Heliyon 8 (2022) e09741

Contents lists available at ScienceDirect

Heliyon

journal homepage: www.cell.com/heliyon

Research article

Preparation of fatty acid-amino diol condensate and its utilization as a durable nonionic softener for PAN fabrics

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ARTICLE INFO

Keywords:
PAN fabric
Softener
Smoothness
Electrostatic charge
Hydrophilic
ANOVA

ABSTRACT

Polyacrylonitrile (PAN) fabrics meet the customers' requirements in many aspects. Nevertheless, PAN fabrics suffer from low moisture regain and the accumulation of electrostatic charges on the fabric surface. Some PAN products exhibit rough a surface which is uncomfortable for human skin. Herein, we synthesized a new hydrophilic nonionic softener by reacting a fatty acid (FA), extracted from wool wax, with 2-amino-2-methyl-1,3-propanediol (AMPD). Adopting the pad-dry-cure technique, the synthesized softener was chemically bound to pretreated PAN fabrics. Without deterioration of the fabrics' mechanical properties, new functions have been imparted to the treated PAN fabrics, viz., silk-like hand, induced resistance to the accumulation of electrostatic charges, and improved wettability. Fourier-transform infrared (FTIR) spectroscopy and carbon-13 nuclear magnetic resonance (^{13}C-NMR) were utilized for the structure elucidation of the prepared softener as well as to determine whether AMPD reacts with the fatty acid through its amino or hydroxyl group. The mechanism of the preparation of the softener as well as its mode of action on PAN fabrics were proposed. The effects of the applied softener on air and water permeability, ultraviolet protection factor (UPF), stiffness, tensile properties, and yellowness of the treated fabric were studied. The scanning electron micrographs of the treated fabric revealed the existence of a layer of the applied softener on the fabric surface. The finished fabric was found to be durable against washing for up to 20 cycles in terms of the fabric smoothness and durability.

1. Introduction

Since the fourth quarter of the previous century, production of synthetic fibers has surpassed natural fibers in the textile sector. Among the other synthetic materials used in the textile and clothing fields, polyacrylonitrile (PAN) fibers exist in 3rd place after polyester and polyamide in market share [1]. Due to their relatively low cost, a wide range of PAN products are utilized in different fields, viz., water purification and antimicrobial products. PAN fabrics merge some desirable features such as chemical tolerance, outstanding elasticity, and aesthetical properties [2]. Moreover, PAN has good resistance to chemicals and corrosive materials, and can retain its tensile strength on repeated use. These properties would allow PAN fibres to replace polyester fibres in some applications [3]. However, most of the synthetic fibres, like PAN fibres, have the disadvantages of being easily caught by fire, having low moisture regain, and being susceptible to accumulation of electrostatic charges [4]. Accordingly, extensive research work has been directed towards improving the performance, comfort, and appearance attributes of man-made fibres [5, 6, 7, 8].

Improving the moisture regain of PAN fabrics was the subject of many investigations. Yang *et al.* used L-cysteine as a coupling agent for covalent bonding of casein or collagen into PAN fibres [9]. The treated fibres exhibited better hygroscopic and mechanical properties. The same goals have been achieved by other methods, including bio-treatment of PAN fibres with nitrile hydratase enzyme [10], grafting with synthetic [11] and natural polymers [12].

Many auxiliaries are used in textile wet processes, which are prepared from textile by-products and waste. Utilization of the by-products produced during processing of textile materials is of prime importance from the technical, ecological, and economic points of views [13, 14, 15]. Lanolin is a greasy material prepared by the purification of wool wax, which is usually extracted from the scouring effluent of raw wool fleece [16]. The discharge of wool wax into the drainage water leads to environmental problems coupled with a loss of possible profits. Within the last few years, purified wool wax has been proposed as a suitable candidate for some textile applications, including hydrophobizer for viscose fabrics [17], binder in pigment printing of cellulosic and synthetic fibres [18], and softener for wool fabric [19].

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https://doi.org/10.1016/j.heliyon.2022.e09741
Received 22 March 2022; Received in revised form 1 May 2022; Accepted 14 June 2022

Wool wax is the greasy secretion of the sebaceous glands of sheep that coats the wool fibers. It has a complex structure in which esters of fatty acids and long-chain aliphatic alcohols or sterols, are the major constituents. Additionally, few fatty acids exist in their free form. Metal salts of fatty acids were detected in wool wax, and the calcium soaps are particularly important [20].

Among the textile auxiliaries which are usually used in the functionalization of textile fabrics are the softening agents. Softeners are widely used to enhance the comfort attributes of textile products. According to the nature of the constituents of the softeners, they are classified into three types. The nonionic softeners, which are usually based on ethoxylates, silicones, esters, or polyethylenes; the cationic softeners, which are usually quaternary ammonium salts; and the anionic softeners, which are sulphates, sulphonates, and sulphosuccinates [21]. An emulsifying agent is usually added with the nonionic softeners to produce stable dispersions.

The nonionic softener encounters some difficulties if applied to acrylic fibres. For instance, when a fatty acid ethoxylate is used on acrylic fibres, the molecules of the softener align themselves so that high fibre-fibre static friction will be generated. Furthermore, if this type of softener is adopted for a dyed fabric with loosely adhering dye molecules on its surface, the colour may be quickly faded together with bad wet and rubbing fastness properties [22].

