

A study on the coloration effectiveness of *Chromolaena odorata* on the worsted wool fabric

Worsted wool fabric

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Abstract

Purpose – The health and environmental hazards associated with synthetic dyes have led to a revival of natural dyes that are non-toxic, environmentally benign and coupled with various functions. The study aims to investigate and develop the potentiality of a popular herb called *Chromolaena odorata* (*C. odorata*) as a sustainable and stable dyestuff in textiles.

Design/methodology/approach – Natural colorant extracted from *C. odorata* leaves is used to dye the worsted fabric, which is one of the premier end-use of wool in fashion, via the padding method associated with pre-, simultaneous and post-mordanting with chitosan, tannic acid and copper sulfate pentahydrate. The effects of extraction, dyeing and mordanting processes on fabric's color strength K/S and color difference ΔE_{CMC} are investigated via International Commission on Illumination's L*a*b* color space, Fourier transform infrared spectroscopy, scanning electron microscope, color fastness to washing, rubbing, perspiration and light.

Findings – The results obtained indicate extraction with ethanol 90% with a solid/liquid ratio of 1:5 within 1 h, and coloration with a liquor ratio of 1:5 (pH 5) within 2 h under padding pressure of 0.3 MPa are the most effective for coloring worsted fabric.

Practical implications – The *C. odorata*'s application as a highly effective dyestuff possessing good colorimetric effectiveness has expanded this herb's economic potential, contributing partly to economic growth and adding value to wool in global supply chain.

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Originality/value – *C. odorata* dyestuff has prevailed over other natural colorants because of its impressive color fastness against washing, rubbing, perspiration and especially color stability for pH change.

Keywords Coloration, Dyeing, *Chromolaena odorata*, Wool, Natural dye, Sustainable

Paper type Research paper

1. Introduction

Recent human awareness and mature demands on eco-preservation, eco-safety and health concerns pressurized the textile industry to use natural colorants that have no negative impact on the environment and aquatic ecosystem (Yusuf *et al.*, 2017). Advanced developments for naturally bio-sourced colorants and their sustainable use for functional clothing have created a revolution in textile research and progress (Yusuf *et al.*, 2017). In recent years, there has been an interest manifested toward the application of these natural dyes because of their biodegradability and higher compatibility with the environment, and as such, the demand for natural dyes is increasing day by day (Salaudun Sk *et al.*, 2021).

Among plenty of globally significant herbs, *Chromolaena odorata* (*C. odorata*), which belongs to the *Asteraceae* zoning dotted around tropical America, Africa, Asia and warm temperate regions in Europe, is well known because of its medical effectiveness for anticancer, antidiabetic, anti-hepatotoxic, anti-inflammatory, antibacterial and antioxidant qualities. *C. odorata* has been also used to heal wounds, burns and skin infections (Zahara, 2019). This incredible capacity for healing is brought on by *C. odorata*'s phytochemical components, including a small concentration of alkaloids, cyanogenic glycosides and flavonoids; a moderate concentration of phytates and tannins; and a very high concentration of saponins (Igboh *et al.*, 2009).

Wool material is now a common choice for high-end fashion design. However, the cost of producing wool remained expensive and correlated with the fineness of the fiber (Hearle, 2001). Worsted wool textiles, which featured very fine fibers in their structure, were frequently used to make upscale clothing. In terms of structure, worsted wools produced by the worsted processes are typically tighter, stronger, more aligned, more condensed and stiffer than woolen wools (O'Haire and Goswami, 2017). Therefore, a series of previous studies have been adopted to enhance this material's properties (e.g. the natural coloration, self-cleaning, microcapsule fixation, etc.) (Bozzi *et al.*, 2005; Mirjalili *et al.*, 2011; Pham *et al.*, 2022). In several studies, researchers applied *C. odorata* extract as an antibacterial and colorant agent on fabrics made of various textile materials, namely, cotton, viscose and so on (Owoyele *et al.*, 2005; Gangadharan, 2014; Dulmalik *et al.*, 2021; Bui and Phan, 2022). However, the colorant effects of this herb have not been studied thoroughly; moreover, previous studies have not yet investigated the relevant parameters of the wool dyeing process.

In this study, colorimetric properties were measured by International Commission on Illumination's $L^*a^*b^*$ color space (CIELab) analysis, Fourier transform infrared spectroscopy (FT-IR) and scanning electron microscope (SEM) images. Assessment of the effect of pH on color strength and the color difference revealed that a pH value of around 5 is the effective pH for wool dyeing with *C. odorata* extract. Color fastness properties of *C. odorata*-finished fabric against washing, perspiration, rubbing and artificial lighting impacts were also evaluated. The study will open up the potentiality of *C. odorata* as a natural colorant that is environmentally benign for the high-end worsted fabric to contribute to sustainable development.

Regarding the structure of this study, Section 2 provides information about materials and methods conducted in the research. Section 3 gives results and discussions. Section 4 concludes the main consequences and the dedications of the study.

2. Materials and methods

2.1 Materials

The worsted plain woven fabric (plain weave made of the worsted yarns, 87 ends per in., 63 picks per in., yarn count of both warp and weft yarns of Nm 40 and an area density of 172.5 g/m²) was supplied by LPTTEX (Vietnam).

Chemicals: ethanol ($\geq 99.5\%$) from Chemsol (Vietnam); acetic acid ($\geq 99.8\%$), sodium hydroxide ($\geq 99\%$), L-histidine monohydrochloride monohydrate (99.0%–101.0%); sodium dihydrogen phosphate dihydrate (99.0%–100.5%), potassium chloride ($\geq 99.5\%$), sodium chloride ($\geq 99.5\%$), disodium hydrogen phosphate dihydrate ($\geq 99.5\%$) and tannic acid from Merck (Germany); copper sulfate pentahydrate $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ ($\geq 98.0\%$) from AR (China); chitosan (EP Grade – extra pure reagent; deacetylation degree – DD $\geq 80\%$) from Nacalai (Japan).

C. odorata leaves were harvested at Ho Chi Minh City (Vietnam), then pre-treated via sorting, cleaning, drying processes at 105°C until no further mass loss by Mesdan Forced Ventilation Oven 251 G (Italy) and crushing into powder.

