Approach Toward Natural Dyes for Coloring Songket Ikat Woven Using Hanjuang (*Cordyline fruticosa*) Extract

Dhony Hermanto[1](✉), Baiq Handayani Rinuastuti[2], Nurul Ismillayli[1], Handa Muliasari[3], Ulul Khairi Zuryati[4], and Rochmad Kris Sanjaya[5]

[1] Department of Chemistry, Faculty of Mathematics and Natural Sciences, University of Mataram, Mataram, Indonesia
{dhony.hermanto,nurul.ismillayli}@unram.ac.id

[2] Department of Management, Faculty of Economics and Business, University of Mataram, Mataram, Indonesia
hrinuastuti@unram.ac.id

[3] Department of Pharmacy, Faculty of Medicine, University of Mataram, Mataram, Indonesia

[4] Laboratory of Analytical Chemistry, Faculty of Natural Sciences, University of Mataram, Mataram, Indonesia
ululkhair@unram.ac.id

[5] Department of Medical Laboratory Technology, Faculty of Health Science, University of Kadiri, Kediri, Indonesia
rochmad@unik-kediri.ac.id

Abstract. Sukarara Village is a woven fabric tourism village on Lombok Island with featured songket and ikat weaving products. Natural dyes in woven fabrics are popular because they are more environmentally friendly, and the colours are more natural than synthetic dyes. However, natural dyes fade quickly, colour consistency is low, and colour variation is limited. This problem was overcome using a pH-based yarn staining technique using Hanjuang leaves (Cordyline Fruticosa). Anthocyanin solutions were varied from pH 2 to 10 using citrate-phosphate buffer solution and potash alum as the mordanting agent. RGB, UV-Visible, and FTIR spectrophotometric analyses were performed to study the effect of pH on anthocyanins and the effect of mordanting agents on the binding of anthocyanin colour. The colour of the anthocyanin solution shifted from red to yellow and turquoise with increasing pH from 2 to 10. Mordanting agents containing metal ions could bridge the binding of anthocyanins with cellulose in yarn by forming a coordination complex. The RGB value of yarn after immersion in anthocyanin solution is higher than the anthocyanin solution indicating more vigorous colour intensity was obtained when it was applied to yarn due to stronger alum-anthocyanin interaction with alum-yarn. Therefore, this method can be used as an alternative for textile colouring using anthocyanin pigments.

Keywords: Anthocyanin · songket · ikat · weaving · Sukarara

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1 Introduction

Crafts generally have a high cultural value and are essential to the nation's economic development. The cultural values of handicraft products are revealed in distinctive patterns, styles, techniques, and patterns that indicate the origin, history, social relations, and ways of life of the community [1], such as in the craft of songket and ikat weaving typical of Sukarara Village, Central Lombok. Songket weaving is a woven fabric made from threads decorated with gold or silver-coloured synthetic threads, with a manufacturing time of about one week to one month to complete one sheet of woven fabric. While ikat weaving usually uses cotton thread with a shorter processing time, one to several days for a piece of cloth. The uniqueness of Sukarara woven fabric that distinguishes it from other regional woven fabrics, in addition to motifs and symbols, is a variety of colour variations so that the woven fabric looks beautiful and charming.

Yarn dyeing as a raw material for woven fabrics is essential in producing beautiful and quality woven fabrics. Good dyeing is not easy to fade and does not damage the thread, so the thread is not brittle or easily broken [2]. Fabrics using chemical dyes will significantly pollute the environment because the dye waste disposed of will pollute the surrounding environment, especially water [3]. In addition, synthetic dyes pose a direct hazard to human health, indicating that they contain carcinogenic chemicals [4, 5]. On the other hand, natural dyes are becoming known and popular nowadays, especially as consumers are increasingly concerned about the issue of environmental pollution due to the use of synthetic dyes and the rise of the "Back to Nature" brand as an elegant product. However, natural dyes have some limitations, especially their low consistency and limited colour variations. So far, the colouring process using natural dyes is more complicated than dyeing using synthetic dyes [6, 7], so it is considered less practical.

