THE EFFECT OF SURFACTANT ON PRE-MORDANTING AND pH OF DYEING PROCESS WITH NATURAL DYES

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Abstract

The dyeing process with natural dyes has disadvantages, including poor color fastness. A mordanting process is required to overcome these disadvantages. Conventional mordanting processes often use metal salts. This research will substitute the mordanting process using surfactants as mordant substances. The pH of the process influences the application of dyeing with natural dyes. This study aims to determine the effect of the pre-mordant process using surfactants and the pH of the dyeing process on the color characteristics of cotton and silk fabrics dyed with Cocos nucifera L dye extract. The surfactants used were cationic and non-ionic polymeric surfactants. This study was conducted in phases. The first phase involved the extraction of coconut fiber, and the second phase involved pre-mordanting the fiber with surfactants. The third stage is the dyeing process, which has variations in pH 3, 7, and 9. The evaluation results show that cationic surfactants have the potential to be used as a mordant and provide an increased anti-bacterial effect on processed fabrics. The optimal use of cationic surfactants was obtained using a concentration of 9 g/L and pH 7 for the cotton dyeing process. The optimum concentration was obtained at 9 g/L for the silk dyeing process, and the dyeing pH was 3.

Keywords: *dyes, pH, pre-mordanting, surfactant*

Introduction

Since prehistoric times, humans have been interested in natural dyes as coloring agents. (Clark, 2011) Coloring with natural materials is an alternative that is widely developed today. One problem with the aquatic environment is a large amount of polluting waste, such as metals and dyes (Nitsae *et al*., 2021). The use of more environmentally friendly natural dyes in textiles has become a very important issue due to increased environmental awareness to avoid using harmful synthetic dyes (Samanta and Agarwal, 2009). Indonesia has a rich diversity of natural plants which are still unexplored. However, the use of natural dyes has drawbacks, including unstable colors, poor color uniformity, low pigment concentration, and limited color spectrum (Paryanto *et al*., 2013).

Natural colors derived from coconut coir (Cocos nucifera L.) were used in this study. The community views the vast amount of coconut coir as garbage. Optimal processing of the waste is not possible. The annual loss to Indonesia from burning waste coconut coir in all of its areas is estimated to be as high as 13 trillion. These losses will be transformed into advantages for the local community with optimal utilization (Andini and Widiawati, 2016).

Waste from coconut coir can be utilized as an environmentally beneficial dye that breaks down quickly in the natural world, preventing pollution that harms the ecosystem. (Anggriani *et al.*, 2017). According to the research, anthocyanin pigments' flavonoid compounds and alkaloids, which have the potential to be natural dyes, are present in immature coconut coir. Light, oxygen, and pH impact the natural dyeing process (Inggrid and Iskandar, 2016). Because there is no attraction between the substance and the material utilized during the coloring process with natural dyes, a mordant must be added—a procedure known as the mordanting process. In order to minimize color fading after processing, modification is employed to create a link between natural dyes and fibers (Siva, 2007).

Generally, the types of mordant mostly used are ash soda, alum, ferrous sulfate and Turkish Red Oil (Sunarya, 2014), where the process can be carried out at different stages depending on existing needs such as pre-mordanting, post-mordanting, and simultaneous mordant (Ding, 2013). The use of synthetic mordant, especially containing metals, can cause pollution to the surrounding environment, so other alternatives can be used, namely by using surfactants. Limitations on color yield and poor fastness properties prompted a search for ideal mordants, the chemicals that increase natural dye uptake by textile fibers. One of the mordant substances derived from metal salts is alum. Among all types of alum, potash alum is cheap, easily available, and safe to use. It usually produces pale versions of the prevailing dye color in the plant. (Samanta *et al.*, 2011). However, alum based on metal salts is less environmentally friendly because it can decompose to produce sulfuric acid and is also a coagulant (Yilmaz *et al.*, 2015).

