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REGULAR ARTICLE

Environmentally Friendly Dyeing of Wool, Cotton and Soybean Fibers by Using Apple *(Malus Domestica)*, Linden Tea *(Tilia Tomentosa)* and Mate Tea *(Ilex paraguariensis plant)* plant wastes as a Natural Dye Source

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Abstract

The growing focus on sustainable coloration strategies has prompted the investigation of agro-industrial by-products as potential dye sources for textiles. In this study, wool, soybean, and cotton fibers were dyed using liquid dye solutions obtained from the extraction of waste materials generated during the production of apple (Malus domestica), linden tea (Tilia tomentosa), and mate tea (Ilex paraguariensis). The color development observed on the textile samples was significantly contributed to by the polyphenolic structures identified in the extracts, as confirmed by high-performance liquid chromatography (HPLC) and ultraviolet–visible (UV–Vis) spectroscopy analyses. Sodium carbonate (Na_2CO_3) and citric acid ($C_6H_8O_7$) were used to modify the surface charge characteristics of the textile substrates, and ferrous sulfate ($FeSO_4$), sodium alginate ($C_6H_7NaO_6$), calcium carbonate ($CaCO_3$), alum ($Al_2(SO_4)_3$), potassium sodium tartrate ($KNaC_4H_4O_6$), aluminum chloride ($AlCl_3$) and magnesium chloride ($MgCl_2$) were employed as mordants during the dyeing process. Optimal extraction and dyeing durations, as well as appropriate mordant concentrations, were systematically investigated. Colorimetric evaluations (CIE$L^*a^*b^*$ coordinates) and colorfastness tests, including washing, crocking, perspiration and saliva fastness, were conducted according to standard protocols. The highest fastness ratings (grade 5) for washing, crocking and perspiration were obtained for wool, cationized cotton and soybean fibers dyed with colorants extracted from linden (Tilia tomentosa) waste. In contrast, samples dyed with extracts derived from apple and mate tea waste showed fastness values ranging from 5 to 3–4. Following the dyeing process, the metal content of the wastewater from the mordanting process was quantified using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). The environmental implications of the detected metal concentrations were subsequently assessed.

Keywords Natural Dyes · Cotton · Soybean · Fastness · Colorimetric properties

1 Introduction

The global rise in environmental pollution and the rapid depletion of natural resources have intensified concerns about the planet's future. These developments have prompted society to become more aware of environmental sustainability. This trend has directly influenced consumer preferences, resulting in a marked increase in demand for organic and eco-friendly products, especially in the textile and food industries. The textile industry, due to its wet processes that require high energy and chemical consumption, is considered a major contributor to global environmental pollution. Research has shown that the discharge of textile chemicals has toxic effects and negatively impacts both aquatic ecosystems and human and ecological health [1, 2]. Specifically, wastewater originating from dyeing facilities contains residual dyes, high concentrations of electrolytes, toxic components (such as heavy metals and unreacted raw materials), and potential carcinogens, posing significant risks to

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both the environment and human health [3]. Environmental strategies within the textile sector have primarily focused on pollution prevention and have been largely shaped by regulatory requirements aimed at mitigating the environmental impacts of wastewater generated from wet processes, including preparation, dyeing, and finishing [4–6]. Currently, these efforts are further supported by active research dedicated to reducing the ecological footprint of dyeing processes, which inherently require wet treatments. In this context, the development of waterless dyeing technologies and the preference for natural dyes over synthetic alternatives have emerged as prominent approaches. Natural dyes are considered a more environmentally friendly and sustainable option compared to synthetic dyes. However, traditional natural dyeing techniques remain relatively costly, offer a limited color palette, and generally exhibit low fastness properties. Additionally, the agricultural land required for cultivating dye plants, the need for irrigation, and the cost and toxicity associated with pesticide use constitute significant environmental burdens that must be taken into account [7]. These limitations have recently driven researchers to explore the use of agricultural plant residues as sources of natural dyes. Utilizing agricultural waste as a dye source can contribute to reducing greenhouse gas emissions by decreasing both storage requirements and overall waste volume. [8, 9] Indeed, a wide variety of agricultural waste materials have been employed to obtain eco-friendly dyes for the textile sector. Nevertheless, the main challenges in this field include low color fastness, a limited range of achievable hues, the need for meticulous optimization of processing parameters, and the difficulties associated with integrating these processes into industrial-scale production [10, 11]. Accordingly, numerous research groups have focused on applying dyes derived from agricultural waste to textile fibers and on developing strategies to overcome these challenges.

For instance, focusing on the meticulous optimization of processing parameters and the evaluation of bio-mordant efficiency, Khan et al. [12] conducted a study to evaluate dyeing conditions for the husks of durum (Triticum durum Desf.) and bread (Triticum aestivum L.) wheat species for use in fabric dyeing. The results indicated that both types of wheat husks could be used as a suitable natural dye source when extracted under alkaline conditions. The most effective performance of the dye derived from durum wheat husks was noted at a temperature of 70 °C, with a pH of 11.0 and a salt concentration of 8.0 g/100 mL of solution. Likewise, in the study, it was found that the alkaline extract of bread wheat peel yielded superior results at 80 °C, a pH of 9.0 for the dyeing solution, and a salt concentration of 8.0 g/100 mL. The study also emphasized that pomegranate peel was the most effective bio-mordant. Moreover, the highest level of color strength was achieved by using 5% tannic acid as a pre-mordant and 5% chrome as a post-mordant, outperforming all other mordant quantities utilized in the process of chemical mordanting.

Another study demonstrated a sustainable method for the eco-printing and dyeing of silk fabrics through the utilization of selected agricultural residues. Specifically, outer onion skins (a natural source of quercetin), along with rose petals and the leaves of eucalyptus, lemon, grape, and peach, were employed as bio-based colorants in the eco-printing process. The resulting textiles exhibited satisfactory wash fastness and demonstrated biofunctional properties, including effective resistance against microbial and insect activity [13]. In a different study, a natural dye was isolated from lotus seedpod petals, and its suitability for textile dyeing was evaluated. This evaluation encompassed the effects of temperature, dyeing time, pH, and the use of a metal mordant. Subsequently, the dye was applied to cotton fabrics using chitosan as a bio-mordant to achieve eco-friendly dyeing and functional finishing [14]. Building upon studies on sustainable dyeing techniques, Sarker et al. [15] developed and optimized a non-toxic, hypoallergenic, and eco-friendly natural dyeing process using factory tea waste as a dye source. In the study, metal mordants, including 10% (w/w) iron sulfate ($FeSO_4$, 99%) and copper sulfate ($CuSO_4$) relative to fabric weight, were employed, and three mordanting techniques—pre-mordanting, simultaneous (meta-mordanting), and post-mordanting—were compared. Among these, the meta-mordanting approach produced the most vivid color tones, with iron sulfate yielding the darkest brown shades. Furthermore, this method achieved the highest performance in both light and wash fastness, with grades of 4–5. Tensile strength tests of the jute fabrics revealed only a slight reduction in breaking strength for the dyed samples compared to the undyed fabrics. Overall, the study demonstrated that meta-mordanting is the most effective technique for achieving vibrant and durable colors on jute packaging materials while maintaining satisfactory mechanical properties.

Xia et al. [16] and Fonseca et al. [17] explored the use of agro-industrial waste materials as natural dye sources for wool, silk, and cotton fibers. Xia et al. [16] focused on spent coffee grounds and tea leaves, demonstrating that a weakly alkaline solvent system (50% ethanol, sodium bicarbonate, pH 8) yielded a pleasing brownish hue along with over 90% antibacterial effectiveness. Fonseca et al. [17] investigated grape pomace extracts, optimizing dyeing duration and assessing wash and light fastness, producing earthy tones with high fastness values. Together, these studies underscore the potential of plant-based wastes as sustainable, non-toxic alternatives for textile coloration [16, 17].

Additionally, Islam et al.[18] investigated the potential of mahogany sawdust extract as a natural dye for cotton and evaluated the fastness properties of the dyed samples using metallic mordants. The results demonstrated that the dyed fabrics exhibited very good to excellent wash fastness (4–5)

and good perspiration fastness (3–4). The study further highlighted the significance of using potassium aluminum sulfate (alum), noted for its low toxicity and minimal environmental impact, in reducing the environmental footprint of dyeing processes involving metal mordants.

The findings summarized above indicate that plant-based waste materials can serve as effective natural dye sources. However, to achieve consistent and high-quality coloration, the determination of optimum extraction and dyeing parameters is essential, as highlighted in the literature [19]. Building upon this foundation, the present study aims to provide an economical and eco-friendly dye source for the textile industry, while characterizing natural colorants extracted from the industrial waste of apple, linden, and mate tea, and exploring their dyeing potential on wool, cotton, and soybean fibers.

Experiments involving the dyeing process were conducted, utilizing a 1:1 ratio of waste to textile materials. 2 g of dried and pre-weighed vegetable waste were extracted, employing a 1:10 liquor ratio. The resulting 20 mL dye solution was then employed to dye 2 g of textile material at a 1:10 bath ratio. The impact of the extraction and dyeing phases, in addition to the varied types and concentrations of non-toxic metal mordants (e.g., ferrous sulfate ($FeSO_4$), sodium carbonate (Na_2CO_3), sodium alginate ($C_6H_7NaO_6$), calcium carbonate ($CaCO_3$), alum ($Al_2(SO_4)_3$), potassium sodium tartrate ($KNaC_4H_4O_6$), aluminum chloride ($AlCl_3$), and magnesium chloride ($MgCl_2$)) on the color attributes of the sample shades and their fastness properties was also investigated. In the present study, the CIELAB color space system was utilized for the evaluation of the samples, with the objective of achieving objective results. CIELAB, otherwise referred to as "CIE 1976 (L^*, a^*, b^*)" color space, is a system that is frequently utilized in the field of textile color measurement. Accurate measurement of CIELAB values is of great importance, especially in color matching and quality control processes. This system facilitates a more objective and precise evaluation of colors. The CIELAB system is comprised of three distinct components: the parameters of this study are L^* (lightness or tone), a^* (red-green axis), and b^* (yellow-blue axis). Furthermore, the colorfastness of the dyed samples was assessed by conducting a series of standard tests, including those for washing, crocking, perspiration, and saliva fastness, in accordance with established protocols. The metal content of the wastewater from the mordanting process was measured using inductively coupled plasma optical emission spectroscopy (ICP-OES). The ICP-OES is a powerful analytical technique that is employed in a variety of settings to determine metal concentrations in diverse samples, including wastewater, textiles, and environmental samples. The mechanism of this process entails the introduction of a sample into a plasma, thereby inducing an excitation of the atoms of the elements present. This excitation subsequently results in the emission of light at characteristic wavelengths. The intensity of the emitted light is then measured and compared with established standards to quantify the metal concentrations. While the core techniques employed in this study are rooted in well-established natural dyeing methodologies, the research proposes specific contributions that augment the extant body of knowledge in this field. In particular, the application of underutilized protein-based fibers, such as those derived from soybean, in combination with optimized mordant concentrations, remains a relatively under-researched area in existing literature. Furthermore, the study emphasizes the utilization of readily available agro-industrial and household waste materials in lieu of cultivated dye plants, thereby aligning with the principles of sustainable local resource utilization. In contrast to numerous preceding studies, this research employs meticulous spectroscopic and chromatographic analyses to enhance comprehension of the composition of the colorant and the interactions between the dye and the fibers. Consequently, the study contributes to the expanding interest in low-impact, waste-based natural dyeing methodologies, which hold potential for both scientific research and small-scale textile applications.

2 Experimental

2.1 Materials

Apple (Malus domestica) and mate tea (Ilex paraguariensis) wastes were obtained from Puccinelli-Elmataş Ltd. and Robertet Company Ltd. (Turkey), respectively. Linden tea (Tilia tomentosa) waste was collected from household sources. Bleached 100% wool yarn (Nm 8/2), cotton fabric (130 g/m^2), and soybean wick (Ne 0.85) were provided by Ipliksan Corp. and Minateks (Turkey). Analytical-grade mordants used in the study included ferrous sulfate ($FeSO_4$), sodium carbonate (Na_2CO_3), sodium alginate ($C_6H_7NaO_6$), calcium carbonate ($CaCO_3$), alum ($Al_2(SO_4)_3$), potassium sodium tartrate ($KNaC_4H_4O_6$), aluminum chloride ($AlCl_3$), magnesium chloride ($MgCl_2$), and citric acid ($C_6H_8O_7$) (Merck, Germany).

