Research article

One-pot multi-functional finishing of wool fabric using reactive nonionic softener

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ABSTRACT

Crossbred wool is widely used in the textile and clothing fields. Nevertheless, the surface roughness of crossbred wool fabric is higher than that of Merino wool. Herein we propose a reactive nonionic softener (RNS) based on fatty acid (FA) and 2-amino-2-methyl propane diol (AMPD) to impart desirable multi-functions for crossbred wool fabric, such as improved smoothness, enhanced wettability, and induced resistance to felting shrinkage. Adopting the pad-dry-cure method, treatment of wool with the said softener was carried out using different concentrations of FA/AMPD condensate at different curing temperatures and durations. The results showed that the highest fabric surface smoothness was attained when wool fabric was treated with 3.5% (o.w.f.) RNS, and the curing temperature and time were 130 °C and 5 min, respectively. The surface smoothness of the treated fabric and its resistance to felting shrinkage were increased by 21.7% and 90% respectively. The effects of treatment of wool with the RNS on its bending stiffness, air and water permeability, yellowness, electrostatic charge, and ultraviolet protection factor (UPF) were monitored. The reaction mechanism between the used RNS and wool was studied using FTIR spectroscopy. Scanning electron microscopic investigation showed a layer of the used RNS on the surface of the treated fabric. The finished fabric retained its air permeability and dyeability with anionic dye. The imparted fabric smoothness was find to be durable against washing until 5 washing cycles, and decreased by 5.5% of after 10 washing cycles respectively.

1. Introduction

The adding value preparatory processes for wool are brought about on its "top" stage by adding chemicals to the sliver to impart certain desired properties such as whiteness, luster, felt-proofing and moth-proofing (Mohammad and Christopher, 2019; Kumari et al., 2021). On the other hand functional finishes of wool, including smoothness, ultra-violet protection, antimicrobial finishing, and flame-proofing, are usually carried out within the "fabric" stage (Akioka et al., 2022; Haque et al., 2022). The reagents which are usually used industrially in textile finishing are expensive causing higher price for the final goods. The utilization of textile by-products discharged into the environment during wet processing of textiles would be of prime importance from the ecological and economic perspectives.

Wool wax is a by-product which is usually discharged to the effluent of wool scouring mills (El-Sayed et al., 2021). The amount of wool wax which can be recycled from municipal sewer around 6% of the mass of scoured wool (López–Mesas et al., 2005). The purified wool wax is called

lanolin and usually used as an additive for some cosmetics (Sengupta and Behera, 2014).Within the last three years, researchers succeeded to use lanolin, either alone or hybridized with other reagents, in textile wet processes (El-Sayed et al., 2021). By virtue of its hydrophobic nature, Khattab et al. utilized lanolin together with silicone rubber to impart enhanced durable hydrophobicity to viscose fabric (Khattab et al., 2019). Other researchers utilized lanolin as a binder in pigment printing of cotton and polyester fabrics (El-Shemy et al., 2020). Lanolin/poly-ethylene oxide condensate was used as a nonionic softener for wool fabrics with improved felting resistance (Abou Taleb and El-Sayed, 2021).

Softness of various fabric surfaces has been the subject of many previous investigations. This was accomplished using different classes of enzymes (Uddin, 2015; Ismail et al., 2022), polymers (Zaman and Zuber, 2015; Bashar and Khan, 2012) and surface etching (Hung and Kan, 2017). More than five decades ago, fabric softeners were introduced into the market to improve the fabric feel and odour. Silicon-based softeners are the most common ones used in the clothing field, presumably due to

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their superior smoothness effect (Zhou et al., 2020). Softeners are categorized mainly into cationic, anionic, and nonionic softeners depending on the nature of the functional group(s) therein (Javadi et al., 2013).

Cationic softeners are widely used to impart soft hand for cellulosic fabrics. Chemically, cationic softeners are chloride, acetate or glycolate salts of primary or tertiary amines and quaternary ammonium salts. Other cationic softeners are salts of amino amides, salts of amino esters, and salts of imidazoline (Saauddin et al., 2021).