The present work aims at the synthesis of a new reactive nonionic softener based on fatty acids extracted from purified wool wax. The synthesized softener will be utilized for multi-functionalization of PAN fabrics to improve their hand and wettability as well as to induce resistance to the accumulation of electrostatic charges on the fabric surface. The utilization of waste materials or by-products in synthesis of textile auxiliary makes the proposed nonionic softener advantageous ecologically and economically. From the technical point of view, the application of reactive nonionic softeners would assure the formation of a permanent durable covalent bond with the treated fabrics, and impart more than one desirable function to the treated fabric. One-way analysis of variance (ANOVA) between different physical and mechanical properties has been applied.

2. Experimental

2.1. Materials

Plain weave (1/1) polyacrylonitrile (PAN) fabrics of medium weight (155 g/m^2) and thickness of 0.4 mm were used in this investigation. For 30 min at 60 °C, the PAN fabric was washed with 2% (o.w.f.) Egyptol PLM® (a nonionic detergent based on nonanoyl phenol ethoxylate). The fabrics were then rinsed thoroughly with running water and air-dried. The fabrics were then rinsed thoroughly with running water and air-dried.

Lanolin was extracted from raw wool fleece according to the method described elsewhere [23]. It is a yellowish brown viscous material with acid, iodine, and saponification values of 5.9, 16.8, and 89.2, respectively.

2-amino-2-methyl-1,3-propanediol (AMPD) was purchased from Sigma-Aldrich, Germany. Sodium hydroxide was supplied by ADWIC, Cairo, Egypt.

2.2. Methods

2.2.1. Extraction of fatty acids from lanolin

The method of hydrolysis of lanolin to obtain fatty acids was conducted as described in our previous work [19].

2.2.2. Synthesis of fatty acid/AMPD softener

Fatty acid (FA)/AMPD softener was prepared by combining equimolar amounts of FA (assuming that it is C_{16} FA) and AMPD, and heating for 2 h at 180 °C in an oil bath with stirring (500 rpm) [24]. The homogenous viscous sticky material was allowed to cool at room temperature.

2.2.3. Treatment of PAN fabrics with the synthesized softener

2.2.3.1. Alkaline hydrolysis of PAN fabric. About 50 g of PAN fabric was soaked in 350 mL of an aqueous solution of 2.5 M NaOH with gentle shaking at 80 °C for 10 min. The hydrolyzed fabric was rinsed thoroughly with distilled water until neutrality, and finally dried at 70 °C till constant weight.

2.2.3.2. Treatment with FA/AMPD softener. The synthesized softener was dissolved in 10 mL of absolute ethanol, and the solution was completed to 100 mL using distilled water. The pretreated PAN fabric was treated with different concentrations of FA/AMPD softener. The fabrics were impregnated for 5 min in the bath, padded between two mangles to a wet pick up of 80%, then dried at 80 °C for 15 min, and finally cured at 150 °C for 4 min.

2.3. Analyses and testing

2.3.1. Structure elucidation

The chemical structure of the extracted fatty acids as well as their softener with 2-amino-2-methyl-1,3-propanediol was investigated using FTIR and ^{13}C NMR. The FTIR spectra were recorded by the FTIR spectrometer JASCO FTIR 4700 with an optical system that conducts data collection over a total range of 4000–400 cm^{-1} with the best resolution of 0.5 cm^{-1}. ^{13}C NMR spectra of the prepared softener were obtained in DMSO-d_6 on a Burker Ascend 400 MHz NMR spectrometer (Burker-Avance III, Fallanden, Switzerland).

2.3.2. Fabric characteristics

The fabric softness was determined using Shirley fabric friction according to the BS 3424 standard test method.

The UV protection factor (*UPF*) values were assessed according to the AATCC Test Method 183:2010-UVA Transmittance using a JASCO V-750 spectrophotometer.

The electrical conductivity of the treated and untreated fabrics was measured using an LRC-bridge (Hioki model 3531zHi Tester, Japan) at a frequency range of 100–100000 Hz.

The degree of yellowness was performed on a Datacolor Colorimeter 3980 (Datacolor Marl). Each value is an average of five measures determined at different positions in the examined sample.

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

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 (the standard test method AATCC 79–2018).

Air permeability was measured on the 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 tensile strength and elongation at break of untreated and some treated PAN fabrics were assessed according to ASTM D 76 standard method using Instron Textile Tester (USA).

2.3.3. Durability test

Adopting the standard AATCC 61–1989 method, the washing durability of the finished fabrics was examined. The treated specimens were washed for 1, 5, 10, and 20 wash cycles. The treated fabric (5 × 15 cm) was mounted in a launder-o-meter with a detergent solution (200 mL) at 40 °C for 45 min. The fabric smoothness and wettability of the washed samples were assessed to indicate the washing durability of the finished PAN fabric.

2.3.4. Fibre morphology

The alteration in the surface structure of the treated fabrics was recorded by scanning electron microscopy using JSM 6360LV, a JEOL

Scanning Electron Microscope (Japan). The samples were mounted on aluminium stubs, and chromium sputtered coated in an S150A with 20 kV scanning voltage. The energy dispersive X-ray spectroscopy (EDX) measurements were reported at 20 kV accelerating voltage and a 15 mm working distance.

