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Multifunctionalization and Increased Lifespan of a Worsted Wool Fabric

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Abstract: A lack of dimensional stability of worsted fabrics when laundering leads to a rapid increase in wool textile waste. Dry cleaning is thus highly recommended; however, it requires solvent(s), which are not eco-friendly. The aim of this study was to produce a machine-washable, 100% worsted wool woven as an outer fabric for men's suit jacket that is also water-repellent in order to reduce the number of washes required during use. Chemical treatments were applied through successive paddings, using a blend of aqueous dispersion of polyurethane and polysiloxane (PUPX) for shrink-proofing/dimensional stability, followed by a second blend of an aqueous emulsion of fluorotelomer methacrylate and paraffin/hydrocarbon waxes (C6PW) polymers for water-repellency. The dimensional change of the finished fabric did not exceed 2%, meeting Woolmark requirements AW-1. Zeta potential measurements confirm that the fabric coated with PUPX has an overall anionic nature, which allows the good adhesion of the successive cationic C6PW polymer blend used in the second padding. Additionally, Scanning Electron Microscopy (SEM) analysis confirmed the good adhesion of the first blend (PUPX) to the wool fiber surface and inter-fiber bonding. After the application of (C6PW) resin, the fabric exhibited durable water repellency with a 5/5 spray test rating after 10 washes and dimensional stability, as well as high resistance to wear and abrasion, while retaining a soft feel and good flexibility.

Keywords: worsted wool; woven fabric; fiber coating; machine washable; shrink-proof; water-repellent; abrasion resistance

Citation: Belhaj Rhouma, A.; Campagne, C.; Massika Behary, N.; François, S.; Lanceron, C.; Vieillard, J. Multifunctionalization and Increased Lifespan of a Worsted Wool Fabric. *Coatings* **2023**, *13*, 736. https://doi.org/10.3390/coatings13040736

Academic Editor: Weizhao Zhang

Received: 22 February 2023
Revised: 24 March 2023
Accepted: 30 March 2023
Published: 4 April 2023

1. Introduction

Textile finishes add new properties to a fabric, enhancing the functional value of textile materials to meet specific consumer requirements and end use. They also provide improved outward appearance and increase lifespan or durability in end-products.

The objective of this study was to develop a sustainable and eco-friendly chemical finishing process for men's worsted jackets. The finished fabric was designed to be both machine-washable and water-repellent, while also improving its functional value and appearance, and increasing its lifespan. By making the fabric machine-washable, it would prevent environmental issues caused by the use of toxic solvents in dry cleaning during the garment's use phase. An additional water-repellent finish would reduce the washing frequency, thus reducing the amount of water consumption and energy consumption needed for washing and drying.

Indeed, previous studies [1,2] have also reported the importance of combining machine washability and water-repellent treatments to obtain durable shrink-proofing treatment.

Wool is a bio-based and a sustainable fiber [3]. It has a unique and complex structure that offers many features, such as comfort. It is also known for its natural breathability, due to its complex physical cell structure on the fiber surface. Wool has a natural insulating

property that makes it ideal for keeping the wearer warm. It is also known for its natural moisture-wicking and good appearance properties [4]. These characteristics make wool a suitable material for outerwear garments, especially for suits, where warmth, comfort, and appearance are important considerations [5].

However, the scales present on the fiber surface are responsible for the tendency of wool fabrics to felt and shrink during machine washing [6]. Woolmark [7] defines felting shrinkage as "an irreversible shrinkage caused by progressive entanglement of the wool fibers induced by washing in an aqueous solution". Additionally a second shrinkage phenomenon is described as relaxation shrinkage by Woolmark [7], which defines relaxation as "the dimensional change caused by the release of strains introduced during manufacture, assessed after the initial wash cycle".

According to Kettlewell [8], the relaxation of wool is mainly due to its behavior when exposed to humid conditions. This phenomenon is reversible and governed by strains imposed during spinning, weaving, knitting, and manufacturing. On the other hand, felting is mainly due to the physical structure of the scaly cuticle layer on the fiber surface. This structure means that the friction in the direction of the scales is lower than in the direction against the scales. When two fibers rub against each other, they tend to migrate preferentially in one direction. In the presence of humidity and agitation, this differential friction effect causes the entanglement of fibers when they move together [9]. Thus, felting shrinkage is an irreversible phenomenon and is an important factor in producing machine-washable wool.

Shrink-resistant treatment can be applied at the stage of loose fiber (sliver) or on fabric. When applied to slivers, it reduces the differential friction effect either by removing scales or by masking them. At the fabric stage, treatments aim to reduce inter-fiber movement by using friction-reducing agents [2]. Many treatments can be performed to obtain machine-washable wool [8,10–12]. The Hercosett treatment involves a degradative process using the common toxic substance chlorine [13], followed by an additive method with the deposition of a synthetic polymer [10,14]. Chlorine-based treatments can be hazardous due to the release of chlorine gas into the environment and the production of effluent containing residual chlorine. These substances can react with ammonia or amines to form chloramines, which are a carcinogen and can be harmful to aquatic animals and humans [10]. Therefore, alternative treatments that are more environmentally friendly have been developed, including enzyme-based treatments [15–18], plasma treatments [19–22], and UV/Ozone treatments [23,24]. Biopolymers, such as chitosan, can be used as an additive fiber coating [25,26].

In the industrial production of machine-washable woven garments, polymer treatments are commonly used to prevent felting by masking the scales and forming inter-fiber bonds that prevent movement between fibers [8]. These treatments are simple and versatile and can easily be applied to fabrics, since most of the processing is complete and the felt resistance imparted by inter-fiber bonding is unlikely to be compromised. In this study, polyurethane and polysiloxane polymers were selected and applied to the fabric surface through padding to prevent felting.

Felt and shrink-proofing through the removal or masking of scales improves the washability of wool fabrics but can also cause loss of the fabric's inherent water repellency. To reduce environmental impact, it is beneficial to add an additional property such as water repellency, as it will decrease the frequency of washing and reduce the likelihood of the garment becoming soiled or stained. Rowen [27] defines the water-repellent fabric as a "one in which the fibers are usually coated with a hydrophobic type of compound and the pores are not filled in the course of the treatment. The latter types of fabric are quite permeable to air and water vapor". Water repellency on wool fabrics can be imparted through functionalization treatments with perfluorinated or hybrid resins [6] or by the application of nanoparticles, such as silica particles [28–31]. The application of nanoparticles to a hydrophobic surface increases surface roughness and mimics the Lotus effect; however, its washing fastness and resistance to rubbing are low [29]. On the

other hand, functionalizing the wool fabric with polymers, such as fluorinated compounds, reduces the fabric's surface energy and creates a durable effect through the formation of covalent bonds between the polymer and the wool. Perfluorinated compounds consist of a polar head that reacts with the textile through covalent bonds and a hydrophobic tail made of a fluorinated chain [32]. Long-chain fluorocarbons, which consist of polymers with lateral chains of eight perfluorinated carbon atoms (C8), are the dominant finishing agents in the market due to their ability to provide fabrics with exceptional durable water and oil repellency, as well as good breathability [33]. However, these long chain compounds are highly toxic as they release toxic chemicals, such as perfluorooctanoic acid (PFOA) and perfluoroctane sulfonic acid (PFOS), into the environment [34]. These substances are known to be bioaccumulative and are suspected of causing cancer. They also have an impact on lipid metabolism, as well as hormonal and reproductive systems [33,35,36]. To prevent the toxicity associated with long-chain fluorocarbons (C8), alternative treatments have been developed that can be categorized into two groups: fluorinated and fluorine-free. Fluorinated alternatives, such as fluorinated silicon-based polymers [37], acrylic and methacrylate monomers, fluorinated waxes, and perfluorinated polyurethanes, are made of short chain fluorocarbons known as C4 and C6. The smaller the fluorocarbon chain, the more rapidly it degrades in the environment, and these compounds has 40 times less bioaccumulative effects than PFOA [32,37,38]. Fluorine-free alternatives, such as silicon-based polymers, alkylamines, acrylic-based monomers and polymers, and non-fluorinated paraffin, can also be used to replace toxic substances [33].