Due to their limited substantivity towards most fabrics and their low softening action, anionic softeners are no longer widely used in textile and clothing fields. They are widely used in industrial scale as anionic surfactants rather than softener for textile fabrics. The structure of this category of softeners involves alkyl sulphonates, sulphates and ether sulphates as well as sulphated or sulphonated amides (Siddique et al., 2021).

The nonionic softeners may have the general formulae $R(OC_2H_4)_n$, OH and $R(C_2H_4)$, OOH (R = alkyl radical). This type of softeners should have non-ionic groups such as paraffins, long chain alcohols, ethoxylated fatty alcohols or amines, and oxidized polyethylene waxes as active component. This type of softeners is appropriate for wool fabrics (Wahle and Falkowski, 2010).

The effects of application of fabric softener on physico-mechanical properties of various textile substrates were studied by many researchers (El-Newashy et al., 2021). Most of the topographical properties of wool, such as surface roughness, friction coefficient, felting shrinkage, wettability, and pilling, could be affected by the applied softener (Kim et al., 2021; Argentou et al., 2022). On the other hand, studying the effect fabric softener on the comfort attributes of wool, such as water vapour and air permeability, static charge, and ultraviolet protection factors, is still stingy and needs further investigations.

Therefore, this investigation is devoted to enhance the smoothness of wool fabric surface with reactive nonionic softener (RNS) containing amino and/or hydroxyl groups. The effect of treatment of wool with the said RNS on some of its comfort characteristics was investigated. The mechanism of interaction of wool with the applied softener was proposed. Compared to previous other related investigations, the proposed softener has the advantage of being able to improve other properties of wool such as induced resistance to felting shrinkage and the enhanced wettability without any adverse effect on the fabric property and dyeability with anionic dye. This reactive nonionic softener was applied successfully to acrylic fabrics to improve their smoothness (Abou Taleb et al., 2022).

2. Experimental

2.1. Materials

Mill-scoured crossbred wool fabrics of mean fibre diameter 21.5 μm in the warp and weft directions were used in this investigation.

Purified wool wax (lanolin), extracted from raw wool fleece as prescribed elsewhere, and was used in this study (Abou Taleb and El-Sayed, 2021). The acid, iodine, and hydroxyl values of the used lanolin were 5.3, 17.0, and 77.9, respectively.

The acid dye C.I. Acid Blue 203 was supplied by BESTCHEM Hungária Kft, Budapest, Hungary. 2-amino 2-methyl propan-1,3-diol (AMPD) was provided by Sigma-Aldrich, Germany.

2.2. Methods

2.2.1. Preparation of nonionic softener

A reactive nonionic softener (RNS) was synthesized by reaction of a fatty acid (FA), extracted from lanolin, with 2-amino-2-methyl propan-1,3-diol (AMPD).The reaction between the said reagents was conducted on equi-molar basis assuming that the major fatty acid in lanolin is stearic acid (Abou Taleb et al., 2022).

2.2.2. Treatment of wool with nonionic softener

The prepared nonionic softener was pre-dissolved in 10 mL absolute ethyl alcohol, and the solution was completed to 100 mL by distilled water. Wool fabric was treated with 1.0, 2.0, 3.5, and 5.0% (o.w.f.) Lanolin/AMPD condensate. The fabric was immersed in the synthesized nonionic softener bath for 5 min, padded between two mangles of a padder to a wet pick up of 100%, dried at 80 °C for 15 min, and finally cured at 120–150 °C for 2–5 min.

2.2.3. Dyeing

The treated and untreated wool fabrics were dyed in an aqueous solution containing C.I. Acid Blue 203 (1.0% shade) for 1 h at 90 °C and pH 4.5 (adjusted by acetic acid). The material-to-liquor ratio (MLR) was 1:50 (Allam et al., 2022).

The percent of dye exhaustion was determined by measuring the absorbance of the residual dye within the dyeing bath on a JENWAY-6405 UV/Vis spectrophotometer (Bibby Scientific Ltd, UK). The dye exhaustion from the dyeing bath was determined at λ_{max}575 nm according to the following equation (El-Sayed and El-Shemy, 2021).

$$\text{Dye exhaustion\%} = [(A_1 - A_2)/A_1] \times 100$$

where "A_1" and "A_2" are the absorbance of dye bath before and after dyeing.

