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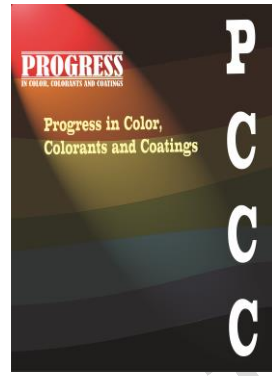

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Effect of Dyeing Temperature and Dye Concentration on Color Characteristics and Fastness of Annatto-Dyed PET/Wool (55/45) Fabrics

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Abstract

Polyester/wool blended fabrics (55:45), conventionally dyed with reactive and disperse dyes, present challenges related to toxicity and environmental sustainability. This study investigates an eco-friendly dyeing approach for PET/Wool fabrics using natural colorants extracted from annatto seeds. The fabrics were dyed at concentrations ranging from 1–30% owf and at temperatures of 90, 110, and 130 °C for 30 minutes using the exhaust dyeing method. Colorimetric analysis showed that increasing dye concentration and temperature generally produced darker, more saturated shades, as reflected in decreasing L* values. Changes in color strength (K/S) indicated that maximum dye uptake occurred at 90 °C for low concentrations, while higher concentrations (15-30%) exhibited superior absorption at 110 °C, with limited additional benefit at 130 °C. Fastness evaluation revealed that wash and rubbing fastness improved significantly at elevated temperatures due to enhanced dye penetration into both fiber components. Sublimation fastness similarly increased with temperature, indicating stronger dye–fiber interactions at 110 and 130 °C. Light fastness remained the principal limitation, with poor resistance across most conditions, showing only moderate improvement at low dye concentration (1%) and the highest dyeing temperature. Overall, the findings highlight dyeing temperature as a critical factor in optimizing color depth and fastness performance in annatto-dyed PET/Wool blends. The study demonstrates the potential of annatto as a natural and environmentally benign alternative for producing moderately fast and aesthetically appealing shades on polyester–wool blended textiles.

Keywords: One bath dyeing, Annatto dye, Polyester-wool fabric, Color Fastness, Colorimetric Analysis.

1. Introduction

It must be emphasized, both in aspects referring to global transformation and the human elements of triggering severe climate change, pollution from several sources, advances made by industries, and degradation of ecosystem resources that have much affected Earth's long-term sustenance [1-3]. The dyeing and finishing sectors in the textile industry rely heavily on synthetic dyes and chemicals, which pollute the aquatic environment by discharging wastewaters [4, 5]. The dangers posed by synthetic dyes arise not only due to their toxic, lethal tissue-destructive capability but even with inflammatory reactions at the local level [2]. Natural dyes, on the other hand, have cancer-free properties, biodegradability, and several other benefits to the environment.

The increasing trend towards sustainable textile practices in recent times has also revived natural dyes [2, 6]. A case in point of natural dyes is annatto, which is obtained from the seeds of the Bixa orellana plant [7-9]. Figure 1 presents a comprehensive visual overview of the annatto plant (*Bixa orellana L.*), highlighting its botanical features and the molecular structures of its key pigments. The image includes the leafy annatto plant, its distinctive spiny red fruits, and the small reddish-brown seeds nestled within, which are the primary source of natural colorants. These seeds are coated with a resinous layer rich in carotenoids, notably bixin and norbixin [10, 11]. Carotenoids, aliphatic compounds, flavonoids, polyphenols and tannins, quinones, and triterpenoids have been found as phytochemicals in the annatto [7, 12]. The chemical structure of bixin, shown in the figure, is a lipophilic apocarotenoid with a long conjugated polyene chain and a methyl ester group, responsible for its reddish-orange hue and solubility in organic solvents. Norbixin, also depicted, is the water-soluble dicarboxylic acid derivative of bixin, formed through saponification, and exhibits a yellow-orange coloration [12-14].

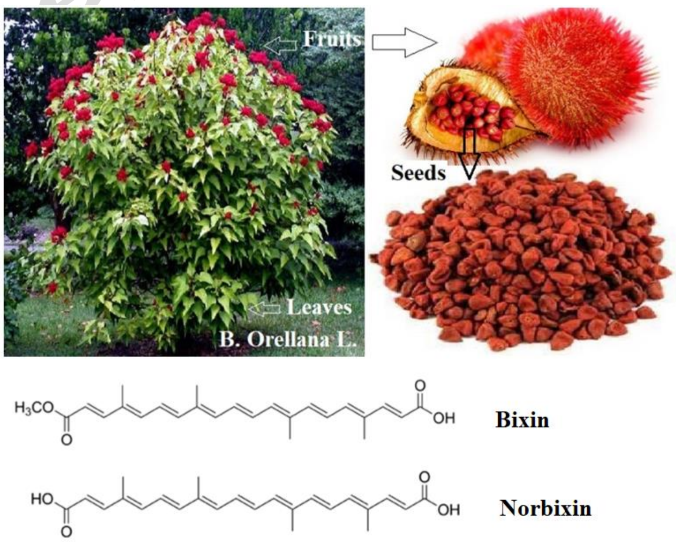


Figure 1. Bixa Orellana L. plant, fruits, and seeds and chemical structure of bixin and norbixin (C.I. Natural Orange 4).

