MORDANT AND FIXATION TEST OF BONE ASH ON NATURAL COLOURS TOWARDS COTTON AND SILK FABRIC

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Abstract: *The environmental pollution due to synthetic textile dye waste has induced an increase in the use of natural dyes. In general, the fixation process using alum, lime and tunjung. The novelty of this research is to make bone ash as an initial mordant and fixator. The CaO in bone ash and its absorbent potential makes it worth testing as a pre-mordant and fixator for cotton and silk fibers using natural dyes. This study aimed to determine the effect of bone ash as a mordant and fixator on the immersion of natural dyes from chromolaena, mango leaves and garlic skin. The test stages include: preparation, extraction, fixation and pelorodan. The resulting colour tends not to change. However, the endurance level of the fabric towards washing, sweat, light and rubbing has improved (grades 4-5). It has been proven that bone ash, which is chemically composed of several metal oxides, (CaO) is quite similar to lime ($CaCO_3$), having a high pH (alkaline) level. Therefore, it can serve as an alternative fixator with sodium carbonate (Na_2CO_3) as the final mordant. The results showed that cow bone ash has a high potential of being used as a pre-mordant material and fixator in the colouring processes of cotton and silk fabrics.*

Keywords*: bone ash, final mordant, natural dyes fixator, cotton, silk fabric.*

1 INTRODUCTION

The use of synthetic dye has existed in the textile industry since the rise of Industrial Revolution in Britain. This was turning point for industrial and economic growth in the world [1]. Textile dyes became very popular in the 21st century [2], as well as batik colouring, which relied on synthetic dyes [3]. Synthetic dyeing in textile industries has become a hereditary tradition in the process of colouring [4]. This is due to its sharpness and colour diversity [5]. An estimated 10,000 types of dyes are used in the textile industry [6]. Textile waste makes clear water colorful and toxic; thus it affects the ecosystem, bringing about chemical and biological changes as well as producing pollutants that disrupt the aquatic ecosystem [7, 8]. Environmental damage and pollution become a long debate in regards to the fulfilment of human economics. This is also known as anthropocentrism behavior, which prioritizes human interests [9]. The high market demand for the activities of textile industries makes it a source of state revenue. These activities contribute about 8.5% of gross domestic product [10]. Therefore, it is a key tool to spurring economic growth [11]. The textile industry needs to collaborate with other textile industries, convection business units, dye supply units and the government for economic progress [12]. The rapid progress of globalization is driving competition in the textile industry, including batik as one of the common commodities in the international market [13]. Many textile businesses have difficulties in economic growth, so innovation is needed [14]. The development of textiles gave rise to colour innovations, including synthetic colouring [12]. These innovations have helped in raising alternative choices of silk batik in long-sleeved and short-sleeved for consumers [15, 16]. This triggers a rapid increase in market demand. Therefore, it results in textile industries choosing synthetic dyes. The use of synthetic dyes can accelerate production, increase product quantity and facilitate economic circulation in the textile industry but conversely, it has an impact on environmental pollution due to its waste [17]. In addition, the use of synthetic dyes in the production process can also become a risk towards health, causing allergies and skin injury [18, 19]. Waste water contains various pollutants including surfactants, salts and acetic acids [20]. Among these components, synthetic dyes are the most difficult components to be melted. This is due to the fact that they are made from synthetic chemicals which have complex structures [21]. Environmental problems arising from accelerated industrial growth have led to the development of safer production strategies [22]. The dangers resulting from the use of synthetic colouring has led to the use of natural dyes by some industries [23]. The use of natural substances can provide a good and sharp colour pattern on the fabric [24]. Natural dye substances can be obtained from plants and animals. It can be extracted from plants through roots and twigs or leaves of many species [25]. Coconut oil can also be used as an enhancement on fabric to absorb natural dye [26]. Extreme climate change also encourages new colour changes on tree leaves [27], which is a natural phenomenon [28]. These dyes can also be obtained from animal skin extract as well as from animal fat [29]. Agents of natural dyes from animals can be found contained in pigments in the animal's body. Each contains substances with different shades [30].