2.3. Analyses and testing

The FTIR spectra of the treated as well as treated wool fabrics were recorded using in FTIR spectrometer JASCO FTIR 4700 equipped with an optical system that provides data in the range of 4000–400 cm^{-1}.

The fibre morphology of the treated as well as untreated wool fibres was monitored by scanning electron microscopy using Bruker Nano GmbH Scanning Electron Microscope D-12489 Berlin, Germany. The samples were mounted on aluminium stubs, and sputter coated with chromium in an S150A with 20 kV scanning voltage.

The percentage add-on was evaluated by determining the difference in the sample weight before and after treatment. The test was carried out in triplicate for each sample using the following equation:

$$\text{Add-one (\%)} = [(W_2 - W_1)/W_1] \times 100$$

where W_1 and W_2 are the dry weights of the untreated and treated samples respectively.

The fabric surface smoothness was determined in accordance with the BS 3424 standard test method using Shirley fabric friction tester.

The felt-area shrinkage of wool fabrics was assessed according to the IWSTM-31 standard method (Wascator).

The electrical conductivity of the fabrics was assessed on an LRC-bridge (Hioki model 3531 zHi Tester, Japan) at frequency range 100–100,000 Hz.

The UV protection factor (UPF) of the modified and unmodified samples was monitored according to the AATCC Test Method 183:2010-UVA Transmittance using a UV/Vis spectrophotometer.

The fabric wettability was assessed in terms of drop disappearance, measured by allowing a drop of water to fall on the sample and recording the time required for drop disappearance (AATCC standard test method).

The bending stiffness was determined according to the standard test method ASTM D1388-2018.

Air permeability was measured on FX 3300 air permeability tester (TEXTEST AG, Switzerland) at a pressure of 100 Pa according to ASTM D737 standard method.

The water permeability was assessed according to ASTM–D 1913 (American Test Method for Water Repellency; Water Spray Test new edition 2010).

The degree of yellowness was performed on Datacolor Colorimeter 3980 (Datacolor Marl). Each value is an average of five measures determined on different positions on the examined sample.
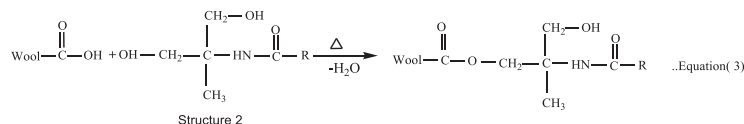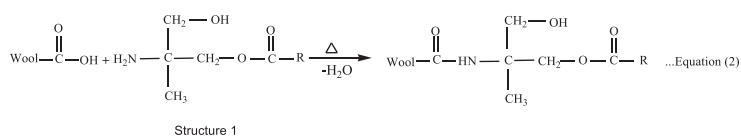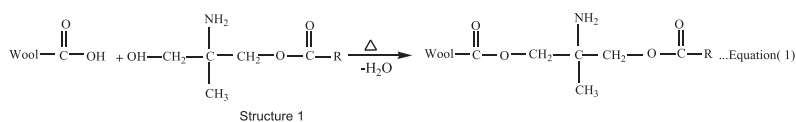
To examine the durability of the treated fabrics, the finished specimens were washed for 1, 5, 10, and 20 washing cycles in accordance with the standard method AATCC 61-1989.The fabric surface smoothness of the washed samples was taken as an indicator of the durability of the proposed finish.

3. Results and discussion

Esterification and amidification reactions are widely used in synthesis of various chemical and pharmaceutical products. The amide and ester bonds represent the backbone of many natural and synthetic polymers (Fattahi et al., 2018). The approved possible structures of the used RNS, shown hereafter, could be suitable candidates for esterification and amidification reactions. These structures were proposed on the basis of FTIR and ^{13}C-NMR analyses (Abou Taleb et al., 2022). This softener has hydroxyl (structure 1) and or amino (structure 2) groups which are the active binding niches for reaction with wool keratin macromolecules.

Figure 1. FTIR spectra of untreated and RNS-treated wool fabric.

