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Research Article

# Curcumin Nanoparticles as a Pigment of Polyamide Textiles: Influence of Conditions in the Exhaust Dyeing and Evaluation of Wastewater Toxicity

Katharina S. Moraes, Aline de Oliveira, Bruna T. M. Ferreira, Evandro Bona, Jaqueline Carneiro, Ana Paula Peron, Marta Fernandes, Andrea Zille, Fernanda V. Leimann, Catia R. Lange de Aguiar, Márcia G. da Silva, Alexandre J. S. Ferreira, Nívea T. Vila, and Odinei Hess Gonçalves*

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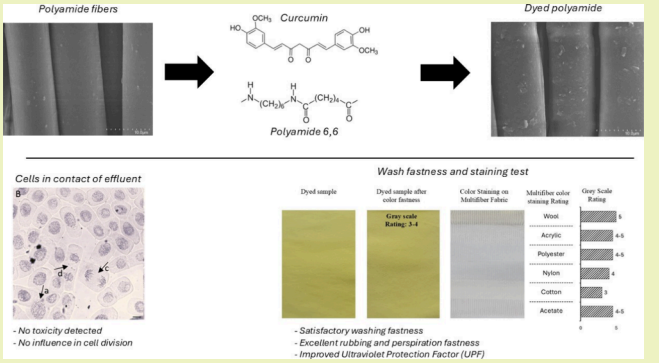
**ABSTRACT:** The growing demand for eco-friendly and sustainable solutions in textile dyeing has attracted interest in natural dyes, since the synthetic counterparts may be potentially damaging to the environment and require the use of additives to efficiently bind them to the fibers. Curcumin, a natural dye derived from *Curcuma longa* L. rhizomes, has attracted attention due to its notable biological activity and vibrant yellow-orange hue; however, its direct application remains limited by challenges related to dye fixation and color fastness. This study aimed to evaluate the use of curcumin nanoparticles in the dyeing of polyamide 6.6, as well as to assess the toxicity level of the effluent generated during the exhaust dyeing process. Colloidally stable nanoparticles with average sizes of 454 and 419 nm were obtained with PVP (polyvinylpyrrolidone) and Poloxamer 407 as the polymeric stabilizer, respectively. A factorial $2^{4-1}$ experimental design was implemented, showing a reduction in color intensity at higher dyeing temperatures, while acidic conditions enhanced dye uptake, leading to improved color depth. Two formulations were selected for a more detailed characterization. Regarding the wash currently used by the textile field, the scores found demonstrated that color did not change during washing. Excellent ratings were found in the rubbing and perspiration fastness and the ultraviolet protection factor for the textiles dyed with PVP and Poloxamer 407 stabilizers. The wastewater of the dyeing process under selected experimental conditions was evaluated with respect to the phytotoxicity, genotoxicity, and cytotoxicity in in vivo models. Based on the results, the wastewater samples analyzed may be environmentally safe.




**KEYWORDS:** *Curcuma longa L., plant-based dyes, clean processes, experimental design, textile effluent, nanotechnology*

## 1. INTRODUCTION

Clothes and other textile fabrics have been dyed with natural colorants for 4000 years; however, the last 150 years have been dominated by synthetically manufactured dyes.[1] Intensive research in synthetic dye technology, coupled with the swift industrialization of textile production, led to nearly complete substitution of natural dyes with synthetic counterparts. Although synthetic dyes have taken precedence, their possible persistence in the environment may lead to contamination of water bodies and soil, resulting in adverse effects on ecosystems and human health. Additionally, the manufacturing and disposal processes of synthetic dyes often emit toxic byproducts, exacerbating air and water pollution.[2] The need for greener textile dyeing processes has been studied over the last years, resulting in a growing demand for natural dyes that are suitable for the textile industry.[3]

Natural dyes from sustainable plant sources are often seen as eco-friendlier and more capable of natural decomposition after disposed.[4] Curcumin, a natural pigment obtained from the turmeric plant (*Curcuma longa* L.), has always attracted considerable interest from the textile industry because of its intense yellow hue. Also, curcumin presented considerable chemical affinity for polymers and has been used as an eco-friendly additive in many fields.[5] The aqueous extract of curcumin was used to dye cotton, wool, polyester, and polyamide fabrics,[6] and the authors have reported high levels of antibacterial and antioxidant activity of the textiles promoted by the curcumin in the surface of the fabrics, but a moderate wash fastness was found. Curcumin extract was

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used as a dyer for cotton and polyester; however, mordants or biomordants are needed, and only moderate gains in color durability have been reported.[7−11]

It was demonstrated that cotton dyeing using curcumin extracts does not present the appropriate fastness and color strength required by industry.[12−14] Also, curcumin's main challenges in terms of application in textiles lie in its poor water solubility[15] and susceptibility to color fading. Nanotechnology has been put to the test to solve a wide range of problems related to the use of natural substances in modern processes, demonstrating that nanostructures such as nanocrystals or nanoparticles may improve stability and dispersibility of biobased biactives.[16,17] Among the variety of techniques to obtain nanostructures, the solid dispersion method is suitable to produce curcumin nanoparticles because the polymer chains act like a stabilizer rather than an encapsulating agent, meaning that the particles are composed mainly of curcumin and not a polymer−curcumin mixture.[15,16,18] Polyvinylpyrrolidone and Poloxamer 407 are excellent choices as polymeric stabilizers for dye nanoparticles because they are readily soluble in water, which may facilitate the interaction of curcumin and the fibers.

Curcumin nanoparticles have already been investigated and may offer benefits to dyeing, such as enhanced dye dispersion and improved color retention.[19] Also, gains in wash fastness and overall stability were reported when curcumin was transformed into colloidally stable nanostructures.[20,21] Han et al.[22] dispersed curcumin using a procedure that is close to a precipitation method by dissolving curcumin in ethanol and then adding this solution to excess water. Results demonstrated that curcumin had a relatively strong affinity to wool, which may be related to the negative charges of curcumin and, therefore, ionic attraction to the positively charged amine groups of wool protein under acidic dyeing conditions.

Toxicity and genotoxicity testing methods play a crucial role in assessing the safety and environmental impact of textile dyes.[3] In fact, Islam and Faqeer[23] have pointed out that toxicity data on natural dyeing compounds are very limited, and further research is needed to elucidate possible toxic effects. There is also a concern about the toxic potential of nanoparticles when they are disposed of in the environment. Acute toxicity testing is one of the widely utilized methods in toxicity assessment, commonly employed for evaluating the adverse impacts of a substance on an organism within a brief time frame.[24] Genotoxicity tests measure the ability of a substance to cause damage to an organism's genetic material, which can lead to mutations and other genetic alterations. They may be used to detect DNA damage, chromosomal aberrations, and induced mutations by textile dyes.[25] The Organization for Economic Cooperation and Development (OECD)[26] cites the species *A. cepa* L. (onion), *Cucumis sativus* L. (cucumber), and *Lycopersicum sculentum* Mill. (tomato), and *Lactuca sativa* L. (lettuce) as excellent test organisms for environmental toxicity analysis, as well as for the evaluation of contaminants in industrial waste. In addition, it is generally accepted that the results obtained using this model are highly similar to those obtained in test systems with other plants and animals in in vivo and in vitro analyses.[27] These experiments may offer an important understanding of the possible mutagenic and carcinogenic characteristics of dyes, aiding in the detection of substances that could present dangers to human health and the ecosystem.

Curcumin is clearly a potential substitute for yellowish hue dyes, and nanotechnology may solve crucial negative aspects, but it is important to understand how operational parameters may affect the dyeing process when curcumin nanoparticles are used. In fact, the temperature and pH may change curcumin keto-enol equilibria and thus compromise curcumin molecular integrity. Also, the stabilizer used to obtain the nanoparticles may interfere with the particle anchoring in the textile fibers. It is also important to determine the toxicity of the dyeing wastewater to assess the sustainability of the process. The main objective of this work was to evaluate the use of curcumin nanoparticles in the exhaust dyeing of polyamide 6.6 textiles. A factorial $2^{4-1}$ experimental design was implemented in order to evaluate the effects of the type of polymeric stabilizer, liquor ratio, temperature, and pH on dyeing capacity and color fastness. The wastewater of the dyeing process of selected experimental conditions was evaluated with respect to the phytotoxicity, genotoxicity, and cytotoxicity in in vivo models.