Bone ash is a white powder which is a residue from the burning (calcination) of bone. It is one type of mineral made from cow bone waste consisting of calcium phosphate. Furthermore, it is a tricalcium phosphate in the form of $Ca_5(PO_4)_3OH$ hydroxyapatite. According to Saragih [32], cow bones contain 58.30% $Ca_3(PO_4)_2$; 7.07% $CaCO_3$; 2.09% $Mg_3(PO_4)_2$; 1.96% CaF_2 and 4.62% collagen. Chemically, bone ash consists of metal oxides in the form of 55.82% CaO; 42.39% P_2O_5; 1.40% MgO; 0.43% CO_2; 0.09% SiO_2; 0.08% Fe_2O_3 and 0.06% Al_2O_3. Bone ash which has a high CaO content can serve as a good absorbent based on the chemical composition of CaO [31].

In general, the fixation process using alum, lime and *tunjung*. The novelty of this research is to make bone ash as an initial mordant and fixator. The development led to many previous studies discussing textile colouring. The study aimed to examine the effect of bone ash as a mordant and fixator of natural dyes obtained from *chromolaena* (Kopasanda), mango leaves, and garlic skins applied to cotton and silk fabrics. White cotton fabric, commonly called *mori* or *Muslin* or cambric, which is made of cotton yarn, was used. It contains 10% mild starch which makes it easily removable while washing. It is a material from natural fibers with high affinity, absorption, heat resistance and heat conductivity. Tests were also carried out using silk that has some special properties which includes: (1) lighter than cotton, (2) having good insulating power against electricity and heat, (3) high water absorption level, and (4) high tensile strength [32].

From some previous studies, researchers did not discover that bone ash can be used as a pre-mordant and fixator in natural fabric colouring. Specifically, this study aims to determine whether bone ash can be used as a substitute for *tunjung* for the initial mordant process. Furthermore, the researcher wanted to find out whether bone ash could be used as an alternative material to replace lime and alum in the fixation process.

2 METHODS

2.1 Materials

Following are the physical properties of fabric primissima cotton: materials (warp 56/60 dtex, weft 50/56 dtex); density (warp 42/50 threads/cm, weft 42/50 threads/cm); 100% pure primissima cotton composition; weight 190 g/m^2. While the physical properties of silk fabric: materials (warp 22.2/24.4 dtex, weft 22.2/24.4 dtex; density (warp 115 threads/cm, weft 90 threads/cm); 100% pure silk composition; weight 175 g/m^2. From the material data, the fabric has met the requirements to do a color fastness test according to the Indonesian National Standard.

Pre-mordant raw materials: bone ash 2 g/L, alum 6 g/L. Raw colouring material: leveling agent, mango leaves, *chromolaena*, garlic skin with the ratio of 1:4. Raw final mordant material (fixator): alum 70 g/L, bone ash 50 g/L, *tunjung* 30 g/L. Equipments: scales, stainless steel pans, leaf grinding machines, process tubs, *pelorodan,* and filters.

2.2 Procedures

There are some processes that need to be passed, which includes: pre-mordant, extraction, fixation, and *pelorodan* (the release of wax).

<u>Pre-mordant process:</u> The mordanting process involves placing alum, lime, *tunjung* or other metal elements into the fabric fibre, therefore allowing them to react with the dye [33, 34]. In preparing ingredients for the mordant (according to the dosage recipe), they are dissolved in a predetermined amount of water and heated; cotton and silk are soaked in a leveling agent for about 10 minutes, then squeezed and placed into mordant solution at a temperature of 60°C; the fabric is heated in the solution for about 1 hour with a maintained temperature of 60-70°C before the source of heat is turned off. After that, it is left in the solution for about 24 hours. Finally, it was neatly washed with plain water, squeezed and dried.

<u>Extraction process:</u> This process began with the preparation of natural dyes according to the 1:4 ratio which was 1 kg of extracted material and 4 litres of water. Natural dyes were chopped and soaked overnight with predetermined amount of water. Furthermore, they were extracted by heating for about 1 hour after reaching the boiling point of water (100°C). The solution was filtered and cooled at room temperature and finally ready to be used as a natural dye. After that, the coloring process begins as shown in Table 1, cotton cloth, plain silk, or batik soaked with a leveling agent first, soak for about 10 minutes, then drain it; the cloth is evenly dipped back and forth and allowed to soak in the natural dye solution for about 10 minutes; the cloth is removed from the soaking solution of natural colors, drained and aerated to dry. Repeat coloring a predetermined number of repetitions (6, 12 and 18 times).

