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SUSTAINABLE DYEING OF COTTON WITH COFFEE HUSKS (COFFEA ARABICA L.) AND POMEGRANATE PEELS (PUNICA GRANATUM) EXTRACTS USING CHITOSAN AND PLANT-BASED BIOMORDANTS: EVALUATION OF COLOR AND PROTECTION FACTOR

TINGIMENTO SUSTENTÁVEL DE ALGODÃO COM EXTRATOS DE CASCAS DE CAFÉ (COFFEA ARABICA L.) E CASCAS DE ROMÃ (PUNICA GRANATUM) UTILIZANDO QUITOSANA E BIOMORDENTES VEGETAIS: AVALIAÇÃO DA COR E DO FATOR DE PROTEÇÃO

TINCIÓN SOSTENIBLE DE ALGODÓN CON EXTRACTOS DE CASCARILLA DE CAFÉ (COFFEA ARABICA L.) Y PIELES DE GRANADA (PUNICA GRANATUM) UTILIZANDO QUITOSANA Y BIOMORDIENTES VEGETALES: EVALUACIÓN DEL COLOR Y DEL FACTOR DE PROTECCIÓN

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ABSTRACT

Interest in the production of sustainable textile products has led to the development of eco-friendly dyeing processes utilizing agro-industrial byproducts. This study investigated a completely bio-based system for cotton fabrics utilizing coffee husk (Coffea arabica L.) and pomegranate peels (Punica granatum) extracts combined with bio-mordants from chitosan, pomegranate peels and guava leaves (Psidium guajava), eliminating synthetic metallic mordants. Experimental results demonstrated that sequential synergy between extracts depends heavily on pH, with acidic media (pH3.0 -5.1) optimizing polyphenol stability and hydrogen bonding with cellulose. Pre-biomordant with guava leaf extract followed by pomegranate dyeing (TpH3.46ExRPMG) yielded the highest color strength (K/S=6.63). For simultaneous biomordant with coffee extract, 50% pomegranate extract TMSRpH3.46 (50%) ExcpH3.46 performed best (K/S=4.64). Color fastness to domestic laundering was excellent (grades 4 to 4/5), with satisfactory perspiration fastness (3 to 4/5). Beyond color yield, the treatment functionalized the substrate into a high-performance sun-protective textile, elevating the ultraviolet protection factor from Undyed (T control) (UPF~ 5.18) to excellent levels (UPF50+), blocking over 95% of UV radiation. Air permeability slightly decreased after

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processing due to fiber swelling and polyphenol deposition but stabilized uniformly post-washing (18.7-25.0 l/m2/s), ensuring that color yield and UV blocking are achieved without sacrificing fabric breathability. In conclusion, combination of coffee, pomegranate, guava, and chitosan offers an efficient, functional, and circular-economy framework for high-value sustainable cotton coloration.

Keywords: Natural Dyeing. Agro-Industrial Waste. Biomordant. UV Protection. Sustainable Textiles.Circular Economy

RESUMO

O interesse na produção de produtos têxteis sustentáveis impulsionou o desenvolvimento de processos de tingimento ecológicos que utilizam subprodutos agroindustriais. Este estudo investigou um sistema de base totalmente biológica para tecidos de algodão, utilizando extratos de casca de café (Coffea arabica L.) e casca de romã (Punica granatum) combinados com biomordentes à base de quitosana, casca de romã e folhas de goiabeira (Psidium guajava), eliminando assim o uso de mordentes metálicos sintéticos. Os resultados experimentais demonstraram que a sinergia sequencial entre os extratos depende fortemente do pH, sendo que meios ácidos (pH 3,0–5,1) otimizam a estabilidade dos polifenóis e as ligações de hidrogênio com a celulose. O pré-tratamento com biomordente de extrato de folha de goiabeira seguido de tingimento com romã (TpH3.46ExRPMG) apresentou a maior intensidade de cor (K/S = 6,63). Para o biomordente simultâneo com extrato de café, a combinação de 50% de extrato de romã (TMSRpH3.46) e 50% de extrato de café (ExcpH3.46) obteve o melhor desempenho (K/S = 4,64). A solidez da cor à lavagem doméstica foi excelente (graus 4 a 4/5), com solidez satisfatória à transpiração (3 a 4/5). Além do rendimento de cor, o tratamento conferiu funcionalidade ao substrato, transformando-o em um têxtil de alto desempenho com proteção solar; o Fator de Proteção Ultravioleta (FPU) elevou-se de níveis baixos na amostra não tingida (controle T, FPU ~ 5,18) para níveis excelentes (FPU 50+), bloqueando mais de 95% da radiação UV. A permeabilidade ao ar diminuiu ligeiramente após o processamento, devido ao inchamento das fibras e à deposição de polifenóis, mas estabilizou-se de forma uniforme após a lavagem (18,7–25,0 l/m²/s), garantindo que o rendimento de cor e o bloqueio de UV fossem alcançados sem comprometer a respirabilidade do tecido. Em suma, a combinação de café, romã, goiaba e quitosana oferece uma abordagem eficiente, funcional e alinhada à economia circular para o tingimento sustentável de algodão de alto valor agregado.

Palavras-chave: Tingimento Natural. Resíduos Agroindustriais. Biomordente. Proteção UV. Têxteis Sustentáveis. Economia Circular.

RESUMEN

El interés por la producción de productos textiles sostenibles ha impulsado el desarrollo de procesos de tintura ecológicos que utilizan subproductos agroindustriales. Este estudio investigó un sistema de base totalmente biológica para tejidos de algodón, empleando extractos de cáscara de café (Coffea arabica L.) y de granada (Punica granatum) en combinación con biomordientes derivados de quitosano, cáscara de granada y hojas de guayaba (Psidium guajava), eliminando así el uso de mordientes metálicos sintéticos. Los resultados experimentales demostraron que la sinergia secuencial entre los extractos depende en gran medida del pH; los medios ácidos (pH 3,0-5,1) optimizaron la estabilidad de los polifenoles y la formación de enlaces de hidrógeno con la celulosa. El pretratamiento con biomordiente de extracto de hoja de guayaba seguido de la tintura con granada