2.2 Extraction

C. odorata was extracted by ethanol (concentrations of 10, 20, 30, 40, 50, 60, 70, 80, 90 and $\geq 99.5\%$ increments) with the solid/liquid ratios of 1:5, 1:10, 1:15, 1:20, 1:25, 1:30, 1:35, 1:40, 1:45, and 1:50 within extraction durations of 1, 24, 48, 72, 96, 120, 144 and 168 h at $28 \pm 2^\circ\text{C}$ in the dark. Solid/liquid ratio means the ratio of material weight in gram (*C. odorata*) by solvent volume in mL (ethanol).

2.3 Coloration

The worsted fabric was dipped with *C. odorata* extract at a liquor ratio of 1:5, 1:10, 1:15, 1:20, 1:25 and 1:30 within surveyed dipping periods of 1, 2, 3, 6, 12, 24 and 48 h. The pH value of the extract was adjusted by either acetic acid or sodium hydroxide 10% w/w to the values of 3, 5, 7, 9 and 11. After that, the dipped fabric was squeezed by the padding method under the nip pressure of 0.2, 0.3, 0.4 and 0.5 MPa. Then, colored fabric is washed in distilled water and dried at 60°C within 1 h. The padding mangle was provided and calibrated by The Branch of Vietnam Textile Research Institute – JSC in Ho Chi Minh City (Vietnam) with rubber-covered rollers (Shore 70 A, diameter 125 mm). Liquor ratio was defined as a ratio of fabric weight in gram by volume of dyeing solution in mL (*C. odorata* extract).

2.4 Mordanting process

The mordanting process was applied on the worsted fabric before, during and after dyeing with *C. odorata* extract. In this study, the used mordants were chitosan 5% o.w.f (on-weight-fabric), $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ 5% o.w.f and tannic acid 5% o.w.f. First of all, chitosan was dissolved by acetic acid 1% w/w to obtain a chitosan solution. In detail, the mordanting process is specified below:

- Pre-mordanting (Pre-M): Fabric was dipped in each mordant (chitosan, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, tannic acid) with a liquor ratio of 1:5 within 2 h. Then, the sample was padded under

the nip pressure of 0.3 MPa. The pre-mordanted fabric underwent the dyeing process as mentioned in *Section 2.3*.

- Simultaneous mordanting and dyeing (S-M and D): Fabric was dipped simultaneously in the combination of *C. odorata* extract and each mordant (chitosan, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, tannic acid) with the liquor ratio of 1:5 within 2 h. Similarly, the dipped sample was padded under the nip pressure of 0.3 MPa.
- Post-mordanting (Post-M): First, the pre-mordanted fabric experienced the dyeing process as mentioned in *Section 2.3*. The colored fabric was dipped again in each kind of mordant (chitosan, $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, tannic acid) with the liquor ratio of 1:5 within 2 h, and then padded under the pressure of 0.3 MPa.

2.5 Characterization

2.5.1 *Color analysis in international commission on illumination's $L^*a^*b^*$ color space.* The color difference (ΔE_{CMC}) and color strength (K/S) of the finished samples in comparison with the original sample were evaluated in the CIELab color space by X-Rite Color Spectrophotometer (USA) and Color iControl software (USA).

2.5.2 Color fastness

2.5.2.1 Color fastness to washing. The sample was assessed color fastness to washing according to ISO 105 C06 A1S-2010. In detail, the sample was washed for 30 min under 30°C with 4 g of European Colorfastness Establishment (ECE) Phosphate reference detergent powders in 1 L of water and ten steel balls. Test color change and color staining were evaluated by the gray scale with five levels (where 1 is the worst and 5 is the best).

2.5.2.2 Color fastness to rubbing. The sample was assessed color fastness to rubbing according to ISO 105 X12-2016 with combed cotton lawn. In detail, the cylindrical grinding head 16 ± 0.1 mm in diameter moved back and forth in a straight line for a distance of 104 ± 3 mm on the cotton sample and applied a downward pressure of 9 ± 0.2 N. Test color change and color staining were evaluated by the gray scale with five levels (where 1 is the worst and 5 is the best).

2.5.2.3 Color fastness to perspiration. The dyed sample was evaluated for color fastness to perspiration following ISO 105 E04 – 2013. In detail, the sample dimension was 40 ± 2 mm \times 100 ± 2 mm. Acidic and alkaline sweats were prepared with specified formulas. In detail, alkaline sweat comprised: $\text{C}_6\text{H}_9\text{O}_2\text{N}_3 \cdot \text{HCl} \cdot \text{H}_2\text{O}$ (0.5 g), NaCl (5.0 g), $\text{Na}_2\text{HPO}_4 \cdot 2\text{H}_2\text{O}$ (2.5 g); distilled water (1,000 mL); NaOH 0.1 M (to adjust pH to 8.0). Besides, acidic sweat included: $\text{C}_6\text{H}_9\text{O}_2\text{N}_3 \cdot \text{HCl} \cdot \text{H}_2\text{O}$ (0.5 g), NaCl (5.0 g), $\text{NaH}_2\text{PO}_4 \cdot 2\text{H}_2\text{O}$ (2.2 g); distilled water (1,000 mL); NaOH 0.1 M (adjust pH to 5.5). The test specimen was completely wetted in an acidic solution at $\text{pH } 5.5 \pm 0.2$ or an alkaline solution at $\text{pH } 8 \pm 0.2$ in a solution ratio of 50:1, and the sample was kept at room temperature for 30 min under nominal pressure of 12.5 ± 0.9 kPa by perspirometer before being heated in the oven for 4 h at $37 \pm 2^\circ\text{C}$. Test color change and color staining were evaluated by the gray scale with five levels (where 1 is the worst and 5 is the best).

2.5.2.4 Color fastness to light. The sample was assessed for color fastness to light according to ISO 105 B02-2014. In detail, the dyed sample was exposed to the artificial light of the Xenon-ARC Fading Lamp. Color fastness grading was assessed by comparing the color change of the test sample with the standard sample used. Eight references have been chosen, where reference 1 is the most fugitive and reference 8 is the most resistant.

2.5.3 *Scanning electron microscope and stereo microscope.* Samples were taken under an SEM at x500 magnification by Hitachi S-4800 High Resolution (Japan). Stereo microscope analysis was conducted by Carl Zeiss stereo microscope (Germany).

