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Sustainable Mordant-Free Dyeing of Nylon with *Terminalia catappa* Leaf Extract: Process Optimization, Dye–Fiber Interaction, and Fastness Performance

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Abstract

The increasing demand for cleaner coloration of synthetic textiles has renewed interest in plant-based dyes; however, limited dye–fiber affinity and shade durability remain major challenges in nylon dyeing. This study developed a mordant-free dyeing process for nylon fabric using *Terminalia catappa* leaf extract by optimizing extraction pH, dyebath pH, dyeing time, and temperature. Mature *T. catappa* leaves were aqueously extracted under acidic, neutral, and alkaline conditions, followed by systematic dyeing optimization. The dyed fabrics were evaluated using color strength (K/S), CIELAB color coordinates, standard fastness tests, FTIR spectroscopy, and FE-SEM analysis. Alkaline extraction followed by acidic dyeing produced the highest dye uptake. The optimum condition was extraction pH 11, dyebath pH 3, 120 °C, and 60 min, yielding a maximum K/S value of 21.94 with ΔL, Δa, and Δb values of −54.25, 8.03, and 24.92, respectively. FTIR and SEM results supported possible dye–fiber interaction through hydrogen bonding, electrostatic attraction, and surface/inter-filament dye deposition without obvious fiber damage. The optimized fabric showed very good washing, dry rubbing, perspiration, and saliva fastness, good light fastness, excellent ultraviolet protection, and slightly lower wet rubbing fastness. Overall, *T. catappa* leaf extract offers a promising mordant-free route for durable nylon coloration.

Keywords: *Terminalia catappa* leaf extract; natural dyeing; nylon; mordant-free dyeing; process optimization; color fastness; textile coloration

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Academic Editor: Tiancheng Mu

Received: 22 May 2026
Revised: 17 June 2026
Accepted: 30 June 2026
Published: 1 July 2026

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1. Introduction

The art of coloration has been in use since ancient times among human societies. The coloration of textiles with natural dyes has long served both aesthetic and functional purposes, while its gradual development has reflected improvements in dye extraction, dye fixation, and fiber-processing practices [1,2]. Traditionally, textile dyes are divided into two main groups, namely natural dyes and synthetic dyes. The ancient dyes obtained from different plant sources were the only source available for artisans in the early days

of the textile industry [3,4]. Natural dyes enjoyed centuries of widespread use, but their prevalence declined sharply with the advent of synthetic dyes, which offered greater practicality, cost efficiency, color vibrancy, and ease of application [5]. However, these benefits come at a great cost to the environment and humans, because synthetic dyes have significant health and environmental effects. The discharge of these in fully treated, partially treated or untreated form presents a very great risk to ecosystems and human health [6,7]. The general consequences include global warming, the pollution of freshwater supplies, soil degradation and desertification in agriculture, which have consequences for the stability of communities around the world [8]. Increasing attention to the dangers has fostered a renewed interest in the use of organics and environmentally friendly products. The interest in natural dyeing practices, which were once widely practiced, has returned to the consumer and research communities, as consumers increasingly seek environmentally responsible textile coloration. This change has resulted in significant research in laboratory dyeing of natural dyes [8–11]. Biodegradability, non-carcinogenic, low allergenicity and overall environmental health compatibility have made them a promising and environmentally friendly alternative to conventional synthetic dyes [12–14].

Nylon (polyamide) is one of the oldest and most popular synthetic fibers known for its strength, extensibility, resistance to abrasion and ease of use in textile technology [15,16]. It is usually formed by condensation polymerization of diamines and dicarboxylic acids (e.g., nylon 6,6) and contains a high percentage of amide groups which form strong affinity towards anionic dyes, especially towards acid dyes [17–19]. Traditional dyeing of nylon is thus largely carried out using synthetic acid dyes that give high color yield and fastness at controlled temperature and pH [20,21]. Synthetic dye effluents, however, have raised environmental concerns, leading to research on alternative sustainable dyeing methods [22]. Several studies have shown that dyes from plant sources such as catechu, madder, mulberry leaf, and pomegranate peel, as well as waste-derived sources such as Cassia fistula pods, can impart good color strength and fastness to nylon when process parameters such as dye concentration (10–70% owf), dyeing temperature (50–100 °C), pH (3–9), and mordanting technique are optimized [13,14]. The use of mineral (alum and ferrous sulphate) and bio-based mordants plays a crucial role in improving color absorption and fastness [23–25]. For instance, Shokrzadeh et al. [26] examined the use of natural dye extracted from *Prangos ferulacea* for dyeing nylon and employed inorganic mordants such as ferrous sulfate, copper sulfate, zinc sulfate, and potassium dichromate for better dye uptake and fastness properties. They also tested some bio-mordants, including yellow Terminalia and *Carthamus tinctorius*, where they found the type of mordant to have a significant effect on the shades developed and on dyeing properties. There are however certain deficiencies, mainly related to light fastness, shade reproducibility, substrate affinity and scalability that hinder the use of these dyes in industry [27,28]. Moreover, the application of metallic mordants can diminish the eco-friendly benefits of the natural dyeing method through the inclusion of extra chemical burden. Emerging innovations incorporating bioengineered dyes and nanotechnology show promise in addressing these challenges, positioning natural dyes as viable, eco-friendly alternatives for nylon dyeing [28]. Abou-Taleb et al. [29] studied the effect of different concentrations of extract, the pH of the dyebath and the dyebath temperature by one factor at a time and compared the dyeing performance of plasma pretreated nylon fabric with non-plasma pretreated nylon fabric in another study. The research conducted has been aimed at enhancing the color yield, fastness, and affinity of the nylon natural dyestuffs by improving their affinity to the substrate through mordanting, bio-mordanting, optimization of the dyeing process or surface pretreatment. However, many studies on nylon natural dyeing still require mordanting or are mainly concerned with color development, and few studies consider systematic optimization of

the extraction–dyeing process, dye–fiber interaction analysis, surface morphology examination and comprehensive fastness assessment in conjunction with the use of natural dyes without mordants.

Terminalia catappa is a large semi-deciduous tree of the tropics and subtropics, reaching 25–35 m in height, and produces broad coriaceous leaves that undergo a striking color transition from deep green to golden-yellow, then vivid red, and finally rust-brown before abscission [30,31]. The visible color change is not just a cosmetic feature but is part of the process of chlorophyll degradation which takes place concurrently with the process of building up and oxidizing phenolic secondary metabolites, the same chemistry that makes the fallen leaves useful as a source of dyes [32,33]. Allyn et al. [34] identified the presence of flavonoids, quinones, phenolics, triterpenoids, and tannins in ethanolic extracts of brown *T. catappa* leaves, which contained 208.72 mg GAE/g dry extract as total phenolic content and 35.77 mg QE/g dry extract as total flavonoid content, placing this material among the more concentrated polyphenol matrices available from plant leaf biomass. The colors of mature leaves arise from four overlapping compound classes, including hydrolysable tannins such as ellagitannins and gallotannins, free phenolic acids, flavonoids and flavonols, and oxidation products formed from all of these during senescence [35]. These colorants attach to nylon by multiple processes simultaneously. Nylon chains have terminal $–NH_2$ and –COOH groups along a –CO–NH– backbone. The terminal amines are protonated to $–NH_3^+$ in acidic dyebaths (pH 3–6) which can create sites for electrostatic interaction with ionizable polyphenolic- and carboxyl-containing dye species, especially when the protonation state of both nylon and the polyphenolic components of plants is controlled by the dyebath [36]. Moreover, the phenolic hydroxyl groups of tannin, flavonoids, and related polyphenolic structures can form hydrogen bonds with the amide groups of nylon, and the increase in temperature can facilitate dye diffusion into the amorphous regions of the polyamide fiber [37]. The chemical and physical factors indicate a possibility of using the extract from the leaves of *T. catappa* as a coloring agent for nylon, provided that the pH of the extract and dyebath conditions are suitably controlled.

To the best of the authors' knowledge, the application of *T. catappa* leaf extract for mordant-free nylon dyeing has not been systematically reported. Therefore, the present study aims to explore the mordant-free dyeing of nylon fabric using a polyphenol-rich natural colorant extracted from mature *Terminalia catappa* leaves. The work systematically examines the effects of extraction pH, dyebath pH, dyeing time, and temperature on color development, with color strength and CIELAB coordinates used to identify the optimized dyeing condition. The optimized dyed nylon was further characterized using FTIR spectroscopy, FE-SEM analysis, and standard fastness tests to understand the interaction between the dye and fiber, the deposition behavior on the surface and the practical durability. The key significance of this study is that it offers a chemically simplified and mordant-free route to the coloration of nylon, with process optimization being linked to dye–fiber interaction, surface morphology, and end-use durability, in a single integrated assessment. This research presents a reproducible approach for the dyeing of polyamide textiles using only plant-based dyestuffs, without the use of any metallic mordants and bio-mordants or synthetic fixing agents through the utilization of alkaline extraction, acidic dyeing, colorimetric optimization, interaction analysis, and durability assessment.

