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# Wood Extracts for Dyeing of Cotton Fabrics—Special View on Mordanting Procedures

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**Abstract:** Natural dyes offer a bio-based opportunity to support the attractive coloration of textile fabrics made from natural fibers like cotton, wool, hemp, and many other textile materials. They can be part of a strategy to realize fully bio-based textiles and clothing materials. In line with this statement, the following study investigates the use of wood extracts for dyeing cotton fabrics. Specifically, extract powders of logwood (*Haematoxylon campechianum* L.), brazilwood (*Caesalpinia* spp.), and quebracho wood (*Schinopsis lorentzii*) are used. The aim of the study is to evaluate which colorations can be obtained by the application of those wood extracts and what fastness properties are reached. For this, different modified process parameters and mordants are evaluated. The dyeing process is modified using different mordants based on iron and aluminum salts. These mordants are applied in pre-, meta-, or post-mordant procedures. The color and fastness properties of prepared textile samples are determined by spectroscopic measurements, color measurements, washing procedures, and a Xenotest for measuring the light fastness. Ultimately, it is shown that a broad range of colorations can be realized through different combinations of wood extracts and mordanting procedures. Notably, stronger color depths are reached with pre- and meta-mordanting compared to post-mordanting. Good wash fastness is obtained for some color shades. However, with post-mordanting, better wash fastness can be achieved. The light fastness of the realized samples is only moderate to low. In conclusion, it can be stated that dyes from wood extracts are excellent materials to dye natural fibers if they are combined with the right mordanting agent in pre- or meta-mordanting procedures. The present study is therefore a good proof-of-concept for the realization of fully bio-based colored textile materials.

**Keywords:** dyeing; coloration; cotton; natural dye; wood extract; mordanting; optical spectroscopy; color measurement; fastness properties



**Citation:** Mai, T.H.; Grethe, T.; Mahltig, B. Wood Extracts for Dyeing of Cotton Fabrics—Special View on Mordanting Procedures. *Textiles* **2024**, *4*, 138–164. https://doi.org/10.3390/textiles4020010

Academic Editors: Ivo Grabchev and Jun Chen

Received: 2 January 2024
Revised: 6 March 2024
Accepted: 28 March 2024
Published: 12 April 2024



## 1. Introduction

Nowadays, the majority of textile fabrics are dyed using synthetic dyes [1–4]. The dominance of synthetic dyes compared to the use of natural dye stuffs is caused by several factors, such as availability, cost issues, and excellent fastness properties [1,4]. Prominent examples for great washing fastness are the application of reactive dyes on cotton or disperse dyes on polyester fabrics [1,5–9]. However, the trend in favor of fully bio-based and biodegradable materials is spreading, and through this, the consumer demand for natural products is increasing [10,11]. Synthetic dyes do not fulfill these demands. In contrast, natural fiber materials like cotton or wool dyed with natural dyes can be part of a strategy to realize a fully bio-based textile product [12–14].

With this background, this study uses extract powder of logwood (*Haematoxylum campechianum*), brazilwood (*Caesalpinia echinata*), and quebracho wood (*Schinopsis lorentzii*) to dye cotton fabrics. The aim of the study is to evaluate which colorations can be achieved by the application of those wood extracts and which fastness properties are obtained. To do this, different modified process parameters and mordants are evaluated. These wood extracts are derived from renewable sources and considered mostly to be non-hazardous. Logwood is an invasive and rapidly spreading tree species. As for brazilwood, its extract produces an orange color. Compared to other wood dyes, quebracho wood is very rich in tannin, which is a natural mordant. Compared to the usage of wood as a coloring agent, the use of extract powder leads to a more reproducible dyeing result and also minimizes transportation costs. In the current study, the effect of dye concentration and mordanting on the resulting color strength and color shade are investigated. To optimize mordanting, different mordanting procedures, mordant types, and mordant concentrations are evaluated. Additionally, color strength, fastness to light, rubbing, and washing are determined.

When using wood extracts for dyeing purposes, it is essential to identify the plant materials correctly by using botanical names to guarantee the reproducibility of sample preparation. In fact, *Caesalpinia echinata*, *C. sappan*, and *Haematoxylum brasiletto* are closely related species. *Haematoxylum brasiletto* can be mistaken with logwood too, and there are many varieties of quebracho trees [15,16]. Logwood belongs to the Fabaceae family; its botanic name is *Haematoxylum* (also known as *Haematoxylon*) *campechianum*, a native plant in the Yucatan Peninsula (Mexico). Due to major economic benefits, many logwood plantations were established in the eighteenth century. Nowadays, logwood trees can be also be found on islands in the Indian and Pacific Oceans, Caribbean islands, Brazil, Australia, India, Malaysia, and West Africa [15,17,18]. Actually, the initiative Plan Alto (ecoforestal y carbono) promotes exclusively logwood and logwood-related products in Mexico as part of a sustainable and environmentally friendly strategy [19]. This type of organization might also be part of a strategy for the development of sustainable dyeing of textile products in the future. Also, for quebracho wood, efforts for reforestation are reported, e.g., in Argentina [20].

The heartwood of logwood contains the chemical component hematoxylin (C. I. 75290; Natural Black 1) [21,22]. The content of hematoxylin in logwood is reported to be in the range of 9 to 12% [21]. Hematoxylin belongs to the category of flavonoids [23]. Pure hematoxylin in the tree is mildly colored, and after oxidation, it is transformed into the coloring agent hematein (compare Figure 1) either by atmospheric oxygen or other oxidative agents [23,24]. There is already a certain amount of naturally oxidized hematein in hematoxylin powder. Hematoxylin dyes are not considered long-lasting, but the discovery of the use of metal salts as mordants to bond the dye chemically to fabrics has offered new chances for fabric dyeing. It is reported that various metallic salts applied as mordant can lead, in combination with logwood extracts, to a variety of colors of dyed fabrics. Colors such as gray to black are obtained with iron salts, green-blue to black with copper salts, blue to black with chromium salts, and violet to gray with aluminum salts. However, the demand for hematoxylin declined in the past due to the emergence of synthetic dyes [25]. Besides applications in the textile area, Hematoxylin finds applications in hair dyeing, histology, and surgical pathology [26,27].

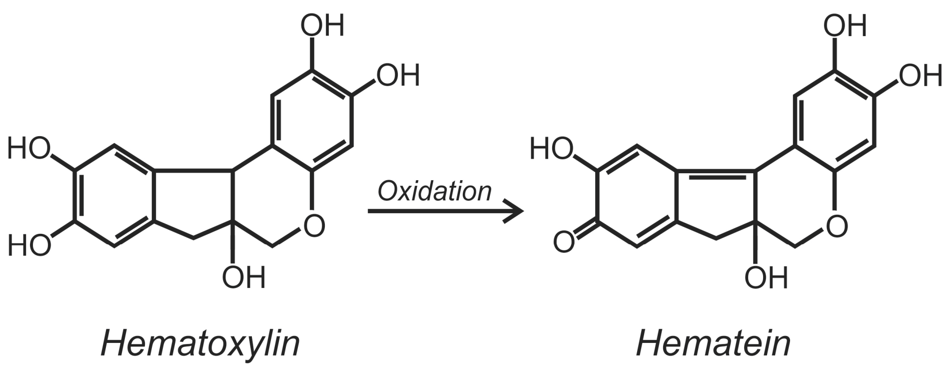


**Figure 1.** Chemical structures of color components in logwood.

Despite being red in color, logwood powder produces brown, violets, greyish brown, and grayish violet in textile dyeing [28]. Previous research by Prasad showed that dyed cotton can exhibit a good washing fastness even without mordant [28]. However, an earlier study by Zarkogianni et al. (2011) indicated that the color change of non-mordanted cotton was quite poor and that pre-mordanting increases wash fastness [29]. Reported data from Prasad showed that without mordant or with same amount of mordants (alum, soda, citric acid, copper, and iron), linen always had better light fastness than cotton [28]. If linen was mordanted with a combination of metallic mordant and oak gall, both wash and light fastness were further improved. Regarding wool, the study by Zarkogianni et al. suggests that, even without mordant, the chroma and color strength of wool were more than double those of cotton [29]. According to the findings of Grifoni et al., the UV protective effect of flax and hemp fabrics could be improved by application of logwood dye [30].

Brazilwood is the common name for *Caesalpinia echinata*, a plant of the Fabaceae family. Its heartwood contains a component called brazilin. In addition to brazilwood, some tree species, including *C. sappan* (sappanwood), *C. violacea,* and *Haematoxylum brasiletto* (peachwood), also have brasilin in their heartwoods. These plants grow in tropical regions such as Brazil, southeast Asia, the Caribbean islands, Central America, and northern South America [15]. Brazilin comes from the same botanical family as hematoxylin. Structurally similar to hematoxylin, brazilin can be easily oxidized to red-colored brazilein (C.I. 75280, Natural Red 24) [21]. The content of brazilin in brazilwood is reported to be quite low at 1.3% [15,22]. However, for other types of woods, the reported brazilin content is significantly higher [15]. Brazilein is attracted to the fiber surface through hydrogen bridges or coordinative bonds in combination with particular metallic mordants. Brazilin is easily converted to brazilein through oxidation with oxygen in the air or other oxidants by forming a carbonyl group [31] (compared Figure 2). A further methylation of brazilein can improve its color stability against changes of pH [32]. There are numerous uses for brazilwood, such as textile dyeing, folk medicine, and histological staining. The population of brazilwood trees was severely overexploited, but strong efforts have been made to rebuild a sustainable plant population [15,25].

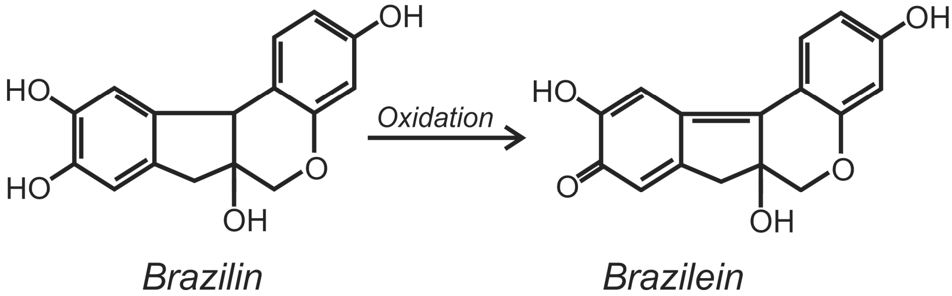


**Figure 2.** Chemical structures of color components in brazilwood.

A comparison between wool and cotton dyed with brazilwood was made by Zarkogianni et al. [29]. The dyed cotton became darker owing to metallic mordants. A pre-mordanting significantly increased the color strength of dyed cotton samples. However, mordanted wool exhibited less improvement in terms of dye adsorption than cotton since its dye adsorption was already significantly better than cotton without mordant. The wash fastness of the dyed cotton was enhanced in most cases with metallic salts used as mordant [29].

Dyeing wool with brazilwood has also been explored in a previous study by Mikropoulou et al. [33]. In this study, it is reported that the usage of mordants leads to an increase in the dye absorption of cotton. However, mordanting does not always improve the washing fastness further. The inclusion of metallic mordants typically only results in a small enhancement in the light fastness of wool. It is reported that for both light and wash fastness, $K_2Cr_2O_7$ and $CuSO_4$ are the most efficient mordants for wool fibers [33].

For more than a century, wild quebracho forests in the Gran Chaco region of Argentina, Bolivia, Chile, and Paraguay have been a source for vegetable tannins and timber [34]. Quebracho wood can contain up to 20% tannins [22,34]. The extract from heartwood chips can be obtained by boiling water, treatment of the warm water-soluble extract with bisulfite, or boiling aqueous bisulfite solution [35]. For this study, the extract used in experiments is *Schinopsis lorentzii*. Two colored components in the heartwood of *Schinopsis lorentzii* were defined, namely catechin and ent-fise-tinidol-4b-ol [35,36]. Their chemical structures are compared in Figure 3 [35].

