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An Environmentally Friendly Dyeing Method for A Sustainable World: Investigation of Mechanical and Fastness Performance of Cotton/Wool Blend via Dyeing with Cinnamon

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Abstract: The world in which we live is changing at a much faster and more uncontrolled rate. This situation brings with it both negative and positive changes. The textile industry is also part of this rapid change. The growth of the world's population and the alterations in consumer habits that this change has brought with it also affect the textile industry. In particular, polluting dyehouses and the synthetic dyes used in them are harming the world. This situation is unsustainable. For this reason, the use of natural dyes instead of synthetic dyes in the coloring of textile products is increasing rapidly. In this study, a blend of 80% cotton and 20% wool was dyed with cinnamon, and its fastness and mechanical performance were tested. In addition, the dyeing results were analyzed using a spectral method, FTIR, and SEM. In this study, natural fiber blends with two different chemical structures were dyed with cinnamon in one bath. The fastness values obtained as a result of the dyeing process were very good. The grey scale value of saliva fastness determined through evaluation was 5. The observed strength increase of approximately 24% in the yarn after dyeing was also remarkable. In conclusion, environmentally friendly dyeing was carried out in this study, and a contribution was made to a sustainable world.

Keywords: cinnamon; cotton–wool blend; natural dyeing; eco-friendly; fastness; sustainability

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1. Introduction

The cinnamon plant is a tropical tree used in food, textiles, and medicine. Specifically, cinnamon is used as a spice in food, as a dye in textiles, and as an antimicrobial agent in medicine. Many studies have shown that cinnamon can aid in the treatment of diseases such as Alzheimer's disease, diabetes, and arthritis. It also gives a unique flavor to food and is used in natural dyeing in the textile industry [1,2]. Cinnamon can be used in different areas, such as food, medicine, and textile dyeing, because of its complex structure [3]. This structure is complex because it contains many different chemical components. As a result of the presence of these different components, the areas of application for cinnamon are quite wide. One of these areas is the dyeing of textile products, which has become interesting in recent years [4–6]. Synthetic azo dyes are mostly used for dyeing textile products. However, these dyes have many negative effects, such as allergic, toxic, and carcinogenic effects. Synthetic dyes can harm human skin when they come into contact with it, and dyeing waste is also harmful for the environment. Alternative methods are being developed to reduce the damage caused by synthetic dyes, which are often used in dye houses. The cationization process used to reduce waste loads in cotton dyeing, the use of impregnation instead of pulping in aqueous processes, or the use of natural dyes instead of synthetic dyes are some of these methods [7–10]. These methods are particularly important for avoiding the damage caused by synthetic dyes and for sustainable production. At this point, the use of natural dyes is essential for sustainable production and securing a greener world [11,12]. Colorful pigments from many plants are used in natural dyes. Cinnamon is one such plant. Many fibers have been dyed with the extraction solution of the bark obtained from the trunk of the cinnamon tree. Positive results have been obtained from these dyeing processes [13]. In addition to dyeing protein fibers such as wool and silk with cinnamon [14,15], cellulose fibers have also been dyed [16]. Fibers such as wool, silk, or cotton can be used in these dyes, and so can blends of these fibers. The fiber blends can be wool/silk blends or protein/cellulose blends [17,18].

In dyeing with cinnamon, the antibacterial and UV-protective properties of cinnamon have been investigated along with its coloring properties. The results of these studies have shown that cinnamon can be used in textiles not only for dyeing but also for other purposes [19–22]. Fastness results are also important when using cinnamon in dyeing. In particular, the resistance of the dyed product to external influences during use influences user satisfaction. For this reason, the fastness properties of cinnamon-dyed products for washing and rubbing have also been investigated [23]. The main parameters affecting these fastnesses in dyed products are dyestuff–fiber interactions, dyestuff fixation, and after-treatment conditions. In addition, one of the most important parameters affecting the fastness of vegetable dyes such as cinnamon is the mordanting process. Mordanting is a very important process for the lifetime fixation of a dye, and many mordants are used for this purpose [24]. These mordants show different effects in dyeing and influence the dyeing results [25–27]. Other parameters that affect the results in these dying processes are temperature, pH, time, and the amount of dye used [28,29]. Some analyses are needed to evaluate the effect of all these parameters on the dyeing results. These analyses can be performed using FTIR and spectral color measurement methods [30].

Dyeing with natural pigments has become increasingly important in recent years. The reason for this is the waste and environmental pollution caused by the incompatibility of synthetic fibers and dyes. This pollution is no longer sustainable. Therefore, the importance of environmentally friendly natural dyes is increasing [31,32].

This study aims to achieve sustainable production in textile dyeing and to minimize the negative impacts of this production. For this purpose, natural products were used in the whole production process from fiber to dye. The cotton–wool blend was used as the finished product, and the coloration pigment obtained from cinnamon was used as the dye. In this study, the interactions between natural dyes and fibers were analyzed using FTIR, and the effects of all the processes on the mechanical performance of the product were investigated. In addition, SEM images were taken at all stages of the processes applied to the product, and the effect of the substances used was analyzed visually. Fastness values important for dyed fabrics, such as wash, perspiration, and saliva fastness, were also analyzed.