2.5.4 *Fourier transformation InfraRed.* The samples were analyzed by FT-IR with a scanning range of wavenumber from 4,000–500 cm^{-1} on the Jasco FT/IR-4700 (Japan).

2.5.5 *Textile pH assessment.* Using ISO 3071:2005, the pH values of colored textiles were measured. The colored fabrics were cut into pieces with sides of around 5 mm. A solution of potassium chloride (0.1 mol/L) was made. Each colored fabric's three cut test specimens (weighing 2 g) were prepared, added to 100 mL of KCl solution in three stoppered glass flasks and physically shaken to extract using a vibrator for 2 h and 5 min at a rotating frequency of 30 min^{-1} . A Mettler Toledo pH meter (Switzerland) was used to measure the pH levels of the extracts.

3. Results and discussion

3.1 Effects of the extraction process on the coloration effectiveness of *Chromolaena odorata* extract on the worsted wool fabric

3.1.1 *Ethanol concentration.* Surveyed samples were denoted from E0 to E100. Figure 1(a) shows the influence of ethanol solvent concentration of *C. odorata* extraction process on the color strength K/S, color difference ΔE_{CMC} of the worsted wool fabric.

It can be seen that the average K/S value of the samples E50 and E90 possessed higher values than the remaining samples. The hypothesis method was used to evaluate the difference between two mean values with a large sample size (36 pieces). The difference between the two mean K/S values of E50 and E90, at a confidence of 95%, was recorded but not remarkable (increase of approximately 5.38%).

The mean ΔE_{CMC} value of samples E80, E90 and E100 were close to the same and higher than the remaining at a confidence of 95%. Therefore, E80, E90 and E100 are among the concentrations with the best results in the color difference in comparison with the undyed fabric.

Moreover, the ethanol/water mixture was proven to be remarkably effective in dye exhaustion (Xia *et al.*, 2018). Especially, the reusability of ethanol as the “as-is” solvent in dyeing mixtures can be conducted by various common methods consisting of azeotropic distillation, adsorption separation, direct reuse and so forth (Xia *et al.*, 2018). The higher concentration of ethanol (90% for instance) in *C. odorata* coloration process on the worsted fabric will yield advantages in reusing post-dyeing effluent in comparison with that of lower concentration (50%) by the way that ethanol used to be utilized radically as an alternative for water in textiles dyeing, degumming and so forth to reduce water usage, enhance the reusability, diminish the volume of effluent for sustainable applications (Xia *et al.*, 2018; Lyu *et al.*, 2022).

Consequently, albeit with slightly lower K/S values compared with E50 (ethanol concentration of 50%), from the above-mentioned results and practical values relating to reusability and sustainable purposes in general, the ethanol concentration of 90% was considered one of effective concentrations in comparison with others.

3.1.2 *Solid/liquid ratio.* Figure 1(b) demonstrates the influence of the solid/liquid ratio of the *C. odorata* extraction process on the values of color strength K/S and color difference ΔE_{CMC} of the worsted fabric.

The K/S and ΔE_{CMC} decreased gradually when increasing ethanol volume per a certain mass of *C. odorata*. And, it can be seen that the average value of the sample with a solid/liquid ratio of 1:5 was the highest in total.

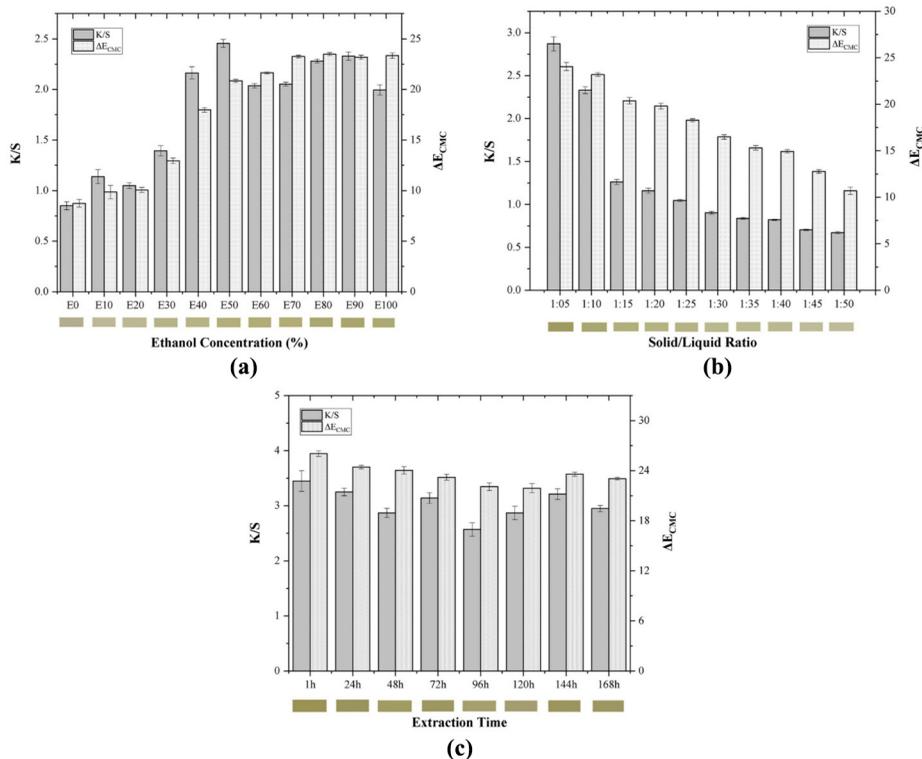


Figure 1. Color difference ΔE_{CMC} and color strength K/S of dyed fabric for various

Notes: (a) Ethanol concentrations (Solid/Liquid ratio of 1:10, Extraction time of 48h); (b) Solid/Liquid ratios (Ethanol solvent 90% concentration, extraction time of 48h); (c) Extraction time (Ethanol solvent 90% concentration, Solid/Liquid ratio of 1:5) in extraction process. To evaluate the effects of the extraction process for coloration efficacy, these extracts were applied on the worsted fabric by padding method under the nip pressure of 0.3 MPa, liquor ratio was 1:10, within 1 hour (intrinsic pH values of extracts were approximately 5)

3.1.3 Extraction time. Figure 1(c) elucidates the influence of the extraction time of the *C. odorata* extraction process on the average value of color strength K/S and color difference ΔE_{CMC} of the worsted fabric.