(TpH3.46ExRPMG) produjo la mayor intensidad de color (K/S = 6,63). En el caso del biomordentado simultáneo con extracto de café, la combinación de extracto de granada al 50 % (TMSRpH3.46) y extracto de café al 50 % (ExcpH3.46) ofreció el mejor rendimiento (K/S = 4,64). La solidez del color al lavado doméstico fue excelente (grados 4 a 4/5), con una solidez satisfactoria frente a la transpiración (grados 3 a 4/5). Más allá del rendimiento del color, el tratamiento funcionalizó el sustrato convirtiéndolo en un textil de alto rendimiento con protección solar, elevando el factor de protección ultravioleta desde el nivel de la muestra sin teñir (control T, UPF ~ 5,18) hasta niveles excelentes (UPF 50+), bloqueando más del 95 % de la radiación UV. La permeabilidad al aire disminuyó ligeramente tras el procesamiento debido a la hinchazón de las fibras y a la deposición de polifenoles, pero se estabilizó de manera uniforme después del lavado (18,7-25,0 l/m²/s), garantizando así un rendimiento de color y bloqueo de rayos UV sin sacrificar la transpirabilidad del tejido. En conclusión, la combinación de café, granada, guayaba y quitosano ofrece un marco eficiente, funcional y basado en la economía circular para la tintura sostenible de algodón de alto valor.

Palabras clave: Tintura Natural. Residuos Agroindustriales. Biomordiente. Protección UV. Textiles Sostenibles. Economía Circular.

1 INTRODUCTION

The textile industry plays a significant role in the global economy; however, it is recognized as one of the most environmentally damaging sectors, relying heavily on synthetic dyes to satisfy consumer demand for vibrant colors (KANT, 2012). The discharge of these synthetic compounds and heavy-metal-based mordants poses severe environmental and public health risks, including carcinogenic and mutagenic effects, while accounting for nearly 20% of global industrial water pollution; (SOYINKA et al., 2026, ISLAM et al., 2025, PERIYASAMY, 2024; (HASSAAN, EL NEMR, 2017). In response to this ecological degradation, adopting sustainable practices and exploring eco-friendly alternatives to petrochemical-derived chemicals have become essential (LELLIS et al., 2019). Reviving the use of natural dyes derived from plants, minerals, and animals provides biodegradable, non-toxic alternatives that offer environmental compatibility and safety for human health (NATERI, NATERI, 2025).

A promising strategy within the circular economy framework is the valorization of agro-industrial waste into high-performance bio-based materials (YUSUF, M., SHABBIR, M. MOHAMMAD, 2017). The food industry generates millions of tons of byproducts annually that are rich in bioactive molecules. Among these resources are spent coffee husks (*Coffea arabica L.*) and pomegranate peels (*Punica granatum L.*). Coffee husks (*Coffea arabica L.*) represent a substantial source of organic waste, with global Arabica coffee production estimated at 99 million bags for the 2024–2025 season (FERREIRA, 2024). Chemically, coffee husks constitute a complex matrix composed of carbohydrates (58–85%) and lignocellulosic fibers, as well as polyphenols and tannins (1–2.3%) exhibiting natural dyeing properties (UNGUREANU, VLĂDUȚ, 2026; CANGUSSU et al., 2021). Pomegranate peels (*Punica granatum* L.) can be utilized as both a colorant and a biomordant (YANG et al., 2023). The content of ellagic acid and hydrolysable tannins in pomegranate residues functions as a molecular bridge, establishing hydrogen bonds and van der Waals interactions between the activated cellulosic structure and the polyphenolic chromophores of the coffee dye (OTAVIANO et al., 2023; BEN SLIMANE et al., 2022).

Despite the composition of plant-derived materials, applying natural dyes to cellulosic fibers, such as cotton, presents challenges regarding color stability and fastness (BROADBENT, 2001). Cotton, selected in this context for its natural origin and comfort properties, exhibits a negatively charged surface in aqueous media, resulting in low affinity for plant-derived chromophores (NAZIR et al., 2022; SHAHID, SHAHID-UL-ISLAM,

MOHAMMAD, 2013). Although metallic mordants (such as alum or ferrous sulfate) are traditionally employed to fix natural pigments, they generate toxic effluents that require complex treatments, shifting the environmental burden rather than eliminating it (SAMANTA, AGARWAL, 2009; SAXENA, RAJA, 2014)

To establish a process with lower environmental impact, multi-component botanical systems and biopolymers emerge as viable pathways (PRANTA, RAHAMAN, 2024). A preliminary bio-activation step using tannin-dense extracts, such as guava leaves (*Psidium guajava*), can serve as a primary bio-mordant (BALOCH et al., 2024). *Psidium guajava* is widely cultivated in humid climates, with global production reaching approximately 50 million units annually across the Americas and Africa. Its phytochemical profile includes secondary metabolites such as sesquiterpenes (β-caryophyllene), monoterpenes (d-limonene and eucalyptol/1,8-cineole), phytol, squalene, methyl palmitate, and methyl linolenate (AL-THAGAFI et al., 2026). The flavonoids and tannic acid present in guava extracts modify the fiber's chemical topography by increasing the density of free phenolic hydroxyl groups, thereby providing new binding sites (MAHAJAN et al., 2025).

Additionally, biopolymers such as chitosan, a deacetylated derivative of chitin obtained from crustacean shells, offer antimicrobial properties, biocompatibility, and biodegradability. Due to its polycationic nature, chitosan can bind to unprotonated, negatively charged surfaces across pH ranges varying from acidic to neutral (NATERI, NATERI, 2025).

The fixation efficiency of these polyphenolic networks depends on certain physicochemical parameters, including the liquor ratio and pH (PEREIRA et al., 2025). Adjusting the pH to slightly acidic conditions optimize the protonation states of both the plant-derived chromophores and the hydroxyl groups of the modified cellulose, thereby minimizing electrostatic repulsion and shifting the chemical equilibrium toward maximum pigment exhaustion (PHAN et al., 2016).