2. Materials and Methods

2.1. Materials

Ne 30/2 ring yarn with an 80% cotton (combed cotton) and 20% wool blend was used for dyeing. Water hardness was controlled, and soft water was used. Water hardness control was performed before each process. The test tubes used were washed with hydrosulfite (H_2S) at 80 °C and then cleaned via a second washing with dilute acetic acid. The pH of the soft water used in this study was checked with a calibrated pH meter and pH paper before each study. The pH of the water used in this study was 7.5–7.7.

The yarn (20 g in total) used in this study was wound in a hank winding machine and tied with yarn from 3 different regions in order to avoid dispersal during the processes.

2.2. Method

In this study, cotton–wool blend yarn was dyed via cinnamon extraction. Pre-finishing, mordanting, and dyeing processes were applied to the blend. A shrinkage method was used in this study. Codes were given according to the processes applied to the yarn. These codes are as follows: untreated (CWE), pretreated (CWPT), mordanting (CWM), cinnamon-dyed without mordanting (CWC-NM), and cinnamon-dyed with mordanting (CWCM).

A pre-finishing process was applied to the cotton–wool blend yarn before the dyeing process was conducted. For this purpose, Tubotex OW, a product of the CHT company; a commercially available ion trap; and a wetting agent were used. The process was applied according to the following recipe: 2.5 g/L of Tubotex OW, 1 g/L of ion scavenger, and 2 g/L of wetting agent heated at 95 °C for 40 min with a flotte ratio of 1/10. The pretreatment bath pH was 7.6, and the pH value was measured as 11 with the addition of pretreatment agents. After treatment, overflow washing and neutralization with citric acid were performed.

Cotton wool blend yarn was mordanted with 10% KAl $(SO_4)_2$ 12 H_2O (alum) at 100 °C for 60 min before dyeing. Since the alum used was coarse-grained, it was first crushed using a mortar and pestle into small granules. Then, a bath was prepared with a flotte ratio of 1/25 and magnetic mixers were used for complete dissolution and homogenous distribution of alum. The pH of the bath prepared with alum was measured as 4.145. After mordanting, the bath outlet pH was noted as 3.8. After mordanting, the samples removed from the bath were squeezed and left in a conditioning chamber for 24 h.

Before staining, cinnamon in the bark was ground into powder, and the extraction of cinnamon was prepared according to the recipe used in this study. Afterward, soft water and cinnamon powder were placed in steel tubes with a flotte ratio of 1/25. The lids of the tubes were closed tightly, and the tubes were placed in the machine. The machine program was adjusted according to the recipe, and the temperature was increased from 40 °C to 100 °C in 15 min. After 60 min at 100 °C, cooling and filtration were performed. The extraction bath was left to rest for 24 h under room-temperature conditions. The extraction bath was stirred before staining, and then staining was performed. The cinnamon extract was filtered, and the pH was measured and found to be 5.6. After dyeing, the pH of the bath outlet was noted as 4.578. After dyeing, the finished product was left in the conditioning room for 24 h after the necessary post-treatments (Figure 1).

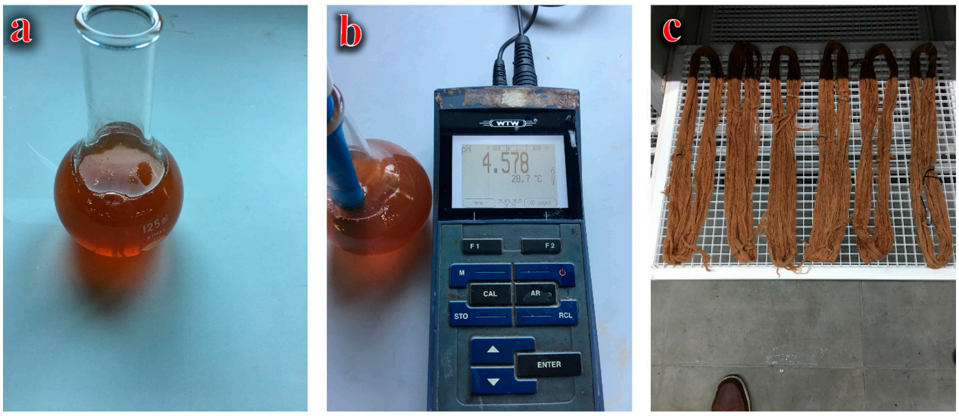

Figure 1. (**a**) Cinnamon extraction, (**b**) extraction pH, and (**c**) conditioning of the dyed product.

The blended yarn used in this study was pretreated, mordanted for mordant dyeing, and prepared, and the filtration of the cinnamon extract was conducted for cinnamon dyeing. Graphs of all processes are shown in Figure 2.

Since the yarn used in this study is a blend, hundreds of trials were conducted to create temperature, time, and pH conditions, among others, that would prevent both fibers from being damaged. The dyeing pH was adjusted so that both fibers would not be damaged. In addition, time and temperature adjustments were made for the dye absorption of cotton with a slightly anionic structure and wool with a cationic structure. This process is very important as it prevents uneven dyeing.

The products obtained by dyeing cotton–wool blend yarn with and without mordanting with cinnamon were wound on bobbins and prepared for the tests (Figure 3).