## 2. MATERIALS AND METHODS

**2.1. Material.** Polyvinylpyrrolidone (PVP, CAS 9003-39-8, 40,000 g $mol^{-1}$, Sigma-Aldrich, 99% purity) and Poloxamer 407 (P407, CAS 9003-11-6, Sigma-Aldrich, 99% purity) were used as polymeric stabilizers. Tween 80 (CAS 9005-65-6, Dinâmica), curcumin (CAS 458-37-7, Sigma-Aldrich, 70% purity), and ethanol (CAS 64-17-5, Vetec, 99.5% purity) were also used to obtain the nanoparticles. Potassium bromide (CAS 7758-02-3, KBr, Sigma-Aldrich, chromatographic grade) was used in the spectroscopic analyses, and ultrapure water was used in the zeta potential and size measurements.

The characteristics of the bleached polyamide 6.6 fabric were as follows: weight per unit area of 135 g $m^{-2}$, warp density of 40 threads per centimeter, and a weft density of 18 threads per centimeter. The pH was adjusted using acetic acid and sodium carbonate. Multifiber DW testing (wool, cotton, acrylic, polyester, polyamide, and acetate fibers) was conducted in accordance with the specifications outlined in ABNT ISO 105 − part F10.[28]

Seeds of *A. cepa*, *C. sativus*, *L. sculentum*, and *L. sativa* were purchased from specialized stores, free of agrochemicals, and with a germination potential above 95%. Mercuric chloride 0.1 wt% aqueous solution ($HgCl_2$, CAS 7487-94-7, Vetec, 99% purity) was used in the sterilization of the seeds. Distilled water was used in the experiments.

**2.2. Nanoparticles Production and Characterization.** The curcumin nanoparticles were obtained using the methodology described by Karavas et al.[29] and Sá et al.[18] with minor modifications. Poloxamer 407 or PVP (3.600 g) and Tween 80 (0.360 g) were solubilized in ethanol (36 mL) with gentle stirring at 25 °C for 5 min. Curcumin (0.360 g) was added to the solution, stirred for 1 min, and then sonicated (Fisher-Scientific, Ultrasonic Dismembrator 120 W, 1/8 in. tip) for 3 min under a pulse regime (30 s on, 10 s off). The ethanol was then evaporated for 18 h at 50 °C in an air circulation oven, and the obtained solid was stored at 10 °C protected from light until use.

The zeta potential was determined using a Stabino Particle Metrix PMX 400 instrument, and samples were diluted to 0.1 g $L^{-1}$. Nanoparticles' average size and polydispersity index were determined in triplicate by dynamic light scattering (DLS) in a NanoDLS Brookhaven equipment (light scattering detector at 90° at 638 nm). The samples were diluted to 0.5 g $L^{-1}$ in ultrapure water before analysis to reduce the level of multiple scattering. The physicochemical properties of the nanoparticles were also characterized by Fourier Transform Infrared spectroscopy (FTIR, Figure S1), X-ray diffractometry (Figure S2), thermogravimetric analyses (TGA, Figure S3), and differential scanning calorimetry (DSC, Figure S4) following the methodologies already described in previous works of our research group[15,16,18] with minor modifications as described in the Supporting Information file.


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**2.3. Experimental Design.** A factorial $2^{4-1}$ experimental design was used to evaluate the influence of four independent variables at two levels (−1 and +1) on the dyeing process: polymeric stabilizer (PVP or Poloxamer 407, $X_1$), temperature ($X_2$, 70 or 100 °C), pH ($X_3$, 3 or 8), and liquor ratio ($X_4$, 20 or 50 wt:vol). The regression coefficients for each response were estimated by using the MATLAB R2022b software. Calculation of the adjusted determination coefficients (adjusted $R^2$) and analysis of variance (ANOVA) were carried out, using the same software, to verify the validity of the obtained models, considering a level of significance of 5%. The dependent variables were the color parameters obtained after the dyeing process and in the wash fastness assay, totaling 12 responses. All experimental conditions were applied in duplicates, and the results in Table 2 are the average of the duplicates.

**2.4. Exhaust Dyeing and Color Properties after Dyeing.** Nanoparticle dye dispersions were prepared by adding 1 g of nanoparticles to 1 L of distilled water. The dispersion process was conducted at 40 °C with continuous stirring for 10 min. In order to dye the polyamide fabrics, different pH values were initially adjusted by using acetic acid and sodium carbonate. Subsequently, the samples were dyed in a Kimak AT1-SW dyeing machine for 60 min under the predetermined conditions of pH, temperature, and liquor ratio as required in the factorial design. Samples were dried for 2 h in an air circulation oven at 40 °C and stored protected from light.

Color parameters of the dried textiles were determined spectrophotometrically in Datacolor 500 equipment located at the Integrated Textile Laboratory/LINTEX (Federal University of Santa Catarina, Brazil) as an average of five measurements in different positions of the textile. The color difference ($\Delta E$) was determined using eq 1 and Chroma (or color intensity, $C^*$) and hue angle ($h°$) were determined using eqs 2 and 3, respectively.

$$\Delta E = \sqrt{(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (1)$$

$$h° = \tan^{-1}\left(\frac{b^*}{a^*}\right) \quad (2)$$

$$C^* = \sqrt{a^{*2} + b^{*2}} \quad (3)$$

Here, $L^*$ is the luminosity, $a^*$ red-green (+ red, − green), and $b^*$ yellow-blue (+ yellow, − blue). $\Delta L$, $\Delta a^*$, and $\Delta b^*$ stand for the differences between color parameters after and before dyeing, and similar differences can also be calculated for Chroma and hue angle ($\Delta C^*$ and $\Delta h°$, respectively).

**2.5. Scanning Electron Microscopy.** Scanning electron microscopy (SEM) was carried out for the polyamide fabrics dyed under the optimized conditions according to the experimental design. The textiles were added onto aluminum stubs by using conductive carbon adhesive tape and coated with a thin layer of Au for 90 s (approximately 7.5 nm) with a sputter coater Quorum Mini QS (London, England). SEM images were taken using a FlexSEM 1000 (Hitachi, Japan) SEM at a voltage of 10 kV.

**2.6. Textile Fastness Testing.** The color fastness of a textile refers to its resistance to change when it is subjected to a specific set of conditions, such as friction, perspiration, or washing. To evaluate color fastness, standardized methods are used, such as those specified in the ISO 105 series.

Color fastness to domestic and commercial laundering, or simply wash fastness, was applied following ISO 105-C06:2006 A1S cycle − Tests for color fastness: Part C-06: Color fastness to domestic and commercial laundering.[30] A sample of the dyed textile was placed in contact with the Multifiber DW fabric (composed of wool, cotton, acrylic, polyester, polyamide, and acetate fibers) that acted as a receptor for any dye that may be released during the washing process. This composite specimen was then laundered, rinsed, and dried. The laundering was carried out under controlled conditions of temperature, alkalinity, bleaching, and abrasive action to achieve a result in a short period.

The wash fastness procedure detailed above was applied to all runs of the experimental design (Table 2) in a Kimak AT1-SW dyeing machine and the change in CIELAB color parameters was determined (Datacolor 500 equipment) for the dried textiles (2 h in an air circulation oven at 40 °C) using eqs 1−3 (comparing the dyed polyamide textiles before and after the test) as an average of five measurements in different positions of the textile. Color transfer (staining) was evaluated by determining the color parameters using eqs 1 and 3 and comparing the Multifiber DW sample before and after the test.

Later, the polyamide fabrics were dyed under optimized conditions according to the experimental design, and then color fastness tests (wash, rubbing, and perspiration) and also the ultraviolet protection factor (UPF) test were carried out for the dried textiles. In these tests, the quantitative evaluation was carried out using the gray scale rating as described in the standard procedure.[31] The gray scale for assessing color change consists of nine pairs of gray chips, ranging from grade 5 (no change) to grade 1 (severe change). The gray scale for assessing staining consists of nine pairs of gray chips, ranging from grade 5 (no staining) to grade 1 (severe staining), and a higher rating indicates better color fastness.