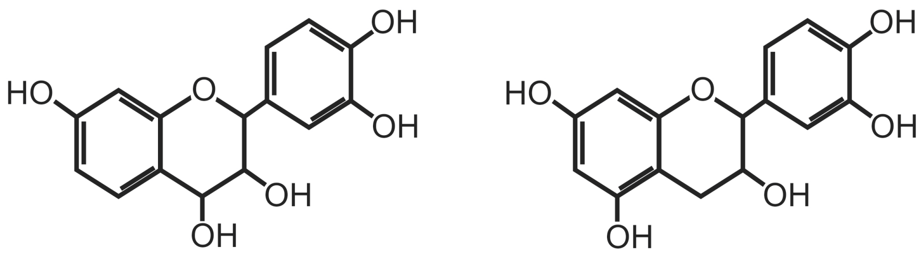


**Figure 3.** Chemical structures of color components in quebracho wood.

Nowadays, tannin from quebracho wood still plays a role in the leather industry. The antibacterial properties of footwear leather may be improved by quebracho extracts [37]. Tannin from *Schinopsis balansae* can be used to produce adsorbent for wastewater remediation due to its capability to absorb cationic pollutants like heavy metals, surfactants, and basic dyes [38]. Quebracho wood is reported to be a victim of overharvesting in several regions [39,40]. Due to this, its use for dyeing purposes may not be considered if fully sustainable dyeing processes are aimed at.

Although there are numerous studies on quebracho, current research in textile dyeing with quebracho remains limited because most studies have exclusively focused on other applications like leather tanning, wastewater treatment, and adhesive manufacturing. Only a small number of reports in the literature consider textile dyeing. For instance, a study by Thakker et al. investigated how plasma surface modification improved the fabric absorbency, color strength, and fastness properties of wool and cotton fabrics which were digitally printed with quebracho-containing ink [41]. Another study by Alves et al. suggested using quebracho extract (*Schinopsis* spp.) in conjunction with aluminum or iron salts in pre-mordanting before dyeing cotton with madder [42]. The findings indicated that K/S values, fastness to washing and UV light, and UV protective properties were enhanced by the pretreatment with quebracho. Utilizing a combination of quebracho and metallic salts is reported to be more effective at improving UV light fastness than solely metal salts. Pre-treated samples with these mordant mixtures also demonstrated better UV protective properties [42].

Quebracho can be used as a dye or as a mordant for cellulosic and protein fibers. To use quebracho as a dye for cotton, dye stuff suppliers suggest quite dissimilar amounts of extract (such as 10%, 20–30% for light to medium colors and pre-mordanting with 7–10% of aluminum acetate; 5–10% for a medium color and pre-mordanting with colorless tannin and 15% alum; and 1–15% for light, medium, and dark colors) [43–46]. In general, they recommend dyeing temperatures of around 85 °C for up to 60 min. Besides aluminum acetate and alum, mordanting with iron is reported to be another useful option. With iron-based mordant, the final coloration is darker [25].

In the current study, the three different types of wood extracts are evaluated for the dyeing of the natural fiber cotton. This evaluation is conducted with the aim of determining which types coloration and levels of color depth can be achieved by the application of these natural dyes. The fastness properties against light, washing, and rubbing are also part of the evaluations. To do this, iron and aluminum salts are considered as mordant agents in different mordanting procedures. Through all of this, this paper aims to be a proof-of-concept that fully bio-based colored textile fabric products are obtainable.

## 2. Experimental Section

### 2.1. Materials

As textile materials, cotton fabrics supplied by Anita Pavani Stoffe OHG (Heuchelheim, Germany) are used. This cotton fabric is plain-woven, undyed, and unbleached. It exhibits a weight per area of 135 g/m$^2$. PERLAVIN NIC, PERIZYM DBS, and caustic soda were supplied by Textilchemie Dr. Petry GmbH (Reutlingen, Germany) and Carl Dicke GmbH and Co. KG(Mönchengladbach, Germany). These three products were utilized for the pre-wash treatment of the cotton fabrics before dyeing was performed. The extract powders of logwood (*Haematoxylum campechianum*), brazilwood (*Caesalpinia echinata*), and quebracho wood (*Schinopsis lorentzii*) were gained from Pflanzenfärber Shop (Hückelhoven-Baal, Germany) (see Figure 4). Iron sulphate ($FeSO_4$ $7H_2O$) and aluminum acetate ($Al(CH_3COO)_2OH$) were obtained from Merck KGaA (Darmstadt, Germany) and Fa. Bernd Kraft GmbH (Oberhausen Germany). Throughout this study, dye extracts as well as metallic mordants were used without further purification. Soft water was applied as a solvent to dissolve the wood extracts and metal mordants.


**Figure 4.** Photographs of the used wood extract powders as supplied.

### 2.2. Sample Preparation

Cotton fabrics were pre-washed using an industrial washing machine HC60 from IPSO (Deinze, Belgium). Both the dyeing and mordanting processes were conducted in a Datacolor Ahiba IR Pro dyeing machine from Datacolor GmbH (Marl, Germany). This is an infrared dyeing machine for textile exhaust dyeing procedures. Five grams of cotton fabric were treated in a dye bath of 50 mL.

#### 2.2.1. Pretreatment

Before dyeing or mordanting, cotton fabrics were washed at 70 °C with a solution containing 3 g/L non-ionic surfactant (PERLAVIN NIC) in an industrial washing machine. After the detergent was added into the washing machine, the pH level of the washing liquor was measured to ensure a pH of 7–8. If the pH was found to be appropriate, 6 g/L PERIZYM DBS was introduced into the washing solution. Finally, the cotton fabric was treated with a solution containing 4 g/L of caustic soda. After treatment, the fabric was thoroughly rinsed with soft water so as to be neutralized as much as possible and was then air-dried at room temperature. The whole washing and rinsing process took 120 min.

### 2.2.2. Dyeing Procedure without Mordant

The pretreated fabric was cut into rectangle pieces with a size of 13 cm × 27 cm, weighing approximately 5 g, and was then soaked in soft cold water for 60 min. Dye solutions were prepared by dissolving wood extracts in 90 °C soft water with a liquor ratio of 10:1. To investigate the impact of dye concentration on the dyeing performance, samples were treated with varying amounts of wood extract, in the range of 2% to 20% of the weight of fabric (owf). The parameters of dyeing temperature, time, and liquor ratio were kept unchanged. As the wood extracts were naturally acidic, the original pH values of dye solutions were mainly in the range from 6.0 to 7.0.

The dye baths were heated from room temperature to 80 °C and then maintained at this temperature for 60 min. The heating rate was set to 2 °C/min. Afterwards, the vessels were cooled down to 50 °C. The whole process took around 90 min. After dyeing, each sample was rinsed in a bath with one liter of soft cold water. Finally, air-drying is performed at ambient temperature.

### 2.2.3. Dyeing Procedure with Mordant

Based on the results of previous experiments without mordant, a specific concentration of each dye was selected for this mordant dyeing process. For dyeing with logwood and brazilwood extracts, the concentration was set to 2% ofw. For quebracho wood, 5% ofw was applied. Cotton fabrics were cut into pieces weighing approximately 5 g and were wetted with soft cold water for one hour. Soft water with a temperature of 90 °C was used for dissolving wood extracts as well as metal salts used as mordants.

Three mordanting methods—pre-mordanting, meta-mordanting, and post -mordanting—were employed to dye cotton fabric samples with wood extracts, using two types of metallic mordant at different concentrations ($FeSO_4$ $7H_2O$ at 0.25% and 3% owf, $Al(CH_3COO)_2(OH)$ at 5% and 10% ofw). The procedures for the different mordanting methods are schematically presented in Figure 5. No adjustment of pH was made. All dyeing and mordanting procedures were carried out with a liquor ratio of 10:1 at the same temperature conditions using an Ahiba IR Pro machine. The temperature was raised from ambient temperature to 80 °C with a heating rate of 2 °C/min. The final process temperature was kept for 60 min. Finally, the bath was cooled down to around 50 °C. The complete dyeing procedure took around 90 min.

In pre-mordanting, wood extracts and mordants were dissolved separately in beakers with a liquor ratio of 10:1. Cotton fabrics were first immersed in metal salt solutions for about 90 min. Then, mordanted samples were lifted from their beakers, given a single rinse with cold soft water to remove loosely bound mordant, and squeezed tightly. Mordanted fabrics were transferred to dye solutions, and the machine ran for another 90 min. When the dye procedure was completed, excess dye on the fabrics was removed by rinsing three times with soft cold water. For meta-mordanting or simultaneous mordanting, samples were treated in dyebaths containing both dyestuff and mordant. To prepare the liquor mixture, dye extract was added in the form of an aqueous solution to the mordanting solution. In the post-mordanting technique, dyeing and mordanting processes were the same as described in the pre-mordanting method but were carried out in the reverse order. Cotton fabric was first impregnated with a dye liquor and then with an aqueous solution of mordant. The fabric samples were washed once with soft cold water to remove unfixed dye before mordanting treatment. For all mordanting methods, the final samples were rinsed three times with soft cold water and dried at room temperature.

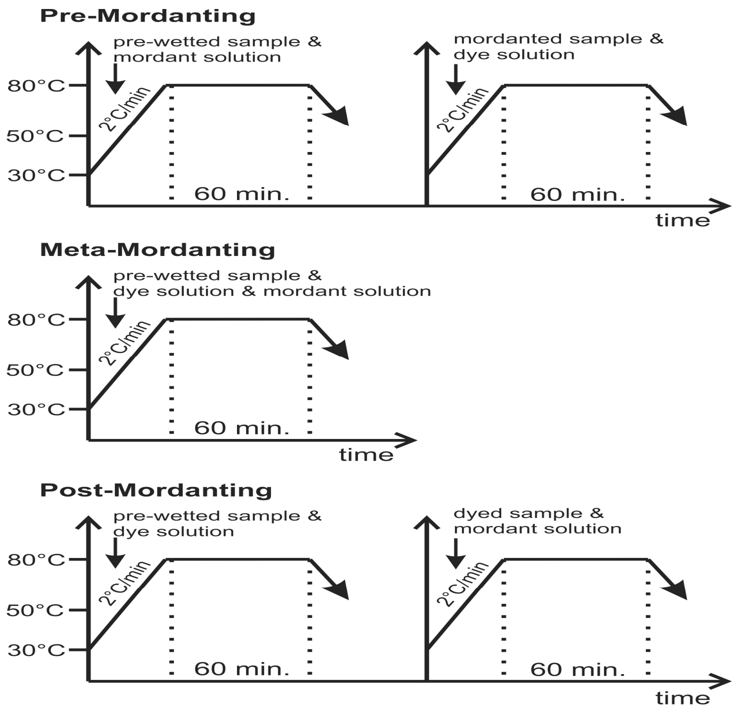


**Figure 5.** Schematic diagrams and temperature profiles for the three different processes with mordant.

## 2.3. Analytical Methods

Color measurements were performed using a Datacolor 400 Spectrophotometer supplied by Datacolor GmbH (Marl, Germany). For washing, rubbing, and light fastness tests, an Ahiba Polymat dyer supplied by Datacolor GmbH (Marl, Germany), a James Heal crockmeter (James Heal, Sterling, VA, USA), and an Atlas Xenotest Alpha LM instrument (Atlas Material Testing Technology GmbH, Linsengericht-Altenhaßlau, Germany) were employed. Color change and color staining during these fastness tests were assessed in an Instrumental Colour Systems Multilight cabinet (VeriVide Limited, Leicester, UK) with an artificial daylight D65 lamp. The detailed procedures for the three different fastness tests are presented in the following three sub-sections.