Based on these principles, this study explores the theoretical synergy of a sequential system employing coffee and pomegranate husks as colorants, alongside chitosan, guava leaves, and pomegranate husks as biomordants, in pH-controlled environments. By valorizing these agro-industrial residues, the study proposes a methodology aimed at reducing the chemical footprint of the textile dyeing process and promoting sustainable innovation

2 MATERIALS AND METHODS

2.1 MATERIALS

Organic coffee husks of the *Coffea arabica* species (participatory certification from Orgânicos Sul de Minas, registered with MAPA) were kindly donated by the Cooperativa dos Camponeses Sul Mineiros, in Minas Gerais. Guava leaves (*Psidium guajava*) were collected on the campus of the School of Arts, Sciences and Humanities of the University of São Paulo - USP Campus - East. The dried pomegranate (*Punica granatum*) peels were purchased from the “Mercado Granel” company, located in Brazil. Chitosan was obtained from Sigma Aldrich, St. Louis, USA, having at least 75% deacetylation. Acetic acid, citric acid, sodium hydroxide, L-histidine monohydrochloride, sodium chloride, monosodium orthophosphate dihydrate, disodium orthophosphate dihydrate and ISO ECE standard detergent were used. The chemicals used in the experiments were of analytical grade. The 100% cotton fabric, plain weave structure, in white, ready to dye, had a grammage of 118.0 ± 1.0 g/m², an average thickness of 0.269 ± 0.02 mm, a mean weft yarn density of 27.6 ± 0.7 yarns/cm, a mean warp yarn density of 38.3 ± 1.0 yarns/cm, and a total mean area density of 65.9 ± 1.2 yarns/cm². The mean yarn count was 16.2 ± 0.3 tex for the weft and 15.9 ± 0.3 tex for the warp. The fabric presented an ultraviolet protection factor (UPF) of 4,697 and color coordinates L* = 93.54, a* = -0.17, b* = 2.82, C* = 2.83, and hº = 93.43.

2.2 DRYING, GRINDING AND GRANULOMETRY

The raw materials were dried at 45 °C for 24 hours and ground in a Spencer SP226/02 knife mill, followed by particle size analysis. Grains with a size of 0.088 mm were used in the extraction processes.

2.3 AQUEOUS EXTRACTION OF CORANTS AND BIOMORDANTS

For this procedure, 200 g of particles with a size of 0.088 mm from coffee husks, pomegranate peel or guava leaves were used and placed in a Becker with 1000 mL of deionized water. The Beckers were placed on a heating plate until they reached a temperature of 70 ºC and maintained at this temperature for 10 minutes. After this period, the Beckers were covered and left to cool for one hour. Subsequently, the extract samples were filtered using a polyester voile fabric (Figure 1) to avoid retaining the dyes. This was followed by two more heating and exhaustion steps.

Figure 1

Coffee husk extract filtration



2.4 CHITOSAN PREPARATION, PRE-MORDANT IN FOULARD

The biomordant solution was prepared by dissolving 0.15 g of chitosan in 1500 mL of a 2% (v/v) aqueous citric acid 2% (v/v) solution in a 2000 mL Becker. To ensure complete dissolution of the polymer, the mixture was kept under continuous stirring for 24 h using a magnetic stirrer (Fisatom 752A). Fabric samples, each weighing 6.0 g, were padded using a laboratory padder (Mathis) with a roller pressure of 1.5 bar and a pick-up of 80% of the dry fabric weight. Alternatively, a comparative bio-mordant bath containing chitosan dissolved in 2% (v/v) acetic acid was also evaluated. Immediately after padding, the fabrics were dried in a laboratory stenter (Mathis) at 120 °C for a strict duration of 5 min. All procedures were conducted at the Textile Chemistry Laboratory of EACH-USP.

2.5 PROCESS OF DYEING

The dyeing processes were carried out with a liquor ratio of 1:20 (m/v) and in accordance with BECHTOLD et al. (2003) using organic coffee husk extract at pH 5.1 (unadjusted) and at pH 3, 7, and 11, in triplicate, without a mordant fabric and pre-biomordant fabric. All dyed samples consisted of cotton fabric measuring 10 x 5 cm and weighing 2 g. The dye and biomordant solutions were used without pH adjustment and with pH adjusted to 3, 3.46, 5.32, 7, and 11, using NaOH and CH_3CO_2H, both (1 M), under the following conditions:

Dyeing without and with pre-biomordant: Dyeing with coffee extract at pH 3, 5.1, 7, and 11 on fabrics that were either untreated or pre-biomordant with chitosan prepared in citric acid (2% w/v) or acetic acid (2% w/v), applied via padding (80% pick-up)—and with pomegranate extract (pH 3.46) impregnated using HT Mathis equipment following the curve of BECHTOLD et al. (2003). Dyeing of fabrics pre-biomordant with guava leaf extract impregnated using HT Mathis equipment following the curve of BECHTOLD et al. (2003)

using coffee extract at pH 3, 5.1, 7, and 11, and pomegranate extract at pH 3, 3.46, 7, 10, and 11 (Table 1).

Dyeing without and with simultaneous biomordant: Simultaneous biomordant and dyeing performed in HT Mathis equipment following the same curve of Bechtold et al. (2003), using pomegranate extract at pH 3.46 at concentrations of 6, 13, 20, and 50% (v/v) and coffee extract at pH 5.1. Dyeing with pomegranate and coffee extract pH values adjusted to 5.32, 3.46, 7, and 11. Simultaneous biomordanting with guava leaf extract at concentrations of 6, 13, 20, and 50% (v/v) and dyeing with coffee extract, with mordant and dye pH values adjusted to 3, 7, and 11 (Table 2).

The dyeing and simultaneous biomordant processes were in accordance with Bechtold et al. (2003), in which the temperature started at 40 °C, then rose to 98 °C with a gradient of 2 °C/min, remained at this temperature for 60 min, and then decreased to 60 °C. After dyeing, the samples were rinsed in running water and dried at 45 °C. All samples were characterized by the colorimetric coordinates in the CIEL*a*b* space (item 2.6), and the K/S values (color strength) were used to select the following samples (Table 3) for characterization regarding wash fastness, acid and alkaline sweat fastness, protection factor, and permeability (items 2.7 to 2.9).

2.6 COLORIMETRIC ANALYSIS

The colorimetric properties of the fabrics were determined using a Konica Minolta CM-26d spectrophotometer, available at the Textile Technical Research Laboratory (LPTT-EACH-USP), FAPESP project 2023/15419-9, under D65 illuminant and a 10° viewing angle, with specular reflection included, a UV filter, and OnColor software. CIEL*a*b* coordinates and their respective ΔE* values were collected, and color intensity (K/S) was also determined.