2. Materials and Methods

2.1. Materials

Mature fallen leaves of *Terminalia catappa* (tropical almond) were collected from trees located on the campus of Northern University Bangladesh, Dhaka, Bangladesh (23.850300° N, 90.424691° E). The leaves were collected during the first week of January, corresponding

to the winter leaf-shedding period in Bangladesh, when mature *T. catappa* leaves fall in relatively high quantity. The collected plant material consisted of mature senescent fallen leaves with predominantly yellowish-brown to reddish/rust-brown coloration, indicating an advanced stage of leaf aging. These details were included to aid in the reproducibility due to the possibility of phytochemical variation in *T. catappa* leaves depending on their maturity and seasonal senescence. The collected leaves were used as the natural dye source because of their characteristic polyphenolic pigmentation. The leaves were collected and then washed thoroughly to remove any impurities on the surface, allowed to dry in air and then ground into fine powder before extraction. The plant material contains a large amount of tannins and other polyphenolic compounds, which are the major coloring constituents of forming natural dyes [38]. A 100% nylon plain-knit fabric of 160 GSM mass per unit area was supplied by Knit Concern Ltd., Narayanganj, Bangladesh, and used as the polyamide substrate. The fabric was ensured to be free from any prior finishing chemicals before use. Acetic acid (CH_3COOH, CAS No. 64-19-7) and soda ash/sodium carbonate (Na_2CO_3, CAS No. 497-19-8) were obtained from Orient Chemicals BD, Nayabazar, Dhaka, Bangladesh, and used for pH adjustment during extraction and dyeing. ECE reference detergent and sodium perborate were supplied by Sabbir Tex Chemicals Ltd., Armanitolla, Dhaka, Bangladesh, for washing fastness evaluation. The artificial saliva solution was prepared using sodium chloride (NaCl, CAS No. 7647-14-5), potassium chloride (KCl, CAS No. 7447-40-7), calcium chloride ($CaCl_2$, CAS No. 10043-52-4), and urea (CH_4N_2O, CAS No. 57-13-6), which were collected from Evergreen Chemicals Ltd., Hatkhola, Dhaka, Bangladesh. Distilled water was used throughout the extraction, dyeing, rinsing, and testing processes to minimize interference from dissolved impurities.

2.2. Methods

2.2.1. Dye Extraction

The leaves of *Terminalia catappa* were washed with distilled water and dried in the air overnight to eliminate moisture in the leaves. The leaves were then ground in a laboratory blender to a fine powder. The amount of leaf powder used per extraction was 5 g and it was dispersed in 100 mL of distilled water to ensure that the solid-to-liquid ratio remained constant.

The effect of acidic, neutral, and alkaline media on the release of colorants was evaluated by aqueous extraction at pH 3, 7, and 11. The pH was adjusted using acetic acid or sodium carbonate solution, as required. All extractions were done at 100 °C under continuous heating of the leaf matrix during 60 min to release tannin-, flavonoid-, and phenolic-based chromophoric compounds into the extraction product. The extraction pH was regarded as a critical variable since it influences the ionization, solubility, and stability of polyphenolic compounds [39]. Alkaline extraction can promote the release of phenolic colorants by partial deprotonation of hydroxyl groups, and acidic extractions might inhibit the solubility of certain chromophoric species [40]. The extraction pH was necessary to determine the available dye and following dye uptake as nylon dyeing relies on the interactions between *T. catappa*-produced polyphenols and the amide/terminal amino groups of polyamide fibers. The dye liquors were extracted and cooled to room temperature and filtered to remove insoluble residues. The filtered extracts were directly applied in nylon dyeing either without metallic mordants or synthetic fixing agents. The extract obtained at pH 11 was chosen to continue dyeing optimization based on the outcomes of the dyed nylon color strength.

2.2.2. Dyeing Process

Dyeing of nylon fabric with *Terminalia catappa* leaf extract was performed using an Ahiba IR dyeing machine. The filtered dye liquor was used directly without applying any metallic mordant or synthetic fixing agent. For each dyeing trial, 5 g of nylon fabric was treated at a material-to-liquor ratio of 1:10 to maintain a constant dye liquor volume and ensure comparability among all samples. The overall workflow for leaf preparation, aqueous extraction, filtration, dye liquor preparation, and representative nylon dyeing outcome is illustrated in Figure 1.

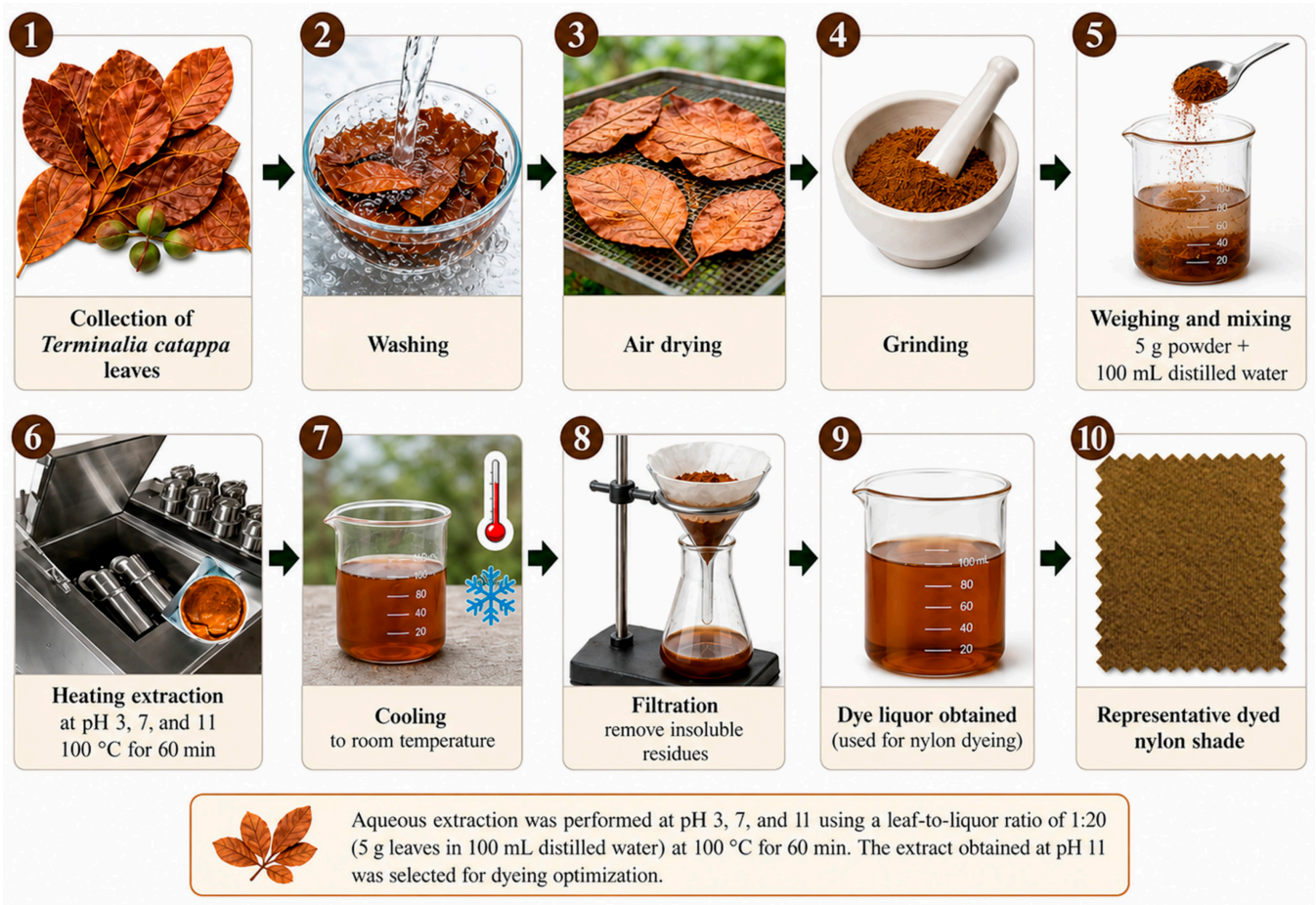

Figure 1. Schematic workflow of mordant-free nylon dyeing with *Terminalia catappa* leaf extraction.