It can be seen that the average value of K/S fluctuated continuously. The values of K/S and ΔE_{CMC} of two samples with an extraction time of 1 and 24 h were higher than those of others. Based on the hypothesis testing method to evaluate the difference between two mean values with a large sample size (36 pieces), the difference between two mean K/S and ΔE_{CMC} of two samples with an extraction time of 1 and 24 h was significant at 95% confidence. Besides, 1 h of extraction time will be more effective, especially in mass production.

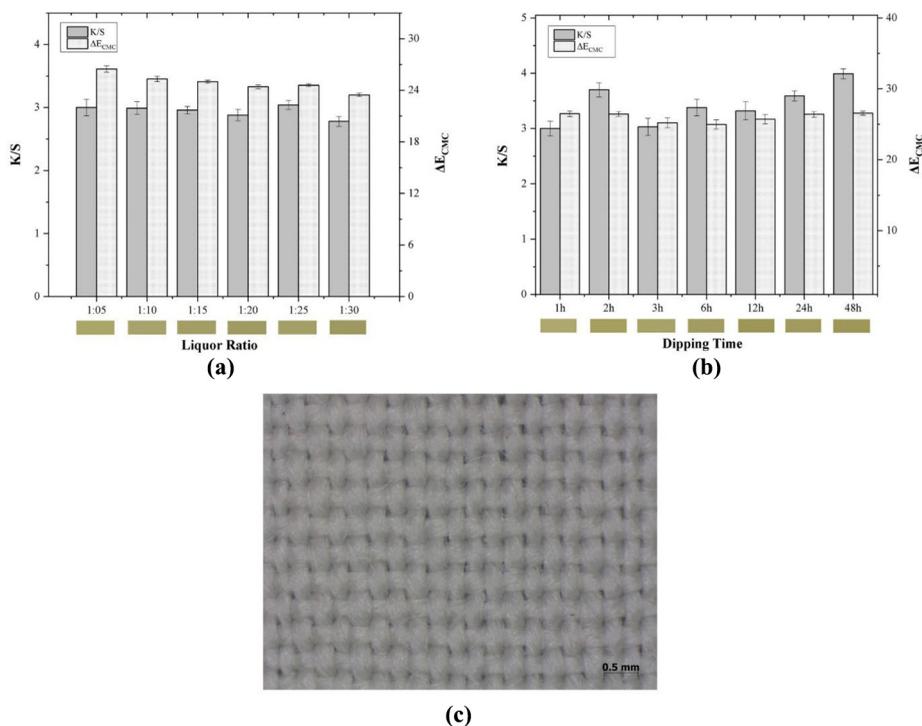
In the same vein, in the previous study, the extraction of a natural dyestuff from *Terminalia arjuna* fruits and *Rubia tinctorum L* with various mixing proportions (w/w) on scoured and bleached wool fabrics indicated that the color strength maximum value at 60 min extraction time, which was equivalent to our study on *C. odorata* (Kamel *et al.*, 2011).

3.2 Effects of the coloration process on the color effectiveness of *Chromolaena odorata* extract on the worsted wool fabric

3.2.1 Liquor ratio. The average K/S value fluctuated with the change in the liquor ratio. From Figure 2(a), it can be seen that the 1:5 and 1:25 ratios presented higher mean values than the rest. The hypothesis testing method at 95% confidence showed that there was no difference between the two samples.

Regarding ΔE_{CMC} , samples of 1:5 and 1:10 liquor ratios were promising contenders for *C. odorata*'s coloration. To determine the optimal liquor ratio, the hypothesis testing for mean ΔE_{CMC} of the ratio 1:5 and 1:10 values with a large sample size (36 pieces) was conducted at the confidence of 95%. As a result, the discrepancy between the two samples was remarkable. Moreover, considering both aspects of color effectiveness and chemical consumption, the liquor ratio of 1:5 was preferred.

When evaluating the influence of liquor ratio on the color efficacy of the worsted wool fabric, the results showed that with the liquor ratio of 1:5, the mean value of color difference ΔE_{CMC} was the largest; the average value of color strength K/S values between 1:5 and 1:25



Notes: (a) liquor ratio (dipping time 1 h, adjusted pH of 5, padding pressure of 0.3 MPa); (b) dipping time (liquor ratio of 1:5, adjusted pH of 5, pressure of 0.3 MPa) in the coloration process. To evaluate the effects of the coloration process on color effectiveness, the optimal *C. odorata* extract (ethanol concentration of 90%, solid/liquid ratio of 1:5 and extraction time of 1 h) were applied on the worsted fabric by padding method, (c) stereo microscope image of the original worsted plain-woven fabric

Figure 2. Color difference ΔE_{CMC} and color strength K/S of dyed fabric for various

ratios have had no significant difference and been higher than the remaining. Therefore, choosing a ratio of 1:5 has yielded a reduction in extract consumed and created impressive coloration effectiveness on the worsted wool fabric. The 1:5 ratio elucidated the most effective coloration with the lowest consumed chemicals.

Although the majority of textile products derived from wool often possess the intrinsically voluminous bulk and hydrophilic property, however, in this study, the used plain-woven fabric made of worsted yarns (yarn count of Nm 40) possesses a very tight, condensed structure [Figure 2(c)], which facilitated to maintain the compact structure intact for swelling of the worsted fabric in dyeing solution in comparison with the fabric composed of woolen yarn (O'Haire and Goswami, 2017). Therefore, because of this ideal structure of the worsted fabric, the liquor ratio of 1:5 applied on this worsted fabric's coloration process is more practically feasible in comparison with other products derived from wool. However, for other wool products, it is recommended to use suitable liquor ratios depending on the structural properties of various wool products.

3.2.2 Dipping time. From Figure 2(b), it can be seen that the ΔE_{CMC} values of the samples dipped 1 and 2 h were higher than the remaining samples. The hypothesis method was used to evaluate the difference between two mean values with a large sample size (36 pieces). The difference between the two mean ΔE_{CMC} values of 1 and 2 h, at a confidence of 95%, was not significant.