### 2.3.1. Washing Fastness Test

To assess color fastness to washing, ISO 105-C06:2010 standard was applied [47]. The temperature of the washing water was set to 40 °C, while the washing duration was 30 min. As a washing agent, 3 g/L non-ionic PERLAVIN NIC detergent was used. For the washing test, a 4 × 10 cm sample fabric was attached to a piece of multifiber-adjacent fabric (wool—WO, acrylic—PAC, polyester—PES, polyamide—PA, cotton—CO, or cellulose acetate—AC) along one of its shorter edges, with the adjacent fabric facing the right side of the specimen. Please compare the examples of washed samples in Figure 6. They were washed separately in stainless steel beakers in the above-mentioned Ahiba Polymat dyer. The liquor volume in each steel beaker was set to 150 mL, and 10 steel balls were added to it. After a single washing test, the samples were rinsed three times with distilled, hard, and soft water and were then air-dried. The color loss of specimens and the staining of adjacent fabrics were graded by comparison with the grey scales ISO 105-A02:1993 and ISO 105-A03:2019, respectively (values 1–5, where 1—poor, 2—fair, 3—good, 4—very good, and 5—excellent) [48,49]. The dyed samples were folded into two layers to make them opaque while evaluating.

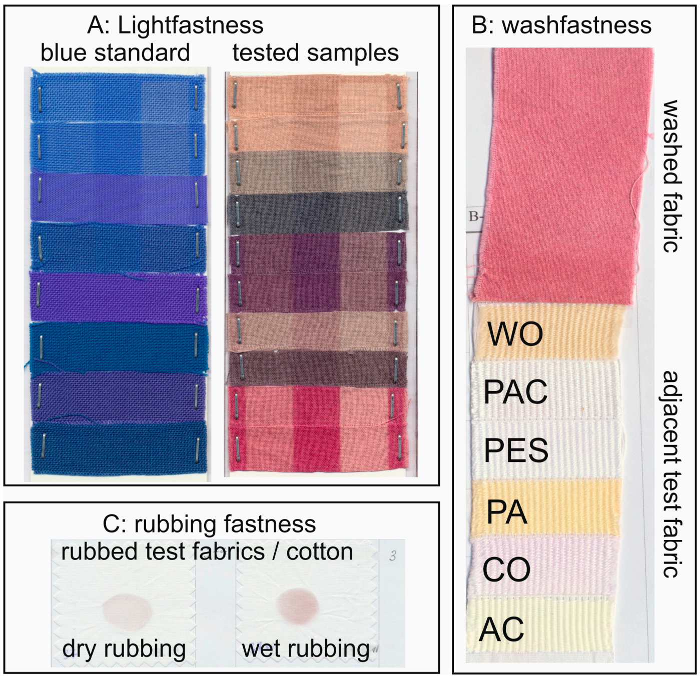


**Figure 6.** Photographs of different samples in the fastness test; examples illustrate the procedures of the used fastness tests—(**A**) lightfastness; (**B**) washfastness; and (**C**) rubbing fastness.

### 2.3.2. Rubbing Fastness Test

Rubbing fastness was evaluated according to ISO 105-X12:2016 on a James H Heal crockmeter [50]. A dry, bleached cotton cloth measuring 5 × 5 cm was rubbed back and forth on the dyed specimen twenty times. Please compare the examples of rubbed cotton specimens in Figure 6. For wet rubbing, the rubbing cloth was wetted with distilled water before conducting the test. The greyscale, complying with ISO 105-A03:2019, was used to rate the staining on the rubbing cloths (rating range 1–5) [49]. To make it completely opaque, each tested rubbing cloth was placed above three layers of white rubbing cloth during assessment.

### 2.3.3. Light Fastness Test

For color fastness to light (ISO 105-B01:2014), strips of dyed fabrics and blue wool references were arranged on mounting cards [51]. An opaque cover was used to cover completely the middle one-third of each test card. The masked cards were then exposed to light under the specified conditions in an Atlas Xenotest Alpha LM instrument equipped with a Xenon arc fading lamp. The measurement temperature was 47 °C, while the relative humidity was set to 40%. The exposure process took place for 16 h until the contrast between the unexposed and exposed areas of the blue wool reference 3 were equal to grade 4 of the greyscale complying with ISO 105-A02:1993 [48]. Test specimens and blue wool references were removed from the source of light, and another opaque cover was used to mask only the right-hand one-third of test cards. All masked cards were placed again into the test chamber for 16 h until the color change of blue wool reference 3 reached grey scale grade 3. The total duration of light exposure was 32 h, which is slightly below the originally recommended duration of light exposure of 40 h according to the mentioned ISO standard. However, a shorter exposure duration can be justified, because already after 16 h of exposure, the color change of the blue wool reference 3 was significant and equal to grade 4 of the greyscale. Finally, lightfastness was graded from 1 = worst to 8 = best by comparing tested fabrics against blue wool references. Please compare the examples of illuminated blue standard and tested fabrics in Figure 6.

## 3. Results and Discussion

The results of the dyeing experiments are presented in the two following sub-sections. First, dyeing experiments conducted without mordanting procedures are presented. These experiments were mainly conducted as a function of increasing dye concentration. Second, the dye concentration was set to a low level, and the combinations of different mordants using different procedures are presented.

### *3.1. Dyeing without Mordant*

The dye applications without mordant were conducted on cotton with an increasing dye concentration from 2 to 20% owf. The color impressions of the resulting cotton samples are documented by photographs and presented in Figure 7. As expected, the color intensity increases with increasing dye concentration. The coloration can be best described as different shades of red and brown. In addition to the description of the visual appearance, the coloration was investigated by spectroscopic measurements and color measurements.

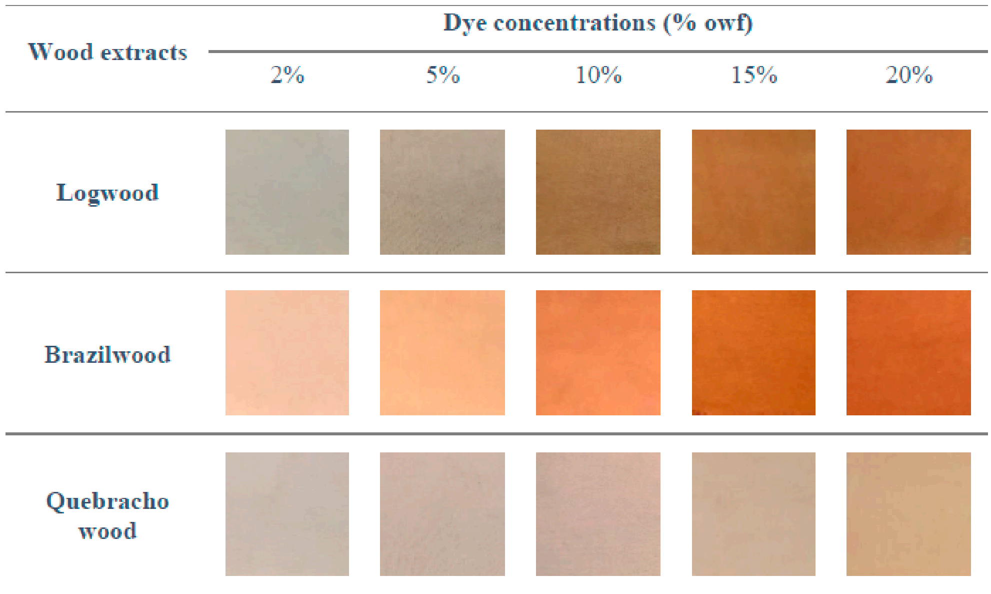


**Figure 7.** Photographs of cotton fabrics after dyeing with wood extracts using increasing dye concentrations without mordanting.

As a general trend, samples dyed with logwood, brazilwood, and quebracho wood showed a maximum absorption wavelength of around 460 nm. For this, the reflection values were recorded at 460 nm and translated into K/S values (Figure 8). Along with the deeper shade of the dyed samples, K/S values moved upwards when extract concentrations were increased.

For logwood and brazilwood, a rapid increase in K/S value occurred from 5% to 15% extract concentrations. At over 15%, color strength continued increasing, but at a lower rate. This is likely due to decreased colorant absorption rate on cotton fiber. When the extract concentration surpassed 15% owf, the dye in the fabric became saturated. From this point onward, adding more dyestuff had less of an effect on the color yield than before. At 20% concentration, the K/S value had not yet achieved equilibrium.

The K/S values of quebracho wood increased with a growth in the percentage of the applied dye, too. However, it displayed a smaller increase compared to logwood and brazilwood. Their K/S values were initially close to each other. However, at 20% dye concentration, the color strength of logwood and brazilwood samples were above fivefold and sevenfold times higher than that of quebracho wood, respectively.

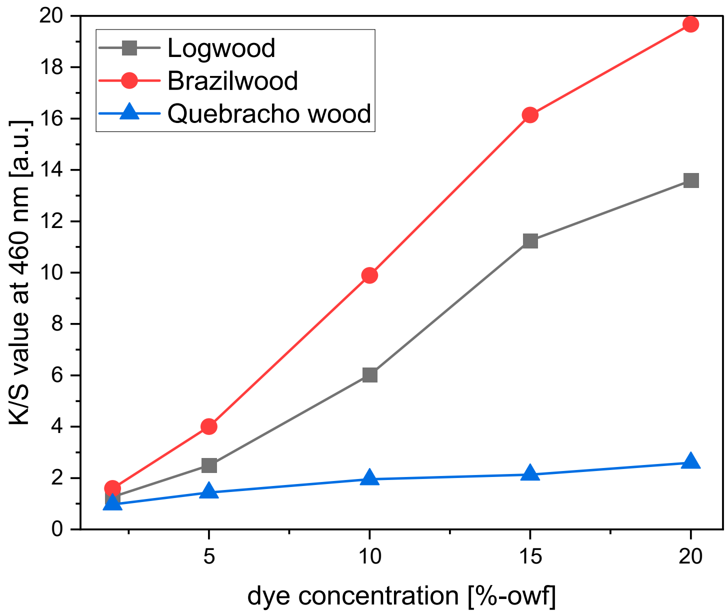


**Figure 8.** K/S-values determined for the wavelength 460 nm of cotton fabrics after dyeing with wood extracts using increasing dye concentrations without mordanting.

For the description of color properties, the L*a*b* values were determined [52]. L*a*b* values of dyed samples were presented as a function of the applied dye concentration (Figures 9–11). At first glance on the L* plane, the reduced value of lightness denoted a darker shade of the color, meaning that the application of more dyestuff generated darker shades. For logwood and brazilwood, luminance fell by more than 22 units when the dye concentrations increased from 2% to 20%. This demonstrated that the visual appearance of samples changed from light- to dark-colored, as can be seen also in Figure 7. Meanwhile, the fall in L* value of quebracho wood dye was just over 10 units. As an overall trend, the redness a* as well as yellowness b* value increased due to higher dye concentrations, indicating more intense color. Among the three extracts, brazilwood had the best color purity. Its samples looked vivid and intense, while with the other dyes, the fabric samples appeared more muted and closer to grey. With regard to color uniformity, dyed fabrics without mordant had nearly a visually uniform color (compare Figure 7).

For logwood, the hue of the color appeared yellowish brown. There is a gradual decrease in hue angle, ranging from 71° to 54° (Figure 9). This tendency meant that the higher the dye concentration, the redder (bluer) the sample became. It is obvious that higher dye concentrations also resulted in a higher level of chroma and lower level of lightness. Specifically, there was roughly a threefold increase in chroma between 2% and 20% concentration. In short, a redder, darker, and stronger color was achieved by applying a higher concentration of dye.

From 5% to 10% concentration, there was a higher increase in C* value, so at this range, the greyish brown should transform to a much purer brown. As seen in Figure 9, greyish brown shades were achieved by 2% and 5%, while a moderate or strong brown was obtained with higher dye concentrations. With higher dye concentrations, the L* value continued decreasing, but with a lower gradient. The data between 15% and 20% concentration illustrated that the three chromatic attributes (L*, h, C*) did not change much.

For samples dyed with brazilwood, an upward tendency of C* value between 2% and 15% dye concentration indicated that the samples became more chromatic or saturated (Figure 10). At 15% concentration, chroma reached a peak of 47, but afterwards, it started to reduce gently. Therefore, a more vivid color was still produced with higher dye concentrations, but not more than 15%. From 2% to 10% concentrations, the L* value went down significantly by nearly one-third. After that, luminous intensity continued to decline but at a slower rate. Similarly, the color difference ΔE did not increase considerably anymore when the dye concentration was greater than 15%.

