



## Utilization of Chili Citrus (*Citrus microcarpa*) as a Natural Fixator for Dyeing Cotton Fabrics with Dayak Onion Tuber Extract (*Eleutherine palmifolia* (L.) Merr)

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### Abstract

The high demand for the textile industry can pollute the environment through waste. Natural dyes from natural ingredients are used as a step to minimize this waste. Dayak onions can be used as a natural dye because they produce a red color from anthocyanin compounds. However, natural dyes have weaknesses, namely that the color is less stable and fades easily, so a fixator is needed. Fixator is used to strengthen the color in fabrics that use natural dyes. Orange chili sauce has the potential to be a natural fixator because it contains citric acid. This research aims to analyze the effect of the concentration of chili pepper fixator (*Citrus microcarpa*) on the color fastness of cotton fabric using natural dyes from Dayak onion bulbs (*Eleutherine palmifolia* (L.) Merr). The research method used was an experiment using a colorimeter application whose results were in the form of  $\Delta E$  values. The test was carried out on the color of the fabric that had been dyed and fixed and then the average value ( $\Delta E$ ) was measured before and after washing the soap. The research results showed that the average value ( $\Delta E$ ) of the smallest to largest fixator was a concentration of 20%, 100%, 0%, 40%, 60% and 80%. Based on the results of the Kruskal Wallis test followed by the Bonferroni test, a significant value of 0.024 ( $p > 0.05$ ) was obtained at a concentration of 20%. This shows that the chili lime fixator has a significant influence on the fastness of the fabric.

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## INTRODUCTION

The textile industry is a major contributor to environmental pollution in many countries, such as China and South Africa (Olisah et al., 2021). The pace of globalization and technological developments has resulted in rapid growth of the textile industry in Indonesia. Dyes used in the textile industry as fabric dyes are the main cause of pollution (Dutta et al., 2024). Every year the textile industry produces more than 70 million tons of synthetic dyes (Chandanshive et al., 2020). These synthetic dyes can produce waste containing fiber reactive dyes, thereby polluting the water. (Firdous et al., 2024).

Waste containing synthetic dyes can affect human health because it contains a variety of persistent pollutants (Ali et al., 2002). One of them is azo dyes which account for 50% of all dyes used widely throughout the world (Zafar et al., 2022). Around 15–50% of azo dye waste from the textile industry is not bound to fibers and fabrics and is therefore discharged into the water (Al-Tohamy et al., 2022). Textile dye waste that is discharged into waters such as lakes, rivers, streams and ponds poses an ecotoxicological threat to living organisms (Parmar et al., 2022). This has an impact on disturbed water ecosystems, such as disrupting the growth of aquatic plants and poisoning animals that live in the water (Elgarahy et al., 2021). The impact

of these synthetic dyes can be felt by humans through direct contact with the skin which can cause irritation (Manickam & Vijay, 2021).

One effort to minimize textile dye waste is to use natural dyes that come from natural and environmentally friendly materials (Enrico, 2019). According to Musripah (2019), natural dyes from plants have the advantage of being easily degraded and decomposed in the environment. However, natural dyes have weaknesses, namely the color is less stable, fades easily, requires a long processing process and the resulting color spectrum is limited (Priambudi et al., 2020). Therefore, a fixator is needed to strengthen the color produced by natural dyes thereby increasing the absorbency of the fabric (Nofiyanti et al., 2018). Plants that have the potential to become natural dyes are Dayak onions (*Eleutherine palmifolia* (L.) Merr). Dayak onions are one of the plant species of the Iridaceae family which has been used as a cosmetic and food coloring (D. Mayasari et al., 2018). Dayak onions have the potential as a natural dye because they contain anthocyanin compounds that produce a red color (Refordayanti et al., 2021).