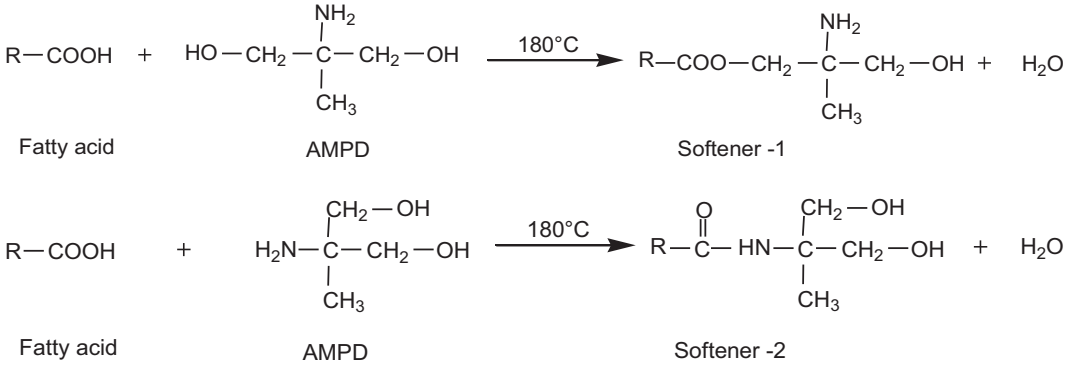
2.3.5. One-way ANOVA analysis

The relationship between the different physical properties as well as the comfort attributes of the treated PAN fabrics was statistically analyzed. The significance of this relationship was investigated using regression analysis and the R^2 value.

3. Results and discussion

3.1. Synthesis of nonionic softener

The reaction of FA with AMPD resulted in the formation of a yellow creamy sticky very viscous softener. The possible mechanism of reaction of AMPD with FA, as shown in equations 1 & 2, comprises either esterification of the hydroxyl group of AMPD with the carboxylic group of FA or amidifcation of the amino group of the former with the carboxylic group of the latter. Nevertheless, the esterification reaction remains the most probable reaction, as it is much easier than the amidification one.



carboxylic group of fatty acid reacted with the hydroxyl and amino groups of amino-2-methyl-1,3-propanediol (AMPD), as evidenced by the formation of ester and amide groups at 1736 cm^{-1} and 1661 cm^{-1}, respectively.

Figures 2 and 3 show the ^{13}C-NMR chemical shifts of the extracted FA and its reaction product with AMPD, respectively. The ^{13}C nucleus with I = 1/2 is an important tool in the structure elucidation of lipids and fatty acids by virtue of its large chemical shift range (*ca.*200 ppm) compared to that of ^{1}H (*ca.*10 ppm) [27]. Generally, four regions are usually encountered in the ^{13}C-NMR chemical shifts of lipids: the carbonyl and carboxyl carbons within the range of 172–178 ppm; unsaturated carbons of 124–134 ppm; carbon atoms of the methyl ester at *ca.* 50 ppm; and aliphatic carbon atoms at 10–35 ppm [28].

The carboxyl carbon atoms of the free fatty acids appear in a narrow but distinct region of ~175 ppm, see Figure 2. The resonances caused a shift of this peak into a higher frequency with respect to the esterified derivatives by ~4 ppm (~169 ppm). Furthermore, in Figure 3, an additional peak appears at *ca.* 68 of the C–O of propandiol moiety, which assures the reaction between the extracted fatty acids and amino-2-methyl-1,3-propanediol.

Based on the above findings, we conclude that AMPD was successfully reacted with the extracted fatty acid to form FA/AMPD softener. This reaction is primarily an esterification reaction between the carboxylic group of FA and the hydroxyl group of AMPD. However, the

To assign which mechanism was taken place between FA and AMPD, we underwent FTIR and ^{13}C-NMR to the prepared softener.

Figure 1 illustrates the FTIR spectra of the extracted FA, AMPD, and FA/AMPD softener. The spectrum of the FA shows a broad band at 3298 cm^{-1}, which belongs to the stretching vibration of –OH group, a band at 2916-2850 cm^{-1} which corresponds to the aliphatic methylene (–CH_2–) group, and a stretching vibration band at 1704 cm^{-1} for the carbonyl group (C=O) of the carboxylic acid. In the FTIR spectrum of AMPD, there is a band at 3325–3190 cm^{-1} with a shoulder peak therein, which is attributed to the stretching vibrations of both amino and hydroxyl groups characteristic (–NH_2 and –OH groups). The stretching vibration band of the aliphatic methylene group appears at 2903-2842 cm^{-1}, and the NH bending vibration band at 1615 cm^{-1} [25,26].