Table 1 Dyeing process test data

Coloring Type	Mordant	Fixator	Dyeing Number		
			6x	12x	18x
Chromolaena	alum 6 g/L	alum 70 g/L	√ (plain)	√	√
		bone ash 50 g/L	√ (plain)	√	√
		tunjung 30 g/L	√	X	X
	bone ash 2 g/L	alum 70 g/L	√	√	√
		bone ash 50 g/L	√	√	X
		tunjung 30 g/L	√	√	√
Mango leaves	alum 6 g/L	alum 70 g/L	√	√	√
		bone ash 50 g/L	√ (plain cotton & batik silk)	X	√
		tunjung 30 g/L	√ (cotton batik)	X	X
	bone ash 2 g/L	alum 70 g/L	√ (plain)	X	√
		bone ash 50 g/L	√	√	√
		tunjung 30 g/L	√ (plain)	X	√ (plain)
Garlic skin	alum 6 g/L	alum 70 g/L	√	√	√
		bone ash 50 g/L	√ (plain)	√	√
		tunjung 30 g/L	√	√	√
	bone ash 2 g/L	alum 70 g/L	√ (plain)	√	√
		bone ash 50 g/L	√ (plain)	√	√
		tunjung 30 g/L	√ (plain)	X	√ (plain cotton and batik silk)

Fixation process: The fixation process was carried out with mordant material (according to the dosage of the recipe), dissolved in predetermined amount of water, stirred evenly and allowed to stand overnight. A portion of the fixation solution was taken as a test sample. The natural-dyed cotton fabric and silk were put into each of the fixation solutions in a dry state. Afterwards, they were dyed back and forth for approximately 2-3 minutes. Thereafter, they were removed, aerated for about 1 minute and washed clean.

Pelorodan process: *Pelorodan* is a term from the Javanese language, Indonesia. *Pelorodan* is the process of removing the wax coating on fabrics. *Pelorodan* on coloured cotton and silk fabrics with water and then heated with bone ash as much as 5 g/L of solution. Afterwards, cotton fabric and batik silk were soaked in starch water solution for approximately 24 hours, squeezed and put into boiling water *pelorodan*. Fabric was stirred in the *pelorodan* solution until the wax was released. Then, it was lifted and put into a leveling agent solution. In addition, the *pelorodan* process should be repeated if wax was not completely released. Finally, the fabric was washed and dried. The results of the last stage of *pelorodan* can be seen in Table 2.

2.3 Data analysis

This research was carried out using the method of implementation experiment in Pilang Village, Masaran District, Sragen Regency and Indonesia.

In the trial, several analysis options can be done. One of them is the Kolmogorov-Smirnov normality test with statistics; if it is included in the test it makes this study not focus on the initial problems of this study. This study focuses more on bone ash as a substitute for mordant and fixator, so that researchers choose another appropriate method, namely the color resistance test based on the Indonesian National Standard to answer the focus of this research problem.

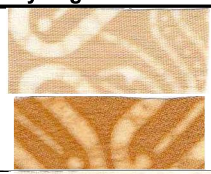
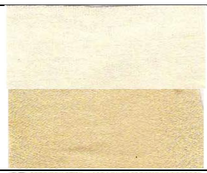
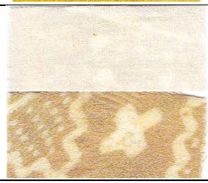
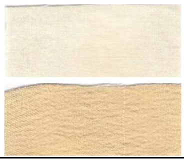
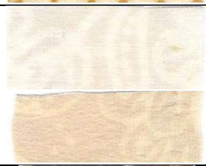
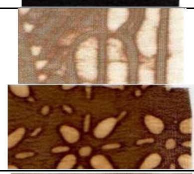
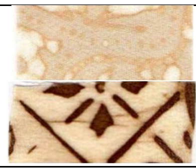
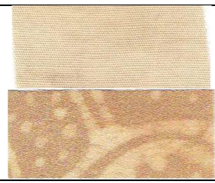
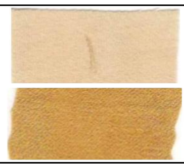
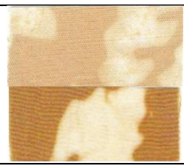
This study uses the standard of Gray Scale to assess the results of dyeing process. There are 4 color fastness tests based on the Indonesian government standard setting through the Indonesian National Standard (SNI) [35]. The quality testing includes color fastness of the fabric against washing (40°C) repeatedly (ISO 105-C06-2010), color fastness of the fabric to sweat (acid) (SNI ISO 105-E04: 2010), color fastness on fabrics against the light (daylight) (SNI ISO 105-B01: 2010) and color fastness on fabrics caused by rubbing (dryness) (SNI 0288-2008).