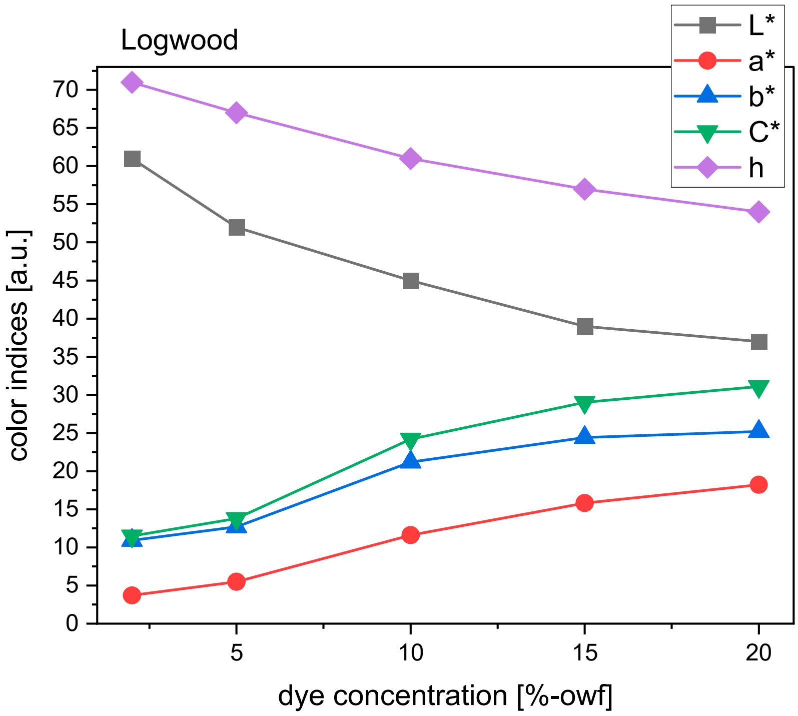


**Figure 9.** Color indices determined for cotton fabrics after dyeing with logwood extract using increasing dye concentrations without mordanting.

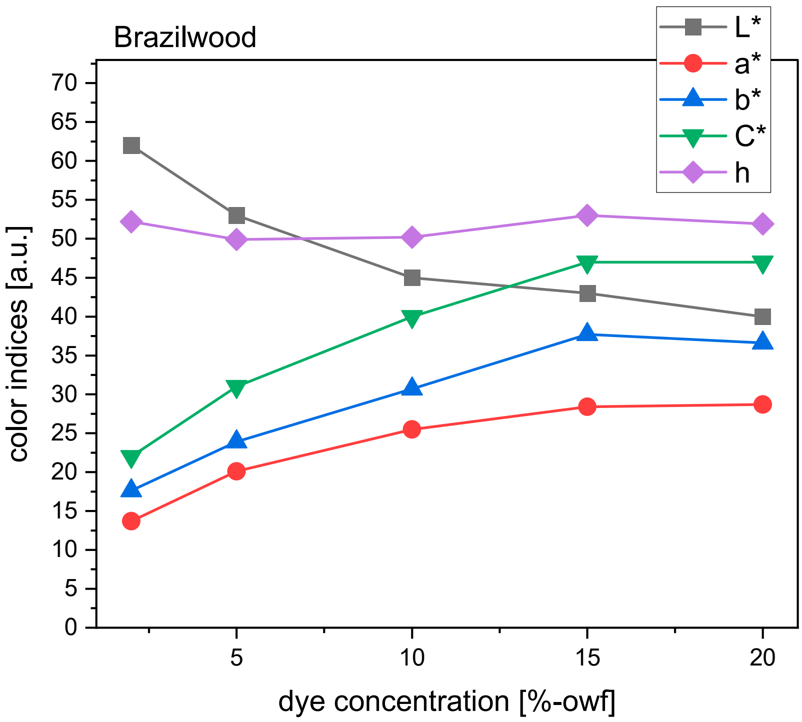


**Figure 10.** Color indices determined for cotton fabrics after dyeing with brazilwood extract using increasing dye concentrations without mordanting.

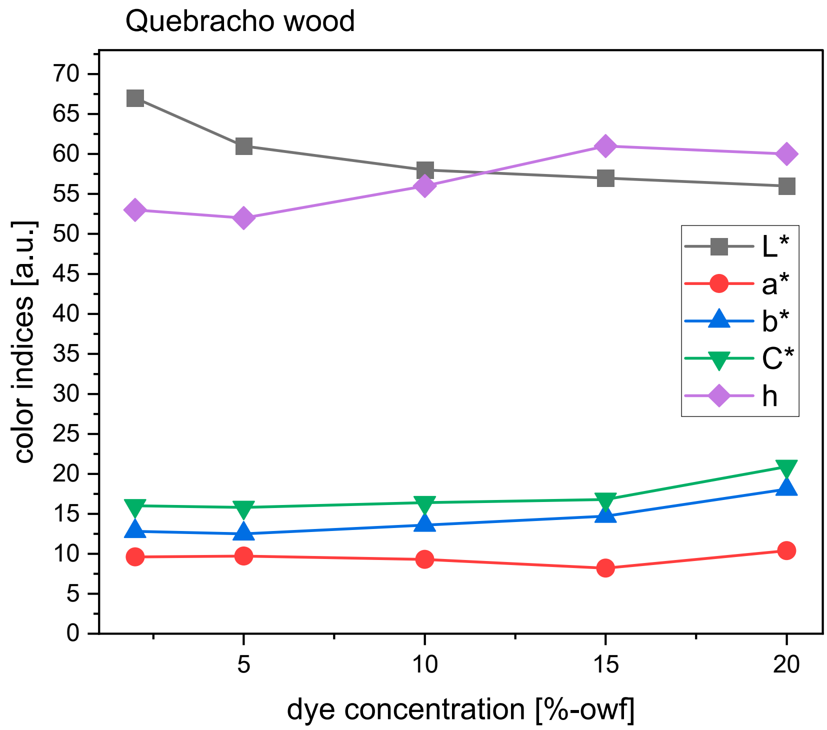


**Figure 11.** Color indices determined for cotton fabrics after dyeing with quebracho wood extract using increasing dye concentrations without mordanting.

Turning to quebracho wood, the hues were described as pale orange-yellow and yellowish brown (Figure 7). The attribute of hue went up and down, but the change moved towards the yellow direction. Therefore, from 10% to 20% dye concentration, the hue was perceived as more yellow than that of the control sample (Figure 11). The hue angle of 5% concentration was a tiny bit smaller than that at 2%, so the fabric looked a little redder. As for saturation, C* remained fairly unchanged when concentrations were below 15%, and it saw a moderate growth when the concentration reached 20%. This explains why its visual color in practice looked much more brilliant than the rest of the samples. Regardless, samples dyed with quebracho wood were generally duller or less chromatic than with the other woods due to lower C*. Higher concentrations witnessed a reduction of lightness, which was indicative of the color being darker. However, the L* value was relatively stable from 10% to 20% dye concentrations (Figure 11).

### *3.2. Dyeing with Mordant*

Cotton fabrics dyed with wood extracts and metallic mordants according to three mordanting methods were evaluated in terms of color strength, color coordinates (CIEL*a*b*), and visual appearance. The visual appearance was documented by photographic images of each prepared sample, as shown in Figure 12. The control sample, which was dyed without a mordant, was used as a benchmark to assess how the mordants affected color and fastness attributes. The visual appearances of the prepared cotton samples exhibit a broad range of different color shades with different intensities.

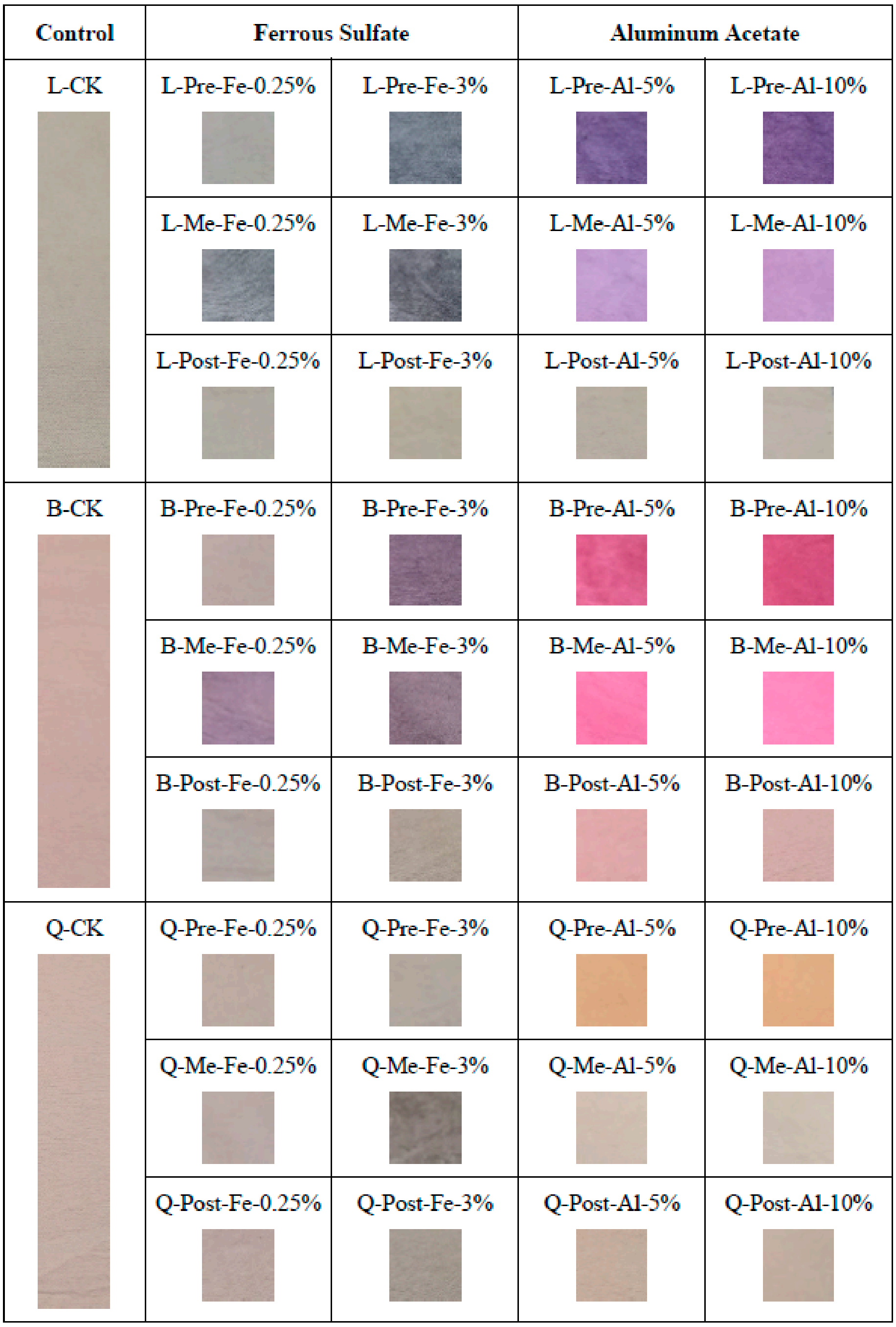


**Figure 12.** Photographs of cotton fabrics after dyeing with wood extracts using different mordants and mordanting processes (pre-, meta-, and post-mordanting).

The color strengths (K/S-values) of the mordanted samples were calculated to be 570, 540, and 450 nm for logwood, brazilwood, and quebracho wood, respectively (Figures 13–15). Mordanting had a positive impact on the dye adsorption of cotton in most cases, as indicated by increased K/S values. For logwood and brazilwood, pre- and meta-mordanting with both metallic salts proved to be more efficient in increasing K/S values than post-mordant. With regards to quebracho, there was no single technique optimized for both iron- and aluminum-based mordant; the best technique actually depends on the mordant concentration and the mordant itself.