The surfactant is usually only used as a substance to accelerate the wetting process on the fabric so that it can help disperse the dye in water and help the penetration of the dye into the fiber (Myers, 2006). The use of surfactants in the textile process can be based on the last property, namely solubilization, often referred to as the process of dissolving organic substances by surfactants assisted by micelles which combine to form surfactant molecules at a certain concentration (Arnelli and Wahyu, 2003). To get the desired properties, the penetration of natural dyes into the fiber is very important. Surfactants containing both hydrophilic and hydrophobic groups play a useful role in textile processing, ideally in aqueous media, by quickly and uniformly wetting the fiber surface. Furthermore, surfactants are also widely used as leveling, dispersing, and wetting agents in dyeing processes. Surfactants can form complexes with ionic dyes or facilitate the absorption of nonionic dyes. The interaction of dyes with surfactants is important to improve the coloring process (Gokturk and Tuncay, 2003). Industrial applications of surfactants depend on the product manufacturing process, the characteristics of the surfactant, and the desired end product. The role of different surfactants is due to their diverse molecular structures. Surfactant molecules can be visualized, like tadpoles having heads and tails. The head is hydrophilic (like water), which is very polar, while the tail is hydrophobic (hates water/likes oil), a nonpolar part. The heads can be anions, cations, or nonions, while the tails can be linear chains or hydrocarbon branches. The head-tail configuration makes surfactants have various functions in the industry. However, more attention should be paid to the interactions between natural dyes and surfactants. There are few reports on natural dye-surfactant interactions (Chandravansh *et al.*, 2012).

This research will be carried out on natural dyes with a pre-mordanting process using non-ionic polymeric and cationic surfactants with hydrophobic and hydrophilic groups in their structural arrangement, often called amphiphilic polymers. In carrying out the dyeing process of natural dyes and the mordant process using surfactants, this research will vary the type of surfactant and dyeing pH in coloring cotton and silk with natural dyes.

Research Methods

Materials

Cotton fabric obtained from PT. Primatexco Batang Central Java and silk fabrics are woven fabrics obtained from IKM Batik Sutera Garut, West Java. Coconut coir material is obtained from traditional markets in the Bandung area. Phospholipid-based Cationic Surfactant (Arlasilk EFA LQ), non-ionic polymeric surfactant (are from CRODA Industrial Chemical Supplier). Experiments and tests were conducted at the Dyeing Chemistry Physics Laboratory and the Textile Evaluation Laboratory at the Politeknik STTT Bandung. Antibacterial activity tests were performed at the Biotechnology Research Laboratory of the Universitas Pendidikan Indonesia (UPI).

Methods

The research methodology begins with making natural dyes, followed by applying these dyes to cotton and silk fabrics pre-mordanted with alum, non-ionic polymeric and cationic surfactants with a concentration of 9 g/L, respectively. The fabric was soaked for 1 hour at room temperature. The coconut fiber extraction process is carried out by cutting old coconut husks that have been dried, then boiling until the color pigment from the coconut husks comes out, with a dose of 2,000 grams of coconut husks in 10,000 mL of water until it is reduced to 5,000 mL. Then, strain the boiling results, separating the remaining material that has been extracted, resulting in the extraction of 5,000 ml of coconut fiber from 2,000 grams of old coconut fiber (Kurniati *et al.*, 2021). The pH of the dyeing process varies from 3, 7, and 9 to see the quality of the color generated. The experiment was carried out twice for each experimental variable. The dyeing results will be evaluated, including the color depth, color direction, color fastness to washing, and antibacterial test.

Results and Discussion

Based on the results of experiments that have been carried out by varying the pre-mordanting pH using surfactants and the pH of the dyeing process, the following analysis can be obtained as follows:

Colorfastness to washing

Colorfastness to washing is an early indication to determine that the dye attached to the fabric has good affinity so that the bond between the dye and the fabric is good. In the results of this study, cotton and silk fabrics dyed with natural dyes from coconut coir showed good color fastness to washing, especially for fabrics made mordant by non-ionic polymeric surfactants, cationic surfactants, and alum mordant. The result of colorfastness can be seen in Table 1.

From Table 1, the use of mordant can improve the color fastness of the dyed fabric. It applies to cotton and silk fabrics. Staining and greyscale values were compared to its scale standard. For staining scale value, the larger number of staining scales indicates that the level of staining that occurs is smaller. Likewise, on the greyscale value, a number close to a scale of 5 means that the fabric does not change color due to the washing process.

The mordant process that is carried out has a function as a bridge between the dyestuff and the fabric so that it is expected to have added value, namely good color fastness to washing, considering that natural dyes have a poor affinity for cotton fabrics, so this process is necessary. From Table 1, it can be seen that fabric that is mordanted with alum and cationic surfactant gives the same result. It can happen because, in mordant dyes, cationic surfactants form complex bonds between fiber-mordant dyes.