Figure 1. Dayak onion bulbs

A fixator is a substance added during the fixation process to strengthen the color in the fabric fibers so that a stable color is produced (Wicaksono & Russanti, 2020). According to their origin, fixators are divided into two types, namely synthetic fixators and natural fixators (Farida et al., 2016). The use of synthetic metal fixators causes poisoning if the prescribed dose is exceeded and pollutes the environment (Choudhury, 2018). Waste produced using synthetic fixators contains heavy metals such as Fe, Cu and Sn which are ecosystem pollutants. In an effort to prevent environmental pollution, natural fixators are used which are more environmentally friendly and easy to find.



Figure 2. Orange sambal

Chili pepper (*Citrus microcarpa*) has the potential to be a fixator for fabric dyeing. Sambal oranges are often found in the West Kalimantan area. According to Junaidi (2011), orange chili sauce contains 2.81% citric acid. Orange chili sauce has a pH of 2.64 (E. Mayasari et al., 2020). This refers to the opinion of Adriani & Atmajayanti (2023) that the pH of the solution can influence the color density of the fabric. When the pH of the solution is high the resulting color tends to be light and at low pH it produces a dark color.

Based on the literature, no research has been conducted that examines the use of chili peppers (*Citrus microcarpa*) as a fixator for dyeing cotton fabrics with Dayak onion (*Eleutherine palmifolia* (L.) Merr) tuber extract. Previous research on Dayak onions has been used as a natural coloring in cosmetic products by D. Mayasari et al. (2018) with a product in the form of lipstick, while the use of chili lime as a natural fixator has never been done.

The orange fixator that has been used previously is lime by Andansari & Nadir (2017) using several types of cloth. Thus, this research was carried out with the aim of finding alternative natural fixators from chili peppers and natural dyes from Dayak onion bulbs in fabric processing. It is hoped that the results of the research can become a reference source for natural materials to overcome fading in fabrics.

## **METHOD**

The research used is experimental research. The aim of this research was to determine the effect of chili lime fixator on the coloring of cotton fabric with Dayak onion (*Eleutherine palmifolia* (L.) Merr) tuber extract. The indicator used to see the effect of chili lime fixator on fabric coloring with Dayak onion bulb extract is color fastness. The color fastness test is carried out by observing the color changes in the fabric during the washing process. The test was carried out using a colorimeter (Lab Tools Apps, Playstore).

### **Time and Place of Research**

This research was conducted at the Chemistry Education Laboratory, Faculty of Education and Science, Tanjungpura University, Pontianak for  $\pm$  4 months.

### **Tools and Materials Used**

The tools used in the research were analytical balance (Aduanx Innotech), mini studio box and colorimeter (Lab Tools Apps, Playstore). The materials used in this research were Dayak onion bulbs (*Eleutherine palmifolia* (L.) Merr) as a natural coloring taken in Pontianak, West Kalimantan and a fixator in the form of chili sauce taken in Kubu Raya, West Kalimantan. Other materials, namely cotton cloth, alum, soda ash, TRO and bar soap.

### **Research Procedures**

#### ***Collection and Preparation of Plant Samples***

Samples in the form of Dayak onion bulbs were collected from the Pontianak area, West Kalimantan, while sambal oranges were taken in Kubu Raya, West Kalimantan. Samples of Dayak onion bulbs and chili peppers were cleaned from dirt and unwanted parts using running water. Dayak onion bulbs are dried by airing at room temperature. Dried Dayak onion bulbs are then cut into small pieces.

#### ***Making Dye Extraction of Dayak Onion Bulb Samples***

A total of 200 grams of dried Dayak onion bulbs are boiled in 2 liters of water for  $\pm$  1 hour or until half of the initial volume. The extract was left for 24 hours then filtered with filter paper. The liquid extract obtained is ready to be used as a fabric dye.

#### ***Making Orange Chili Fixator (*Citrus microcarpa*)***

The chili orange fixator is obtained by squeezing the chili orange fruit with a squeezer then filtering it with filter paper and a fixator with a concentration of 100% is obtained. Fixator with concentrations of 80 mL, 60 mL, and 40 mL, 20 mL of 100% orange juice with water to a volume of 100 mL.