A schematic summary of the details of the study is presented in Fig. 1.

The apple (Malus domestica) is a cultivated fruit species within the Rosaceae family, and Turkey ranks fourth with 2.5 million tons [20]. The apple is a fruit that contains water, protein, invert sugar, sucrose, tannin, quercetin, flavone, crude fiber, ash, and trace amounts of minerals including manganese, copper, fluorine, magnesium, calcium, and potassium, among others [21]. It has been established that certain phenolic compounds, including quercetin, a constituent of the apple, also serve the function of coloring agents,

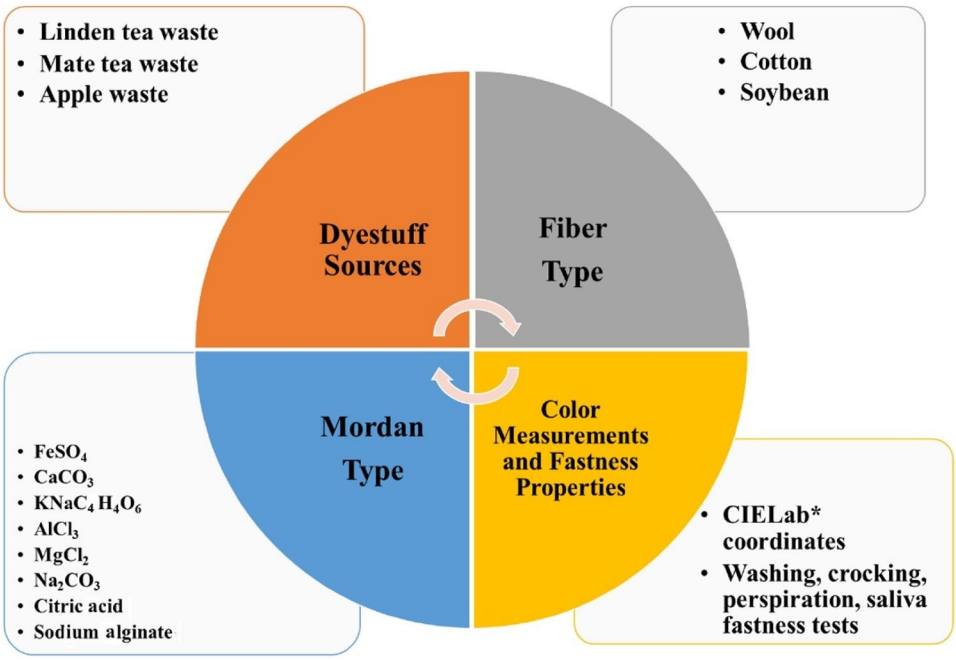

Fig. 1 Schematic summary

with the presence of oxachromium groups [22].The chemical composition of linden tea (Tilia tomentosa) encompasses various categories of phenolic compounds, predominantly consisting of quercetin glycosides (specifically rutin, quercitrin, and isoquercitrin), a kaempferol glycoside known as tiliroside, procyanidins, and various phenolic acids (including caffeic acid, p-coumaric acid, and chlorogenic acids) [23, 24]. Furthermore, the presence of polysaccharides, condensed tannins, and terpenoids was identified in the mentioned study. Mate tea (Ilex paraguariensis plant) is one of the most popular beverages in South America. It contains a variety of bioactive compounds, including caffeine, phenolic acids, and saponins. These compounds are absorbed by the body and can function either as antioxidants or as scavengers for free radicals. The bioactive compounds in question include phenolic acids, gallic acid, furans, furanones, and terpene oxides [25].

2.2 Methods

The primary activities encompassed in the study encompass the optimization of extraction time for dye extraction from designated sources, the optimization of dyeing time, the optimization of mordant quantity, the characterization of the color properties of dyed products, and the analysis of the metal content in dyeing wastewater. The details of the study and the workflow diagram are given in Fig. 2. The study also included activities such as analyzing the chemical composition of dye

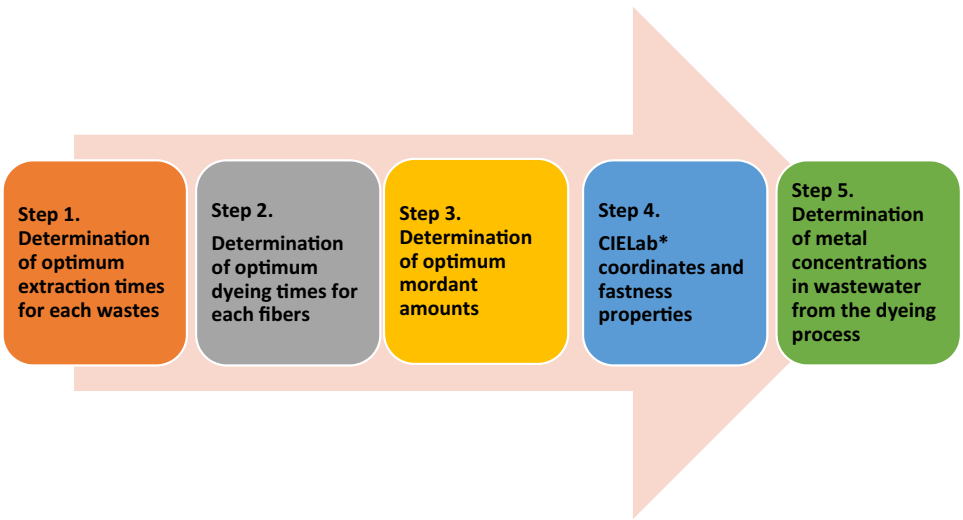

Fig. 2 Paramaters examined in the study

extracts, cationizing cotton fabric for effective dyeing, and characterizing cationic fabric.

2.2.1 Cationization of Cotton

Several studies have concentrated on modifying the ionic properties of cotton to enhance its dyeability [26–28]. In an aqueous environment, cotton fibers exhibit a slight negative charge, which can be converted into a cationic form through a process known as cationization. This modification enhances the affinity of the fibers for anionic dyes. In this study, Zetesal 2000—a formaldehyde-free quaternary polyammonium compound certified by GOTS and OEKO-TEX—was utilized for cotton cationization. A solution with a concentration of 2% (based on the weight of the fabric) was prepared at a pH of 5 using 1% acetic acid. Cotton samples were subjected to the exhaustion method at a temperature of 40 °C for a duration of 20 min, employing a liquor ratio of 1:20. The process details are provided in Table 1. Following cationization, the samples were subjected to dyeing process.

2.2.2 Optimization of Extraction Times for Plant Dyes

Vegetable waste materials such as mate tea, apple, and linden—both domestic and industrial—were utilized as natural dyestuff sources. Following a process of shade drying, the waste was subjected to grinding using an herb grinder. Each material contains a distinct combination of dyestuff components, which diffuse into the dyebath at varying rates depending on temperature and extraction time. Therefore, it is necessary to determine the optimum extraction times for each material individually.

For the extraction process, 2 g of dried plant waste was refluxed with 20 mL of water (1:10 liquor ratio) in a beaker, reflecting standard industrial dyeing machine conditions. The duration of boiling ranged from 30 min to 4 h, with boiling times of 30–60 min. The solutions were filtered and utilized to dye 2 g of wool yarn in a 1:1 (w:w) waste-to-wool ratio for a duration of 1 h. Following the dyeing process, the yarns were immersed in a solution of 1 g/L olive oil soap (Ayteks Chemicals) at a temperature of 40 °C for a duration of 30 min. This was followed by a thorough rinsing with cold water on two separate occasions. CIELAB values were measured using a Datacolor 600 spectrophotometer (Datacolor AG, Switzerland). The optimum extraction time was determined based on the sample with the highest *K/S* value, which indicates maximum color saturation [29]. The same sample also exhibited the lowest L^* value, corresponding to the darkest shade observed on the dyed fabric. Consequently, the observation that the sample with the highest *K/S* value also yielded the lowest L^* value serves to demonstrate that both parameters are mutually supportive in terms of the reliability of the obtained result. The selection of wool yarn was made because of its proven superiority in terms of affinity for natural dyes [30].

Table 1 Cationization recipe

Amount of cationization agent (%)	pH	Temperature	The time of the period
2	5–6	40 ^{O}C	20 min

2.2.3 Optimization of Dyeing Times

At this stage, the dyeing process was executed using previously determined optimum extraction times. Two grams each of raw wool yarn, cationized cotton, and soybean fiber were dyed at a 1:10 liquor ratio using the exhaustion method for 30–120 min, with 15-min interval sampling. Following the dyeing process, the samples were rinsed, treated with olive oil soap, and subsequently air-dried. The *K/S* values were utilized to evaluate the color depth, and the point at which the tone stabilized at the highest *K/S* value was designated as the optimum dyeing time for each fiber–dyestuff pair.

2.2.4 Optimization of Mordant Amount

Mordants have been shown to play a pivotal role in the process of natural dyeing, through the formation of coordinate covalent bonds between textile fibers and dye molecules. It has been demonstrated that mordanting is an effective process for enhancing the fixation of dyes onto fibers [10, 26, 31]. While multivalent metal salts, including aluminum, iron, copper, chromium, and tin, are extensively utilized as mordant agents, a considerable number of these salts are categorized as hazardous, according to the criteria delineated in Directive 2006/11/EC, due to their inherent toxicity to humans and the environment. In this study, non-toxic metal salts have been selected as mordants. Furthermore, the employment of specific acidic and basic compounds was instrumental in modifying the surface charges of fibers and dyes. This modification enhanced dye affinity through the mechanisms of hydrogen bonding and van der Waals interactions [10].

In the study, the mordants were added directly to the dye baths obtained from previous extractions and stirred for 5 min (co-mordanting method). Wool, cotton, and soybean fibers were subjected to the dyeing process, as illustrated in Fig. 3.

To ascertain the optimal mordant concentration, each mordant was tested at concentrations ranging from 1 to 5 g/L. The evaluation of the dyed samples was conducted utilizing the CIELab color space coordinates (L^*, a^*, b^*). The determination of the optimal concentrations was derived from the lowest L^* values, which are indicative of the darkest and most stable color tones [32]. The validity of these

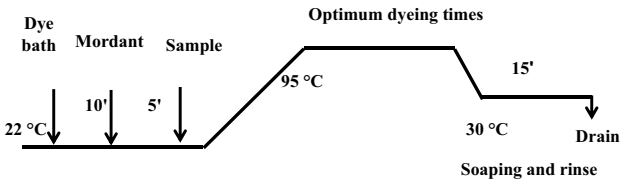

Fig. 3 Dyeing diagram for all samples

findings was further substantiated by the *K/S* values, which also demonstrated maximum color yield at the mordant concentrations corresponding to the lowest L^* values across all samples.

2.3 Characterization

The phenolic compound content of the extracts was determined via HPLC (Shimadzu, Kyoto, Japan) using an Agilent Eclipse XDB C-18 column (250 × 4.6 mm, 5 µm) and a PDA detector at 278 nm. The main phenolics identified were catechin, chlorogenic acid, caffeic acid, p-coumaric acid, and rutin. Furthermore, UV–Vis spectra of the aqueous extracts were recorded using a Perkin Elmer spectrophotometer, thus confirming the presence of flavonoids and polyphenols.

The untreated and Zetasal 2000-treated cotton fabrics were examined using Fourier transform infrared (FTIR) spectroscopy. The FTIR spectra were obtained through the utilization of a PerkinElmer FTIR System Spectrum BX, with the employment of potassium bromide disks. The total number of scans performed for each sample was 32. The absorption peaks from wave numbers 4000 to 400 cm^{-1} were analyzed.

Colorimetric measurements of the dyed products were performed using a Datacolor 600 spectrophotometer (Datacolor AG, Switzerland) under D65/10° standard observer conditions, scanning between 360–700 nm. The L^*, a^*, b^*, and ΔE values were recorded in order to assess the color properties. In this system, L^* is used to denote lightness (0 = black, 100 = white), a^* is used to denote the red-green axis (positive = red, negative = green), and b^* is used to denote the yellow-blue axis (positive = yellow, negative = blue). It is evident that elevated a^* and b^* values are indicative of heightened chromatic intensity. Color strength (*K/S* at 400 nm) and whiteness degree (Stensby) were also determined using the same equipment, and *K/S* values were calculated based on the Kubelka–Munk equation:

$$\frac{K}{S} = \frac{(1-R)^2}{2R} - \frac{\left(1-R_0\right)^2}{2R_0}$$

where R is the decimal fraction of the reflectance value of the dyed sample at the maximum absorption wavelength (nm), R_0 is that of the undyed one, and K is the absorption coefficient, and S is the scattering coefficient [33].