2.7 COLOR FASTNESS ASSESSMENT

The color fastness properties of the dyed fabrics were evaluated for resistance to acidic and alkaline perspiration, washing, and artificial light exposure, in accordance with the following standards: ABNT NBR ISO 139:2008, ISO 105-E04:2013, ISO 105-C06:2010, and ISO 105-A03:1993, respectively.

2.8 EVALUATION OF THE TEXTILE UV PROTECTION FACTOR

A T391 UV Protection Tester (Sataton Instruments Technology Co., Ltd.) spectrophotometer and the associated UV anti-ultraviolet property testing software were used to evaluate the UPF of the undyed and dyed fabrics. The tests were based on the standards "Sun Protective Clothing – Evaluation and Classification" (AS/NZS 4399:2017) and "Clothing – Ultraviolet Protection Factor – Requirements and test methods" (ABNT NBR 16695:2018). Table 1 presents the UPF ranges and classification categories.

Table 1

UPF range according to ABNT NBR 16695:2018

UPF rating	Protection	%RVR blocked
15 - 24	Good	93.3-95.9
25 -39	Very good	96.0-97.4
40 -50, 50+	Excellent	Greater than 97.5

2.9 AIR PERMEABILITY

Air permeability tests on undyed fabrics and fabrics dyed with extracts were conducted in accordance with ISO 9237:1995 – Textiles – Determination of the permeability of fabrics to air. Specimens measuring 15 x 15 cm were evaluated using a Textest FX 3300-III air permeability tester with electronic flow control, a 5 cm² test head, and range 4. The programmed pressure was 100 Pa, and results were obtained in L/m²/s. The test was performed at the Textile Technology Laboratory of the Institute for Technological Research (LTT - IPT).

3 RESULTS AND DISCUSSION

3.1 PRELIMINARY ASSESSMENT AND SAMPLE SELECTION

To establish a robust baseline for further investigation, a comparative analysis of the tinctorial efficacy of the different bio-mordanting methods was performed. While chitosan pre-mordanting provides a functional surface modification, the simultaneous meta-mordanting process using pomegranate peel (*Punica granatum*) and guava leaf (*Psidium guajava*) extracts demonstrated a superior performance in terms of color yield.

Pre-biomordant (Table 1) using guava leaves followed by dyeing with pomegranate peel extract at an acidic pH (TpH3.46ExRPMG) yielded the highest color strength (K/S > 6.0), surpassing the simultaneous method.

Regarding the dyeing of cotton fabric with coffee, Table 2 shows that for the pomegranate extract (TMSR), the K/S value scales almost linearly as the concentration increases from 6% to 50%, for example: TMSRpH3.46 (6%) ExcpH3.46 (K/S = 1.04), TMSRpH3.46 (13%) ExcpH3.46 (K/S = 1.55), TMSRpH3.46 (20%) ExcpH3.46 (K/S = 1.71), and TMSRpH3.46 (50%) ExcpH3.46 (K/S = 4.64). Increasing the concentration of the biomordant raises the mass of tannins available in the dye bath solution. A higher tannin content can increase the probability of collisions and the deposition of dye aggregates within the pores of the cotton fiber.

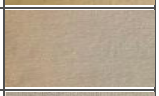
Regarding the dyeing of cotton with a mixture of coffee extract and guava leaf extract (TMSG), acidic pH levels (pH 3.46 to 5.32) resulted in slightly higher fixation compared to neutral and alkaline media (pH 7 and 11), for instance, in samples TpH3ExcPMG (K/S=0.78), TpH5.1ExcPMG (K/S=0.57), TpH7ExcPMG (K/S=0.34), and TpH11.0ExcPMG (K/S=0.70). For guava leaf extract (TMSG) at pH 3 (50%) sample (TMSGpH3 (50%) ExcpH3) the K/S value reached 0.80, whereas at pH 7 and 11, it did not exceed 0.38 and 0.44, respectively. Acidic pH can preserve the integrity of the tannins and flavonoids and prevent extensive ionization of their esterified phenolic hydroxyl groups, thereby maintaining the stability of hydrogen bonds with the cellulose hydroxyl groups. Conversely, in an alkaline medium, massive deprotonation (formation of phenoxides, $–O^-$) and the degradative oxidation of phenols into low-molecular-weight quinones can occur; this generates electrostatic repulsion against the cotton (which also acquires a negative charge in alkaline media) and weakens the dye affinity.

Under all simultaneous biomordant conditions, pomegranate peel extract (TMSR) outperformed guava leaf extract (TMSG) in terms of K/S values and color variation. Pomegranate peel contains a higher concentration of punicalagin and ellagic acid (tannins with lower steric hindrance and high reactivity) (OTAVIANO et al., 2023) than guava leaves, allowing for better diffusional penetration and retention within the tissue's amorphous matrix.

Table 2

*Colorimetric measurement values in the CIEL*a*b* color space for control (undyed) fabric samples and samples dyed with coffee and pomegranate extracts, with or without chitosan pre-biomordant prepared in citric acid (2% w/v), acetic acid (2% w/v), pomegranate extract, or guava leaf extract. The ΔE value was calculated using the undyed sample (control T), and the K/S value was determined at 400 nm*

Fabrics samples	Colorimetric reading						Image of the fabrics
	L*	a*	b*	C	ΔE*	K/S	
Undyed (T control)	93.54	-0.17	2.82	2.83		-	
PMQui	93.51	-0.17	3.02	3.03	0.2	-	
T pH3 ExcSM	80.57	3.6	13.53	14	17.24	0.54	
T pH3ExcPMQuiACi	86.93	2.09	10.03	10.25	10.04	0.85	
TpH3ExcPM QuiACe	76.54	4.64	16.02	16.68	73.84	0.88	
T pH5.1ExcSM	87.31	1.83	8.75	8.94	8.83	0.25	
T pH5.1Exc PMQuiACi	87.97	1.58	7.75	7.91	7.65	0.30	
TPH5.1ExcPM QuiACe	82.06	3.17	13.97	14.32	77.23	0.55	
T pH 7ExcSM	77.36	4.34	16.12	16.7	21.43	0.21	
T pH 7 ExcPM QuiACi	84.01	2.69	12.25	12.54	13.71	0.37	
T pH 11Exc SM	84.15	2.82	12.13	12.45	13.55	0.85	
T pH 11ExcPM QuiACi	83.37	2.91	11.5	11.86	13.72	0.38	
TPH11ExcPM QuiACe	84.63	2.64	10.05	10.39	75.23	0.30	
TPMR	75.08	2.98	20.89	21.1	81.88	3.31	
TpH3ExcPMR	73.41	3.78	15.54	15.99	76.32	1.43	

