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Temperature Optimization in the Ultrasonic Dyeing Process of Cotton Fabric Using Natural Dye Powder from Sappan Wood (*Caesalpinia sappan* L.)

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Abstract
Sappan wood (Caesalpinia sappan L.) is one of the natural dye sources with potential for use in textile dyeing because it contains brazilin, which can be oxidized into brazilein and produce colors ranging from red to brownish-red. The use of the ultrasonic dyeing method can assist the dye absorption process into textile fibers, but the effect of temperature on the dyeing results of sappan wood still needs to be examined. This study aims to determine the effect of temperature variations of 40°C, 50°C, 60°C, and 70°C in the ultrasonic dyeing process with a frequency of 22 kHz for 20 minutes on the dyeing results of cotton fabric. The parameters observed include color strength (K/S), CIELAB values, color fastness, and functional group characteristics using FTIR. The results showed that the K/S value increased from 2.105 at 40°C to 2.627 at 60°C, then decreased to 2.146 at 70°C. This decrease is thought to be related to changes or degradation of the dye compound due to higher temperatures. Color fastness to washing was at grade 1–1–2, while color fastness to wet rubbing was at grade 4–5 at 40–50°C and decreased to grade 4 at 60–70°C. FTIR analysis results showed a relatively similar absorption band pattern across all treatments and did not indicate the formation of new bands. Based on the overall results, a temperature of 60°C produced the highest K/S value, so it can be used as the optimum condition in the ultrasonic dyeing process of cotton fabric using sappan wood dye powder. The CIELAB analysis was also conducted to examine the color characteristics of the dyed fabrics, including lightness (L*), redness (a*), and yellowness (b*). The hue angle values ranged from 19°C to 21.9°C indicating relatively similar color tones across the temperature variations. The apparent exhaustion (Eapp) values were also recorded to provide additional information about the dyeing process. The results of this study provide information on the relationship between dyeing temperature and the color characteristics of cotton fabric dyed with sappan wood using ultrasonic technology. These findings may serve as a reference for further research on natural dyeing methods and efforts to improve the color fastness of naturally dyed textiles.

Keywords: *sappan wood; ultrasonic dyeing; cotton fabric; color strength; color fastness*

INTRODUCTION

The textile industry is one of the sectors that continues to grow and plays an important role in meeting society's clothing needs. Along with increasing textile production, the use of synthetic dyes has become dominant in the dyeing process because they offer many color variations, are easy to apply, and are relatively cheap. However, the use of synthetic dyes also has a negative impact on the environment. However, the use of synthetic dyes also has a negative impact on the environment (Islam et al., 2025). The waste produced contains hazardous chemical substances that are difficult for nature to decompose (Al-Tohamy et al., 2022). This condition has encouraged the use of natural dyes from biological sources as a more environmentally friendly and sustainable alternative. Various types of local plants containing compounds such as flavonoids, tannins, anthocyanins, and phenolic compounds have the potential to be utilized as sources of natural dyes for textiles (Setyaningsih et al., 2025). Sappan wood (Caesalpinia sappan L.) is a tropical plant with potential as a source of natural dye for textiles (Mai, Grethe and Mahltig, 2024). Sappan wood is used as a natural dye because it is

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able to produce colors ranging from red to brownish-red and has an abundant availability of raw material in Indonesia (Pujilestari and Salma, 2017).

The dyeing characteristics of sappan wood as a natural dye are related to its phenolic compound content, particularly brazilin, which can undergo oxidation into brazilein, thereby producing a higher color intensity. The color characteristics of sappan wood extract are determined by pH conditions and the use of mordant, which influence the direction and intensity of the resulting dye color (Rahayuningsih et al., 2018). In its application, the natural sappan wood dye powder is first redissolved to form a dye solution, which is then used as the dyeing medium for fabric. Textile dyeing using natural dyes is generally still carried out using the conventional dyeing method. In this method, fabric is immersed in the dye solution with the aid of heating to assist dye absorption into the textile fibers. The conventional dyeing method is still widely used because the process is relatively simple and can be applied to various types of textile materials.

