



Article Calcium Chloride Treated Highly Elastane Cotton Fabrics as Antibacterial, Comfortable and Environmentally Friendly Materials

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Abstract: In the paper, the results of experiments on high elastane cotton crochet warp knitting fabrics treated with different calcium chloride solutions by the pad-dry-cure method have been reported. The tests for evaluating the antibacterial activity, thermophysiological comfort properties, physical performance of the samples were performed. In addition, the characterization analyses, including SEM, EDX, FTIR-attenuated total reflectance (ATR), and XRD, afforded indications of the good crosslinking of the CaCl₂ onto the cotton/elastane samples. At a higher degree of activity, the calcium-chloride-treated fabrics were rated as having good antibacterial activity, being exceptionally breathable, and being comfortable with good physical properties such as stiffness, handle, and whiteness properties.

Keywords: cotton; calcium chloride; antibacterial; thermophysiological comfort; elastane; XRD

1. Introduction

The most common organic compound on the planet is cellulose. It is a polysaccharide made up of a β -1,4-linked linear chain of d-glucose units. Unlike cellulose, amylase starch consists of glucose sugar units in α -1,4 linkage and exhibits widely different chemical, physical, and biological characteristics. Cellulose is a very highly crystalline, infusible, and insoluble polymer that often plays a structural role in nature. Fabric made from cellulose fibers is characterized by its ability to absorb and wick away or remove by capillary action, any moisture that it contacts [1]. Moisture management refers to a fabric's or garment's ability to manage moisture (especially sweat) by transporting or wicking moisture away from the skin to the fabric's outer surface, avoiding perspiration from lingering adjacent to the skin. Moisture vapor can flow through gaps in fibers or yarns. Hydrophilic fibers, for example, can act as a buffer by capturing moisture vapor and enhancing the fabric's comfort characteristics. Water or sweat must be routed through a fabric structure and then evaporated from the outside. Several studies have been conducted on elements influencing moisture management and comfort properties, such as fiber and yarn type, blend, fabric construction, and fabric density [2–9]. The optimum heat and moisture regulation, rapid moisture absorption, and conveyance capacity are the essential requirements of a good moisture management fabric. Polyester, with its low moisture absorption, polypropylene, with its good moisture wicking, and polyamide, with its wicking and durability attributes, are all popular fibers for moisture management.

Comfort characteristics are influenced by fiber type and moisture wicking, as well as the characteristics of double- and single-layer knit structures [10]. Because of their affinity for water molecules, hydrophilic fibers are not ideal for functional applications or unique materials. Liquid conveyance and release through capillary wicking without liquid retention is possible with hydrophobic fibers such as nylon and polyester [11]. Sweat



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Copyright: © 2021 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). production and the wearer's skin type have an impact on moisture management, which affects clothing's thermophysiological comfort [12].

The absorbency of textile materials is further affected by surface modification and fabric finishing processes [13–16]. The wicking treatment improves the rate of absorption of aramid fabrics but has no effect on their capacity or water vapor absorption [17,18]. Although the flame-resistant rayon blends enhance vapor absorption, they have a negative impact on liquid moisture management. Yoo et al. investigated the impact of hydrophilic fiber mixing and wicking finishes on moisture management capabilities in another investigation. It was reported that the former has no effect on the liquid moisture management qualities, whilst the latter significantly increases the absorption rate [17,18].

The impact of stitch length and single jersey, single airtex, and honeycomb knit structures on comfort attributes of moisture management finished microdenier polyesterknitted fabrics was the subject of Sampath's research. The fabric samples were treated with a chemical mixture of an amino silicone polyether copolymer and a hydrophilic polymer, and the finishing treatments improved the absorbency and wicking properties, according to the findings [19]. The durability of the finish is also important for the success of the chemical treatment [4]. Chance et al. investigated the use of formaldehyde in calcium chloride solutions to improve textile wrinkle recovery. There were significant differences between 10 wt % and 50 wt % calcium chloride solutions, with 35 wt % calcium chloride solutions being the best [20]. Glampedaki et al. studied the moisture absorption capacity of novel fabrics after functionalizing the surface of polyamide 6,6 fabrics with chitosan-based hydrogels. Chitosan considerably enhanced the wetting periods of polyamide fabrics in all situations [21]. In addition, calcium chloride has been determined to be generally recognized as safe (GRAS) based on scientific procedures and has been evaluated as environmentally safe by US FDA, as chloride toxicity has not been observed in humans.