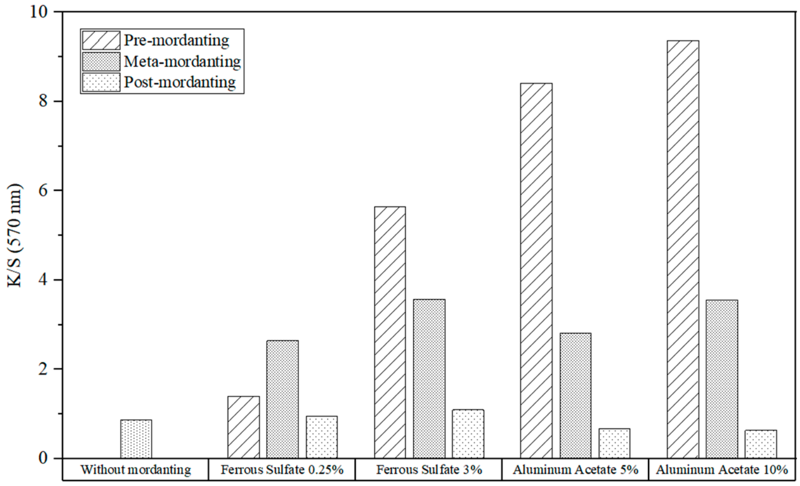


**Figure 13.** K/S-values of cotton fabrics determined for the wavelength 570 nm after dyeing with logwood extract using different mordanting procedures.

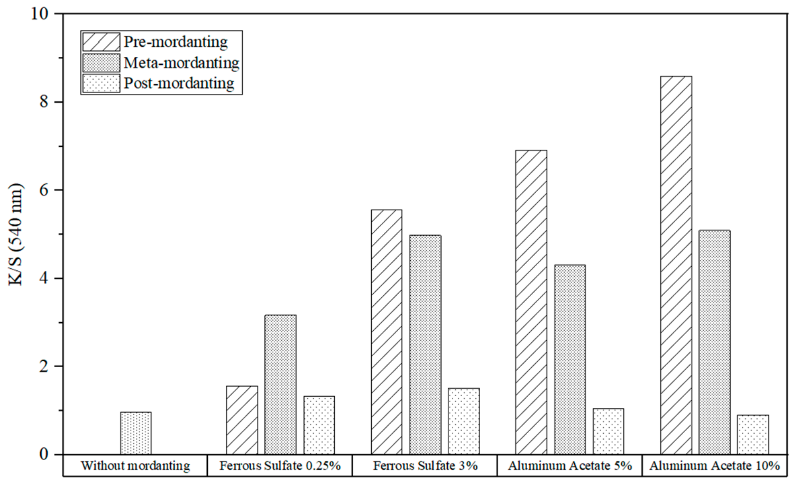


**Figure 14.** K/S-values of cotton fabrics determined for the wavelength 540 nm after dyeing with brazilwood extract using different mordanting procedures.

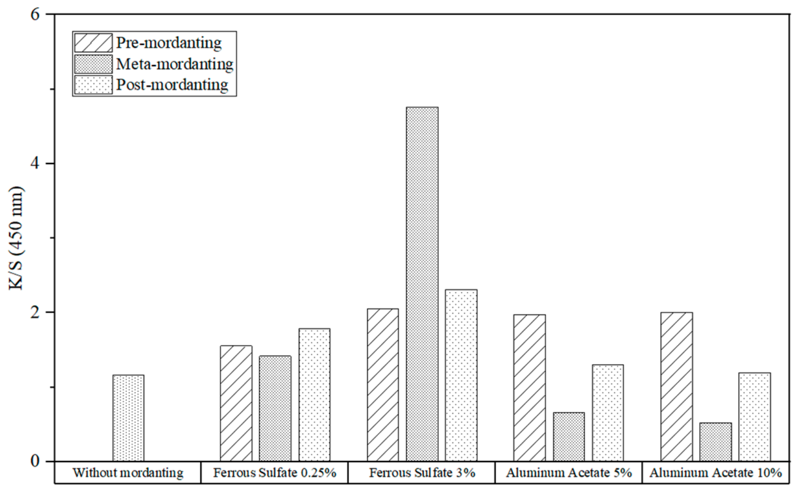


**Figure 15.** K/S-values of cotton fabrics determined for the wavelength 450 nm after dyeing with quebracho wood extract using different mordanting procedures.

Figure 13 depicts the impact of mordanting on the K/S values of the cotton fabrics dyed with logwood extract. A remarkable improvement in color strength was observed with the pre- and meta-mordanting methods using 3% iron sulfate and 5% and 10% aluminum acetate.

The results confirmed that all samples had improved K/S values due to the used iron sulfate, and higher iron concentrations were necessary to increase the color intensity. However, when it was utilized in the post-mordant procedure, a small loss in color strength was observed compared to the reference without mordant.

Similarly, higher aluminum acetate concentrations led to better K/S values except for the post-mordant process, in which its color strength even got lower with larger mordant concentrations.

When utilized in the pre-mordant procedure, aluminum guaranteed better color yields than iron sulfate. Overall, the pre- and meta-mordanting processes gave fabrics greater color strength than the post-mordanting technique. With only 0.25% iron sulfate, one-bath mordant generated the best color strength. However, pre-mordanting was a far more effective method when using a 3% iron concentration. In the case of aluminum acetate, pre-mordanting obtained the best results no matter what concentration of the mordant was applied.

The K/S-values of the cotton fabrics dyed with brazilwood extracts were compared in Figure 14. As can be observed, the biggest enhancement in K/S-value was revealed using the pre- and simultaneous-mordanting processes with a 3% concentration of iron sulfate and 5% and 10% concentrations of aluminum acetate. Similar to logwood, this analysis demonstrated that mordanting with iron salt always enhanced K/S values, and a higher concentration of iron salt yielded a visible improvement in color strength.

Aluminum acetate provided higher K/S values than the reference sample, except for the post-mordanting method with 10% aluminum acetate. For pre- and one-bath mordanting, but not for post-mordanting, using more aluminum had a favorable impact on K/S values. When using the pre-mordanting method, mordanting with aluminum provided greater color strengths than using iron sulfate. However, less favorable results were observed with meta- and post-mordanting methods.

Between three mordanting methods using the same mordant concentrations, pre- or meta-mordanting produces the highest K/S value. With 0.25% iron sulfate, the K/S value of the dyed fabric was highest with one-step mordanting. Despite this, pre-mordanting would be the most effective method for a 3% iron sulfate concentration. As for aluminum acetate, pre-mordanting always leads to the best results regardless of the salt concentration.

The influence of mordanting on the K/S values of cotton fabrics dyed with quebracho extracts is depicted in Figure 15. The maximum color strength was obtained using one-bath mordanting with a 3% concentration of iron salt.

For all three mordanting techniques, the K/S values of the iron-mordanted fabrics exceeded that of the control sample at all concentrations. These values also increased as the iron sulphate concentration rose from 0.25% to 3%. With a low concentration of iron sulphate, there was little change in the K/S values of the mordanted samples. For significantly greater color strength, a concentration of 3% is recommended.

Due to a moderate increase in K/S values, the pre-mordant procedure was best suited for aluminum acetate, whereas meta-mordanting resulted in unsatisfactory outcome. With post-mordanting, the color strength was only slightly improved. It is not always beneficial to use more aluminum acetate because K/S values can decrease, as in meta- and post-mordanting methods. As for pre-mordanting, there is just a small jump between the K/S values of samples mordanted with 10% and 5% concentrations. Hence, it is generally not efficient to use more aluminum acetate.

Iron sulphate and aluminum acetate were found to have similar color strengths in the pre-mordant method. However, in meta- and post- mordanting, iron-treated fabrics had much higher K/S values than aluminum-treated ones. Therefore, iron sulphate generally achieved better results. When comparing the three mordant processes, it is hard to identify a single method optimized for both iron sulphate and aluminum acetate. With just 0.25% iron sulfate, the post-mordant process was improved for dyeing. With a 3% concentration, however, it was better to employ simultaneous mordanting because the K/S value was more than doubled compared to other methods. For dyeing processes using aluminum acetate, pre-mordanting was the best procedure. However, adding more aluminum acetate was not very effective because it only marginally increased color strength, as mentioned above.

In addition to the discussion of visual appearance and K/S-values, the color indices are determined and presented in Figures 16–18.

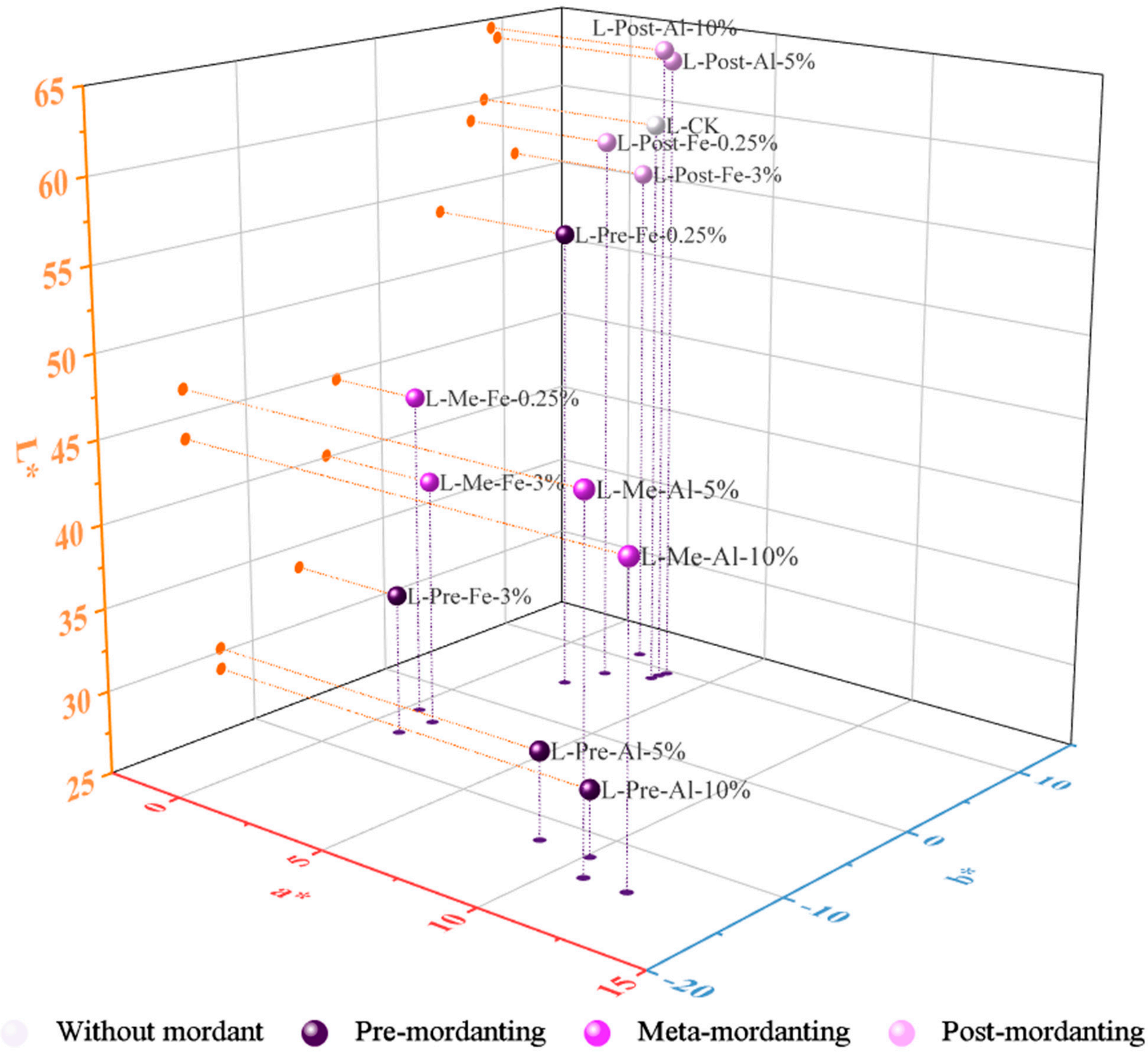


**Figure 16.** Color indices determined for cotton fabrics after dyeing with logwood extract using different mordanting procedures— relation of indices a* and b* and comparison of indices a* and b* with L*.