However, this conventional method requires relatively large amounts of energy and water, as well as a fairly long dyeing time for the dye to be properly absorbed into the fibers (Hassan and Saifullah, 2023). On this basis, various efforts continue to be made to develop more efficient dyeing methods, one of which is through the application of ultrasonic dyeing (Shipan et al., 2017). Along with the development of textile dyeing technology, the ultrasonic dyeing method has become one of the innovations in dyeing using natural dyes. This method utilizes ultrasonic waves that generate a cavitation phenomenon, namely the formation and collapse of microscopic bubbles within the dye solution. This cavitation effect helps increase the contact between the dye solution and the fiber surface, making the dyeing process more effective (Farooq et al., 2018). The cavitation effect can also increase color intensity while producing more even dyeing on the fabric surface (Tissera, Wijesena and de Silva, 2016). In the application of ultrasonic dyeing, temperature plays an important role in determining the effectiveness of dye absorption into textile fibers. Changes in temperature during dyeing can affect the migration of dye molecules into the fiber.

At an appropriate temperature, ultrasonic waves are able to enhance the migration of dye molecules into the fiber, thereby increasing the amount of dye absorbed and the color strength value (Ali et al., 2019). However, at excessively high temperatures (Banna et al., 2023), this can reduce the stability of the natural dye compound, so that the resulting color intensity has the potential to decrease. Therefore, temperature regulation needs to be considered in order to obtain optimal dyeing results during the application of ultrasonic dyeing (Rehman et al., 2024). Previous studies on the utilization of sappan wood as a natural dye, the application of the ultrasonic dyeing method, and the effect of temperature on dyeing results have provided information on the characteristics of natural dyes as well as the role of temperature in dye absorption into fibers. Nevertheless, the characteristics of the dyeing results of cotton fabric using sappan wood natural dye solution at various temperature variations during the application of ultrasonic dyeing still need further examination. Based on this condition, this study aims to analyze the effect of temperature variation during the application of ultrasonic dyeing on the dyeing characteristics of cotton fabric based on UV-Vis analysis, FTIR, and color fastness testing.

METHOD

This research used sappan wood (Caesalpinia sappan L.), cotton fabric, water, alum ($Al_2(SO_4)_3$), soda ash (Na_2CO_3), and lime as research materials. The equipment used in this research included an ultrasonic dyeing device, a digital scale, a beaker glass, a measuring cup, a thermometer, and other supporting equipment. The dyed fabric that had undergone the fixation stage was then used as a sample for UV-Vis analysis, FTIR, and color fastness testing carried out in the testing laboratory.

The sappan wood dye was first prepared through an extraction stage using water. A total of 1 kg of sappan wood was soaked in 8 L of water for approximately 15 hours. After soaking, the sappan wood was boiled for 2 hours, starting with heating until boiling and continuing with low heat. The resulting extract was left to reach room temperature, then filtered using a fabric with a not-too-tight weave. The filtered extract was then dried using a spray dryer to obtain the dye in powder form. Before the dyeing process, the cotton fabric was given a pre-mordanting treatment using alum and soda ash to help increase the fiber's affinity for the dye. The sappan wood dye powder was then dissolved using water at a concentration of 10% (w/v). This concentration was obtained from the use of 12 g of dye powder in 120 mL of water, based on the ratio of powder mass to water volume using the equation:

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$$\text{Concentration} = \frac{12\text{g}}{120mL} \times 100\% = 10\%$$

This solution was used to dye cotton fabric weighing 6 g, so that the ratio of fabric weight to the solution volume was 1:20. The fabric that had undergone pre-mordanting was then placed into the dye solution and processed using an ultrasonic dyeing device at a frequency of 22 kHz for 20 minutes. The treatment was applied at four temperature variations, namely 40°C, 50°C, 60°C, and 70°C. During the dyeing process, the fabric was kept submerged and occasionally turned over so that the solution could evenly reach both surfaces of the fabric. After the treatment time was completed, the fabric was removed from the device and dried at room temperature before fixation was carried out.