The FTIR spectrum of FA/AMPD softener has a broad band at 3284 cm^{-1} which corresponds to the stretching vibration of the –NH– of the amide linkage. The aliphatic methylene group appears at 2916–2850 cm^{-1}, the stretching vibration of the carbonyl group of ester (C=O) appears at 1736 cm^{-1}, a weaker band appears at 1661 cm^{-1} for the (C=O) stretching vibration of the amide linkage, and a weak band at 1577 cm^{-1}, which is characteristic of the NH bending vibration of the amide group. The

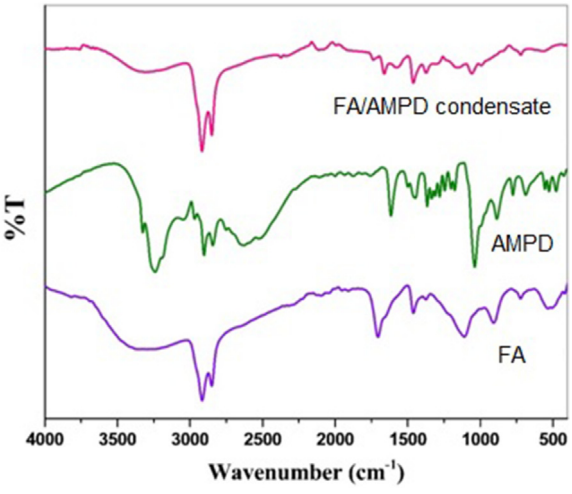


Figure 1. FTIR spectra of FA, AMPD, and FA/AMPD softener.

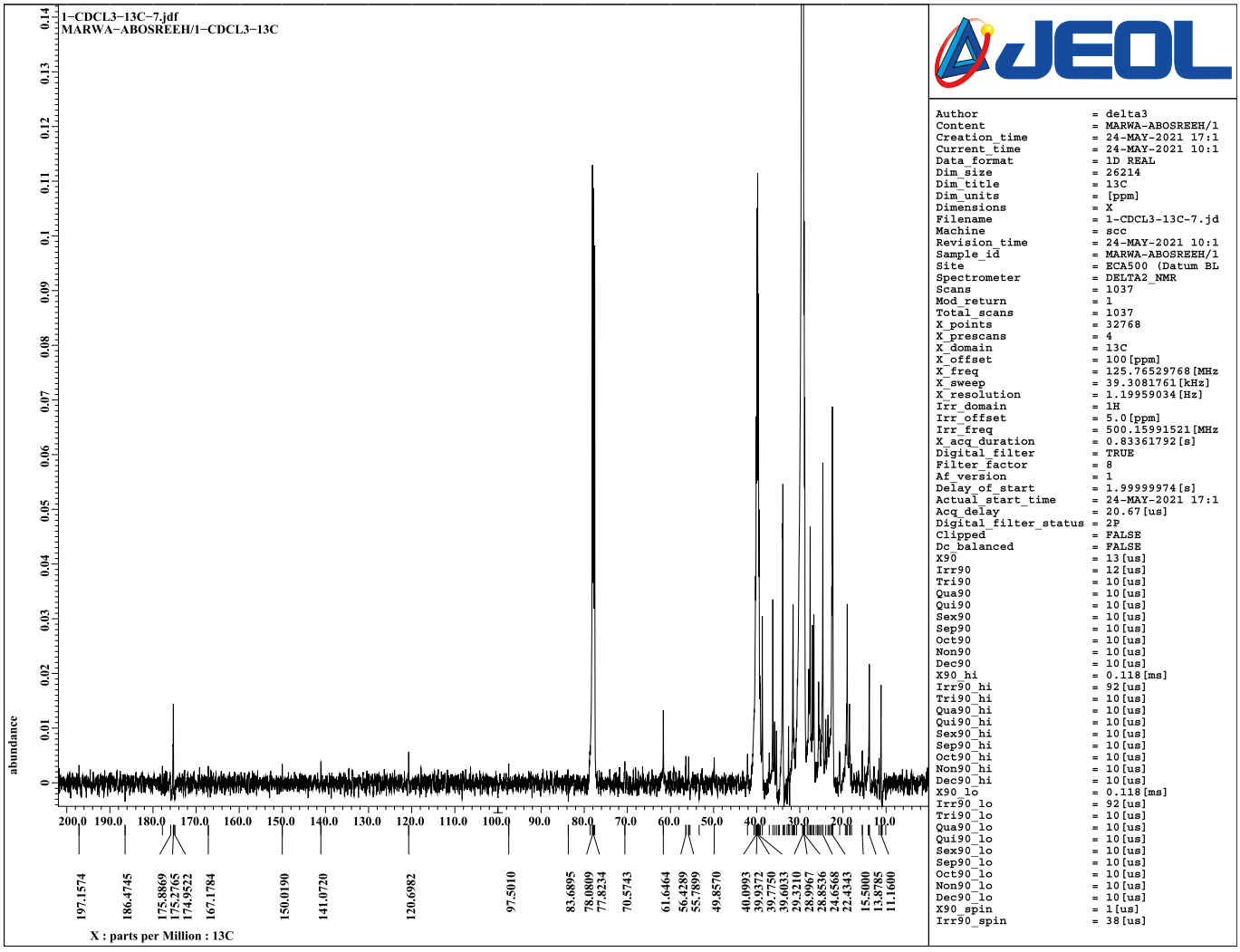


Figure 2. ^{13}C-NMR spectra of free fatty acid.

FTIR spectrum of the prepared softener also reveals that an amide link was formed between the carboxylic group of the FA and the amino group of AMPD.

3.2. Fabric properties

The effects of treatment of PAN fabrics with different concentrations of the prepared nonionic softener on some of its properties were assessed, and the results are summarized in Table 1.