TpH5,1ExcPMR	76.52	2.81	13.93	14.21	78.58	0.99	
TpH7ExcPR	76.32	2.9	14.25	14.54	78.49	1.00	
TpH11ExcPMR	76.42	2.94	15.78	16.06	79.44	1.01	
TPH3ExcPMG	77.36	3.9	13.32	13.88	73.66	0.78	
TpH5,1ExcPMG	79.98	3.55	12.14	12.65	73.68	0.57	
TpH7,0ExcPMG	84.58	2.82	11.71	12.04	76.47	0.34	
TpH11,0ExcPMG	77.67	3.86	12.86	13.43	73.29	0.70	
TpH3ExRPMG	74.6	4.06	20.34	20.74	78.72	5.44	
TpH3.46ExRPMG	73.34	4.32	21.71	22.13	78.75	6.63	
TpH7.0ExRPMG	62.63	-0.76	14.9	14.92	92.92	4.45	
TpH11.0ExRPMG	55.49	4.42	17.24	17.8	75.63	5.23	

T = Fabrics; PM = pre-biomordant; Exc = coffee extract; ExR = pomegranate extract; SM = unmordanted; QuiACi = chitosan prepared in citric acid (2% w/v) using a padder; QuiACe = chitosan prepared in acetic acid (2% w/v) using a padder; PMR = pre-biomordant with pomegranate (HT); PMG = pre-biomordant with guava leaf (HT)

Table 3

*Colorimetric measurement values in the CIEL*a*b* color space readings for dyeing using simultaneous biomordant with pomegranate extract at pH 3.46 (concentrations of 6%, 13.20%, and 50% v/v) and dyeing with coffee extract at pH 5.1. The pH of the pomegranate and coffee extracts was adjusted to 5.32, 3.46, 7, and 11. Guava leaf extract with a biomordant was also used. ΔE was calculated using Undyed (T control) fabric, and K/S was determined at 400 nm*

Fabrics samples	Colorimetric reading						Image of the fabrics
	L*	a*	b*	C	ΔE*	K/S	
TMSRpH3.46 (6%) ExcpH5.1	82.89	1.51	14	14.09	83.86	0.67	
TMSRpH3.46 (13%) ExcpH5.1	80.85	1.66	15.97	16.05	84.07	0.98	

TMSRpH3.46 (20%) ExcpH5.1	78.65	1.79	18.23	18.32	84.4	1.50	
TMSRpH3.46 (50%) ExcpH5.1	76.65	2.28	20.35	20.48	83.61	2.28	
TMSRpH5.32 (6%) ExcpH5.32	81.03	1.79	13.92	14.03	82.68	0.812	
TMSRpH5.32 (13%) ExcpH5.1	79.4	2.02	16.99	17.11	83.22	1.29	
TMSRpH5.32 (20%) ExcpH5.32	76.29	2.6	17.32	17.51	81.45	1.53	
TMSRpH5.32 (50%) ExcpH5.32	74.4	2.18	20.93	21.05	84.05	3.02	
TMSRpH11 (6%) ExcpH11	80.57	1.97	14.27	14.41	82.14	0.870	
TMSRpH3.46 (6%) ExcpH3.46	80.11	2.65	15.21	15.44	80.13	1.04	
TMSRpH3.46 (13%) ExcpH3.46	77.87	2.35	16.87	17.03	82.06	1.55	
TMSRpH3.46 (20%) ExcpH3.46	78.35	2.72	16.62	16.84	80.7	1.71	
TMSRpH3.46 (50%) ExcpH3.46	69.77	4.93	20.58	21.16	76.54	4.64	
TMSRpH7 (6%) ExcpH7	81.75	2.09	14.06	14.22	81.56	0.72	
TMSRpH7 (13%) ExcpH7	79.74	1.88	16.06	16.17	83.32	1.09	
TMSRpH7 (20%) ExcpH7	77.53	1.89	18.11	18.21	84.05	1.56	
TMSRpH7 (50%) ExcpH7	70.64	0.51	18.02	18.03	88.38	2.61	
TMSRpH11 (6%) ExcpH11	83.62	1.63	13.92	14.02	83.31	0.64	
TMSRpH11 (13%) ExcpH11	78.22	1.26	17.48	17.53	85.87	1.33	
TMSRpH11 (20%) ExcpH11	82	0.28	17.28	17.28	89.08	1.08	
TMSRpH11 (50%) ExcpH11	65.6	3.15	16.45	16.45	78.96	2.41	
TMSGpH3 (6%) ExcpH3	84.68	2.7	11.07	11.39	76.29	0.51	
TMSGpH3 (13%) ExcpH3	82.59	3.11	12.76	13.13	76.29	0.76	
TMSGpH3 (20%) ExcpH3	78.95	3.72	14.07	14.55	75.19	0.79	

TMSGpH3 (50%) ExcpH3	79.47	3.41	13.59	14.01	75.91	0.80	
TMSGpH7 (6%) ExcpH7	82.36	2.7	12.97	13.25	78.23	0.30	
TMSGpH7 (13%) ExcpH7	85.35	2.73	10.28	10.63	75.14	0.29	
TMSGpH7 (20%) ExcpH7	85.23	2.67	10.1	10.45	75.19	0.35	
TMSGpH7 (50%) ExcpH7	84.3	2.95	10.4	10.81	74.17	0.38	
TMSGpH11 (6%) ExcpH11	83.96	2.95	10.81	11.21	74.73	0.20	
TMSGpH11 (13%) ExcpH11	87.8	1.87	8.43	8.64	77.49	0.30	
TMSGpH11 (20%) ExcpH11	85.56	2.54	9.85	10.17	75.56	0.34	
TMSGpH11 (50%) ExcpH11	84.73	2.75	10.3	10.66	75.04	0.44	

T = Fabric, MSR = simultaneous mordant with pomegranate (HT), MSG = pre-biomordant with guava leaves (HT), Exc = coffee extract

The systematic selection of samples based on their K/S values serves as a rigorous criterion for optimization. The prioritization of higher K/S values was based on the literature (PUCCINI et al. 2026; OTAVIANO et al. 2023). In the dyeing of cotton fabrics with coffee extract reported by PUCCINI et al. (2026), K/S values were 1.58 at pH 5.08, 2.28 at pH 7, and 3.05 at pH 11. Regarding pomegranate husks, results obtained by OTAVIANO et al. (2023) for dyeing cotton fabrics showed K/S values of 5.42 at pH 4.06, 3.08 at pH 3 and 3.58 at pH 7.