In this study, a worsted wool used as an outer fabric for men's suit jackets was subjected to a padding-curing process to produce a multifunctional fabric that is both machine-washable and water-repellent. Shrink-proofing treatments were carried out using polyurethane and polysiloxane polymers blend, while water repellency treatments utilized fluorotelomer methacrylate and paraffin-based polymers.

Characterization methods were used to study dimensional stability, wettability, fiber surface morphology, resistance to wear through abrasion and pilling tests, air permeability and adiathermic power. Multiple wash cycles were performed on the resulting samples to evaluate the durability of the treatments.

SEM and zeta potential analysis allowed analyzing, respectively, the fiber surface morphology and the surface charges before and after the application of coatings. Furthermore, air permeability and adiathermic power measurements were carried out in order to confirm that the treatments did not modify the breathability and comfort properties. The results show the importance of the two-step padding process in obtaining a uniform coating on the fibers, leading to an improvement in the water-repellent properties and an increase in the lifespan of the modified fabrics.

2. Materials and Methods

2.1. Materials

A 100% worsted dyed wool woven fabric weighing 180 g/m^2 and made from 80/2 Nm twisted yarns was supplied by Miroglio (Sliven, Bulgaria) for this study. The fabric was a thin plain weave of 0.31 mm thickness.

Chemicals used for the chemical finishing process are summarized in Table 1. Two commercial polymers provided by supplier S1 were used for the shrink-proofing treatment. The first is an aqueous dispersion preparation of a modified polyether urethane named (PU) anionic polymer, and the second is an emulsion of a cationic functional polysiloxane and fatty acid amide named (PX).

Table 1. Chemicals used for shrink-proofing and water-repellent treatments.

Chemicals	Supplier	Composition [1]	Ionic Character
PU	S1	Aqueous preparation of a modified polyether urethane	Anionic
PX	S1	Emulsion of a functional polysiloxane and fatty acid amide	Cationic
C6	S2	Fluorotelomer methacrylate polymer	Weakly cationic
PW	S3	Paraffin and hydrocarbon waxes	Weakly cationic

[1] Taken from the technical and security data sheets.

Two other commercial polymers, an aqueous emulsion of fluorotelomer methacrylate polymer C6 and paraffin and hydrocarbon wax-based compounds provided by suppliers S2 and S3, respectively, were blended and used for the water-repellent treatment. The C6 resin is a fluorotelomer methacrylate polymer obtained from short-chain fluorotelomer chemistry, as shown in Figure 1, and is free of PFOA and PFOS. The second polymer consists of paraffin and hydrocarbon waxes (PW), which are fluorine-free polymers. Paraffin and hydrocarbon waxes are both made from petroleum. Paraffin wax is a mixture of high molecular weight straight-chain alkanes and is obtained through the emulsion polymerization, as seen in Figure 2.

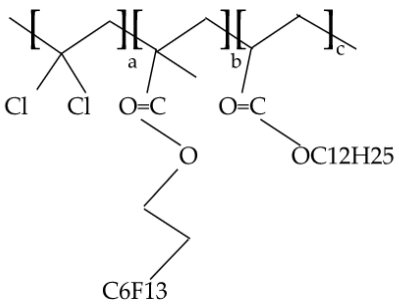


Figure 1. Schematic of a 6:2 fluorotelomer methacrylate polymer [39].

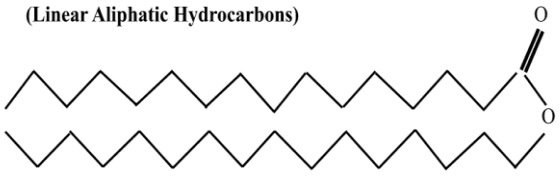


Figure 2. Chemical structure of paraffin (linear aliphatic hydrocarbons) [40].

PUPX is the resulting polymer made from blending PU and PX resins.

C6PW is the resulting polymer made from blending C6 polymer and paraffin and hydrocarbon waxes (PW).

Table 1 shows the characteristics of the chemicals, based on the manufacturers' information. According to the safety data sheet, formaldehyde was not used in chemicals.

2.2. Methods

2.2.1. Finishing Application on the Textile: Two-Step Padding Treatment

Finishing treatments were performed using the padding curing process. The chemicals were diluted in water and the pH was adjusted to 5 using acetic acid. Samples were then immersed in the bath solutions and squeezed by the rollers of the padding machine to achieve a 70%–80% pick-up ratio. Finally, the treated samples were dried and cured in an oven.

However, for confidentiality reasons, the specific formula and treatment conditions cannot be disclosed.

2.2.2. Textiles Testing

Prior to testing, samples were conditioned at 20 °C and 65% relative humidity for 24 h.

Dimensional Stability

Dimensional stability was evaluated using the International Wool Textile Organization (IWTO) test method TM31 [7]. Washing tests were carried out in a household washing machine (Samsung) using the wool/delicate program and a commercial detergent special for wool garments.

The fabric sample size of 17 cm × 17 cm was prepared, and a square was marked inside with dimensions of 14 cm × 14 cm. The marked fabrics were subjected to one relaxation cycle, using the 7A program; a gentle washing cycle at 40 °C with no spin; and three felting cycles, using the 5A program at 40 °C and 800 rpm. Then, area shrinkage was calculated as in Equation (1):

$$\text{Area shrinkage (\%)} = ((\text{OM} - \text{FM})/\text{OM}) \times 100 \quad (1)$$

where OM is the original measurements (cm) of the marked square (14 cm × 14 cm), which represents the sum of shrinkage in both warp and weft directions before washing, and FM is the final measurement (cm) of the marked square, the sum of warp and weft variations after washing. A sample was tested from both the untreated and treated fabric.

Spray Test

The tests were performed according to NF EN ISO 4920 [41]. Each test was performed with 250 mL of distilled water and sprayed on a test specimen that has been mounted on a ring at a 45° angle. The spray rating was determined by comparing the appearance of the specimen with descriptive standards and photographs, where:

- 5 is attributed when no sticking or wetting of the specimen;
- 4 corresponds to a slight random sticking or wetting of the specimen face;
- 3 corresponds to the wetting of specimen face at spray points;
- 2 is the partial wetting of the specimen face beyond the spray points;
- 1 is the complete wetting of the entire specimen face beyond the spray points;
- Additionally, 0 represents when a complete wetting of the entire face of the specimen is obtained [41].

The measurements were performed after 0, 1, 5, and 10 washing cycles in a domestic washing machine (Samsung) with the wool/delicate cycle at 40 °C and no drying between washes.

Two different liquids were used for the spray tests. First, it was conducted with 100% distilled water. This was followed by a second test using 250 mL of mixture 90/10 water/isopropyl alcohol. In fact, the surface tension of the isopropyl alcohol is about 21.9 mNm^{-1}, which is much lower than that of the distilled water, about 72.8 mNm^{-1}. The resulting solution, with a very low surface tension, allows textile samples to be tested under more severe conditions.

Abrasion Tests

Abrasion tests were performed according to the corresponding standards NF EN ISO 12947-1 [42] and NF EN ISO 12947-3 [43], following the weight loss and two yarns breakage methods, in order to evaluate the resistance to wear of the treated fabrics. The Martindale abrasion tester subjects a circular specimen to a defined load and rubs it against an abrasive medium (standard specimen, wool based) with a translational movement, tracing a Lissajous figure.

First, the untreated fabric was subjected to abrasion cycles in order to determine the cut-off point, which is the number of rubs required until the two-yarn breakage. Then, the treated samples were exposed to the same number of rubs corresponding to the cut-off

point of the untreated fabric. The evaluation of the abrasion resistance of all fabric samples was determined using the mass loss rate, according to Equation (2):

$$ML\ (\%) = ((Mi - Mr)/OM) \times 100 \quad (2)$$

where ML (%) is the mass loss rate, Mi is the initial weight of the specimen, and Mr is the weight of the rubbed specimen.