Fixation was carried out using a lime solution made by dissolving 15 g of lime into 4 L of water. The dyed fabric was placed into the solution and gently rubbed for 5 minutes. After the treatment was completed, the fabric was dried at room temperature. The fabric that had undergone all treatments was then prepared for testing. Color characteristic analysis was carried out to determine the dyeing results for each temperature variation using samples measuring 15 x 15 cm. The parameters analyzed included color strength (K/S), CIELAB values, and Eapp. FTIR analysis was carried out to determine the functional group characteristics of the fabric after the dyeing process using samples measuring 2 x 2 cm. Meanwhile, color fastness testing was carried out to determine the fabric's ability to retain color after being subjected to the testing treatment, using samples measuring 25 x 10 cm and 10 x 5 cm. All testing was carried out in the testing laboratory using instruments and equipment appropriate to each parameter being analyzed.

RESULTS AND DISCUSSION

Effect of Temperature on Color Strength (K/S) and CIELAB Values

Color characteristic testing was carried out to determine the changes in the dyeing results of cotton fabric at various ultrasonic dyeing process temperatures. The temperature variations used consisted of 40°C, 50°C, 60°C, and 70°C. The color characteristics observed included the Eapp value, color strength (K/S), and CIELAB values consisting of L*, a*, b*, and ΔE*. The color characteristic test results for each temperature variation are presented in Table 1.

Table 1. Eapp, K/S, and CIELAB (ΔE*) Test Results of Cotton Fabric Dyed with Sappan Wood at Various Temperatures

Parameter	40°C	50°C	60°C	70°C
Eapp (%)	−13.77	−41.30	−70.29	−102.17
K/S	2.105	2.354	2.627	2.146
L*	53.72	56.73	2.627	56.18
a*	30.05	29.77	2.627	29.59
b*	10.47	10.92	10.56	11.87
ΔE*	3.4	4.46	—	4.16

Based on Table 1, the K/S values show a change in color strength at each temperature variation. The K/S value increased with rising temperature from 40°C (2.105) to 50°C (2.354) and reached its highest value at 60°C (2.627), then decreased at 70°C (2.146). This pattern indicates that increasing temperature up to a certain point promotes the more optimal diffusion of brazilin molecules into the cotton fabric fiber, consistent with the ultrasonic cavitation effect that enhances contact between the dye solution and the fiber surface. At 70°C, the decrease in K/S is related to the thermal degradation of the brazilin/brazilein compound, so that the color intensity actually decreases even though the process temperature is higher. This is consistent with the potential decrease in stability of the natural dye compound at excessive temperatures. The CIELAB values show a more varied pattern. The L* value (brightness level) does not show a monotonic trend, ranging from 52.37–56.73, with the lowest value (darkest) at 60°C, coinciding with the highest K/S, which indicates the most intense color at that temperature. The a* value is relatively stable (29.59–30.64), while the b* value tends to increase with rising temperature, from 10.47 at 40°C to 11.87 at 70°C, indicating a shift in color direction toward a stronger yellowish tone at high temperatures.

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To deepen the color analysis, the chroma value ($C^* = \sqrt{(a^2 + b^2)}$) and hue angle ($h^\circ = \arctan(b^*/a^*)$) were also calculated from the CIELAB data in Table 1. The C* value across the four temperature variations ranged from 31.71–32.41, while the hue angle ranged from 19.0°–21.9°, representing the typical orange-red color of sappan wood and consistent with the characteristics of brazilin oxidized into brazilein (Ngamwonglumlert et al., 2020). The relatively narrow range of hue angle variation across temperatures indicates that the color direction did not change much even though the intensity (K/S) differed, so that the change in process temperature affected color depth more than it affected the shift in color tone produced. The Eapp value shows a consistently decreasing (increasingly negative) trend as temperature rises, from –13.77% at 40°C to –102.17% at 70°C. This trend is interesting to examine because it does not align with the K/S trend, which actually increased up to 60°C before finally decreasing at 70°C.