A series of tests were conducted in order to ascertain the colorfastness of the samples under scrutiny. The testing process encompassed evaluations of washing, crocking, perspiration and saliva fastness, in accordance with standardized protocols. The washing fastness of dyed samples was evaluated according to EN ISO 105-C06 using grayscales (ISO 105-A02, A03). The rubbing fastness was tested through the implementation of a crockmeter in accordance with the ISO 105-X12 standard. The evaluation of staining was conducted under appropriate illumination conditions, utilizing grayscales in accordance with the ISO 105-A01, A03 protocol. The evaluation scale employed ranged from 0, indicating poor performance, to 5, denoting excellent performance. The material was deemed acceptable if the rating was 3.0 or higher. The assessment of perspiration fastness was conducted under conditions of acidity and alkalinity, as delineated in ISO 105-E04. The evaluation of saliva fastness was undertaken in accordance with DIN 53160–1, employing filter papers that had been saturated with test solutions. Color changes and staining were recorded after testing.

Subsequent to the dyeing process, an analysis was conducted on the metal content present in the wastewater result from the mordant, and an assessment was undertaken of its environmental impact. For this study, the concentrations of sodium (Na), iron (Fe), calcium (Ca), potassium (K), aluminum (Al), and magnesium (Mg) in the waste dye liquor were then quantitatively analyzed through ICP-OES. The environmental impact of the wastewater was then compared to existing water quality data for textile effluents. Furthermore, the amounts of mordant remaining in the solution can be evaluated based on these analyses, allowing for further optimization.

3 Results and Discussion

3.1 Phenolic Composition and Spectroscopic Characterization of the Extracts

The phenolic profiles of aqueous extracts of apple peel, linden flower, and mate tea were analyzed using reverse-phase high-performance liquid chromatography (RP-HPLC) equipped with a diode-array detector. The following catechins were identified: (1) catechin, (2) chlorogenic acid, (3) caffeic acid, (4) p-coumaric acid, and (5) rutin. The identification of these compounds was achieved by comparing their retention times with those of authentic standards. The standard chromatogram employed for peak assignment can be found in the Supplementary Material (Fig. S1). The chromatograms of each extract are presented in Fig. 4a–c, and the quantitative results (in ppm) are provided in Table 2.

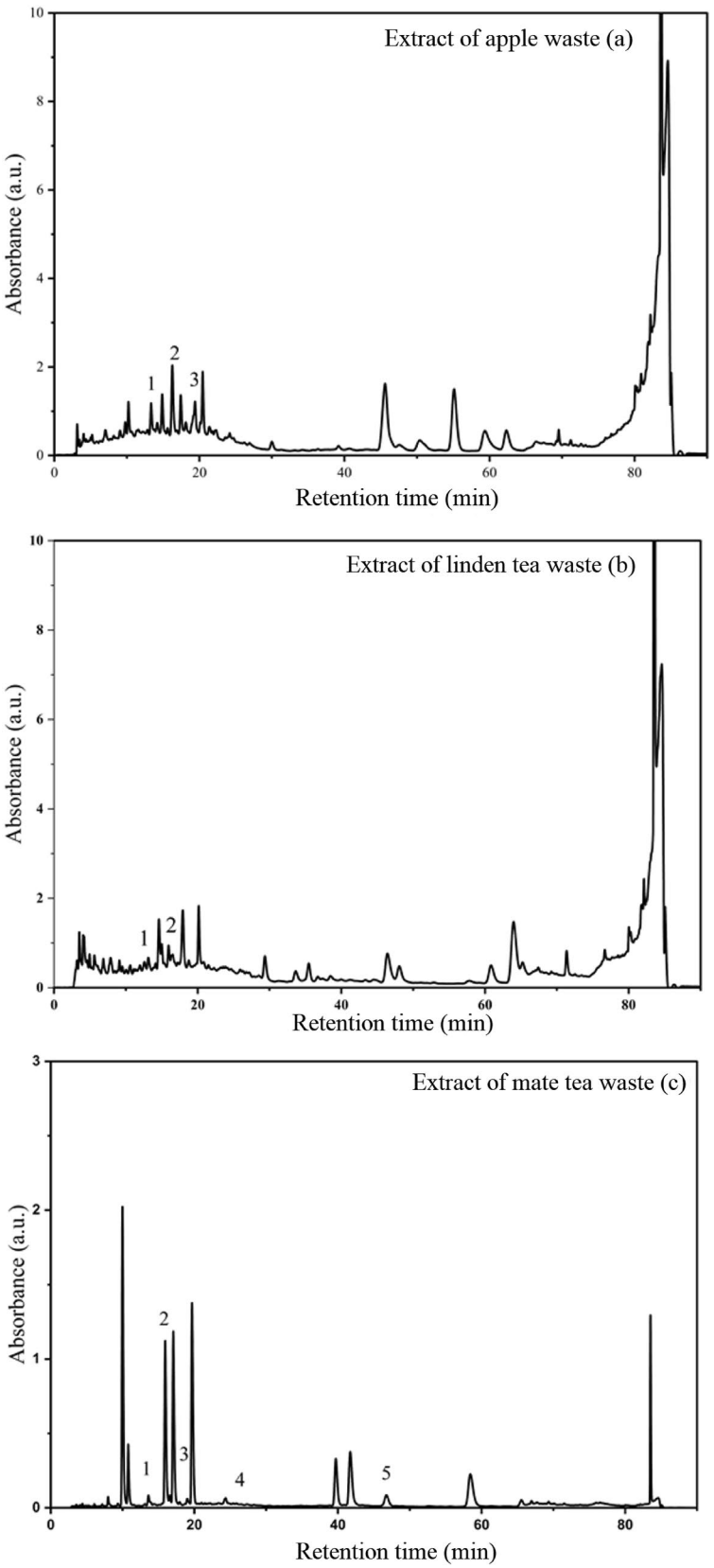

Fig. 4 HPLC chromatograms of the aqueous extracts of apple waste **a**, linden tea waste **b**, and mate tea waste **c** obtained at 278 nm. The absorbance values of the chromatograms are presented in arbitrary units (a.u.) for clarity. The raw detector signals were normalized by dividing the data by a factor of 10,000 for chromatograms (a) and (b), and by a factor of 100,000 for chromatogram (c)

Table 2 Phenolic profiles of apple, linden, and mate extracts ıdentified by HPLC analysis

Sample	Catechin (ppm)	Chlorogenic acid (ppm)	Caffeic acid (ppm)	p-Coumaric acid (ppm)	Rutin (ppm)
Apple peel extract	10.1	8.7	1.3	0.1	* (Not detected)
Linden waste extract	4.5	2.8	* (Not detected)	* (Not detected)	* (Not detected)
Mate tea waste extract	9.1	406.1	4.8	1.0	161.5

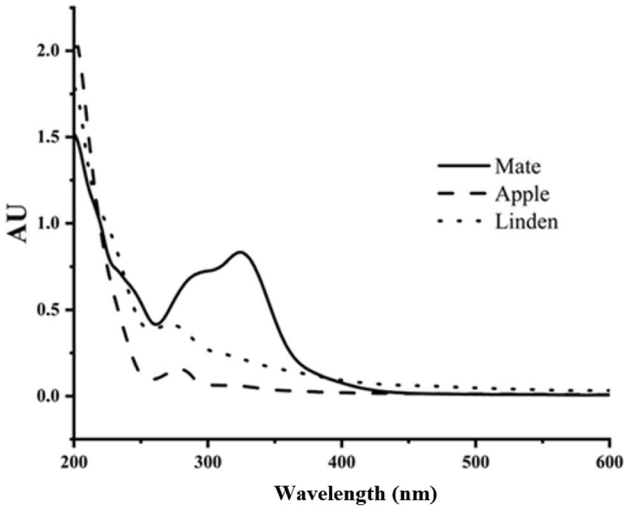

Fig. 5 UV–Vis absorption spectra of apple, linden, and mate extracts measured in the range of 200–600 nm

The results of the HPLC analysis revealed the presence of catechin (1), chlorogenic acid (2), caffeic acid (3), and p-coumaric acid (4) in the apple peel extract, while catechin (1) and chlorogenic acid (2) were identified in the linden extract. The most abundant phenolic profile was identified in the mate tea extract, which contained all five targeted compounds (1, 2, 3, 4, and 5) and exhibited the highest total phenolic content among the samples. These bioactive compounds contribute not only to the biological properties of the extracts but also play a key role in color development during the dyeing process, thus supporting their potential as natural colorants.

As demonstrated in Fig. 5, a comparison was made of the UV–Vis absorption spectra of all three samples across the 200–600 nm range. In accordance with the extant literature, it was demonstrated that all extracts exhibited maximum absorption within the 200–400 nm region. This finding thus confirmed the presence of polyphenolic structures [34]. As demonstrated by the UV–Vis spectroscopic analysis, the primary absorption bands of the apple peel and linden extracts were observed at approximately 260 nm and 380 nm, respectively. The strong absorption bands between 200 and 300 nm correspond to the characteristic range of phenolic compounds, including phenols and phenolic acids [35]. It is well established that phenols and phenolic acids typically absorb within 250–290 nm, whereas flavones and flavonols generally exhibit absorption bands between 250 and 350 nm [36].

The spectral curves of all samples remained above the baseline in the 400–450 nm region, with this feature being particularly pronounced in the mate extract. This finding indicated not only a high polyphenol content but also the presence of additional chromophoric compounds that might act as natural colorants. The findings of this study suggested that the spectra obtained for the apple peel and linden extracts were attributable to polyphenolic compounds, a conclusion that was further substantiated by the HPLC results.

When the UV spectra of the samples in the characteristic absorption region of phenolic compounds between 200 and 400 nm were examined individually (Fig. 5), the apple peel extract ranked second in terms of the intensity of the absorption bands and showed peaks that are typically associated with phenolic acids and flavonols. The predominant peak observed at 270 nm signified the presence of flavonols, including quercetin derivatives, in conjunction with other phenolic acids [37].

The UV–Vis spectrum of the linden extract exhibited relatively low absorbance intensity, indicating a lower abundance or concentration of polyphenolic compounds. The broad and low-intensity peaks provided further evidence for the presence of polyphenols, albeit in smaller quantities. It was noteworthy that a distinctive shoulder was detected in the range of 270–275 nm, which corresponded to gallic acid. This finding indicated the presence of tannin-type polyphenols in the extract, which might be responsible for the color formation observed [38, 39].

The mate extract demonstrated the highest degree of absorption intensity and the most distinct peaks among the three samples, indicating a high concentration of flavonoids, particularly chlorogenic acid derivatives. The presence of a distinct shoulder at approximately 320 nm was indicative of chlorogenic acid and caffeoylquinic acid derivatives, thereby highlighting the richness of mate tea in these compounds [40, 41]. The present findings provided further evidence for the potential of these plant-based extracts to serve as natural colorants, thereby demonstrating their applicability for the dyeing of fabrics.

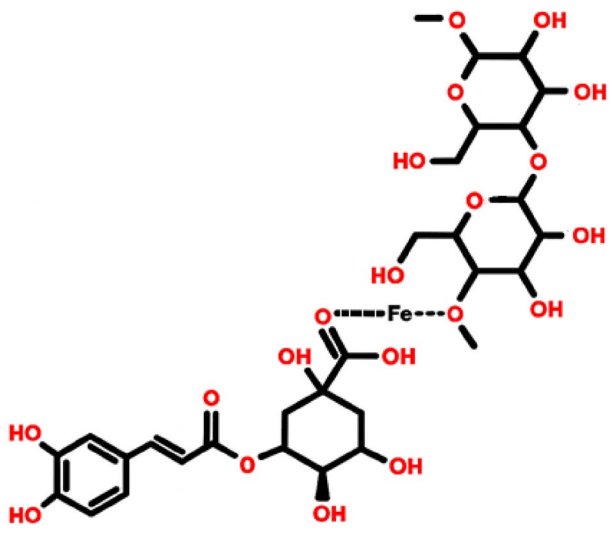

Fig. 6 Representative binding mechanism of chlorogenic acid with cellulose via divalent iron mordant

3.2 Proposed Dye–Fiber Interaction Mechanism

Figure 6 presents a proposed dyeing mechanism intended to illustrate the possible chemical interaction among a natural dye with a phenolic structure, a metallic mordant, and raw cotton fiber.