In addition, the addition of an acidic pH to the dye solution can give the dye partially positive properties because a positive charge will interact with the negative dipole that the natural dye has. Surfactants will accumulate with the formation of micelles and can deliver dyes to the fabric. This process will help the dispersion of the dye in the solution and the penetration of the dye into the fabric. Because the type of surfactant used is a non-ionic polymer, many possible bonds can occur, which can cause many dyes to bind and form the micellization of the dye. In contrast, for cationic surfactants, this can be due to ionic interactions between the dye carrier surfactant and the fiber.

The results of testing the color fastness to washing found that fabrics processed with pre-mordanting methods using cationic surfactants as mordant substances can increase color fastness to washing fabrics and can reduce color staining on upholstery compared to the use of alum mordant substances. Based on this, the use of cationic surfactants is good as a mordant and can form complex bonds between fibers, mordant, and dyes so as to increase color fastness to washing. The formation of a complex bond between the fiber, cationic surfactant as a mordant, and the dye occurs in hydrophilic and hydrophobic interactions by utilizing the surfactant's hydrophilic (polar) and hydrophobic groups to bridge the dye with the fiber.

Table 1 Color fastness of cotton and silk processed surfactant pre-mordanting with variations in pH dyeing

Dyeing pH of Cotton	Staining scale	Grey Scale	Dyeing pH of Silk	Staining scale	Grey scale
With no mordan					
pH 3	4	3–4	pH 3	3	3
pH 7	4	3	pH 7	3	2–3
pH 9	3	3	pH 9	3	2–3
Mordan with alum					
*pH 7	4–5	3	*pH 7	4	3
Mordan with non-ionic polymeric surfactant					
pH 3	4	4	pH 3	3–4	3–4
pH 7	4	3	pH 7	3–4	3–4
pH 9	4	3	pH 9	3–4	3–4
Mordan with cationic surfactant					
pH 3	4	4	pH 3	4	3
pH 7	4–5	4	pH 7	4	3
pH 9	4	4	pH 9	4	3

*conventional methods

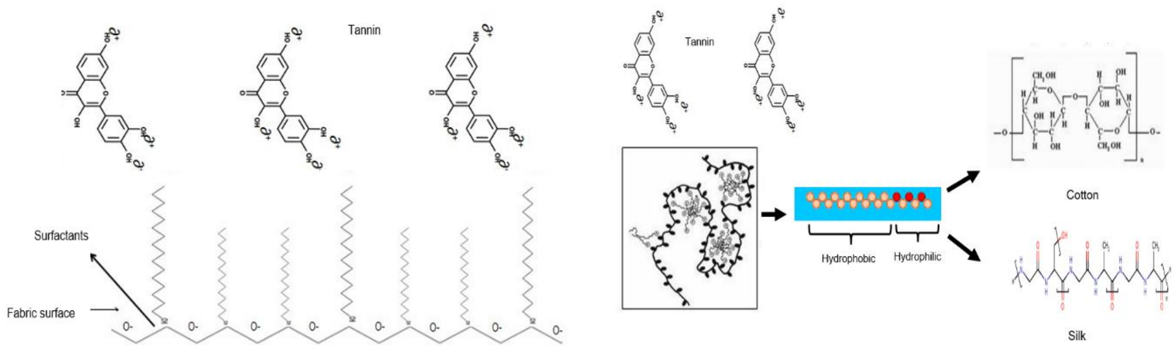


Figure 3. Interaction of tannin, surfactant and fiber, (a) cationic surfactant and (b) non-ionic

Color depth (K/S)

A color depth test was conducted to see the effect of the pre-mordanting process and pH dyeing variations on color depth value. A spectrophotometer instrument measured the color depth. The higher the K/S value, the darker the color resulted. The processed value can be seen in Table 2.

The content of the coconut coir extract, which carries dyes in the form of tannins, has an acidic pH. It is because the majority of the color pigments found in extracts from plants, such as flavonoids, tannins, and xanthonoids, have a positive charge of H+ (acidic) and a negative charge (O-) of the groups.