## **Fabric Dyeing**

The fabric dyeing process consists of 4 stages.

### ***Scouring***

The cotton fabric scouring process refers to Astuti & Widiastuti (2021) as much as 2 grams of TRO dissolved in 1 liter of water. 20 pieces of cotton cloth measuring 10 cm x 10 cm were soaked in 0.2% TRO solution for 15 minutes then rinsed with running water and dried in the sun until dry.

### ***Mordant***

The mordant process refers to Kharisma & Sudiarmo (2020) as much as 84 grams of alum and 16 grams of soda ash in 1 liter of boiling water. The cloth is put into the solution and boiled for 1 hour. The stew is left for 24 hours in a closed container, rinsed and dried in the sun by airing.

### ***Fabric Dye***

Dyeing cotton fabric is done by soaking cotton fabric that has been given mordant in the form of Dayak onion bulb extract for 24 hours. After 24 hours, the cotton cloth is dried in the sun by airing it.

### ***Fixation***

After the cloth is dry, it is fixed with chili lime juice with concentrations of 0%, 20%, 40%, 60%, 80% and 100% by soaking the cotton cloth in chili sauce solution with various concentrations. After 15 minutes, dry the cotton cloth in the sun until it is dry.

## **Color Fastness Test**

The color fastness test of cotton fabric by dyeing Dayak onion bulb extract with chili lime fixator was carried out using a Samsung A20s cellphone camera. When taking pictures the application will stand by in live mode (Nairfana & Rizaldi, 2022)). Color measurements on cotton fabric that has been fixed are carried out using the colorimeter application (Lab Tools Apps, Playstore) which has been downloaded on the cellphone. Measurements were carried out by repeating three times. The color fastness test was carried out by washing with a solution of bar soap, soaking the fabric for 5 minutes and drying in the air.

The color components to be measured are brightness ( $L^*$  value), green-red ( $a^*$ ) and blue-yellow ( $b^*$ ). The CIE values, namely  $L^*$ ,  $a^*$  and  $b^*$ , were obtained from photographs of cotton fabric using a colorimeter application (Saputri et al., 2023). Based on the  $L^*$ ,  $a^*$  and  $b^*$  values, the  $\Delta E$  value will be calculated, here is the formula to determine the color fastness of fabric (Kartina et al., 2022)

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

While,  $\Delta E$  = Color Difference;  $\Delta L^*$  = Brightness Difference ( $L^*$  before –  $L^*$  after);  $\Delta a^*$  = Red to green color difference ( $a^*$  before –  $a^*$  after);  $\Delta b^*$  = Difference between yellow and blue ( $b^*$  before –  $b^*$  after)

## **Data analysis**

Data analysis was carried out to see the effect of variations in the concentration of chili lime fixator using natural dye Dayak onion bulb extract using the CIELAB method consisting of three values, namely  $L^*$  represents brightness,  $a^*$  represents color in the green-red range and  $b^*$  represents color in the blue-yellow range. The test was carried out using a colorimeter application (Lab Tools Apps, Playstore) to obtain the  $\Delta E$  value. Data are presented as mean  $\pm$  SD then analyzed using the Kruskal Wallis test and followed by the Bonferroni test to determine the optimal fixator concentration.