The misalignment between the Eapp and K/S trends is based on the difference in aspects measured by the two parameters: K/S reflects the color intensity that is truly fixed onto the fiber, while Eapp describes the efficiency of dye absorption from the mother liquor. At high temperatures, increased solubility and mobility of dye molecules in the solution can accelerate the adsorption-desorption equilibrium process, such that some of the dye that had been absorbed has the potential to be released back into the solution before it is permanently fixed onto the fiber (Hassan and Saifullah, 2023). This condition may explain why the Eapp value continued to decrease even though K/S was still increasing up to 60°C, before the two parameters finally showed a more aligned trend at 70°C. This interpretation is tentative and requires further verification, given that the definition of the Eapp calculation in this study has not been precisely confirmed.

Theoretically, the K/S value obtained through the Kubelka-Munk equation ($K/S = (1-R)^2/2R$, where R is the reflectance of the fabric at the maximum wavelength) is directly proportional to the concentration of dye fixed onto the fiber (Safi and Amirshahi, 2023), so that the increase in K/S value from 40°C to 60°C can be interpreted as a direct indicator of the increasing number of brazilin/brazilein molecules that successfully diffused and bound to the cellulose structure of the cotton fabric. Conversely, the decrease in K/S at 70°C indicates that excessive thermal energy no longer contributes to increased dye absorption, but instead promotes degradative reactions that reduce the number of active chromophores both in the solution and those already fixed onto the fiber.

This tendency toward decreased color intensity at excessive temperatures is also consistent with findings from previous research on the effect of sappan wood extraction temperature on batik color quality, which reported that excessively high process temperatures can reduce color quality due to degradation of the active dye compound (Pujilestari and Salma, 2017). Although that study focused on the extraction stage while this study focuses on the dyeing stage, the similarity in the pattern of color quality decline at high temperatures reinforces the indication that brazilin/brazilein in sappan wood generally has a thermal stability limit that needs to be taken into account throughout the process chain, both at the extraction and dyeing stages.

Effect of Temperature on Color Fastness

Color fastness was tested to determine the ability of the cotton fabric dyed with sappan wood to retain its color after being subjected to washing and wet rubbing treatments. Testing was carried out on fabric dyed at four temperature variations, namely 40°C, 50°C, 60°C, and 70°C. The parameters observed included color fastness to washing and color fastness to wet rubbing. The color fastness test results for each temperature variation are presented in Table 2.

Table 2. Color Fastness Test Results of Cotton Fabric Dyed with Sappan Wood at Various Temperatures

Parameter	40°C	50°C	60°C	70°C
Washing	1	1	1	1-2
Wet Rubbing	4-5	4-5	4	4

Based on the test results in Table 2, color fastness to washing was relatively constant at grade 1 (poor) for temperatures of 40–60°C, and slightly improved to grade 1–2 at 70°C. In contrast, color fastness to wet rubbing showed a decreasing trend, from grade 4–5 at 40–50°C to grade 4 at 60–70°C. This decrease indicates that although high temperature increases the initial penetration of dye into the fiber, the dye–fiber bond on the fabric surface tends to be less stable against mechanical friction at

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higher process temperatures. In general, the color fastness values obtained in this study are consistent with the general characteristics of ultrasonic-assisted textile dyeing using natural dyes, which tend to produce good rubbing fastness but lower washing fastness due to the nature of natural dyes being bound to the fiber mainly through physical interactions and hydrogen bonding, rather than permanent covalent bonds as in synthetic reactive dyes (Islam et al., 2024). The low washing fastness across all temperature variations (grade 1–1–2) indicates that the dyeing temperature factor alone is not sufficient to significantly strengthen the dye–fiber bond, and needs to be combined with optimization of the type and concentration of the fixing agent so that the color fastness of sappan wood dye can be further improved in subsequent research.