The acidic and alkaline perspiration fastness was performed according to the standard EN ISO 105-E04[32] with standard chemicals and apparatus. The color change and the staining on the adjacent Multifiber fabric were classified visually according to the grayscale (1−5) in a VeriVide D65 (UK) light cabinet under the artificial daylight illuminant D65.

The rubbing fastness test was conducted according to standard EN ISO 105-X12[33] in a Roaches crockmeter (Roaches International, Birstall, UK). The polyamide fabrics were rubbed against a wet and a dry cotton fabric with ten forward and ten backward movements. The color changes in shade and the staining on the adjacent white fabric were graded using the grayscale (1−5).

The AU/NZS 4399[34] standard assesses the sun protection offered by fabrics through the UPF determination (eq 4).

$$\mathrm{UPF} = \frac{\sum_{290}^{400} E\lambda S\lambda \Delta\lambda}{\sum_{290}^{400} E\lambda T\lambda \Delta\lambda} \quad (4)$$

Here, $E\lambda$ is the relative erythemal spectral efficacy (W m$^{-2}$ nm$^{-1}$), $S\lambda$ is the solar spectral irradiance (Melbourne), $\Delta\lambda$ corresponds to the measured wavelength range (nm), and $T\lambda$ is the spectral transmittance of the sample (%). The UV−vis spectrophotometer (Shimadzu UV 2600 at the Centre for Textile Science and Technology, Portugal) was used to measure the transmittance of the fabrics in the range of 290−400 nm. Results were categorized into three ranges: Low (15−24), good (25−39), and excellent (40−50+).

**2.7. Phytotoxicity, Cytotoxicity, and Genotoxicity Assessment of the Dyeing Wastewater.** The wastewater generated in the dyeing process under the optimized conditions was collected and evaluated regarding in vivo toxicity models as follows.

*2.7.1. Phytotoxic Potential.* To assess phytotoxic potential, the protocol of OECD[26] was adopted. Seeds of similar sizes of *A. cepa*, *C. sativus*, *L. sculentum*, and *L. sativa* were separated from each species. For every treatment, five repetitions for each species were made, totaling 100 seeds for each concentration tested and for the control (using distilled water). After dropping 1 mL of their respective treatment into the dishes, they were wrapped in plastic film and placed in a closed chamber at 25 °C, protected from light, for 5 days. A seed was considered germinated when the radicle protruded. The germination percentage ($G$, %) was determined as follows (eq 5):

$$G\ (\%): \frac{\text{number of seeds germinated}}{\text{number of seeds evaluated}} \times 100 \quad (5)$$

After 5 days, 10 roots of each dish were measured using a digital caliper to determine the relative growth index (RGI) and the germination index (GI%) based on Biruk et al.[35] (eqs 6 and 7, respectively).

$$\mathrm{RGI} = \frac{\mathrm{RLI}}{\mathrm{RLC}} \quad (6)$$



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$$\text{GI}\ (\%) = \frac{\text{RLI} \times \text{GSI}}{\text{RLC} \times \text{GSC}} \times 100 \quad (7)$$

Here, RLI is the average length of roots exposed to wastewater, RLC is the average length of roots of the control, GSI is the number of germinated seeds resulting from exposure to wastewater, and GSC is the number of germinated seeds of the control. Three classifications were considered for RGI: inhibition of root growth: 0.1< RGI < 0.8; no effect: 0.8 ≤ RGI ≤ 1.2; and stimulation of root elongation: RGI > 1.2.

*2.7.2. Cytotoxicity and Genotoxicity Assessments.* Potentials for cytotoxicity and genotoxicity were determined according to Filipi et al.[36] using the Mitotic Index (MI) and Cellular Alteration Index (CAI).

Bulbs of *A. cepa* used in our experiments came from a store specializing in the sale of organic foods. Initially, dehydrated cataphylls were removed, and the bulbs were washed with deionized water. Soon after, bulbs were placed in contact with the treatments (control with distilled water or concentrations of nanoparticles) and allowed for root growth for 5 days. To evaluate each treatment, five repetitions of bulbs were used. The roots collected from the bulbs were placed into 3:1 Carnoy fixative (methanol: acetic acid) for 12 h. For slide mounting, the meristematic regions of the roots were crushed and stained with 2% acetic orcein. These slides were analyzed with an optical microscope. MI (eq 8) was established for each treatment, obtained by counting 2000 cells per bulb and 10,000 per treatment.

$$\text{MI:}\ \frac{\text{Total number of dividing cells}}{\text{Total number of cells analyzed}} \times 100 \quad (8)$$

IAC (eq 9) was calculated to check for the genotoxic potential of the dyeing wastewater. 1000 cells were analyzed per treatment (200 cells per bulb). The cellular alterations considered were micronuclei, sticky chromosomes, chromosome disorganization at the different stages of mitosis (prophase, metaphase, anaphase, and telophase), chromosome breaks and bridges, and polyploidy.

$$\text{CAI:}\ \frac{\text{Number of cell changes}}{1000} \times 100 \quad (9)$$

As a complement to the analysis of cytotoxicity and genotoxicity potential, the presence of hook-shaped roots and changes in the thickness and color of the roots were observed.

**2.8. Statistical Analysis.** Average size, the polydispersity index, and the zeta potential were evaluated by the one-way ANOVA test with the level of significance of $p < 0.05$ and are presented along with their standard deviation. The toxicity effects of the wastewater were analyzed using Kruskal–Wallis analysis of variance, followed by Dunn's test ($p \leq 0.05$), because the Lilliefors test evidenced that the data were non-normal. The UPF test was evaluated using the Tukey test, considering the level of significance of $p < 0.05$. The statistical tests used in the experimental design were described in detail in Section 2.3.

## 3. RESULTS AND DISCUSSION

**3.1. Nanoparticles Characterization.** Table 1 presents the average size, the polydispersity index, and the zeta potential of the curcumin nanoparticles stabilized with PVP and Poloxamer 407. Supporting Information presents the physicochemical analyses of the curcumin nanoparticles: FTIR (Figure S1), X-ray diffraction (Figure S2), TGA (Figure S3), and DSC (Figure S4).

Curcumin particles presented sizes in the nanometric range and moderately polydisperse samples.[37] Zeta potential values indicated that particles presented low stability and thus they may be prone to coagulate or destabilize over time. No statistically significant difference was found between the nanoparticles produced with PVP or Poloxamer 407 as the stabilizer ($p < 0.05$).

**Table 1. Average Size, the Polydispersity Index, and the Zeta Potential of the Curcumin Nanoparticles Stabilized with PVP and Poloxamer 407**

| | PVP-curcumin nanoparticles[a] | P407-curcumin nanoparticles[a] |
|---|---|---|
| average size (nm) | 454 ± 17 | 429 ± 44 |
| polydispersity index (−) | 0.3 ± 0.1 | 0.4 ± 0.1 |
| zeta potential (mV) | −32 ± 3 | −29 ± 4 |

[a]Value ± standard deviation.

Taking into account the results above and those presented in the Supporting Information, the physicochemical characterization of the nanoparticles strongly suggested interactions between curcumin and PVP formed during solid dispersion, likely through hydrogen bonding, leading to the inclusion of curcumin within the polymer matrix. They also indicated that curcumin was converted from a crystalline to an amorphous form in the solid dispersions obtained with both PVP and Poloxamer 407. The morphological and physicochemical characterization strongly indicated that a colloidal dispersion of curcumin nanoparticles would form when the solid dispersion was added to water, as the polymers dissolved completely and released the nanometric particles into the medium.

**3.2. Experimental Design.** Table 2 presents the levels of independent variables for each experiment and the responses obtained for different experimental conditions. Figure 1 presents the results of the dyeing of the polyamide textiles, showing the absolute values of the normalized effects on color difference ($\Delta E$) and on the variations of $L^*$, $a^*$, $b^*$, $C^*$, and $h^\circ$. Figure 2 presents the results of the wash fastness showing the absolute values of the normalized effects color difference ($\Delta E$) and the variations of $L^*$, $a^*$, $b^*$, $C^*$, and $h^\circ$. It is worth noting that the variation values ($\Delta$) account for the differences between the values after and before the fastness test.

When discussing these results, it is worth pointing out that the polyamide textile presented the following color parameters before dyeing: $L^* = 92.85$, $a^* = -1.31$, $b^* = 2.69$, $C^* = 2.99$, and $h^\circ = 116^\circ$.