Chlorogenic acid is an ester formed from caffeic acid and quinic acid and contains a carboxylic acid group and numerous phenolic –OH groups. As previously stated, cellulose is a natural polymer formed by the polymerization of β-D-glucose units via glycosidic bonds, and it contains numerous hydroxyl (–OH) groups in the chain. In transition metal complexes, carboxylate ions function as ligands, coordinating with metal ions to form stable complexes. Divalent metal ions, including Zn^{2+}, Cu^{2+}, and Fe^{2+} facilitate bond formation between the carboxylate group and the metal ion. The bond is established through the involvement of one or two oxygen atoms. Consequently, the oxygens in the deprotonated carboxylate group of chlorogenic acid form a coordination bond with the metal center.

As illustrated in Fig. 6, the metal cation (M^{2+}) bonds to the carboxylate group of chlorogenic acid and the hydroxyl group (–OH) on cellulose. It is therefore evident that the metal cation establishes a bond between the carboxylate oxygen of chlorogenic acid and the oxygen atom in cellulose, thereby enhancing the stability of the cellulose–mordant–dye complex. This mechanism represents the potential interaction pathway in cellulose-based fibers. Similar coordination or ionic interactions are also expected to occur in protein-based fibers, such as soy and wool, depending on the availability of functional groups. In cationized cotton, positively charged ammonium groups ($–NH_3^+$) are present on the fiber surface. These groups have the capacity to interact electrostatically with negatively charged functional groups, such as the carboxylate or phenolate moieties found in phenolic dye molecules. Consequently, even in the absence of a mordant, ionic interactions or hydrogen bonding may occur between the fiber and the dye, driven by complementary charges and hydrogen donor–acceptor dynamics. Soy fiber, as reported in the literature, is protein-based and contains amino acid side chains with functional groups such as $–NH_2$ and –COOH. In a similar manner, wool fiber (keratin) comprises sulfur-containing amino acids in addition to amine and carboxyl groups. The presence of hydrogen bonds is possible between phenolic dye molecules and both soy and wool fibers, due to the fibers' functionalities. Furthermore, coordination complexation through metal ions is possible when mordants are employed. These alternative interaction routes support the applicability of the proposed binding mechanism, or its variations, across different fiber types.

3.3 Optimization of Extraction Times

At this stage, the study aimed to determine the extraction time for achieving the highest color yield from each type of waste. Figure 7 presents the *K/S* values of wool yarn samples dyed with dyes extracted at varying durations from apple, linden, and mate tea wastes. A thorough examination of the *K/S* values (i.e. color saturation) was conducted, revealing that the optimal extraction time for apple and linden was 4 h, while for mate, it was 3 h. These optimum extraction durations were then utilized as a reference for the subsequent preparation of dyeing solutions in dyeing processes. Figure 7

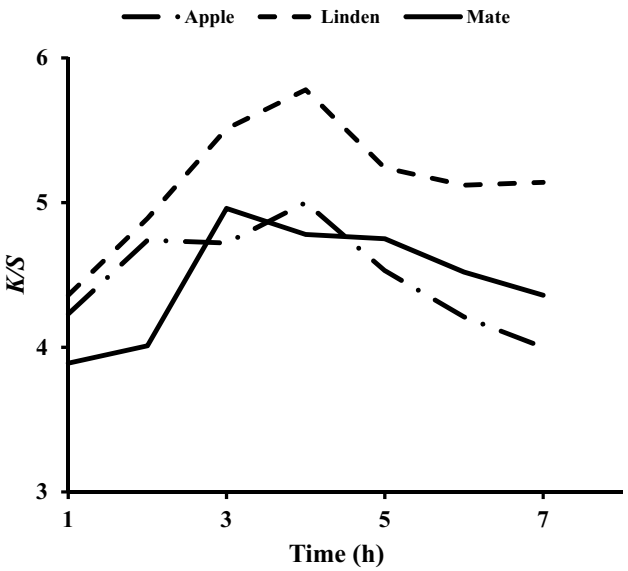

Fig. 7 Optimum extraction times for each plant-based waste material

presents the *K/S* values of wool yarn samples dyed with dyes extracted at varying durations from apple, linden and mate tea wastes.

3.4 FTIR Analysis

In this study, FTIR absorption spectroscopy was employed to verify the presence of the cationic agent Zetasal 2000 on cationized cotton fabric. The FTIR spectra of the untreated cotton fabric (black), the cotton fabric treated with the cationizing agent Zetasal 2000 (red), and the cationic agent Zetasal 2000 itself (blue) are presented in Fig. 8. In the FTIR spectrum of Zetasal 2000, characteristic N–H stretching bands between 3000–3400 cm^{-1} and a distinct carbonyl (C = O) peak around 1650 cm^{-1} are observed. In addition, the presence of C–O stretching bands near 1100 cm^{-1} is clearly evident. When the FTIR spectrum of the Zetasal-treated fabric is examined, a noticeable decrease in transmittance appears at approximately 3000–3400 cm^{-1}, 1650 cm^{-1}, and 1100 cm^{-1} compared with the untreated fabric. This reduction can be attributed to the incorporation of N–H, C = O, and C–O functional groups into the cotton fiber structure due to the application of the cationic agent. These findings confirm the successful attachment of the cationic agent to the cotton substrate. Furthermore, the reduction in band intensity within the range of 3375–1057 cm^{-1} indicates the presence of nitrogen-containing functional groups introduced during the cationization process. The appearance of these characteristic absorption bands is consistent with the presence of quaternary ammonium groups, which are typical of cationic agents such as Zetasal 2000.

3.5 Optimization of Dyeing Times

In the preliminary phase of the experimental process, the optimal extraction times for each category of waste material were ascertained. In this phase, the optimal duration for dyeing for each fiber type was identified. As previously mentioned, the highest *K/S* value observed in the dyed samples indicated the optimum dyeing time at which the textile fibers achieved maximum color yield. For the samples that were dyed with linden tea waste, the optimum dyeing time was found to be 60 min for all three fiber types. In the case of mate tea waste, the optimum dyeing time was determined to be 45 min. The optimum dyeing times for wool, cotton, and soybean fiber dyed with apple waste were found to be 75, 75, and 60 min, respectively. The optimal dyeing times corresponding to each fiber and waste type are presented in Fig. 9a-c.

3.6 Optimization of Mordant Amount

In the process of determining optimal mordant concentration, a series of dyeing experiments were conducted using different mordant concentrations. For each fiber type and mordant, the dyeing procedure was repeated three times under identical processing conditions. Table 3, 4, and 5 presents the CIELab color values of samples dyed with 1, 2, 3, 4, and 5 g/L mordant concentrations for all combinations of mordant, fiber, and dye types.

Following the triplicate dyeing processes, the CIELab color values of the dyed samples were measured, and the average values were calculated for analysis. In this study, L^* values were taken as reference; since a lower L^* value indicates a darker shade, the optimal mordant concentration for each sample was determined based on this criterion. Furthermore, the color strength values (*K/S*) also reached their highest levels at the mordant concentrations identified as optimal based on the L^* values. Consequently, it can be concluded that the L^* and *K/S* values were consistent with each other in determining the optimal mordant concentration and support the achievement of maximum color yield. The results obtained unequivocally demonstrated that elevating the mordant concentration beyond the ascertained optimal level did not result in a substantial enhancement of color depth or yield. As illustrated in Table 6, the maximum

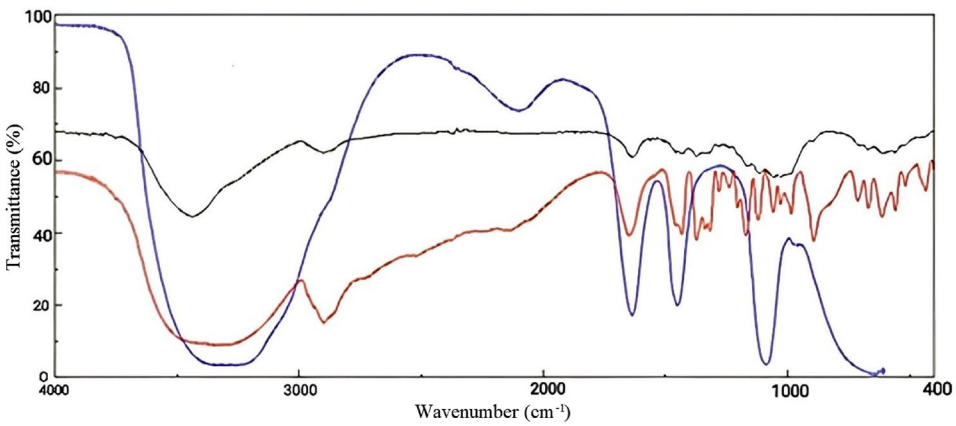

Fig. 8 FTIR spectra of Zetasal 2000 (blue), untreated cotton fabric (black), and cotton fabric treated with Zetasal 2000 (red)

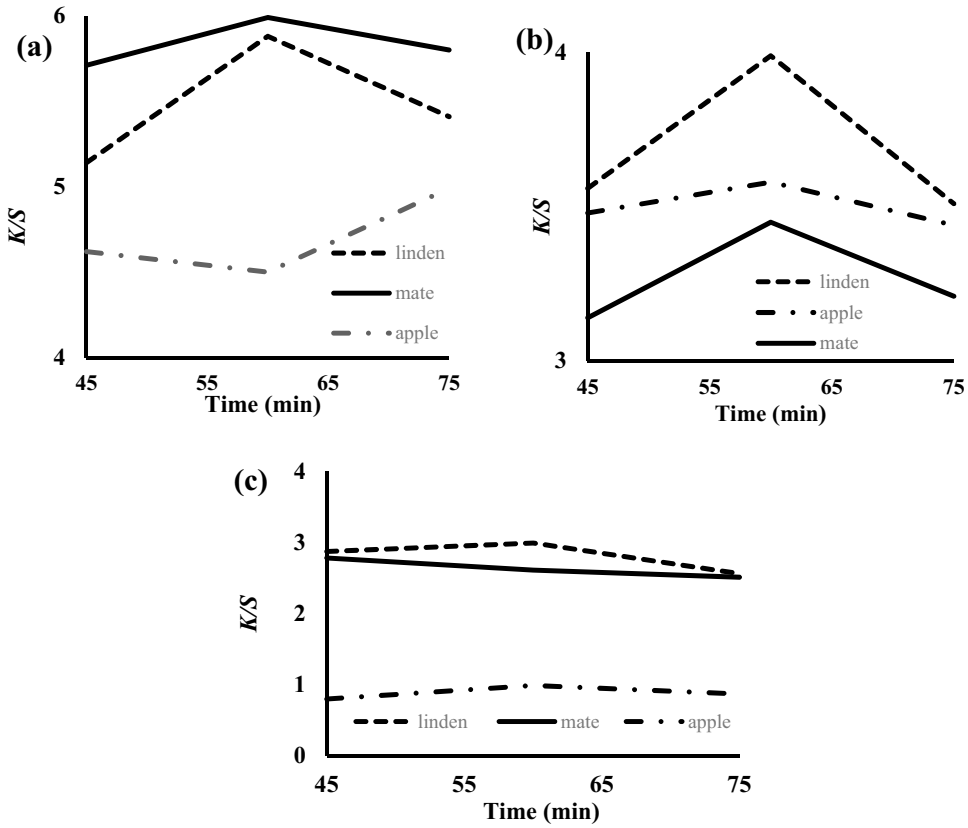

Fig. 9 The optimum dyeing times of wool fiber **a**, cotton fiber **b**, and soybean fiber **c** for each type of waste materials

permissible amounts of mordant for each dyestuff and fiber type were summarized, as outlined in the examples presented in Tables 3, 4, and 5.

3.7 Dyeing Outcomes

The photos of samples dyed with apple, mate tea, and linden tea plant extracts using different mordants were presented in Table 7. As shown in Table 7, the study resulted in a diverse range of colors that could appeal to consumers of all age groups. These shades were commonly featured in the color charts of apparel collections from various brands, as they constituted basic colors within different segments.