## RESULTS AND DISCUSSION

Dyeing using Dayak onion bulb extract produces colors that can be used to dye cotton fabric. Based on the opinion of Refordayanti et al. (2021) Dayak onions contain anthocyanin pigments which can produce color. According to Hidayat et al. (2022) Dayak onions have anthocyanins of 4.3 mg/100 g. This content is what makes Dayak onions have a maroon or brownish red color (Senorita et al., 2018). The color is produced after going through the process of scouring, mordanting and dyeing using Dayak onion bulb extract to produce a pink color. The process of scouring and mordanting the fabric functions to open the pores of the cotton fabric and sharpen the color of the fabric thereby forming a chemical bridge between the natural dye and the fiber so that the absorbency of the dye increases (Utami et al., 2023). The mordant process is carried out using alum and lime (CaO) mordant substances. Maghfiroh & Widowati (2020) said that the mordant was chosen because it has a strong bond to the dye due to the presence of covalently bonded metal compounds. This is based on the opinion of Utami et al. (2023) the mordant process is carried out to form a bridge between the fabric fibers and natural dyes so that the absorbency of the fabric increases.

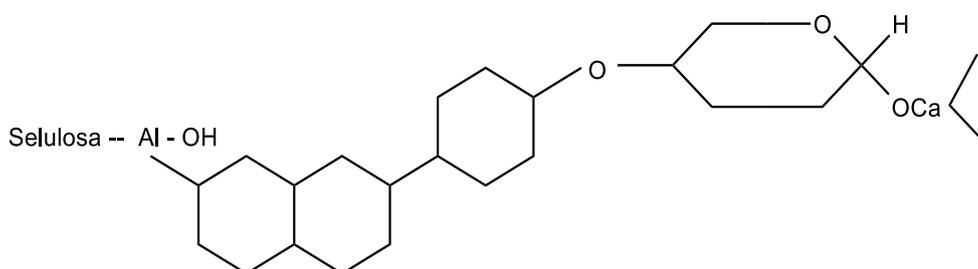


Figure 3. Chemical bonds in the mordant process (Harmini, 2011)

According to Adelina et al. (2014), alum produces a color that tends to be the same as the original color, while tunjung produces a darker color. This is caused by alum which has tannin bonds with  $Al^{3+}$  metal ions, while tunjung has bonds between tannin and  $Fe^{2+}$  metal ions so that a complex salt (ferrous tannate) is formed through coordinating covalent bonds between metal and non-metal ions (Hasanudin & Widjiati, 2002).

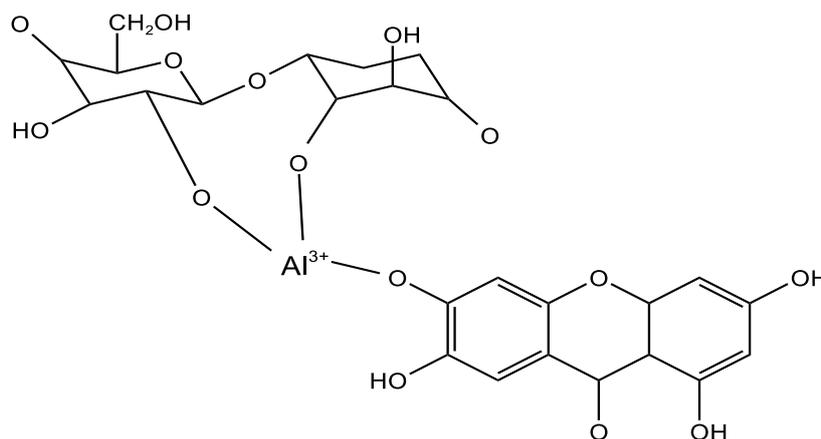
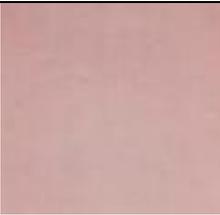
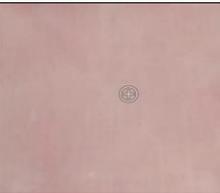
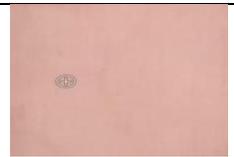


Figure 4. Covalent bonds of aluminum (from alum) and oxygen from (filament-cellulose fiber and dye-tannin) (Lalang et al., 2023)