Considering the K/S, the values of all samples were not too different, the 48-h sample showed the largest value. Combined with the ΔE_{CMC} result, results of the samples dipped in 2 and 48 h were selected to compare the differences between the two average values by the hypothesis testing method. As a result, there was a significant difference between the two samples. However, when the dipping time increased 24-fold from 2 to 48 h, there was a slight raise in K/S by around less than 10% (from approximately 3.70 to 3.99). Therefore, it can be seen that the 48-h dipping option was not necessary because the time difference was very large compared to 2 h, causing a waste of time, but the coloration was not too remarkable. In conclusion, the 2-h dipping time option was preferred.

3.2.3 Padding pressure. Figure 3(a) presented that the color strength value K/S and color difference ΔE_{CMC} of the sample with the padding pressure of 0.3 MPa were more effective than the rest. It can be observed from SEM analysis from Figure 3(b) that at the padding pressure of 0.3 MPa, the wool structure had certain effects, fiber scales were broken to create debris, but very little. The padding pressures of 0.4 and 0.5 MPa greatly affected the surface structure of wool fibers, significantly affecting the fiber's appearance. In conclusion, considering the effect of padding pressure on the color strength and color difference of treated fabric, coupled with the SEM results, the padding pressure of 0.3 MPa was the most effective.

3.2.4 Effect of pH value of colorant solution. This section figured out the impact of pH on the dyeing process under the same conditions. Based on Figure 4(a), the values of K/S and ΔE_{CMC} at the alkaline condition (pH = 11) of the dyeing solution were higher than the others. Nevertheless, the gap in this discrepancy was not truly noticeable. By contrast, in the previous study, the extraction of a natural dyestuff from *Terminalia arjuna* fruits and *Rubia tinctorum* L with various mixing proportions (w/w) on scoured and bleached wool fabrics had proven that the highest color strength was obtained at a pH value of 4 (Kamel et al., 2011).

It can be seen from Figure 4(b) that there was not any change in the position of the K/S peaks representing the colorant stability due to the alteration of the pH value of the dyeing solution. While with other natural dyestuffs (Brazilein, Anthocyanin, etc.) derived from plants (e.g. *Caesalpinia sappan* L., *Clitoria ternate*, Red cabbage, etc.), there was a significant

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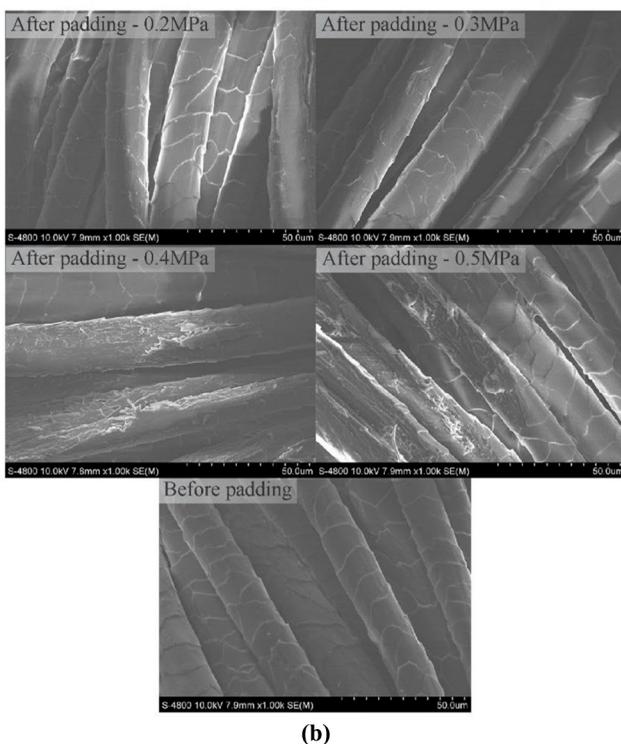
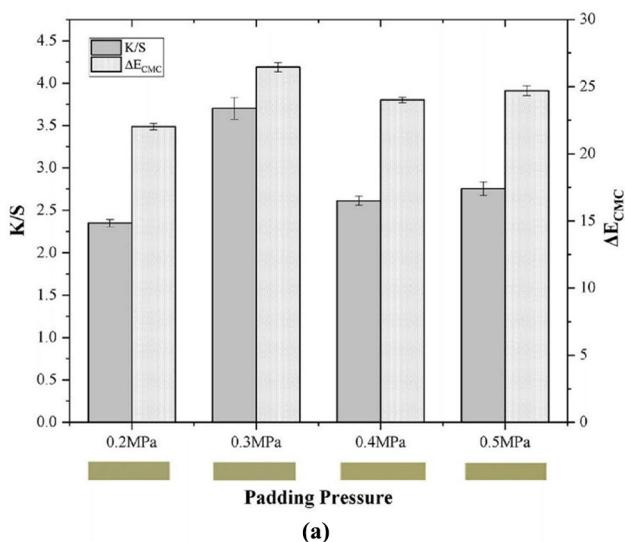


Figure 3. Effect of padding pressure on (a) Color difference ΔE_{CMC} and color strength K/S; and (b) SEM images of undyed (before padding) and dyed (after padding) wool structure (Liquor ratio of 1:05, dipping time of 2 hours, adjusted pH value of 5)