Recently, various types of plants that have the potential to be used as textile colours have attracted researchers. It is to increase the amount of availability and types of natural dyes that can be used as textile dyes. In addition, the technique/method of staining is also getting attention to get more optimal staining results in a slightly shorter time. Processing or taking natural dyes from plants is done in 2 ways; extraction and fermentation [8]. The anthocyanin pigments in hanjuang leaves can be used as natural dyes with more varied and attractive colours [9, 10]. Anthocyanins are unsaturated organic substances and belong to the flavonoid group. Its main structure is characterized by two benzene aromatic rings (C_6H_6) linked by three carbon atoms. The three carbon atoms are bonded by an oxygen atom, thus forming a ring between the benzene rings. In solution, anthocyanins exist in five equilibrium states depending on pH conditions. The five forms are flavilium cation, carbinol base, chalcone, quinoidal base, and anionic quinoidal [11]. At very acidic pH (pH 1–2), the dominant form of anthocyanins is the flavilium cation. In this form, anthocyanins are in the most stable and colourful state. When the pH increases above 4, it takes the form of yellow anthocyanin compounds (chalcone form), blue compounds (quinoids form), or colourless compounds (carbinol bases) [12, 13]. In other words, different pH treatments can produce different colours according to the stable anthocyanin form under these conditions [14]. Anthocyanins are hydrophilic, meaning they can dissolve in water so that during the washing process, the colour absorbed by the yarn (as a raw material for woven fabrics) can be separated from the fabric fibres causing the colour to fade. Therefore, a fixation process is needed, namely the binding of dyes using a fixator [15, 16] such as potash alum. The content of metal ions in the fixator can react to form complex compounds with anthocyanin dyes [17] so that they remain in the fabric fibres. In other words, they do not fade when washed. For that purpose, hanjuang leaves are used as natural dyes in yarn and silk, knowing the colours produced at variations in pH, and potash alum as a fixator. Dyeing is carried out using the room temperature method correctly to get optimal colouring results and reduce energy consumption.

2 Method

A. *Chemicals*

The hanjuang plant is grown and cultivated locally in Lombok, West Nusa Tenggara. For this purpose, the hanjuang leaves are extracted using water as a solvent. The woven yarn used in this study was hand-spun cotton yarn 12S for weaving purchased from the local trader in Lombok as mordanting agent potash Alum [$K_2Al_2(SO_4)_3.24H_2O$] is used and procured from Merck, Germany. All other reagents used were of analytical grade. Distilled water was used as the primary solvent.

B. *Instrumentations*

The colour produced from natural dyes is visually observed regarding the colour produced. To test the quality of the staining results, a colour difference test was carried out for each treatment using a UV-Visible Spectrophotometer (Labo-Hub, China) and image colour analysis (ImageJ) as the average RGB value. Meanwhile, the functional group test to study the interaction between matrix and colour was used by Fourier Transform Infra-Red (FTIR) (Perkin Elmer, USA).

C. *Hanjuang Extract Preparation*

Anthocyanin solution was obtained from red leaf extract of hanjuang. Initially, the hanjuang leaves were washed under running tap water and rinsed with distilled water. Hanjuang leaves are left overnight at room temperature to reduce the moisture content. The next step is to chop the leaves as small as possible and immerse them in a solvent with a mass ratio of leaves and solvents is 1:2. The solvent used was a mixture of distilled water and 37% HCl with a volume ratio of 99:1. The extraction process was carried out in a closed container, dark room at room temperature for one night. In the final stage, the extraction results are filtered using Whatman filter paper no. 41, and its filtrate is used as a natural dye for yarn. The proposed natural dye was tested in a citrate-phosphate buffer solution with a pH range of 2–10, and a colour change was observed.

D. *Colouring Process*

The dyeing process is carried out in ambient conditions to reduce heat energy, with the following steps: the initial stage of the woven yarn is activated first with 0.1 M

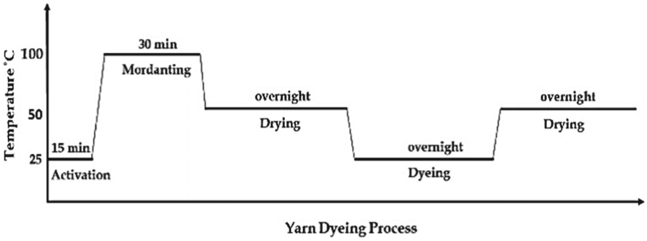


Fig. 1. Diagram of Yarn Dyeing Process

HCl, followed by 0.1 M NaOH, then rinsed with distilled water. The next step is to soak the samples in 15% potash alum as mordanting agent solution while heating it at a temperature of 90–100 °C for 30 min. The samples are drained and dried overnight. Ready-to-dye woven yarn was placed in a 500 mL glass beaker and immersed in a hanjuang leaves extract at various pH variations from 2 to 10. For this purpose, the weight ratio between the samples and the natural dye solution was 1:1, and the soaking was carried out overnight. Then the samples were washed and air-dried. Post-mordant was carried out on samples stained using the same type of mordant agent and concentration as that used in pre-mordant under ambient conditions for 30 min. Finally, the dyed threads were drained and dried under ambient conditions. The thread colour was observed visually and quantitatively measured using a UV-Visible spectrophotometer and ImageJ software. The yarn dyeing process is described in Fig. 1.