Table 4 presents the 16 selected samples, including detailed descriptions; these served as the basis for all subsequent analyses, ensuring an experimental foundation for future research on sustainable textile coloration.

Table 4

Description of fabric dyeing and biomordant with the best K/S results

Sample codes	Description of fabric dyeing and biomordant	Image of the dyed fabrics	Color strength (K/S)
TPMR	Pre-biomordant with pomegranate peel extract		3.31

TpH3ExcPMR	Pre-biomordant with pomegranate peel extract and dyed with coffee husk extract at pH 3		1.43
TpH3ExRPMG	Pre-biomordant with guava leaf and dyed with coffee husk extract adjusted to pH 3		5.44
TpH3.46ExRPMG	Pre-biomordant with guava leaf extract and dyed with pomegranate extract at pH 3.46 (unadjusted)		6.63
TpH7.0ExRPMG	Pre-biomordant with guava leaf extract and dyed with pomegranate extract at pH 7		4.45
TpH11.0ExRPMG	Pre-biomordant with guava leaf extract and dyed with pomegranate extract at pH 11		5.23
TMSRpH3.46 (20%) ExcpH5.1	Dyed with coffee extract at pH 5.1 with simultaneous biomordant using 20% pomegranate extract at pH 3.46		1.50
TMSRpH3.46 (50%) ExcpH5.1	Dyed with coffee extract at pH 5.1 with simultaneous biomordant using 50% pomegranate extract at pH 3.46		2.28
TMSRpH5.32 (20%) ExcpH5.32	Dyed with coffee extract at pH 5.32 with simultaneous biomordant using 20% pomegranate extract at pH 5.32		1.53
TMSRpH5.32 (50%) ExcpH5.32	Dyed with coffee extract at pH 5.32 with simultaneous biomordant using 20% pomegranate extract at pH 5.32		3.02
TMSRpH3.46 (13%) ExcpH3.46	Dyed with coffee extract at pH 3.46 with simultaneous biomordant using 13% pomegranate extract at pH 3.46		1.55
TMSRpH3.46 (20%) ExcpH3.46	Dyed with coffee extract at pH 3.46 with simultaneous biomordant using 20% pomegranate extract at pH 3.46		1.71
TMSRpH3.46 (50%) ExcpH3.46	Dyed with coffee extract at pH 3.46 with simultaneous biomordant using 50% pomegranate extract at pH 3.46		4.64
TMSRpH7 (20%) ExcpH7	Dyed with coffee extract at pH 7 with simultaneous biomordant using 20% pomegranate extract at pH 7		1.56
TMSRpH7 (50%) ExcpH7	Dyed with coffee extract at pH 7 with simultaneous biomordant using 50% pomegranate extract at pH 7		2.61
TMSRpH11 (50%) ExcpH11	Dyed with coffee extract at pH 11 with simultaneous biomordant using 50% pomegranate extract at pH 7		2.41

T = Fabrics; PM = pre-biomordant; Exc = coffee extract; ExR = pomegranate extract; SM = unmordanted; QuiACi = chitosan prepared in citric acid (2% w/v) using a padder; QuiACe = chitosan prepared in acetic acid (2% w/v) using a padder; PMR = pre-biomordant with pomegranate (HT); PMG = pre-biomordant with guava leaf (HT)

3.2 COLOR FASTNESS PERFORMANCE

Beyond color strength (K/S), the fastness properties of dyed textiles are critical parameters determining their end-use suitability and industrial potential. Therefore, the color fastness to domestic laundering (ISO 105-C06) and perspiration (ISO 105-E04) were

systematically evaluated for the optimized dyeing routes. The comprehensive results, including numerical ratings for both color change and staining on a standardized multifiber adjacent fabric, are consolidated in Tables 5, 6 and 7.

Overall, the bio-mordanted and dyed cotton specimens displayed remarkable resistance to wet treatments. The synergy established between the tannin-rich plant extracts (pomegranate and guava) and the organic coffee husk pigments facilitate the formation of stable complexes within the cellulose matrix. These interactions significantly limit hydrolytic desorption and dye migration during rigorous washing and sweat exposure.

The laundering fastness ratings, summarized in Table 5, demonstrate excellent resistance to wet treatments, with color change ratings consistently between 4 and 5. This performance is attributed to the formation of a robust macromolecular network where tannin-rich bio-mordants act as molecular bridges. Under acidic dyeing conditions (pH 3.46), the phenolic hydroxyl groups of pomegranate and guava extracts form strong hydrogen-bonded bridges with the cellulose matrix, effectively anchoring the coffee pigments and preventing hydrolytic desorption during the ISO 105-C06 standardized laundering process.