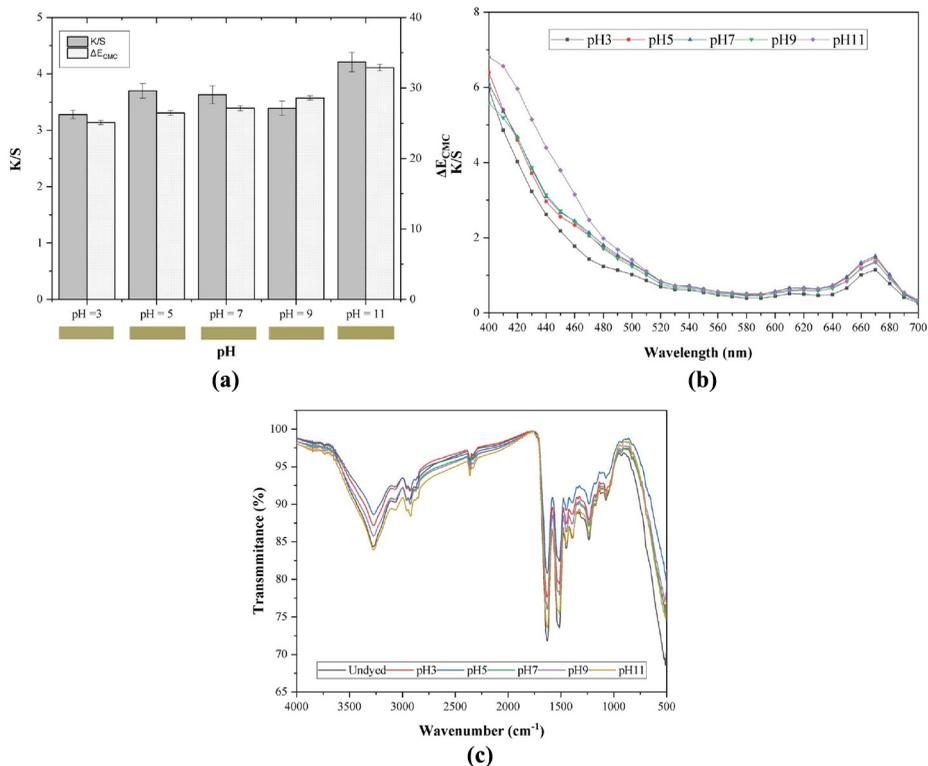


Figure 4.

Effects of colorant solution's pH value on (a) Color difference ΔE_{CMC} and color strength K/S; (b) K/S spectra; and (c) FT-IR spectra of dyed fabric (liquor ratio of 1:5, dipping time of 2 hours, pressure of 0,3 MPa)

change in color depending on pH value of dyeing solution (Wiczkowski *et al.*, 2013; Khoo *et al.*, 2017; Ngamwonglumlert *et al.*, 2020; Roy and Rhim, 2021; Vidana Gamage *et al.*, 2021). Thus, our investigation opened up the step forward to the naturally stable dyestuff.

Turning into the safety aspect for the textiles that contact directly with human skin, the pH value of colored fabrics had been assessed. The textile pH assessment is truly significant that is required to be skin-friendly for direct contact purposes as numerically defined by various national regulations, standards of textile and fashion brands, international organizations (such as standards of Oeko Tex, GOTS Goods and so forth) (Das, 2013). In detail, the safe pH for skin is in the range of 4.1–5.8 (Proksch, 2018). If the pH value of the material is above 6, there will be the risk of being attacked by human-pathogenic bacteria, *Candida albicans* yeast and so forth (Schneider *et al.*, 2007). In Table 1, it is obvious that the strongly acidic and alkaline pH values of colorant solutions (pH = 3 and pH = 11,

Table 1.

The dyed fabric pH in accordance with pH values of colorant solutions

pH value	Colorant solutions	3	5	7	9	11
	Colored fabric	3.99 ± 0.03	4.84 ± 0.02	5.32 ± 0.03	5.78 ± 0.03	6.45 ± 0.03

respectively) were not safe for skin contact (Das, 2013). The rest levels were in the safe range; moreover, the intrinsic pH value of the *C. odorata* extract was around 5, while the adjusted pH value was 5, which gave the most color efficacy. The results of the FT-IR analysis [Figure 4(c)], represented the identification of the combination of wool and primary compounds in *C. odorata* (flavonoids, alkaloids, saponins, tannins) (Hong, 2015; Zargarkazemi et al., 2015; Shwe and Win, 2019; Bui and Phan, 2022), indicated that these samples were all similar. It can be concluded that there was no any change in the material in various pH values of the dyeing solution, although wool is usually dyed in acidic conditions (Kamel et al., 2011). Therefore, in this case, extracted substances derived from *C. odorata* did not serve as an acidic dyestuff but instead operated as a direct dye.

The fastness properties of *C. odorata* dyed worsted fabric are shown in Table 2. In general, the *C. odorata* extract gave relatively good results to color fastness assessments for all ranges of pH values. Based on the results of the washing color fastness assessment, it can be seen that the color strength was the same between the samples. As for the color staining, it was almost equivalent and relatively good. Based on the test results, it can be deduced that the *C. odorata* extract bound well to wool and that the pH did not affect the color fastness to washing too much. In the same vein, the color fastness to rubbing of the samples was relatively good, both longitudinal and transverse friction color fastness in wet and dry conditions were similar. Regarding the

List of color fastness tests			pH3	pH5	pH7	pH9	pH11
<i>Color fastness to washing</i>							
Color change			3	3	3	3	3
Color staining	Acetate		4-5	4	4-5	4	4
	Cotton		4-5	4-5	4-5	4	4
	Nylon		4	3-4	4	4	4
	Polyester		4-5	4-5	4-5	4-5	4-5
	Acrylic		4-5	4-5	4-5	4-5	4-5
	Wool		4-5	4-5	4-5	4-5	4-5
<i>Color fastness to rubbing</i>							
Vertical	Dry		4	3-4	3-4	3-4	3-4
	Wet		3-4	3-4	3-4	3-4	3-4
Horizontal	Dry		4	3-4	3-4	3-4	3-4
	Wet		3-4	3-4	3-4	3-4	3-4
<i>Color fastness to perspiration</i>							
Color change	Acidic		4	4	4	4	3-4
	Alkaline		4	4	4	4	4
Color staining	Acetate	Acidic	4	4	4	4	4
		Alkaline	4	4	3-4	3-4	3-4
	Cotton	Acidic	4-5	4	4	4	4
		Alkaline	4	4	3-4	3-4	3-4
	Nylon	Acidic	3-4	3-4	3-4	3-4	3-4
		Alkaline	3-4	3-4	3-4	3-4	3-4
	Polyester	Acidic	4-5	4	4	4	4-5
		Alkaline	4-5	4	4	4	3-4
	Acrylic	Acidic	4-5	4	4	4-5	4-5
		Alkaline	4-5	4	4	4	3-4
	Wool	Acidic	4-5	4-5	4-5	4-5	4-5
		Alkaline	4-5	4	4	4	3-4
Color fastness to light			1	1	1	1	1