Structure 1
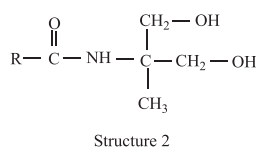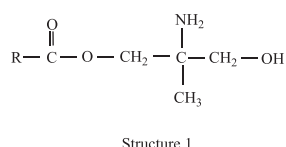
Structure 2

Through their amino or hydroxyl group, both structures 1 and 2 have the potential to covalently bind to wool via esterification or amidification reaction on the carboxylic groups along keratin macromolecules (see Eqs. (1), (2), and (3)).

1630 cm^{-1}, 1518 cm^{-1}, and 1236 cm^{-1} refer to the amide I, amide II, and amide III, respectively which are characteristics peaks for wool (El-Sayed and El-Hawary, 2021). The FTIR of the wool fabrics treated with the synthesized RNS showed a sharp strong band at 1735 cm^{-1} which is specific to the stretching vibration of the carbonyl group in esters. This

Structure 1

Structure 1

Structure 2

The proposed mechanism for the reaction of wool and the synthesized RNS was confirmed by FTIR investigation. Figure 1 shows the FTIR spectra of untreated as well as RNS-treated wool fabrics. In the FTIR spectra of untreated wool, the distinguishing bands beyond 3200 cm^{-1} are associated with the stretching vibration of the amino and hydroxyl groups along the keratin macromolecules of wool. The bands around
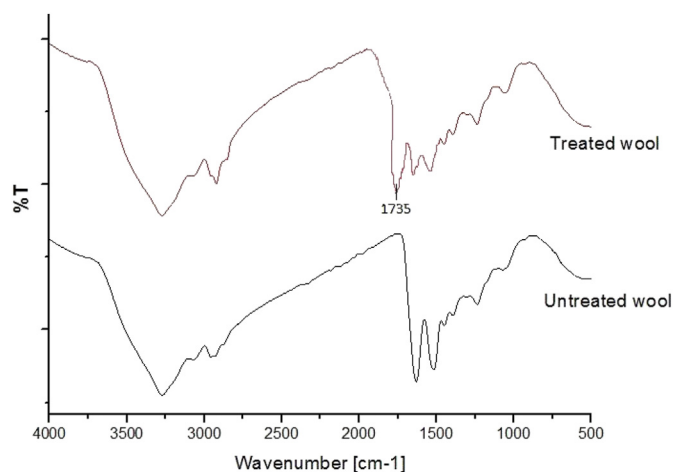
emphasized our hypothesis that the synthesized RNS bound to wool through an esterification reaction between the carboxylic groups of glutamic and aspartic acids residues in wool keratin macromolecules and a hydroxyl group in the synthesized RNS as shown in Eq. (3). Nevertheless, the synthesized RNS can bind to wool keratin through an amidification reaction according to Eq. (2).

3.1. Process optimization

Treatment of wool with FA/AMPD condensate was conducted using different softener concentration as well as different treatment temperatures and durations. The optimum reaction conditions were assigned according to the fabric surface smoothness as well as the add-on percent. Results of this investigation, summarized in Tables 1, 2, and 3, imply that the highest fabric surface smoothness was attained upon treatment of wool fabric with 3.5% (o.w.f.) FA/AMPD condensate, and the curing temperature and time were 130 °C for 5 min, respectively. Although curing of the treated fabric at 150 °C led to slight improvement in the fabric surface smoothness, yet it caused fabric yellowing. Similar trend was observed upon curing the treated fabric at 150 °C for more than 5 min. Fabric yellowness is undesirable and lessens the final product's quality (Hassan, 2018).

Results indicate also that the weight of wool samples increased upon treatment with the synthesized RNS, and the extent of increment was a function of the concentration of the used RNS, the curing temperature, and the curing time. The maximum add-on was attained upon treatment of wool with 5% (o.w.f.) of the RNS and the curing was conducted for 5 min at 150 °C. Yellowing of the treated wool at this temperature for 5 min favoured the use of lower temperature and time during curing of the treated samples.

3.2. Performance characteristics

The effects of treatment of wool with FA/AMPD condensate on some of its physical properties were assessed and the results were shown in Table 4. The data in this table indicate that the wettability of the treated fabrics was highly improved to the extent that the drop of water spreads instantaneously on the surface of the treated fabric, while it takes more than 30 min without spreading on the untreated fabric surface. This finding indicates that the applied FA/AMPD condensate imparts two important functions to wool fabric which are the enhanced smoothness and wettability. The enhanced wettability of the treated wool fabric could be explained in terms of the induction of additional polar –OH or –NH$_2$groups within the chemical structure of the RNS-treated samples (*c.f.* Eqs. (1), (2), and (3)).