Regarding dyeing capacity, a remarkable increase in color difference was found for all experimental points, meaning that the dyeing procedure was successful in coloring the textile. This may be attributed to the high surface area of such small particles deposited on the fiber, and that a small amount of nanopigment dispersions is usually required to attain a comparable color value in comparison with conventional dispersion.[38] This may also indicate the presence of the nanoparticles on the textile's surface. The luminosity variation ($\Delta L^*$) presented negative values, meaning that a small yet significant ($p = 0.0090$) decrease in luminosity was detected. Dyed textiles presented more negative values of $a^*$, denoting a tendency to green, while a remarkable increase in $b^*$ was found, demonstrating again the dyeing capacity of curcumin nanoparticles. The use of PVP as an encapsulant led to a statistically lower luminosity gain ($\Delta L^*$) when compared to Poloxamer 407 ($p = 0.0061$). The temperature had a significant effect ($p = 0.0139$) as an increase in temperature caused a reduction in color difference ($\Delta E$), which was indirectly caused by the effect of temperature on $\Delta b^*$ ($p <$ 0,0001) and also a decrease in $L^*$. $\Delta a^*$ slightly decreased when the dyeing process was conducted in pH 8, when compared to


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Table 2. Factorial $2^{4-1}$ Experimental Design Runs with Coded/decoded Independent Variables and the Responses (Polyamide Values before Dyeing: $L^* = 92.85$, $a^* = -1.31$, $b^* = 2.69$, $C^* = 2.99$, and $h^\circ = 116^\circ$)

| | | | | | dyeing capacity[a] | | | | | wash fastness[b] | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| run | $X_1$ polymeric stabilizer | $X_2$ T (°C) | $X_3$ pH | $X_4$ liquor ratio (wt:vol) | $\Delta L^*$ | $\Delta a^*$ | $\Delta b^*$ | $\Delta C^*$ | $\Delta h^*$ | $\Delta E$ | $\Delta L^*$ | $\Delta a^*$ | $\Delta b^*$ | $\Delta C^*$ | $\Delta h^*$ | $\Delta E$ |
| 1 | −1 (P407) | −1 (70) | −1 (3) | −1 (1:20) | −4.41 | −6.18 | 80.16 | 80.20 | −20.84 | 80.52 | 0.53 | 1.61 | −7.46 | −2.38 | 0.50 | 2.54 |
| 2 | +1 (PVP) | −1 (70) | −1 (3) | +1 (1:50) | −1.36 | −8.20 | 80.29 | 80.53 | −19.46 | 80.71 | −1.82 | 1.69 | −9.17 | −2.92 | 0.34 | 3.48 |
| 3 | −1 (P407) | +1 (100) | −1 (3) | +1 (1:50) | −5.96 | −5.83 | 69.91 | 69.96 | −20.38 | 70.41 | −0.63 | 1.87 | −6.97 | −2.36 | 0.66 | 2.54 |
| 4 | +1 (PVP) | +1 (100) | −1 (3) | −1 (1:20) | −4.04 | −6.52 | 67.61 | 67.76 | −19.63 | 68.04 | −0.59 | 1.59 | −6.55 | −2.24 | 0.49 | 2.39 |
| 5 | −1 (P407) | −1 (70) | +1 (8) | +1 (1:50) | −4.58 | −4.97 | 79.38 | 79.32 | −21.62 | 79.67 | −0.51 | 1.19 | −7.02 | −2.23 | 0.47 | 2.41 |
| 6 | +1 (PVP) | −1 (70) | +1 (8) | −1 (1:20) | −4.10 | −7.23 | 75.89 | 76.05 | −19.80 | 76.34 | −0.37 | 1.21 | −3.90 | −1.29 | 0.41 | 1.47 |
| 7 | −1 (P407) | +1 (100) | +1 (8) | −1 (1:20) | −5.67 | −5.21 | 69.08 | 69.07 | −20.81 | 69.50 | −0.68 | 1.18 | −5.33 | −1.80 | 0.39 | 1.97 |
| 8 | +1 (PVP) | +1 (100) | +1 (8) | +1 (1:50) | −4.50 | −7.03 | 68.12 | 68.30 | −19.29 | 68.62 | −0.23 | 1.09 | −5.54 | −1.88 | 0.24 | 1.91 |

[a]Differences (Δ) were found for the color parameters before and after dyeing. [b]Differences (Δ) were found for the color parameters before and after the wash fastness test.

pH 3 ($p = 0.0795$), and the use of PVP as an encapsulant led to a statistically higher $\Delta a^*$ when compared to Poloxamer 407 ($p = 0{,}0001$). Chroma ($C^*$) or color intensity or saturation was also negatively impacted by the increase in the temperature ($p < 0{,}0001$). A significant decrease in hue was found when PVP was used as encapsulant ($p < 0{,}0001$).

Regarding the wash fastness test, the increase in pH led to a slight but significant decrease in both $\Delta a^*$ ($p = 0.0384$) and, consequently, $\Delta E$ ($p = 0{,}0001$). Values of $a^*$ were more negative after washing, meaning that the textiles presented a small tendency to green. The values of luminosity variation in wash fastness found were close to zero, meaning that no appreciable change was caused by the washing process. Within the experimental range evaluated, only a small yet significant ($p = 0.0542$) effect was found on $b^*$, which stands for the yellow tendency as well as on color intensity ($C^*$, $p = 0.0434$). Remarkably, changes in hue angle were not detected. These results strongly demonstrated that the dyeing process using curcumin nanoparticles was robust and that curcumin was strongly attached to the textile fibers.

Table S1 and Figures S5−S9 (Supporting Information) present the results of the staining test on wool, cotton, polyamide, polyacetate, polyester, and acrylic textiles. Based on the staining evaluation conducted across different textile fibers, cotton, wool, and polyamide exhibited the highest overall $\Delta E$ values, suggesting a greater susceptibility to staining by curcumin nanoparticles. In contrast, polyacetate and polyester demonstrated significantly lower color changes, indicating higher resistance to staining under the tested conditions. It is worth noting that wool showed consistently elevated $\Delta E$ values with relatively low variability between experimental runs, reflecting a stable interaction with the dye. Wool and polyamide, on the other hand, presented higher variability in $\Delta E$ values, indicating that their interaction with the dye may be more sensitive to changes in the experimental parameters.

Schmidt et al.[39] dyed polyamide and cotton using a *C. longa* L. ethanolic extract in an exhaust process similar to the one applied here. The dye uptake from the solutions was higher for polyamide than cotton, indicating that curcumin interacted with a positively charged, protonated polyamide chain. Washing fastness presented a low gray rating, suggesting low binding with the fibers. They demonstrated that curcumin uptake was positively influenced by temperature, the same trend observed by Yin and Wang.[40] This could indicate that the lower dyeing capacity found here may be due to other factors, such as curcumin degradation. Mirjalili et al.[41] indirectly investigated the fixation of curcumin using the turmeric powder directly in the dyeing bath and carried out the washing fastness test to determine the antibacterial activity of the textile after several washing cycles. They found a sharp loss of antibacterial activity after the first wash and a decrease of approximately 75% in the activity after 20 cycles, but only a small loss was detected when mordants were used, indicating that the dyeing method was not efficient in binding curcumin onto polyamide fibers.