In the fixation process, natural ingredients derived from orange chili sauce are used. Natural fixators that have been used previously are lime, vinegar, palm sugar, lime and molasses (Farida et al., 2016). The fixation process aims to strengthen the color of the fabric and determine the direction of the fabric color (Setiyani & Yulistiana, 2023). The fixation process uses a hot lime fixator with concentrations of 0%, 20%, 40%, 60%, 80% and 100%. Fixator

variations were carried out to determine the effect of concentration on the fastness of cotton fabric using natural dyes from Dayak onion bulb extract. Then the color fastness test was carried out to soap washing. The purpose of a color fastness test is to determine color changes in dyed fabric (Putri et al., 2023). The results of visual staining obtained a pink color (table 1).

Table 1. Staining cotton fabric using Dayak onion bulb extract with chili orange fixator before and after washing with soap

Fixation Concentration (%)	Before Washing Soap	After Washing Soap
0	 <i>Light Pink</i>	 <i>Pink</i>
20	 <i>Light Pink</i>	 <i>Light Pink</i>
40	 <i>Wheat</i>	 <i>Vanilla Ice</i>
60	 <i>Wheat</i>	 <i>Vanilla Ice</i>
80	 <i>Wheat</i>	 <i>Light Pink</i>
100	 <i>Wheat</i>	 <i>Vanilla Ice</i>

Testing the color fastness of cotton fabric uses the CIELAB method, which is a space that includes all colors that can be perceived directly by the eye (Pujiwisata et al., 2016). The color

space consists of three values, namely  $L^*$  represents brightness,  $a^*$  represents colors in the green-red range and  $b^*$  represents colors in the blue-yellow range. Based on Putra et al. (2012) if the  $L^*$  value is high, the color will be brighter. The  $L^*$  value ranges from 0-100. A value of 0 means dark while 100 means bright. The  $a^*$  (green-red) value if  $+a^*$  between 0-60 leads to red while the  $-a^*$  value of 0- (-60) leads to green. The  $b^*$  value (blue-yellow) if the  $+b^*$  value from 0-60 leads to yellow while the  $-b^*$  value from 0-(-60) leads to blue. The results of the average values are shown in (table 2).

Table. 2 Average Values of Cloth Before and After Soap Washing on Dayak Onion Bulb Samples Using Sambal Orange Fixator

Concentration (%)	$L^*$		$a^*$		$b^*$	
	Before	After	Before	After	Before	After
0	79.5	83.4	23.7	19	10.7	5.6
20	79.3	79.2	15.4	17.1	8.7	8.6
40	81.7	81.7	13.7	14	15.5	5.8
60	83.9	83.6	14.5	14	15.5	5.8
80	81.2	83.6	14.2	14	15.8	5.8
100	81.2	83.2	14.2	9.9	15.8	7

Based on (table 2), the  $L^*$  value for cotton fabric is towards bright or white because it is close to 100. This is influenced by the strength of the bond between the fabric fibers from natural dyes that have gone through a fixation process (Ramadhan et al., 2020). The red-green color value ( $a^*$ ) in the graph before and after shows changes at concentrations of 0%, 20% and 100%, while at concentrations of 40%, 60% and 80% there is no significant change. According to Kasmudjo & Saktianggi (2019), color fastness to soap washing is influenced by the strength of the bond between the fiber and the dye in the fabric. The yellow-blue color value ( $b^*$ ) on the graph before soap washing is higher than after soap washing. This is in accordance with research conducted by Anggraeni et al. (2023). Based on Haerudin et al. (2020) the color becomes darker after washing due to the interaction between soap and natural dyes in the fabric fibers. Alkaline soap causes increased solubility of natural dyes. Anthocyanin in Dayak onion bulbs has a phenol group in the form of hydroxyl (-OH) which is bound to a benzene ring so it is very easy to ionize in alkaline conditions which increases the solubility of natural dyes.