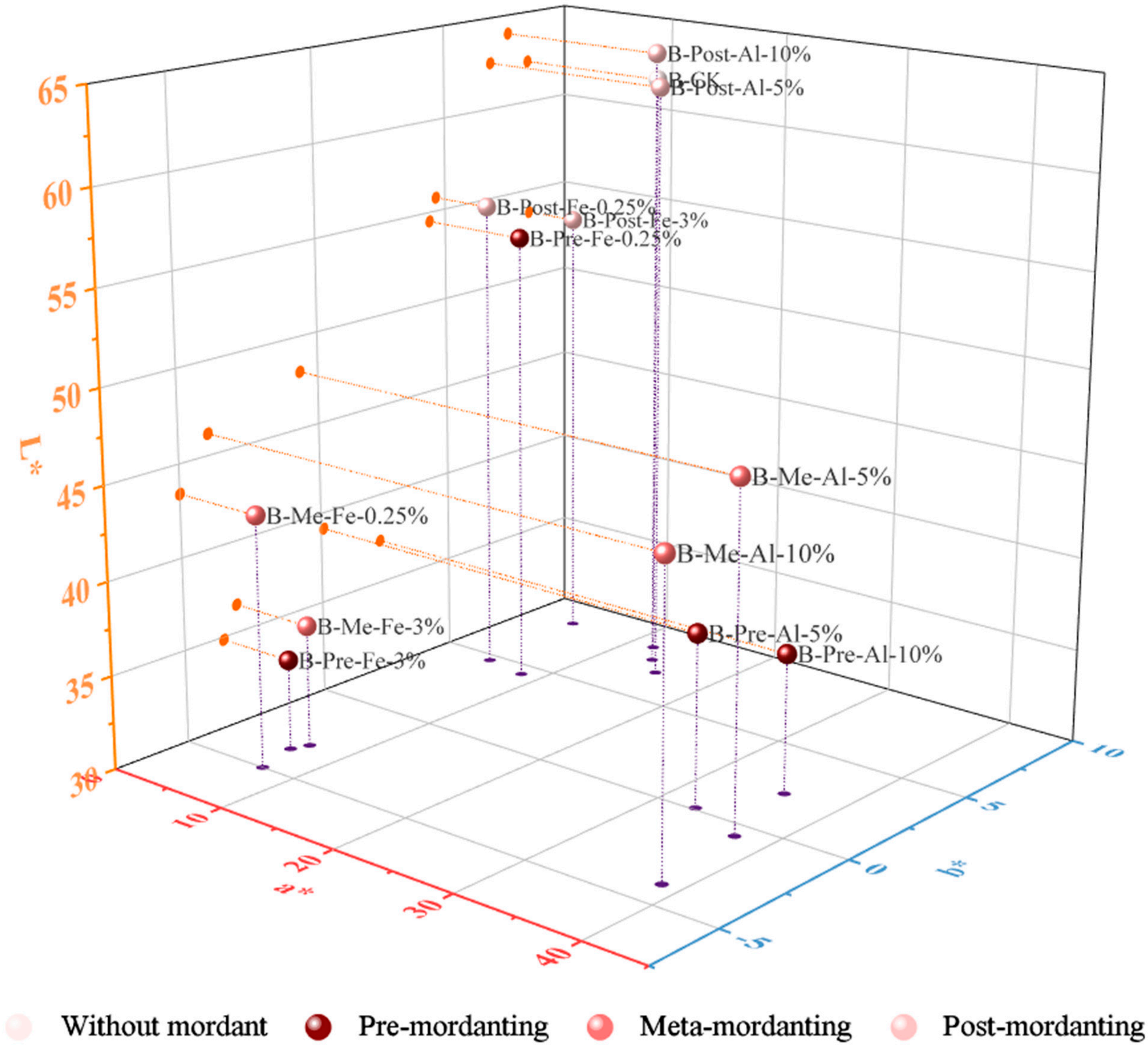


**Figure 17.** Color indices determined for cotton fabrics after dyeing with brazilwood extract using different mordanting procedures—relation of indices a* and b and comparison of indices a* and b* with L*.

With the addition of iron sulphate, the lightness and chroma values of almost all samples across the three dyes decreased, making them darker and more greyish (Figures 16–18). On the other hand, aluminum acetate always increased color saturation. In short, the L* data demonstrated that iron sulfate had a comparatively greater darkening effect than aluminum acetate, whereas the C* value indicated that aluminum acetate produced more saturated colors. Additionally, most mordanted samples had high ΔE values (total color difference), particularly those that were pre- or meta-mordanted.

The same wood extract produced a variation of hues or even a new color by applying two mordants. There was a remarkable change in the color gamut when logwood and brazilwood extracts interacted with metallic mordants through pre- and simultaneous mordanting techniques. This is in contrast to the post-mordanting procedure either with iron or aluminum salt, because the colors obtained were relatively similar to those of the control samples. Turning to quebracho, metallic salts did not change its original color completely.

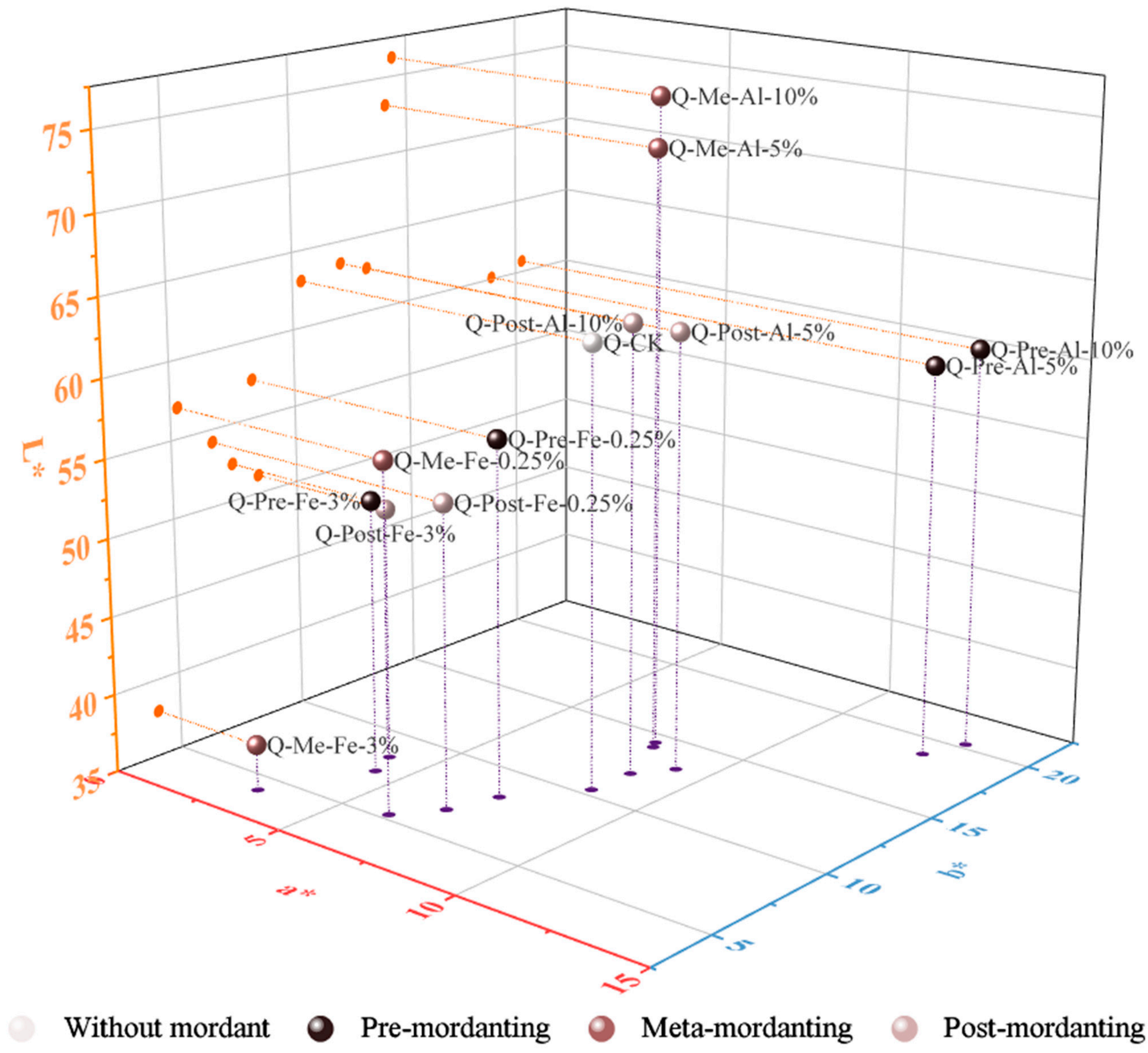


**Figure 18.** Color indices determined for cotton fabrics after dyeing with quebracho wood extract using different mordanting procedures—relation of indices a* and b* and comparison of indices a* and b* with L*.

The colorimetric data of samples dyed with logwood in terms of L*, a*, and b* values are plotted in two- and three-dimensional space and presented in Figure 16.

The hue of non-mordanted fabric was specified by a very light yellowish brown color. Generally, the color shade was darker and duller with iron sulfate but lighter and stronger with aluminum acetate. This is consistent with findings from previous research. As presented in Figure 12, the use of metallic mordants produced new colors. Although a few mordanted samples still stayed in the orange range (35–70°) of the hue circle like the non-mordanted one, all others shifted to 70–105° yellow, 195–285° blue, and 285–350° violet (Figure 16).

When iron sulfate was combined with logwood in the pre-mordant process, there were two different colors depending on the concentration of iron sulfate as well as the mordant method. The obtained data show that cotton fabrics meta-mordanted with iron sulfate (0.25% and 3%) and pre-treated with iron sulfate (3%) produced a bluish grey because hue angles were inclined towards the blue coordinate of the blue–red zone. Meanwhile, the other iron-mordanted samples had a similar brown color to the non-mordanted one since their hue angles moved fractionally from the standard of 67.11°. In general, iron mordant had a darkening effect (lower L*, C* values) by generating black precipitates when it reacted with the logwood extract. Only in the case of post-mordanting with 3% iron sulfate 3% (owf) did the sample became slightly saturated, but the darkening effect persisted. The darkest shade was obtained from pre-mordanting with 3% iron sulfate followed by simultaneous mordanting.

Aluminum acetate also generated two different hues. The pre- and meta-mordanted cotton fabrics were violet, as evidenced by hue angles of 304.77° to 310.27°. Meanwhile, post-mordanted samples were still located in the yellow–red zone and very close to the control sample. Consequently, post-treatment with aluminum acetate created the closest hue to the non-mordanted one. Likewise, a larger amount of aluminum acetate did not significantly modify the color, as demonstrated by minor differences in all L*, C*, and h° values. The photographs shown in Figure 12 visualize how comparable their colors are.

As can be observed, the post-mordanting method did not bring about such a substantial change in colorimetric data as did the pre- and simultaneous methods. Compared to the control samples, post-mordanted samples guaranteed the most equivalent outcomes of color measurement and visual assessment. Moreover, the L*a*b* values remained fairly static, and thus, post-treated samples had the least color differences (ΔE).

When the visual appearances of all samples were mutually compared, it was found that meta-mordanted fabrics suffered most from uneven colorization. The cotton samples treated in the pre-mordanting method showed a more uniform color distribution, but this still needs slight improvement. For these two methods, a smaller amount of iron sulfate and a larger amount of aluminum acetate resulted in better color uniformity. The post-mordanting process achieved a high degree of uniformity regardless of the types and concentrations of the used mordant. One-bath mordant is the most convenient preparation and should come to extensive use.