**3.3. Proposed Mechanism of Dyeing Polyamide Using Curcumin Nanoparticles.** The distinction between "dyes" and "pigments" is a fundamental principle in textile chemistry and dyeing technology, primarily based on solubility and application mechanism. Dyes are typically colored organic compounds that are water-soluble and that absorb into the pores of the fiber, where they bond with polymer molecules. Pigments are insoluble, particulate solids that are applied as a


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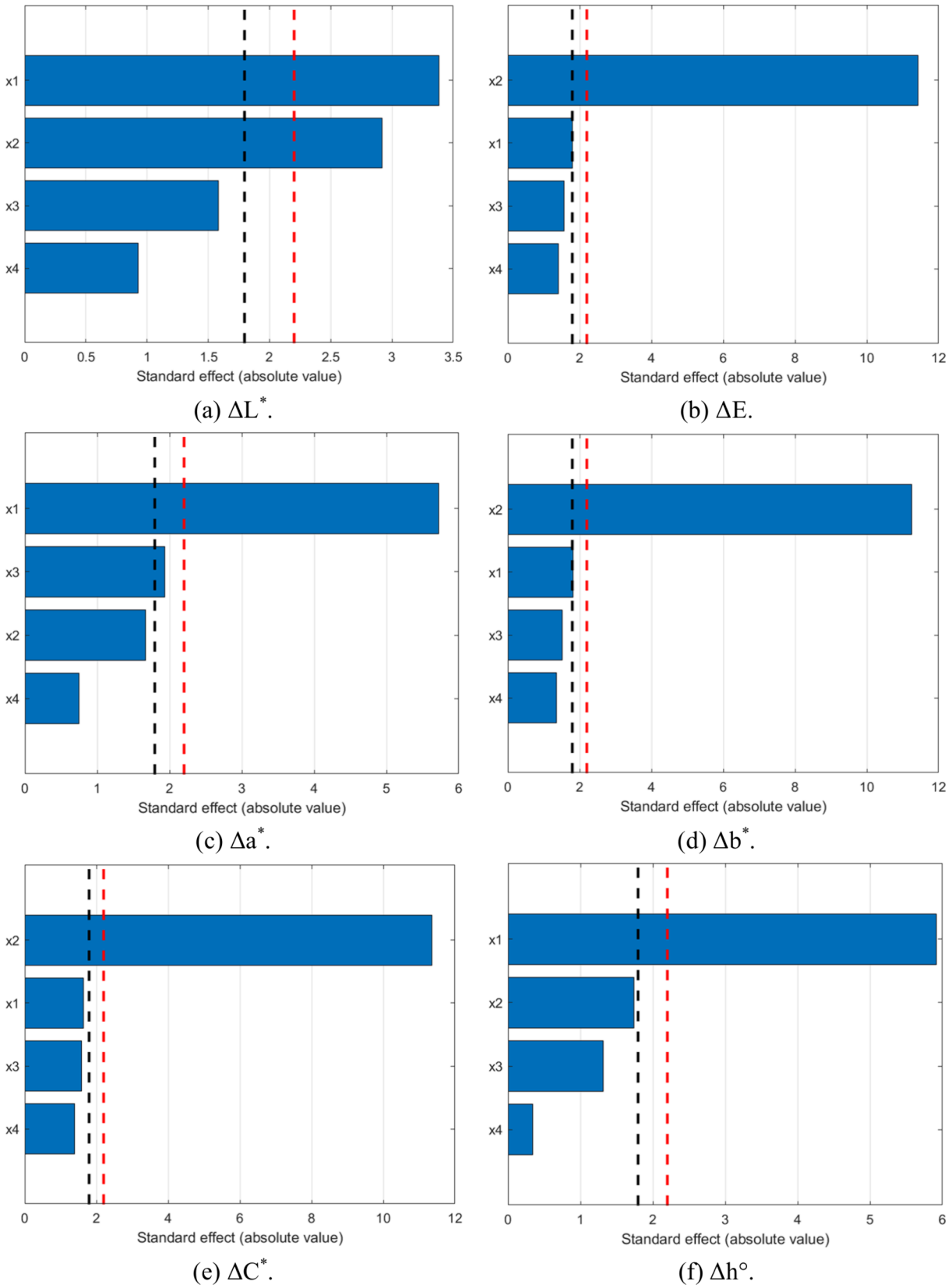


**Figure 1.** Absolute values of the normalized effects of $X_1$ (encapsulant), $X_2$ (temperature), $X_3$ (pH), and $X_4$ (liquor ratio) on color difference ($\Delta E$) and on the variations of luminosity, $a^*$, $b^*$, Chroma or color intensity ($C^*$), and hue angle ($h°$). Effects are statistically significant at $p < 0.05$ (red lines) and $p < 0.1$ (black lines). Differences ($\Delta$) were found for the color parameters before and after dyeing.

dispersion and fixed to the textile surface with a binder since they lack a direct chemical affinity for the fiber.

Curcumin is inherently insoluble in water in its native crystalline form, which aligns it more closely with the physical



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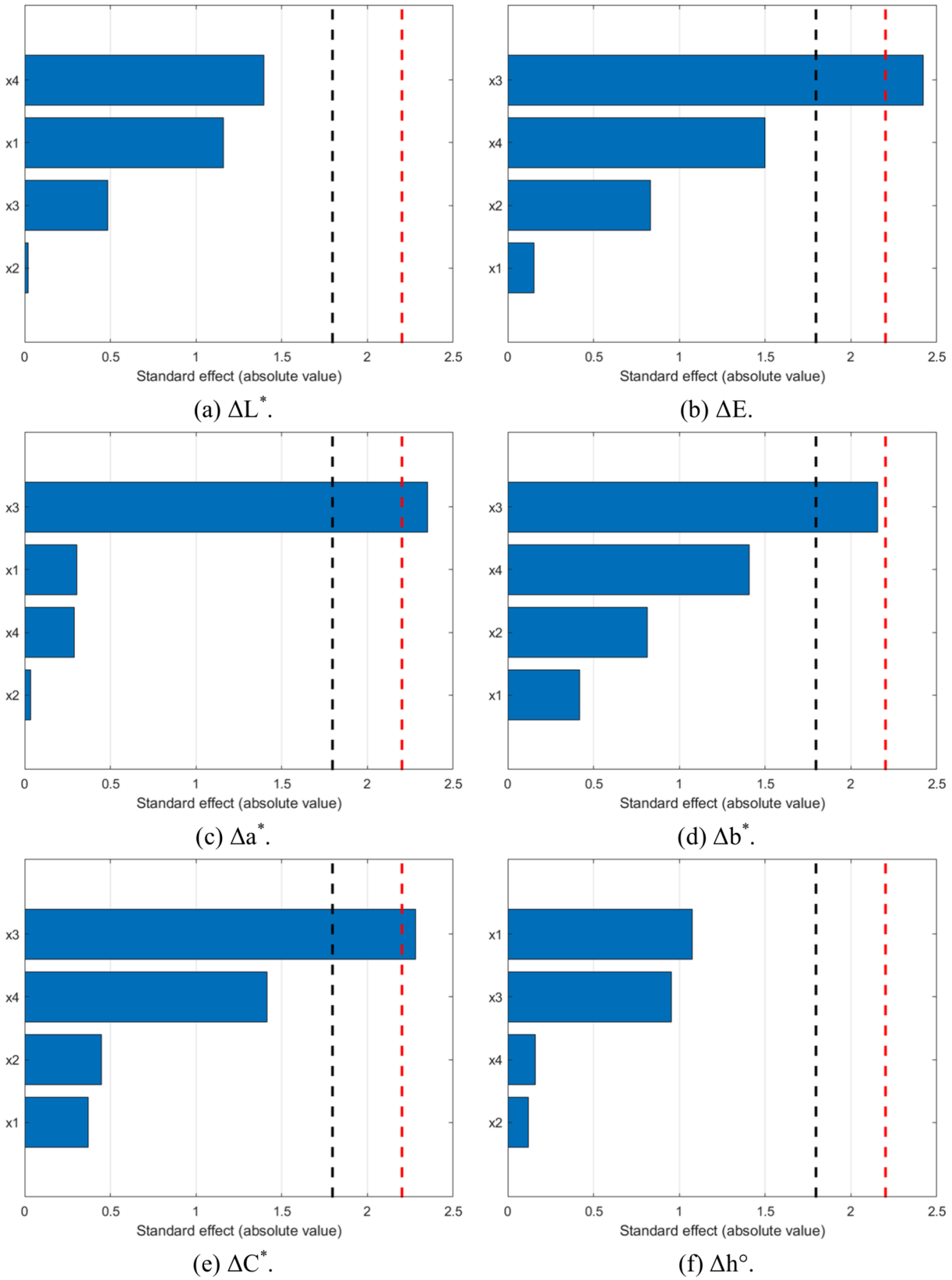


**Figure 2.** Results of the wash fastness test with the absolute values of the normalized effects of $X_1$ (encapsulant), $X_2$ (temperature), $X_3$ (pH), and $X_4$ (liquor ratio) on color difference ($\Delta E$) and on the variations of luminosity, $a^*$, $b^*$, Chroma or color intensity ($C^*$) and hue angle ($h°$). The black dashed line is the $t_{critic}$ for alpha = 10% and the red dashed line is the $t_{critic}$ for alpha = 5%. Differences ($\Delta$) were found for the color parameters before and after the wash fastness test.

properties of a pigment. Taking into consideration the most accepted definition used in textile dyeing technology, the transformation of curcumin into nanoparticles creates a nanopigment. Unlike conventional pigments, the reduced



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size and large surface area of these nanoparticles allow them to be dispersed in an aqueous solution,[38] and the results showed that they were successfully bonded to the polyamide fibers via an exhaust dyeing process.[7−9]

It is worth pointing out that curcumin is a polyphenol that presents an enol form at high pH, while the keto form is prevalent in acidic medium.[9,42] The experimental design revealed that optimal dyeing results, indicated by improved dyeing capacity, were achieved under acidic conditions (pH 3) and at 70 °C. This is consistent with the known dyeing mechanism of polyamide fibers, which are typically dyed with acid dyes.[39] Under acidic conditions, the amino groups within the polyamide molecular chain become protonated, creating positively charged dye sites.