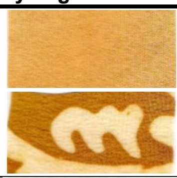
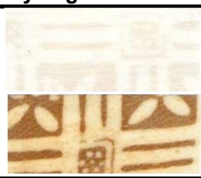
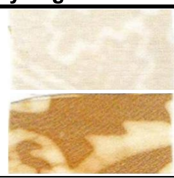
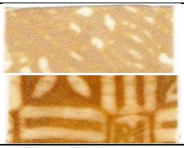
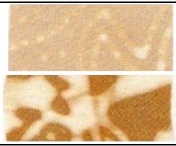
3 RESULT

3.1 Result of staining

Table 2 is the result of the coloring process starting from the initial mordant process, the extraction process in which there is a staining test process in Table 1, the fixation process, the *pelorodan* process (the release of wax). Based on the conducted dyeing results (Table 2), it showed that the differences were not significant, except for fixation with *tunjung* which appeared darker. There are several dyeing repetitions that show a darker colour with a smaller number of repetitions in the same mordant or type of fixation. This situation has led to a difficulty in making conclusions regarding the most appropriate dyeing technique. The fixation result also showed that batik silk fabric absorbs colour easily and therefore produces a darker shade than the batik cotton fabric.

Table 2 Test results of natural dyed fabric

Dyeing process	Type of fabric	Dyeing number 6x	Dyeing number 12x	Dyeing number 18x
bone ash - mango leaves - bone ash	cotton silk fabric			
bone ash - *chromolaena* - bone ash	cotton silk fabric			
bone ash - garlic skin - bone ash	cotton silk fabric			
bone ash - mango leave s- alum	cotton silk fabric			
bone ash - *chromolaena* - alum	cotton silk fabric			
bone ash - garlic skin - alum	cotton silk fabric			
bone ash - mango leaves - *tunjung*	cotton silk fabric			
bone ash – *chromolaena* - *tunjung*	cotton silk fabric			
bone ash - garlic skin - *tunjung*	cotton silk fabric			
alum - mango leaves - bone ash	cotton silk fabric			

Dyeing process	Type of fabric	Dyeing number 6x	Dyeing number 12x	Dyeing number 18x
alum - *chromolaena* - bone ash	cotton silk fabric			
alum - garlic skin - bone ash	cotton silk fabric			
alum - mango leaves - alum	cotton silk fabric			
alum - *chromolaena* - alum	cotton silk fabric			
alum - garlic skin - alum	cotton silk fabric			
alum - mango leaves - *tunjung*	cotton silk fabric			
alum - *chromolaena* - *tunjung*	cotton silk fabric			

3.2 *Color fastness test*

There are 4 color fastness tests conducted in this study. Color fastness test based on the Indonesian government standard-setting through the Indonesian National Standard (SNI). The basis of colour fastness test lies in the overall difference or contrast between the original and the sample to be tested. Quality test for dyeing includes: fastness to washing (40°C), sweat (acid), light (daylight) and iron (wet). This test is based on SNI ISO 105-C06-2010, SNI ISO 105-E04: 2010, SNI ISO 105-B01: 2010 and SNI 0288-2008. Readings from the test results of colour dyeing and colour change was conducted in the form of gray scale with the following values:

In Table 3, the value 5 means there is no change in color change and so on until the value 1 means that there is a very large color change. The test results in Table 4 show color fastness to washing 40°C. The results on the types of cotton and silk fabrics scored 4-5 (good), the results of the color fastness test to acid sweat, on 7 samples of cotton fabrics, 2 samples got a value of 4-5 (good) and 5 samples got a value of 4 (good), while in 5 samples of types of silk fabric 4 samples scored 4 (good) and 2 samples scored 3-4 (quite good). From the results of the color fastness test to light (daylight), the results on the types of cotton and silk fabrics showed a score of 4-5 (good). From the results of the color fastness test to rubbing with dry cotton, 1 sample got a score of 4-5 (good), 3 samples got a score of 4 (good), 3 samples got a score of 3-4 (pretty good), while on silk, 1 The sample scores 4-5 (good) and 4 samples score 4 (good).