Extracting and dyeing processes are fairly inexpensive, and quite fast consume lower environmental time in comparison to synthetic ones. In addition, using eco-friendly mordants and optimizing dyeing conditions can further reduce the much ecocide footprint of the dyeing techniques [15-18]. Annatto dye is primarily extracted from the resinous pericarp of Bixa orellana seeds using various methods, with solvent-based and microwave-assisted techniques being the most effective [13, 19, 20]. Traditional extraction involves boiling the seeds in water or acidic media, while modern approaches utilize organic solvents such as ethanol, methanol, and ethyl acetate, or employ ultrasonic radiation or microwave-assisted aqueous extraction to enhance pigment yield [7, 13, 21-24]. The process parameters-temperature, time, and seed-to-solvent ratio—are optimized using statistical models like Box-Behnken design or response surface methodology to maximize the yield of bixin (fat-soluble) and norbixin (water-soluble) pigments [14, 25-28]. Microwave-assisted extraction, in particular, improves efficiency by accelerating solvent penetration and pigment release through cell wall disruption, resulting in higher yields with reduced energy and solvent consumption [29, 30]. The solubility and color characteristics of annatto dye depend on its chemical constituents-bixin and norbixin-and the solvent used during extraction. Bixin is lipophilic and dissolves well in organic solvents like ethanol, chloroform, and ethyl acetate, producing reddish-orange hues, while norbixin is hydrophilic and soluble in alkaline aqueous solutions, yielding yellow-orange tones [13]. The extracted dye exhibits pH sensitivity, with color shifting from pale yellow in alkaline media to greenish-yellow in acidic conditions. Spectrophotometric analysis shows maximum absorbance for bixin around 457-487 nm and for norbixin near 453-482 nm, confirming their distinct chromatic profiles [13, 20, 31, 32]. The dye’s color intensity and stability are influenced by solvent polarity, concentration, and extraction conditions, making annatto a versatile natural colorant for food, textiles, and cosmetics. Research topics focused on this feature of this natural dye continued and included the various textile fibers under investigation among those for dyeing on natural (e.g., wool, silk, cotton, jute, pineapple) [33-36] and synthetic fibers (e.g., polyester, nylon, acrylic, viscose) [8, 31, 37-42]. Among the earliest studies concerning the emerging importance of new fibers and this material in blend with wool was written by Dennison and Leach back in 1952. They wrote a report that emphasized many advantages possessed by fabrics of a polyester/wool blend [43]. This was derived from the knowledge that the then much-quoted ratio of 55:45 polyester/wool blend was the minimum polyester content necessary for permanent pleating in wool blends [44, 45]. Thus, every fiber would require a different dye due to difference in fibers in physical and chemical composition, particularly in polyester and wool [46, 47]. Polyester does not absorb the soluble anionic dyes (acid, metal-complex, or reactive dyes) to which wool is generally subjected; however, the superficial staining presents an opportunity for poorly fastness-withstanding rubbings and high luminosity exposures [48]. Two baths of dissimilar dyes can be used in this case, or residents may use one bath with all necessary auxiliary agents [48]. There would, therefore, be an apparent encouragement to dye a polyester-wool fiber blend without a dye. In relation to terms of the quality of colors, annatto dye appears to have very good compatibility with wool and polyester fabrics, where colors range from orange to yellow. Factors like dye concentration, temperatures, and pH determine the color strength (K/S) values. The dyeing is indeed affected by both the temperature and the pH of the medium. In general, dye exhaustion and yield increase with increasing temperatures, though excessive heat may damage the dye. The optimal pH for annatto dyeing lies between 4.5 and 6, where the dye molecules will most effectively interact with the fabric [37, 40, 42]. Annatto dye concentration is very important in influencing the shade and intensity of the color. Higher concentrations lead to darker colors, whereas more intensive dye concentration may lead to aggregation and decreased fastness levels. Optimal dye concentration is mostly determined experimentally balancing color yield and fastness properties [42]. Wool-polyester fabrics are generally best dye-treated at temperatures ranging between 80 and 130 ^{o}C, with pH readings around 4.5 to 6. These conditions improve the interaction between the dye and fabric, thus producing deeper and richer colors [37, 40, 42]. Hence, this study aims at evaluating the viability of employing a single natural dye in body dyeing a polyester-wool blended fabric in one dye bath.

The dyeing properties of annatto on wool-polyester fabrics may vary according to the type of fabric and the blend ratio. Dye uptake and retention would be higher for wool, a protein fiber, compared to polyester, a synthetic fiber. The blend ratio of wool versus polyester may also have an effect on the overall color yield and fastness, thereby requiring condition adjustments during dyeing to obtain results that are as consistent with desired targets as possible.

2. Experimental

2.1. Materials and Methods

The woven fabric composition consists of 55% polyester and 45% wool fiber and was provided by Motahari Spinning and Weaving Co. Iran. The details of the fabric used in this study are shown in Table 1. The natural dye annatto was obtained from YaseSpid Company (Iran). The extraction of dye from annatto seeds was achieved using a sodium NaOH solution (0.5 M) at a temperature of 60^{o}C for a duration of 5 hours with a weight-to-solvent volume ratio of 1:10. Thereafter, the solution was filtered and the resultant solution neutralized with HCl (3M) to a final pH of 7. Freeze-drying was then carried out, which converted it into a powder.

Table 1. Fabric details

Fabric	Wool	Polyester	Warp/cm	Weft/cm	Yarn count	Wool fineness	Polyester fineness
Wool/Polyester	45%	55%	27	22.5	52/2 Nm	23 microns	3.6 dTex

2.2. Dyeing procedure

Specifically, to remove contaminants and spinning oils, the fabric was boiled for 30 minutes and then rinsed with a non-ionic detergent before dyeing. The polyester-wool fabric, with a composition ratio of 55:45, was dyed in a one bath using annatto dye at concentrations ranging from 1 to 30 % on the weight of the fabrics (owf). It was then added with a dispersing agent (Dekamol PES, China) at the concentration of 1% owf. The tartaric acid solution was used to buffer the dye bath for pH adjustment. Dyeing was done using a dyeing machine of Eco-Dye Laboratory (Xiamen Rapid Co. LTD., China). The initial temperature was set at 40 ^{o}C, while the desired dyeing temperature of 90 ^{o}C was reached gradually at a rate of 1^{o}C/min to 110^{o}C and then 130^{o}C. The dyeing process lasted for this temperature ranges for 30 minutes.

2.3. FTIR analysis

Fourier Transform Infrared spectroscopy (FTIR) studies were conducted on the polyester-wool blend fabric, annatto dye powder, and the annatto-colored fabric. For each of the samples, the respective material was mixed with potassium bromide (KBr) powder in a ratio of 1:100 and subsequently hand-pressed under controlled conditions to yield pellets of defined dimensions. The pellets were then placed on the sample-holder of an FTIR spectrometer (Shimadzu, model FTIR-8400S spectrum, Japan) which operated within the 4000-400 cm^{-1} spectral range, with a resolution of 4 cm^{-1} and 8 scans.

2.4. Color characteristics

Measurement of the dyeing color on fabric dyed with annatto dye was made with the use of a Color-Eye 3100 reflectance spectrophotometer (GretagMacbeth, USA), attuned through a computer. In this system, L* is the lightness (where 0 is the darkest and 100 is the brightest), a* indicates the red- green axis, b* denotes the yellow-blue axis, C indicates the color purity and ho the hue angle. Every measurement gave three separate points where the average was automatically computed. The samples were compared on the basis of color strength (K/S value at 440) derived from their reflectance values (R) at the wavelengths of the greatest absorption by Kubelka-Munk's equation (Eq. 1).

$$\frac{K}{S} = \frac{(1-R)^2}{2R} \qquad (1)$$

The total color difference (ΔE) was utilized to compare fabrics dyed with annatto at a concentration of 1% and other concentrations. The values were calculated as follows (Eq. 2):

The measurement of total color difference (ΔE) was made for fabrics dyed in annatto at the concentration of 1% and various concentrations. The values were calculated as follows:

$$\Delta E = \sqrt{\{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2\}} \qquad (2)$$

The color difference, represented as ΔL*, indicates the variation between the L* values of the fabric dyed at 1 % owf and those dyed at other concentrations (e.g. 5, 10, 15, 20, and 30 % o.w.f.). The values of Δa* and Δb* were computed based on the a* and b* values of the fabrics dyed at 1% owf in comparison to those dyed at the other concentrations.