The dyeing process was modeled as a step-wise optimization process to test the influence of the dyebath pH, the dyeing time and temperature on the dye uptake and shade formation of nylon as summarized in Table 1. Initially, the influence of dyebath pH was investigated at pH 3, 4.5, 11, and 12. The pH of the dyebath was adjusted using acetic acid or sodium carbonate solution, as required. These pH values were chosen in order to compare the behavior of nylon dyeing in acidic and alkaline environments. The selected pH values were chosen to represent acidic and alkaline dyeing conditions. The pH 3 is a very acidic pH at which nylon amino end groups are highly protonated and promote attraction to ionizable polyphenolic dye species, while the pH 4.5 is an intermediate acidic pH used in acid dyeing of polyamide. The dyeing behavior at alkaline pH (11 and 12) was tested due to reduced protonation of nylon and potentially increased ionization of the phenolic dye compounds at these pH values. The more strongly acidic pH 2 or lower was avoided as acidity can cause hydrolytic damage to the polyamide chain and result in a lower integrity of the fabric during high-temperature dyeing. Hence, these pHs were chosen to understand the effect of protonating fiber and the ionization of dyes on the dye

uptake falling in a practically relevant processing window. The terminal amino functional groups of nylon can be protonated under acidic conditions to create $–NH_3^+$ sites and can be used to increase the attraction of the polyamide fiber with the anionic or partially ionized polyphenolic dye components [41,42]. Conversely, in alkaline conditions, the decreased availability of protonated amino groups can weaken dye–fiber affinity and inhibit dye exhaustion [43,44].

Table 1. Sequential optimization strategy for dyeing nylon fabric with *Terminalia catappa* leaf extract.

Step	Parameter Optimized	Conditions Investigated	Selected Condition	Purpose/Observation
1	Extraction pH	3, 7, 11	11	Improved release of polyphenolic colorants from the leaf matrix
2	Dyebath pH	3, 4.5, 11, 12	3	Favored protonation of nylon amino groups and enhanced dye–fiber attraction
3	Dyeing time	60 and 90 min	60 min	Longer dyeing did not provide meaningful improvement in color yield
4	Dyeing temperature	80, 100, 120 °C	120 °C	Enhanced dye diffusion, shade depth, and color strength

The initial dyeing experiments were conducted at 100 °C with a duration of 60 min. This condition was chosen as the baseline because high-temperature accelerates the mobility of dye molecules and an increase in the movement of segments in the amorphous regions of nylon, thus, promoting the diffusion of dye into the fiber structure [45]. The effect of dyeing time was studied after determining the most effective dyebath pH, through comparing the results at 60 and 90 min under the preferred conditions. Optimization of temperature was then performed at 80, 100, and 120 °C to establish the importance of thermal energy in the process of dye penetration, color depth, and fixation. This selected temperature range also has some commercial applications in nylon dyeing. The temperature of 80 °C is a low temperature dyeing condition, 100 °C is an almost boiling atmospheric dyeing condition and 120 °C is a high-temperature dyeing under pressure condition, which is widely used in the industry to get better dye diffusion in synthetic fibers. Therefore, these temperatures were chosen to compare the energy saving condition with the conventional and the high temperature condition for dyeing and to check whether the higher temperature brings any improvement in terms of color strength.

The samples were withdrawn from the dyebath followed by thorough rinsing with distilled water to remove any unfixed or loosely deposited dye. A soaping treatment was then performed to remove unfixed and loosely adhered dye from the nylon surface, ensuring that the subsequent colorimetric and fastness evaluations reflected the retained dye fraction rather than weakly attached surface deposits. The samples were finally dried at room temperature. The colored samples were then assessed in terms of color strength and CIELAB color-coordinate analysis. The optimal dyeing condition was determined based on the overall colorimetric performance, and the dyeing conditions which resulted in the highest color strength on nylon fabric were extraction pH 11, dyebath pH 3, 120 °C, and 60 min.

3. Characterization

3.1. Color Properties of Dyed Fabric

3.1.1. Color Yield

The color yield of the dyed nylon samples was evaluated using the Kubelka–Munk theory, which provides a quantitative relationship between the reflectance of a dyed substrate and its color strength [46]. The color strength (K/S) values were determined from the reflectance data obtained using a spectrophotometer equipped with a pulsed xenon lamp, simulating standard daylight illumination (D65). The measurements were taken in an 8° viewing geometry and the wavelength range of 400 to 700 nm to measure the visible reflectance spectrum of samples.

To achieve accurate and reproducible measurements, each dyed fabric was folded to obtain a four-ply structure, thereby eliminating background interference and ensuring optical opacity. The values of reflectance (R) were measured, and the corresponding K/S values were calculated automatically by the instrument using Equation (1) [47].

$$K/S = \ (1 - R)^2/2R, \quad (1)$$

where R is the reflectance of the dyed fabric at a particular wavelength, K is the absorption coefficient, and S is the scattering coefficient. The strength of the color of the dyed sample is directly proportional to K/S value; the higher the value, the higher the rate of uptake of the dye and the intensity of the color [48]. The procedure provides an excellent comparison of the performance of dyeing of the nylon samples prepared under varied extraction pH, dyebath pH, dyeing time and temperature. This study has used K/S analysis to give a solid ground for the study of the effectiveness of process optimization in the natural dye uptake of nylon fabric.

3.1.2. Color Coordinates

The color properties of the samples of dyed nylon were assessed using a spectrophotometer (Datacolor 650™, Datacolor Inc., Lawrenceville, NJ, USA) with appropriate calibration against a standard reference. The measurements were performed by placing the dyed samples in comparison with corresponding undyed nylon fabric to ensure proper evaluation of color development. The samples were folded accordingly to form a four-layered structure before measurements to minimize the background interference and enhance optical signal transmission. The color values of samples were measured from multiple representative areas of the dyed fabric by moving and rotating the sample, and the three closest values for each sample were averaged for reporting K/S and CIELAB coordinates. The procedure was followed to reduce the effects of local non-uniformity, fabric direction, and knitted surface texture on the instrumental color data. Thus, the reported colorimetric readings were an average of the response of each dyed sample and not a single point of the visual selection.

The spectrophotometric analysis offers reflectance spectra and CIELAB color coordinates (L, a, b) which are commonly used in the quantitative evaluation of colors in textile applications. The CIELAB color space describes color in three dimensions: lightness (L*) and chromaticity coordinates (a* and b*). The L* value represents the lightness of the sample on a scale from 0 (black) to 100 (white), where lower L* values indicate darker shades and higher values correspond to lighter shades. The a* coordinate represents the red–green axis, with positive values indicating redness and negative values indicating greenness. Similarly, the b* coordinate is a yellow–blue axis with positive values denoting yellow and negative values denoting blue [49].

These parameters provide a complete description of the color properties of dyed fabrics and provide the opportunity to draw accurate comparisons between the samples of nylon dyed under various processing conditions. The application of the CIELAB values in the study allowed assessing variations in shade depth, hue, chromatic intensity, and total color difference in the nylon dyeing process under the investigated extraction and dyeing conditions.

3.2. Color Fastness

The performance of the dyed nylon samples in terms of durability and end-use performance was evaluated based on the standard color fastness tests, including washing, rubbing, perspiration, saliva, and light exposure. Prior to testing, all specimens were conditioned for 4–6 h under standard atmospheric conditions (20 ± 2 °C and 65 ± 2% relative humidity) to ensure measurement consistency. The fastness ratings have been measured using standardized grayscale (1–5) and blue wool scale (1–8), where higher grades are associated with better resistance to change and staining of color.

The fastness of the color to washing was tested in accordance with ISO 105-C06 (C2S) [50]. The composite specimens (10 × 4 cm) were prepared by stitching the dyed sample onto multifiber adjacent fabric and treating with a detergent solution containing 4 g/L standard detergent (without optical brightener) at a liquor ratio of 50:1. This washing was performed at a temperature of 40 °C and a period of 30 min under controlled agitation. The extent of color change and staining was determined by grayscale ratings after rinsing and drying. The significance of this test is that it shows what the dye fiber system can withstand in the domestic laundering process and therefore it reflects some practical service durability.

Color fastness to rubbing (crocking) was determined according to ISO 105-X12 under both dry and wet conditions [51]. Test specimens (approximately 14 × 5 cm) were subjected to controlled friction using a standard crockmeter with a load of about 9 N for 10 cycles. Then ratings were assessed using a standard white rubbing cloth. For wet rubbing, the rubbing cloth was wetted to a moisture content of approximately 95–100%. The degree of color transfer was evaluated using the grayscale for staining. This test is important because it reflects the tendency of surface-bound dye to transfer during wear, handling, and abrasion.

Color fastness to perspiration was assessed according to ISO 105-E04 using both acidic and alkaline perspiration solutions [52]. Composite specimens of approximately 10 × 4 cm were stitched with adjacent multifiber fabric, immersed for 30 min, adjusted to about 100% wet pick-up, and then subjected to a pressure of about 12.5 kPa. The specimens were then incubated at 37 ± 2 °C for 4 h. After drying, the degree of color change and staining was evaluated using grayscale ratings. This test is important as it simulates the effect of human sweat on dyed textiles under real wearing conditions.