The data in this table indicates that the synthesized nonionic softener has a smoothing action on the treated PAN fabric. When the concentration of the synthesized softener increased from 2 to 3.5 %, the surface smoothness increased (as indicated by the decrease in the fabric smoothness). Further increases in the softener concentration from 3.5 to 5% have limited improvement in the fabric surface smoothness.

The results shown in Table 1 also indicate that there is an increase in the UPF of the treated PAN fabric compared to the untreated sample, but not to the acceptable limit. The increase in the UPF of the treated sample may be attributed to two factors. Firstly, the increase in the wetness of the treated PAN fibres led to fibre swelling, which in turn decreased the fabric porosity; and thus the UPF increased [29]. The second factor, which is the yellow tint exhibited by the treated fabrics, is more important. It has been reported that coloration of white fabrics results in a significant increase in the UPF [30].

Compared to the untreated PAN fabric, there is a limited discrepancy in the tensile strength and elongation at break of the hydrolyzed as well as the treated samples. The maximum loss in the tensile strength was 2.4% and the highest increase in the elongation at break was approximately 4.6%.

The wettability of the treated fabrics' surfaces was highly improved by virtue of the induced hydrophilic functional groups (amino and/or hydroxyl groups). Treatment of PAN fabrics had a negative effect on their degree of yellowness, presumably under the influence of the high temperature used during curing of the treated fabrics. The bending stiffness of the treated fabric is close to that of the untreated one. This indicates that the applied non-ionic softener layer on the treated PAN fibres is not stiff and would not affect the fabric drape.

Results of the statistical analysis, shown in Table 2, indicate that there is a strong correlation between the yellowing index and the UPF, as well as between the wettability and tensile strength. On the other hand, there is a strong inverse relationship between wettability and UPF. These findings are of strong significance, as indicated by the respective *p*-value (far less than 0.01).

3.3. Fabric durability

The fabric smoothness and wettability of the finished PAN fabric were assessed after different washing cycles to assign the washing durability of the proposed treatment. The results of this investigation, summarized in Table 3, indicate that the finished fabrics are almost durable against

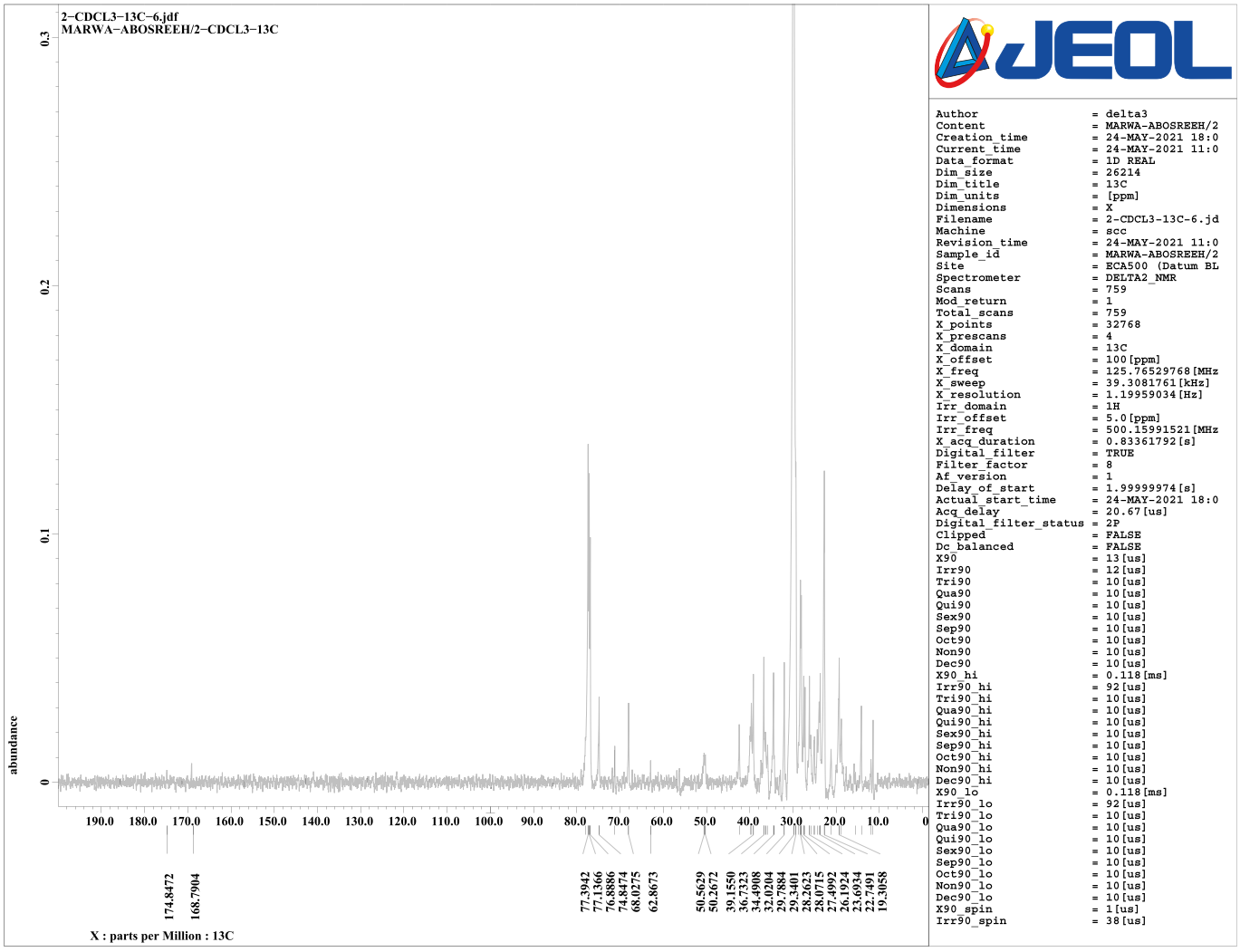


Figure 3. ^{13}C-NMR spectra of FA/AMPD softener.