2.5. Color fastness

Wash fastness of color was analyzed based on the prediction of ISO-105-C06:2010 whereby the color change was assessed using the ISO 105-A02 gray scale (1-5). ISO 105-B02, 2014 was used to measure light fastness under Xenon Test Chamber (Reessanj Co., Model XTC97) of 1000 W original xenon lamp. Determination of color fastness to dry heat (sublimation) was done in compliance with ISO 105-X11; 2001. Dyed fabric samples are subjected to sublimation at a temperature of 150 ^{o}C ± 2 for a duration of 30 seconds. The difference in color change after sublimation will be rated using a gray scale of 1-5 [49, 50]. An ADSL international Ltd (Shirley developments Ltd. UK) apparatus was used for the rub fastness test (ISO 105 X12, 2016). The tested rubbing fabric was evaluated against grey scale.

3. Results and Discussion

3.1. FTIR Analysis

The FTIR spectrum of polyester-wool (55:45), annatto dye powder, and dyed fabric is presented in Figure 2. Annatto dye has norbixin and bixin as derivatives of carotenoid compounds. The spectral examination of annatto dye displays many peaks that are of considerable importance. A broad O-H stretching band that is characteristic of carboxylic acid groups present inside norbixin and bixin is observed around 3400 cm^{-1} [39, 51] The C-H stretching associated with aliphatic groups is seen around 2920 cm^{-1}. A sharp C=O stretching peak, attributed to the carboxylic in bixin, is noted at approximately 1725 cm^{-1}.

Additionally, the C=C stretching related to conjugated double bonds, typical of carotenoids, is located at around 1620 cm^{-1}. The C-O stretching is, however, in the region of 1250-1000 cm^{-1}, which corresponds to functionalities associated with esters or ethers. Further, there are out-of-plane C-H bending vibrations suggestive of aromatic structures found between 750 and 900 cm^{-1}. Collectively, all these spectral characteristics confirm the presence of hydroxyl (OH), carbonyl (C=O), and conjugated C=C groups in annatto dye [13, 39, 51].

Spectral data reveals a broad peak comparable to the N-H stretch (Amide A from wool) around 3300 cm^{-1} in the context of a blended fabric made from polyester and wool. The C-H stretching band appears in the range of 2950-2850 cm^{-1} in common to both polyester and wool. The strong peak around 1735 cm^{-1} is correlated to the ester carbonyl (C=O stretch) from polyester [39, 52]. The Amide I (C=O stretch from wool) and Amide II (N-H bending and C-N stretch) are recognized at 1640 and 1530 cm^{-1}, respectively [21]. The C-O-C stretching from polyester, along with Amide III from wool, is located at 1240 and 1100 cm^{-1}, respectively [33]. Furthermore, the range of around 700-900 cm^{-1} includes the bending of aromatic C-H associated with the ring of polyester. These spectral characteristics are true to the expected peaks of a wool-polyester blended fabric.

Noteworthy changes between the spectra of dyed and undyed fabric comprise that wider and possibly sharper peak at 3400 cm^{-1} close to being suggestive of an overlap of O-H and N-H stretching, indicating hydrogen bonding between the dye groups and the fabric mostly being wool. There was also evidence for a slight broadening or intensity increase within the peak between 725 cm^{-1} to about 1735 cm^{-1}. Since this broadening would occur because of possible overlap of signals arising from selected functional groups of the annatto dye and from ester structures from polyester, it is suggested that they are likely arising due to the dye. The peak in the range of 1620-1640 cm^{-1} is broader, covering what seems to be a slight shift, possibly due to the π-π or hydrogen bonding interactions between the dye's C=C and the Amide I of wool. Evidence points more towards chemical interactions, probably hydrogen bonding and π-π interactions, being much pronounced among the annatto dye and wool as compared with polyester. Hence, the spectral windows show that adsorption and possibly bonding between annatto and fabric are confirmed, whereas the high hydrophobicity of polyester limits its contribution to the dye-fiber bond. The broadening and shifts of key peaks, specifically around 3400, 1730, and 1650 cm^{-1}, further support a successful dyeing process [39].

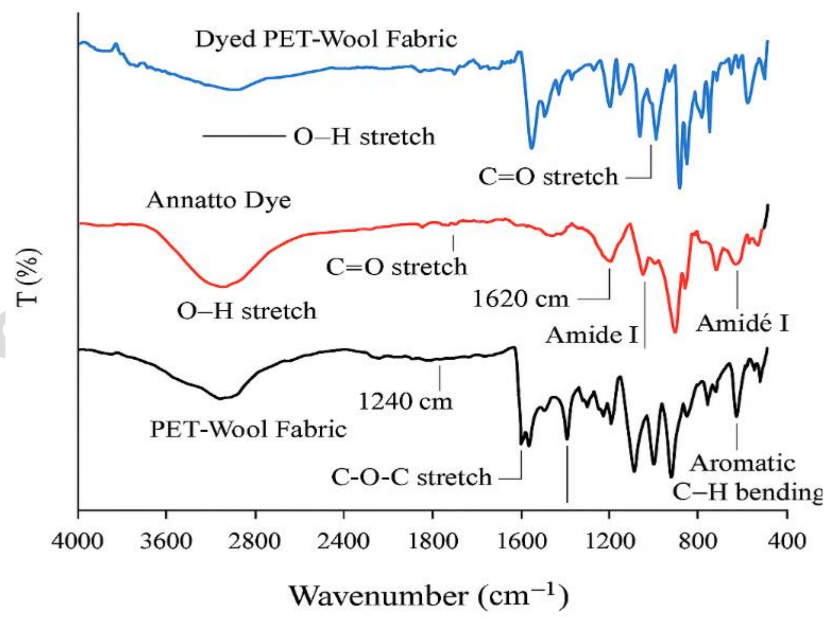


Figure 2. FTIR spectra of polyester-wool blend fabric, annatto dye powder, and the colored fabric.

3.2. Color specifications of dyed fabric at 90 °C

As shown in Table 2, fabrics dyed at 90 °C have their color specifications analyzed. Increase in concentration of annatto dye gives rise to reductions in lightness (L*) and hue angle (h°) while contributing to increases in redness (a*), yellowness (b*), chroma (C*), and color strength (K/S). The fact that L* decreases with increasing concentration indicates that the fabric turns darker. This is indeed true since, with more colorant, more will be absorbed by the fabric. Figure 3 is a digital image showing the dyed fabrics next to the a*b* plot. As the results show, CIElab index profiles for the PET/Wool 55:45 blended fabric dyed with annatto dye exhibit all samples located in the first quadrant of the a*b* plot, while color changes progress from yellow-orange to red-orange with increasing dye concentration.