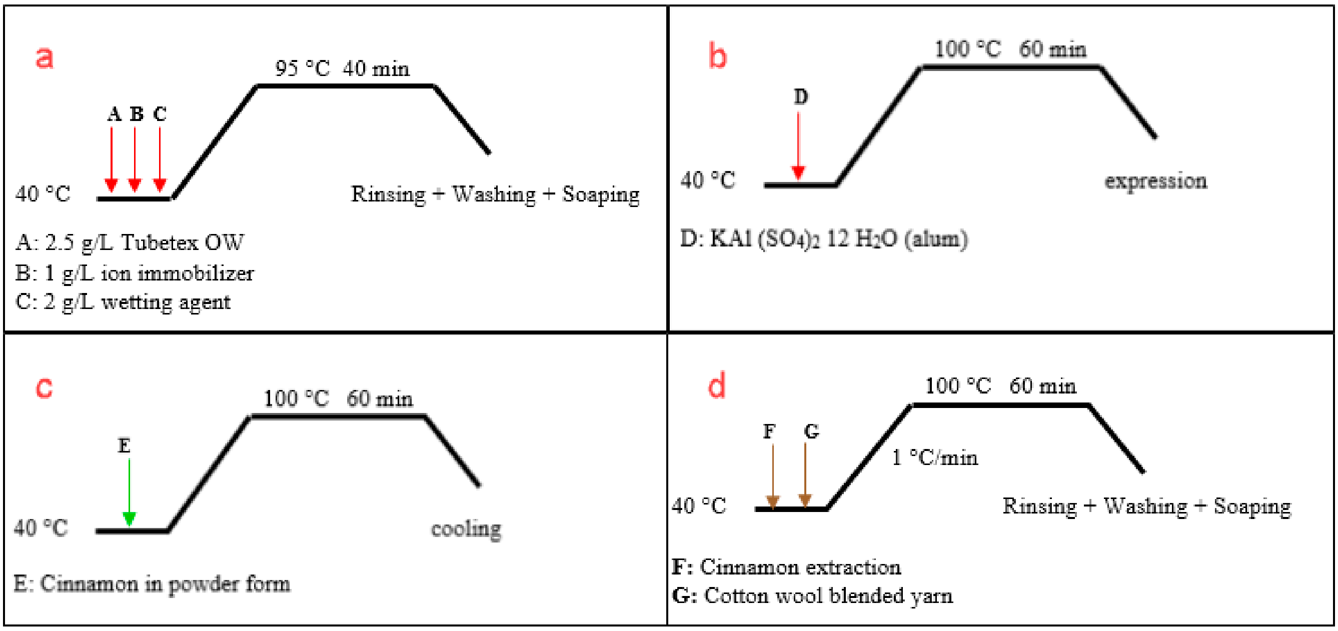

Figure 2. Graphs of treatment of cotton–wool blend yarn: (**a**) pre-treatment, (**b**) mordanting, (**c**) extraction process, and (**d**) dyeing of cotton–wool blend with cinnamon extract.

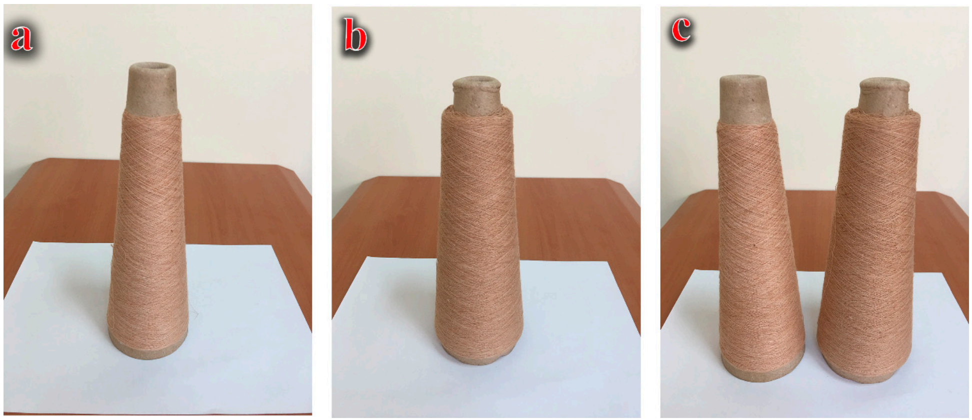

Figure 3. Cotton wool blend yarn dyed with cinnamon: (**a**) non-mordanted, (**b**) mordanted, (**c**) non-mordanted–mordanted.

2.3. Fastness Testing

Washing, rubbing, saliva, water, and perspiration fastness tests were performed on cotton–wool blends. Washing fastness was tested according to ISO 105-C06 [33] standard, rubbing fastness was tested according to ISO 105-X12 [34] standard (Figure 4a), saliva fastness was tested according to DIN 53160-1 [35] standard (Figure 4b), water fastness was tested according to ISO 105-E01 [36] standard (Figure 4c), and perspiration fastness was tested according to ISO 105-E04 [37] (Figure 4d). Images of the tests are shown in Figure 4. The test results were evaluated on a grey scale, and the results obtained were noted. A light box with standard light sources was used in the evaluation.

2.4. Yarn Strength Measurements

The changes in the strength and elongation properties of Ne 30/2 blend yarn consisting of 80% cotton and 20% wool were measured before and after dyeing with cinnamon. The strength values of the yarn in untreated (CWE), pretreated (CWPT), mordanted (CWM), non-mordanted and cinnamon-dyed (CWC-NM), and mordanted and cinnamon-dyed (CWCM) forms were measured. USTER® UTR 5 20.8.4.6f19301 device was used for the measurement (Uster Technologies AG, Uster, Switzerland). The test speed of the device was 5000 mm/min, and the number of test repetitions was 10. The tests were performed at 23 °C and at 62.1% relative humidity. The tested yarn length was 500 mm.