Table 5

Color fastness to domestic washing (ISO 105-C06) of biomordant and dyed cotton fabrics

Fabrics samples	Color Change	Color staining					
		CA	CO	PA	PES	PAC	WO
TPMR	4/5	5	4/5	5	5	5	4/5
TpH3ExcPMR	4	5	4/5	5	5	5	4/5
TpH3ExRPMG	4/5	4/5	5	5	5	5	4/5
TpH3.46ExRPMG	4/5	4/5	5	5	5	5	4/5
TpH7.0ExRPMG	2/3	5	4/5	4	5	4	3
TpH11.0ExRPMG	2	5	4/5	5	5	4/5	3/4
TMSRpH3.46 (20%) ExcpH5.1	4	5	5	5	5	5	4/5
TMSRpH3.46 (50%) ExcpH5.1	4/5	5	5	5	5	5	4/5
TMSRpH5.32 (20%) ExcpH5.32	3/4	5	4/5	4/5	5	4/5	4
TMSRpH5.32 (50%) ExcpH5.32	3/4	5	4/5	4/5	5	4/5	4
TMSRpH3.46 (13%) ExcpH3.46	3/4	5	4/5	4/5	5	4/5	3/4
TMSRpH3.46 (20%) ExcpH3.46	4	5	4/5	4/5	5	4/5	4
TMSRpH3.46 (50%) ExcpH3.46	3/4	5	4/5	4/5	5	4/5	3/4
TMSRpH7 (20%) ExcpH7	4/5	5	4/5	4/5	5	4/5	4
TMSRpH7 (50%) ExcpH7	3	5	3/4	4/5	4/5	4/5	3/4
TMSRpH11 (50%) ExcpH11	2/3	5	4/5	4/5	4/5	4/5	3/4

CA – Acetate; CO – Cotton; PA – Polyamide; PES – Polyester; PAC – Acrylic; WO – Wool. Ratings range from 1 (lowest) – 5 (highest).

Table 6

Color fastness to acid perspiration testing of samples dyed

Fabrics samples	Acid Perspiration Testing						
	Color Change	Color staining					
		CA	CO	PA	PES	PAC	WO
TPMR	4	3	1/2	1	4	3/4	3
TpH3ExcPMR	3	3	3	3	4/5	4/5	4
TpH3ExRPMG	3	3/4	1/2	2	4	3	2
TpH3.46ExRPMG	4/5	3	1	2	2	3	2
TpH7.0ExRPMG	4/5	4	2/3	3	4	4	3
TpH11.0ExRPMG	2	4/5	3	3/4	4/5	4/5	3
TMSRpH3.46 (20%) ExcpH5.1	4/5	4	3	3	4/5	4	3
TMSRpH3.46 (50%) ExcpH5.1	4/5	3	1	1	4/5	3/4	2/3
TMSRpH5.32 (20%) ExcpH5.32	4	3/4	3	3	4	4/5	2/3
TMSRpH5.32 (50%) ExcpH5.32	3	4/5	3	3	4/5	4	2
TMSRpH3.46 (13%) ExcpH3.46	3/4	4	3	3	4/5	4	2
TMSRpH3.46 (20%) ExcpH3.46	4	4/5	2/3	2	4	4	3
TMSRpH3.46 (50%) ExcpH3.46	4	3	1/2	2	4	4	3
TMSRpH7 (20%) ExcpH7	4	4	3/4	3	4/5	4	2
TMSRpH7 (50%) ExcpH7	3	4	3/4	4	4/5	4	3
TMSRpH11 (50%) ExcpH11	3	4/5	4	4/5	4	4	3/4

CA – Acetate; CO – Cotton; PA – Polyamide; PES – Polyester; PAC – Acrylic; WO – Wool. Ratings range from 1 (lowest) – 5 (highest).

Table 7

Color fastness to alkaline perspiration testing of samples dyed

Fabrics samples	Alkaline Perspiration Testing						
	Color Change	Color staining					
		CA	CO	PA	PES	PAC	WO
TPMR	4/5	2	2	2/3	3	3	2
TpH3ExcPMR	4	3/4	3	3/4	4/5	4	3
TpH3ExRPMG	4	3/4	2	3	3	2/3	1/2
TpH3.46ExRPMG	4	3	2	3	3/4	3	2/3
TpH7.0ExRPMG	3	4	3	4	4/5	4	3/4
TpH11.0ExRPMG	3	3/4	3	4	4/5	4	2/3
TMSRpH3.46 (20%) ExcpH5.1	4/5	4	3	3/4	4/5	4	3/4
TMSRpH3.46 (50%) ExcpH5.1	4	3/4	3	3/4	4	3/4	2
TMSRpH5.32 (20%) ExcpH5.32	4/5	3/4	3	4	4/5	4	3/4
TMSRpH5.32 (50%) ExcpH5.32	4	4	3	3/4	4	4	3/4
TMSRpH3.46 (13%) ExcpH3.46	4	4	3/4	4	4	4	3/4
TMSRpH3.46 (20%) ExcpH3.46	4/5	4	3/4	4	4	3/4	3/4
TMSRpH3.46 (50%) ExcpH3.46	4	3/4	3	3/4	4	4	3
TMSRpH7 (20%) ExcpH7	4/5	4	3/4	4	4/5	4	3
TMSRpH7 (50%) ExcpH7	4	4	3/4	4	4/5	4	4

TMSRpH11 (50%) ExcpH11	4	4/5	4	4/5	4/5	4/5	4/5

CA – Acetate; CO – Cotton; PA – Polyamide; PES – Polyester; PAC – Acrylic; WO – Wool. Ratings range from 1 (lowest) – 5 (highest).

The results presented in Tables 6 and 7 demonstrate a clear pH dependence regarding the stability of the complexes formed with the biomordant. Overall, the acid perspiration fastness ratings were superior to those observed under alkaline conditions.

The reduction in fastness observed at higher pH levels (pH > 7) clearly demonstrates the susceptibility of phenolic compounds to deprotonation, a process that increases dye solubility and promotes pigment migration to adjacent fibers. These findings underscore the necessity of maintaining an acidic to neutral medium to ensure the durability of the system treated with the biomordant.

The TMSRpH3.46 (20%) ExcpH5.1 sample (simultaneous mordanting with 20% pomegranate extract at pH 3.46 and dyeing with coffee at pH 5.1) yielded the best results. This suggests that, at pH 3.46, the tannins in the pomegranate extract (ellagic acid and punicalagin) are likely protonated, facilitating anchoring to the cellulose via hydrogen bonds. Furthermore, at pH 5.1, the coffee extract allows for adequate penetration of chlorogenic acids into the fabric-mordant complex without hydrolyzing the fiber or causing severe electrostatic repulsion (which is common at an alkaline pH of 11).

At a biomordant concentration of 50%, there appears to be an excess of tannins accumulated on the surface of the cotton fiber; upon contact with sweat (whether acidic or alkaline), these unfixed tannins easily detach, causing the fabric to fade and staining adjacent fibers (as explained in notes 1 and 2 regarding the 50% samples). A concentration of 20% appears to offer the ideal stoichiometric ratio: sufficient tannins to fix the coffee dye without leaving any free excess on the surface.