3 Results and Discussion

Hanjuang leaves extracted with water solvent in acidic conditions give a dark red colour. Hanjuang leaves extract testing was carried out in citrate-phosphate buffer pH 2 to 10. The anthocyanin colour of Hanjuang leaves extract was found to differ according to the number of hydroxyl groups (-OH) bound to the molecule (especially those bound to the second aromatic ring attached to the chroman ring on the molecule). The position of the C2 atom. The more hydroxy groups, the colour will shift from red to blue. Four anthocyanin molecular structures have been identified: flavilium cation, quinoidal base, pseudo basic carbinol, and chalcone [18, 19]. At equilibrium, anthocyanins have different chemical structures depending on the pH solution [20].

Anthocyanins are generally more stable in acidic solutions than in neutral or alkaline solutions. In an acidic medium (pH $<$ 1), anthocyanins in flavium cations are the only species stable at that equilibrium, and it is red. At pH 2–4, anthocyanins are a mixture of flavinium and quinoidal cations. At a higher pH of 5–6, there are two colourless compounds, pseudo basic carbinol and chalcone. According to Castañeda-Ovando [13], below pH 3, the anthocyanin solution showed the most intense red colour. It was also seen from the application of hanjuang leaf extract at pH 2 and 3, which showed a red colour. As pH is raised, the red colour usually fades to a colourless appearance in the pH range of 4 to 5. As the pH of the solution increases, its concentration and pigmentation decrease as the cations are hydrated to a colourless carbinol base. Chalcone and quinoid anhydrobases begin to form at a pH close to the equilibrium of flavylium cations and

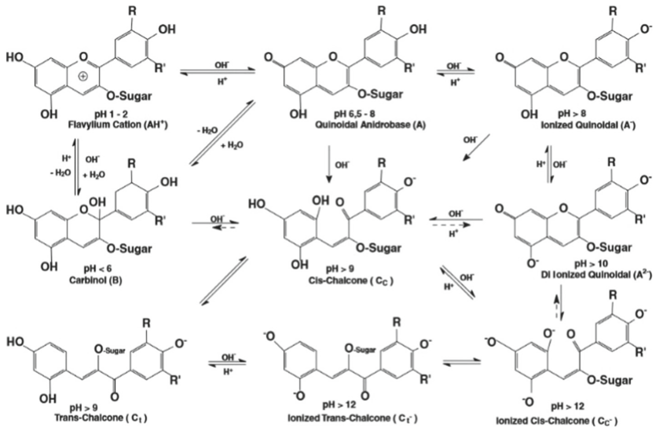


Fig. 2. Anthocyanin structure in pH-dependent speciation balance was suggested in previous studies [22]

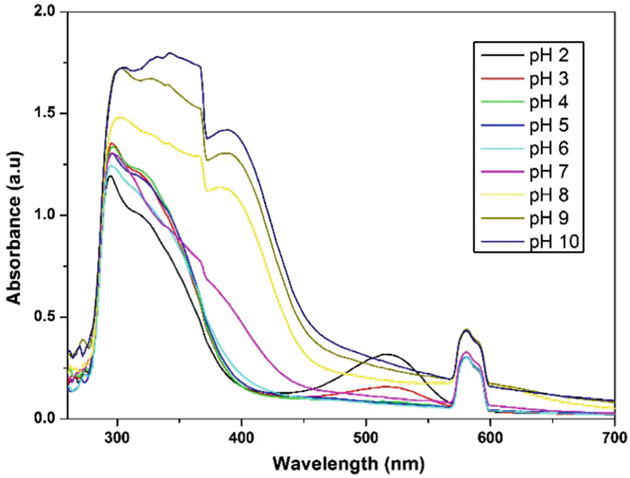


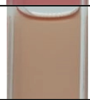
Fig. 3. Scanning of the wavelength of hanjuang leaves extract