Pilling Tests

Pilling tests were conducted according to standard NF EN ISO 12945-2 [44,45]. The objective of these tests was to assess the effect of the various treatments on the appearance and aesthetics of wool fabrics. Untreated and treated samples were subjected to pilling tests using Martindale testing machine where samples rub against each other following circular movements. Then, the levels of pilling, fuzzing or matting were assessed by visual analysis, according to standard NF EN ISO 12945-4 [45].

Bending Length

The standard NF EN ISO 9073-7 [46] defines the bending length as the length of a rectangular sample (25 cm × 2.5 cm), fixed at one end and free at the other. The specimen has a tendency to bend under its own weight. Then, flexural rigidity was calculated from the bending length using the following Equation (3):

$$G = m \times C^3 \times 10^{-3} \quad (3)$$

where G is the mean flexural rigidity (mN/cm), m is basis weight of the specimen (g/m^2), and C is the overall mean bending length of the specimen (g).

The flexural rigidity is a ratio of small changes in bending moment per unit width of the material to corresponding small changes in curvature.

Since the fabric had a plain weave, and the warp and weft yarns were similar, three measurements were carried out only in the weft direction for each untreated and treated fabrics.

Air Permeability Test

The air permeability was tested as the bulk of the conveyed atmosphere via the unit region of the fabrics in a given time horizon (unit $L/m^2/s$). Tests were performed following ISO 9237-1995 [47] standard method. The dimension of the fabric was 20 cm^2 and the differential-pressure was 100 Pa. Ten measurements were carried on each sample. The objective of these tests was to evaluate the effect of the different treatments on the porous structure and the breathability of the wool fabric.

Adiathermic Power

The measurements of the adiathermic power PA (%) were carried according to the NF G07-107-1985 standard [48] using the cylindrical heater method, as in Figure 3. These tests were performed in order to assess the impact of the coatings on the initial thermal properties of the wool fabric used for this study.

Two rectangular samples measuring 150 mm × 130 mm each were taken from both the untreated and treated fabrics. The smaller dimension corresponds to the chain direction. The adiathermic power PA (%) is then determined using the calibration curve provided, using the following Equation (4):

$$PA\ (\%) = f((1 - {U_1}^2/{U_0}^2) \times 100) \quad (4)$$

where U_0 (V) is the supply voltage of the heating resistance for the cylinder without the sample being tested, U_1 (V) is the supply voltage of the heating cylinder covered with the tubular specimen.

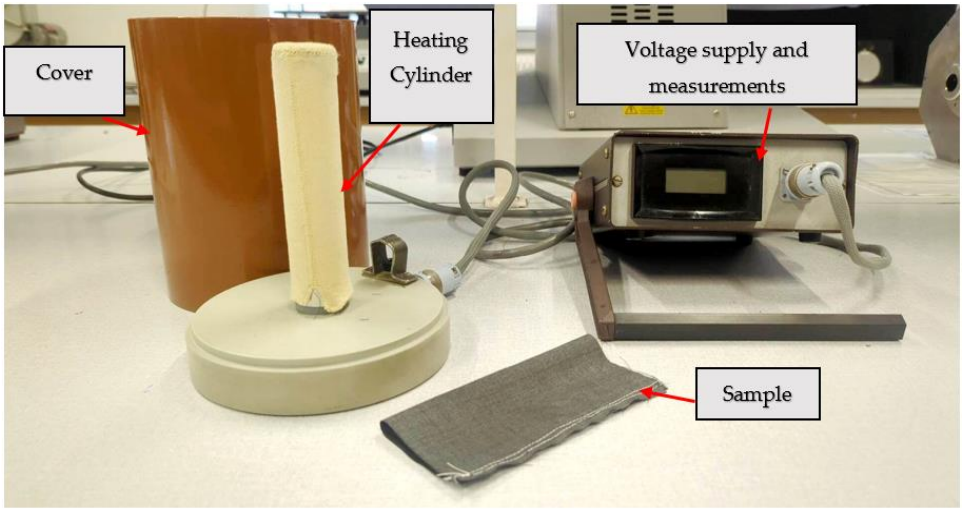


Figure 3. Experimental device for determining adiathermic power.

Two test specimens were tested of both the untreated and treated fabric and the average was calculated.

2.2.3. Fiber-Surface Physico-Chemical Analysis

SEM Analysis

The surface morphology of treated fabric was investigated using a scanning electron microscope.

A ZEISS EVO 15 electron microscope (Zeiss Group, Oberkochen, Germany) was used for SEM analysis. Materials were previously metallized by a gold layer at 18 mA for 360 s with a Bio-Rad E5200 device (Bio-Rad, Hercules, CA, USA). Tests were carried out after each treatment, washes, and abrasion tests.

Zeta Potential Analysis

Zeta potential is defined as the electrical charge density and ionic character on the surface of a particle. It is measured to evaluate polymer adhesion to fiber surfaces and determine optimal treatment conditions, with pH being the most crucial parameter in textile finishing. The zeta potential values of both textiles and polymers at various pH levels were measured using two different methods.

First, the flow potential method was used to measure the electronic charge of the fabric using the ZETACAD instrument from CAD Instruments (CAD, Naucelle, France). ZETACAD determines the density of the electrical charge, known as the zeta potential. Then, the ZETASIZER device was used to determine whether the polymers and mixed polymers in colloidal solutions were anionic or cationic through electrophoresis—the movement of charged particles in a liquid under an electrical field. The zeta potential of the fabric and each polymer was measured in aqueous solutions at pH values of 3, 4, and 8, adjusted using acetic acid or sodium hydroxide.

Water Contact Angle and Water Sliding Angle

The water contact angle (WCA) and water sliding angle (WSA) were measured using the GBX Digidrop (GBX Scientific Ltd, Dublin, Ireland) apparatus to evaluate the wetting properties of wool fabrics after each step of the functionalization process. The contact angle measurements define the three-phase interaction between solid, liquid, and vapor at the edge of a sessile droplet.

As per the standard NF EN 828 [49], the WCA is the angle formed by a tangent to the droplet's contour that passes through one of the triple points and the baseline.

Ten random positions were selected for water contact angle measurements, and the average value and standard deviation were calculated. Due to the rough nature of the

fabric, there is some uncertainty in defining the interface between the fabric and the testing liquid.

The water sliding angle (WSA) was determined as the angle at which the droplet rolls over the solid surface. The volume of the water droplets used for both WCA and WSA measurements was 10 µL.

Roll-off angle measurements were conducted by placing a water droplet on the horizontal fabric, followed by gradually tilting the fabric at a rate of 2°/s until the droplet rolled off, as shown in Figure 4. The WSA values were determined by taking the average of the measurements made at 10 different points on each sample surface.

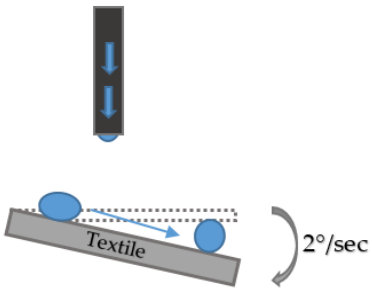


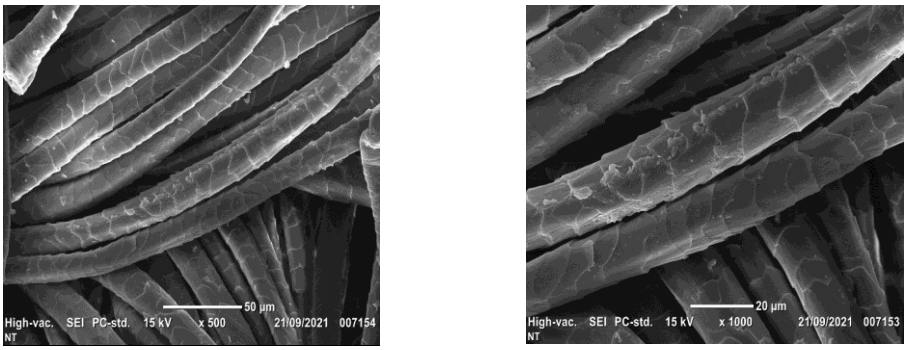
Figure 4. Water sliding angle "WSA" measurements.