The proposed mechanism of exhaust dyeing using insoluble curcumin nanoparticles on polyamide may involve the following aspects:

*Electrostatic attraction and adsorption:* As observed with other insoluble nanoparticles, the negatively charged curcumin nanoparticles (zeta potential around −30 mV) are attracted to the positively charged amino groups of the polyamide fibers in the acidic dye bath (pH 3). This strong electrostatic interaction facilitates the adsorption of the nanoparticles onto the fiber surface.

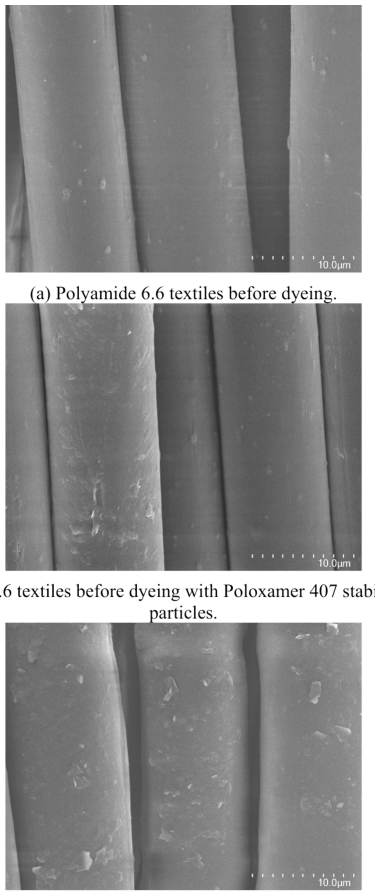
*Fiber swelling and entrapment:* polymer swelling caused by heat may allow the nanoscale curcumin particles, along with their polymeric stabilizers, to penetrate deeper into the fiber matrix, rather than merely adhering to the surface. This phenomenon has been observed with other insoluble nanopigments during exhaustion dyeing.

*Role of polymeric stabilizers:* The polymeric stabilizers (PVP and Poloxamer 407) play a crucial role beyond just maintaining nanoparticles dispersion. Their interaction with curcumin by hydrogen bonding might also facilitate anchoring of the nanoparticles within the polyamide fiber structure. These polymers could act as a bridge, enhancing the affinity of the curcumin nanoparticles for the fiber or aiding in their mechanical entrapment within the swollen fiber matrix.

The combination of strong adsorption, potential penetration, and entrapment within the fiber structure, facilitated by the optimized dyeing conditions and the polymeric stabilizers, leads to a more robust and durable fixation of the curcumin nanoparticles compared to that of conventional surface-bound pigments. This enhanced integration into the fiber matrix may be the underlying reason for the observed improvements in wash fastness and overall stability, effectively mimicking the performance characteristics traditionally associated with molecularly diffused dyes.

**3.4. Textiles Characterization.** A detailed characterization of the dyed polyamide textiles was performed under two selected dyeing conditions, chosen based on the influence of experimental parameters on dyeing performance and wash fastness, as identified in the experimental design. Instead of using the $\Delta E$ values from Table 2, the statistical data generated by the experimental design (Figures 1, 2, and S5−S9 in the Supporting Information) were employed, since this approach accounts for factor interactions and provides a more precise analysis. Considering these criteria, the best results were found in acidic conditions (pH 3) and at a lower temperature (70 °C). Besides, liquor ratio ($X_4$) did not significantly influence dyeing. The dyed textiles obtained in runs 1 (Poloxamer 407 as a polymeric stabilizer) and 2 (PVP as a polymeric stabilizer) (Table 2) were then selected for further characterization.

Figure 3 presents the SEM images of the (a) polyamide textiles before dyeing and the textiles dyed with (b) Poloxamer 407-stabilized particles and (c) PVP-stabilized particles. Figure 4 presents the washing fastness results of the textiles dyed with curcumin nanoparticles produced with PVP and Poloxamer 407 as stabilizers. The rubbing fastness, and perspiration fastness, and the UPF of the polyamide samples dyed with curcumin nanoparticles are shown in Table 3.




(a) Polyamide 6.6 textiles before dyeing.

(b) Polyamide 6.6 textiles before dyeing with Poloxamer 407 stabilized curcumin particles.

(c) Polyamide 6.6 textiles before dyeing with PVP stabilized curcumin particles.

**Figure 3.** Scanning electron microscopy images of the polyamide 6.6 textiles (a) before and after dyeing with (b) Poloxamer 407 and (c) PVP-stabilized curcumin particles. (Magnification 10,000×).

SEM images of the polyamide fibers prior to dyeing revealed a smooth and uniform surface typical of synthetic fibers. After dyeing, clusters were observed on the fiber surfaces, likely resulting from the deposition of curcumin nanoparticles. As shown in Figure 3, the dye appeared to be evenly distributed across the textile surface, indicating a visually acceptable degree of levelness.

Washing fastness scores showed comparable statistical levels for textiles dyed with nanoparticles stabilized by either PVP or Poloxamer 407, with ratings ranging from moderate to excellent. This indicates that the choice of stabilizer did not significantly affect the binding of nanoparticles to the fibers, corroborating the experimental design results. However, PVP-stabilized nanoparticles exhibited slightly lower transfer ratings on both cotton and polyamide fibers, with fastness values between "slightly fair" and "good." Overall, curcumin nanoparticles stabilized with Poloxamer 407 conferred greater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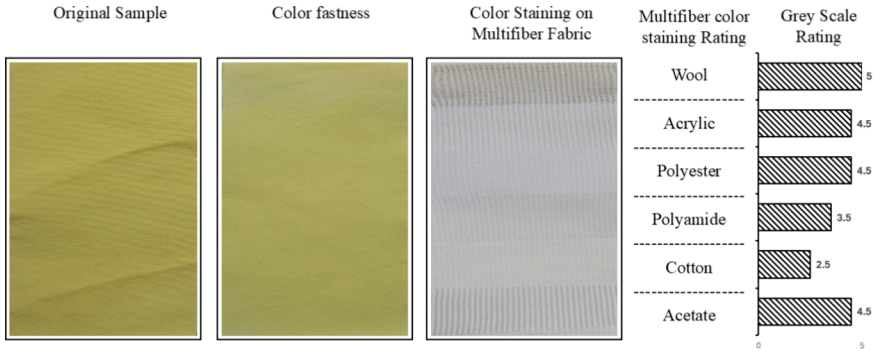
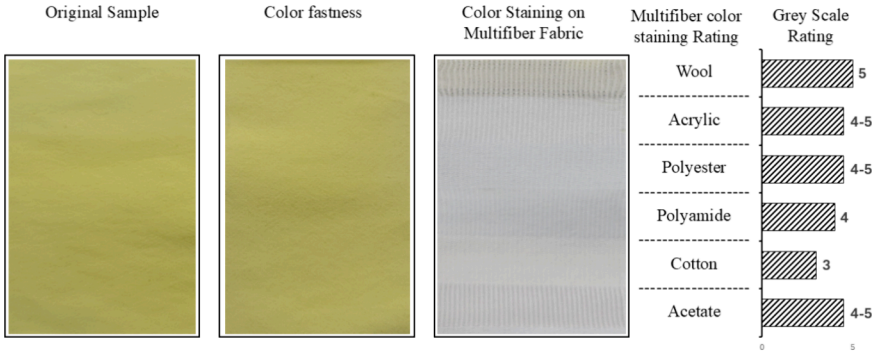



(a) PVP as stabilizer.