The goal of this work is to use the calcium chloride pad-dry-cure method to improve antibacterial activity and thermophysiological comfort qualities of highly elastane cotton fabrics for functional reasons. Antibacterial activity and thermophysiological comfort features, bending length, bending rigidity, and yellowness-whiteness properties were also included while evaluating the effects of various chemical agent level treatment processes. Furthermore, surface morphology was evaluated using SEM, and the chemical structural features of the cotton/elastane samples were investigated using EDX, FTIR-attenuated total reflectance (ATR), and XRD.

2. Materials and Methods

The fabric samples used in this study were 60/40 cotton/elastane fabric with a weight of 294 g/m² and a thickness of 0.90 mm. Yarn density was 7 wales/cm and 20 courses/cm (yarn number: Co: 36 tex, elastane: 163 tex). The cotton/elastane fabrics were all warp knitting structures produced on a 16 gauge Comez 816/LT type crochet machine, and the crochet warp knitting structure was based on attaching columns, which were formed by the warp yarns, using a continuous weft yarn.

Calcium chloride (CaCl₂), 37% (w/w) hydrochloric acid (HCl), sodium bicarbonate (NaHCO₃), and other reagents used were analytically pure. CaCl₂ with a molecular weight (MW) of 110.99 g/mol was supplied from Carlo Erba while NaHCO₃, and HCl reagents were supplied from Sigma-Aldrich. Laundry tests were conducted using an Atlas LaunderOmeter washing machine for washing durability following AATCC Test Method 61 (2A): 2012 [22].

2.1. Treatments with Calcium Chloride Solutions

Firstly, calcium chloride solutions were prepared with three different percentages (10 wt %, 25 wt %, and 50 wt %) and were labeled as "a", "b", and "c", respectively, and for "b" samples after 5 and 10 washes, they were labeled as "e" and "f", respectively. as the recipe in Table 1. To achieve an ionic bonding on cotton/elastane samples, the pH of the solutions was adjusted to pH 12 using sodium bicarbonate as a weak base.

Recipe	C	nemical Agent (g/100 n	nL)
	CaCl ₂	HCl	NaHCO ₃
1	10.00	3.00	1.77
2	25.00	3.00	1.77
3	50.00	3.00	11.12

Table 1. CaCl₂ treatment recipes of the cotton/elastane fabrics.

The solutions were stirred at room temperature for 24 h after they were prepared according to Table 1. The CaCl₂ finish was then applied to the pre-scoured cotton/elastane fabrics using the pad-dry-cure process. In the laboratory-type padding machine (Prowhite/Y002), the solutions were impregnated to the samples at 21 °C for 2 h with an 85 percent wet-pick-up on the weight of fabric (owf) and at a 1.5 m/min speed and a 2.5 bar pressure. Fabrics were then dried at 120 °C for 2 min and then cured at 130 °C for 2 min (Prowhite/Y003).

2.2. Add-On Calculation

Equation (1) was used to determine the add-on values of the finished cotton/elastane fabrics:

$$W_{add-on} = \frac{(W_2 - W_1)}{W_1} \times 100,$$
 (1)

where W_1 denotes the weight of the untreated fabrics, while W_2 indicates the weight of the treated (finished) cotton/elastane fabric. Each sample was measured 10 times, and the average value was computed. The solution-treated cotton/elastane fabrics' add-on percentage values (%) were reported.

2.3. Antibacterial Activity

The experimental method used to determine the antibacterial effects was AATCC Test Method 100: 2004 "Assessment of Antibacterial Finishes on Textiles" Standards, using Staphylococcus Aureus ATCC 6538 and Escherichia Coli ATCC 25922 (2.00×10^5 CFU/mL) test inoculum [23]. The AATCC Test Method 61 (2A): 2012 "Colorfastness to Laundering: Accelerated" was used to evaluate the washing durability. The samples were evaluated after 5 and 10 washes. The percentage reduction of bacteria was calculated by the following Equation (2):

$$R = \frac{100 \ (C - A)}{C} \,, \tag{2}$$

where *R* is the percentage reduction of bacteria, *A* is the number of bacteria recovered from the inoculated sample at 0 contact time (CFU/mL) and *C* is the number of bacteria recovered from the inoculated sample after 24 h oscillation (CFU/mL).