Table 2. Effects of pH values of colorant solutions on color fastness of *C. odorata* dyed worsted fabric

impacts of perspiration, based on the results of the evaluation of color change with acid/alkali sweat, the samples were relatively similar and good, ranging from 3–4. The assessment results of the samples for color staining with acidic perspiration possessed the same trend with their color change. In which adjacent fabric samples from nylon fiber gave lower results than the others (3–4). However, the results for color fastness to the artificial light of all samples were truly poor (at the lowest level), which is a common drawback of natural dyes (e.g. Madder, Alkanna, Safflower, Henna, Sandalwood, Cochineal, Brazilwood and so forth), albeit with good protection against sunlight/ultraviolet radiation impact of wool materials (Padfield and Landi, 1966; Gambichler *et al.*, 2001; Zarkogianni *et al.*, 2011). It can be concluded that the *C. odorata* extract confirmed its effectiveness for worsted fabric's dyeing due to high color fastness for living activities (e.g. washing, rubbing, perspiration); nevertheless, it is highly recommended to prevent direct exposure to the sun, especially for drying purpose for a long time.

3.2.5 Effect of mordanting. The impact of the mordanting process was studied in various methods (pre-mordanting, simultaneous mordanting and dyeing, post-mordanting) as mentioned in Section 2.4 and mordants ($\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, tannic acid and chitosan mordants). The results were illustrated in Figure 5. In terms of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ mordant, the K/S and ΔE_{CMC} of the Pre-M (pre-mordanting) were higher than the others and $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ mordanted samples. However, these mordanting processes with $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ have had higher K/S and ΔE_{CMC} values compared with unmordanted ones and been strongly different from the original color of this natural dyestuff, which was considered as a significant downside of this mordant. Whereas, there was a similar trend in the cases of tannic acid and

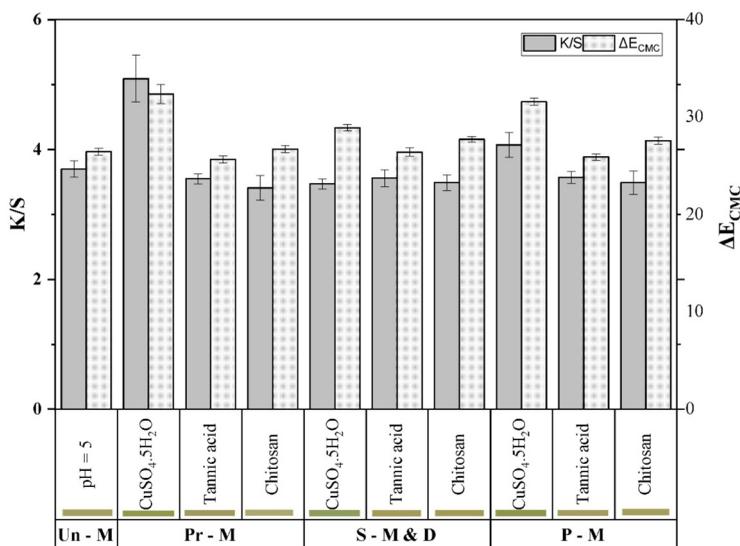


Figure 5. The effects of the mordanting method using $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, tannic acid and chitosan on the value of color strength K/s and color difference ΔE_{CMC}

Notes: The surveyed samples were denoted in the order Pre-M, S-M and D, Post-M, and Un-M, respectively, with pre-mordanting, simultaneous mordanting and dyeing, post-mordanting and unmordanted samples

chitosan mordants, in which the S-M and D samples (simultaneous mordanting and dyeing) showed the most positive results relating to color strength, color difference and especially process shortening and simplifying. Furthermore, it can be seen clearly that tannic acid and chitosan mordanted samples had lightly lower K/S and ΔE_{CMC} levels in comparison with the unmordanted ones. Hence, these bio-based mordants (tannic acid and chitosan) will be prioritized for *C. odorata* dyeing to maintain the original color of this herb. Tables 3, 4 and 5 elucidate the improvement for color fastness owing to mordanting, especially for color fastness to washing, and light; however, the color fastness's enhancing efficacy was not significant in comparison with unmordanted samples (Table 2). Besides, in comparison with other natural dyestuff (e.g. Madder, Alkanna, Safflower, Henna, Sandalwood, Cochineal, Brazilwood, Logwood, Indigo and so forth) with addition of mordants (Alum, SnCl₂, K₂Cr₂O₇, CuSO₄, ZnCl₂, FeCl₃, FeSO₄, etc.), *C. odorata* was proven its effectiveness for color fastness against washing, rubbing, perspiration without addition of any mordant. Moreover, adding CuSO₄ mordant facilitated a slightly improvement in color fastness against light of *C. odorata* dyed fabric that is strongly related to a variety of studies about mordanting with CuSO₄ of natural dyes (Zarkogianni *et al.*, 2011; Mansour *et al.*, 2022). In conclusion, the color

List of color fastness tests		Pre-mordanted	Simultaneously mordanted and dyed	Post-mordanted	
<i>Color fastness to washing</i>					
Color change		3	2	4	
Color staining	Acetate	4-5	4-5	4-5	
	Cotton	4-5	4-5	4-5	
	Nylon	4	4	4	
	Polyester	4-5	4-5	4-5	
	Acrylic	4-5	4-5	4-5	
	Wool	4-5	4-5	4-5	
<i>Color fastness to rubbing</i>					
Vertical	Dry	2-3	3	3	
	Wet	3	3	3	
Horizontal	Dry	2-3	3	3	
	Wet	3	3	3	
<i>Color fastness to perspiration</i>					
Color change	Acidic	4	4	4	
	Alkaline	4	4	4	
Color staining	Acetate	Acidic	4	4	
		Alkaline	4	4	
	Cotton	Acidic	3-4	4	3
		Alkaline	3-4	3-4	4
	Nylon	Acidic	3-4	3-4	3
		Alkaline	3-4	3-4	3
Polyester	Acidic	4	4	4	
	Alkaline	4	4	4	
Acrylic	Acidic	4	4	4	
	Alkaline	4	4	4	
Wool	Acidic	4	4-5	4	
	Alkaline	4	5	4	
Color fastness to light		2	1	2-3	