Tests were carried out after several washing cycles at 40 °C using the wool/delicate program on a household washing machine.

3. Results

3.1. Dimensional Stability

The shrink-proofing finish aims to modify the scale structure of wool fibers to reduce friction and increase resistance to washing [50]. This study aimed to produce a machine-washable worsted wool fabric, which has not undergone any felting treatments, as confirmed by the SEM images in Figure 5 and the results in Table 2. The SEM images in Figure 5a,b demonstrate the overlapping cuticular scales present on the outer surface of the fibers.



(**a**) (**b**)

Figure 5. SEM images of the non-treated fabric used for this study (**a**,**b**).

Table 2. Area shrinkage ratio.

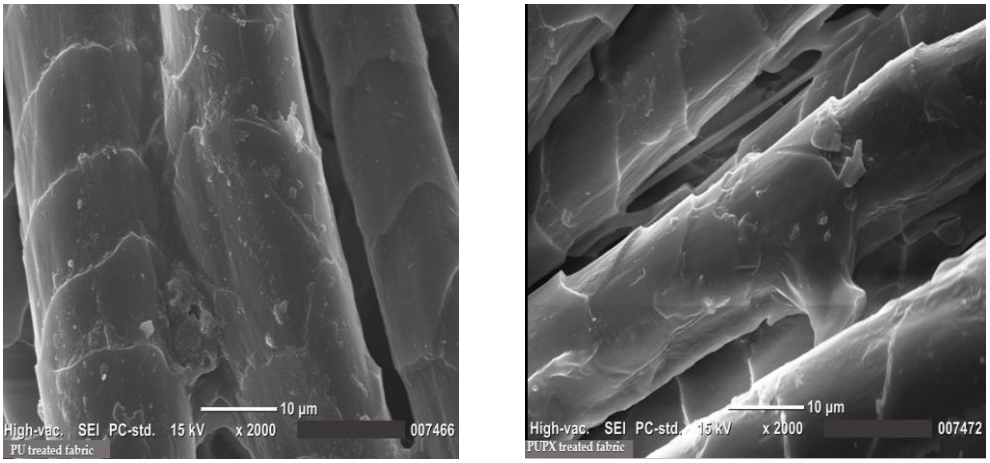
	Area Shrinkage (%)
Initial fabric	−6
Fabric treated with PU	−4
Fabric treated with PUPX	−2

Table 2 displays the percentage of area dimensional change before and after the various treatments. The area dimensional change is approximately 6%, caused by an increase in

the directional friction effect during washing, which is a result of the scales present on the fiber surface.

In order to meet the requirements of the machine washable standard AW-1, the area dimensional change must not exceed (−3%). When the fabric was treated with PU resin alone, the dimensional variation was approximately (−4%), which did not meet the Woolmark standard. However, after padding with the PUPX polymer blend, the shrinkage rate dropped to 2%.

SEM images in Figure 6b reveal a uniform and smoother coating on the fiber surface and inter-fiber bonding when the PUPX coating is used, in comparison with the fabric coated with PU (Figure 6a).



(**a**) (**b**)

Figure 6. SEM images of the fabric treated with PU (**a**) and with PUPX (**b**).

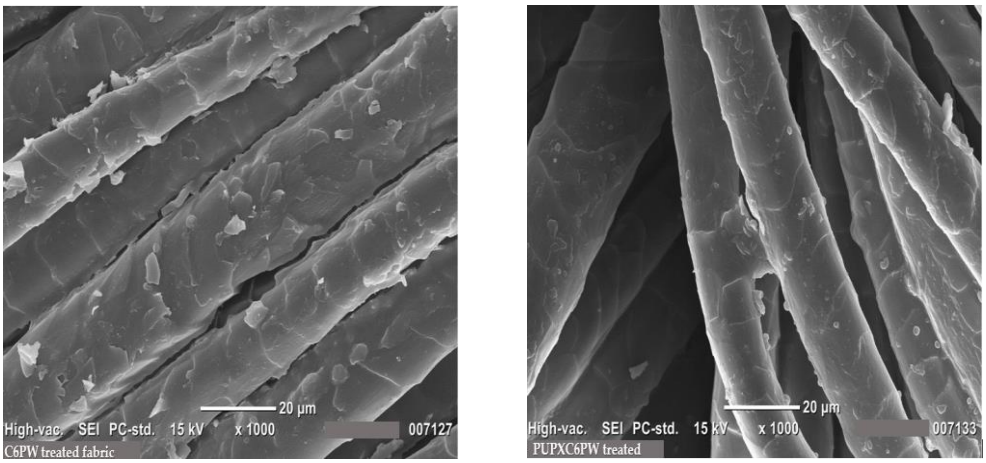
In conclusion, the dimensional stability of wool fabric was improved by coating it with a polymer blend of polyurethane and polysiloxane. The final treated fabric had an area shrinkage rate of 2%, which meets the Woolmark specification AW-1 for machine washability.

3.2. Water Repellency

Several combinations using single-step or two-step padding with the water-repellent polymer C6PW alone or in combination with the PUPX blended polymers were tested. SEM images in Figure 7a reveal that treating the fabric with only the C6PW polymer blend resulted in a non-homogeneous coating on the fiber surface. In contrast, combining both treatments, as shown in Figure 7b, resulted in a uniform coating of the fiber surface and allowed for inter-fiber bonding. The fabric treated with both polymer blends is referred to as the PUPXC6PW fabric.

Table 3 shows the results of spray tests performed on samples treated with the PUPXC6PW polymer blend. The tests were conducted using 100% water and a mixture of (90/10) water and isopropyl alcohol. After 10 washing cycles using a wool/delicate program, the spray test rating with water was around 5, indicating that there was no sticking or wetting of the sample face. In contrast, the spray test performed with the (90/10) water/isopropyl alcohol mixture had a rating of around 3, with wetting observed at the spray points where the droplet was absorbed.

The surface tensions of water, 100% isopropyl alcohol, and the (90/10) water/isopropyl alcohol mixture were 72.8 mNm^{-1}, 21.9 mNm^{-1}, and 38.7 mNm^{-1}, respectively. This explains the difference in wetting behavior between the two tests, as the mixture of water and isopropyl alcohol has a lower surface tension.



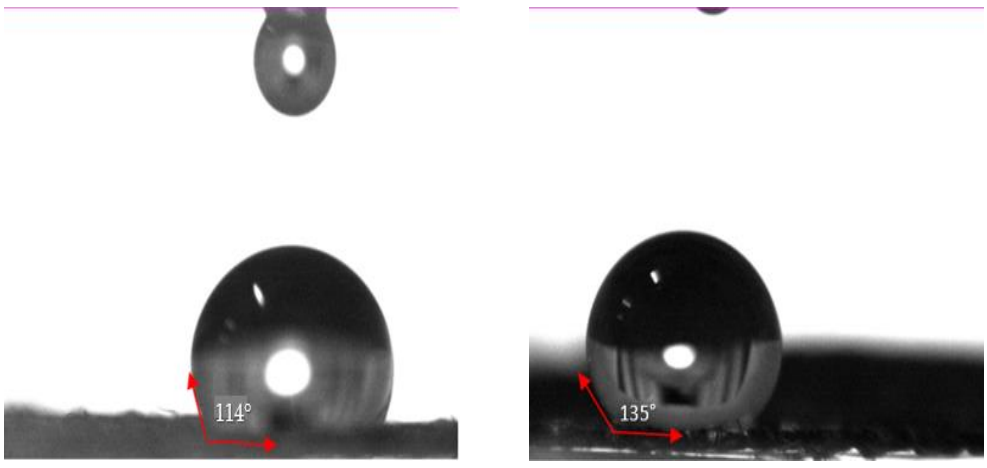
(**a**) (**b**)

Figure 7. SEM images of the fabric treated with C6PW (**a**) and combination PUPXC6PW (**b**).

