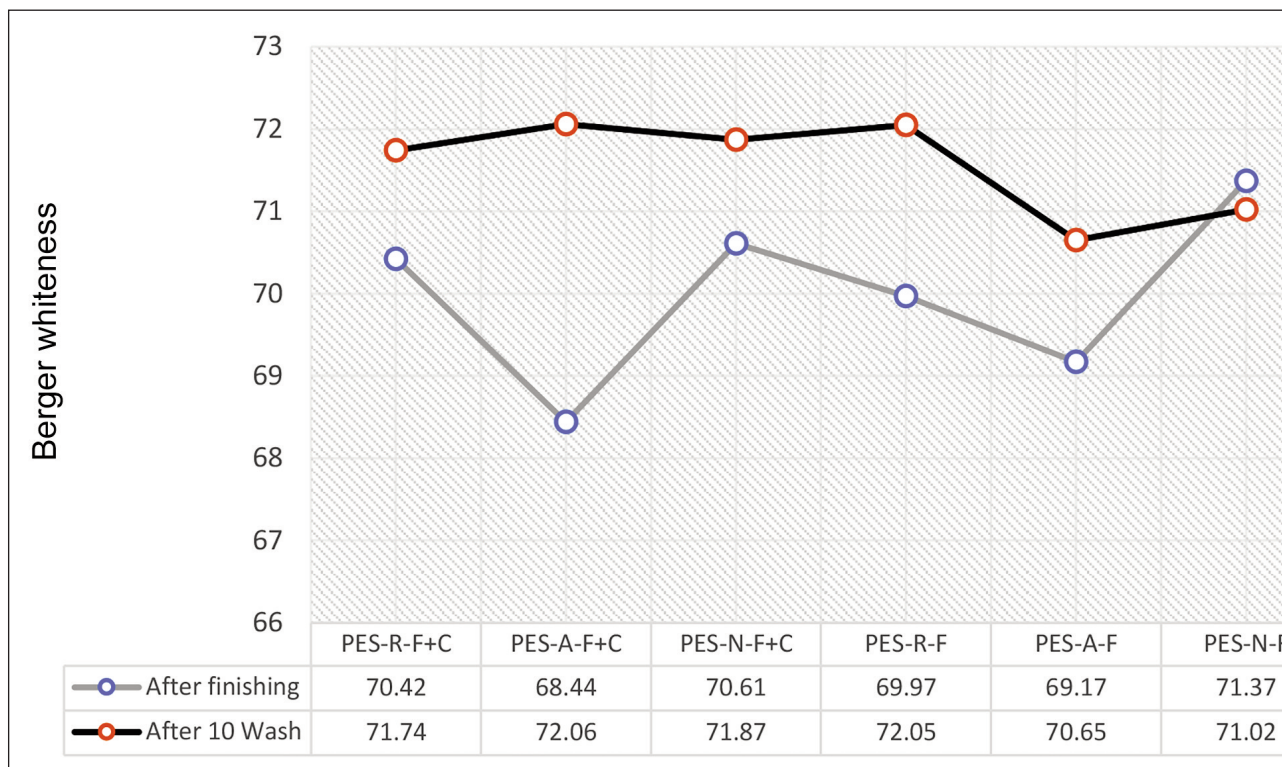


Fig. 4. Berger whiteness values

of the samples treated with nitrogen plasma were very close to the reference sample. The whiteness values of the samples washed 10 times after the water-repellent finishing process were higher than the whiteness values immediately after finishing, except for the PES-N-F coded sample. The whiteness values of the PES-N-F coded sample were close to each other after finishing and after 10 washings. To examine the permanence of the finish, repeated home washing was performed. Due to the bleach in the detergent used, the whiteness values after repeated washings were higher than the whiteness values immediately after finishing.

CONCLUSION

This study aims to investigate the use of plasma pre-treatment before finishing instead of cross-linking chemicals to ensure the permanence of the water-repellency finish. Pre-treated polyester-based fabrics, prepared for dyeing, underwent air and nitrogen plasma treatment. Fluorocarbon solutions with and without cross-linkers were applied to fabrics according to the pad-dry-cure method. Contact angle measurements, FT-IR analysis, SEM analysis, and whiteness measurements were conducted. The samples were subjected to repeated washing 10 times and measurements were made again.

It was found that applying air plasma, along with cross-linker and fluorocarbon, after the water-repellent finishing process led to an increase in the contact angle values of the fabrics. However, it was observed that both air and nitrogen plasma treatments had a detrimental effect on the contact angle values in samples subjected to 10 repeated wash cycles for this finishing type.

Plasma pretreatment enhanced the initial contact angle values of the fabrics. After 20 seconds of the droplet being applied to the fabric, the contact angle

values of the samples exhibited similar behaviour and improvement compared to the reference sample. Upon examining the contact angle values measured after 10 repeated wash cycles, it was observed that the contact angle values of the plasma-treated samples were higher than those of the reference sample. The level of permanence achieved in the fabric after repeated wash cycles with cross-linker chemicals and the level of permanence in the fabric pre-treated with plasma (without the use of cross-linker) were very similar. Plasma pre-treatment eliminates the necessity of employing cross-linkers and yields superior water-repellency values.

SEM analysis results show that after multiple washings, the contact angle of the PES-N-F sample was found to be higher than that of the other samples, despite not using any cross-linker. According to FT-IR analysis, it has been noted that the washing process applied to the fabric does not effectively remove the finishing chemicals.

While nitrogen plasma has no effect on the whiteness after finishing, air plasma reduces the whiteness of the sample. In the water repellency finishing process using only fluorocarbon, air plasma decreased the whiteness value of the sample after finishing, while nitrogen plasma increased the whiteness value of the sample.

After repeated wash cycles, although the whiteness values of the samples treated with fluorocarbon and crosslinker were similar, the application of air and nitrogen plasma reduced the whiteness level of the samples treated with fluorocarbon.

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