Table 1. Effect of treatment of PAN fabrics with different amounts of FA/AMPD softener on their surface smoothness as well as some of their physical and mechanical properties (curing time: 4 min and curing temperature: 150 °C).

FA/AMPD softener conc. (% w/v)	Smoothness (°) [a]	UPF	Yellowing Index	Tensile strength (Kgf/cm^2)	Elongation at break (%)	Bending length (cm)	Wettability (S)
Untreated	44	4.4	3.84	78.0	36.4	3.8	40
Hydrolyzed	43	5.4	3.76	76.2	38.1	3.3	30
2%	43	6.6	11.13	76.4	37.5	3.2	26
3.5%	39	6.5	12.53	76.3	37.2	3.4	25
5%	40	7.4	12.26	76.1	36.9	3.8	21

[a] as the degree decrease, the fabric smoothness increases.

Table 2. The correlation coefficient between the different physical and mechanical properties of the untreated, hydrolyzed, and treated PAN fabrics.

	Smoothness	UPF	Yellowing Index	Tensile strength	Elongation at break	Bending length	Wettability
Smoothness	1						
UPF	-0.73354	1					
Yellowing Index	-0.78888	0.90552*	1				
Tensile strength	0.598046	-0.803	-0.57829	1			
Elongation at break	0.039766	0.16551	-0.07802	-0.688997	1		
Bending length	-0.08154	-0.1284	-0.10079	0.4919349	-0.831283	1	
Wettability	0.75582	-0.9759*	-0.83961	0.9095205*	-0.343543	0.244441	1

* Significant results (*p-value* less than 0.01).

Table 3. The smoothness and wettability of the treated PAN fabrics after washing for different washing cycles.

PAN sample	Smoothness (°) after					Wettability (S)				
	0 wash	1 wash	5 wash	10 wash	20 wash	0 wash	1 wash	5 wash	10 wash	20 wash
Treated with 2% of the synthesized softener	43	43	43	43	44	26	25	26	27	29
Treated with 3.5% of the synthesized softener	39	39	40	40	40	25	25	25	26	26
Treated with 5% of the synthesized softener	40	40	40	41	41	21	22	21	22	23

washing for up to 20 cycles. The fabric smoothness and wettability of the PAN fabrics treated with different concentrations of AMPD softener, after being subjected to the standard durability test, are nearly similar to those of the unwashed treated samples. This indicates that the applied AMPD softener was covalently bonded to the PAN fabrics, and hence the finished fabric can withstand the washing conditions inside domestic washing machines.

3.4. Comfort characteristics

The effects of treatment of PAN fabrics with the prepared nonionic softener on some of their comfort characteristics, viz. air and water permeability, and accumulated electrostatic charges, were monitored and the results are summarized in Table 4.

The data in this table reveals that almost the entire air permeability of PAN fabric was slightly changed compared to the untreated sample. The water permeability of the treated PAN fabrics was improved to a very limited extent. These imply that the main factors which affect the air and water permeability of fabrics were not drastically changed, viz. fabric structure, fabric type, and yarn structure. The slight change in air and water permeability may be attributed to the change in the fabric porosity.

One of the major problems in all synthetic fabrics, like PAN, is the accumulation of electrostatic charges on the surface of the fabrics causing a bad sensation on the human skin. As declared in Table 4, treatment of PAN fabrics with the prepared nonionic softener resulted in an improvement in their electrical conductivity and hence a reduction in the accumulated electrostatic charges. This is, presumably, due to the creation of active niches, namely hydroxyl and/or amino groups, along the macromolecules of PAN fabrics treated with FA/AMPD softener.

We can conclude from the findings in Tables 1 and 3 that the applied nonionic softeners resulted in enhancing the fabric softness and reducing the accumulation of electrostatic charges without negative any impact on their comfort attributes, viz. air and water permeability.

The data in Table 5 elucidates that there is a very strong correlation between the electrical conductivity and the water permeability of PAN fabrics, with strong significance as indicated by the respective *p*-vale (far less than 0.001).

Table 5. The correlation coefficient between the some comfort characteristics of the untreated, hydrolyzed, and treated PAN fabrics.

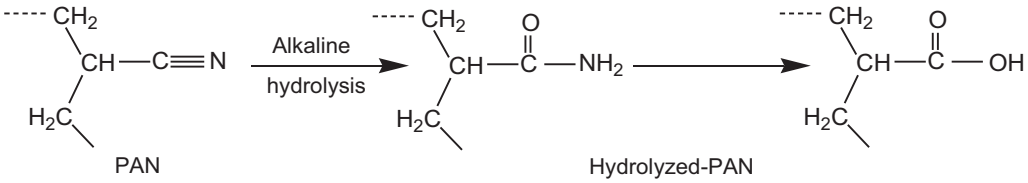
	Air permeability	Water permeability	Electrical conductivity
Air permeability	1		
Water permeability	-0.06187	1	
Electrical conductivity	-0.22909	**0.985714***	1

* Strong significant results (*p-value* less than 0.001).