2.4. Thermal Resistance

Zweigle Alambeta T675 was used to test the thermal resistance of the fabric. The guarded sweating hot plate apparatus, also called skin model, was used to measure the thermal resistance (R_{ct}) (m²K/W) values of the fabrics under steady-state conditions according to ISO 11092 (ISO, 1993) [24]. The temperature of the guarded hot plate was fixed at 35 °C (the temperature of human skin), and typical ambient circumstances (65% relative humidity (RH) and 20 °C) were used to determine the R_{ct} of the materials. The test device was placed in a climatic chamber with an airspeed of 1.1 ± 0.05 m/s created by an airflow hood. The test part was in the center of the plate, surrounded by a guard and a lateral heater that prevented heat leaking. The samples were placed on a porous metal plate surface for the R_{ct} test, and the heat flux from the plate to the environment was

monitored. When the system reached a steady state, the total thermal resistance of the fabric was determined using Equation (3):

$$R_{\rm ct} = \left[\frac{(T_m - T_a) \cdot A}{H - \Delta H_c}\right] - R_{ct(0)},\tag{3}$$

where T_m is the measuring unit's temperature (°C), T_a is the test enclosure's air temperature (°C), A is the measuring unit's area (m²), H is the heating power given to the measuring unit (W), ΔH_c is the heating power correction term (W), and $R_{ct(0)}$ is the thermal resistance without the sample.

2.5. Thermal Conductivity and Thermal Diffusion

The thermal conductivity and thermal diffusion values of the fabrics were also tested with the Alambeta device. Alambeta is a computer-controlled tool that measures textiles' essential static and dynamic thermal properties [24].

2.6. Water Vapor Permeability (WVP) and Water Vapor Resistance (WVR)

WVR and WVP were measured using Permetest equipment in accordance with the ISO 11092:2014 (sweating guarded hot plate test) standard [24]. WVR was calculated by dividing the difference in water vapor pressure between the test specimen's two faces by the evaporative heat flux per unit area in the direction of the water vapor pressure gradient. WVP refers to a material's ability to enable water vapor to travel through it. The relative WVP (%) is a non-standardized, practical statistic that reflects the tested sample's WVP as a percentage of that of a free measuring surface (where the WVP is 100%). The ratio of heat loss f is used to compute *P*. WVR is the water vapor pressure difference between the two faces of the test specimen divided by the resultant evaporative heat flux per unit area in the direction of the water vapor pressure gradient. Equation (4) was used to determine the ratio of heat loss from the measuring head with a fabric (q_s) and that without a fabric (q_0) [25]:

$$P = (q_{\rm s}/q_0) \times 100\% \tag{4}$$

2.7. Bending Length and Bending Rigidity Measurement

To evaluate tactile characteristics, the bending length and bending rigidity values of cotton/elastane fabrics were measured using the ASTM D1388 SDL Atlas Shirley Rigidity Testing Apparatus [26]. The average results were derived after measuring each sample 10 times.

2.8. Color Measurement

Chlorine chemical applications are known to cause yellowing when used on cellulosic fibers such as cotton and can return natural polymers to their original color (yellow). To explore the color change, the whiteness index (WI) and yellowness index (YI) values were measured with a DataColor 600TM Spectrophotometer, and the results were reported as Stentsby WI and E313 YI values, respectively. Each sample was measured 10 times, and the average values were obtained.

2.9. Characterization Analyses

SEM (Zeiss EVO 40) was used to analyze the surface morphologies of cotton/elastane fabrics at magnifications of $355 \times$ and $1100 \times$. To improve conductivity, the samples were coated with a thin gold film layer prior to testing. To acquire elemental information from cotton/elastane samples, EDX analysis (Zeiss Supra 40VP) was used. The samples were coated with a thin carbon layer prior to EDX examination to make them conductive. The infrared analysis was carried out using a Perkin Elmer Spectrum TwoTM Infrared Spectrometer (FTIR) in the ATR mode with a diamond universal ATR accessory and a diamond crystal with an effective depth of penetration of 1 µm and a resolution of 4 cm⁻¹. The test was performed in the 4000 cm⁻¹ to 650 cm⁻¹ range, and the recorded spectrum

for each sample was the average of four scans. To identify the crystallographic structure of the samples, XRD analysis was performed using the GNR APD 200 PRO brand X-ray diffractometer and CuK α radiation. The wavelength of the X-ray used was 1.54059 Å. The scanning range 2θ for the samples was between 5° and 50°, and the measurement was performed using an integration time of 2 s for each angle value. The average particle sizes of the samples were calculated according to the Debye-Scherrer equation (Equation (5)):