Color fastness to saliva was assessed according to DIN 53160-1 [53], using an artificial saliva solution made of sodium chloride (NaCl), potassium chloride (KCl), calcium chloride ($CaCl_2$), and urea with a pH of about 6.8–7.0. Composite samples (10 × 4 cm) were soaked in the saliva solution for 30 min, and then adjusted to about 100% wet pick-up. The specimens were then positioned between plates under a pressure of approximately 12.5 kPa and held at 37 ± 2 °C for 2–4 h. The specimens were then dried and grayscale values were used to determine the extent of staining and discolorations that may have occurred during the test. This is an important test in babywear and infant products, as the goal of eco-friendly natural dyes is to produce dyeing to be used in highly sensitive applications where mouth contact is possible. Coloration using natural dyes could be better for sensitive uses, as it is organic and free from synthetic complex dyes. How-

ever, its biological safety is not confirmed, and further toxicity, migration, and regulatory tests are needed before claiming its use in babywear or products with prolonged mucous-membrane contact.

Light fastness was evaluated following ISO 105-B02 using artificial light from a xenon arc lamp and rating the change in color using the blue wool scale from 1 to 8 [54]. The dyed samples were irradiated under controlled conditions using a xenon arc lamp and a section of each sample was protected from light to act as a reference standard. The higher the value, the greater the resistance to photodegradation. This test is important because it determines the resistance of the dyed sample to photodegradation in real-life storage and use, which indicates the retention of color in the presence of light.

Generally, the selected fastness protocol provided a complete picture of the reliability of the natural dye on nylon fabric. The overall tests establish whether the dyeing system is able to achieve the necessary durability of the apparel, the textile in direct contact with the skin and the infant-oriented materials.

Alongside conventional fastness evaluation, the dyeing approach was considered in relation to the Natural Organic Dye Standard (NODS), which provides a framework for naturally dyed textiles based on dye-source authenticity, permitted auxiliaries, mordanting practices, fastness performance, and restriction of hazardous substances [55]. The present system is consistent with the main principles of NODS because coloration was achieved using only *Terminalia catappa* leaf extract, with acetic acid and sodium carbonate used solely for pH adjustment. No metallic mordants, synthetic dyes, fixing agents, binders, or additional harmful auxiliaries were applied, reducing the chemical burden commonly associated with mordant-assisted natural dyeing. The washing, rubbing, perspiration, saliva, and light fastness tests therefore provide a durability-based evaluation of this mordant-free dyeing route within the context of natural dye standardization. The good to excellent fastness ratings indicate that practical color durability can be achieved without environmentally or health-concerning mordants. However, full NODS compliance would still require dedicated screening for restricted substances, heavy metals, and synthetic dyestuff contamination, which should be addressed in future work.

3.3. FTIR Analysis Method

Fourier transform infrared (FTIR) spectroscopy was used to identify functional groups in both undyed nylon and the optimized nylon sample dyed with *Terminalia catappa* leaf extract and to assess possible dye–fiber interactions. Since both undyed and dyed nylon samples were analyzed, spectral interpretation was based on the characteristic absorption bands of polyamide substrate with those of the optimized dyed sample, allowing changes related to retained plant-derived dye constituents and possible dye–fiber interactions to be evaluated more reliably. The analysis was performed using an ALPHA FT-IR spectrometer (Bruker Optics, Billerica, MA, USA) in ATR mode over 4000–400 cm^{-1}, with 20 scans at a resolution of 4 cm^{-1}. The main spectral regions taken into consideration were the N–H/O–H stretching, aliphatic C–H stretching, Amide I, Amide II, and fingerprint-region bands.

3.4. SEM Analysis Method

The morphological structure of dyed nylon fabric was characterized by means of a Schottky Field Emission Scanning Electron Microscope (FESEM; JSM-7610F, JEOL Ltd., Akishima, Tokyo, Japan). The samples were coated with a thin layer of gold to improve electrical conductivity and to achieve high-resolution images.

Surface morphologies were examined at various magnifications (200×, 500×, 1000× and 2000×) to assess surface characteristics and structural changes resulting from dye

deposition and dye–fiber interaction. Special emphasis was placed on the surface texture of the fibers, coating coverage, and dye particles. The SEM images were also examined to evaluate fiber-dye interaction, surface coverage and adhesion of dye molecules to the nylon surface. This interpretation gives an understanding of the morphological changes to the surface of the fiber and complements the colorimetric data on dye uptake.

3.5. Ultraviolet Protection Analysis

The ultraviolet protection performance of the undyed and optimized dyed nylon fabrics was evaluated in terms of ultraviolet protection factor (UPF) using a UV protection tester (ChiuVention, Dongguan, China). The test was performed according to AATCC TM183-2020 [56], which determines the transmission or blocking of erythemally weighted ultraviolet radiation through textile fabrics. Fabric samples were conditioned at standard atmospheric conditions before being tested to reduce the effect of moisture level variation on the UV transmittance. The UPF value was determined by measuring the spectral transmittance of the ultraviolet radiation that passes through the fabric in both the UVA and UVB regions using the formula in Equation (2).

$$UPF = \frac{\sum_{290}^{400} E_\lambda S_\lambda \Delta_\lambda}{\sum_{290}^{400} E_\lambda S_\lambda T_\lambda \Delta_\lambda}, \quad (2)$$

where E_λ is the relative erythemal spectral effectiveness, S_λ is the solar spectral irradiance, T_λ is the measured spectral transmittance of the fabric at wavelength λ, and $\Delta\lambda$ is the wavelength interval. This analysis was carried out to assess whether coloration with *Terminalia catappa* leaf extract improved the UV shielding ability of nylon fabric in addition to providing coloration and fastness performance.

4. Results and Discussion

4.1. Best Extraction Condition

The extraction pH had a prominent influence on the color strength of dyed nylon. Under the same dyeing conditions of pH 3, 100 °C, and 60 min, the K/S value increased from 16.19 for extract prepared at pH 7 to 17.19 for extract prepared at pH 3, and reached 21.53 for extract prepared at pH 11. This corresponds to an approximately 33% higher color strength compared with neutral extraction, confirming that alkaline extraction was more effective in releasing color-active compounds from *T. catappa* leaves. The improvement at pH 11 can be attributed to the enhanced solubilization and partial ionization of tannin-, flavonoid-, and phenolic-based chromophores, which increases their availability in the dye liquor [57]. During subsequent acidic dyeing, these polyphenolic compounds can interact with nylon through electrostatic attraction between anionic dye species and protonated $–NH_3^+$ sites, together with hydrogen bonding involving the amide groups of nylon [58]. The highly negative ΔL value of −54.28 further confirms the formation of a much darker shade. Therefore, extraction pH 11 was selected as the optimum extraction condition for further dyeing optimization.

4.2. Colorimetric Properties of Dyed Fabrics

The colorimetric performance of nylon fabric dyed with *Terminalia catappa* leaf extract was evaluated by varying extraction pH, dyebath pH, dyeing time, and temperature. The strength of colors was expressed as K/S, and the transformation of lightness and chromatic direction was interpreted as ΔL, Δa, and Δb. The dyeing behavior of nylon is highly dependent on the protonation state of the fiber used, and the presence of polyphenolic dye components in the extract. The general findings are summarized in Table 2 and discussed in a systematic manner in the following subsections.

Table 2. Color strength (K/S) and CIELAB colorimetric properties (ΔL, Δa, and Δb) of nylon fabric dyed with *Terminalia catappa* leaf extract under different extraction and dyeing conditions.

Extraction pH	Dyeing Parameters			K/S	ΔL	Δa	Δb	Sample
	pH	Temp. (°C)	Time (min)					
7	3	100	60	16.19	−38.53	5.1	27	
7	4.5	100	60	14.26	−36.82	4.11	27.15	
7	11	100	60	1.66	−28.95	5.36	21.68	
7	12	100	60	2.09	−29.2	5.4	22.91	
3	3	100	60	17.19	−41.28	6.86	24.4	
11	3	100	60	21.53	−54.28	7.24	21.85	
11	11	100	60	0.49	−11.79	2.5	14.52	
11	3	100	90	20.94	−51.75	6.44	21.43	
11	3	80	60	11.67	−29.11	5.72	30.22	
11	3	120	60	21.94	−54.25	8.03	24.92	

Table 2 shows that the dyeing performance of nylon was controlled by both chemical and thermal factors. The increase in K/S values was observed when alkaline extraction was coupled with acidic dyeing which indicated that the availability of colorants in the dye liquor and protonation of nylon both contributed to enhancing dye–fiber interaction.

The deepest shade with the highest K/S value of 21.94 was obtained at extraction pH 11, dyebath pH 3, 120 °C, and 60 min. Table 2 shows only the color of sample images visually; all the samples were prepared from the same lot of nylon fabric, and photographed under the same lighting, distance, and background conditions. Minor texture differences may result from knitted-loop orientation, creasing, fabric relaxation after dyeing, and image resizing, whereas the reported K/S and CIELAB values were obtained separately from averaged instrumental measurements.

4.3. Effect of pH, Time and Temperature

After identifying extraction pH 11 as the most effective extraction condition, dyebath pH, dyeing time, and temperature were further investigated to clarify their individual contributions to nylon dyeability. Dyebath pH mainly controls the protonation state of nylon and the ionic interaction between fiber and dye molecules. Dyeing time determines whether sufficient contact is provided for adsorption and diffusion, while temperature governs the mobility of dye molecules and the accessibility of the amorphous regions of the polyamide structure. These variables are discussed separately to avoid overlapping interpretation.