The degree of yellowness of the treated sample was higher than that of the untreated one by 5.24% which is acceptable from the appearance point of view. The fabric drape was highly improved as indicated by the remarkable increase in the bending length of the treated sample relative to its corresponding untreated one. The enhanced fabric drapability is of prime importance in improving the aesthetic properties of the textile product (El-Newashy and El-Sayed, 2022).

The untreated and treated wool fabrics have very good ultraviolet protection (>15%). The treated wool fabric had *ca.* 8% UPF value lower than that of the untreated fabric. This can be attributed to the enhanced smoothness of the treated fabric surface. It has been reported that the smoother fabric surface has lower ability to scatter the ultraviolet radiation; thus, the UV radiation will have higher ability to transmit through the fabric, which decreases its UPF (Rietzler et al., 2019). The negative impact of the fabric smoothness on the UPF value of the treated fabric was counteracted by the increase in the fabric yellowness. This assumption is in harmony with previous work reported by Grifoni et al. in which they concluded that deeper colours enhance remarkably the fabric UPF (Grifoni et al., 2009).

Treatment of wool fabric with FA/AMPD condensate highly reduced its felt-area shrinkage to 4.8% compared to 44.7% for the untreated sample. This could be attributed to masking of the scales on wool fibre surface by an adhered layer from the used RNS. It has been agreed that the scaly structure of wool fibre surface is the reason of its felting shrinkage during mechanical agitation (Mohammad and Christopher, 2019; El-Sayed, 2021); thus, controlled removal of this scales using any degradative method, or covering these scales with polymeric materials would result in woolen products with enough resistance to shrinkage (Gupta and Natarajan, 2017; Kantouch et al., 2011).

3.3. Fabric comfortability

From the factors which affects the comfort characteristics of clothes are their air and water permeability as well as their ability to

Table 1. Effect of treatment of wool fabrics with different concentrations of FA/AMPD on their surface smoothness (curing time: 3 min and curing temperature: 130 °C).

Concentration of FA/AMPD condensate (%, o.w.f.)	Smoothness (°)[a,b]	Add-on (%)
-	46[c]	-
1.0	44[d]	0.65
2.0	43[e]	1.10
3.5	40[f]	1.69
5.0	40[g]	1.77

[a] The smaller smoothness angle, the smoother will be the fabric surface.
[b] Average of 7 measurements in 7 places in the inspected sample.
[c] Standard deviation (SD) 1.18,
[d] SD1.93,
[e] SD 1.23,
[f] SD1.90,
[g] SD 1.42.

Table 2. Effect of curing temperature of wool fabrics treated with FA/AMPD on their surface smoothness (FA/AMPD conc. 3.5% o.w.f., and curing time 3 min).

Curing temperature (°C)	Smoothness (°)	Add-on (%)
Untreated	46[a]	-
120	41[b]	1.43
130	39[c]	1.69
140	39[d]	1.70
150	38[e]	1.78

[a] SD 1.25,
[b] SD1.87,
[c] SD 1.07,
[d] SD1.97,
[e] SD 1.35.

Table 3. Effect of curing time of wool fabrics treated with FA/AMPD on their surface smoothness (FA/AMPD conc. 3.5% o.w.f., curing temperature 130 °C).

Curing time (°C)	Smoothness (°)	Add-on (%)
Untreated	46[a]	-
2	41[b]	1.25
3	39[c]	1.69
4	38[d]	1.73
5	36[e]	1.81

[a] SD 1.40,
[b] SD1.31,
[c] SD 1.29,
[d] SD1.14,
[e] SD 1.22.

Table 4. Effect of treatment of wool with FA/AMPD condensate on some of its physical properties (FA/AMPD conc. 3.5%; w/v, curing time: 5 min and curing temperature: 130 °C).