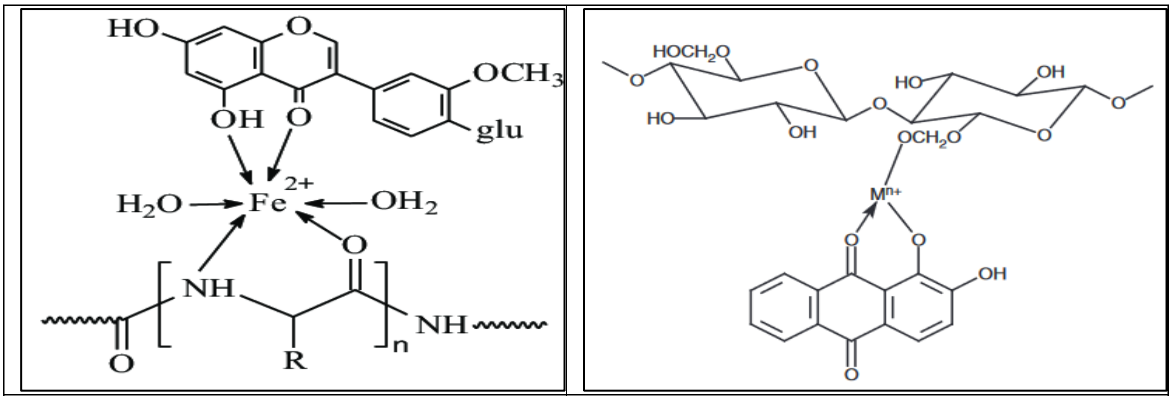
Mordant-processed fabrics with alum and surfactants had higher K/S values than those without mordant. A cotton fabric consisting of cellulose contains a lot of OH groups, where the tendency of the cotton fabric can be affected by the pH activity given to the system (solution). Cotton fabric tends to be partially negative. Natural dyes are then added to the dye bath in an acidic condition. The interaction between the dye and the pH will cause a new interaction. However, in this case, the dipole or positive charge in the acidic pH will interact with the dye, which has a negative dipole, resulting in a positive partial remaining, which will interact with the cotton fabric and assist the penetration process by surfactants. The cationic surfactant process gave a better depth color value than another pre-mordanting process.

Table 2. The color value of cotton and silk processed pre-mordant using surfactant with variations in pH dyeing

Dyeing pH	Color Depth (K/S)	
	Cotton	**Silk**
	Dyed fabrics with no mordant	
pH 3	1.15	1.22
pH 7	0.96	0.39
pH 9	0.96	0.42
	Dyed fabrics with alum mordant	
*pH 7	1.47	0.64
	Dyed fabrics with non-ionic polymeric surfactant	
pH 3	1.73	0.71
pH 7	1.25	0.49
pH 9	1.18	0.37
	Dyed fabrics with cationic surfactant	
pH 3	2.83	1.38
pH 7	2.59	1.28
pH 9	1.51	1.35

*Conventional methods

Silk fabrics showed that fabrics without mordant dyed using a pH of 3 and the pre-mordanting fabric with cationic surfactant yield the same value. Silk fabrics have two active site groups, namely the NH_2^+ and $COOH^-$ groups. Acidic conditions will cause the NH_2^+ present in the silk to change to NH_3^+, causing the polyphenols from natural dyes to interact with the NH_3^+ from the fiber to form ionic bonds. Alum mordant contains Al_3^+, which binds to the carboxyl group on silk and then binds the dye to the fiber and produces ionic bonds when it binds to the alum group. The cationic surfactant will give the same phenomenon with interactions between alum, fiber and dyes. It will form a complex bond between dye and fiber with ionic interactions, while the nonionic polymer surfactant only forms physical bonds. The illustration of the mordant process with the fiber can be seen in Figure 4.



(a)	(b)

Figure 4. Bonds formed between mordant and dyes on (a) cotton (Ding Freeman, 2017) and (b) silk (Jiang *et al.*, 2019)

Tannins, when dissolved in water, will be weakly acidic (Ismarani, 2012). The bond formed in this dyeing is a complex bond between the dye-surfactant-cotton (Baliarsingh *et al*.,2013). Based on the test results, it can be seen that the pre-mordanted fabric with cationic surfactants has a higher color depth than the non-pre-mordanted fabric. Pre-mordanted fabric using cationic surfactant can be substituted for alum mordant. It happens because cationic surfactants have two roles, as a mordant and as a surfactant, which have the property of accelerating dyes into the fiber. Cationic surfactants have a polar group that will interact with the polar part of the cotton or silk fiber, while the hydrophobic part will interact with the tannins. As the process progresses, micelles from surfactants will form, enveloping the dye so that the dye can easily enter the fiber. The presence of surfactants can increase the affinity of the fiber for the dye by lowering the surface tension on the fiber, which will make the dye penetrate more and more into the fiber. It will result in a high value of ageing on the dyed fabric. However, compared to the pre-mordant process using non-ionic polymer surfactants, the pre-mordant with cationic surfactants will provide maximum color depth due to ionic interactions between the fiber and the surfactant.