4.3.1. Effect of Dyebath pH

Dyebath pH showed a dominant influence on the color development of nylon dyed with *T. catappa* leaf extract. When the extract prepared at pH 7 was applied, the K/S value was high under acidic dyeing conditions, reaching 16.19 at dyebath pH 3 and 14.26 at pH 4.5. In contrast, a sharp reduction was observed under alkaline dyeing conditions, where the K/S values decreased to 1.66 at pH 11 and 2.09 at pH 12. This indicates that acidic dyeing produced substantially greater color strength than alkaline dyeing. The CIELAB results also support this observation, as the acidic dyed samples showed more negative ΔL values, confirming darker shade formation. For example, ΔL was −38.53 at pH 3 and −36.82 at pH 4.5, whereas it decreased in depth to −28.95 and −29.20 at pH 11 and 12, respectively.

A similar behavior was observed when extract prepared at pH 11 was used. Dyeing at pH 3 produced a high K/S value of 21.53, with a highly negative ΔL value of −54.28, indicating the deepest shade among the samples dyed at 100 °C for 60 min. However, when the same extract was applied at dyebath pH 11, the K/S value dropped drastically to 0.49, with ΔL reduced to −11.79. This poor color strength confirmed that alkaline dyeing was unsuitable for nylon under the present system. Therefore, the combination of extraction pH 3 with dyebath pH 11 was not further investigated, because the results obtained at alkaline dyebath pH already demonstrated weak dye uptake and unsatisfactory shade development compared with acidic dyeing. This outcome is in line with the known dyeing properties of polyamide, which usually require an acidic dye bath to absorb the dyes effectively. The alkaline dyebath was neither a recommended dyeing route nor was it used for the main purpose of studying the influence of pH. Under alkaline conditions, nylon may undergo hydrolytic degradation due to saponification [59], whereas anthocyanin- or polyphenol-based chromophores in the extract may also undergo structural transformation, degradation, or shade change [60]. High-temperature alkalinity can also impact the polyamide substrate, as well as alter the pH-sensitive natural dye chromophores. Hence, the extremely low K/S value corresponding to the alkaline pH further indicates that alkaline dyeing is unsuitable for this dyeing system of nylon/*T. catappa*.

The superior performance at acidic pH can be explained by the chemical structure of nylon. Under acidic conditions, the terminal amino groups of nylon are protonated to $–NH_3^+$, creating positively charged sites that can attract anionic or partially ionized

polyphenolic compounds from *T. catappa* extract [58]. This electrostatic attraction enhances dye adsorption and fixation. In addition, hydrogen bonding between phenolic –OH groups of the dye molecules and the amide groups of nylon may further stabilize the dye–fiber interaction [37]. Conversely, under alkaline conditions, the protonation of nylon amino groups is suppressed, resulting in fewer cationic binding sites and weaker dye–fiber affinity [44]. This explains the low K/S values and less negative ΔL values observed at pH 11 and 12. Therefore, dyebath pH 3 was selected as the optimum dyeing pH for achieving high color strength and deeper shade formation on nylon fabric.

4.3.2. Effect of Dyeing Time

The impact of dyeing time was assessed by comparing the dyeing time of 60 and 90 min at extraction pH 11, dyebath pH 3 and 100 °C. The K/S value slightly decreased from 21.53 at 60 min to 20.94 at 90 min, showing that prolonging the dyeing duration did not improve color strength. This indicates that the dye uptake became close to equilibrium in 60 min under acidic conditions. Once the available binding sites on nylon were saturated, additional dyeing time did not contribute to further fixation [61].

The slight decrease in K/S after 90 min might be due to desorption occurring at this time, to the redistribution of loosely bound dye in the sample or to the instability of some polyphenolic components during extended heating [62,63]. The processing aspect is more favorable with 60 min as it gives a higher color strength and less energy use and time than that of longer dwell times. Therefore, a dyeing time of 60 min was chosen as the optimum because of this.

4.3.3. Effect of Dyeing Temperature

Dyeing temperature strongly influenced color strength, shade depth, and chromatic development of nylon dyed with *T. catappa* leaf extract. The temperature increment from 80 to 100 °C at extraction pH 11 and dyebath pH 3 for 60 min resulted in a significant enhancement of dye uptake, with the K/S value increasing from 11.67 to 21.53. The CIELAB values also showed an increase with the increase in temperature, from −29.11 at 80 °C to −54.28 at 100 °C, resulting in a very dark color. The Δa value also rose from 5.72 to 7.24, reflecting an increase in the reddish component, and the Δb value also dropped from 30.22 to 21.85, reflecting the shift towards a deeper brownish tone.

The further increase to 120 °C resulted in the maximum K/S value of 21.94, with ΔL remaining highly negative at −54.25. The optimized sample showed a further increase in Δa value of 8.03 and Δb value of 24.92 which revealed that the optimized sample maintained its deep shade with stronger red–yellow chromatic contribution. The significant improvement between 80 and 100 °C indicates that higher thermal and kinetic energy increased dye molecular mobility and promoted internal structural mobility in the amorphous regions of nylon, thereby facilitating diffusion of polyphenolic dye molecules into accessible regions of the polyamide substrate rather than through fiber swelling, which is generally limited in PA fibers [64]. The smaller improvement between 100 and 120 °C, however, suggests near-saturation of dye uptake at 100 °C, and that 120 °C only yielded incremental improvement in dye uptake and fixation. The color strength at 120 °C is the highest, but the improvement from 100 to 120 °C is relatively small compared with the improvement from 80 to 100 °C, and thus 120 °C can be regarded as the optimum temperature for maximum color strength, while 100 °C might be a more energy-conscious option when slightly lower color strength is acceptable. This is significant in the industrial application because the sustainability value of the present process is primarily associated with the removal of the metallic mordants, bio-mordants, and synthetic fixing agents, although the energy requirement of the high-temperature dyeing should not be neglected during scale-up.

This behavior is in line with the thermal responses of polyamide fibers. The nylon chains have higher segmental mobility at elevated temperatures, which leads to the creation of more free volume to facilitate the diffusion of polyphenolic dye molecules to internal binding sites [65]. Hence, the optimum dyeing temperature of 120 °C was chosen as it provides the maximum color strength and also the color remained deep with strong chromatic characteristics under the chosen conditions of extraction pH 11, dyebath pH 3 and 60 min.

4.4. FTIR Spectral Interpretation

FTIR analysis was performed on the undyed nylon and optimized nylon sample dyed with *Terminalia catappa* leaf extract to identify the functional groups present after dyeing and to support the possible dye–fiber interaction mechanism. The inclusion of the undyed nylon spectrum allows the dye-induced spectral changes to be interpreted more reliably, as the characteristic absorption bands of the nylon substrate can be directly compared with those of the dyed sample. The FTIR spectrum is presented in Figure 2.

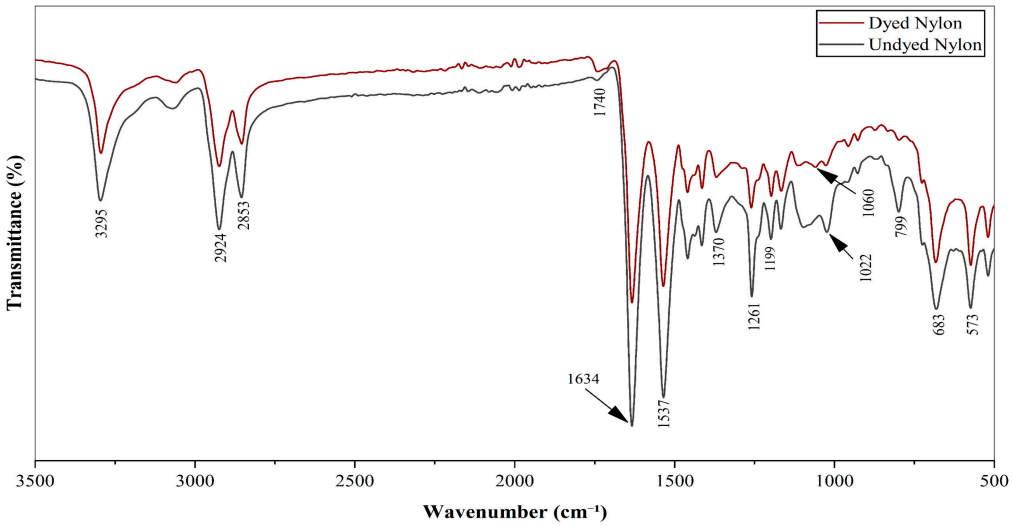

Figure 2. FTIR spectra of undyed and *T. catappa*-dyed nylon, showing polyamide bands and dye–fiber interaction features.