The mordanting process is crucial when dyeing fibers with natural dyes due to their low affinity. In other words, mordanting enhances the colorimetric and fastness properties of dyed fibers [4, 42]. To investigate the impact of mordants in this study, non-toxic metal salts were utilized. The color strength (*K/S*) behavior of dyed wool, cationized cotton fibers, and soybean fibers using apple, linden, and mate waste extracts is presented in Figs. 10, 11, and 12, respectively. As shown in Figs., the use of mordants increased the *K/S* value of dyed fibers because the formation of chemical complexes resulting from the interaction between the mordant and the fiber surface enhanced the dyeability potential [42, 43]. In this study, $FeSO_4$ demonstrated the best performance as a mordant, while apple, linden, and mate tea wastes exhibited excellent performance as eco-friendly dyes. The highest color yield on a per-fiber basis was achieved with wool fibers. This can be attributed to the greater presence of functional groups in the structure of wool fibers that enable interaction with the anionic dyestuff [30]. Satisfactory color yield performance was observed with cationized cotton, but *K/S* values were lower for soybean fibers. This difference may be related to the morphology and chemical composition of the fibers.

Soy protein fibers are typically produced by blending soy protein with other polymers such as polyvinyl alcohol and cellulose, because pure soy protein has insufficient mechanical strength for fiber formation. Therefore, the soy protein content in these fibers is usually below 50% [44, 45]. Zhan and Zeng [46] stated in their study that the shape of globular proteins is roughly spherical. They reported that the polypeptide chain folds compactly, with the hydrophobic amino acid side chains on the inside of the molecule and the hydrophilic side chains on the outside, exposed to the solvent. The globular protein structure contained in soy fibers, along with additives such as PVA added to improve fiber properties, may cause these fibers to absorb less dye. Chemical groups exposed on the surface of the protein network affect the interaction with dyes. For this reason, the *K/S* values of soybean fibers are lower than those of other fibers.

Table 3 Mean CIELab color coordinates of wool samples dyed in triplicate using different mordant concentrations and three different dye sources

Dye source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
Apple	$FeSO_4$	1	43.18	2.04	5.62	17.27	5.01
		2	35.77	3.02	5.76	18.17	5.54
		3	**34.52**	1.09	7.32	18.04	6.88
		4	35.6	0.57	6.97	17.95	6.09
		5	34.58	1.15	7.31	18.31	5.41
	Alum	1	44.77	1.15	7.25	21.35	4.74
		2	**43.40**	1.95	7.41	23.55	5.62
		3	45.81	0.63	7.24	23.72	5.21
		4	46.29	2.90	7.38	22.88	5.14
		5	48.77	3.21	7.1	20.82	5.20
	$CaCO_3$	1	**45.98**	0.69	7.6	25.86	5.92
		2	51.31	3.84	7.38	24.12	5.81
		3	52.71	0.50	7.92	24.66	5.26
		4	55.26	2.37	8.19	26.63	5.44
		5	51.68	0.76	8.21	26.34	4.21
	Citric acid	1	54.4	0.76	7.85	25.83	5.22
		2	**54.12**	0.73	7.99	26.00	5.93
		3	57.51	5.85	8.62	26.18	5.47
		4	54.57	0.71	8.10	27.37	5.81
		5	64.4	2.02	6.25	25.77	5.62
	Sodium alginate	1	56.59	2.92	22.66	6.11	5.57
		2	55.01	3.96	24.58	7.13	5.48
		3	50.98	3.28	25.07	7.62	5.67
		4	49.50	2.09	25.38	8.41	5.41
		5	**46.80**	0.69	24.70	8.27	5.82
	$AlCl_2$	1	54.73	1.48	7.33	25.73	4.43
		2	54.39	1.06	8.92	30.91	4.58
		3	50.45	2.10	11.72	35.56	4.99
		4	52.11	0.64	10.88	33.18	5.01
		5	**49.51**	2.69	10.66	33.17	5.18
	$KNaC_4H_4O_6$	1	59.04	2.21	5.21	24.26	5.01
		2	53.88	1.44	7.64	28.31	4.87
		3	52.39	0.71	7.98	29.23	5.66
		4	**51.19**	1.73	7.27	28.33	5.98
		5	53.06	0.47	8.19	27.67	5.27

Table 3 (continued)

Dye source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$MgCl_2$	1	**63.57**	2.56	3.99	21.52	5.74
		2	64.85	0.75	3.62	22.65	5.48
		3	64.15	1.56	3.65	23.00	5.41
		4	65.3	1.52	3.95	22.47	5.22
		5	65.87	0.77	3.44	22.90	4.06
	Na_2CO_3	1	52.69	1.72	0.78	23.95	4.71
		2	**51.64**	1.56	0.79	22.83	5.02
		3	55.18	1.82	0.77	22.19	4.87
		4	52.33	1.53	0.72	22.45	4.91
		5	55.32	1.21	0.81	22.23	4.36
Lınden	$FeSO_4$	1	42.82	5.75	2.19	7.77	5.87
		2	40.47	0.51	2.31	10.60	5.94
		3	**32.08**	1.08	2.95	12.05	6.44
		4	33.06	0.87	3.24	12.80	6.02
		5	36.37	1.10	3.25	12.88	5.71
	Alum	1	**51.00**	1.83	15.01	15.39	4.89
		2	53.29	0.53	15.58	14.86	3.39
		3	51.91	0.73	15.41	14.70	4.28
		4	51.29	0.97	16.51	15.13	4.51
		5	50.17	0.15	17.59	14.53	4.19
	$CaCO_3$	1	43.88	1.17	16.47	21.83	4.72
		2	46.30	0.54	15.65	22.64	4.16
		3	43.67	1.22	17.15	23.43	4.89
		4	**41.03**	1.07	19.26	24.54	5.09
		5	44.45	0.62	17.64	23.26	3.58
	Citric acid	1	49.13	0.42	10.22	14.91	3.02
		2	**49.0**8	0.27	11.73	15.69	3.61
		3	50.81	2.71	11.50	15.38	3.23
		4	53.61	1.84	11.20	16.23	3.48
		5	53.42	1.29	11.69	15.93	3.32
	Sodium alginate	1	**46.37**	4.69	14.05	14.82	5.64
		2	47.26	1.47	14.65	16.10	5.21
		3	48.10	0.80	14.57	15.65	5.17
		4	48.08	0.53	14.31	14.95	4.98
		5	48.26	0.27	16.22	14.93	4.56
	$AlCl_3$	1	53.91	1.20	11.25	22.38	3.21
		2	**53.17**	2.24	11.26	21.74	3.92
		3	56.25	0.38	11.15	22.21	3.57
		4	57.06	0.39	10.64	20.35	3.42
		5	54.63	0.37	10.83	20.01	2.85
	$KNaC_4H_4O_6$	1	45.59	3.32	16.32	15.19	4.93
		2	46.83	0.83	16.48	15.73	4.88
		3	**45.18**	1.35	17.48	14.74	5.22
		4	46.95	1.77	17.54	14.79	5.07
		5	46.14	0.36	15.00	15.37	4.74

Table 3 (continued)

Dye source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$MgCl_2$	1	**46.16**	0.50	14.28	15.63	5.87
		2	50.70	3.22	11.38	15.48	5.24
		3	46.75	1.33	12.91	15.08	5.63
		4	47.20	2.02	13.89	14.73	4.58
		5	48.69	0.62	11.76	14.97	4.33
	Na_2CO_3	1	47.73	3.51	15.31	16.26	4.56
		2	49.26	0.92	15.64	16.28	4.87
		3	47.55	2.02	16.53	14.99	4.12
		4	49.30	0.90	16.74	14.91	4.92
		5	**46.11**	0.30	15.69	14.30	5.12
Mate	$FeSO_4$	1	35.69	1.18	-1.22	6.72	5.81
		2	34.47	0.99	-1.19	6.57	5.92
		3	35.82	0.63	-1.12	6.68	5.77
		4	35.06	0.32	-0.98	4.93	6.08
		5	**34.01**	0.54	-0.92	5.02	6.23
	Alum	1	64.56	1.78	2.76	26.10	5.21
		2	65.40	1.67	2.63	25.66	4.89
		3	67.38	0.88	2.66	26.20	4.95
		4	64.23	4.17	2.95	25.53	5.17
		5	**63.93**	1.25	2.65	25.58	5.32
	$CaCO_3$	1	**52.09**	0.19	0.95	15.85	5.39
		2	53.00	1.36	0.93	15.89	5.11
		3	52.76	0.76	0.80	15.90	5.07
		4	53.90	1.65	0.56	15.41	4.87
		5	54.67	0.15	0.23	13.83	4.51
	Citric acid	1	**61.92**	0.08	6.94	24.70	5.76
		2	63.69	0.76	7.01	25.25	5.26
		3	64.78	0.35	6.37	24.15	5.14
		4	62.81	0.92	6.95	25.04	4.89
		5	63.00	1.72	6.73	24.50	4.52
	Sodium alginate	1	64.57	1.84	2.87	15.73	4.51
		2	61.94	0.10	3.93	17.33	4.72
		3	61.83	0.38	4.04	17.38	4.83
		4	62.13	0.84	3.95	17.08	5.14
		5	**60.03**	0.31	3.81	16.96	5.88
	$AlCl_3$	1	66.77	1.47	1.72	19.32	5.01
		2	**65.70**	1.77	2.42	19.56	5.28
		3	68.94	0.45	2.00	20.23	4.85
		4	68.53	0.04	1.64	19.69	4.92
		5	68.90	0.24	1.66	19.90	5.07

Table 3 (continued)

Dye source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$KNaC_4H_4O_6$	1	63.38	2.38	3.36	16.35	5.27
		2	62.19	0.82	3.89	17.26	5.18
		3	**61.08**	1.17	3.95	17.30	5.89
		4	62.17	0.49	3.87	17.12	4.83
		5	62.49	0.75	3.94	17.34	4.95
	$MgCl_2$	1	**50.66**	0.82	0.22	16.01	5.64
		2	52.99	1.26	0.65	14.53	5.16
		3	52.74	0.83	0.93	15.78	5.08
		4	53.59	1.71	0.85	15.41	4.27
		5	52.39	1.01	0.78	15.15	5.50
	Na_2CO_3	1	51.96	0.17	5.39	12.42	4.67
		2	53.41	1.55	5.37	12.62	4.78
		3	53.65	1.20	4.47	12.12	4.58
		4	52.99	1.99	5.34	11.75	4.35
		5	**51.05**	1.12	5.52	12.21	5.01

*The CIELAB color coordinates presented are the mean values obtained from the 1st, 2nd, and 3rd dyeing processes. These processes were performed under optimized chemical conditions using three natural dye sources and five different mordant concentrations (1, 2, 3, 4, and 5 g/L)

The values indicated in bold represent the lowest L* values obtained during the optimization of mordant concentration. These values correspond to the darkest shades measured within the respective dyeing processes. The lowest L* values, determined through both visual assessment and spectrophotometric measurements, indicate that the highest color depth was achieved at these specific mordant concentrations

3.8 Fastness Properties

The data pertaining to the fastness properties of the material under consideration is summarized in Tables 8, 9, 10, 11, and 12. The data indicated that the washing and rubbing fastness of the samples were excellent (4–5 to 5). Comparable outcomes were evident in relation to the staining intensity of the dyed specimens, ranging from 4 to 5 on a scale of 5. Furthermore, the samples that had undergone the dyeing process demonstrated notable fastness to perspiration when tested on all three types of fibers. The fastness levels obtained in this study are consistent with those achieved using commercially prevalent dyes in industrial dyeing processes. The findings of the study demonstrate that the fastness levels obtained from vegetable waste dyes are suitable for cotton, wool, and soybean fibers [33, 45, 47]. As demonstrated in Tables 8, 9, and 10, minimal to no color transfer or staining was observed from dyed cotton, wool, and soybean fibers to the adjacent multifiber fabric. This suggested that the vegetable waste dye was suitable for various fabric blends. This outcome is particularly noteworthy when considering that a substantial proportion of commercial fabrics are composed of two or more distinct fiber types.

Saliva fastness test results reveal that the samples generally exhibited lower fastness values, except when dyed with the potassium sodium tartrate ($KNaC_4H_4O_6$) mordant under both acidic and basic conditions. While the dyestuff extracts and mordants utilized in this study do not fall into the toxic category, these findings may restrict the application of the described procedure as a colorant for textiles intended for infants aged 1–2 years.

Finishing treatments can be applied to textile fibers to enhance saliva fastness. For this purpose, polycarboxylic acids, which can act as cross-linking agents, are particularly effective in improving fastness on cotton fabrics by enhancing fiber–dyestuff interactions. As reported in the literature [48], citric acid, a natural polycarboxylic acid, provides effective cross-linking while offering an eco-friendly and economical finishing alternative. Furthermore, additional washing steps to remove unfixed dye can contribute to improving the overall fastness performance.