3.5. Fibre morphology

The fibre morphology of the untreated, hydrolyzed, and nonionic softener-treated PAN fabrics was illustrated in Figure 4. The scanning electron micrographs elucidate the smooth even surface of the untreated and hydrolyzed fabrics. On the contrary, there is a layer of the applied nonionic softener on the surface of the treated fabric.

The results of EDX showed that the nitrogen content of untreated PAN fabric decreased sharply upon hydrolysis, presumably due to the conversion of the nitrile groups along PAN macromolecules into carboxylic groups as shown in equation 3.

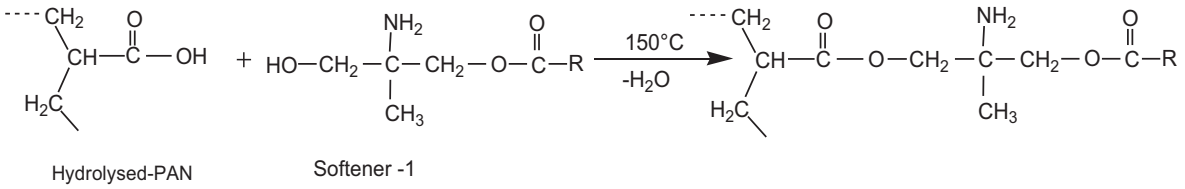


(3)

Reaction of the hydrolyzed PAN fabric with FA/AMPD softeners 1 and 2 led to a sharp increase in the nitrogen content of the fabric due to the introduction of free amino or amide groups within the modified macromolecules. Softener–1 may react via its hydroxyl group (Equation 4) or amino group (Equation 5) with the hydrolyzed fabric, whereas the reaction of the softener–2 with the hydrolyzed PAN fabric took place between the hydroxyl group of the former and the carboxylic group of the latter (Equation 6).

Table 4. Comfort attributes of PAN fabrics treated with FA/AMPD softener (3.5 %, curing time: 4 min and curing temperature: 150 °C).

Sample	Air perm. ($cm^3/cm^2/S$)	Water perm. (L/sec)	Dielectric const. at 100 Hz (ε')	Dielectric loss at 100Hz (ε'')	Electrical conductivity (Ω^{-1} cm^{-1})
Untreated	77.00	0.960	5.64	0.18	1.0×10^{-11}
Hydrolyzed	78.34	0.993	6.15	0.28	1.4×10^{-11}
Treated with the synthesized softener	77.32	1.098	12.3	1.22	6.8×10^{-11}