Table 3. Spray test results with 100% water and (90/10) (water/isopropyl alcohol).

	0 Washes		1 Wash		5 Washes		10 Washes	
	100% W	**90% W + 10% Isop**	**100% W**	**90% W + 10% Isop**	**100% W**	**90% W + 10% Isop**	**100% W**	**90% W + 10% Isop**
PUPXC6PW-treated Fabric	5	5	5	5	5	4	5	3

Furthermore, the water contact angle (WCA) and water sliding angle (WSA) were determined. These parameters provide the objective indications of wettability, surface energy, and adhesion. Figure 8 shows the contact angle of a drop deposited on the fiber surface of the untreated fabric (a) and PUPXC6PW-treated sample (b). The initial water contact angle of the untreated fabric is around 114°, due to the natural hydrophobic properties of wool fiber. For the PUPXC6PW-treated fabric, the WCA increased to 135°. This increase in WCA, from 114° to 135°, suggests that the treated fabric has a more hydrophobic surface, which is reflected in the improvement of the spray test results.



(**a**) (**b**)

Figure 8. Water contact angles measured from water droplets deposited on the surface of the untreated fabric (**a**) and PUPXC6PW-treated fabric (**b**).

In addition, according to Table 4, the water sliding angle (WSA) was around 69° before treatment and decreased to 33° after treatment with the PUPXC6PW, confirming improved

water repellency of the coated sample. However, the WSA increased with the number of washes, reaching an angle of 44° after 10 wash cycles, which still shows more efficient water repellency than the untreated fabric.

Table 4. Area shrinkage, WCA and WSA measurements of non-treated and PUPXC6PW-treated fabric.

Samples	Area Shrinkage (%)	WCA (°)	WSA (°)			
			0 Washes	1 Wash	5 Washes	10 Washes
Non-treated	−6	114	69 ± 3.9	-	-	-
Fabric treated with PUPXC6PW	−2	135	33 ± 0.6	36 ± 1.5	40 ± 2.7	44 ± 2.9

The combination of both treatments resulted in better polymer adhesion to the fiber surface, resulting in a durable treatment. Additionally, the treatment did not alter the dimensional stability achieved by the first treatment, which remained stable at 2% with the PUPXC6PW polymer blend and met the Woolmark AW-1 specifications. These results demonstrate that the use of the PUPXC6PW polymer blend effectively improved the water repellency and dimensional stability of the wool fabric, making it suitable for machine washable applications.

3.3. Abrasion and Pilling Tests

Initially, the untreated fabric was tested in order to find the endpoint which corresponds to the number of abrasion cycles until rupture, two or more yarns break, or hole appearance, while also measuring weight loss. The untreated fabric reached its endpoint at 30,000 cycles with a 30% weight loss as shown in Figure 9. The treated samples were then, tested under the same conditions. The PUPX- and PUPXC6PW-treated samples showed improved durability with only 5% and 10% weight loss, respectively. The PUPXC6PW treatment made the fabric three times more durable than the untreated fabric.

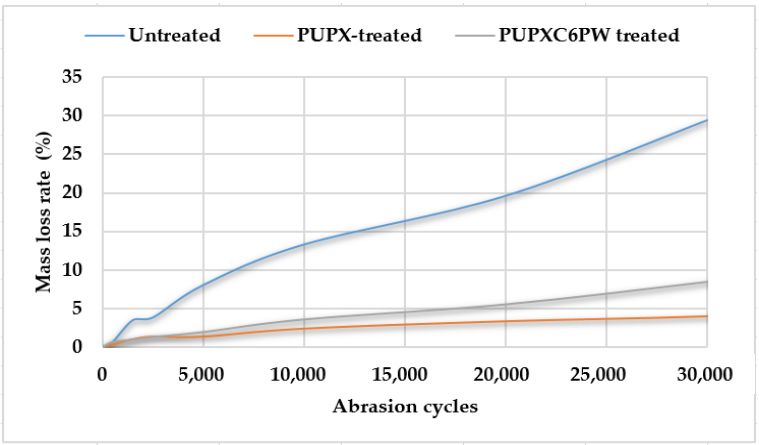


Figure 9. Mass loss rate variation during abrasion cycles before any washes.

The fabric's lifespan was evaluated through pilling and washing tests to assess the effect of treatments on the fabric's appearance. The untreated fabric showed pilling after 500 cycles, while the treated fabric with PUPXC6PW combination showed resistance to 2000 cycles before pilling. After 25,000 cycles, Figure 10, the untreated fabric (a) had significant matting, pilling, and fuzzing, while the treated fabric (b) had only slight fuzzing but no significant matting or pilling.

(**a**) (**b**)

Figure 10. Samples of untreated fabric (**a**) and treated with a PUPXC6PW combination, (**b**) tested after 25,000 pilling cycles.

However, the combination of two treatments improved dimensional stability, water repellency, and resistance to abrasion and pilling but negatively influenced the fabric's handling, causing it to become more rigid and rough. Hence, in order to improve fabric handling and flexibility, the chemical concentrations used need to be optimized.

3.4. Chemical Treatment Optimization and Increase in Lifespan

The main drawback of using polymers in fabric surface treatment is their negative effects on fabric softness and handling when used in large quantities. The deposited polymers represent 14% of the final treated fabric weight. Using fewer polymers will not only improve fabric handling, but it will also help to reduce the overall environmental impact of the worsted wool fabric.

Several combinations have been tested for optimum conditions and are presented in Table 5. The first line corresponds to the combination used for the above reported results. First, the concentrations of each padding bath were varied and samples were dried and cured under specific conditions. Dimensional change, spray tests, and abrasions were performed in order to evaluate the impact on the shrink-proofing and hydrophobicity performances previously imparted. Additionally, bending length tests were carried out in order to investigate the impact of the fiber coating on the fabric handling and softness.

Table 5. Different combinations tested.

1st Padding Bath	2nd Padding Bath
(4/5; 1/5) (PU; PX)	(1/3; 2/3) (C6; PW)
(3/4; 1/4) (PU; PX)	(1/2; 1/2) (C6; PW)
(2/3; 1/3) (PU; PX)	(2/3; 1/3) (C6; PW)
(1/2; 1/2) (PU; PX)	(1; 0) (C6; PW)

Figure 11 shows mass loss rate of the PUPX-treated fabric samples with the four different polymer blend combinations (described in Table 5). The mass loss was recorded for 30,000 abrasion cycles performed after zero, one, five, and ten washing cycles, respectively. The use of PU in high percentages enhanced the abrasion resistance of wool fabric. In fact, mass loss for the (4/5; 1/5) PUPX was only 4% initially and reached almost 8% after 5 washes. On the other hand, this loss was as high as 6 to 7% with the other combinations for zero washes and was further enhanced to 10% after 10 washes.

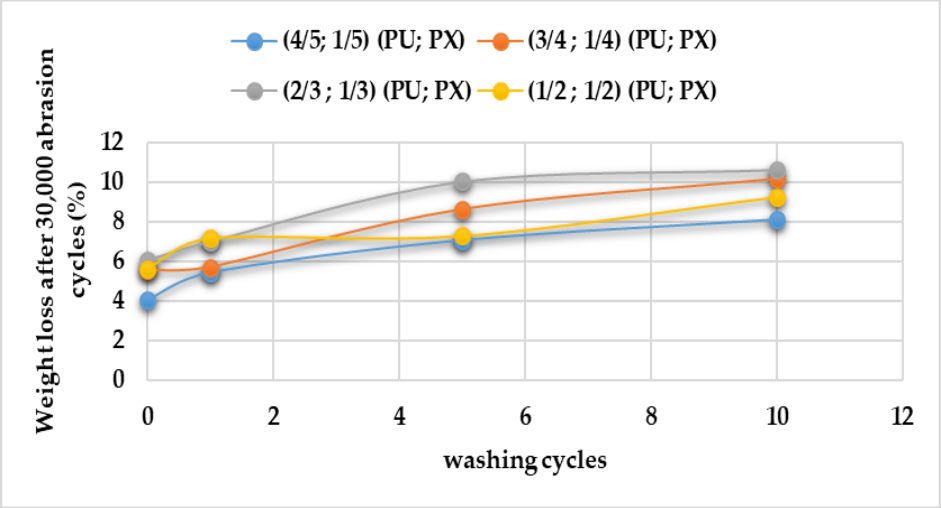


Figure 11. Mass loss rate of fabric, treated with the four different polymer blend combinations (PU;PX), measured after zero, one, five, and ten wash cycles, with 30,000 abrasion cycles for each.