3.9 Concentration of Metals in Wastewater from the Dyeing Process

In this section, considering environmental sustainability, dyeing experiments were conducted on wool, cotton, and

Table 4 Mean CIELab color coordinates of cotton samples dyed in triplicate using different mordant concentrations and three different dye sources

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
Apple	$FeSO_4$	1	53.36	0.26	6.45	8.11	2.84
		2	54.04	0.96	5.23	8.32	2.52
		3	**50.16**	0.30	2.66	8.35	3.19
		4	50.57	1.09	2.77	7.61	2.93
		5	54.01	2.79	2.57	7.24	3.01
	Alum	1	62.50	0.74	7.76	14.17	2.01
		2	62.51	0.68	8.01	14.36	2.56
		3	**61.43**	0.16	8.27	14.40	2.84
		4	63.96	1.56	7.88	13.02	2.41
		5	71.53	4.09	5.60	12.04	2.38
	$CaCO_3$	1	**66.28**	0.38	11.85	12.94	1.17
		2	68.05	0.18	11.72	12.64	0.75
		3	68.02	0.15	11.80	12.80	0.87
		4	68.49	0.18	12.32	14.68	0.96
		5	71.19	0.13	12.25	15.58	1.07
	Citric acid	1	**66.17**	0.65	7.41	11.68	2.37
		2	68.11	6.05	7.04	11.70	2.08
		3	74.95	0.58	5.83	11.18	1.52
		4	75.13	0.72	5.63	11.18	1.87
		5	74.21	0.68	5.83	11.38	1.94
	Sodium alginate	1	**59.86**	0.61	8.41	11.25	2.69
		2	62.44	1.54	7.55	11.89	1.76
		3	64.81	1.55	5.88	11.46	1.47
		4	66.21	0.25	5.57	11.11	2.08
		5	64.68	3.15	5.81	11.40	2.24
	$AlCl_2$	1	77.18	0.15	9.14	11.29	1.58
		2	76.91	0.24	8.69	10.86	2.08
		3	77.16	0.33	8.70	10.90	2.17
		4	77.26	0.18	9.06	11.05	1.39
		5	**74.66**	1.13	7.83	10.44	2.31
	$KNaC_4H_4O_6$	1	62.93	0.23	14.28	14.06	1.05
		2	62.78	0.83	14.07	13.99	0.96
		3	**60.78**	0.03	14.85	14.65	1.89
		4	64.67	0.39	14.01	13.99	0.84
		5	64.88	0.83	13.36	13.59	1.20

Table 4 (continued)

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$MgCl_2$	1	**73.29**	1.64	8.87	11.34	2.01
		2	75.65	0.49	8.55	10.83	0.58
		3	76.15	0.29	8.83	11.06	0.91
		4	75.95	0.34	9.23	11.38	0.77
		5	73.33	1.18	7.64	10.18	1.65
	Na_2CO_3	1	55.56	0.03	15.89	16.49	1.12
		2	56.13	0.26	15.90	16.48	1.08
		3	55.82	0.63	15.65	16.41	0.74
		4	55.51	0.15	15.05	16.09	1.06
		5	55.23	0.33	15.20	16.13	1.47
Lınden	$FeSO_4$	1	55.58	2.40	2.70	12.22	4.72
		2	55.31	1.10	2.96	15.26	4.12
		3	**46.07**	1.41	3.18	10.86	5.35
		4	46.26	0.71	3.32	11.17	4.08
		5	47.01	1.54	3.38	6.96	5.03
	Alum	1	74.83	0.86	6.20	16.04	1.22
		2	**73.94**	0.56	7.44	16.96	**2.27**
		3	75.09	0.33	6.98	16.33	1.48
		4	82.27	0.08	3.33	16.85	1.53
		5	78.20	3.36	4.97	16.57	1.89
	$CaCO_3$	1	65.58	1.17	14.54	12.35	2.09
		2	65.79	1.13	14.43	12.25	3.07
		3	64.07	0.73	15.50	13.16	2.94
		4	**55.44**	0.57	16.58	14.05	3.51
		5	56.78	0.96	16.02	13.27	3.08
	Citric acid	1	**65.55**	1.67	5.96	9.53	3.67
		2	67.86	0.58	5.23	8.73	3.52
		3	67.98	1.56	3.39	6.35	3.23
		4	67.82	0.27	3.46	6.55	3.17
		5	68.32	0.34	3.24	6.40	2.92
	Sodium alginate	1	73.80	0.51	8.14	15.16	3.10
		2	71.80	1.74	8.12	16.76	2.75
		3	71.17	0.49	8.20	17.48	2.30
		4	72.08	0.29	7.25	16.36	1.93
		5	**70.89**	0.33	7.65	16.92	**3.28**
	$AlCl_3$	1	72.62	0.08	6.72	15.90	2.98
		2	**71.00**	0.33	6.91	16.35	**3.64**
		3	77.66	0.18	3.21	15.76	3.34
		4	77.38	0.62	3.01	15.60	3.04
		5	77.64	0.31	3.33	16.10	2.74
	$KNaC_4H_4O_6$	1	56.22	0.88	16.90	13.48	2.59
		2	53.85	0.19	15.73	12.23	2.47
		3	**51.45**	0.51	15.80	12.22	3.81
		4	56.85	0.78	15.21	10.20	3.58
		5	56.28	0.74	15.29	11.45	3.14

Table 4 (continued)

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$MgCl_2$	1	**52.46**	0.70	9.40	13.73	3.05
		2	59.75	1.11	8.83	16.02	2.69
		3	55.85	0.43	9.53	14.08	2.25
		4	55.80	0.39	9.72	14.40	1.84
		5	62.16	0.89	9.09	15.39	1.61
	Na_2CO_3	1	60.10	1.27	0.14	22.59	2.75
		2	58.99	1.94	0.20	22.17	2.05
		3	59.35	1.03	0.18	20.90	2.84
		4	59.70	0.60	0.17	21.89	2.61
		5	**57.18**	0.96	0.21	22.09	3.06
Mate	$FeSO_4$	1	38.41	0.67	-0.15	5.49	4.89
		2	37.98	0.34	-0.08	5.41	4.28
		3	**36.10**	6.27	-0.06	5.52	5.44
		4	38.96	0.41	-0.04	5.55	4.95
		5	39.13	0.16	0.25	5.63	5.00
	Alum	1	61.75	0.31	4.00	26.94	1.87
		2	**60.04**	0.14	4.28	27.76	2.41
		3	64.20	0.60	3.32	17.18	1.93
		4	65.28	0.90	3.49	26.73	2.20
		5	64.20	0.60	3.23	16.51	2.16
	$CaCO_3$	1	58.79	0.71	3.55	15.88	2.94
		2	58.70	1.01	3.53	15.91	2.29
		3	59.62	0.64	3.58	15.71	2.93
		4	**55.58**	0.29	3.66	11.64	3.47
		5	56.76	0.07	3.58	11.74	1.94
	Citric acid	1	**54.05**	0.47	3.72	10.39	3.74
		2	55.84	0.17	3.69	10.36	1.53
		3	56.15	0.36	3.80	10.41	1.93
		4	56.11	0.54	3.73	10.33	2.20
		5	63.94	0.28	3.43	11.46	1.93
	Sodium alginate	1	**60.05**	0.14	4.96	15.18	3.51
		2	62.89	1.13	4.84	15.37	1.94
		3	64.73	1.30	4.63	15.67	2.29
		4	65.91	0.24	4.42	15.76	1.93
		5	64.84	0.77	4.11	15.59	2.70
	$AlCl_3$	1	81.34	0.10	-0.92	17.63	3.21
		2	82.40	2.25	-0.86	17.47	2.20
		3	**76.91**	0.24	-1.57	18.38	3.86
		4	81.98	3.75	-1.02	21.02	1.93
		5	79.89	0.44	-0.41	22.38	2.44

Table 4 (continued)

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$KNaC_4H_4O_6$	1	**54.0**4	0.17	3.52	13.47	3.97
		2	54.55	0.72	3.51	13.53	1.54
		3	54.69	0.52	3.56	13.50	2.78
		4	54.87	0.65	3.52	12.77	2.99
		5	55.89	0.62	3.54	12.41	3.01
	$MgCl_2$	1	**62.38**	0.31	3.28	25.93	3.36
		2	63.77	1.33	4.05	19.00	2.21
		3	64.15	1.46	3.35	24.84	3.04
		4	65.70	0.55	3.30	24.70	1.57
		5	65.41	0.72	4.17	15.60	2.81
	Na_2CO_3	1	**50.97**	0.18	4.53	11.62	3.01
		2	54.16	0.29	5.38	12.40	0.91
		3	54.15	0.08	5.39	12.42	0.85
		4	53.41	0.66	5.43	12.37	1.26
		5	53.82	0.69	5.37	12.65	2.36

*The CIELAB color coordinates presented are the mean values obtained from the 1st, 2nd, and 3rd dyeing processes. These processes were performed under optimized chemical conditions using three natural dye sources and five different mordant concentrations (1, 2, 3, 4, and 5 g/L)

The values indicated in bold represent the lowest L* values obtained during the optimization of mordant concentration. These values correspond to the darkest shades measured within the respective dyeing processes. The lowest L* values, determined through both visual assessment and spectrophotometric measurements, indicate that the highest color depth was achieved at these specific mordant concentrations

soybean fibers using vegetable waste extracts with various mordant types. In the study, mordant optimizations were performed based on color darkness. For this reason, the exhaustion rate of the mordants were analyzed (i.e., how much bound to the fiber versus how much remained in the dye bath as waste). Because one of the primary objectives of the study was to optimize the mordant quantity based on wastewater parameters and to ensure a genuinely environmentally friendly process. In order to assess the environmental implications of the mordants and auxiliary chemicals used, their regulatory status and sustainability profile were examined based on current literature and relevant directives. None of the auxiliary chemicals and mordant metals used in the study are included in the "Materials Restricted Substance List" of the IPPC's "Best Available Techniques Reference Document for the Textile Industry" published in 2023 [49].The same IPPC document also states that natural dyes are "free from priority chemicals (hazardous or toxic species), thus reducing the impact of the dyeing process in the wastewater streams." Furthermore, a Life Cycle Assessment (LCA) indicated that the use of natural dyes has a significantly smaller water and CO_2 footprint and a less harmful impact on human health. As the literature lacks specific regulations on metal concentrations for textile or industrial wastewater, we based our evaluation of wastewater and mordant concentrations on the European Union's Urban Wastewater Treatment Directive (91/271/EEC) and the "Pollution from Hazardous Substances Discharged to the Water Environment" directive (2006/11/EC) [50]. It is important to note that none of the mordants utilized in this work fall under the toxic classification according to both EU directives.