Table 3 The results of the color fastness value

Score	Different color	Tolerance for work standards	Reading
5	0	± 0.2	very good
4-5	0.8	± 0.2	good
4	1.7	± 0.3	good
3-4	2.5	± 0.3	good enough
3	3.4	± 0.4	enough
2-3	4.8	± 0.5	less
2	6.8	± 0.6	less
1-2	9.6	± 0.7	bad
1	13.6	± 1.0	bad

Table 4 The results of the color fastness value of the samples of marenggo shrubs, mango leaves and onion skin on cotton and silk fabrics

Type of fabric	Test type	Test results			
		40°C color fastness resistance to washing	***Color fastness resistance to acid sweat***	***Lightfastness (daylight)***	***Fastness resistance to dry rubbing cotton***
Cotton	bone ash - marenggo 18x - *tunjung*	4 -5	4	4 - 5	3 -4
	alum - 18x onion skin - alum	4 -5	4 – 5	4 – 5	4
	alum - 18x onion peel - bone ash	4 -5	4 – 5	4 – 5	4 -5
	bone ash - mango leaves 18x - bone ash	4 -5	4	4 – 5	4
	bone ash - mango leaves 18x - alum	4 -5	4	4 – 5	3 -4
	bone ash - mango leaves 18x - bone ash	4 -5	4	4 – 5	4
	bone ash - mango leaves 18x - bone ash	4 -5	4	4 – 5	3 -4
Silk	alum - onion skin 18x - alum	4 -5	4	4 – 5	4
	alum - 18x - bone ash - mango leaves	4 -5	3 -4	4 – 5	4
	bone ash - mango leaves 18x - alum	4 -5	4	4 – 5	4
	bone ash - mango leaves 18x - bone ash	4 -5	4	4 – 5	4 – 5
	bone ash - marenggo 18x - bone ash	4 -5	3 -4	4 – 5	4

From the test, samples were examined for colour density and colour fastness towards washing, sweat, rubbing and direct sunlight and the following results were obtained. From the results of table 4 on cotton fabric, it can be concluded that the most stable value was the use of alum as a pre-mordant with garlic skin and bone ash as the final mordant. This was due to the low content of colour pigments on garlic skin waste. Therefore, the possibility of colour loss due to fading process is minimal compared with colouring of *chromolaena* and mango leaves which contain more colour pigments.

Conversely, the highest value for silk fabric was found in mango leaf dyeing using bone ash a pre-mordant and colour fixator (final mordant) as well. This was a result of the presence of mangiferin pigment found in mango leaves which binds more strongly to the silk fibers in bone ash (mordant) through covalent bonds. The estimated result changes due to various factors, including suboptimal extraction, unstable treatment of the dyeing process, the condition of the material and its preparation process (the difference in wax used on a batik stamp). This can be minimized by maintaining similar natural materials, waxing on the initial preparation, and the treatment consistency during the dyeing process. With the combination of alum and bone ash, the dyeing results only compared the mordants (alum and bone ash) and fixators (alum, bone ash and *tunjung*). The results showed little difference except for fixations that showed darker results when tides was used. There are several dyeing repetitions that show more intense colour with fewer repetitions on the same type of mordant or fixation. Therefore, making a decision regarding the most appropriate colouring technique was difficult. Colouring results after fixation also showed that batik silk absorbs more colour extract thereby producing a darker colour than the cotton fabric.

4 DISCUSSION

Besides using synthetic dyes, the fabrication and dyeing process also utilizes natural mordant dyes which are generally extracted from plant parts, such as *Soga Alam*, Gambier, *secang* wood, indigofera leaves etc. This type of dye works optimally in colouring protein fibers (silk, wool) and polyamide fibers. In the natural mordant colouring process, the preliminary mordant (pre-mordant) and final mordant (fixation) processes are carried out [36]. This process was carried out to provide good washing resistance and colour determination. Binding of dyes to fibers is facilitated by the presence of metal ions from the mordanting process [37]. Metal ions act as electron acceptors to form coordination bonds with dye molecules, making mordants insoluble in water and ensuring colour fastness to the fabric [38]. In Sarwono's research, it was stated that alum, lime, and *tunjung* can be used as fixators [39].