$$D = \frac{k\lambda}{\beta cos\theta} \tag{5}$$

The sample's crystallinity index was calculated using Equation (6):

$$C.I. = \frac{I_c - I_{am}}{I_c} \times 100 \tag{6}$$

2.10. Statistical Analysis

JMP version 8.0.2 software was used to perform statistical analysis on the experimental data (SAS Institute, Inc., Cary, NC, USA). The statistical analysis included the ANOVA, in which "SS" is the sum of squares of the deviations from the means, "df" is the degrees of freedom, "MS" is the mean squares, "F" is the ratio of two independent chi-square variables divided by their respective degrees of freedom, and "*p*-value" is the probability of obtaining test results (at least as extreme as the results observed during the experiment). *p*-values less than 0.05 were considered statistically significant for the one-way ANOVA. All of the data were also shown as average \pm standard deviation.

3. Results and Discussion

3.1. Antibacterial Activity

Figures 1 and 2 show the bactericidal capabilities of the control (untreated 60/40 cotton/elastane fabric) and calcium-chloride-treated cotton/elastane samples. Each sample had its complete population of *Staphylococcus aureus* (*S. aureus*) ATCC 6538 and *Escherichia coli* (*E. coli*) ATCC 25922. Following the antibacterial tests, the live vibrio concentrations of the standard blank sample at zero contact time, a standard blank sample oscillated for 24 h, and an antibacterial fabric sample oscillated for 24 h were compared. Figures 1 and 2 depict the effect of contact time on the percentage decrease of bacteria with treated fabrics against *S. aureus* and *E. coli*. A minor but statistically significant elevation in CaCl₂ concentration was reported, while a small but statistically significant decrease in wash cycles was observed. It was shown that the antibacterial actions of CaCl₂ induced a minor significant rise with the concentration increase and created long-lasting antibacterial resistance.

A study on the mechanism of *Pseudumonas* bacteria attachment on nylon fibrous media interaction with various levels of salt concentration of calcium chloride also showed that the created high-energy barriers resist the deposition of the bacteria based on the collision efficiency and change in the surface properties of bacteria [27].

Since the AATCC 100 test method is a quantitative test method, reliable test results were obtained about the antibacterial activity on Gram-positive *S. aureus* and Gram-negative *E. coli*. Although a stable and good antibacterial activity was achieved, the growth rates of *S. aureus* and *E. coli* decreased with increasing wash cycles due to the weak-ening of the bonds. It was found that the calcium chloride-treated elastic cotton fabrics retained their effectiveness even after 10 wash cycles. Studies on polyester/viscose fabrics treated with CaCl₂ cross-linked on the surface due to calcium cation showed excellent antibacterial activity against *S. aureus* and *K. pneumoniae* test organisms using AATCC Test Method 100 conducted by Kilinc et al. [28]. In another study by Dabay et al., bicarbonate (HCO₃⁻) significantly inhibits the growth of *S. aureus* and *E. coli*, suggesting that HCO₃⁻ can, in general, suppress bacterial growth [29].



Figure 1. Antibacterial activities using *Staphylococcus aureus* (*S. Aureus*) ATCC 6538 based on one-way ANOVA (p < 0.01). a: untreated; b: 10 wt % CaCl₂; c: 25 wt % CaCl₂; d: 50 wt % CaCl₂). The data are represented as mean \pm standard deviation (SD).



Figure 2. Antibacterial activities using *Escherichia coli* (*E. coli*) ATCC 25922 based on one-way ANOVA (p < 0.01). a: untreated; b: 10 wt % CaCl₂; c: 25 wt % CaCl₂; d: 50 wt % CaCl₂. The data are represented as mean \pm SD.

3.2. Thermal Resistance

Fabrics with low thermal resistance are often assumed to be more comfortable since they allow wearers to maintain normal control over their body temperatures. The thermal resistance values showed a statistically significant decrease with the increase in CaCl₂ chemical agent concentration in Figure 3. It was found that a samples had a higher thermal resistance than b, c, and d samples, depending on the calcium chloride concentration level. As a result, in terms of relative warmth, the untreated fabrics had the greatest effect on body temperature, and the fabrics treated with 50 wt % CaCl₂ had the least. The fabrics after 5 and 10 washes treated with 10 wt % CaCl₂ acted similarly with a samples and affected body temperature most significantly, creating the most thermal insulation capacity.