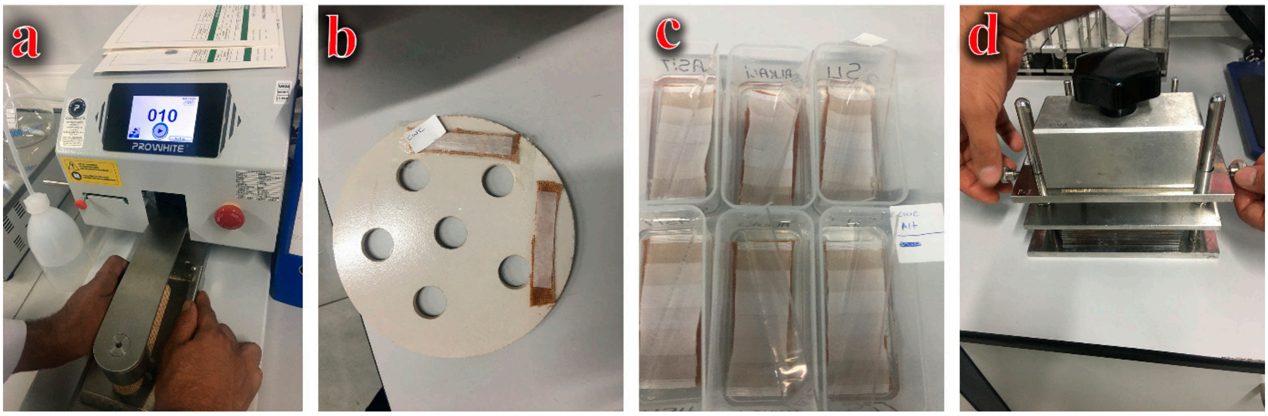

Figure 4. (**a**) Rubbing fastness, (**b**) saliva fastness, (**c**) water fastness, and (**d**) perspiration fastness.

3. Results and Discussion

3.1. Fastness Test Results

The test results obtained from the dyeing of cotton–wool blend yarn with cinnamon without being mordanted and after being mordanted are shown in Table 1. The washing fastness of the yarn dyed with cinnamon without mordanting was evaluated in the presence of a multifiber fabric and evaluated on a grey scale. In the results, scores of 4–5 correspond to good and very good. All fibers in the multifiber fabric showed little or no contamination. Again, the yarn dyed with cinnamon without mordanting was knitted, and dry and wet rubbing fastnesses were examined. The dry rubbing fastness of the fabric dyed with cinnamon without mordanting was 4–5, and the wet rubbing fastness was 4. These values are quite good. The perspiration fastness of the same fabric was examined under acidic and alkaline conditions. Among the multifiber fabrics, the lowest grey scale value was 4, obtained for cotton, and 4–5 for the others. In addition, the water fastness of our fabric dyed without mordanting was found to be 4–5.

The washing fastness of the yarn dyed with mordanted cinnamon was evaluated on a grey scale in the presence of multifiber fabrics. In the results, scores of 4–5 correspond to good and very good. All fibers in the multifiber fabric showed little or no contamination. Again, yarn dyed with mordanted cinnamon was knitted, and dry and wet rubbing fastnesses were examined. The dry rubbing fastness of the mordanted cinnamon-dyed fabric ranged from four to five, and the wet rubbing fastness was four. These values are quite good. The perspiration fastness of the same fabric was examined in acidic and alkaline conditions. In the multifiber fabric, the lowest grey scale value was 4 in cotton and 4–5 in the others. In addition, the water fastness of our mordanted dyed fabric was found to range from four to five.

The saliva fastness results of cotton–wool blend dyed with cinnamon with and without mordanting were evaluated on a grey scale, and a very good value of five was obtained. It can be concluded that cinnamon-dyed products can be easily used for coloring baby clothes due to their saliva fastness.

3.2. Colorimetric Analysis

In this study, CIELAB values, K/S graphs, reflectance graphs, and color space images of the cinnamon-dyed product were obtained using a spectrophotometer. The device used for this purpose was the datacolor500.

In the spectral measurement of cotton–wool blends dyed with and without mordanting with cinnamon, the results were obtained with different standard light sources and the same measurement geometry. When the CIELAB values in Table 2 are analyzed, it can be seen that the L values are higher in the dyeing of cinnamon without mordanting (CWC-

NM). This shows that in the mordant dyeing of cinnamon (CWCM), the product is dyed darker via consuming more dye. It can be said that some degree of a decrease is expected in mordant dyeing, where the reflectance values of X, Y, and Z filters in the CIELAB system are compatible. This is also seen in the higher reflectance values of the CWC-NM yarn, which consumes less dye and is lighter, in all filters. In the D65—10° measurement, the X, Y, and Z values of CWC-NM were 24.92, 24.17, and 15.90 respectively, while these values were 22.52, 21.41, and 13.20 in CWCM.

Table 1. Fastness results of cotton–wool blend dyed with cinnamon with and without mordanting.