From the considerable variations in color indices between 1% and 5% owf, it would be inferred that within this range, an increase in dye concentration could lead to significant changes in the color properties of the fabric. The minor differences observed at higher concentrations imply that saturation among the wool fibers is reached, and continued increases in concentration may be less effective. This seems to suggest that saturation level in maximum absorption capacity of wool fibers has been reached, while polyester fibers have lower absorption levels owing to their disposition. Color difference values (ΔE) denote the overall difference in color between samples. $\Delta E=7$ is between 1% and 5% owf, which signifies that a significant difference is observed in color, whereas the $\Delta E=3$ for 5-15% owf and $\Delta E=1$ for 20-30% owf indicate lesser color differences. Wool and polyester fibers usually do not absorb dye in the same manner. Annatto dye is more compatible with wool fibers than polyester, and the absorption of polyester fibers at 90°C is very limited.

Table 2. Color evaluation results of dyed PET/Wool 55:45 blended fabric at 90ºC

Dye (owf%)	L*	a*	b*	c	hº	R_{min} % (460 nm)	K/S	ΔE
1	67.92	14.13	29.46	32.67	64.37	18.13	1.85	-
5	61.68	22.79	42.71	48.41	61.92	9.05	4.57	6.97
10	60.09	24.71	46.73	52.86	62.13	7.24	5.94	8.85
15	55.64	25.73	45.38	52.17	60.45	5.8	7.65	9.95
20	53.69	32.12	49.25	58.8	56.89	4.38	10.44	13.02
30	52.9	33.6	51.26	61.29	56.75	3.64	12.75	14.21

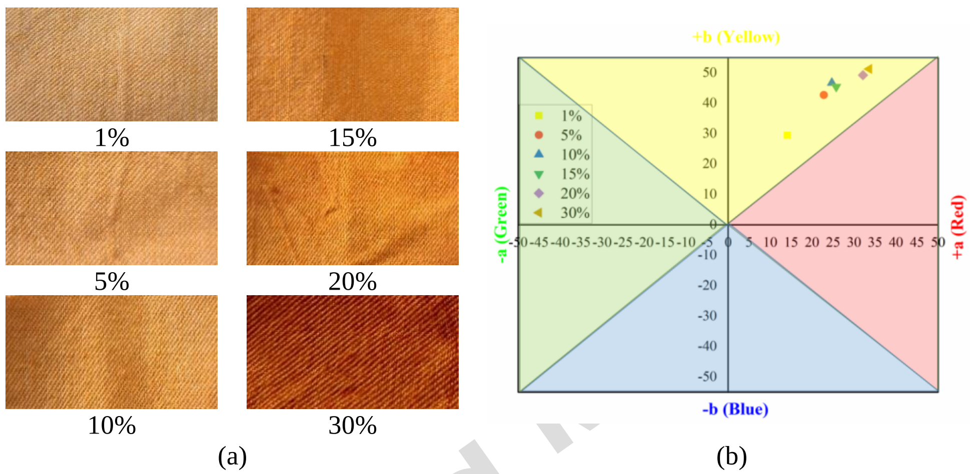


Figure 3. (a) Appearance color and (b) chromatic plot of the dyed PET/Wool 55:45 blended fabric with annatto at 90ºC with dye concentration of 1 to 30% owf.

3.3. Color specifications of dyed fabric at 110 ºC

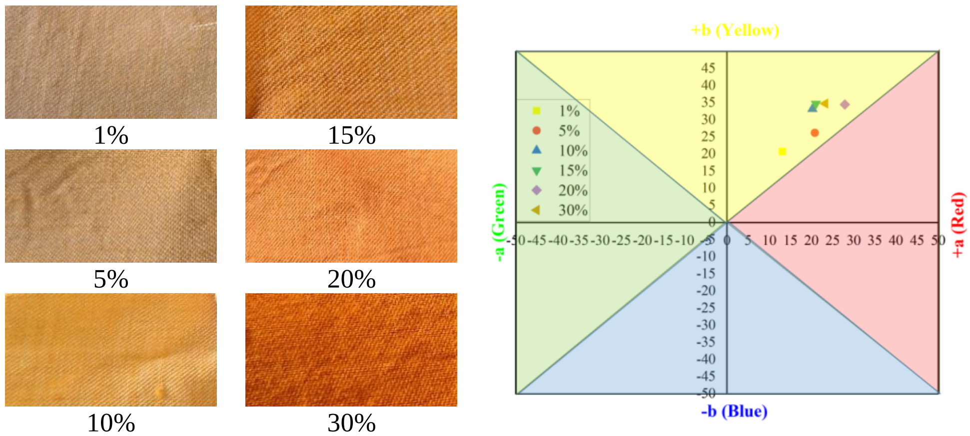
Table 3 details the colorimetric investigations into the PET/Wool (55/45) fabric dyed at 110 °C with different concentrations of the annatto dye. From the results, it can be deducted that, as at 90 °C, an increase in the annatto dye concentration brings improvement in redness, yellowness, purity, color strength, and color differences in the samples while the parameters of lightness and hue angle decrease. Lightness decreases, without exception, as an increasing amount of dye is incorporated. The L* value starts out at 72.67 for 1% on-weight fiber (o.w.f.) dye and at 30% o.w.f. dye falls to a low value of 49.20, indicating that incorporation of a greater concentration of dye darkens a fabric. Minimum reflectance (R_{min}) measured at 460 nm (in the blue region) decreased as dyeing concentration increased as expected since more dye is absorbed, thereby reducing the reflectance. R_{min} falls from 22.35 percent at 1 percent o.w.f down to 5.2 percent for 30 percent o.w.f. Redness increases due to increasing dye concentration. The a* vale increases from 14.59 at 1% o.w.f. to 31.06 at 30% o.w.f., indicating that the hue is shifting toward a redder appearance. Increases in yellowness were also found with increased dye concentrations, but the increase in yellowness was not as strong as that in the redness. The b* value increases from 30.13 at 1% o.w.f. to 38.31 at 30% o.w.f. The initial increase reflected in the chroma (c) as dye concentration increased seems to level off at higher dye concentrations. It starts out as 33.48 for 1% and reaches 49.32 at 30% o.w.f. With an increasing amount of dye being applied, hue angle (ho) is reduced, thus changing hue from a more yellow-orange to a more red-orange consistent with the increasing a* and b* values. The hue angle decreases from 64.16 for 1% o.w.f to 50.97 for 30 % o.w.f. Color strength (K/S), a measure of uptake of a dye or depth of color, showed a substantial increase with increasing concentrations of dye. It increased from 1.35 at 1 % o.w.f. to 9.08 at 30% o.w.f. The total difference (D) increases significantly with the added amount of dye, suggesting the greater change in color than with the 1 % o.w.f. sample. The L from 4.1 at 5% o.w.f to 22.18 at 30% o.w.f. This in turn can be interpreted as saying that the color in higher concentrations is entirely different from the initial 1% concentration.