The colorimetric data of samples dyed with brazilwood in terms of L*, a*, and b* values are plotted in two- and three-dimensional space and presented in Figure 17.

The apparent color of the control sample was greyish orange, while those treated with iron sulfate and aluminum acetate could obtain new color shades (Figure 12). In general, both iron sulfate and aluminum acetate caused a reduction in yellowness but an increase in bluish nuance compared to the control sample. Aluminum-treated fabrics have greater C* values and lower L* than the iron-mordanted ones, and thus, their samples are perceived as more vivid and lighter. The application of these two mordants created new colors (see Figure 12).

For logwood, iron sulfate was used to achieve two distinct hues. Similarly, iron sulfate had two distinct hues for brazilwood-dyed cotton. It was not only the iron itself but also its concentration and the mordanting procedure that determined the hue. Pre-mordanting with iron sulfate (0.25%) and post-mordanting with iron sulfate (0.25% and 3%) produced a shade comparable to that of the control sample. Their hue angles varied from 27.52° to 63.62°, indicating a red-orange hue. The minor difference was that post-mordanted fabrics looked less red than the others. On the other hand, a violet color was obtained with iron salt by pre-mordanting (3%) or meta-mordanting (0.25% and 3%). The hues stayed within a small range of 324.92–334.72°, specifying violet color. Violet fabrics that were pre-mordanted seemed slightly darker than the meta-mordanted ones.

Similarly to iron sulfate, aluminum acetate could result in two different hues. When pre- or meta-mordanting, the hue angle shifted clockwise towards the red direction (+a* axis) in a range of 354.13° to 3.6°, indicating a pink color. The L* of the samples that had been pre-treated with aluminum acetate decreased considerably, roughly about two-thirds of the control sample. However, in meta-mordanted samples, there was a lower loss in L*, accompanied by better C* and lower −b*. As such, the former is a dark pink, and the latter is light pink but stronger and bluer. In contrast to pre- and meta-mordanting methods, the post-mordanting method did not significantly alter the a*, b*, and L* values of samples treated with aluminum acetate. Their hue angles of 24.02–29.23° indicated a slightly redder hue compared to the non-mordanted sample. Their samples were most similar to the control sample due to minimal ΔE. As a result, aluminum acetate post-treatment produced the most comparable shade to the non-mordanted one.

Visual comparisons among all samples revealed that meta-mordanted fabrics were less evenly dyed than those using other techniques. Hence, dye evenness is not feasible with brazilwood in one-bath mordanting, as with logwood. The cotton after pre-mordant treatment displayed quite good degrees of uniformity. In order to obtain better color uniformity, it is recommended to use less iron sulfate and more aluminum acetate for these two procedures. No matter what kind or how much of a mordant was employed, the post-mordanting process produced highly uniform colors.

The colorimetric data of samples dyed with quebracho wood extract are plotted in two- and three-dimensional space and presented in Figure 18.

In the absence of a mordant, quebracho dyeing resulted in yellowish brown shades on cotton (Figure 12). The hue of cotton dyed without mordant was not entirely different from those treated with mordants, which was attributed to the positions of the datapoints only moving along the red (+a*) and yellow (+b*) axes. Their h° values were in the same range as the control sample, namely 35–70° (orange) in the color circle. The hue difference between a mordanted sample and the non-mordanted sample was not so large as in logwood- or brazilwood-dyed samples.

In comparison to iron sulfate-mordanted fabrics, aluminum acetate-treated fabrics had greater CIEL*a*b* values, reflecting stronger and lighter colors. Regardless of the mordanting method, dyeing with iron sulfate produced darker and less saturated shades, reflected in lower L* and C* values. Results also revealed that fabrics became redder in hue at lower iron concentrations (0.25%) and yellower at higher concentrations (3%). Higher iron sulfate concentrations did not exert a profound darkening effect, with the exception of meta-mordanting with 3% iron sulfate. In this case, the lightness value declined sharply from 63.02 to 37.92. This explains why its sample appeared much darker than all other samples, and its ΔE value of 26.43 exhibited a huge difference from the control sample. Undesirably, this sample suffered from a serious uneven dyeing defect, whereas the colors of all other samples looked completely or mostly uniform.

Compared to the control sample, dyeing with aluminum acetate mordant (pre-, meta-, and post-mordanting) yielded a more yellow and brilliant shade, as evidenced by higher C* and hue difference. As can be seen from the above table, the hue of the standard sample was 50.51°, while the hue of samples treated with aluminum acetate ranged from 54.45° to 61.35°, adding a slight yellowish nuance to the brown color. The increase of the C* value indicated that the fabrics were not only more yellow but also stronger in coloration. The chroma levels were at their best with the pre-mordanting method, giving an earth-yellow color. Furthermore, a higher concentration of aluminum acetate did not alter the color drastically, as evidenced by small differences in all color attributes (L*, C*, h°). The samples that look nearly identical to the control sample are the post-mordanted ones with aluminum acetate because of a slight change in L*, C*, h° values and low color change ($\Delta E < 3$). Meta-mordanted samples are pale orange-yellow due to the highest L* and hue angles closest to the yellow direction.

Except for one-bath dyeing at a 3% iron sulfate concentration, the outcome of color uniformity was excellent when utilizing iron sulfate as a mordant. A small degree of unevenness appeared when using aluminum acetate in both pre- and meta-mordant methods. Greater quantities of aluminum acetate used in the pre-mordant process resulted in better evenness but performed worse in the one-bath dyeing. Additionally, colors produced by the post-mordanting process were highly uniform no matter what type of mordant or how much was applied.

### 3.3. Effects of Mordanting on Colour Fastness Properties

Mordanted fabrics were also tested for fastness to demonstrate their practicability. Tables 1–3 summarize the data from the evaluation of wash, rubbing, and light fastness for logwood, brazilwood, and quebracho wood applications, respectively. The fastness values are listed in grades from 1 = worst to 5 = best. The staining during washing is shown against an adjacent multifiber fabric containing different types of fiber: (wool—WO, acrylic—PAC, polyester—PES, polyamide—PA, cotton—CO, cellulose acetate—AC).

**Table 1.** Color fastness properties of cotton fabrics after dyeing with logwood extract using different mordanting procedures. The sample code shows the type of mordanting used (Pre, Me, or Post) and the mordanting agent (Fe or Al) with indicated concentrations. The staining is tested against an adjacent multifiber fabric (wool—WO, acrylic—PAC, polyester—PES, polyamide—PA, cotton—CO, cellulose acetate—AC). The fastness values are listed in grades from 1 = worst to 5 = best.

| Sample Code | Washing Fastness | | | | | | | Rubbing Fastness | | Light Fastness |
|---|---|---|---|---|---|---|---|---|---|---|
| | Colour Change | Staining | | | | | | Dry | Wet | |
| | | WO | PAC | PES | PA | CO | AC | | | |
| L-CK | 3 | 3 | 4 | 4–5 | 4 | 4 | 4–5 | 4–5 | 4–5 | 2 |
| L-Pre-Fe-0.25% | 2 | 3 | 4–5 | 4–5 | 3–4 | 4–5 | 4–5 | 4–5 | 4–5 | 2 |
| L-Pre-Fe-3% | 1 | 2 | 4–5 | 4–5 | 3–4 | 4–5 | 4–5 | 3 | 2–3 | Better than 3 |
| L-Pre-Al-5% | 1 | 2 | 4 | 4–5 | 2–3 | 3–4 | 3–4 | 3 | 2–3 | 1 |
| L-Pre-Al-10% | 1 | 2 | 4 | 4–5 | 2–3 | 3–4 | 4 | 3 | 2–3 | 1 |
| L-Me-Fe-0.25% | 1–2 | 2 | 4 | 4–5 | 3 | 4 | 4–5 | 4 | 3–4 | Better than 3 |
| L-Me-Fe-3% | 1 | 2 | 4 | 4–5 | 4 | 4 | 4–5 | 3 | 3 | 2–3 |
| L-Me-Al-5% | 2 | 3 | 4–5 | 4–5 | 4 | 4–5 | 4–5 | 4 | 3–4 | 1 |
| L-Me-Al-10% | 2 | 4 | 4 | 4–5 | 4 | 4–5 | 4–5 | 3 | 3 | 1 |
| L-Post-Fe-0.25% | 2 | 4 | 4–5 | 4–5 | 4 | 4–5 | 5 | 4–5 | 4–5 | 2–3 |
| L-Post-Fe-3% | 3 | 4 | 5 | 5 | 4–5 | 4–5 | 5 | 4–5 | 4 | Better than 3 |
| L-Post-Al-5% | 3 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 5 | 4–5 | 4–5 | 1 |
| L-Post-Al-10% | 4 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 1 |

In general, the dyed fabrics without mordant displayed good color staining and rubbing fastness. However, brazilwood and quebracho wood showed fair to poor color change and light colorfastness, while logwood had acceptable color change but only fair colorfastness to light.

The findings show that the addition of metal ions can enhance the color fastness of fabrics. However, the majority of mordanted and dyed samples did not show good performance in all three fastness tests simultaneously. For instance, iron sulfate often improved light fastness but not color change, while the opposite behavior was observed for mordanting with aluminum salt. The mordant effect of iron sulfate on light fastness was better compared to applications with aluminum salts.

The results obtained for logwood-dyed samples are displayed in Table 1. The rubbing and washing fastness of the control sample were all above grade 3. However, the color faded quickly when exposed to light, indicated by a grade of 2 in light fastness.

**Table 2.** Color fastness properties of cotton fabrics after dyeing with brazilwood extract using different mordanting procedures. The sample code shows the type of mordanting used (Pre, Me, or Post) and the mordanting agent (Fe or Al) with indicated concentrations. The staining is tested against an adjacent multifiber fabric (wool—WO, acrylic—PAC, polyester—PES, polyamide—PA, cotton—CO, cellulose acetate—AC). The fastness values are listed in grades from 1 = worst to 5 = best.

| Sample Code | Washing Fastness | | | | | | | Rubbing Fastness | | Light Fastness |
|---|---|---|---|---|---|---|---|---|---|---|
| | Colour Change | Staining | | | | | | Dry | Wet | |
| | | WO | PAC | PES | PA | CO | AC | | | |
| B-CK | 2 | 4 | 4 | 4–5 | 3–4 | 4–5 | 4 | 4–5 | 4–5 | 1 |
| B-Pre-Fe-0.25% | 2 | 3 | 4–5 | 4–5 | 3–4 | 4–5 | 4 | 4–5 | 4–5 | 2 |
| B-Pre-Fe-3% | 1 | 2 | 4–5 | 4–5 | 3 | 4 | 4 | 4 | 3 | Better than 3 |
| B-Pre-Al-5% | 2 | 2 | 4 | 4 | 1–2 | 3 | 3–4 | 3 | 2–3 | 1 |
| B-Pre-Al-10% | 1–2 | 2 | 4 | 4 | 1–2 | 3 | 3–4 | 3 | 2–3 | 1 |
| B-Me-Fe-0.25% | 2 | 2 | 4 | 4–5 | 3 | 4 | 4 | 4–5 | 3–4 | 2 |
| B-Me-Fe-3% | 1 | 2 | 4 | 4–5 | 3–4 | 4 | 4–5 | 3–4 | 3 | Better than 3 |
| B-Me-Al-5% | 1–2 | 2–3 | 4–5 | 4–5 | 2–3 | 3–4 | 3–4 | 4 | 3 | 1 |
| B-Me-Al-10% | 2 | 2–3 | 4–5 | 4–5 | 2–3 | 3–4 | 3–4 | 4 | 3–4 | 1 |
| B-Post-Fe-0.25% | 2–3 | 4 | 4–5 | 4–5 | 3–4 | 4–5 | 5 | 4–5 | 4–5 | Better than 3 |
| B-Post-Fe-3% | 3 | 4 | 4–5 | 4–5 | 4 | 4–5 | 4–5 | 4–5 | 4 | Better than 3 |
| B-Post-Al-5% | 3 | 4–5 | 4–5 | 4–5 | 4 | 4–5 | 4–5 | 4–5 | 4–5 | 1 |
| B-Post-Al-10% | 3–4 | 4–5 | 4–5 | 5 | 4 | 5 | 5 | 4–5 | 4–5 | 1 |

The following is a sequence starting from the highest to the lowest ratings of staining and rubbing fastness with iron and aluminum: post-mordant > meta-mordant > pre-mordant. Regarding color change, post-mordanted samples also showed the highest grade for both metallic mordants. Therefore, post-mordanting is generally the most reliable method for logwood in terms of fading properties. Despite yielding deeper shades, pre- and meta-mordanting did not really improve fastness properties.