alkaline carbinol. With increasing pH, the concentration of chalcone, quinoidal base, and carbinol increased while the flavylium cation decreased [21] until it reached a minimum amount at pH 4–5. Low concentrations of these two-coloured species (carbinol and flavylium cations) caused less pigmentation in the solution, as indicated by the hanjuang leaves extract at pH 4, and 5 was a pale red. Further increasing the pH, the concentration of chalcone, quinoidal base, and carbinol was more dominant, resulting in the colour of hanjuang leaves extract at pH 6 to 10 being blue to bluish green. Changes in pH cause reversible structural anthocyanin changes, as shown in Fig. 2. These changes and their distribution are characterized by intensity changes and maximum wavelength shifts shown in Fig. 3.

Anthocyanin compounds generally have a maximum wavelength in the 400–600 nm region. Based on Fig. 3, the anthocyanin spectrum of hanjuang leaf extract shows a maximum wavelength shift in line with changes in pH. At pH 2 and 3, anthocyanins have a maximum absorption peak at a wavelength of 525 nm and a decrease in absorbance to pH 4. It indicates a change in the anthocyanin structure of the dominant red flavilium cation. The increase in pH causes a bathochromic shift to a wavelength of 570–600 nm.

At pH 8 to 10, the red flavilium cation changes to a quinoidal base through the loss of one proton, and then this cation converts to carbinol by the nucleophilic addition of water. Above pH 7, pigmentation changes from blue to bluish-green due to chalcone

Table 1. Value RGB and colour change at the pH of the solution

Sample	Value			Photograph
	R	G	B	
Extract	116	47	64	
pH 2	160	29	47	
pH 3	171	44	71	
pH 4	171	100	100	
pH 5	168	138	114	
pH 6	150	115	96	
pH 7	32	42	52	
pH 8	20	43	37	
pH 9	27	49	47	
pH 10	25	51	48	

formation through cleavage of the anhydrobase ring. Chalcone, a colourless compound, can be ionized to form a bluish-green compound. The colour change of hanjuang leaf extract at various pH variations from 2 to 10, as well as the value of the Red-Green-Blue colour composition, commonly known as RGB, can be observed in Table 1.

The results of the FTIR analysis (Fig. 4) on the woven yarn show the IR spectra of the specimen before treatment (Fig. 4a) and after activation and mordanting (Fig. 4b). The peak value of the spectrum in the woven yarn before treatment was defined as a broad band at 3349 cm^{-1} associated with the –OH stretching vibration for bound H_2O absorption and the peak at 2901 cm^{-1} corresponded to the long-chain asymmetric stretching of methylene ($-CH_2-$) for the wax-residue at yarn. For H_2O adsorbed onto the yarn specimen, the peak appeared at 1641 cm^{-1}. The peak at 1429 cm^{-1} was associated with rocking C-H carbohydrates and lignin, supported by a band at 1319 cm^{-1} for lignin. Furthermore, the two peaks at 1163 and 1113 cm^{-1} correspond to an asymmetric ether relationship (-C-O-C-). For the contribution of cellulose, hemicellulose and minor lignin Spectra show a strong band at 1058 cm^{-1} due to the symmetrical C-O-C bond strain in-alkyl ether and C-O stretching vibrations and peaks at 898 cm^{-1} attributed to glycoside preference and asymmetrical out-phase ring stretching of C1- O-C4. For threads that were activated and mordanted with potash alum, four significant changes appeared at the peaks of 3407, 1639, 1131, and 899 cm^{-1}, respectively. Due to the presence of inorganic salts (potash alum) at the peak, the cellulose was exhibited at 899 cm^{-1}. The peaks at 1131 cm^{-1} correspond to C-H and C-O deformation bands, while 3407 and 1639 cm^{-1} are responsible for the deformation of OH and H_2O adsorbed into yarn specimens.