Property	Untreated fabric	Treated fabric
Wettability (S)	Up to 30 min	0
Yellowing index	26.68	28.08
Bending length (cm)	2.4	3.4
Ultraviolet protection factor (UPF)	30.9	28.4
Felt-area shrinkage (%) after 5 washing cycle	44.7	4.8

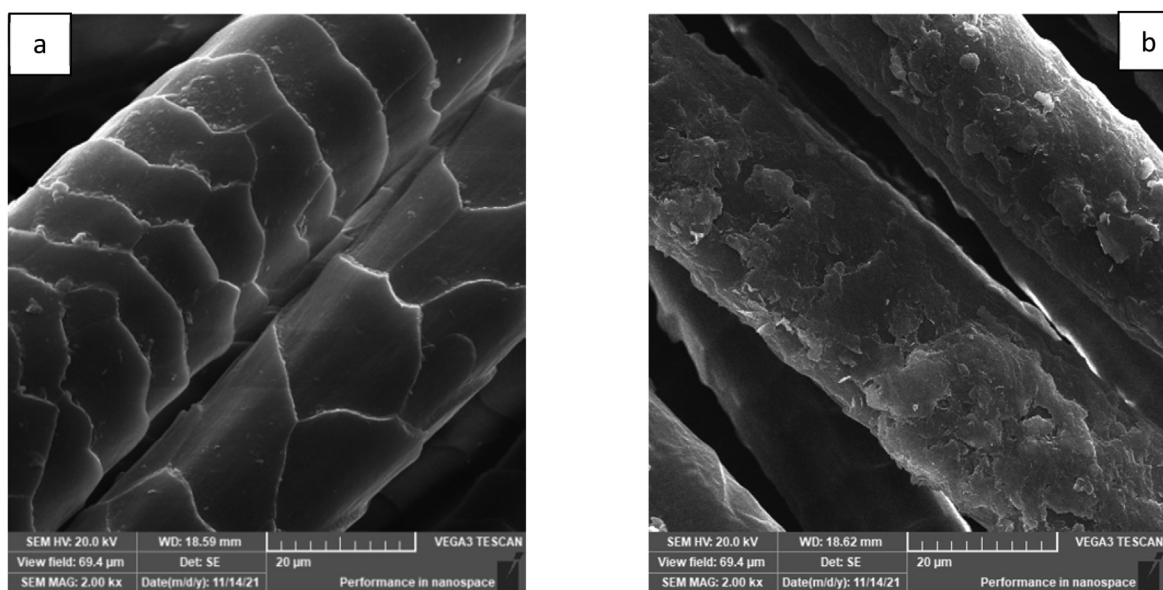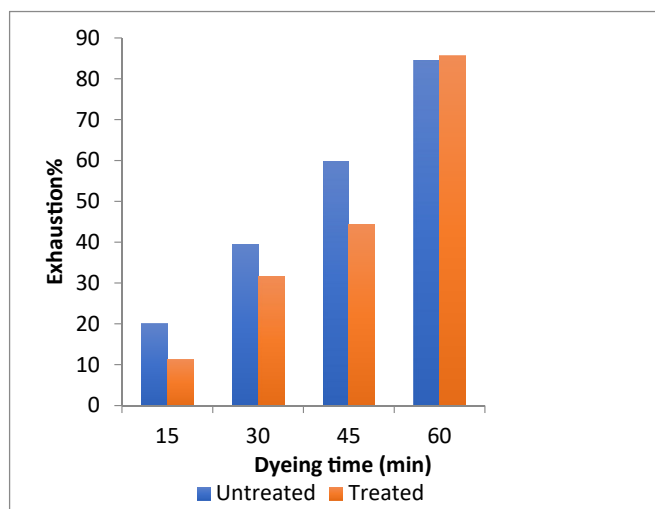**Table 5.** Comfort attributes of wool fabrics treated with FA/AMPD condensate.

Comfort attribute	Untreated fabric	Treated fabric
Air permeability (cm^3/cm^2. S)	64.87	64.59
Water permeability (L/sec)	0.787	0.901
Dielectric const. at 100 Hz (ε')	4.52	5.82
Dielectric loss at 100Hz (ε'')	0.081	0.227
Electrical cond.σ_{dc} (S/cm) or Ω^{-1} cm^{-1}	0.38×10^{-11}	1.08×10^{-11}

diminish the accumulation of electrostatic charge on its surface. Table 5 abridges the effects of treatment of wool fabric with FA/AMPD condensate on its water and air permeability, and surface static charge. The data in this table reveals that there is a limited decrease in air permeability of the treated fabric when compared to the untreated sample. The water permeability was increased by about 20.6%, relative to the untreated sample. These findings indicate that treatment of wool with the said RNS led to significant increase in the fabric pore size.