Based on these regulatory and sustainability considerations, the residual metal concentrations in the dye baths were analyzed to evaluate the efficiency of mordant usage in the present study. As illustrated in Table 13, residual metal concentrations in the dye bath after dyeing. Non-toxic metal salts were utilized as mordants, the analysis of residual metals in the dye bath was conducted to with objective of minimizin waste and optimize mordant usage. For instance, the optimal $FeSO_4$ amount for wool dyeing with apple pulp was found to be 3 g/L. This means it contains approximately 1100 ppm iron. Reducing the $FeSO_4$ concentration from 3 g/L to 2 g/L decreased the iron content in the bath to approximately 738 ppm, with average L^* values of 35.77 and 34.52, respectively, indicating only a slight darkening despite a 362 ppm decrease in mordant concentration. However, wastewater analysis showed 986 ppm of iron remained in the bath. This

Table 5 Mean CIELab color coordinates of soybean samples dyed in triplicate using different mordant concentrations and three different dye sources

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
Apple	$FeSO_4$	1	60.19	3.08	0.87	-7.12	1.27
		2	60.87	2.49	0.38	-7.13	0.91
		3	59.67	3.90	0.34	-6.52	0.78
		4	**57.67**	2.13	0.54	-6.07	1.49
		5	58.71	2.37	0.37	2.24	1.17
	Alum	1	**73.08**	0.50	2.15	10.85	1.12
		2	74.52	1.01	2.61	11.14	1.51
		3	78.30	0.55	2.43	11.10	1.09
		4	76.69	1.39	2.71	11.39	0.76
		5	77.26	0.73	2.67	11.12	0.79
	$CaCO_3$	1	**81.34**	2.04	3.93	14.09	0.97
		2	82.13	0.67	3.73	13.16	0.27
		3	82.09	0.16	3.52	13.51	0.18
		4	82.75	0.26	3.67	13.95	0.59
		5	82.57	0.12	3.18	13.49	0.44
	Citric acid	1	**73.74**	0.85	2.48	8.99	2.02
		2	77.30	2.15	3.57	14.03	0.75
		3	79.94	0.72	3.48	14.52	0.87
		4	80.02	1.84	3.64	14.63	1.24
		5	80.95	1.55	3.58	14.33	1.69
	Sodium alginate	1	84.94	1.25	3.45	14.46	0.98
		2	84.39	0.53	3.22	14.74	1.29
		3	85.04	1.14	2.86	14.47	1.57
		4	**80.08**	0.72	3.04	14.35	**2.07**
		5	81.66	0.58	2.89	14.58	1.64
	$AlCl_2$	1	80.59	1.46	3.74	13.66	0.12
		2	79.11	1.07	3.52	13.61	0.48
		3	**78.88**	**0.87**	3.76	13.89	0.69
		4	80.77	2.44	3.70	13.78	0.21
		5	81.90	1.14	3.72	13.90	0.38
	$KNaC_4H_4O_6$	1	**79.71**	1.22	4.19	14.30	0.84
		2	81.60	0.83	4.15	13.90	0.59
		3	81.93	0.69	3.98	13.62	1.07
		4	80.42	1.05	4.25	13.76	1.26
		5	82.42	0.76	4.30	14.04	1.18

Table 5 (continued)

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$MgCl_2$	1	**79.96**	0.31	2.59	12.81	1.79
		2	81.71	1.57	2.48	12.34	1.22
		3	82.53	0.29	2.72	12.85	1.47
		4	83.36	0.29	2.83	13.12	0.96
		5	84.60	0.92	3.06	13.12	0.84
	Na_2CO_3	1	83.68	0.47	2.89	13.98	0.62
		2	82.69	2.76	3.33	14.70	0.57
		3	83.49	1.74	3.33	14.45	0.64
		4	**80.69**	1.53	3.13	14.40	0.74
		5	83.49	1.06	2.88	13.49	0.21
Lınden	$FeSO_4$	1	44.03	1.97	1.55	2.87	1.99
		2	**41.35**	1.85	1.42	3.06	3.18
		3	45.29	0.33	51.66	2.37	2.24
		4	46.10	0.13	1.44	2.88	2.71
		5	46.85	0.28	1.48	2.83	2.08
	Alum	1	63.78	2.34	11.39	14.27	1.52
		2	65.17	0.78	10.35	13.87	1.87
		3	64.08	0.90	11.14	14.08	1.76
		4	**60.71**	0.43	11.19	14.66	**2.11**
		5	61.51	0.62	11.56	15.08	1.83
	$CaCO_3$	1	64.34	1.01	8.96	14.24	2.27
		2	63.34	1.22	9.40	14.40	1.85
		3	**59.63**	0.55	9.52	13.94	2.84
		4	61.52	0.73	9.35	15.38	1.97
		5	61.49	0.65	8.59	15.71	2.06
	Citric acid	1	68.27	1.29	9.27	15.00	1.45
		2	65.70	1.39	9.30	15.41	0.86
		3	**64.97**	1.51	9.18	15.45	2.02
		4	66.85	2.48	9.21	14.59	0.73
		5	68.44	0.70	9.59	15.21	1.58
	Sodium alginate	1	**64.67**	0.65	8.72	15.41	2.04
		2	65.33	0.50	9.04	14.43	1.69
		3	66.70	0.48	9.57	15.51	1.85
		4	65.57	0.68	8.70	15.46	1.52
		5	65.96	0.82	9.30	15.40	1.74
	$AlCl_3$	1	63.54	1.28	10.65	12.64	1.47
		2	**61.62**	0.52	10.20	12.64	**2.17**
		3	62.56	0.45	9.80	12.73	1.82
		4	63.19	0.75	9.73	12.68	1.94
		5	64.46	0.45	9.73	12.77	2.01
	$KNaC_4H_4O_6$	1	**63.68**	0.93	12.12	13.45	**2.48**
		2	64.38	0.13	12.36	13.50	1.90
		3	64.25	0.29	13.06	13.49	1.84
		4	64.64	0.33	13.02	13.60	2.14
		5	65.08	0.79	13.31	14.21	2.03

Table 5 (continued)

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$MgCl_2$	1	74.20	0.83	3.57	14.23	2.08
		2	**72.75**	0.40	4.54	13.79	2.76
		3	75.09	0.15	4.58	13.72	1.86
		4	74.64	0.45	4.29	13.65	1.09
		5	75.06	0.80	4.22	13.41	1.91
	Na_2CO_3	1	61.43	1.46	6.87	8.47	1.94
		2	60.63	2.16	6.70	8.47	1.85
		3	61.20	1.04	6.36	8.04	2.07
		4	**59.64**	0.60	6.45	7.98	2.37
		5	62.25	1.13	7.04	8.41	2.00
Mate	$FeSO_4$	1	51.78	0.83	− 1.33	2.75	1.81
		2	**50.98**	2.06	− 1.30	2.66	2.54
		3	52.73	0.56	− 1.41	2.45	1.73
		4	53.52	0.27	− 1.45	2.60	2.08
		5	54.66	0.57	− 1.55	2.76	2.13
	Alum	1	**64.98**	1.05	0.06	20.25	2.43
		2	72.46	0.57	0.58	22.95	1.94
		3	72.86	0.67	0.36	22.79	2.01
		4	71.72	0.58	1.30	21.65	1.82
		5	73.69	1.38	1.15	21.82	1.47
	$CaCO_3$	1	75.72	1.01	2.43	19.72	1.08
		2	**71.20**	2.13	2.35	19.91	1.88
		3	73.24	0.79	2.18	19.21	1.19
		4	73.60	1.21	2.22	20.57	0.78
		5	74.28	0.77	2.04	21.04	1.07
	Citric acid	1	75.34	1.08	3.48	17.05	1.98
		2	75.14	0.45	2.93	17.41	1.72
		3	**74.94**	0.35	3.31	17.77	2.84
		4	75.14	0.24	2.99	17.40	1.54
		5	75.64	0.38	3.85	17.62	2.04
	Sodium alginate	1	76.08	1.16	2.19	15.81	1.92
		2	76.52	2.01	2.77	15.93	2.09
		3	**74.04**	0.43	2.48	15.92	**2.27**
		4	74.87	0.28	2.38	14.84	0.98
		5	75.63	0.58	1.92	15.62	1.47
	$AlCl_3$	1	73.45	1.86	0.04	20.51	1.87
		2	**68.46**	5.61	0.28	18.05	2.72
		3	71.07	1.79	0.31	18.47	2.35
		4	71.17	1.14	0.36	15.89	2.45
		5	72.74	0.32	-0.52	17.03	1.58

Table 5 (continued)

Dye Source	Mordant	Mordant Amount (g/L)	CIELab color coordinates ($L^*/a^*/b^*$)				
			L^* Mean	L^* SD	a^* Mean	b^* Mean	*K/S* Mean
	$KNaC_4H_4O_6$	1	75.31	4.15	0.36	20.30	2.08
		2	74.48	1.82	0.28	19.77	1.25
		3	**73.81**	0.68	0.50	20.11	2.84
		4	75.16	0.47	0.31	20.07	1.69
		5	74.97	0.13	0.43	19.61	1.94
	$MgCl_2$	1	73.96	0.41	− 0.07	21.82	1.57
		2	**72.76**	2.24	− 0.05	21.13	2.67
		3	74.83	1.06	− 0.18	19.53	1.41
		4	74.51	0.50	− 0.16	20.53	1.95
		5	75.03	0.41	− 0.19	20.51	2.01
	Na_2CO_3	1	75.41	1.53	− 0.46	11.59	1.24
		2	**72.98**	0.69	− 0.59	11.43	1.56
		3	73.80	0.19	− 0.37	11.28	0.85
		4	74.56	0.58	− 0.47	44.42	0.97
		5	75.14	0.46	− 0.66	11.29	1.12

*The CIELAB color coordinates presented are the mean values obtained from the 1st, 2nd, and 3rd dyeing processes. These processes were performed under optimized chemical conditions using three natural dye sources and five different mordant concentrations (1, 2, 3, 4, and 5 g/L)

The values indicated in bold represent the lowest L* values obtained during the optimization of mordant concentration. These values correspond to the darkest shades measured within the respective dyeing processes. The lowest L* values, determined through both visual assessment and spectrophotometric measurements, indicate that the highest color depth was achieved at these specific mordant concentrations

Table 6 The optimum mordant amounts determined for each dye and fiber type

Sample	Waste	Optimum mordant amounts (g/L)								
		Alum	$CaCO_3$	Citric acid	Sodium alginate	$AlCl_3$	$KNaC_4H_4O_6$	$MgCl_2$	$FeSO_4$	Na_2CO_3
Wool	Apple	2	1	2	5	5	4	1	3	2
	Linden	5	4	2	1	2	3	1	3	5
	Mate	5	1	1	5	2	3	1	2	5
Cotton	Apple	3	1	1	1	5	3	1	4	5
	Linden	2	4	1	5	2	3	1	3	5
	Mate	2	4	1	1	3	1	1	3	1
Soybean	Apple	1	1	1	4	3	1	1	3	4
	Linden	4	3	2	1	2	1	2	1	4
	Mate	1	2	3	3	2	3	2	2	2

indicates that a significant proportion of added metal remained as waste, suggesting that minimizing mordant amounts not only reduces environmental load but also lowers process costs. Thus, careful consideration of both dyeing performance and environmental sustainability allows for an optimized, eco-friendly dyeing process.

4 Conclusions

In the field of sustainable textiles, researchers have proposed a variety of strategies. Among these, one of the most effective approaches for achieving textile sustainability is

Table 7 The color tones of samples dyed with apple, linden, and mate tea waste extracts using different mordants

Cotton		Wash Fastness (Staining)							Rubbing Fastness		
	Mordant	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	Change	Dry	Wet	Change
Linden	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	5	5	5	5	5	5	5	5	5	5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
Apple	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	5	5	5	5	5	5	4/5	5	4/5	5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
Mate	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	5	5	5	5	5	5	4/5	5	5	5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5

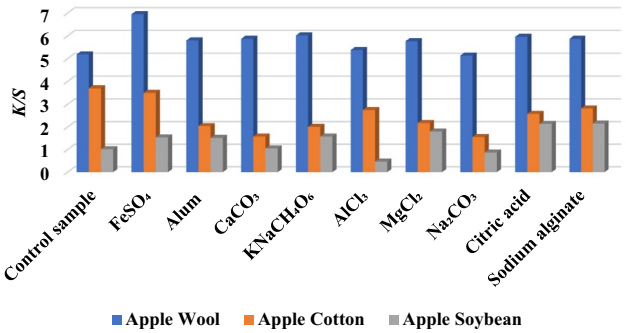

Fig. 10 *K/S* values (at 400 nm) of samples dyed with apple waste extract according to various mordant concentrations

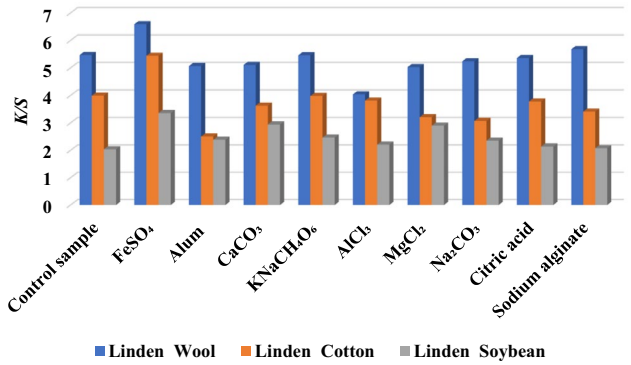

Fig. 11 *K/S* values (at 400 nm) of wool samples dyed with linden tea waste extract according to various mordant concentrations

the application of environmentally friendly chemicals in the dyeing, printing, and finishing processes of textile substrates. This is particularly important because textile wet processing is recognized as one of the most polluting aspects of the textile industry, contributing significantly to the generation of colored wastewater. One promising approach to mitigate this pollution is the utilization of natural materials. In this context, apple, linden, and mate tea plant residues represent environmentally friendly and cost-effective sources of natural dyes for wool, cotton, and soybean fibers.