Fastness Type	**Multifiber Fabric**	**Test Conditions (Acidic–Alkaline)**	**CWC-NM Greyscale Value**	**CWCM Greyscale Value**
Perspiration fastness (staining)	Acetate	Acidic	4–5	4–5
		Alkaline	4–5	4–5
	Cotton	Acidic	4	4
		Alkaline	4–5	4–5
	Nylon 6.6	Acidic	4–5	4–5
		Alkaline	4–5	4–5
	Polyester	Acidic	4–5	4–5
		Alkaline	4–5	4–5
	Acrylic	Acidic	4–5	4–5
		Alkaline	4–5	4–5
	Wool	Acidic	4–5	4–5
		Alkaline	4–5	4–5
Fastness type	Multifiber Fabric		CWC-NM Greyscale value	CWCM Greyscale value
Washing fastness (contamination)	Acetate		4–5	4–5
	Cotton		4–5	4–5
	Nylon 6.6		4–5	4–5
	Polyester		4–5	4–5
	Acrylic		4–5	4–5
	Wool		4–5	4–5
Fastness type	Test conditions		CWC-NM Greyscale value	CWCM Greyscale value
Rubbing fastness	Dry		4–5	4–5
	Wet		4	4
Fastness type			CWC-NM Greyscale value	CWCM Greyscale value
Saliva fastness			5	5

Table 2. CIELAB values of cotton–wool blend yarns that were dyed with cinnamon and non-mordanted.

Standard	**Sample Code**	**L***	**a***	**b***	**C***	**h°**	**X**	**Y**	**Z**	**x**	**y**
D65—10°	CWC-NM	56.26	8.83	18.76	20.73	64.79	24.92	24.17	15.90	0.38	0.37
	CWCM	53.39	10.53	20.18	22.76	62.43	22.52	21.41	13.20	0.39	0.38
A—10°	CWC-NM	58.40	12.36	21.49	24.79	60.09	32.85	26.39	5.36	0.51	0.41
	CWCM	55.80	14.17	23.33	27.29	58.73	30.14	23.71	4.46	0.52	0.41
TL84—10°	CWC-NM	57.40	8.37	21.40	22.98	68.64	28.22	25.33	9.12	0.45	0.40
	CWCM	54.62	9.92	23.00	25.05	66.67	25.58	22.56	7.55	0.46	0.41

In the color space of the cotton–wool yarn blend dyed with cinnamon without mordanting and dyed with cinnamon with mordanting, both dyeing results were in the +a* and +b* region, and C was slightly higher in the cotton-wool dyed with cinnamon without mordant. It was expected that the affinity of the fiber for the dyestuff would be higher in cinnamon dyeing with mordanting and that the L* value would therefore be lower (Figure 5).

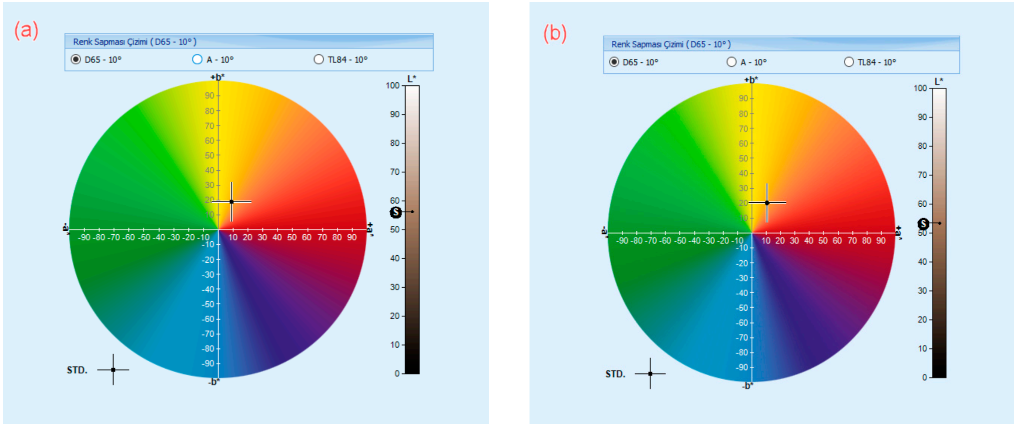

Figure 5. CIELAB color space of a cotton–wool blend dyed with cinnamon: (**a**) non-mordanted; (**b**) mordanted.

The L*-axis describes the lightness: a white object has an L* value of 100, and the L* value of a black object is 0. The so-called achromatic colors, the shades of grey, are on the L*-axis. Each color is represented by a color point (L*, a*, b*) in the color space; L*, a*, and b* are the color coordinates of the color points.

According to the results of the measurement at a 360–700 nm wavelength, it was observed that the K/S values were higher in the 360–560 nm region of mordant cinnamon staining, but these values gradually approached each other and equalized after 660 nm (Figure 6).