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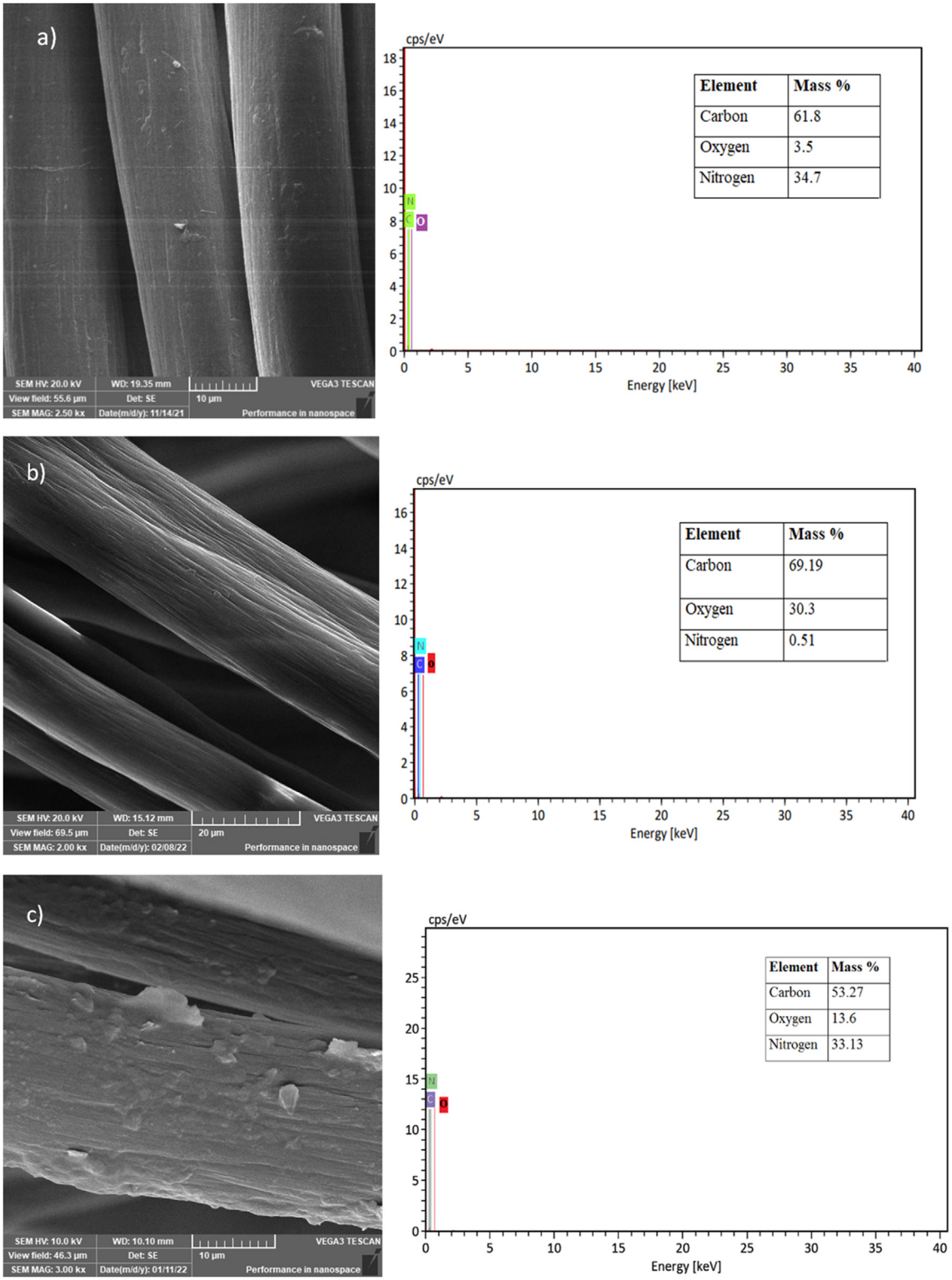
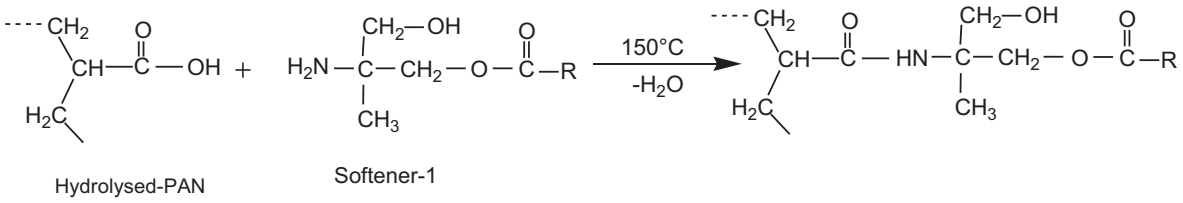
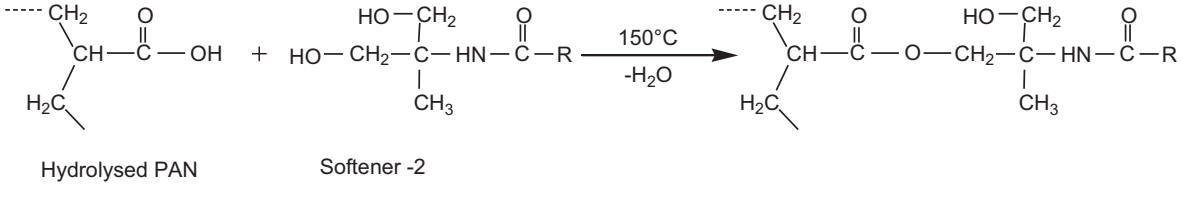


Figure 4. Scanning electron micrographs and EDX of (a) untreated; (b) hydrolyzed; and (c) treated PAN fabrics.



Hydrolysed-PAN Softener-1 (5)



Hydrolysed PAN Softener -2 (6)

Results of EDX analysis show that the oxygen content of the hydrolyzed fabric increased sharply upon hydrolysis due to the introduction of carboxylic groups, then decreased remarkably upon reaction with the prepared softener owing to the liberation of water molecules.

4. Conclusion

Based on the above findings, we can conclude that fatty acids (FAs) extracted from purified wool wax can be used for the synthesis of a nonionic softener through reaction with aliphatic amino diol. Spectroscopic analyses assured that the used amino diol reacts with the extracted fatty acid through ester formation as well as amide formation reactions.

The prepared nonionic softener was found to be an appropriate candidate to enhance the softness of PAN fabric. By virtue of its polar hydroxyl and/or amino groups, the used softener improved the fabric wettability and reduced the accumulated electrostatic charges on the fabric surface. The inherent properties of the treated fabrics, viz. air and water permeability, UPF and stiffness were not adversely affected. The degree of yellowing of the treated fabric increased remarkably, which makes it better to dye the fabric using dark colours only. The finished fabrics exhibited outstanding washing durability as they retained their smoothness and wettability after 20 wash cycles, indicating the formation of permanent covalent bonds between PAN fabric and the applied AMPD softener.

Declarations

Author contribution statement

M. Abou Taleb: Performed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

A. M. Hussien: Performed the experiments; Analyzed and interpreted the data; Wrote the paper.

A. F. Al-Fiky: Performed the experiments.

H. El-Sayed: Conceived and designed the experiments; Analyzed and interpreted the data; Contributed reagents, materials, analysis tools or data; Wrote the paper.

Funding statement

This work was supported by the Science, Technology, & Innovation Funding Authority (STIFA) (Project number: 38107).

Data availability statement

Data will be made available on request.

Declaration of interests statement

The authors declare no conflict of interest.

Additional information

No additional information is available for this paper.

Acknowledgements

The authors would like to thank Prof. Salwa Abdel Massieh for testing the electrical properties of the fabric surface.

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