At 110 °C, wool fibers are subjected to high-temperature dyeing, but they are degraded under such conditions. Furthermore, under the said temperature, a considerable amount of dye absorbed is expected to leach and migrate into polyester fibers. Very nice; at 110 °C the dyeing of polyester fibers is more appropriate than at 90 °C. If the size of dye molecules were smaller, adsorption on the polyester fibers would be greater at this temperature.

The L* values form most of the differences using dye concentrations less than 10% owf, which are accompanied by increased L* values and decreased a* and b* values as compared with the respective fabrics made with the same concentrations at 90 °C. The yellowness of samples dyed at 110 °C was minimally affected by alteration in dye concentration in the bath compared to that of redness. Since annatto dye is defined as an orange dye having parts that are red and yellow, we can also say that the yellow one at 110 °C would remain fairly constant despite this change in concentration at the dye bath, while the red part would have more readily absorption by fibers (Figure 4). As a result, the increase in colorimetric values of redness is anticipated because of that constant yellowness with increasing redness. This is understandable since lower concentrations would be absorbed by the yellow part of the dye into the wool fibers; with higher concentrations, no more are absorbed, or part of the absorbed dye goes loose from the fiber and goes into the solution instead of being resumed by polyester fibers.

Table 3. Color evaluation results of dyed PET/Wool 55:45 blended fabric at 110ºC

Dye (owf%)	L*	a*	b*	C	hº	R_{min} % (460 nm)	K/S	ΔE
1	72.67	14.59	30.13	33.48	64.16	22.35	1.35	-
5	67.70	13.87	32.48	35.32	66.87	16.97	2.03	4.1
10	64.83	18.14	38.23	42.32	64.62	12.42	3.09	6.69
15	56.24	25.36	38.86	46.4	56.87	7.53	5.68	14.84
20	51.66	28.32	37.58	47.06	52.60	6.05	7.29	19.34
30	49.20	31.06	38.31	49.32	50.97	5.2	9.08	22.18



(a) (b)

Figure 4. (a) Appearance color and (b) chromatic plot of the dyed PET/Wool 55:45 blended fabric with annatto at 110 ºC with dye concentration of 1 to 30 % owf.

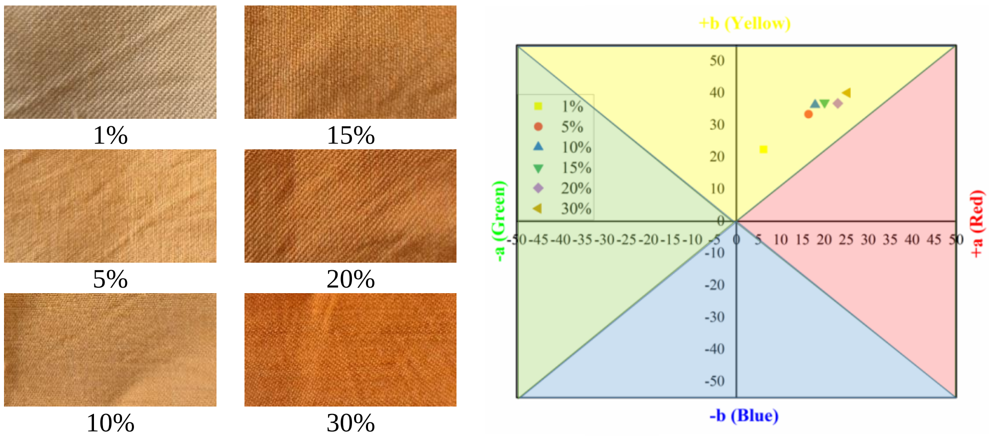
3.4. Color specifications of dyed fabric at 130 ºC

Dyeing polyester fibers using 130 °C is generally considered appropriate since the temperature opens the fiber structure and creates spaces for the dye to penetrate. For wool, however, this temperature is unfavorable: that is, high temperature can damage wool fiber structure, and dyes escape from the fiber structure into the dye solution or penetrate into neighboring fibers. The results of annatto dye uptake by PET-wool 55:45 blended fabric, as shown in Table 4, were less than those of the previous two temperatures (90° and 110°).

Figure 5 shows the dyed fabrics and an a*b* plot corresponding to these samples at this temperature. As a rule, an increase in the percentage of dye (Dye (o.w.f. %)) goes along with an increase in K/S (color intensity), ΔE (overall color change), a* and b* values (color coordinates of the L*a*b* color space), while simultaneously diminishing R_{min} (minimum reflectance) and L* (brightness). This suggests that the higher the dye content, the darker and more saturated the fabric is becoming. The ΔE measures the color differences between the dyed test sample and the undyed control; thus, a higher ΔE signifies a larger perceived color difference. In the present study, the ΔE increased from 9.77 at a 5 % dye concentration to 17.39 at a 30 % concentration, thus indicating that aging concentration results in higher color change of the fabric. These K/S values signify the dyeing extent the fabric absorbs, such that the higher the K/S value, the more intense the color of the fabric. In this study, K/S values increased from 2.64 at 5 % dye concentration to 6.91 at 30 %, demonstrating that color intensity increases with higher dye concentrations. R_{min} indicates reflectance at maximum absorption wavelength from the fabric surface whereby lower R_{min} signifies increased absorption. Herein, R_{min} reduced from 13.98 at 5 % dye concentration to 6.35 at 30 %, thus indicating higher absorption at higher dye concentration. Finally, the L* value, which denotes brightness, decreases with increasing dye percentages, reinforcing the point that higher percentages of dye concentration bring about darker fabric coloration.