3.3 FABRIC PROTECTION FACTOR BEFORE AND AFTER WASHING

Table 8 presents the dyeing results, including the best K/S values (indicating color strength), ultraviolet protection factor (UPF) data, and wash durability. The undyed control fabric showed no UV protection, with a value of 5.18. All other dyed samples exhibited a protection factor. Specifically, sample TpH3ExRPMG showed a value of 113.45; although it experienced a 63.16% reduction after washing, it still maintained a protection value of 41.79.

Higher protection values generally corresponded to higher K/S values. However, sample TpH3ExcPMR showed a K/S of 1.43 and a protection value of 55.08 (categorized as

"excellent"), along with a smaller reduction after washing (31.35%). The smallest reductions in UV protection were observed in samples pre-mordanted with pomegranate. This may be attributed to the fact that pomegranate peel (*Punica granatum*) is a source of hydrolysable tannins, which possess multiple hydroxyl (-OH) and phenolic groups that bind simultaneously to the textile fiber and the dye molecules (OTAVIANO et al., 2023). The washing process employed corresponds to five domestic wash cycles.

Table 8

Fabric protection factor (UPF) results before and after washing

Samples	UPF before washing	UPF after washing	Percentage difference (%)
Undyed (T control)	5.18	-	-
TPMR	70.85	43.46	38.66
TpH3ExcPMR	55.08	37.81	31.35
TpH3ExRPMG	113.45	41.79	63.16
TpH3.46ExRPMG	95.84	41.21	57.00
TpH7.0ExRPMG	103.23	42.39	58.94
TpH11.0ExRPMG	80.26	29.46	63.29
TMSRpH3.46 (20%) ExcpH5.1	42.59	20.47	51.94
TMSRpH3.46 (50%) ExcpH5.1	48.24	32.21	33.23
TMSRpH5.32 (20%) ExcpH5.32	52.69	25.53	51.55
TMSRpH5.32 (50%) ExcpH5.32	50.3	27.9	44.53
TMSRpH3.46 (13%) ExcpH3.46	56.3	21.71	61.44
TMSRpH3.46 (20%) ExcpH3.46	58.67	21.5	63.35
TMSRpH3.46 (50%) ExcpH3.46	76.33	34.45	54.87
TMSRpH7 (20%) ExcpH7	42.66	19.88	53.40
TMSRpH7 (50%) ExcpH7	53.3	29.6	44.47
TMSRpH11 (50%) ExcpH11	50.31	18.69	62.85

T = Fabrics; PM = pre-biomordant; Exc = coffee extract; ExR = pomegranate extract; SM = unmordanted; QuiACi = chitosan prepared in citric acid (2% w/v) using a padder; QuiACe = chitosan prepared in acetic acid (2% w/v) using a padder; PMR = pre-biomordant with pomegranate (HT); PMG = pre-biomordant with guava leaf (HT)

3.4 AIR PERMEABILITY OF FABRICS

Table 9 shows that the air permeability of the cotton fabric (control: 29.5 l/m^2/s) decreased by 20–40% after dyeing (ranging from 17.6 to 26.4 l/m^2/s), likely due to thermal fiber swelling and tannin deposition within the pores. Following the washing process, the values stabilized within a consistent range (18.7–25.0 l/m^2/s), driven by the removal of unfixed excess biomordant and structural fabric relaxation. This stabilization demonstrates that the

functional finish preserves inter-yarn airflow. The results in Tables 8 and 9 indicate that the process imparts color and advanced UV protection without compromising the breathability or thermal comfort of the textile substrate.

Table 9

Air permeability of undyed and dyed cotton fabric with pre-biomordant and simultaneous biomordant, before and after washing.

	Permeability before washing ($l/m^2/s$)	Permeability after washing ($l/m^2/s$)
Undyed (T control)	29.5	-
TPMR	23.8	20.2
TpH3ExcPMR	18.8	20.9
TpH3ExRPMG	19.7	20.4
TpH3.46ExRPMG	19.3	20.2
TpH7.0ExRPMG	20.7	20.0
TpH11.0ExRPMG	22.6	20.0
TMSRpH3.46 (20%) ExcpH5.1	22.6	20.8
TMSRpH3.46 (50%) ExcpH5.1	26.4	19.3
TMSRpH5.32 (20%) ExcpH5.32	20.1	19.7
TMSRpH5.32 (50%) ExcpH5.32	23.6	18.7
TMSRpH3.46 (13%) ExcpH3.46	20.4	25
TMSRpH3.46 (20%) ExcpH3.46	17.6	20.5
TMSRpH3.46 (50%) ExcpH3.46	19.1	25
TMSRpH7 (20%) ExcpH7	19.9	22.2
TMSRpH7 (50%) ExcpH7	21.2	20.2
TMSRpH11 (50%) ExcpH11	22.0	21.6

T = Fabrics ; PM = pre-biomordanted; Exc = coffee extract; ExR = pomegranate extract; SM = unmordanted; QuiACi = chitosan prepared in citric acid (2% w/v) using a padder; QuiACe = chitosan prepared in acetic acid (2% w/v) using a padder; PMR = pre-biomordanted with pomegranate (HT); PMG = pre-biomordanted with guava leaf (HT)

4 CONCLUSIONS

This study successfully demonstrated a sustainable, bio-based approach to dyeing and functionalizing cotton fabrics using coffee husks, pomegranate peels, guava leaves, and chitosan. Acidic media (pH 3.0–5.1) proved ideal for both pre-biomordant and simultaneous biomordant, maximizing polyphenol stability and hydrogen bonding with cellulose. The sample pre-biomordant with guava leaf extract, followed by dyeing with pomegranate extract (TpH3.46ExRPMG), exhibited the highest color intensity (K/S = 6.63). Regarding simultaneous biomordant with coffee extract, the pomegranate peel extract at a 50% concentration (TMSRpH3.46 50%) showed the best performance (K/S = 4.64). The optimized dyeing processes demonstrated excellent domestic wash fastness (grades 4 to 4/5) and

satisfactory perspiration fastness, meeting quality requirements. All optimized samples exhibited a UV protection factor categorized as excellent.

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