(b) P407 as stabilizer.

**Figure 4.** Washing fastness of the textiles dyed with curcumin nanoparticles produced with PVP and Poloxamer 407 as stabilizers.

**Table 3. Rubbing and Perspiration Fastness and Ultraviolet Protection Factor (UPF) of the Textiles Dyed with Curcumin Nanoparticles Produced with PVP and Poloxamer 407 as Stabilizers**[a]

| | rubbing fastness | | perspiration fastness | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | | staining on adjacent fibers[b] | | | | | | UPF | |
| sample | dry | wet | color change | wool | acrylic | polyester | polyamide | cotton | acetate | UPF | UV protection category |
| run 1 (P407 as stabilizer) | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 105.6 ± 7.7[b] | excellent |
| run 2 (PVP as stabilizer) | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 5 | 104.8 ± 6.2[b] | excellent |
| textile before dyeing | | | | | | | | | | 80.7 ± 6.1[a] | excellent |

[a]Different letters in a row indicate a statistically significant difference in the Tukey test ($p < 0.05$). [b]WO: wool; PAC: acrylic; PES: polyester; PA: polyamide; CO: cotton; CA: acetate.

resistance to color loss during washing, making this stabilizer the preferred option for applications requiring high durability.

Rubbing fastness results were found to be excellent for both stabilizers. This uniformity indicated that both P407 and PVP did not interfere with the penetration of the dye into the fibers, but rather merely coated the surface.[6] This deep penetration is crucial as it enhances the durability of the dye under mechanical stress, such as rubbing, which is a common occurrence in the everyday use of textile products.

The sweat fastness results were classified as "excellent," revealing that the color resistance of the treated fabrics remained unaffected by the pH of the sweat. The absence of color change in the samples and the lack of color transfer to adjacent fabrics further reinforce the reliability and effectiveness of curcumin nanoparticles in preserving color under varied environmental conditions.

The transformation of curcumin into colloidally stable nanostructures, as achieved with PVP and Poloxamer 407 stabilizers, directly addressed the solubility and stability issues of native curcumin. This nanosizing approach has been shown to improve dye dispersion and enhance color retention. The wash fastness, rubbing fastness, and perspiration fastness results (Table 3 and Figure 4) demonstrated significant improvements compared with conventional curcumin dyeing, aligning with previous reports of gains in wash fastness and overall stability when curcumin is nanostructured. This enhanced fastness is a critical improvement for textile applications, as it ensures the durability and longevity of the dyed fabric. Nanoparticles, such as carbon black and zinc oxide, have been shown to achieve good to very good light and washing fastness (gray rating 4−5) when applied to textiles, supporting the potential for similar improvements with curcumin nanoparticles.[43]

Regarding UPF, there was a significant increase in UV protection for the dyed samples, with both samples showing an increase of more than 25% compared to the undyed fabric ($p <$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0.05). The difference between the dyeing processes was not statistically significant, confirming that both dyeing methods were effective in enhancing the UPF of the fabric. Several studies in the literature support our findings regarding the use of curcumin as a dye. Silk samples dyed with curcumin exhibited effective ultraviolet protection, achieving UPF values up to 88%, depending on the concentration of curcumin used during dyeing.[21] Botalo et al.[44] prepared various edible films, including alginate-only, alginate with whey protein, and alginate with whey protein isolate, along with two different concentrations of curcumin. Results indicated that incorporating whey protein and curcumin reduced film transparency while increasing the hydrophobicity, antioxidant activity, and UV-blocking efficiency.

Nanoparticles were highly effective UV blockers, probably due to their small size and large surface area, which allowed them to scatter and absorb UV radiation efficiently. Therefore, the incorporation of curcumin nanoparticles was expected to impart significant UV protective properties to the polyamide fabric, adding a valuable functional benefit beyond mere coloration. This multifunctional aspect, where the colorant also provides protective features, represents a key advantage of nanotechnology in textile applications.[38]

**3.5. Phytotoxicity, Cytotoxicity, and Genotoxicity Assessment of the Dyeing Wastewater.** Table 4 presents the phytotoxic potential of wastewater in *A. cepa* L., *C. sativus* L., *Lycopersicum esculentum* Mill., and *L. sativa* L. Table 5 shows the mitotic indices and cell change index in root meristems of *A. cepa* L. bulbs, while the meristematic cells from *A. cepa* L. bulb roots are shown in Figure 5 (the letters inside the image indicate the different cell division stages found: a, prophase; b, metaphase; c, anaphase; d, telophase).

**Table 4. Phytotoxic potential of wastewater in *Allium cepa* L., *Cucumis sativus* L., *Lycopersicum esculentum* Mill., and *Lactuca sativa* L. based on Germination (*G*), Relative Growth Index (RGI), and Germination Index (GI)[a]**

| | *G* (%) | RGI (−) | GI (%) |
|---|---|---|---|
| *Allium cepa* L. | | | |
| control | 100 | 1.00 | 100 |
| wastewater from P407-stabilized nanoparticles dyeing | 98 | 0.90 | 88.5 |
| wastewater from PVP-stabilized nanoparticles dyeing | 97 | 0.86 | 83.1 |
| *Cucumis sativus* L. | | | |
| control | 100 | 1 | 100 |
| wastewater from P407-stabilized nanoparticles dyeing | 100 | 0.80 | 80.3 |
| wastewater from PVP-stabilized nanoparticles dyeing | 100 | 0.89 | 88.8 |
| *Lycopersicum esculentum* Mill. | | | |
| control (distilled water) | 100 | 1 | 100 |
| wastewater from P407-stabilized nanoparticles dyeing | 93 | 0.87 | 81.5 |
| wastewater from PVP-stabilized nanoparticles dyeing | 94 | 0.90 | 84.1 |
| *Lactuca sativa* L. | | | |
| control (distilled water) | 100 | 1 | 100 |
| wastewater from P407-stabilized nanoparticles dyeing | 100 | 0.84 | 84.3 |
| wastewater from PVP-stabilized nanoparticles dyeing | 95 | 0.86 | 8.14 |

[a]Significant differences between concentrations and their respective controls, according to Kruskal−Wallis H followed by Dunn's post hoc test ($p \leq 0.05$).

**Table 5. Mitotic Indices and Cell Change Index in Root Meristems of *Allium cepa* L. Bulbs Exposed to Different Concentrations of Avobenzone for 120 h[a]**

| | MI (%) | CCI (%) |
|---|---|---|
| control | 100.00 ± 0.9 | 0.9 ± 0.5 |
| wastewater from PVP-stabilized nanoparticles dyeing | 90.7 ± 0.9 | 0.8 ± 0.4 |
| wastewater from P407-stabilized nanoparticles dyeing | 92.4 ± 1.0 | 0.7 ± 0.6 |

[a]For MI, data are expressed as a percentage of control values. Significantly different from the control group (distilled water), according to Kruskal−Wallis H followed by Dunn's post hoc test ($p \leq 0.05$).

Curcumin is known to be nontoxic, and even the chemicals (PVP, Poloxamer 407, and Tween 80) applied in this work present no toxicity at the concentrations used. The question of toxicity was raised mainly due to the nanoparticulate characteristic of curcumin. In fact, there is concern in academia and society about the possible toxicity of nanoparticles and how they interact with living organisms.

In Table 4, considering the four plant species assessed, it was verified that exposure to the wastewater samples did not result in significant impairment of seed germination (*G* (%) > 90%) or radicle elongation (RGI > 80%). These findings were statistically supported by the Kruskal−Wallis H test followed by Dunn's post hoc test ($p \leq 0.05$). The GI values (GI (%) > 80%) indicate the absence of phytotoxic potential,[45] thereby demonstrating that the initial developmental processes of the tested plants remain unaffected under the experimental conditions.

Additionally, when compared to the control group, the wastewater did not induce measurable alterations in cellular proliferation (MI > 90%) nor in chromosomal organization and dynamics during mitotic activity (CCI < 1%) in the root meristematic cells of *A. cepa* (Table 5), as confirmed by the same nonparametric statistical approach. Microscopic analyses (Figure 5) revealed that meristematic cells exposed to the wastewater progressed through the canonical phases of mitosis, exhibiting structural and functional characteristics indistinguishable from those observed in untreated controls. Such results strongly suggest that the cellular machinery involved in DNA replication, chromosome segregation, and cytokinesis was not perturbed by the wastewater samples.