Figure 3. Thermal resistance values for each specimen versus % content in CaCl₂ based on one-way ANONA (p < 0.01). a: untreated; b: 10 wt % CaCl₂; c: 25 wt % CaCl₂; d: 50 wt % CaCl₂; e: 5 washes for b samples; f: 10 washes for b samples. The data are represented as mean \pm SD.

3.3. Thermal Conductivity and Thermal Diffusion

Since thermal conductivity is defined as the amount of heat that travels in unit time via the unit area and thickness, the coating procedure did not significantly hinder heat flow even after 5 and 10 washes. A small statistically significant increase in thermal conductivity was observed with the increase in calcium chloride concentration level, as presented in Figure 4. It can be seen that d samples showed a higher thermal conductivity value required for the transmission of heat from the skin to the outside.



Figure 4. Thermal conductivity values for each specimen versus % content in CaCl₂ based on oneway ANONA (p < 0.01). a: untreated; b: 10 wt % CaCl₂; c: 25 wt % CaCl₂; d: 50 wt % CaCl₂; e: 5 washes for b samples; f: 10 washes for b samples. The data are represented as mean \pm SD.

Thermal diffusion is a property associated with heat transfer through the air within the fabric structure. Because of the significant amount of air inside the structure, bulky fabric architectures have a higher thermal diffusion value. Figure 5 shows the coatings with the highest calcium chloride chemical concentrations created the bulkiest fabrics that could trap the air inside the fabric and would make the wearer feel warmer.



Figure 5. Thermal diffusion values for each specimen versus % content in CaCl₂ based on one way ANONA (p < 0.01). a: untreated; b: 10 wt % CaCl₂; c: 25 wt % CaCl₂; d: 50 wt % CaCl₂; e: 5 washes for b samples; f: 10 washes for b samples. The data are represented as mean \pm SD.

3.4. WVR

The WVR (R_{et}) values showed a statistically significant increase with the increase in chemical agent concentration level, as presented in Figure 6. Fabrics treated with higher concentrations of calcium chloride did not allow moisture transmission through the fabrics, and the moisture transmission decreased significantly with the increase in the calcium chloride concentrations for b, c, and d fabrics. After 5 and 10 washes for e and f samples, the water-resistance values tended to decrease, which indicated higher moisture transmission through the fabrics would make the wearer feel more comfortable.



Figure 6. Water vapor resistance values for each specimen versus % content in CaCl₂ based on one-way ANONA (p < 0.01). a: untreated; b: 10 wt % CaCl₂; c: 25 wt % CaCl₂; d: 50 wt % CaCl₂; e: 5 washes for b samples; f: 10 washes for b samples. The data are represented as mean \pm SD.

3.5. WVP

The relative WVP (%) values showed a statistically significant decrease with the increase in calcium chloride concentration levels, as shown in Figure 7. The untreated fabrics had a higher WVP value compared with the treated samples. When compared to the untreated materials, calcium chloride coatings caused poorer WVP, indicating that perspiration would not evaporate off the skin as expected, and high rates of wicking would not transfer moisture away from the skin via capillary action. After 5 and 10 washes for e and f samples, the WVP values tended to increase, which makes the fabric more comfortable for the wearer in further designs.



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Figure 7. Water vapor permeability values for each specimen versus % content in CaCl₂ based on one-way ANONA (p < 0.01). a: untreated; b: 10 wt % CaCl₂; c: 25 wt % CaCl₂; d: 50 wt % CaCl₂; e: 5 washes for b samples; f: 10 washes for b samples. The data are represented as mean \pm SD.

3.6. Bending Length and Bending Rigidity Results

Bending length and bending rigidity results in the machine direction (MD) and the cross-direction (CD) of the cotton/elastane samples treated in three different ways (10 wt % CaCl₂; 25 wt % CaCl₂; 50 wt % CaCl₂) by the pad-dry-cure method are given in Table 2.

Sample	Bending Length (cm·S)		Bending Rigidity (mg·cm·S)		
	MD	CD	MD	CD	
*a	2.16	1.53	454.68	175.04	
*b	4.14	1.79	2709.59	226.32	
*с	3.43	1.66	1927.45	221.38	
*d	4.35	1.55	8099.58	532.04	

Table 2. Bending length and bending rigidity results.

*a: untreated, *b: 10 wt % CaCl₂, *c: 25 wt % CaCl₂, *d: 50 wt % CaCl₂ (*p* < 0.01; one-way ANOVA).