Table 4. Color evaluation results of dyed PET/Wool 55:45 blended fabric at 130 ºC

Dye (owf%)	L*	a*	b*	c	hº	R_{min} % (460 nm)	K/S	ΔE
1	75.98	6.21	22.43	23.27	74.52	31.81	0.73	-
5	64.16	16.41	33.41	37.22	63.84	13.98	2.64	9.77
10	64.22	17.9	36.34	40.51	63.77	12.93	2.93	10.94
15	58.18	20.03	37.04	42.11	61.60	9.01	4.59	13.66
20	54.28	23.08	36.79	43.92	56.90	7.22	5.96	16.35
30	54.13	25.27	40.1	47.4	57.78	6.35	6.91	17.39



(a) (b)

Figure 5. (a) Appearance color and (b) chromatic plot of the dyed PET/Wool 55:45 blended fabric with annatto at 130ºC with dye concentration of 1 to 30% owf.

3.5. Color strength and lightness

Lightness data of dyed fabrics are represented in column graph format, allowing comparison of results. The results show that the lightness of the fabric decreases with an increasing amount of dye being absorbed during dye application, with lower dye absorption being noted in respect of the L* values pertaining to the fabric dyed at 130 °C. Generally, an increase in dye concentration in the dye bath leads to decreased lightness of the samples, and the decreasing tendency is felt more at lower dye concentrations. On the contrary, decrease in lightness is at a more gradual slope for dye concentrations above 15 %. It can be inferred that dyeing fabric at a temperature of 130 °C with dye concentrations greater than 15 % does not show a significant difference in lightness among any of the samples. It is also of interest, below 20 % concentration, the lowest brightness levels of dyed fabrics correspond to samples dyed at 90 °C, whereas for above 20 %, it is at 110 °C that the samples show the lowest brightness.

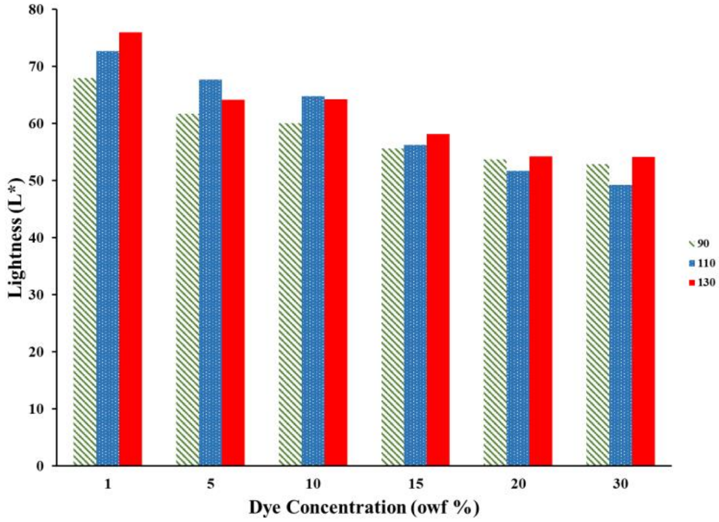


Figure 6. The Lightness (L*) vs. dye concentration of dyed PET/Wool 55:45 blended fabric at 90, 110, and 130 ºC.

The Results of the Color Strength of Samples Dye at 90, 110, and 130 °C with varying dye concentrations present in Figure 7. An increase in the concentration of dye favors increasing the color strength (K/S) of the samples. All exhibits maximum dye absorption at a dyeing temperature of 90 °C with the highest K/S value to the other two temperatures, irrespective of concentration employed. The concentration used ranges from 1-10 %; raising the temperature from 110 to 130 °C does not change the color strength significantly. From 15% to 30% o.w.f., however, a dying temperature of 110 °C gives a superior result for dye absorption to that at 130 °C. Therefore, one can say a concentration of dye does not really influence much the color strength of dyed fabrics at 130 °C.

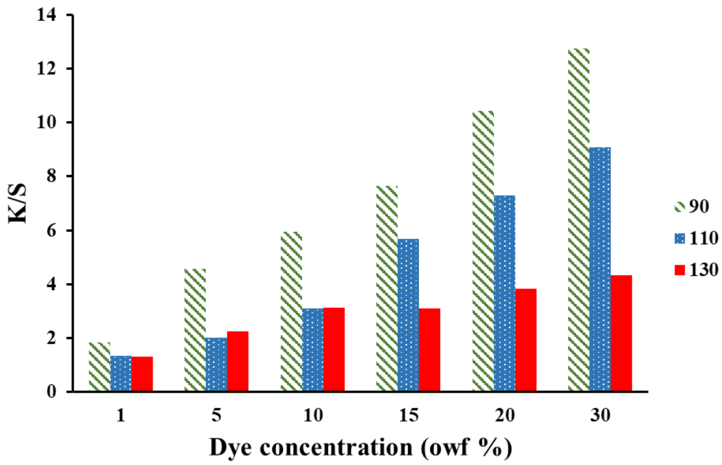


Figure 7. The color strength (K/S) vs. dye concentration of dyed PET/Wool 55:45 blended fabric at 90, 110, and 130 ^oC.

3.6. Color fastness to washing

The fastness of fabric dyed with annatto dye towards washing is shown in Table 5. The wash fastness variable shows that at 90 ^oC, the fabrics dyed exhibit weak stability to washing, which indicates large changes in the color after washing, with fastness rated between 2 to 3. Increased dyeing temperatures have been proven to improve color fastness in the fabrics; that is, at 110 ^oC and, in particular, 130 ^oC, the color fastness against washing is rated between 4 and 5. The results show that at 110 ^oC, an increase in dye concentration in the dye bath corresponds to an increased absorption of dye; in relation to this, such absorption is only superficially deep and does not strengthen within the fiber, making it easily removed during the washing process. Such findings could also be explained that the dyed fabric consists of both polyester and wool fibers, each of which would exhibit different modes of dyeing. At 90 ^oC, wool fibers absorb more of the dye than do polyester fibers. Furthermore, the data on color fastness indicates that the fusion of annatto dye to wool fibers is termed to be "weak interaction". The chemical structure of the dye suggests a lopsided number of specific functional groups capable of forming ionic or hydrogen bonds with the decolorized wool fibers, hence indicating that the bonding of annatto dye onto wool fibers mainly occurs under van der Waals forces. At higher temperatures, the relative contribution of polyester fibers surpasses that of wool fibers in the absorption of annatto dye. Considering the small size of its molecules, the said dye finds will find more openings to penetrate the structure of polyester fibers even at temperatures above 110 °C. It should also be noted that full penetration of dye into the fiber structure cannot be realized, owing to the compact structure of polyester and a dyeing duration of 30 minutes. However, the dye that does penetrate the fiber structure shows a good wash fastness. Thus, the color fastness of fabrics dyed at 130 ^{o}C is higher than that of fabrics dyed at the lower temperatures, such as 90^{o}C and 110^{o}C.