The absorption bands of polyamide were observed from both undyed and dyed nylons. The presence of a wide absorption band at approximately 3294 cm^{-1} in both spectra was attributable to the N–H stretching vibration of the amide groups present in nylon, and possibly some overlapping contribution from the O–H stretching vibration of hydroxyl containing polyphenolic compounds which remain in the *T. catappa* extract after dyeing [32,66,67]. The broadness of this band is particularly important, as free N–H or O–H groups generally show sharper absorption, whereas hydrogen-bonded groups usually appear broader [32]. In the dyed nylon spectrum, this region remained clearly visible without the appearance of a separate new band, indicating that dyeing did not chemically disrupt the polyamide backbone. The slight intensity variation in this region may be associated with changes in hydrogen-bonding environment caused by the interaction between phenolic –OH groups of the dye constituents and the amide N–H/C=O groups of nylon. The spectrum of dyed nylon is similar to that of undyed nylon, with the absorption in this region being slightly more intense indicating the presence of hydroxyl-containing dye components and possible hydrogen bonding between the phenolic –OH groups of dye molecules and the amide C=O/N–H groups of nylon. The bands near 2924 and 2853–2854 cm^{-1} correspond to asymmetric and symmetric C–H stretching vibrations of

aliphatic methylene groups in the nylon backbone [68,69]. These bands were retained after dyeing, indicating that the aliphatic structure of nylon was not degraded during the optimized acidic high-temperature dyeing process.

The strong absorption bands at approximately 1634 and 1537 cm^{-1} are characteristic of the polyamide structure. The band at 1634 cm^{-1} is attributed to the Amide I band, which is primarily attributed to the C=O stretching vibration, and the band at 1537 cm^{-1} to the Amide II band, which is mainly attributed to the N–H bending coupled with the C–N stretching vibration [70,71]. These two bands were observed in both undyed nylon and dyed nylon, which again resulted in the maintenance of the framework of the nylon amide. The relative intensities and profiles of these bands, however, were altered following the dyeing process, indicating a change in the local amide environment but not a complete change in its chemical structure. This may be considered as evidence of possible association between dyes and the fibers through the involvement of the nylon amide groups with the phenol-OH group in the constituents that are present in *T. catappa* extract, such as tannin, flavonoids, and phenolic acids [32,37,67]. Another weak shoulder/band around 1740 cm^{-1} was found in the dyed nylon spectrum, possibly related to carbonyl-containing groups such as in carboxylic acid, ester, galloyl or hydrolysable tannin structures from the plant extract. The present band, however, is weak and partially overlaps with the bands in the nylon spectral region, and therefore should be considered as supporting evidence rather than as proof of a new covalent bond.

The presence of co-existing plant-derived dye constituents and nylon functional groups is further supported by several absorption bands in the fingerprint region. The bands located at 1462, 1415, and 1370 cm^{-1} can be correlated with CH_2 bending, C–N stretching, and possible aromatic skeletal vibrations [68,69]. The absorptions at 1261, 1199, and 1167 cm^{-1} could be due to the C–N stretching of nylon and the overlapping of C–O stretching of phenolic compounds [72,73]. The band around 1060 cm^{-1} is especially significant since the vibration of the C–O and C–O–C bonds of hydroxylated aromatic structures such as tannins, flavonoids and other polyphenolic compounds could be present [72,73]. The intensity variation in this region is not observed in the case of dyed nylon in the present spectrum. It is therefore worth considering this area as a sign of overlapping nylon and dye vibrations, but also as evidence of the change in the surface chemistry after dyeing. Similarly, the band near 799 cm^{-1} and the lower wavenumber bands near 683 and 573 cm^{-1} could be attributed to out-of-plane bending, skeletal/ring deformation, low-frequency bands of the polyamide, and possibly aromatic residues of the natural dye [74]. The higher transmittance observed for the dyed nylon at some fingerprint bands should not be considered as absence of dye uptake; rather, it reflects changes in relative absorbance caused by dye deposition, surface coverage, hydrogen-bonding modification, and ATR contact differences between the dyed and undyed fabrics.

The overall comparative FTIR spectra validate that the optimized dyeing method does not significantly damage the nylon structure, since the spectra of main polyamide bands were clearly visible in both dyed and undyed samples. Simultaneously, the lower transmittance values for the dyed nylons in the N–H/O–H stretching region, the Amide I, Amide II and C–O/C–N fingerprint bands indicate a successful binding of the polyphenolic compounds from *T. catappa* to the nylon fabric. At the same time, the changes in band intensity/profile, the weak carbonyl contribution near 1740 cm^{-1}, and the modified fingerprint-region features indicate the presence of retained *T. catappa* dye constituents on or within the nylon substrate. The results show that the dye fixation was primarily through non-covalent interactions, particularly hydrogen bonding between the –OH groups of phenolic components of the dye and the amide C=O/N–H groups of nylon [32,37,67]. In addition, under the optimized acidic dyeing condition, protonated terminal amino groups

of nylon may contribute to electrostatic attraction with ionizable phenolic or carboxyl-containing dye species [43]. Therefore, the FTIR results provide molecular-level support for the successful attachment of *T. catappa* dye constituents onto nylon fabric and support the proposed dye–fiber interaction mechanism, indicating that hydrogen bonding and electrostatic interactions between the functional groups of the dye molecules and the amide linkages of nylon are the major contributors to dye fixation.

4.5. SEM Morphological Interpretation

The surface morphology of nylon fabric dyed with *Terminalia catappa* leaf extract in the optimized condition was studied by SEM analysis. The micrographs were captured at 200×, 500×, 1000× and 2000× magnifications to investigate the structure of the yarn, the integrity of the filaments, surface deposits and the potential interactions between the dye and the fiber. The SEM images are presented in Figure 3.

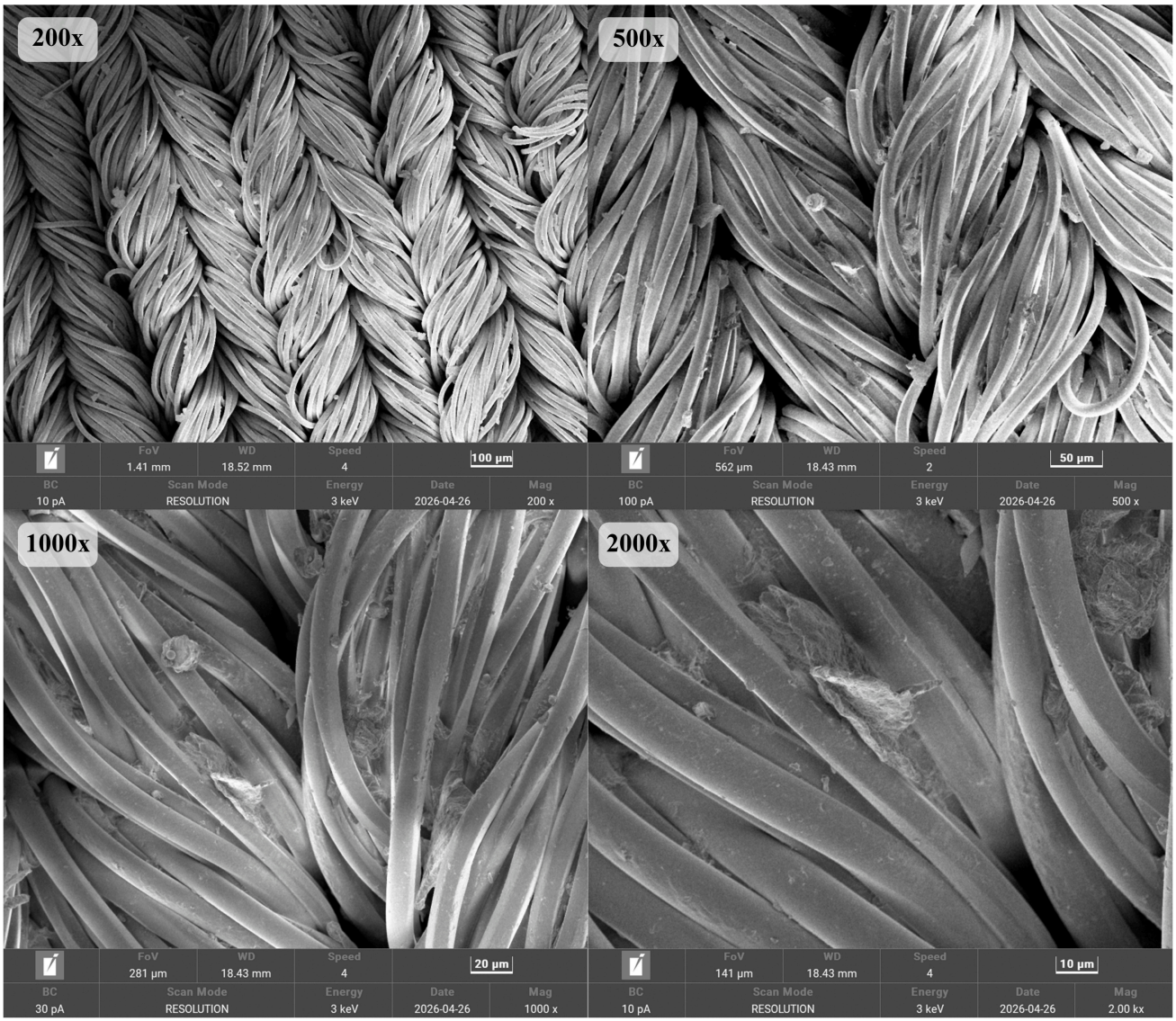

Figure 3. SEM micrographs of nylon fabric dyed with *Terminalia catappa* leaf extract under the optimized dyeing conditions at different magnifications.