When the values of the samples in terms of CaCl₂ concentrations were compared to the values of the untreated control sample, the bending length and bending rigidity results showed a statistically significant increase after calcium chloride treatments at the MD. The results steeply increased, depending on the amount of coating layer of fabrics. Similar results were found for all samples at the CD after calcium chloride treatments with different concentrations. It was considered that the calcium chloride treatments created a softener structure and decreased the bending length and the bending rigidity of the fabrics after higher chemical concentration levels.

3.7. WI and YI Results

Table 3 presents the WIs (Stentsby) and the YIs (E313) of three different methods to treat cotton and elastane samples (10 wt % CaCl₂; 25 wt % CaCl₂; 50 wt % CaCl₂).

Sample	WI (Stentsby)	YI (E313)
*a	72.25	7.57
*b	67.40	11.87
*с	66.53	11.77
*d	71.26	9.31

Table 3. Whiteness index (WI) and yellowness index (YI) results.

*a: untreated, *b: 10 wt % CaCl₂, *c: 25 wt % CaCl₂, *d: 50 wt % CaCl₂ (*p* < 0.01; one-way ANOVA).

After calcium chloride treatments, a statistically significant loss in WI and a statistically significant increase in YI were noted, as expected and described in the literature [30]. The WIs of the cotton/elastane fabrics remained almost unchanged or steeply decreased, depending on the amount of coating layer of fabrics. This could confirm the decrease of the fabric WI was higher for the fabrics treated with 50 wt % CaCl₂ chemical agents. The YI of the cotton/elastane fabrics increased according to the amount of the coating chemical. As shown in Table 3, a higher YI resulted in a lower WI. Calcium chloride concentrations were found to be strongly related to the WI and the YI, with the decrease in WI resulting from an increase in YI at the same chemical concentration level.

When the WI values of d samples were compared to the values of the untreated control sample in terms of calcium chloride concentrations, a decrease of roughly 1.4% was seen and a 23% increase in the YI value was noted. Therefore, the changes can be considered as an acceptable range.

3.8. SEM Results

The SEM images of the cotton/elastane samples were treated in three different ways (10 wt % CaCl₂; 25 wt % CaCl₂; 50 wt % CaCl₂) by the pad-dry-cure method are given in Figure 8.



Figure 8. The SEM results of the cotton/elastane samples: (**a**) untreated; (**b**) 10 wt % CaCl₂; (**c**) 25 wt % CaCl₂; (**d**): 50 wt % CaCl₂.

From Figure 8a, it can be seen that the sample had a bright surface structure. It can be seen in Figure 8b–d that this surface structure changed due to the calcium chloride treatment process and became compact. According to the images, the calcium chloride treatment process promoted swelling in fiber cross-section, indicating that higher swelling was induced with the increase in chemical concentration level.

3.9. EDX Results

EDX analysis was performed on the untreated and treated cotton/elastane samples to validate the CaCl₂ coating on the fabrics. Table 4 shows the atomic percentages of elements found in the cotton/elastane samples. Furthermore, comparable EDX spectra are shown in Figure 9. The carbon (C) content was projected to increase after CaCl₂ coating treatment, according to Table 4. The percentage amounts of C content decrease computed for b, c, and d samples were 38.18%, 46.54%, and 84.76%, respectively. The oxygen (O) content was found to decrease after CaCl₂ coating treatment for b, c, and d samples, parallel to the decrease in C content. The percentage drops in O concentration for b, c, and d samples were computed as 44.20%, 50.14%, and 77.89%, respectively. The O content was found to decrease after CaCl₂ coating treatment for d samples, which was computed as a 3.18% decrease, in parallel to the decreases in O content for b, c, and d samples. After CaCl₂ solution operations, the EDX findings validated the existence of Ca, Cl, and Na elements in b, c, and d samples, respectively. The Ca and Cl contents increased for b, c, and d samples, and d samples showed a higher increase than c and b samples, which also confirmed the achievement of CaCl₂ chemical agent bonding onto the cotton/elastane samples.

Table 4. Atomic percentages of different elements in the cotton/elastane samples.