Table 5. Wash fastness of the dyed PET/Wool 55:45 blended fabric according to Color Eye 3100, D65/10/IN

Temp. (^{o}C)	Dye conc. (% owf)	ΔL*	ΔC*	Δh^{o}	ΔE	W.F
90	**1**	3.76	-7.16	3.02	8.63	2
	5	2.24	-3.95	1.38	4.74	3
	10	2.18	-5.37	0.24	5.80	3
	15	2.59	-5.04	1.01	5.75	3
	20	4.83	-6.26	4.17	8.94	2
	30	4.20	-8.54	2.81	9.92	2
110	**1**	-1.35	-0.96	0.04	1.66	4
	5	0.53	-1.03	0.63	1.32	4-5
	10	0.34	0.56	0.85	1.07	4-5
	15	2.87	-0.14	3.40	4.45	3
	20	3.79	0.21	4.31	5.74	2-3
	30	5.01	-1.86	5.25	7.49	2
130	**1**	0.84	-1.26	0.16	1.53	4
	5	0.45	-1.44	0.22	1.52	4-5
	10	-0.65	-1.55	0.64	1.79	4
	15	2.87	- 0.14	3.40	4.45	3
	20	1.21	1.96	1.50	2.75	4
	30	1.26	-0.07	1.93	2.30	4

3.7. Color fastness to light

Light fastness measures the resistance of a fabric toward fading or discoloration upon exposure to light. The standard light fastness scale operates on a scale of 1-8, where 1 corresponds to the least resistance, and 8 indicates the highest. Light fastness of the PET/Wool 55:45 blended fabric dyed with annatto is presented in Table 6. The results clearly indicate a compromise in light fastness by the annatto dye on this polyester-wool blend, and this concurs with other studies. Nakpathom et al. found that fading of polyester fabric dyed with annatto had occurred rapidly during the first 8 hours of xenon arc light exposure and the fading thereafter took place at a relatively low rate. The control annatto-dyed sample was rated as fair, scoring 3 on the blue wool scale.

Broadly speaking, light fastness is expected to decrease or remain constant with temperature; higher temperature generally means lower light fastness (Table 6). In addition, higher dye concentration lowers light fastness, and at 90°C, light fastness at first decreases with increased dye concentration and stabilizes thereafter. This could be attributed to poor dye penetration into the fiber structure. Dyes lying on the fiber surface are more susceptible to fading by light radiation due to the light-sensitive nature of the chromophore structure. At 110°C, light fastness remained consistently low for all dye concentrations, but at 130°C the 1% dye concentration showed a light fastness rating of 6, giving it relatively good light resistance. Beyond that concentration, with increasing dye concentration, a declining trend of light fastness rating is evident from 3. Remarkably, light fastness at 1% dye concentration at 130°C was better than that at lower temperatures. It also finds that, with higher concentrations of dye (above 5%), stability against light was generally poor for all temperature conditions investigated.

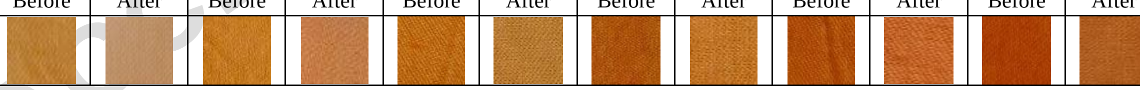
Table 6. Light fastness of the dyed PET/Wool 55:45 blended fabric according to Color Eye 3100, D65/10/IN

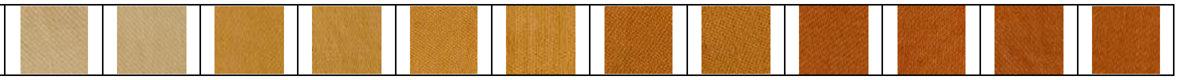
Temp. (ºC)	90ºC		110ºC		130ºC	
Dye conc. (% owf)	**ΔE**	**Light fastness**	**ΔE**	**Light fastness**	**ΔE**	**Light fastness**
1	13.8	4-5	15.2	4	7.3	6
5	19.2	3	15.0	4	19.8	3
10	15.9	4	18.6	3	20.0	3
15	24.4	3	23.4	3	18.5	3
20	26.3	3	27.4	3	23.2	3
30	26.1	3	22.5	3	16.6	4

3.8. Color fastness to dry heat

Sublimation fastness (S.F.) increases with higher dyeing temperatures, according to Table 7. This means that at higher temperatures, the penetration of annatto dye into the fiber and its adhesion are improved, further enhancing sublimation fastness. There seems to be an extremely significant impact of concentration on ΔE values and S.F. at lower temperatures, specifically at 90 °C. This explanation may be inversely correlated to more uneven uptake being favored at lower temperature, very much deserved by the concentrations prior to dyeing. At increased temperature, however, concentration may pose decreasing influence toward uptake. Changes can be expressed as ΔL*, Δa*, and Δb*; each figure has meaning in understanding the color transformation due to sublimation. The generally positive ΔL* values indicate that the sublimated fabric should be just slightly lighter than before sublimation. A less consistent pattern of Δa* suggests that the red-green balance could also be shifting during sublimation; with predominant overall negative Δb* values suggesting a slightly bluer color shift post-sublimation. The sublimation fastness is relatively low at 90 °C (sublimation fastness S.F. = 3-3.4) and increases to 4-5 at 110 and 130 °C. Considerable color difference (high ΔE value) is highly noticeable at dye concentrations of 10 % and 30 % at 90 °C. On the other hand, the changes decrease at 110 °C and 130 °C while being constant at all dye concentrations. In this regard, relatively stronger values of negative Δb* in samples dyed at 130 °C compared to those dyed at 90 °C led to the conclusion that the retention of the yellow component has been enhanced. It is clear that dyeing at 90 °C is insufficient for achieving acceptable sublimation fastness indications for annatto dye on wool-polyester blends. While, dyeing at 130 °C generally yields better results, the practical significance across all concentrations may be negligible. Thus, the analysis should consider the cost versus benefits, as increasing the temperature during the dyeing process will lead to higher energy consumption.

Table 7. Sublimation fastness of dyed polyester-wool (55:45) fabrics measured using Color Eye 3100 (D65/10/IN).