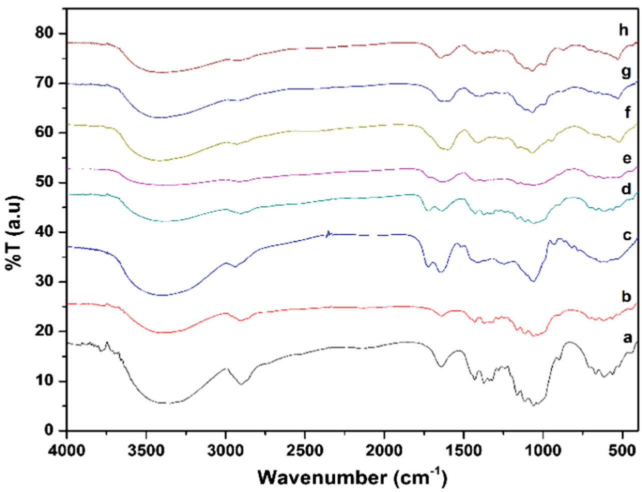


Fig. 4. FTIR spectra of a) woven yarn before treatment; b) weaving yarn after activation and mordanting; c) hanjuang leaf extract; then yarn is woven after dyeing d) pH 3; e) pH 5; f) pH 6; g) pH 7; and h) pH 9

Table 2. Value RGB and colour change at the pH of the woven yarn

Sample	**Value**			**Photograph**
	R	**G**	**B**	
Ekstrak	135	93	107	
pH 2	192	129	156	
pH 3	176	128	150	
pH 4	154	123	129	
pH 5	177	164	155	
pH 6	160	149	143	
pH 7	97	107	98	
pH 8	107	125	109	
pH 9	84	109	88	
pH 10	77	94	76	

Figure 4c shows the FTIR spectrum of the hanjuang leaf extract dye extracted using acid water as a solvent. The spectrum of the peak at 1061 cm^{-1} is associated with the C-O stretching vibration representing the ester group. The peak at 1423 cm^{-1} corresponds to the C-C stretching vibration of the aromatic group. The peak at 1638 cm^{-1} represents the N-H stretch. The peak at 1722 cm^{-1} is associated with the C = O stretch. The presence of a carbonyl group (C = O) and an amine group (N-H) in natural dye extracts was also presented by previous researchers [23, 24]. The peak at the wave number 3384 cm^{-1} corresponds to the O-H stretching vibration. In comparison, the FTIR spectra of the application of hanjuang leaf extract on woven yarn as a natural colouring agent at various pH variations can be seen in Fig. 4d to 4h. For threads stained with hanjuang leaf extract at acidic pH (Fig. 4d and Fig. 4e), it was seen that the peak representing the N-H stretch was still visible, and the OH was seen to decrease in intensity. The one responsible for the red colour decreasing the pH is the methoxy group or C-H stretch (CH_3-O-), which in the FTIR spectra is indicated by the high intensity at wave numbers 2903 and 2914 cm^{-1}. The symmetrical C-O-C bond strain in alkyl strengthens it. The peak indicates ether at 1060 cm^{-1}. Furthermore, with increasing pH, it was seen that the intensity of the methoxy group decreased, and the equilibrium shifted to the formation of the hydroxyl group (-OH), where the intensity of the hydroxyl group at wave number 3399 cm^{-1} increased. It was strengthened by an increase in intensity at the peak of 1650 cm^{-1}, which was associated with the C = O stretch. In addition, the presence of metal, in this case, potash alum used as a mordanting agent, can be observed at peaks around 800 cm^{-1}.

This interaction between cellulose (yarn)-metal dye is a coordination complex. Potash alum forms coordination bonds with weak complexes in the yarn but is strong against natural dye molecules. They do not block the dye and reduce interaction with the yarn, which causes a high value of colour strength [6, 15]. It can be seen in the RGB values, which increase when dyes from extract solutions at various pH variations are applied to the yarn, as shown in Table 1 and Table 2.

4 Conclusions

Changes in pH cause a reversible change in the anthocyanin structure. At a pH of 2–3, anthocyanins are in the dominant form of flavylium cations which are red and fade in the range of 4–5. An increase in pH to 10 causes chalcone, a quinoidal base, and carbinol to predominate, producing a yellow-to-turquoise colour. The colour change causes a red shift in the UV-Vis spectra of anthocyanin. Potassium alum interacts with anthocyanins more strongly than its interaction with yarn, resulting in a more vigorous colour intensity when applied to the yarn. It is indicated by the increasing RGB value when anthocyanin solutions at various pH variations are applied to the yarn.

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Author Contributions. DH conceptualization, methodology, writing-original draft. NI. validation, data curation. UKZ formal analysis, investigation. HM visualization, project administration. BHR software, supervision. RKS methodology, resources. All authors contributed to writing, review & editing.

Conflict of Interest. The authors declare that there are no conflicts of interest.

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