Results in Table 6 show the variation of flexural rigidity and the area shrinkage with the variation of PUPX polymers concentrations in the first padding bath. The mean flexural rigidity decreases by reducing the PU percentage. In fact, when using 4/5 of PU resin blended with only 1/5 PX, flexural rigidity was about 21.86 mN/cm, and it decreased to 9.07 mN/cm when using (1/2; 1/2) (PU; PX). The reduction in PU polymer percentages increased only slightly the surface shrinkage. According to abrasion resistance and flexural rigidity, the combination (1/2; 1/2) (PU; PX) was chosen for the remaining tests.

Table 6. Area shrinkage and flexural rigidity results of the samples treated with the 1st padding bath.

1st Padding Bath	Area Shrinkage (%)	Flexural Rigidity G (mN/cm)
Untreated fabric	−6	8.87 ± 0.1
(4/5; 1/5) (PU; PX)	−2	21.86 ± 0.2
(3/4; 1/4) (PU; PX)	−2	15.99 ± 0.2
(2/3; 1/3) (PU; PX)	−3	12.46 ± 0.1
(1/2; 1/2) (PU; PX)	−3	9.07 ± 0.2

Then, in the next step, the concentrations of the polymers used in the second padding bath were varied (see Table 5). For the remainder, the samples were first treated with (1/2; 1/2) (PU; PX) and followed up with the four different polymer blend combinations (C6; PW) of the second padding bath.

Figure 12 shows the mass loss rate at 30,000 abrasion cycles after zero, one, five, and ten washing cycles for fabrics treated with (1/2; 1/2) (PU;PX) for first padding followed by a second padding with one of the three different combinations of C6PW. Even after 10 wash cycles, all treated samples have a mass loss rate, which is below 10%. In contrast, the untreated fabric loses 30% of its initial weight after the same number of abrasion cycles before washing.

Tables 6 and 7 show that the mean flexural rigidity increased from 8.87 mN/cm for the untreated sample to 37.8 mN/cm after treatment with the first combination (1/2; 1/2) (PU; PX) (1/3; 2/3) (C6; PW). McNeil [51] explains this increase in bending rigidity as being due to the coatings effectively pinning the edges of the wool cuticle scales and introducing inter-fiber bonding, which is also confirmed by the SEM pictures in our study (see Figure 7a,b and Figure 8b).

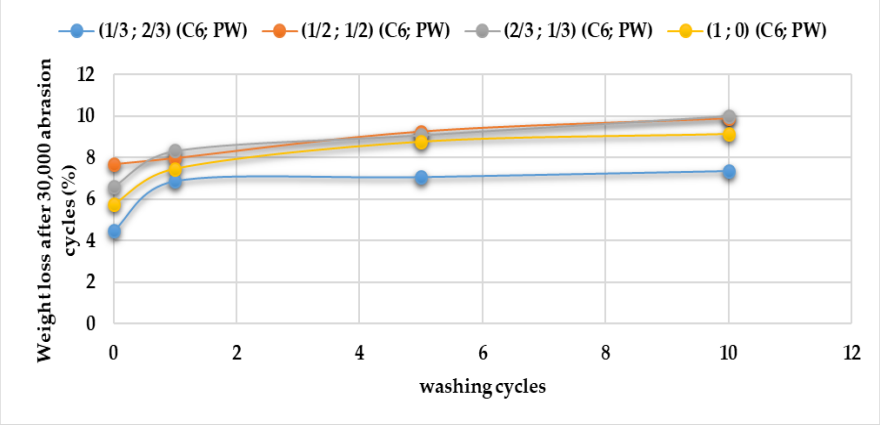


Figure 12. Mass loss rate fabric, treated with (1/2; 1/2) (PU;PX) for the first padding and then with the four different polymer blend combinations (C6; PW), measured after zero, one, five, and ten washes with 30,000 abrasion cycles for each.

Table 7. Area shrinkage and flexural rigidity results of the samples first treated with (1/2; 1/2) (PU; PX) and then with different combinations of the second padding bath C6PW.

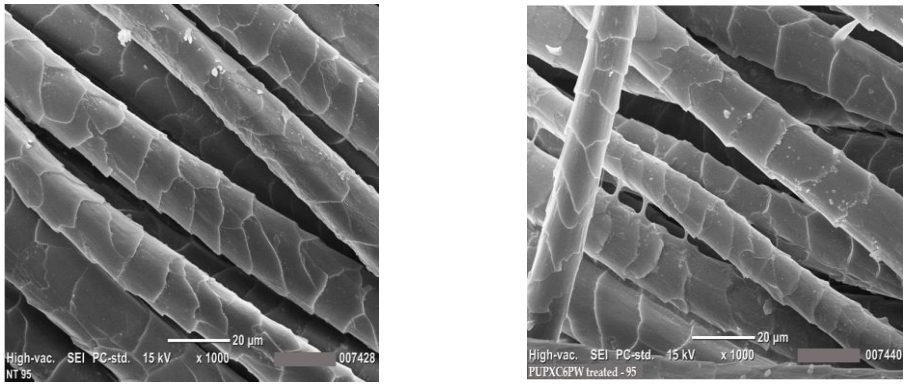
2nd Padding Bath	Area Shrinkage (%)	Flexural Rigidity G (mN/cm)	Spray Test Results							
			0 Wash		1 Wash		5 Washes		10 Washes	
			100% W	90% W + 10% Isop	100% W	90% W + 10% Isop	100% W	90% W + 10% Isop	100% W	90% W + 10% Isop
(1/3; 2/3) (C6; PW)	−2	37.8 ± 0.2	5	5	5	5	5	4	5	4–3
(1/2; 1/2) (C6; PW)	−2	19.36 ± 0.3	5	4	5	4	5	4	5	4–3
(2/3; 1/3) (C6; PW)	−2	17.67 ± 0.2	5	4	5	4	5	4	5	4–3
(1; 0) (C6; PW)	−3	19.4 ± 0.5	5	4	5	4	5	3	5	3–2

However, reducing the concentration of the PW resin decreases the flexural rigidity of the treated fabric with the first combination from 37.8 mN/cm to 17.67 mN/cm. In fact, the use of PW in low concentration not only slightly improves the mean flexural rigidity but also enhances the resistance to wettability. Furthermore, the results of the spray tests have been improved with the optimization of the first padding bath.

The combination for optimal results of abrasion resistance, flexural rigidity, and the spray test is the case of the fabric sample that was first padded with (1/2; 1/2) (PU; PX) and then subjected to a second padding with (2/3; 1/3) (C6; PW).

Moreover, this combination increases product lifespan. In fact, in the literature, many studies refer to abrasion resistance as a significant parameter linked to product durability [52,53]. According to the abrasion results before washing in Figure 10, the combined PUPXC6PW finishing treatment made the fabric three times more durable than the untreated fabric. While the untreated fabric lost 30% of its initial weight, the PUPXC6PW-treated fabric lost only about 10% of its initial weight at zero washing cycles. The fabric's lifespan was also evaluated by pilling to assess the impact of treatments on the fabric's appearance, as seen in Figure 11. The treated fabric showed only slight fuzzing, but no remarkable matting or pilling, even after multiple pilling tests. To accelerate the aging of the fabric, a washing cycle at 95 °C was performed.