Iron-mordanted samples achieved the same or better lightfastness than the control sample, reaching a grade of 2 to better than 3. The staining might be worsened, but in many cases, their gradings were still good enough. The main issue of iron-treated fabrics was the poorer color change it achieved relative to the control sample. Only one sample (L-Post-Fe-3%) could be regarded as good because it showed a rating of 3 or above for all colorfastness to washing (color change and staining), wet and dry rubbing, and light.

Aluminum acetate generated worse washing, rubbing, and light fastness than the control sample when used as a mordant in the pre- and meta-mordanting methods. With the post-mordanting process, it achieved better washing fastness and retained good rubbing fastness, but the light fastness was still less than that of the control sample. As a result, it is not recommended to use aluminum acetate with logwood extracts if a certain light fastness is demanded.

**Table 3.** Color fastness properties of cotton fabrics after dyeing with quebracho wood extract using different mordanting procedures. The sample code shows the type of mordanting used (Pre, Me, or Post) and the mordanting agent (Fe or Al) with indicated concentrations. The staining is tested against an adjacent multifiber fabric (wool—WO, acrylic—PAC, polyester—PES, polyamide—PA, cotton—CO, cellulose acetate—AC). The fastness values are listed in grades from 1 = worst to 5 = best.

| Sample Code | Washing Fastness | | | | | | | Rubbing Fastness | | Light Fastness |
|---|---|---|---|---|---|---|---|---|---|---|
| | Colour Change | Staining | | | | | | Dry | Wet | |
| | | WO | PAC | PES | PA | CO | AC | | | |
| Q-CK | 2–3 | 3 | 4–5 | 4–5 | 4 | 4 | 4–5 | 4–5 | 4–5 | 2 |
| Q-Pre-Fe-0.25% | 3 | 3 | 3–4 | 4–5 | 3–4 | 4 | 4 | 4–5 | 4–5 | 2 |
| Q-Pre-Fe-3% | 3 | 3 | 4 | 4–5 | 4 | 4 | 4 | 4–5 | 4 | Better than 3 |
| Q-Pre-Al-5% | 3 | 3 | 3–4 | 3–4 | 3–4 | 3–4 | 4 | 4 | 3–4 | 2 |
| Q-Pre-Al-10% | 4 | 3 | 3 | 3–4 | 3–4 | 3 | 4 | 4 | 3–4 | 2 |
| Q-Me-Fe-0.25% | 2–3 | 2–3 | 3–4 | 4 | 3–4 | 3–4 | 4 | 4–5 | 4–5 | 3 |
| Q-Me-Fe-3% | 2 | 2–3 | 3–4 | 3–4 | 3–4 | 3–4 | 4 | 3–4 | 3 | Better than 3 |
| Q-Me-Al-5% | 3–4 | 3 | 3–4 | 3–4 | 3–4 | 3–4 | 4 | 4–5 | 4–5 | Better than 3 |
| Q-Me-Al-10% | 4 | 4 | 4 | 4 | 4 | 4 | 4 | 4–5 | 4–5 | Better than 3 |
| Q-Post-Fe-0.25% | 2–3 | 4 | 4–5 | 4–5 | 4 | 4–5 | 5 | 4–5 | 4–5 | 2 |
| Q-Post-Fe-3% | 3 | 4 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | Better than 3 |
| Q-Post-Al-5% | 4 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 5 | 4–5 | 4–5 | 1 |
| Q-Post-Al-10% | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 4–5 | 1 |

The results for fastness tests obtained for brazilwood-dyed samples are displayed in Table 2. For samples without mordanting, a color change of 2, staining of 3–4 to 4–5, rubbing fastness of 4–5, and light fastness of 2 are demonstrated. As shown in Table 2, the results of color staining and rubbing fastness with iron sulfate followed a sequence in following order: post-mordant > pre-mordant > meta-mordant. With aluminum acetate, they exhibited a different order: post-mordant > meta-mordant > pre-mordant. The best light fastness for samples treated with iron sulfate was likewise achieved with the post-mordant method. Overall, when it comes to fading properties, post-mordanting is the best procedure for brazilwood.

In many cases, iron-mordanted samples outperformed control samples in terms of lightfastness, with grades exceeding 3. Even though the stains frequently got worse, the grades could be nonetheless acceptable. The main problem with iron-treated fabrics was that it generally produced no enhancement in color change. Only one sample (B-Post-Fe-3%), which was graded 3 for color change and better than 3 for all other tests, could be considered good.

Regardless of concentration or mordanting method, aluminum acetate resulted in a lower light fastness than the control sample, although it can improve other fading properties to some extent. The best washing and rubbing fastness were seen in the post-mordanted sample with 10% aluminum acetate; however, its light fastness was still 1. Consequently, the use of aluminum acetate for brazilwood extract was disadvantageous.

Logwood and brazilwood have several results in common. First, the color change and lightfastness ratings of both non-mordanted and mordanted sampled were frequently poor to very poor (1–2). Second, a higher concentration of aluminum acetate could slightly improve washing and rubbing fastness in some cases, but under no circumstances did it enhance the light fastness. Another similarity is that the most appropriate process for both dyes was post-treatment with a 3% concentration of iron. Lastly, although pre- and meta-mordanted samples had much higher color strength, their washing and rubbing fastness were actually worse than post-mordanted ones. This is because the dye adsorption has vastly improved, but much of the dye was only superficially absorbed on the surface of the cotton material, making it easy to remove during wash and crock fastness tests. Although having significantly faded from their original hues after wash, many of them still had much deeper color than post-mordanted samples.

The results for fastness tests obtained for quebracho-dyed samples are displayed in Table 3. The Staining and color fastness to crocking of the control sample were found to be in the range of 3 to 4–5. However, the ratings of color change and light fastness were 1 or 2–3 (poor) (Table 3). The results demonstrated that color change in mordanted samples was better than in the control sample except for meta-mordanted ones with iron sulfate. More than half of the dyed samples had staining grades of good to very good (3 to 4–5). Additionally, half of them showed improvement in light fastness. There would are three effective mordanting processes, as described below.

Based on the control sample, iron sulfate was consistently effective in improving light fastness and, depending on the mordanting technique, could also increase washing fastness. The best solution for quebracho was found to be post-mordanting with 3% iron sulfate, which enhanced both wash and light fastness while maintaining good rubbing fastness. Pre-mordanting with 3% iron sulfate was also effective, but its staining is marginally worse than in the control sample.

With aluminum acetate, lightfastness values remained unchanged with pre-mordanting, decreased with post-mordanting, and increased with meta-mordanting. Light fastness was impacted more by the mordanting method than the aluminum acetate concentration. In the post-mordanting method, it was undesirable that both washing and rubbing fastness were outstanding while light fastness was only 1, making this method less efficient. The most effective way to treat cotton with aluminum acetate was one-bath mordanting since the light fastness this produced was considerably better than that of the control sample, although washing fastness was not that excellent but still acceptable. For an overall good fastness, a minimum concentration (5%) of aluminum acetate was sufficient. However, a 10% owf concentration would be recommended for marginally better color change and staining.

Overall, the fastness grades of quebracho-mordanted samples were higher than those of logwood and brazilwood. In terms of coloration durability for logwood and brazilwood, post-mordanting with 3% iron sulfate is the best option. Turning to quebracho wood, besides pre- or post-mordanting with iron sulfate at 3%, cotton can be also treated well with aluminum acetate. One-bath dyeing is a definite advantage of dyeing with quebracho wood extract because this method saves time and cuts down on water and energy consumption. In summary, although logwood and brazilwood offer attractive hues like pink and violet, the color durability of these colors is lower and thus less attractive for commercial use. The color produced by all three extracts is greyish brown, and each of them had a different nuance. Wood extracts clearly contain less tannin than raw wood. Tannin and cellulose fibers form strong connections, and aluminum ions can effectively interact with the complex of tannins and fibers [53]. For this, a further improvement could be reached in subsequent investigations by the addition of tannins together with the metal mordant agents.

## 4. Summary and Conclusions

The use of three different extracts from colored wood was investigated in this study using a setup with three different mordanting procedures and two different mordanting agents based on aluminum and iron salts. A total of 12 different combinations were investigated for each wood extract for the use of dyeing cotton fabric. For each combination, only one dyeing experiment was performed. However, there was a clear correlation between the chosen parameters and gained dyeing results, so the reliability of the results can be assumed. By using aluminum acetate and iron-(II)-sulfate, several color shades were generated for the application of extracts from logwood and brazilwood. Using the same metallic salt, different mordant methods may result in drastically different hue color (h°) and CIELab values. Nevertheless, regarding quebracho dye, the color of the mordanted samples did not significantly differ from the control sample prepared without a mordanting agent. Overall, with iron-based mordant, darker (lower L*) and less-intense (lower C*) colors were gained compared to recipes with aluminum-based mordant. Depending on type, concentration, and methods, metallic mordants can be utilized to raise or lower color strength and be effective or ineffective at raising fastness qualities. In most cases, cotton fabrics treated with mordant achieved higher K/S values than those without mordant. As demonstrated by an increase in color strength, mordanting mostly enhanced the dye adsorption on cotton fabric.

Many appealing shades were produced with the combination of wood dyes and two mordants. Nevertheless, to develop textile products that are feasible for commercial use, the improvement of fastness properties is a principle goal. The best fastness results were obtained using the extract from quebracho wood with pre- or meta-mordanting procedures. Using this method, a variation of brown shades was provided, which is typical of natural dyes. The primary benefit of quebracho dyeing in combination with aluminum acetate is the elimination of a second mordanting process or two-bath dyeing. A major similarity between logwood and brazilwood is that pre- and meta-mordanting with metallic salts did not have a beneficial influence on washing and rubbing fastness. However, these mordanting methods resulted in the highest color strength. Post-mordanting yielded generally better fastness results than the others due to fewer unfixed pigments remaining on the fiber surface. Further, cotton mordanted with iron salt displayed better light fastness compared to samples with aluminum-based mordant.

This study shows that the use of wood extracts for the purpose of dyeing textiles made from natural fibers is an interesting approach to reach fully bio-based and biodegradable textile materials. The presented investigations show that a broad range of attractive colorations can be reached through different wood extract applications by modifying the mordanting procedure. However, for some industrial applications, the light fastness properties of the developed samples might be too low. This issue could be solved in subsequent investigations by combined application with UV-absorbing components. As to this, compounds with a cinnamate structure can be useful UV absorbers [54]. Nevertheless, a first proof-of-concept on the suitability of the method is made in this study, which could be a starting point for the development of further dyeing processes. These future developments should focus especially on the improvement of fastness properties.

**Author Contributions:** Methodology, T.H.M. and B.M.; formal analysis, T.H.M.; investigation, T.H.M.; resources, T.G.; data curation, T.H.M.; writing—original draft, T.H.M., T.G. and B.M.; editing and writing, B.M.; supervision, T.G. and B.M. All authors have read and agreed to the published version of the manuscript.

**Funding:** This research received no external funding.

**Data Availability Statement:** Data are contained within the article. Dataset available on request from the authors.

**Acknowledgments:** The authors owe many thanks to Simone Wagner and Gudrun Lieutenant-Bister for their helpful advice during the dyeing experiments and color analytics done in the finishing laboratory (Niederrhein University of Applied Sciences, Faculty of Textile and Clothing Technology). All product and company names mentioned in this article may be trademarks of their respected owners, even without labelling.

**Conflicts of Interest:** The authors declare no conflicts of interest.

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