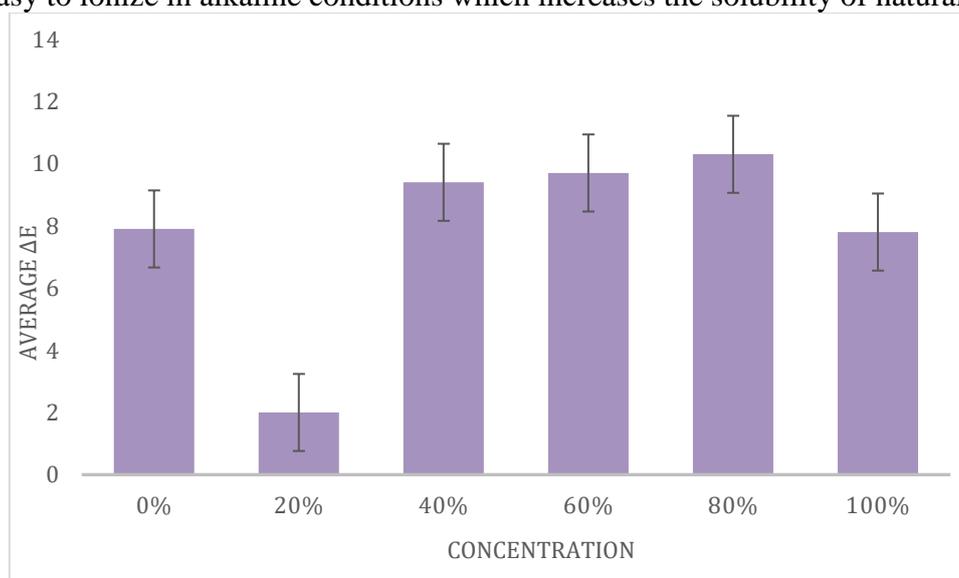


Figure 3. Graph of Average Color Difference Value ( $\Delta E$ )

Based on (figure 5), the smallest  $\Delta E$  value when repeated three times at a concentration of 20% has a value of  $2 \pm 0.42$  so that the fade on the fabric is low. This refers to the opinion of

Anggraeni et al. (2023) the  $\Delta E$  value is influenced by the  $L^*$ ,  $a^*$ , and  $b^*$  values. If the  $\Delta E$  value is large, the fabric fade is high, while the  $\Delta E$  value is small, the fabric fade is low.

Hypothesis testing of the effect of chili lime fixator concentration on the color fastness of cotton fabric using natural Dayak onion bulb extract dye was carried out using non-parametric ANOVA analysis, namely Kruskal Wallis with a significance level of  $\alpha = 0.05$  in SPSS for data processing. Kruskal Wallis was carried out as an alternative to the one way ANOVA test and the normality test showed that the data was not normally distributed. In the Kruskal Wallis test, the significant value obtained was 0.024 ( $p > 0.05$ ), which stated that the chili orange fixator had an influence on the color fastness of cotton fabric, so the Bonferonni test was carried out to determine the best concentration for color fastness. Based on the results of the Bonferonni test, it was found that a concentration of 20% was the best concentration with a significant  $p < 0.05$ . This can be seen from (figure 3) the graph of the average color difference value ( $\Delta E$ ) at a concentration of 20%, the  $\Delta E$  value is low so that the fade on the fabric is low.

## CONCLUSION

Based on the research results, it can be concluded that the color fastness test on cotton fabric before and after washing has an effect so that the chili orange fixator which uses natural dye extract from Dayak onion bulbs (*Eleutherine palmifolia* (L.) Merr) can resist the color fading of the fabric. Based on the test results, the best  $\Delta E$  value was at a concentration of 20% with  $2 \pm 0.42$ , so that chili sauce can be used as an alternative natural fixator. This can be an alternative to using fixators made from natural materials to minimize environmental pollution and utilize local resources.

## RECOMMENDATIONS

A fastness test is required using a more quantitative method using a gray scale and a fastness test method to dry rubbing, drying, staining, etc. that is in accordance with SNI.

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