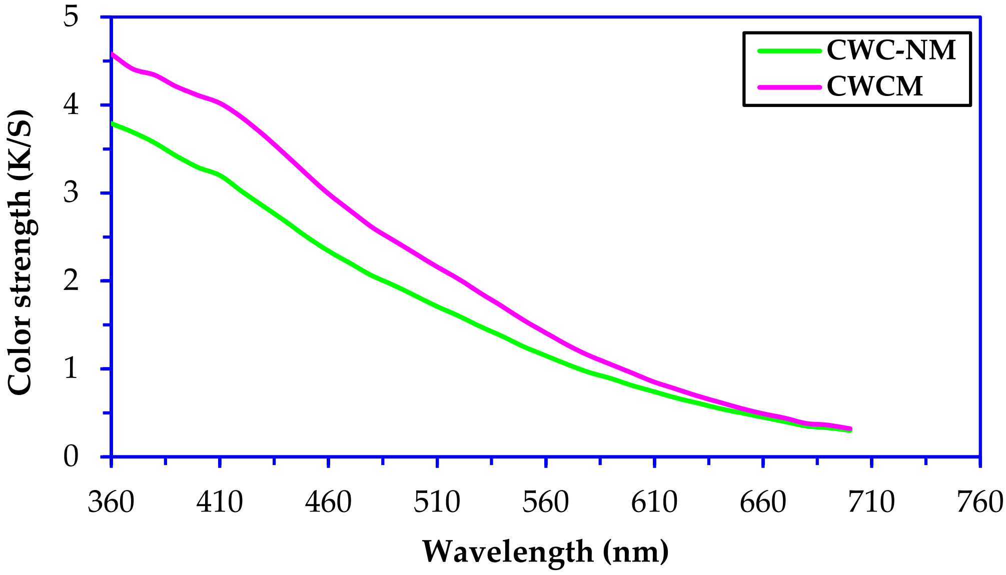

Figure 6. Color strength (K/S) wavelength graph of cotton–wool blend yarn dyed with cinnamon.

The %R value was slightly higher in the yarn dyed with cinnamon without mordanting. This shows that there is greater dye consumption in dyeing with mordanted cinnamon. In addition, it can be seen that the colorant pigments in cinnamon are located deeper in the red region (Figure 7).

3.3. Mechanical Performance of the Product before and after Dyeing

The tensile strength of cotton–wool blend yarn was evaluated. The pretreatment resulted in a significant increase in the tensile strength of the untreated (CWE) yarn. This increase was partial after mordanting. As a result of dyeing, for the finished products that were not mordanted and those that were mordanted, the increase in tensile strength was quite high. It was also observed that the treatments made a positive contribution to the % elongation. Except for some chemical treatments, such as mercerization, a decrease in strength is generally observed as a result of finishing processes. Looking at the results in Table 3, an increase of approximately 24% in tensile strength and 26.81% in % elongation was achieved as a result of pre-treatment. In addition, increases of 20.64% in tensile strength and 22.12% in % elongation were observed in the product dyed without mordanting. In mordanted dyeing, these increases were 21.17% in tensile strength and 17.82% in % elongation (Table 3).

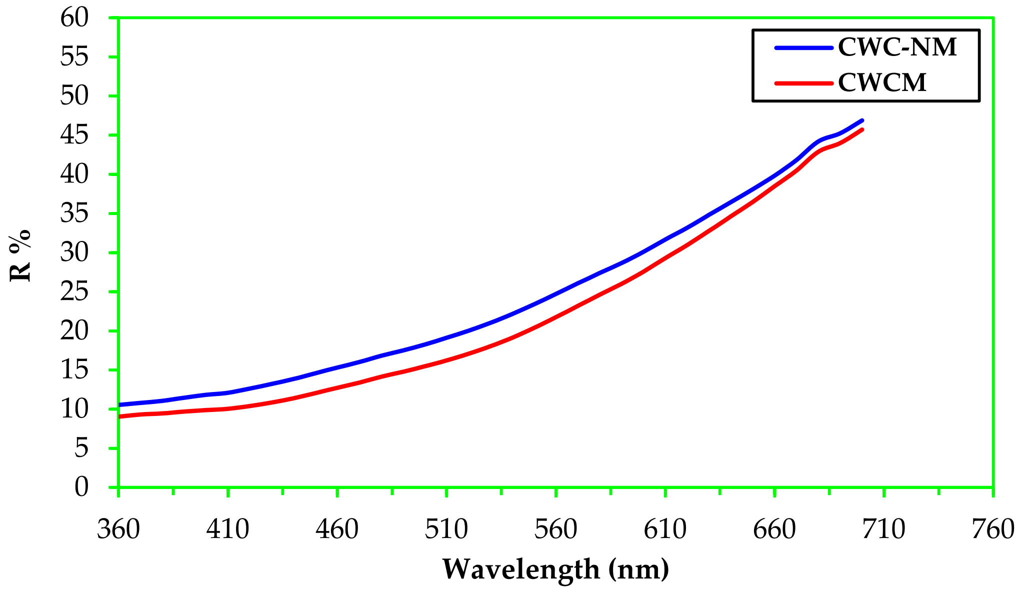

Figure 7. R% wavelength graph of cotton–wool blend yarn dyed with cinnamon.

Table 3. USTER values of cotton–wool blend yarn.

Sample Code	Sample Name	B-Force Average (N)	Elongation Average (%)	Tenacity Average (Rkm)	B-Work Average (N.cm)
CWE	Untreated	4.73	5.35	12.25	6.64
CWPT	Pre-treated	6.22	7.31	16.11	10.33
CWM	Mordanted	5.19	7.00	13.43	8.45
CWC-NM	Dyed without mordanting	5.96	6.87	15.44	9.66
CWCM	Dyed with mordanting	6.00	6.51	15.55	9.24

3.4. FTIR Analysis

Untreated cotton/wool blend (CWE), pretreated cotton/wool blend (CWPT), mordanted cotton/wool blend (CWM), cinnamon-dyed cotton/wool blend without mordanting (CWC-NM), and mordanted cinnamon-dyed cotton/wool blend (CWCM) were analyzed using FTIR to determine the effects of the substances transferred to the product at all stages of the study (Figure 8).