The function of the initial mordant is to assist and increase the adhesion of the dye to the fabric. The purpose of mordanting in the fabric process is to increase color absorption, remove oil, grease, and wax that inhibit the color absorption process, increase color adhesion, connect natural dyes with fabric fibers so that affinity (the attraction of the dye increases to the fiber). Fixation is to strengthen the color and change the color of natural dyes according to the type of metal binder.

So far, a lot of cow bones have been wasted, not used. The results show new findings that bone ash can be used as an alternative material to substitute lime and soda ash for the final mordanting process, as a substitute for *tunjung* for initial mordant.

The test results also show that dyeing on cotton and silk fabrics without undergoing the *pelorodan* process did not experience a drastic colour reduction due to the absence of heat (temperature) which greatly affects the quality of natural colouring. The results of garlic skin dyeing gave a yellowish-white colour (ivory), mango leaves gave a light yellow colour [40], and the greenish yellow colour was shown in the dyeing results using *chromolaena* [41]. The use of the final mordant (fixator) alum gave a colour like the original colour before fixation, bone ash became slightly reddish yellow while *tunjung* became the darkest.

The colour direction obtained by the three types of natural dyes is as a result of the substance present in each material. The leaves of *chromolaena* and mango contain chlorophyll, a colouring pigment which is responsible for trapping sunlight energy which is used in photosynthesis. The leaves have other pigments, such as xanthophyll which gives a yellowish colour as shown in the results of *chromolaena* and mango leaf dyeing. Meanwhile, garlic skin which is a waste from white food does not have a pigment that can absorb certain colours. Therefore, the colour direction produced tends to be yellowish white. Garlic skin dyeing can be done by the traditional method of boiling in water [42]. However, the use of garlic skin waste as an alternative to natural dyes for batik became more optimal with pastel colours produced by a combination of *chromolaena* dyeing, mango leaves and garlic skin, from the initial level using alum fixation which later becomes darker with *tunjung* fixation.

Bones reflect an excellent affinity for pollutants. These pollutants become bound to the absorbent through the absorption process [43]. Bone ash is very important due to its unique cellular structure from bone which is maintained through calcination. Bone ash has excellent non-wetting properties for chemical moisture, free of organic material and has a very high heat transfer resistance [44]. The experimental results show that cow bone ash has high potential of being used as a pre-mordant material and a colour lock (fixator) as well in the process of colouring cotton and silk batik fabric. Bone ash is chemically composed of several metal oxides, one of which is CaO (55.82%), which implies that bone ash has a similarity with calcium oxide having a high pH (alkaline) and therefore can be an alternate fixator in substitution for calcium oxide and sodium carbonate as the re-mordant.

5 CONCLUSION

Synthetic staining can accelerate production and increase the quantity of products in the textile industry. Conversely, it contributes to environmental pollution due to its waste. The arising environmental problems caused by synthetic dyeing have led a number of producers in switching to natural dyeing. Natural dyeing can be obtained through elements of natural substances that can reduce production costs and are environmentally friendly with better quality. Natural substances provide various shades and stronger colouring. Bone ash reflects very good affinity for pollutants that are bound in the absorbent through the absorption process, and has some similarities with calcium oxide having high pH content. The test for the effects of mordant and fixation with bone ash on cotton and silk fabrics, using natural dyes obtained from *chromolaene*, mango leaves, and garlic skin, has shown results in colour direction which tends to change. Chemically, bone ash consists of several metal oxides (CaO) which is quite similar to lime ($CaCO_3$) with quite a high pH content (alkaline). Therefore, it can serve as an alternative fixator in substituting for lime ($CaCO_3$) and sodium carbonate (Na_2CO_3) as the final mordant. This was proven by the better value of fastness towards washing, perspiration, light, and iron (grades 4-5). The results showed that cow bone ash has a good potential as a pre-mordant material and fixator in the process of colouring cotton fabric and silk fabrics. This research was limited to cotton and silk fabrics. This condition allows the use of a more diverse fabric. Therefore, it can give a different colour dyeing and fastness in future research.

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