Table 3.
Effects of mordanting using copper sulfate pentahydrate on color fastness of *C. odorata* dyed worsted fabric

List of color fastness tests		Pre-mordanted	Simultaneously mordanted and dyed	Post-mordanted	
<i>Color fastness to washing</i>					
Color change		3	2-3	3	
Color staining	Acetate	4-5	4	4	
	Cotton	4-5	4	4	
	Nylon	4	4	4	
	Polyester	4-5	4-5	4-5	
	Acrylic	4-5	4-5	4-5	
	Wool	4-5	4-5	4-5	
<i>Color fastness to rubbing</i>					
Vertical	Dry	3-4	3-4	3-4	
	Wet	3-4	3-4	3-4	
Horizontal	Dry	3-4	3-4	3-4	
	Wet	3-4	3-4	3-4	
<i>Color fastness to perspiration</i>					
Color change	Acidic	4	4	4	
	Alkaline	3-4	3-4	3-4	
Color staining	Acetate	Acidic	4	4	
		Alkaline	3-4	3-4	
	Cotton	Acidic	4	4	4
		Alkaline	3-4	3-4	3-4
	Nylon	Acidic	3-4	3-4	3-4
		Alkaline	3-4	3-4	3-4
	Polyester	Acidic	4	4	4
		Alkaline	3-4	3-4	3-4
	Acrylic	Acidic	4	4	4
		Alkaline	3-4	3-4	3-4
	Wool	Acidic	4-5	4	4
		Alkaline	3-4	3-4	3-4
Color fastness to light		1	1	1-2	

Table 4.
Effects of mordanting using tannic acid on color fastness of *C. odorata* dyed worsted fabric

fastness before and after mordanting of *C. odorata* was thoroughly investigated which will contribute to further references in natural dye studies as well as strongly confirmed the coloration effectiveness and fastness of pristine *C. odorata* dyestuff for worsted wool fabric.

4. Conclusion

Natural dyestuff was extracted from *C. odorata* at various conditions, and its dyeing on wool fabric was optimized. To figure out the effects of the extraction on color strength K/S and color difference ΔE_{CMC} , there were alterations in many factors, including ethanol solvent concentration, solid/liquid ratio, extraction time. As a result, the optimal extracting conditions giving the most promising result on color efficacy were ethanol solvent at 90% concentration, solid/liquid ratio of 1:5 and extraction time of 1 h. In the same vein, the impacts of the coloration process on the color effectiveness of *C. odorata* extract on the wool fabric were also studied by conducting the coloration period under different conditions. The results obtained indicate that a liquor ratio of 1:5, dipping time of 2 h, padding pressure of 0.3 MPa and adjusted pH value of 5 were truly effective for coloring worsted wool fabric. It was found that despite the changes in pH of the dyeing solution, there was no change in the position of the K/S peaks, representing the colorant stability. Hence, it can be concluded that

Worsted wool fabric

List of color fastness tests		Pre-mordanted	Simultaneously mordanted and dyed	Post-mordanted	
<i>Color fastness to washing</i>					
Color change		4	3	3-4	
Color staining	Acetate	4-5	4	4-5	
	Cotton	4	4	4-5	
	Nylon	4	4	4-5	
	Polyester	4-5	4-5	4-5	
	Acrylic	4-5	4-5	4-5	
	Wool	4-5	4-5	4-5	
<i>Color fastness to rubbing</i>					
Vertical	Dry	3	3-4	3-4	
	Wet	3-4	3-4	3-4	
Horizontal	Dry	3	3-4	3-4	
	Wet	3-4	3-4	3-4	
<i>Color fastness to perspiration</i>					
Color change	Acidic	3-4	4	4-5	
	Alkaline	4	3-4	4	
Color staining	Acetate	Acidic	4	4	
		Alkaline	4	4	
	Cotton	Acidic	4	4	4
		Alkaline	4	3-4	3-4
	Nylon	Acidic	4	3-4	3-4
		Alkaline	3-4	3-4	3-4
	Polyester	Acidic	4-5	4	4-5
		Alkaline	4-5	4	4
	Acrylic	Acidic	4-5	5	4-5
		Alkaline	4-5	4	4
Wool	Acidic	4-5	4-5	4-5	
	Alkaline	4-5	4-5	4	
Color fastness to light		1	1-2	1-2	

Table 5.
Effects of mordanting using chitosan on color fastness of *C. odorata* dyed worsted fabric

natural dyestuff extracted from *C. odorata* was deemed to be a stably natural dyestuff. In addition, the color fastness properties of treated fabric were also evaluated by testing the color fastness of fabric against washing, acidic and alkaline perspiration, rubbing and artificial light. The results (all were above 3 out of 5) indicated that good fastness properties were achieved. In the cases of the mordanting process, it was studied in various methods (pre-mordanting, simultaneous mordanting and dyeing and post-mordanting) with three kinds of mordants. It is noticed that mordanting with $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ before dyeing showed a significant change in both K/S and ΔE_{CMC} , while tannic acid and chitosan have had advantages in preserving the original color of these natural herb dyes. It is obvious that *C. odorata* yielded good color fastness; therefore, it is implied that mordanting process is not vital for this innovative sustainable dyestuff.

In conclusion, it is clear from the results that dyestuff extracted from *C. odorata* is a stable one with highly colored resistance to various impacts, including mechanical and chemical actions. Furthermore, the intrinsic extract's pH value of 5 is safe for skin contact. With the desire to replace artificial dyes that are being used on the market with natural resources, the use of *C. odorata* as a highly effective dyeing dyestuff and good colorimetric effectiveness has expanded the economic potential of this plant, thus contributing in part to

economic growth and added value to wool in the global supply chain. For the future perspective of the study, to commercialize this natural dyestuff and actualize the aforementioned targets, the cost-effectiveness and the comparison with synthetic dyes are required other thorough and dedicated studies in both economic and engineering fields. Finally, because of the abundant availability and strong vitality of *C. odorata* in the natural environment in combination with simplified and effective extraction and coloration processes proposed by our study, which required less liquor ratio (1:5) for dyeing, less solvent used for extraction (solid/liquid ratio of 1:5), *C. odorata* will yield novelty and innovation in eco-textile product manufacturing.

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