For silk fiber, according to the theory obtained, the value of the color depth differences in the pH of the solution greatly influences the results of the degree of color depth. Dye solution in an acidic pH atmosphere produces darker colors than the alkaline pH dye solution (Haerudin and Satria, 2018). If the dye is in nature, immersion in alkaline pH will cause the formation of groups that can allow cross-linking in the protein structure, where serine residues will form lysioalanine residues. It can cause a reduction in the reactivity of silk fiber to the dye source contained in the dye extract solution during the dyeing process. Although the residue formed is small, it can affect the ageing value of the dyeing results (Rosyida and Subiyati, 2018).

Color direction (Hue)

The results of testing the color direction resulting from the experiment using surfactants as mordant and variations in pH can be seen in Table 4.

Cotton fabric variants have positive values, which means the resulting colors are more brownish. Fabrics processed with non-ionic polymer surfactants produced higher a* and b* values than the other sample variants, both fabrics with pre-mordanting with alum or without mordant. The use of cationic surfactants shows that the processed fabric gives a lower lightness (L*) color compared to fabrics without the use of a mordant, meaning that the resulting color is darker. It is because cationic surfactants play a role in increasing the surface tension of cotton fabrics so that more dyes bind to the fiber than non-mordant fabrics. The values a* and b* show a high difference in values between fabrics without mordant and fabrics with cationic surfactants mordanted. The a* value with cationic surfactant mordant will shift the color towards yellow, while the b* value with cationic surfactant mordant will shift the color towards red. The color shift towards yellow is due to the presence of a type of flavonoid, namely flavones and flavanols, which bind to the cotton fiber.

For silk dyed fabric, pre-mordanting processed silk fabrics using polymeric surfactants show an a* value towards the red (reddish), while the resulting b* value is towards a yellowish color because the b* value is much greater than the a* value. Then, the color produced in this study is browner (brownish). For silk fabrics processed pre-mordanting using cationic surfactants, the a* value indicates the red direction (reddish), the b* value indicates the yellowish direction, and the L* value indicates the color brightness. Silk fabric made without immersion and dyeing process with neutral pH (control cloth) produced an L* value of 77.26. It is because there is not too much dye interaction that enters the material, so it produces bright colors.

Table 4. The effect of mordant substances and dyeing pH on the color direction of fabrics dyed with Coconut Coir Dyes

Dyeing pH	**Value of L*a*b*C*H**					
	Cotton			**Silk**		
	L*	**a***	**b***	**L***	**a***	**b***
Dyed fabrics with no mordant						
pH 3	72.69	10.68	15.62	69.97	4.76	15.24
pH 7	75.32	8.71	10.04	77.64	1.85	6.34
pH 9	76.65	8.23	8.24	77.31	1.93	6.39
Dyed fabrics with alum mordant						
Alum (pH 7)	70.15	9.38	13.72	75.88	2.32	9.83
Dyed fabrics with non-ionic polymeric surfactant						
pH 3	68.35	10.78	17.35	76.77	1.65	6.78
pH 7	72.33	9.49	12.35	76.07	2.23	7.85
pH 9	72.62	8.76	12.36	75.85	2.12	7.66
Dyed fabrics with cationic surfactant						
pH 3	61.356	9.344	16.828	68.27	5.13	14.56
pH 7	62.45	9.208	15.258	68.00	6.76	13.79
pH 9	70.374	7.162	11.044	67.54	6.99	14.46

Note: L* showed brightness and a*, b* are chromatic coordinates. a* and b* are color direction: +a * is red axis, -a ‘is green axis, + b * is yellow axis and -b * is blue axis.

Optimum condition process

After the sort of surfactant and pH dyeing optimum were obtained, concentration variations of the selected surfactants were carried out at optimum conditions. The following data on the results of variations in the concentration of cationic surfactants can be seen in Table 5.