In the FTIR analysis of CWE, it was observed that the graph obtained corresponded to a 92% D-cellulose3 structure that was similar to cotton and a 91.8% D-cellulose5 structure that was similar to ramie. In the FTIR analysis of CWPT, there was an 84.3% D-cellulose5 structure that resembled stenter and an 84.2% D-cellulose3 structure that resembled cotton. We estimate that this was due to the pre-treatment. The fact that the process was carried out in a basic environment and the product's being a mixture of cotton and wool may be the reasons for this result. Unlike the FTIR graph of CWE, a new peak was observed in the 2300 cm^{-1} band as a result of the pre-treatment. This peak was also observed in the FTIR graphs of the mordanting, cinnamon dyeing without mordanting, and mordant dyeing processes. It can be seen that new bonds were formed in this region, and these bonds are C≡C and C≡N triple bonds. As a result of the mordanting of CWE, it was observed that the %T values in the 400–1000 cm^{-1} band increased from 73% to 85%, and frequent and numerous peaks were formed in the 1000–2300 cm^{-1} band.

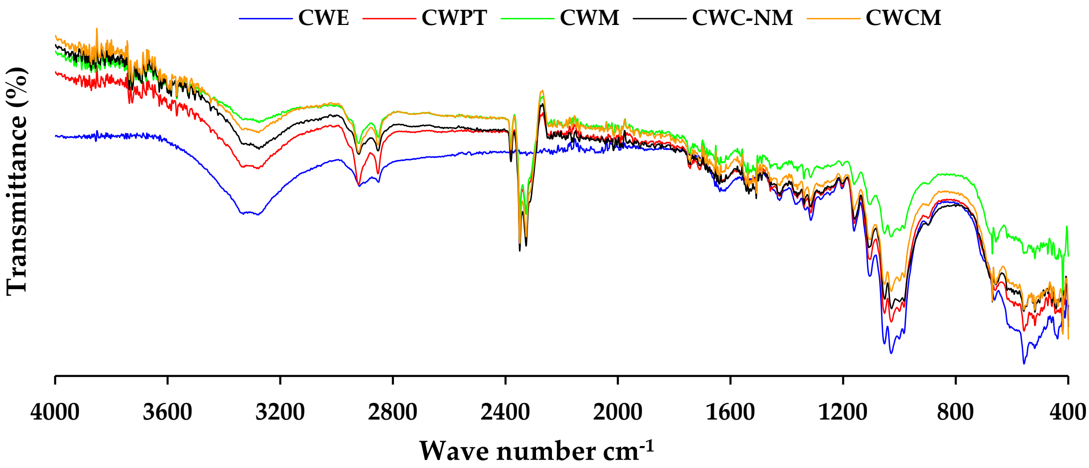

Figure 8. FTIR graphs of cotton–wool blend yarn at all stages.

In the 3300 cm^{-1} band, it can be seen that there is a weakening in the peak as a result of mordanting, and the number of C=C double bonds is reduced. In the 2850 cm^{-1} band, the %T value of the peak decreased from 98% to 93%. It was observed that weak but frequent peaks were formed in the 1500–2200 cm^{-1} band as a result of non-mordanted and mordanted staining with cinnamon and that these were C≡C, C≡N, R-CO-NH2, and R-CO-H bonds. The peaks in the 1690–1760 cm^{-1} band are thought to be cinnamaldehyde from cinnamon.

FTIR analyses were performed for all the processes applied to the cotton–wool blend yarn. The results of these processes, especially the peak that formed in the 2300 cm^{-1} band as a result of pre-treatment, will attract attention.

3.5. SEM Analysis

SEM images of the untreated, pretreated, pre-treated, mordanted, non-mordanted cinnamon-dyed, and mordanted cinnamon-dyed cotton–wool blend products were analyzed. From the SEM images, it can be said that the treated state of the structure is more uniform, and the substances transferred by the processes provide smoothness to the surface of the structure. In addition, it can be seen that the surfaces of the cotton and wool forming the mixture are covered with dyestuff as a result of dyeing the product with cinnamon without mordanting and with mordanting (Figure 9).

The study yields important results in terms of dyeing two fiber blends with very different chemical structures in a single bath. In addition, the fact that a product consisting of natural fibers was dyed with cinnamon, which is a natural colorant, and the finding that the results obtained were good are also noteworthy in terms of proving the accuracy of the dyeing method applied in this study.