The structure of knitting and arrangement of multifilament were clearly visible at the 200× magnification and showed no evidence of filament breakage, melting or significant change in structural shape. This indicates that the optimized acidic dyeing process at

elevated temperature did not cause noticeable damage to the nylon substrate. The deposits observed along the filaments and within inter-filament spaces suggest surface coverage and possible retention of extract-derived materials, which may include dye constituents, fine sediments, or residual particles from the natural extract.

The individual filaments captured at 500× and 1000× magnifications exhibited a surface with a low degree of surface roughness and some deposits at discrete locations. These features are possibly related to the adhesion and uptake of polyphenolic constituents on the nylon surface that are produced by *T. catappa* [75]. The presence of deposited material along the filaments and in the inter-filament spaces indicates that the dye was not only confined to the external surface of the yarn but could also penetrate the yarn structure during the dyeing process. This is significant because it can result in greater apparent color depth by increasing the amount of surface dyed and minimizing the amount of undyed surface area in the yarn bundle [75].

The filament surface at 2000× magnification revealed a thin and unevenly coated surface with small particulate or aggregated features. This morphology could be due to retained extract-derived residues, including tannin-, flavonoid-, and phenolic-based colorant components; however, the possible contribution of non-colorant sediments or dust-like particles from the natural extract cannot be excluded [37]. Importantly, no apparent cracking, peeling or loss of surface was seen, making the dye-related deposits appear to have good adhesion to the nylon surface. The adhesion could be a hydrogen bond between –OH groups of phenolics and amide groups of nylon and electrostatic interactions between ionizable dye components and protonated amino groups during acid dyeing conditions [76]. However, it is important to note that this high K/S value may not arise solely from dye diffusion into the internal polymer matrix. The FE-SEM observations indicate that deposition of dye components in the surface and inter-filament spaces might also play a role in the apparent color strength, which is due to the increased optical absorbing effect of the fiber/yarn surface. The combined effect of the rinsing and soaping pre-treatment and the characteristic fastness performance, however, suggested that a part of the dye retained was not just loosely deposited on the nylon substrate, but was also associated with it through dye–fiber interactions.

Overall, the SEM observations suggest that the nylon surface was modified with *T. catappa* dyeing, but the original filament structure was retained. The results obtained from the surface coverage, inter-filament deposition and lack of significant morphological damage indicate the retention and possible association of extract-derived dye constituents/residues on nylon. The results of these morphological observations correspond to the interaction mechanism proposed by FTIR and the improved color strength obtained during the optimized dyeing parameters.

4.6. Color Fastness Results

The color fastness performance was evaluated to check the practical durability of the nylon fabric dyed under the optimized dyeing condition using the extract of the *Terminalia catappa* leaf. The assessment consisted of washing, rubbing, acidic and alkaline perspiration, saliva, and light fastness, which reflected the major conditions that would be encountered when laundering, wearing, skin contact, and light exposure. The ratings were interpreted by the standard gray scale for color change and staining, and the blue wool scale was used for rating light fastness. The summary of the results is presented in Table 3.

The dyed nylon fabric showed very good overall fastness performance, with most ratings ranging from 4 to 5. Washing fastness exhibited a color change rating of 4–5, indicating very good to excellent resistance to laundering. Interestingly, the washed sample appeared darker than the original dyed sample. This behavior may be associated with

the pH-sensitive nature of plant-derived polyphenolic dyes. Natural dyes containing phenolic, flavonoid, and tannin-based chromophores often show halochromic behavior, where changes in pH alter the protonated/deprotonated state of the dye molecules and consequently modify their light absorption and visible shade [77]. Natural dyes are widely reported to be pH-sensitive because protonation–deprotonation changes affect their absorption behavior and color appearance [11].

Table 3. Color fastness properties of nylon fabric dyed with *Terminalia catappa* leaf extract under the optimized dyeing conditions.

Fastness Property	Test Condition	Rating	Assessment
Washing Fastness	Color Change	4–5 *	Very good to excellent
	Staining (Acetate)	5	Excellent
	Staining (Cotton)	4–5	Very good to excellent
	Staining (Nylon)	5	Excellent
	Staining (Polyester)	5	Excellent
	Staining (Acrylic)	4	Very good
	Staining (Wool)	4	Very good
Rubbing Fastness	Dry	4–5	Very good to excellent
	Wet	3–4	Fair to very good
Perspiration Fastness (Acidic)	Color Change	4–5	Very good to excellent
	Staining (Acetate)	4–5	Very good to excellent
	Staining (Cotton)	4–5	Very good to excellent
	Staining (Nylon)	3–4	Fair to very good
	Staining (Polyester)	4–5	Very good to excellent
	Staining (Acrylic)	4	Very good
	Staining (Wool)	3–4	Fair to very good
Perspiration Fastness (Alkaline)	Color Change	4–5	Very good to excellent
	Staining (Acetate)	5	Excellent
	Staining (Cotton)	4	Very good
	Staining (Nylon)	4–5	Very good to excellent
	Staining (Polyester)	5	Excellent
	Staining (Acrylic)	5	Excellent
	Staining (Wool)	3–4	Fair to very good
Saliva Fastness	Color Change	4–5	Very good to excellent
	Staining (Acetate)	5	Excellent
	Staining (Cotton)	4–5	Very good to excellent
	Staining (Nylon)	4	Very good
	Staining (Polyester)	5	Excellent
	Staining (Acrylic)	5	Excellent
	Staining (Wool)	3–4	Fair to very good
Light Fastness	Xenon arc lamp	5	Good

* the processed sample shade was found to be darker than the original.

In the present washing test, the detergent system containing sodium perborate could create a mildly alkaline and oxidative environment. Borate- and carbonate-containing detergent systems are commonly associated with alkaline washing conditions, while sodium perborate can act as an oxidizing bleaching component [11]. Therefore, the post-wash darkening may be explained more plausibly by a halochromic effect, accompanied by possible bathochromic and/or hyperchromic changes in the natural dye chromophores under alkaline washing conditions. A bathochromic shift would move absorption toward longer wavelengths, while a hyperchromic effect would increase absorption intensity, both of which may contribute to a visually deeper shade. In addition, removal of weakly adsorbed surface impurities or loosely held dye fractions during washing may reduce

surface scattering and make the remaining fixed shade appear deeper [78]. Thus, the darker appearance after washing is not necessarily evidence of dye instability; rather, it may reflect pH-induced chromophore transformation and improved visual clarity of the retained dye layer.

Staining results during washing were excellent for acetate, nylon, and polyester, with ratings of 5, while cotton showed 4–5 and acrylic and wool showed 4. The conclusions from these results are the limited migration of dyes to adjacent fibers during laundering. This slightly reduced staining resistance on wool might be due to the presence of amino, carboxyl and polar functional groups in the protein structure which can interact with the migrated polyphenolic dye molecules through hydrogen bonding and ionic bonding [79]. The staining to cotton was also very good, indicating a minimal amount of dye was released from the nylon substrate during washing.

Rubbing fastness showed a dry rubbing rating of 4–5 and a wet rubbing rating of 3–4. A higher dry rubbing resistance indicates that the dye was fairly well retained by the nylon surface under dry rubbing. Natural dye systems can be expected to have a lower wet rubbing rating due to swelling of the fiber surface, causing more fiber rubbing cloth contact, and to mobilize a small percentage of the weakly bound dye molecules [80]. However, a wet rubbing rating of 3–4 is fair to very good and implies that the majority of the dye molecules were still bound to the nylon substrate. The good washing fastness can be attributed to the combined effect of dye retention within nylon and the pH-sensitive behavior of the natural dye. When dyeing at 120 °C, some of the dye may enter the amorphous regions of nylon and become trapped in the polymer as it cools. Small amount of color loss and staining of adjacent wool/acrylic fabrics was seen, but sodium perborate in the washing bath may lead to alkaline pH and a darkening effect of the polyphenolic dye. Thus, high washing fastness does not mean that there is no dye loss, but rather a fair amount of dye remains with shifting color in a darker shade. The difference between washing fastness and wet rubbing fastness can be attributed to the different stresses being applied. Washing predominantly involves the fabric coming into contact with a dilute detergent solution in a controlled manner, whereas wet rubbing involves the direct mechanical friction in the presence of water. Water may act as a plasticizer and can help to soften surface/inter-filament dye deposits, which can increase the ease of dye transfer to the rubbing cloth, even if laundering fastness is maintained [81].