In this study, to evaluate the dyeing potential of these plant residues, the phenolic composition of aqueous extracts

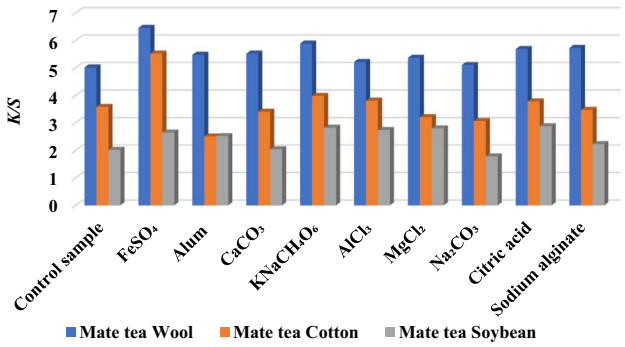

Fig. 12 *K/S* values (at 400 nm) of samples dyed with mate tea waste extract according to various mordant concentrations

obtained from apple peel, linden flower, and mate tea residues was first characterized using reverse-phase high-performance liquid chromatography (RP-HPLC) equipped with a diode-array detector. The phenolic compounds specifically targeted for detection in this study were catechin, chlorogenic acid, caffeic acid, p-coumaric acid, and rutin. According to the results, all five phenolics were detected in the mate tea extract, which also exhibited the highest total phenolic content, while apple and linden extracts contained varying levels of catechin, chlorogenic acid, and other compounds. These phenolic compounds, in addition to being responsible for the bioactive properties of the extracts, play a key role in color formation during dyeing.

Further confirming the presence of polyphenolic structures, UV–Vis absorption spectra showed strong absorption bands in the 200–400 nm region, consistent with phenolic acids and flavonols. Notably, mate extract exhibited the highest absorbance intensity with distinct shoulders around 320 nm, indicating the predominance of chlorogenic acid derivatives. These spectroscopic and chromatographic analyses together highlight the potential of these plant-based extracts as natural dyes.

Given their promising chemical composition, the study also explores the environmental benefit of repurposing plant residues. By transforming apple, linden, and mate tea wastes

Table 8 Wash and rubbing fastness properties of the dyed wool samples

Wool		Wash fastness (Staining)							Rubbing fastness		
	Mordant	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	Change	Dry	Wet	Change
Linden	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	5	5	5	5	5	5	5	5	5	5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
Apple	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	4	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	4	4	4	4	4	5	5	5
	$KNaC_4H_4O_6$	5	4/5	4	4	4	4	4	5	5	5
	$MgCl_2$	5	4/5	4	4	4	4	4	5	5	5
	$FeSO_4$	4/5	5	5	5	5	5	5	5	4	4/5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
	Alum	4	4/5	4	4	4/5	4	4	4	4	4
Mate	$CaCO_3$	4	4/5	4	4	4/5	4	4	4	4	4
	Citric acid	4	4/5	4	4	4/5	4	4	4	4	4
	Sodium alginate	4	4/5	4	4	4/5	4	4	4	4	4
	$AlCl_3$	4	4/5	4	4	4/5	4	4	4	4	4
	$KNaC_4H_4O_6$	4	4/5	4	4	4/5	4	4	4	4	4
	$MgCl_2$	4	4/5	4	4	4/5	4	4	4	4	4
	$FeSO_4$	4	4/5	4	4	4/5	4	4	4	4	4
	Na_2CO_3	4	4	4	4	4/5	4	4	4	4	5

Table 9 Wash and rubbing fastness properties of the dyed cotton samples

Cotton		Wash Fastness (Staining)							Rubbing Fastness		
Linden	Mordant	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	Change	Dry	Wet	Change
	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	5	5	5	5	5	5	5	5	5	5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
Apple	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	5	5	5	5	5	5	4/5	5	4/5	5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
Mate	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	5	5	5	5	5	5	4/5	5	5	5
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5

into natural dye sources, this research presents a potential solution to plant waste management. Additionally, a diverse range of color shades was achieved across all three fiber types, appealing to a broad consumer base.

To ensure the development of a fully eco-friendly dyeing process, optimization studies were conducted for both extraction and dyeing stages. *K/S* values, which indicate color saturation, were used as the primary criterion for determining optimal extraction durations. For apple and linden waste, the highest *K/S* values were obtained after 4 h of extraction, while for mate tea waste, the optimal time was 3 h. These extraction times corresponded to lower L values, confirming deeper and more saturated shades.

Regarding fiber-dye interactions, Fig. 6 illustrates a proposed dyeing mechanism showing possible chemical bonding among a phenolic natural dye, a metallic mordant, and raw cotton fiber. In this model, chlorogenic acid—richly present in all extracts, particularly mate—forms coordination complexes with divalent metal ions such as Fe^{2+} or Zn^{2+}. These metal ions bridge the dye and fiber by bonding with the carboxylate groups of the dye and the hydroxyl groups of cellulose, thereby enhancing dye fixation. This mechanism was further extended to other fiber types, including cationized cotton, soy, and wool. In cationized cotton, positively charged $–NH_3^+$ groups may interact ionically with negatively charged dye molecules. In protein-based fibers such as soy and wool, hydrogen bonding and coordination via available amino acid side chains can occur. These mechanisms collectively support the versatility and applicability of the proposed model.

Table 10 Wash and rubbing fastness properties of the dyed soybean samples

Soybean		Wash Fastness (Staining)							Rubbing Fastness		
	Mordant	Wool	Acrylic	Polyester	Nylon	Cotton	Acetate	Change	Dry	Wet	Change
Linden	Alum	5	5	5	5	5	5	5	5	5	4/5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	5	5	5	5
	$AlCl_3$	5	5	5	5	5	5	4/5	5	5	5
	$KNaC_4H_4O_6$	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5	4/5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	4/5	4/5	4/5	4/5	4/5	4/5	4	4/5	4	4
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
Apple	Alum	5	5	5	5	5	5	5	5	5	5
	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	5	5	5	5	5	5	5	5	5	5
	Sodium alginate	5	5	5	5	5	5	4/5	5	4	4
	$AlCl_3$	5	5	5	5	5	5	5	5	5	5
	$KNaC_4H_4O_6$	5	5	5	5	5	5	5	5	5	5
	$MgCl_2$	5	5	5	5	5	5	5	5	5	5
	$FeSO_4$	4	4	5	5	4	4/5	4/5	4	3/4	4
	Na_2CO_3	5	5	5	5	5	5	5	5	5	5
	Alum	4/5	5	5	5	5	5	5	5	5	4/5
Mate	$CaCO_3$	5	5	5	5	5	5	5	5	5	5
	Citric acid	4	4	4	4/5	4/5	4/5	4	5	5	5
	Sodium alginate	4	4	4	4	4	4	4	4	4	4/5
	$AlCl_3$	5	4	5	5	5	5	4/5	5	5	5
	$KNaC_4H_4O_6$	4/5	4	5	5	5	5	4/5	5	5	5
	$MgCl_2$	4/5	5	5	5	5	5	4/5	5	5	5
	$FeSO_4$	4	4	4	4	4	4	3	4	4	4
	Na_2CO_3	5	5	5	5	5	5	4/5	5	5	5

The dyeing procedures were optimized according to the *K/S* values of the dyed samples. Optimal dyeing times were determined as follows: for apple extract, 75 min for wool and cationized cotton fibers, and 60 min for soybean fiber; for linden extract, 60 min across all fibers; and for mate extract, 45 min. These parameters ensured uniform dye uptake and strong color development. The dyed fabrics exhibited excellent fastness properties to washing, rubbing, and perspiration across all fiber types, consistently above the commonly accepted threshold of 3–4. Although slightly lower values were observed for saliva fastness, the use of non-toxic dyestuffs and mordants—such as potassium sodium tartrate ($KNaC_4H_4O_6$)—ensures no restrictions for their use in textile applications.

Ultimately, this proposed dyeing process converts agro-industrial waste into valuable textile dyes without relying on hazardous materials. By avoiding heavy-metal-containing auxiliaries and producing biodegradable effluents, the method fully aligns with the principles of cleaner production. As such, it should be considered a more sustainable alternative not only in terms of environmental impact, but also in production and textile application practices.

Table 11 Perspiration Fastness properties of dyed samples

Sample	Waste	Solutions	Type of the mordant								
			Alum	$CaCO_3$	Citric acid	Sodium alginate	$AlCl_3$	$KNaC_4H_4O_6$	$MgCl_2$	$FeSO_4$	Na_2CO_3
Wool	Apple	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
	Linden	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
	Mate	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
Cotton	Apple	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
	Linden	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
	Mate	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
Soybean	Apple	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
	Linden	Acidic	5	5	5	5	5	5	5	5	5
		Alkaline	5	5	5	5	5	5	5	5	5
	Mate	Acidic	5	5	4/5	5	5	5	5	4	5
		Alkaline	5	5	4	5	5	5	5	3	5

Table 12 Saliva fastness properties of dyed samples

Type of the mordant	Apple		Linden		Mate	
	Acidic solution	Alkaline solution	Acidic solution	Alkaline solution	Acidic solution	Alkaline solution
Alum	+	+	+	+	+	-
$CaCO_3$	+	-	+	-	+	+
Citric acid	-	-	+	-	+	-
Sodium alginate	+	+	-	-	-	-
$AlCl_3$	+	-	+	-	+	-
$KNaC_4H_4O_6$	+	+	+	+	+	+
$MgCl_2$	+	+	-	-	-	-
$FeSO_4$	+	+	+	-	-	-
Na_2CO_3	+	-	+	-	-	-

Table 13 Characteristics of the wastewater from dyeing wool, cotton and soybean fibers with apple, linten and mate tea waste extract

Waste	Sample	Alum	$CaCO_3$	$KNaC_4H_4O_6$		$AlCl_3$	Sodium alginate	$FeSO_4$	Na_2CO_3
		Al (mg/L)	Ca (mg/L)	Na (mg/L)	K(mg/L)	Al (mg/L)	Na (mg/L)	Fe (mg/L)	Na (mg/L)
Apple	Wool	245.6 ± 2.02	296.7 ± 0.56	146.1 ± 0.46	100 ± 0.42	218.7 ± 4.06	146.1 ± 0.46	986.6 ± 0.15	239 ± 0.72
	Cotton	182.1 ± 1.19	164.3 ± 1.16	37.14 ± 1.16	103.9 ± 1.05	0.491 ± 0.075	37.14 ± 1.16	872.2 ± 0.19	131.8 ± 1.27
	Soybean	92.27 ± 1.5	140 ± 1.05	117.3 ± 10.17	101.6 ± 9.2	141.2 ± 1.17	104.3 ± 1.15	958.8 ± 1.08	91.69 ± 1.56
Linden tea	Wool	373.4 ± 0.89	361.9 ± 1.36	45.6 ± 0.47	199.3 ± 2.64	386.3 ± 0.43	45.6 ± 0.47	179.7 ± 0.99	151 ± 0.35
	Cotton	124 ± 2.4	267.4 ± 0.54	71.01 ± 0.99	204 ± 1.02	215.2 ± 0.39	71.01 ± 0.99	669.3 ± 0.18	64.11 ± 0.64
	Soybean	410.2 ± 0.82	121.3 ± 1.02	201.1 ± 0.51	160.7 ± 0.49	101.9 ± 0 .153	13.1 ± 0.51	170.0 ± 2.93	48.65 ± 1.394
Mate tea	Wool	232.73 ± 0.52	180.2 ± 0.59	146.9 ± 0.68	146.8 ± 0.85	71 ± 5.55	146.9 ± 0.68	303.8 ± 0.74	505.1 ± 0.57
	Cotton	141.02 ± 1.03	343.2 ± 1.311	122 ± 0.99	99 ± 1.09	215.2 ± 0.39	122 ± 0.99	745.6 ± 0.21	116 ± 0.48
	Soybean	11.93 ± 0.153	127,37 ± 2.55	121 ± 1.23	179 ± 1.93	177.9 ± 3.48	121 ± 1.23	537.2 ± 1.03	95.22 ± 2.105

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Author contributions This research article was carried out in collaboration between the following authors. Enfal Kayahan had responsibilities including extraction of natural dyes, dyeing of textile substrates and doing fastness properties, analyzes, supervision, writing-original draft, conceptualization, visualization, writing-review, and editing manuscript. Mustafa Karaboyaci has scientifically guided the work for smooth running of experiments, did writing-review and editing, analyzes. Mehmet Dayık did review and editing. All authors have read and agreed to the published version of the manuscript.

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Data availability The authors reported all data and materials in the main text.

Declarations

Conflict of interest The authors declare that there are no known competing financial interests or personal relationships that could have influenced the work reported in this paper.

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