The electrical conductivity of the treated wool fabric was significantly enhanced relative to the untreated one, indicating a decrease in the accumulated electrostatic charges on the fabric surface. This would be attributed to the creation of additional hydrophilic groups along the keratin macromolecules of the treated fabric; Viz. the polar hydroxyl and/or amino groups, which can liberate any accumulated electrostatic charge.

3.4. Fibre morphology

Figure 2 shows the scanning electron micrographs of the untreated as well as the finished wool fabric. While the scaly structure of untreated wool fibre surface is obvious in Figure 2-a, there is a clear layer from the applied RNS adhered on the fibre surface of the treated fabric (Figure 2-b). This layer is responsible for the smoothness effect imparted to wool fabric treated with FA/AMPD condensate. Furthermore, this layer led to masking of the scales on the fibre surface imparting high resistance to felting shrinkage (*c.f.* section 3.2). It has been agreed by many authors that the additive method of shrink-proofing is more favorable than the subtractive

Figure 3. Dye exhaustion of wool fabric dyed with C.I. Acid Blue 203 at λ_{max} 575 nm.

methods using chemicals or enzymes (Mohammad and Christopher, 2019; El-Sayed, 2021).

3.5. Dyeing

Dyeing of textiles is of prime importance as it highly influences the appearance attributes of the final product. Therefore, it was necessary to study the effect of the applied softening agent on the dyeability of wool fabric using C.I. Acid Blue 203 and the results were presented in Figure 3.

Thorough inspection of Figure 2 elucidates that at a short dyeing time up to 45 min, the dye exhaustion of the untreated fabric was significantly higher than that of the treated fabric. This may be explained in terms formation of new bonds between wool keratin macromolecules and the used FA/AMPD condensate; thus, the mobility of polypeptide chains of wool decreased and the penetration of dye molecules into the fibre interior got slower. Prolonged heating of wool within the dyeing bath for

Figure 2. Scanning electron micrographs of (a) untreated wool, (b) treated wool.

Table 6. Surface smoothness of wool fabric treated with FA/AMPD.

Number of washing cycles	Smoothness (°)
0	36
1	36
5	36
10	38
20	40

60 min resulted in an increase in the mobility of the macromolecular chains of wool keratin affording more accessible pathways through which the dye molecules can stab faster (El-Sayed, 2006).

3.6. Durability

The durability of the treated fabric against washing in launderometer was assessed after up to 20 washing cycles. The results of this investigation, shown in Table 6, indicate that the finished fabric is durable against washing up to 20 wash cycles as indicated by the degree of smoothness of the washed fabric surface. This could be attributed to the formation of covalent bonds between wool keratin macromolecules and FA/AMPD condensate which are able to withstand the effect of washing.

4. Conclusion

The fatty acid(FA)/2-amino-2-methyl propan-1,3-diol (AMPD) condensate was found to be an appropriate reactive nonionic softener (RNS) for wool fabric using pad-dry-cure technique. The finished wool fabric exhibited new multi-functions including smoother surface, shrink-resistance, enhanced wettability, and improved drapeability. Through its hydroxyl and/or amino groups, the FA/AMPD condensate was able to form covalent bond with the carboxylic groups within wool keratin macromolecules through ester or amide linkages, respectively. The finished wool fabrics retain the imparted smoothness even after 20 washing cycles. The applied RNS caused slight increase in the fabric yellowness together with small decrease in the ultraviolet protection factor.

The water permeability and the resistance to static charge of the finished fabrics were improved; whereas the air permeability of the treated fabric was not significantly affected. The dyeability of the treated fabric towards anionic dye is comparable to that of the untreated one especially upon dyeing at the boil for at least 60 min.

Declarations

Author contribution statement

H. El-Sayed: Conceived and designed the experiments; Wrote the paper.

S. Mowafi, A. S. Basuoni: Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data.

Data availability statement

Data included in article/supplementary material/referenced in article.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

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