The color fastness testing in this study refers to the grey scale standard of textile testing, which assesses the color change of the test fabric and staining on the comparison white fabric within a range from grade 1 (very large/poor change) to grade 5 (no change/very good) (D'Cruz, 2020). The consistently low washing fastness across all temperature variations (grade 1–1–2) indicates that the binding mechanism of sappan wood dye onto the cellulose fiber of the cotton fabric is dominated by physical interactions, such as hydrogen bonding and van der Waals forces, rather than the stronger covalent bonds that are more resistant to repeated washing.

Comparison with ultrasonic-assisted dyeing research on other fibers using different natural dyes shows that the rubbing fastness obtained in this study (grade 4–5) is fairly good and comparable to ultrasonic dyeing results in general, which tend to produce higher rubbing fastness compared to washing fastness due to the effect of ultrasonic cavitation on the distribution of dye on the fiber surface (Banna et al., 2023). This confirms that the main limitation in this study lies in washing fastness, not rubbing fastness, so that improvement efforts in subsequent research should be focused on the fixation stage or the addition of supplementary binding agents to strengthen the dye–fiber bond against exposure to water and detergent.

Functional Group Analysis (FTIR)

FTIR analysis was carried out to identify the functional group characteristics of the cotton fabric after the sappan wood dyeing process at various temperature variations. Testing was carried out on samples dyed at 40°C, 50°C, 60°C, and 70°C. Identification was based on the main absorption bands that appeared in each treatment. The identification of FTIR absorption bands is presented in Table 3.

Table 3. Main FTIR Absorption Bands (cm^{-1}) of Cotton Fabric Dyed with Sappan Wood at Various Temperatures

Functional Group	40°C	50°C	60°C	70°C
O–H stretching	3333	3341	3333	3312
C–H stretching	2909	2909	2898	2895
H–O–H bending	1644	1643	1647	1647
CH_2 bending	1416	1444	1428	1427
C–H/O–H bending	1365	1363	1363	1360
C–O–C glycosidic	1168	1157	1157	1165
C–O stretching	1044	1044	1043	1024
β-glycosidic	891	894	881	884

The FTIR analysis results for the four temperature variations (Table 3) show a relatively consistent absorption pattern, with no new absorption bands appearing and no existing bands disappearing, indicating that the cellulose structure of the cotton fabric did not undergo degradation due to the process temperature variation. The O–H stretching band was observed to shift from 3341 cm^{-1} (50°C) to 3312 cm^{-1} (70°C), which may indicate a weakening of the hydrogen bond interaction between the hydroxyl groups of cellulose and the dye molecules at high temperatures. The shift in the CH_2 bending band (1416→1444→1428→1427 cm^{-1}) further supports the indication of a change in the interaction between the dye and the fiber, although it does not show a fully linear pattern with respect to temperature (Rotich, Wangila and Cherutoi, 2022).

The finding of consistency in the FTIR absorption pattern in this study is in line with the results

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of research on ultrasonic-assisted dyeing of cotton fabric using a different natural dye, which also reported that the ultrasonic-assisted dyeing method did not produce new functional groups or significant band shifts in the cellulose structure of the fabric, thereby indicating that the ultrasonic energy and process temperature variations used did not cause chemical degradation of the fiber. Thus, the temperature variation of 40–70°C in this study can be categorized as a process condition that is safe for the chemical structure integrity of the cotton fabric, so that the observed differences in dyeing results are purely caused by differences in the physical interaction between the dye and the fiber, rather than by damage to the fiber structure itself.