Dye (%)	**1**		**5**		**10**		**15**		**20**		**30**	
	Dyeing temperature of 90 °C											
ΔL*	4.15		2.29		3.5		10		0		6.04	
Δa*	-5.06		-2.02		-7.24		-5.08		-0.46		-9.64	
Δb*	-13.43		-15.66		-15.07		-8.9		-13.08		-14.65	
ΔE	14.94		15.95		17.08		14.13		13.09		18.54	
S.F	3		3-4		3		3		3-4		3	
	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After
Samples												
	Dyeing temperature of 110°C											
ΔL*	1.76		2.46		-1.09		1.1		2.05		0.23	
Δa*	1.62		-1.24		-1.2		0.7		3.9		1.61	
Δb*	-3.06		-1.76		-4.59		-3.47		2.56		1.86	
ΔE	3.88		3.27		4.86		3.71		5.09		2.47	
S.F	5		5		4-5		4-5		4		4-5	
	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After
Samples												
	Dyeing temperature of 130°C											
ΔL*	0.05		2.24		1.8		3.96		1.32		2.08	
Δa*	0.01		-1.24		-1.65		-0.51		0.06		-0.71	
Δb*	1.49		0.86		-3.49		2.49		-0.26		-1.17	
ΔE	1.49		2.71		4.26		4.71		1.35		2.49	
S.F	5		4		4		4-5		4-5		4-5	
	Before	After	Before	After	Before	After	Before	After	Before	After	Before	After

Samples	

3.9. Color fastness to rubbing

The fastness values to rubbing of PET/wool samples dyed with annatto are given in Table 8. The rubbing fastness values obtained for the PET/Wool (55:45) blended fabric dyed with annatto demonstrate clear dependencies on both dyeing temperature and dye concentration. In general, the fastness grades ranged from 3-4 to 5, indicating moderate to very good performance for a natural dye. The results highlight the complex behavior of annatto components (primarily bixin and norbixin) when applied to a blend of hydrophobic polyester and protein-based wool fibers.

Temperature had a pronounced influence on rubbing fastness. At 90°C, rubbing fastness was generally lower, especially at concentrations above 5% owf, where most samples exhibited ratings of 3-4. This temperature is sufficient for wool dyeing but remains below the threshold required for significant dye diffusion into PET, which has a compact and highly crystalline structure. As a result, a greater portion of annatto pigment remains on the fiber surface, increasing susceptibility to mechanical removal during rubbing. Rubbing fastness improved substantially at 110 and 130 °C, particularly for lower dye concentrations. At 130 °C, the sample dyed at 1% owf reached a grade of 5, and several others achieved 4-5, indicating excellent stability. These findings align with previous studies showing that natural dye uptake in PET increases at temperatures above 110 °C due to enhanced chain mobility and fiber swelling, which facilitate deeper pigment penetration. Therefore, the higher temperatures reduce the amount of loosely bound dye on the surface and consequently improve rubbing resistance.

The influence of dye concentration followed the typical behavior observed for natural dyes. Increasing annatto concentration beyond 10 % o.w.f. did not improve rubbing fastness; instead, the values remained within 3-4, regardless of temperature. This can be attributed to saturation of the fiber surface at high pigment loads. Natural dyes generally exhibit limited diffusion into the fiber interior, especially in polyester-containing fabrics. Once the surface layer is saturated, excess pigment tends to accumulate externally, leading to decreased rubbing fastness. Similar trends have been reported for annatto and other plant-based dyes when applied to wool, cotton, and blended fabrics.

The highest and most consistent rubbing fastness grades were achieved at 5-10% o.w.f when dyed at 110-130°C, indicating an optimal balance between available dye molecules and fiber capacity for absorption and fixation. These conditions minimize the amount of unfixed dye on the surface while allowing sufficient penetration into both fiber components.

The blended structure of the fabric plays a critical role in dyeing performance. Wool readily absorbs annatto due to its amino and carboxyl functional groups, which interact favorably with the dye components. In contrast, PET has limited affinity for annatto unless subjected to high temperature, where its amorphous regions become more accessible. Thus, the superior fastness observed at elevated temperatures reflects improved dye penetration within both phases of the blend. The intermediate scores across all conditions are consistent with the hybrid nature of the substrate, exhibiting performance between pure protein and synthetic systems. The results of this study align closely with existing literature [33, 40, 53]. Prior investigations into annatto dyeing of wool and cellulosic fibers report rubbing fastness values typically between 3 and 4, with improvements observed upon mordanting or higher dyeing temperatures [54, 55].

Research on dyeing polyester with natural colorants also highlights the necessity of temperatures near 130 °C for achieving acceptable fastness levels. In blended fabrics, studies consistently show that optimal performance occurs at moderate dye concentrations due to reduced surface deposition. The current findings support these trends, confirming the critical influence of temperature-driven diffusion and concentration-dependent surface saturation.

Table 8. Dry Rubbing fastness of the dyed PET/Wool 55:45 blended fabric according to Color Eye 3100, D65/10/IN

Dye conc. (% owf)	**Temp. (ºC)**		
	90 ºC	**110** ºC	**130** ºC
1	4-5	4	5
5	3-4	4-5	5
10	3-4	4-5	4
15	3-4	3-4	4
20	3-4	3-4	3-4
30	4	3-4	3-4

4. Conclusion

This study demonstrates that annatto can be effectively used as a natural dye for PET/Wool (55/45) blended fabrics, with dyeing temperature and dye concentration significantly influencing both colorimetric properties and fastness performance. Analysis of lightness (L*) values revealed that increasing dye uptake reduces fabric brightness, with the lowest L* values generally observed at higher dyeing temperatures and moderate to high dye concentrations. While dye concentrations above 15 % produced minimal further reductions in lightness, dyeing at 130 °C consistently resulted in lower L* values, reflecting improved penetration of the water-soluble dye into both wool and polyester components. Color strength (K/S) measurements corroborated these findings, indicating that moderate concentrations at 90 °C provided maximum pigment uptake for low-concentration dyes, while at higher concentrations (15-30 % o.w.f.), 110 °C achieved superior K/S values compared to 130 °C. These results suggest that optimal color intensity is achieved through careful adjustment of temperature and concentration, with limited incremental benefit at the highest temperature. Evaluation of fastness properties demonstrated a similar dependence on dyeing conditions. Wash, rubbing, and sublimation fastness all improved with higher dyeing temperatures, particularly at 130 °C, reflecting deeper dye penetration and stronger anchorage within both fiber types. Light fastness remained the most limiting factor, due to the inherent photolabile nature of annatto chromophores, with higher dye concentrations and lower penetration exacerbating surface exposure and fading. These results emphasize the necessity of balancing color depth with stability, as excessive dye concentrations or suboptimal temperatures may compromise practical durability despite achieving darker shades. Overall, the findings confirm that annatto is a promising natural colorant for PET/Wool blends, capable of producing visually appealing and moderately fast-dyeing fabrics under optimized conditions. Practical challenges, including limited light fastness, high-temperature requirements, and careful control of dye concentration, must be considered to ensure reproducibility and industrial feasibility. The combined analysis of K/S, L*, and multi-dimensional fastness properties provides a robust framework for guiding future eco-friendly dyeing protocols, highlighting strategies for achieving balanced color strength, brightness, and durability in blended textile systems.

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