SEM pictures (Figure 13) show the untreated fabric (a) and the fabric treated with the PUPXC6PW combination (b) where both were washed at 95 °C. The photos of the untreated samples, Figure 13a reveals slight damage to the fiber surface, while for the treated fabric, as seen in Figure 13b, slight damage occurs but it concerns only the coating layer on the fiber surface. As a result, the product lifespan was improved by these treatments due to better resilience to abrasion and pilling, as well as wash-durable dimensional stability and water repellency.



(**a**) (**b**)

Figure 13. SEM images for untreated fabric (**a**) PUPXC6PW-treated fabric (**b**) and washed once at 95 °C.

3.5. Air Permeability and Adiathermic Power

The samples treated with the final combination were tested to evaluate the impact of the treatments on the initial air permeability and thermal behavior of the worsted wool fabric used for this study. The results of the adiathermic power PA (%) and the air permeability (L/m^2/s), of the initial untreated fabric, the sample treated with (1/2; 1/2) (PU; PX), and the sample treated with the final optimized combination (1/2; 1/2) (PU; PX) and (2/3; 1/3) (C6; PW) are presented in Figure 14.

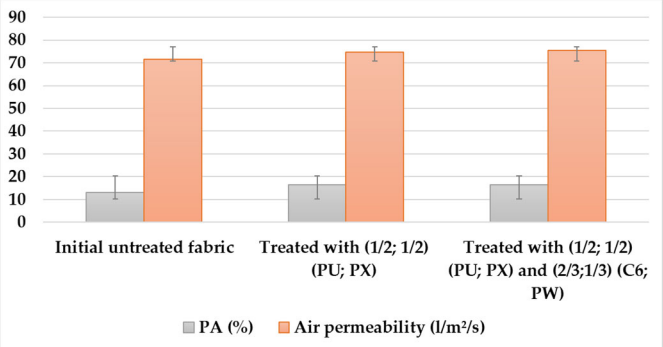


Figure 14. Variation of the adiathermic power PA (%) and the air permeability (L/m^2/s) of the initial untreated fabric, the sample treated with (1/2; 1/2) (PU; PX), and the sample treated with the final optimized combination (1/2; 1/2) (PU; PX) and (2/3; 1/3) (C6; PW).

The air permeability results, as seen in Figure 14, show that the applied treatments do not affect the porous structure of the initial worsted wool fabric used for this study. In fact, after the first padding with (1/2; 1/2) (PU; PX), a slight increase in the air permeability of the untreated fabric, of the order of 4%, was obtained. Then, the sample treated with the final optimized combination of (1/2; 1/2) (PU; PX) and (2/3; 1/3) (C6; PW) showed a further improvement in air permeability, with a 5% increase compared to the untreated fabric.

Furthermore, an increase in adiathermic power was observed after treating the sample with both dimensional stability and water-repellent treatments. In fact, adiathermic power refers to the rate at which a material can transfer heat without conducting it. It is known from the literature that wool fibers have natural insulating properties that makes it ideal for keeping the wearer warm [4]. This property is relative to higher adiathermic power.

However, the applied treatments caused only a slight change in the air permeability and adiathermic power, and as a result, did not have a negative influence on the initial properties of the fabric used.

4. Discussion

The study investigated the effect of combining machine washability and water-repellent treatments on worsted wool fabric and evaluated the impact on the performance of each finishing process as well as on the product durability.

4.1. Influence of a First Padding with PUPX Blend

The dimensional stability of a wool fabric was improved by coating with a polymer blend made of polyurethane and polysiloxane polymers (PUPX). The area dimensional change ratio of the final treated fabric was up to (−2%), which meets with Woolmark specifications AW-1 for machine washability.

In fact, potential zeta measurements confirm the good adhesion of the PUPX polymer blend to the fiber surface. Figure 15 shows the zeta potential values of (1) the non-treated worsted wool fabric, (2) PUPX polymer blend aqueous solution, and (3) the fabric padded with the PUPX polymer blend. The initial worsted wool fabric, as a received sample used for this study, has a negative charge regardless of what the pH value is and is highly receptive to the cationic polymer blend of PUPX. It is important to remember that the PU resin has an anionic character, and when blended with the PX, it yields a cationic character and that the polymer blend was applied to the fabric surface at pH 5. Therefore, the adequate adhesion of the polymer blend PUPX to the wool fiber surface is achieved with the inter-fiber bonds resistant to the washing process.

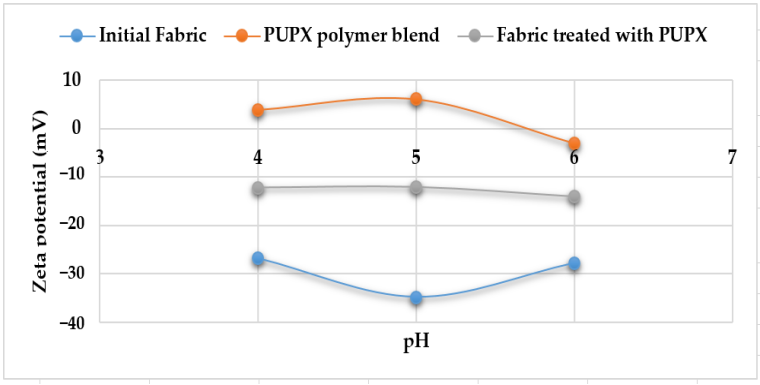


Figure 15. Zeta potential of the non-treated fabric, PUPX polymer blend, and PUPX-treated fabric.

4.2. Influence of a Second Padding with C6PW for Increased Water Repellency

In addition, potential zeta measurements confirm that the fabric coated with PUPX has an overall anionic nature, which is therefore highly receptive to cationic C6PW polymer blend used in the second padding. Hence, padding with the first blend of polymers contributed to the subsequent uniformity of the fluorocarbon coating (C6PW) on the fiber surface. This may also be due to the fact that the polyurethane polymer used is a crosslinking agent that will have a synergistic effect with the polymers of the second padding bath. In fact, Roy Choudhury et al. [37] reported that the durability of fluorocarbon finishes can be improved by the use of a polyurethane polymer as a cross-linking agent.

The treated fabric PUPXC6PW showed good water repellency results. The spray tests with water were rated 5 even after 10 washes. For testing under more severe conditions, a second series of spray test measurements was carried out using (90% water, 10% isopropyl alcohol). The impact of isopropyl alcohol on the wetting behavior was reported by C.-M. Tåg et al. [54], where it was found that mixing water and isopropanol reduces the surface tension of water, changes the solubility parameters and polarity of the mixture, and thus improves fabric wettability. There was no difference in the spray tests carried out with water after many washes, while the spray tests carried out with the water/isopropanol mixture allowed us to distinguish the impact of washing on the wetting behavior of the treated fabric samples. In fact, a spray test rating of 5 was obtained before washing, and this rating declined to reach 3–2 after 10 washes, with the partial wetting of the fabric

beyond the sprayed droplets. These results can be explained by the fact that washing cycles damaged the coating on the fiber surface. The SEM analysis in Figure 16 shows the fibers of the fabric treated with PUPXC6PW: the unwashed fabric (a), the fabric washed only once (b), five times (c), and ten times (d). After ten washing cycles, the fiber coating and inter-fiber bonding are still visible, which confirms the obtained results of water spray tests. Hence, these images demonstrate also that the finishing treatments were slightly impacted during multiple washing cycles. It is shown that the surface coating on the fiber surface became less uniform. This may occur due to the high friction during laundering. These results explain the variation of the spray ratings after multiple washes with the water/isopropanol mixture.