The absence of new absorption bands or drastic band shifts in the FTIR spectrum also reinforces the findings in the color fastness section, namely that the interaction between the sappan wood dye and the cellulose fiber of the cotton fabric is physical in nature (physisorption) through hydrogen bonding, rather than a chemical interaction (chemisorption) involving the formation of new covalent bonds. If chemisorption occurred, new absorption bands or significant wavenumber shifts would generally be observed due to the formation of new functional groups resulting from the reaction between the dye and cellulose, whereas the pattern observed in this study shows small shifts that are more consistent with changes in hydrogen bond strength due to temperature variation.

This physical binding nature coherently explains why the sappan wood-dyed fabric in this study has relatively low washing fastness, because hydrogen bonds are more labile and are easily broken by water and mechanical agitation during the washing process compared to covalent bonds. This finding forms an important basis for the development of fixation strategies in subsequent research, for example through the use of metal mordants or crosslinking agents capable of forming more permanent chemical bonds between the dye and the fiber (Benli, 2024).

Determination of Optimum Temperature

Based on the combination of the K/S, CIELAB, and color fastness parameters, 60°C showed the best performance in terms of color strength, although accompanied by a slight decrease in wet rubbing fastness compared to 40–50°C. Therefore, 60°C can be considered as the optimum temperature for the ultrasound-assisted dyeing process of sappan wood, with a trade-off in wet rubbing fastness that remains within the good category (grade 4). From the perspective of application on a wider scale, the selection of the optimum temperature needs to consider not only color strength, but also energy efficiency and the consistency of the resulting color fastness. A temperature of 50°C can be considered as an alternative if the main priority is to maintain wet rubbing fastness at a higher level (grade 4–5), although with the consequence of a slightly lower K/S value compared to 60°C. Further research is recommended to test the combination of the optimum temperature with variations in the type and concentration of fixing agents, in order to improve the washing fastness that is still relatively low across all temperature variations tested.

In terms of energy efficiency, the use of a temperature of 60°C is also relatively advantageous compared to 70°C, because it is able to produce the highest color strength without requiring additional heating energy. This principle is in line with one of the main advantages of the ultrasonic dyeing method compared to the conventional method, namely its ability to achieve optimal dyeing results at a relatively moderate process temperature thanks to the acoustic cavitation effect, which increases the rate of dye diffusion without relying entirely on thermal energy (Hassan and Saifullah, 2023). Thus, the application of a temperature of 60°C is not only optimal in terms of color quality, but also has the potential to support the principle of more energy-efficient and sustainable textile dyeing when applied on a larger production scale, for example in the natural-dye batik industry.

CONCLUSION

The test results show that temperature variation in the ultrasonic dyeing process affects the dyeing characteristics of cotton fabric using natural sappan wood (Caesalpinia sappan L.) dye powder. The K/S value increased from 2.105 at 40°C to 2.627 at 60°C, then decreased to 2.146 at 70°C. The decrease in the K/S value at 70°C is based on changes or thermal degradation of the brazilin/brazilein compound at higher temperatures. The CIELAB values show a relatively similar color direction across all treatments, with the hue angle in the range of 19.0°–21.9°. Meanwhile, the Eapp value decreased with increasing temperature, although this pattern did not show the same tendency as the K/S value. Color fastness to washing was at grade 1-1-2 across all temperature variations, while color fastness to wet rubbing was at grade 4-5 at 40-50°C and decreased to grade 4 at 60-70°C. The FTIR analysis

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results showed a relatively consistent absorption band pattern across all treatments, and no new absorption bands were found. This indicates that temperature variation during the ultrasonic dyeing process did not cause significant changes in the functional groups of the cotton fabric. A temperature of 60°C produced dyeing results with the highest K/S value, so it can be used as the optimum temperature in the ultrasonic dyeing process using sappan wood dye powder. Although wet rubbing fastness decreased slightly at this temperature, its value remained at grade 4. The use of different types and concentrations of fixing agents can be considered to improve color fastness to washing.

DECLARATION OF GENERATIVE AI

The authors declare that no generative AI or AI-assisted technologies were used in the writing process of this manuscript.

CONFLICTING INTEREST STATEMENT

The author declares that there is no conflict of interest in the publication of this article.

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