Table 5. Concentration effect to washing fastness and color depth of dyed cotton and silk which pre-mordanted using cationic surfactant

Fabric	Concentration	Staining scale		Grey scale	K/S
		Cotton	Silk		
Cotton	3 g/L	4	4	4–5	26.861
	6 g/L	4–5	4	4–5	26.208
	9 g/L	4–5	4–5	4–5	27.122
	12 g/L	4–5	4–5	4	28.313
Silk	3 g/L	4–5	4	4	21.179
	6 g/L	4	4–5	4–5	27.883
	9 g/L	4–5	4–5	4–5	29.849
	12 g/L	4–5	4	3–4	34.457

Based on several experiments on dyeing results that have been tested for color fastness, K/S value (color depth) and color direction can be stated that the best results are obtained from fabrics processed with a cationic surfactant as a mordant substance. The use of cationic surfactants as a mordant for the pre-mordanting method yielded the same value as the alum mordant as a comparison for the color change scale. In contrast, the color staining scale produced a value higher than the mordant alum. Based on the test results, cationic surfactants have better mordant power to produce less color staining and the same discoloration as alum mordant.

The surfactant will form micelles that envelop the dye so that the dye can bind to the fiber. In addition, the general structure of the surfactant, which is slender and long, makes it easier for the surfactant to penetrate the pores of the fiber so that more bonds are formed and reach the fiber core. The more surfactant concentrations are used, the more bonds are formed, so the color fastness to washing will be better. It is along with the test results on cotton and silk fabrics, which are processed by varying the concentrations under optimum conditions for each type of surfactant.

For cotton fabrics, the best conditions were obtained in the pre-mordanting process with cationic surfactants, dyeing pH at pH 7 and 9 g/L., For silk fabrics, the best conditions were obtained using cationic surfactants and dyeing pH 3 at 9 g/L.

Antibacterial activity testing

Test of anti-bacterial activity was done under SNI ISO 20743: 2011 procedure. Based on the results of the anti-bacterial activity test, which can be seen in Table 6. Anti-bacterial testing was carried out to confirm the anti-bacterial properties as an effect of using cationic surfactants. Cationic surfactants are the best-known class of antimicrobial, particularly when applied at alkaline to neutral pH, showing a high affinity for the interfaces of all three microbial classes of interest due to the negative charge of the microbial interfaces. The antimicrobial action of quats demonstrates the effect beyond adsorption to the interface. First, adsorb and penetrate the cell wall. Second, reacts with the cytoplasmic membrane to cause disorder. Third, leakage of lower-MW intracellular material. Fourth, degradation of proteins and nucleic acids. Fifth, cell lysis and death. Maehara's in Falk studies noted earlier that polyethoxylated lauryl ether had only a minor antimicrobial effect in laundry applications (Falk, 2019).

Table 6. Effect of surfactant as mordant substances to antimicrobial properties on cotton and silk dyed with coconut coir

Name of test bacteria	*Escherichia coli*			
Inoculum concentration (CFU/ml)	1.22×10^5			
Concentration subtances	Non-ionic Polymeric Surfactant 9 ml/L	Cationic Surfactant 9 ml/L	Non-Ionic Polymeric Surfactant 9 ml/L	Cationic Surfactant 9 ml/L
Growth value F ($F = lgC_t – lgC_0$)	1.49	1.49	1.41	1.41
Growth value G ($G = lgT_t – lgT_0$)	0.98	0.79	1.09	0.59
Antibacterial activity value (A = F – G)	0.51	0.70	0.32	0.81
Material	Cotton		Silk	
Sterilization model	Autoclave			
Incubation time	20 hours			

The antibacterial activity increases with the use of cationic surfactants. It is because, in cationic surfactants, the hydrophobic groups in surfactants can facilitate the penetration of bacteria. Cation ions enter the hydrophobic structures of the cell wall, such as the lipid domain, especially the outer membrane of gram-negative bacteria. Gram-positive bacteria have only a thicker peptidoglycan layer and an inner plasma membrane without an outer membrane. It may not further enhance the interactions between the hydrophobic cationic ion layers killing gram-negative E. coli. The antibacterial mechanism is related to the electrostatic interaction between the cationic layer of the cationic surfactant and the negatively charged bacteria. Tannin compounds are capable of inhibiting the activity of bacteria. (Ruwanda, *et al*, 2021) So that all samples processed with natural dyes have antibacterial activity.

Conclusions

Based on the data and discussions above, the following conclusions are that cationic surfactants have capabilities to substitute the use of alum mordant based on color fastness to washing, color depth obtained, the optimum concentration of cationic surfactant as mordant agent obtained at 9 g/l for cotton and silk. pH dyeing process after the fabric is pre-mordanted is 7 for cotton and 3 for silk. Besides being able to substitute the use of alum mordant, the use of cationic surfactants also provides antibacterial properties.

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