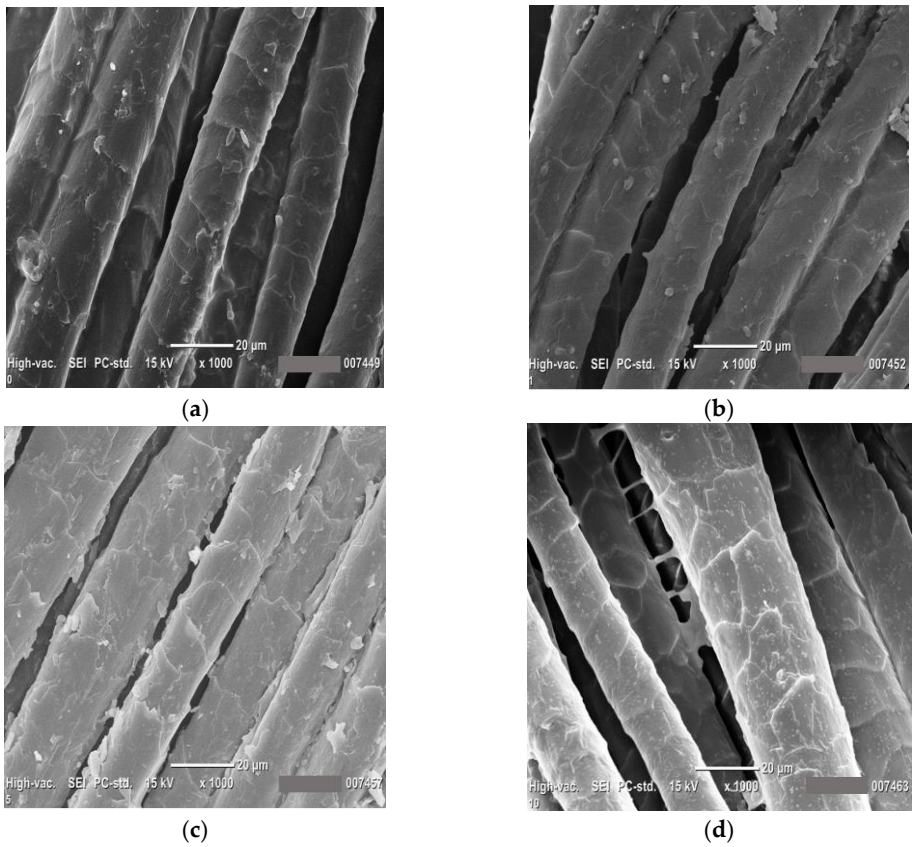


Figure 16. SEM photos of PUPXC6PW-treated samples after zero washing cycles (**a**), one washing cycle (**b**), five washing cycles (**c**), and ten washing cycles (**d**).

As described above, the treated fabric had a WCA above 135° and a WSA around 33°, while the untreated fabric has a WCA above 114° and WSA 69°.

Furthermore, combining both treatments has allowed for not only a dimensional change rate of around 2% but also an increase in the product's lifespan by improving its resistance to use and wear. The PUPXC6PW treatment has improved the abrasion resistance of the initial wool fabric. In fact, the untreated sample was damaged after 30,000 cycles and lost 30% of its initial weight, whereas in the case of the PUPXC6PW-treated fabric, only two yarns broke at 40,000 cycles with a weight loss rate of only 10% after 30,000 abrasion cycles, thus increasing the product's lifespan. However, the fabric's handling was impacted and the sample became more rigid. To improve the fabric's handling, the chemical concentrations were optimized. Several combinations showed that reducing the quantity of polyurethane and paraffin polymers decreased the main flexural rigidity.

Optimization study shows that two paddings using (1/2; 1/2) (PU; PX) and (2/3; 1/3) (C6; PW) reduced the bending rigidity to 17.67 mN/cm while maintaining good results in terms of hydrophobicity and dimensional stability.

Furthermore, the previously and recently reported results on samples of 100% wool fabrics are summarized in Table 8 and the data are comparable to our findings. The reported results in this study show acceptable shrink-resistant and water-repellent performances. Better dimensional stability results have been found with Benisek, L [2], but water-repellent results are much lower. The main advantage of the findings reported in this study is that the shrinkage resistance obtained will be easily applicable on an industrial scale. In fact, polymer treatments are simple and versatile. Additionally, the padding method has several advantages such as the lower consumption of water, electricity, and chemicals as well as higher productivity. All these results highlight the effectiveness of the two-step padding method chosen to apply both blend polymers on the fabric surface. Furthermore, the rate of weight loss after the abrasion tests allowed a subjective evaluation of durability of the treated fabrics.

Table 8. Shrink-resistant and water repellency results of previous and recent studies.

Textile	Treatment	Method	Number of Washes	Area Shrinkage (%)	Spray Test Water (10 Washes)	Weight Loss Rate after 30,000 Abrasion Cycles (%)	References
Dyed worsted wool fabric	Shrink-resistant + water repellent	Polymers	1 7A + 3 5A [1]	−2	5	10	This study
Plain weave worsted fabric	Shrink-resistant/stain and soil repellent	Polymers	1 7A + 3 5A	−9 [2]	3–4	-	[1]
Undyed serge	Shrink-resistant/flame retardant/water repellent	Polymers	1 7A + 3 5A	−1	4 (0 W) and 2 (after 20 W)	-	[2]
plain-weave wool fabric	Shrink-resistant + water and oil repellent	Polymers	1 7A + 3 5A	[−3.4; 13.1]	1 (5 W)	-	[55]
Loom state worsted wool	Shrink-resistant	Polymers	-	<−8%	-	-	[56]
Merino woven wool fabric	Shrink-resistant	Enzyme + biopolymer	1 7A + 3 5A	−6	-	-	[57]
Wool tops	Shrink-resistant	Enzyme	1 7A + 5 5A	−2.4	-	-	[50]
plain-weave pure wool fabric	Shrink-resistant	Plasma +polymer	1 7A + 5 5A	−2	-	-	[22]

[1] 7A and 5A are the washing cycles for dimensional stability. [2] A was chosen where properties are similar to the fabric used for this study.

5. Conclusions

The research work carried out here enabled the production of a machine-washable 100% worsted wool woven as an outer fabric for men's suit jackets. These results were achieved through successive paddings, with a first blend of the aqueous dispersion of polyurethane and polysiloxane (PUPX) for shrink-proofing and a second blend of a fluorotelomer methacrylate and paraffin and a hydrocarbon waxes polymer blend (C6PW) for water repellency. An improved adhesion to wool fiber surface as well as inter-fiber coating, as confirmed by the potential zeta measurements and SEM analysis, would explain better dimensional stability, the better durability of water repellency, the higher abrasion resistance, and thus, an increased lifespan. To improve fabric handling, the chemical concentrations were optimized to reduce the flexural rigidity while maintaining the durability of water repellency and washing treatments. In addition, a very slight change in air permeability and the adiathermic power was obtained, meaning that the coatings do not negatively influence the initial properties of the used wool fabric.

The main objective of this study is to eco-design a worsted wool fabric for men's suit jackets. The European Environment Agency [58] defines eco-design as a method or process that considers environmental aspects at all stages of the product development process, aiming for products with the lowest possible environmental impacts throughout their life cycle. Reducing polymer quantities will help to lower the environmental impact of the treated men's suit jackets. A study is also being conducted to analyze the life cycle of the treated fabric and the objective of the next study is to compare it with market products and propose ways to further improve the environmental impact of the end product.

Author Contributions: Conceptualization, A.B.R., C.L. and N.M.B.; data curation, A.B.R., C.L., N.M.B., C.C. and S.F.; methodology, A.B.R., C.L., C.C. and N.M.B.; validation, S.F.; writing—original draft, A.B.R.; writing—review and editing, A.B.R., C.L., N.M.B., C.C. and S.F.; data, A.B.R. and J.V.; supervision, C.C., N.M.B. and S.F. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by INDUO and ENSAIT with the contribution of ANRT.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: Not applicable.

Acknowledgments: The authors would like to express their appreciation to Miroglio (Bulgaria) for providing wool fabrics, as well as to other suppliers for providing chemical products.

Conflicts of Interest: ENSAIT declare no conflicts of interest. INDUO uses a specific formula and treatment conditions for commercial interests either in their current form or in an improved version, and it is therefore required that they remain confidential. ANRT had no role in the design of the study; in the collection, analyses, or interpretation of data; in the writing of the manuscript, and in the decision to publish the results.

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