Treatment –	Elements (Atomic %)				
	С	0	Na	Ca	Cl
*а	71.68	28.32	-	-	-
*b	44.31	15.80	2.79	25.27	11.83
*с	38.32	14.12	0.54	33.43	13.58
*d	10.92	6.26	1.80	50.30	30.73

*a: untreated, *b: 10 wt % CaCl₂, *c: 25 wt % CaCl₂,*d: 50 wt % CaCl₂ (*p* < 0.01; one-way ANOVA).



Figure 9. EDX spectra of the cotton/elastane samples coated with CaCl₂: (**a**) untreated; (**b**) 10 wt % CaCl₂; (**c**) 25 wt % CaCl₂; (**d**) 50 wt % CaCl₂).

3.10. FTIR-ATR Results

The FTIR-ATR spectra of the cotton/elastane samples were treated in three different ways (10 wt % CaCl₂; 25 wt % CaCl₂; 50 wt % CaCl₂) by the pad-dry-cure method are given in Figure 10. The FTIR-ATR spectra of all cotton/elastane fabrics show characteristic bands of cellulosic compounds at 3400 cm⁻¹ and 3290 cm⁻¹, which referred to the O–H stretching of the hydroxyl group of cellulose. Short peaks at 2900 cm⁻¹ and 1430 cm⁻¹ corresponded to methylene groups (–CH₂) and aliphatic C–H stretching, respectively. Calcium chloride characteristic peaks at 872 cm⁻¹ and 874 cm⁻¹ in C3 in the IR spectrum are the joint contributions of the bending vibrations of OH and CH. The peaks were greater for d samples in this band range, which could be due to the Ca⁺² increased reactivity to the hydroxyl functional group (–OH) of cellulosic fabric. This is in line with Jabar et al. [31]. The FTIR spectrum for the system Co/elastane/CaCl₂ shows a characteristic absorption band for the –C–O–C– stretching vibration at 1094 cm⁻¹ [32]. This again confirmed the crosslinking between cotton/elastane fabrics and CaCl₂.



Figure 10. FTIR-ATR spectra analyses of fabrics coated with CaCl₂: (**a**) untreated; (**b**) 10 wt % CaCl₂; (**c**) 25 wt % CaCl₂; (**d**) 50 wt % CaCl₂.

3.11. XRD Results

The XRD analyses of the cotton/elastane samples were treated in three different ways (10 wt % CaCl₂; 25 wt % CaCl₂; 50 wt % CaCl₂) by the pad-dry-cure method are given in Figure 11. The approximate particle sizes of the cotton/elastane samples using the Debye-Scherrer equation (Equation (5)) were found as 91.42 Å for a samples, 89.61 Å for b samples, and 139.21 for c samples.

The crystallinity indices of the cotton/elastane samples using Equation (6) were found as 35.476% for a samples, 51.714% for b samples, and 53.613% for c samples. According to the measured X-ray diffractogram for d samples, the calculation could not be made, because the healthy peaks required for the crystal index and crystallinity index calculations could not be obtained.



Figure 11. XRD analysis of fabrics coated with $CaCl_2$: (**a**) untreated; (**b**) 10 wt % $CaCl_2$; (**c**) 25 wt % $CaCl_2$; (**d**) 50 wt % $CaCl_2$.

4. Conclusions

The purpose of this study is to improve the antibacterial activity and thermophysiological comfort properties of high elastane cotton fabrics using a calcium chloride pad-dry-cure method, as well as to evaluate the effects of three different calcium chloride concentration level treatment processes. This is the first time to use the pad-dry-cure method in high elastane cotton fibers. The results demonstrated that the calcium chloride agent treatment had a substantial impact on antibacterial activity against S. aureus and E. coli as well as thermal comfort characteristics. It was also shown that using the pad-dry-cure process to apply calcium chloride solutions did not result in significant changes in physical attributes such as stiffness or visual properties such as color. At a greater degree of activity, calciumchloride-treated fabrics were rated as having good antibacterial activity, being exceptionally breathable, and being comfortable. In addition, the SEM analysis confirmed the swelling in fiber cross-section and the reformation on the fiber surface. The EDX analysis verified the presence of Ca and Cl elements, depending on the pad-dry-cure coating, and the FTIR-ATR analysis showed the changes in chemical structures of the cotton/elastane fabrics after chemical processes. The XRD analysis showed the calcium chloride application increased the degree of crystallinity (crystallinity %) due to crosslinking and bonding onto the cotton/elastane fabric structure. As a result of the aforementioned benefits, it is recommended that the calcium chloride process could be employed in other textile chemical processes and other textile materials, and prospective studies could be conducted for future advances.

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