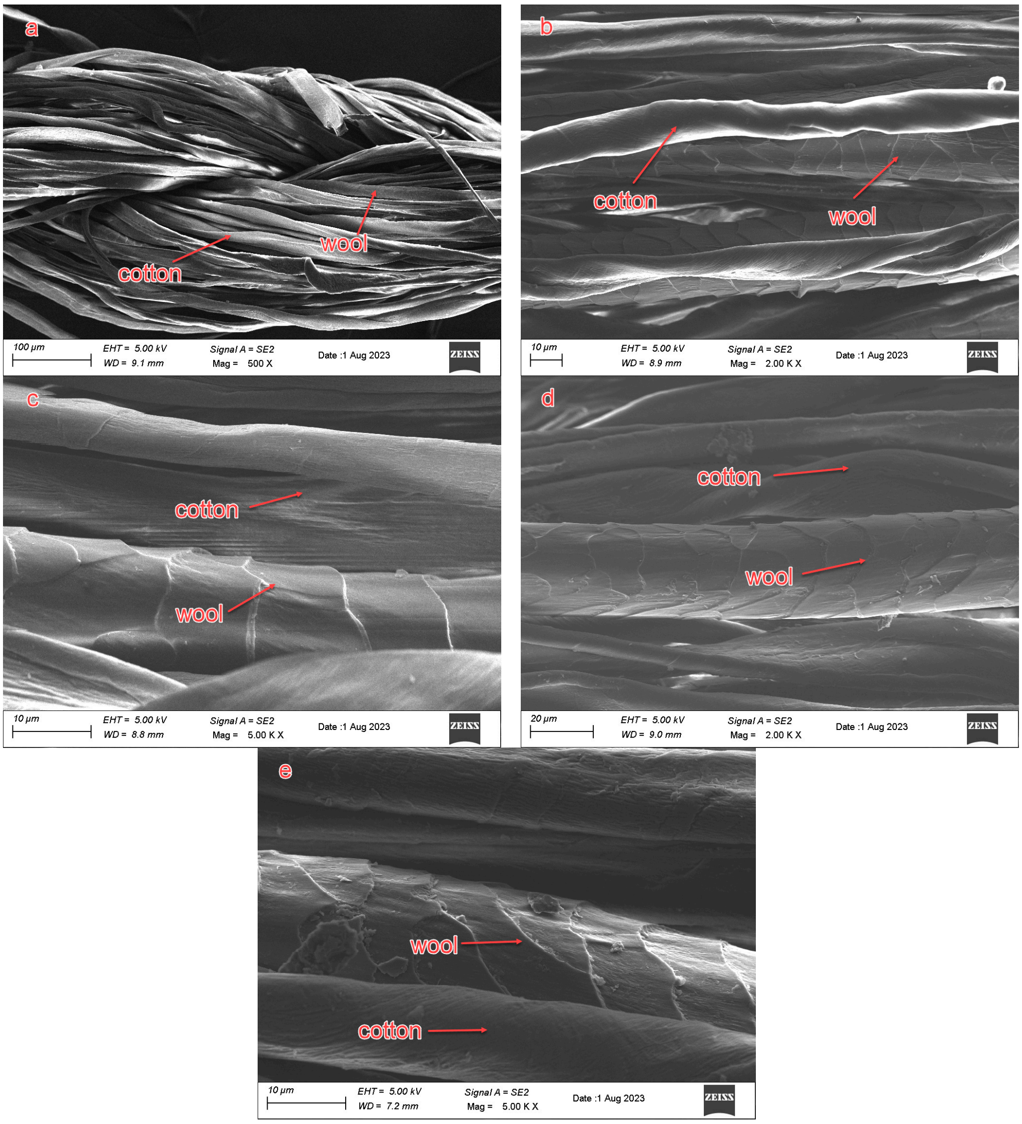

Figure 9. SEM images of cotton/wool blend: (**a**) untreated cotton–wool blend, (**b**) pre-treated cotton–wool blend, (**c**) mordanted cotton–wool blend, (**d**) non-mordanted cotton–wool blend dyed with cinnamon, and (**e**) mordanted cotton–wool blend dyed with cinnamon.

4. Conclusions

A natural cotton–wool blend textile product was dyed with cinnamon, which is also a natural dyestuff. In addition to the ecological advantages of this dyeing procedure, positive results were obtained in terms of fastness and mechanical performance. According to the results of the tensile tests performed on the cotton–wool blend product, the highest values were obtained in the pretreated yarn. Again, an increase was observed in the mechanical performance of the mordanted and dyed product compared to the raw product. The tensile elongation was also greater in the pretreated product. This situation reveals the positive effects of the processes applied to the raw product. The new chemical bonds formed and the coating effect of the colorant obtained from cinnamon on the surface of the product played an important role in obtaining these results. The new chemical bonds formed can also be seen in the FTIR test and SEM images.

In this study, the fastness results of dyeing processes incorporating cinnamon were also examined, and the values were found to be very good. In particular, saliva fastness was evaluated on a grey scale, and the obtained value, 5, was a very good result. Again, desired values such as 4–5 were obtained from dry and wet rubbing fastnesses. In addition, acidic and alkaline perspiration fastness and water fastness were also found to be in the 4–5 range. In addition, the grey scale values of washing fastness, which is the most important fastness test, were in the 4–5 range. These values correspond to good and very good. The fastness tests with cinnamon showed us that the results were quite good and acceptable.

The cinnamon plant contains high amounts of color pigments in its structure. According to a CIELAB space image taken using a spectrophotometer, these pigments were found to be concentrated in the region where yellow and red are predominant, especially in the first region.

Cinnamon can be easily used in textile dyeing for applications pertaining to both human health and the protection of natural life. This is very necessary for a sustainable world. We recommend the use of cinnamon, especially for dying children's clothes.

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