The absence of phytotoxic manifestations in cucumber, tomato, and lettuce seedlings (Table 4) is consistent with the low frequency of mitotic and chromosomal abnormalities detected in onion root tips (Table 5). This coherence across bioassays reinforces the interpretation that wastewater samples did not compromise radicle development (Table 4), a critical parameter that integrates seedling vigor, cell division regularity, and tissue differentiation capacity.

The employment of nanoparticles derived from natural compounds, such as curcumin, has been increasingly proposed as a sustainable alternative for textile dyeing, given their reduced ecological burden when compared to conventional synthetic dyes. Nonetheless, as highlighted earlier, the environmental toxicity of such nanostructures has remained insufficiently explored. In this context, the absence of systemic, cytotoxic, and genotoxic effects in plants exposed to curcumin nanoparticles-based wastewater (Tables 4, 5 and Figure 5) is a


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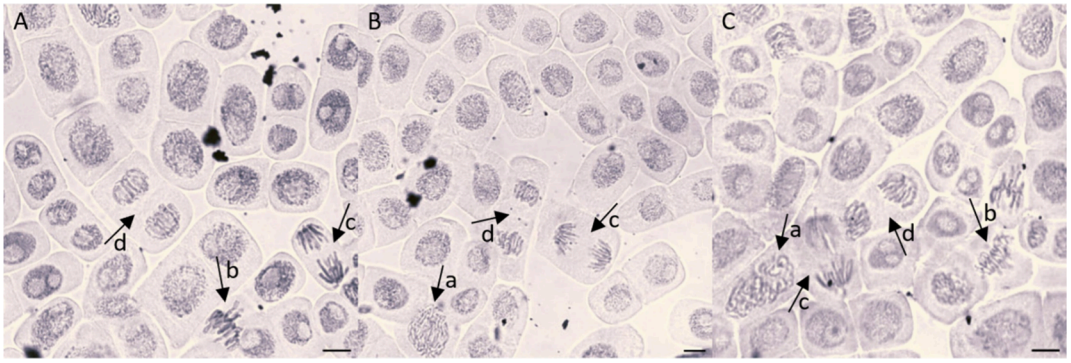


**Figure 5.** Meristematic cells from *Allium cepa* L. bulb roots exposed for 120 h to (A) control root, (B) P407 wastewater, and (C) PVP wastewater.

particularly promising result. This is relevant because the cell division process is a highly sensitive biological process that can be readily disrupted in seedling radicles upon exposure to environmental stressors, such as industrial textile effluents. The vulnerability arises from the fact that apoplastic barriers in seeds do not selectively restrict the entry of anomalous compounds into the seedling metabolism.[46]

It is also well established that inhibition of radicle growth constitutes a sublethal effect in higher plants, as it reduces the capacity of seedlings to absorb water and essential nutrients, thereby compromising overall plant fitness.[36] Therefore, the combined evidence from germination, growth, and cytogenetic endpoints supports the conclusion that, under the experimental conditions evaluated, the wastewater containing curcumin nanoparticles does not elicit phytotoxic, cytotoxic, or genotoxic effects. Collectively, these findings strongly suggest that curcumin nanoparticles represent an environmentally safe alternative for textile applications, with low ecological risk potential.[45]

## 4. CONCLUSIONS

This study systematically evaluated the use of curcumin nanoparticles in the exhaust dyeing of polyamide 6.6, combining the application of a natural dye source explored from an optimization standpoint, the development of an optimized and reproducible dyeing protocol for a particular textile substrate, and the detailed quantification of its practical performance through fastness tests. Also, the environmental safety of dyeing wastewater was evaluated using in vivo standardized tests.

Curcumin nanoparticles successfully imparted color to the polyamide fabrics. Dyeing performance was strongly influenced by process parameters: higher temperatures reduced the color intensity, likely due to curcumin degradation, while acidic conditions enhanced dye uptake and color depth. Washing fastness results were comparable for both PVP- and Poloxamer 407-stabilized nanoparticles; however, Poloxamer 407 conferred greater resistance to color transfer, making it the preferred stabilizer for applications requiring high durability. The dyed textiles also exhibited excellent rubbing and perspiration fastness and demonstrated minimal color loss after washing. In addition, the UPF was significantly improved, highlighting the functional benefits of curcumin as a dye.

Environmental assessments confirmed that dyeing wastewater was nonphytotoxic, noncytotoxic, and nongenotoxic, supporting the ecological safety of the process. Collectively, these findings demonstrate that curcumin nanoparticles represent a sustainable alternative to synthetic dyes and reinforce the potential of nonfood plant derivatives in producing eco-friendly textile products.

The application of curcumin nanoparticles in exhaust dyeing represents a notable advancement in textile coloration. By exploiting the unique properties of nanoscale materials and optimizing process conditions, this approach overcomes the limitations of conventional curcumin extracts, achieving a superior dyeing performance, enhanced fastness, and added functionality. Importantly, the incorporation of insoluble nanoparticles into the fiber structure, traditionally a property associated only with soluble dyes, challenges existing paradigms in dyeing science and opens new pathways for sustainable textile technologies.

## ■ ASSOCIATED CONTENT

### Supporting Information

The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acssuschemeng.5c06849.

Statistical analysis of the factorial $2^{4-1}$ experimental design runs for the staining test (PDF)

Characterization of the nanoparticles (methodology, results and discussion) (PDF)

## ■ AUTHOR INFORMATION

### Corresponding Author

**Odinei Hess Gonçalves** − *Textile Engineering Department (DET) and Post-Graduation Program of Textile Engineering (PGETEX), Federal University of Santa Catarina, Blumenau 89036-002, Brazil; Centro de Investigação de Montanha (CIMO), Instituto Politécnico de Bragança, 5300-253 Bragança, Portugal;* orcid.org/0000-0002-9528-8187; Email: odinei.hg@ufsc.br

### Authors

**Katharina S. Moraes** − *Textile Engineering Department (DET), Federal University of Santa Catarina, Blumenau 89036-002, Brazil*

**Aline de Oliveira** − *Textile Engineering Department (DET), Federal University of Santa Catarina, Blumenau 89036-002, Brazil*

**Bruna T. M. Ferreira** − *Post-Graduation Program of Textile Engineering (PGETEX), Federal University of Santa Catarina, Blumenau 89036-002, Brazil*

**Evandro Bona** − *Federal University of Paraná − Technology (PPGTA), Campo Mourão 87301-899, Brazil*

**Jaqueline Carneiro** − *Biopol, Chemistry Department, Federal University of Paraná, Curitiba, PR 81531-980, Brazil*



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**Ana Paula Peron** − *Department of Biodiversity, Federal University of Technology − Paraná, Campo Mourão 87301-899, Brazil*

**Marta Fernandes** − *Centre for Textile Science and Technology, University of Minho, Guimarães 4800-058, Portugal;* orcid.org/0000-0002-5814-415X

**Andrea Zille** − *Centre for Textile Science and Technology, University of Minho, Guimarães 4800-058, Portugal;* orcid.org/0000-0001-5299-4164

**Fernanda V. Leimann** − *Federal University of Paraná − Technology (PPGTA), Campo Mourão 87301-899, Brazil;* orcid.org/0000-0001-6230-9597

**Catia R. Lange de Aguiar** − *Textile Engineering Department (DET) and Post-Graduation Program of Textile Engineering (PGETEX), Federal University of Santa Catarina, Blumenau 89036-002, Brazil*

**Márcia G. da Silva** − *Textile Engineering Department (DET), State University of Maringá, Goioerê 87360-000, Brazil*

**Alexandre J. S. Ferreira** − *Textile Engineering Department (DET), Federal University of Santa Catarina, Blumenau 89036-002, Brazil; Federal University of Paraná, Jandaia do Sul 86900-000, Brazil*

**Nívea T. Vila** − *Textile Engineering Department (DET), State University of Maringá, Goioerê 87360-000, Brazil*

Complete contact information is available at: https://pubs.acs.org/10.1021/acssuschemeng.5c06849

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### Notes

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