The fastness of the perspiration was also good both in acid and alkaline conditions. Color change was rated as 4–5 in acidic perspiration and staining was 3–4 to 4–5 depending on the adjacent fiber in acidic perspiration. The color change for alkaline perspiration was also 4–5, and the staining resistance was excellent on acetate, polyester and acrylic. These results indicate that the dye–fiber association was relatively stable under simulated sweat conditions. The slightly lower staining ratings observed for nylon and wool in some cases may be due to their greater ability to bind polyphenolic compounds than more hydrophobic synthetic fibers [76,82]. The stable color change rating under acidic and alkaline perspiration also indicate that the dyed nylon is suitable for moderate pH changes during use.

Saliva fastness showed a color change rating of 4–5, with excellent staining resistance on acetate, polyester, and acrylic, and very good to excellent staining resistance on cotton. Nylon and wool showed slightly lower but still acceptable ratings of 4 and 3–4, respectively. This is a significant result for skin contact and/or potential mouth contact textile applications where resistance to mild aqueous biological media is desired. The good saliva fastness suggests that the dye constituents retained on nylon were not easily released under near-neutral aqueous conditions [83].

The light fastness was rated on blue wool scale 5, which shows good resistance to xenon arc light exposure. This is still a promising result as many natural dyes have been found to undergo degradation under light exposure because of oxidation or breakage of chromophoric structures [84,85]. The good light fastness may be associated with the presence of polyphenolic compounds in *T. catappa* extract, which can contribute to antioxidant activity and partial stabilization of chromophoric groups. The chemical basis of the natural dye activity was confirmed by the studies that reported the presence of tannins, flavonoids, phenols and other polyphenolic components in *T. catappa* leaves [86].

Overall, the fastness results demonstrate that *T. catappa* leaf extract can produce a durable natural dyeing effect on nylon without the use of metallic mordants. The combination of very good washing, rubbing, perspiration, saliva, and light fastness suggests that the dye molecules were effectively retained by the polyamide substrate. This performance is consistent with the proposed fixation mechanism involving electrostatic attraction between protonated nylon amino groups and ionizable dye constituents, supported by hydrogen bonding between nylon amide groups and phenolic hydroxyl groups of the natural dye.

4.7. Ultraviolet Protection Performance

The undyed nylon fabric showed UVA and UVB transmittance values of 10.90% and 2.49%, respectively, corresponding to UVA and UVB blocking of 89.10% and 97.51%. It had a mean UPF rating of 26.39, which represents an adequate to very good level of UV protection. Once dyed under the optimized condition, the UVA and UVB transmittance values dropped significantly to 0.03%, resulting in 99.97% blocking of UVA and UVB radiation. The mean UPF value of the dyed nylon was 1995, which falls far above the highest commonly used textile UV-protection rating category and is therefore reported as UPF 50+ [14]. This significant enhancement may be explained by the dark shade formation and the presence of retained polyphenolic dye components on and inside the nylon structure. The chromophoric and aromatic constituents of *T. catappa* extract may absorb and scatter incident UV while the process of surface/inter-filament dye deposition seen in the SEM may also reduce the amount of UV transmission through the fabric. These results indicate that *T. catappa* leaf extract not only provides effective mordant-free coloration of nylon but also imparts excellent UV-protective functionality.

5. Conclusions

This study demonstrates the feasibility of using *Terminalia catappa* mature leaf extract as a mordant-free natural dye for nylon fabric. The work addresses the need for bio-based coloration systems for synthetic polyamide textiles while maintaining acceptable color strength and end-use durability. Since nylon dyeing is strongly governed by terminal amino group protonation, dye diffusion, and dye–fiber affinity, the study systematically optimized extraction pH, dyebath pH, dyeing time, and temperature to establish an effective dyeing route.

The results validated that the most effective strategy was the combination of alkaline extraction and acidic dyeing. The extraction at pH 11 resulted in better release of color active polyphenolic compounds resulting in a K/S value of 21.53 compared to 16.19 obtained for neutral extraction when the dyeing conditions were pH 3, 100 °C, 60 min. The most important dyeing parameter was the dyebath pH, as the dye uptake was strong with dyeing in an acidic medium at pH 3, while an alkaline dyeing bath at pH 11 resulted in low dye uptake; for instance, the dye uptake in terms of K/S value was 21.53 at dyebath pH 3 and 0.49 at dyebath pH 11. Extending the dyeing time from 60 to 90 min did not improve color yield, while increasing the temperature from 80 to 120 °C enhanced K/S from 11.67 to 21.94. The optimized condition was then determined to be extraction pH 11, dyebath pH 3,

120 °C, and 60 min, which resulted in a deep shade with ΔL of −54.25, Δa of 8.03 and Δb of 24.92. Although the highest color strength was achieved at 120 °C, the relatively small difference between 100 °C and 120 °C suggests that further optimization should consider energy consumption together with color yield for industrial applications.

The FTIR analysis supported the presence of hydrogen bonds and possible electrostatic interactions between the *T. catappa* derived polyphenolic compounds and the terminal amino groups of nylon amide. SEM observations also confirmed the presence of surface deposition and inter-filament coverage with dye without any significant damage to the fibers. The optimized sample also showed promising fastness performance, including washing color change of 4–5, dry rubbing of 4–5, wet rubbing of 3–4, perspiration color change of 4–5, saliva color change of 4–5, light fastness of grade 5, and excellent ultraviolet protection with a UPF 50+ rating. The outcomes of this research revealed the capability of the dyeing system to be used for durable dyeing without any metallic mordant.

The overall conclusion can be made that the *T. catappa* leaf extract is a potential natural dye source for nylon, especially when extracted under alkaline and dyed under acidic conditions. This study, however, included only one nylon fabric, and was not accompanied by detailed chromatographic identification or repeated laundering testing or full Natural Organic Dye Standard compliance testing. Furthermore, the present study did not include detailed chemical characterization of the dye components extracted from the dyes and the pH-dependent chromatic behavior. A further LC–MS, HPLC, UV–Vis chemical profiling was not possible due to limited access to advanced analytical facilities. Thus, the relationship between specific coloring compounds and extract color variation at different pH levels should be investigated in future work. The stability of the dye liquor extracted during storage was also not studied which could be significant for practical batch-to-batch reproducibility. Furthermore, the effect of various fabric constructions, yarn fineness and nylon grades on dye uptake and fastness behavior was not assessed in the study. The lack of testing for antimicrobial properties and antioxidant activity further restricts the general functional interpretation of the dyed nylon, though polyphenolic compounds with bioactivity have been found in *T. catappa* leaves. Therefore, future work should be directed towards LC–MS/HPLC profiling, dye exhaustion and fixation quantification, repeated wash durability, ΔE-based evaluation of color change, and pilot-scale evaluation of process sustainability. Future research should also address issues of extract stability, reproducibility of shade from multiple dyeing batches, optimization of lower temperature dyeing processes to lower the energy requirement, and evaluation of the performance of these extracts with various commercial acid dyes and other known natural dyestuffs. Life-cycle assessment, effluent characterization, and techno-economic analysis would also be valuable to calculate the real industrial feasibility of a mordant-free natural dyeing process.

Author Contributions: Conceptualization, B.C.M., A.A.F. and M.M.A.; methodology, B.C.M., A.A.F. and M.M.H.; validation, B.C.M., M.M.A., C.E.C. and M.H.M.; formal analysis, B.C.M., A.A.F. and M.K.U.; investigation, B.C.M., A.A.F., S.A.M. and M.H.M.; resources, B.C.M., M.M.H., M.K.U., M.M.A. and C.E.C.; data curation, B.C.M. and A.A.F.; writing—original draft preparation, B.C.M.; writing—review and editing, B.C.M., M.M.A., C.E.C., M.K.U. and M.M.H.; visualization, B.C.M., S.A.M. and M.H.M.; supervision, M.M.A. and C.E.C.; project administration, B.C.M., M.M.A. and C.E.C.; funding acquisition, M.M.A. and C.E.C. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Data Availability Statement: The original contributions presented in this study are included in the article. Further inquiries can be directed to the corresponding authors.

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

Abbreviations

The following abbreviations are used in this manuscript:

FTIR	Fourier transform infrared
ATR	Attenuated total reflectance
FE-SEM/FESEM	Field-emission scanning electron microscopy/microscope
SEM	Scanning electron microscopy
K/S	Kubelka–Munk color strength value
CIELAB	Commission Internationale de l'Éclairage L*a*b* color space
L*	Lightness coordinate
a*	Red–green coordinate
b*	Yellow–blue coordinate
ΔL, Δa, Δb	Difference values of L*, a*, and b*
GSM	Grams per square meter
pH	Potential of hydrogen
ISO	International Organization for Standardization
DIN	Deutsches Institut für Normung/German Institute for Standardization
NODS	Natural Organic Dye Standard
CAS	Chemical Abstracts Service
GAE	Gallic acid equivalent
QE	Quercetin equivalent
LC–MS	Liquid chromatography–mass spectrometry
HPLC	High-performance liquid chromatography
UV	Ultraviolet

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