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IDENTIFICATION OF CHANGES IN MAGNETIC CLAY MINERAL LATTICE DUE TO BATIK FABRIC DYEING PROCESS USING X-RAY DIFFRACTION

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ABSTRACT

Clay in West Sumatra has been developed as an industrial material, one of which is a natural dye for batik. The use of clay as a natural dye was previously known to have magnetic minerals contained in clay so as to produce color and resistance to fabrics. Currently, no one has investigated the types of magnetic minerals found in clay and their effect on color resistance in batik cloth, so this study aims to determine the types of magnetic minerals in clay and their effect on color resistance in batik cloth. Clay samples were taken from two areas in West Sumatra, namely Pesisir Selatan and Sijunjung. Samples were characterized from the extraction of clay using X-Ray Diffraction, where measurements were taken before and after the dyeing process on the fabric. The results showed that the types of magnetic minerals found in the clay before and after dyeing the fabric were Maghemite. In addition, the non-magnetic mineral found in clay is Quartz. The color resistance test on the CL-SPPS-210314-2 fabric sample did not change color, on the contrary, the CL-PSBSJJ-210421 fabric sample experienced quite a change from the original color of the fabric or before washing.

Keywords: clay; color resistance; mineral type; XRD

INTRODUCTION

West Sumatra is located at the confluence of two plates, namely the Indo-Eurasia Plate and the Indo-Australian Plate[1] and the area is traversed by the Bukit Barisan mountains[2] As a result of plate movement, hot rock beneath the earth's surface slowly penetrates to the surface, then cools and occurs weathering is clay. The process of weathering the earth's crustconsists of feldspathic rocks, in the form of granite and igneous rocks a very long time resulting in a chemical reaction to produce alumina and silica and minerals [3,4,5]. Clay is composed of one of the chemical elements, namely iron (Fe). The content of iron (Fe) is in the form of Hematite, Magnetite or Ilmenite[6] and is included in the classification of Alfisol or Aluminum Iron Earth and contains Alumina (Al_2O_3) and Silica (SiO_2), mixed with Potash (K_2O) and Soda (Na_2O)[7]. Clay is flexible and impermeable to water. This property is determined by the type of soil mineral that dominates it[8]. Clay can almost be found in all areas of West Sumatra. Clay was developed as an industrial material, one of which is natural batik dye which has high economic value [9].Clay in Lubuk Alung is used as raw material for decorative ceramics[10]. Clay in the District of Rambatan, Tanah Datar Regency is used as a material for producing pottery and bricks[11]. Clay has the potential to be used as a natural dye in batik[12].The natural color of clay occurs due to the presence of iron oxide and organic elements[13]. The brown color of batik comes from the color of clay[14]. The process of making clay batik is the same as batik in general, only the coloring process uses natural dyes from clay [15].Batik coloring with clay is carried out in several stages, namely cooking, soaking, and washing. Cooking on clay is done so that the fabric on batik is not easily weathered. Soaking the batik cloth so that the color in the clay adheres to the cloth. The washing time of batik cloth usually causes discoloration or colored material to separate from the cloth or causes the color of the batik cloth to fade.In this research,the ease or difficulty of fading the color of the fabric is investigated by the type of magnetic mineral attached to the fabric, whereThe mineral Hematite gives a red color, Magnetite gives a brown color and Ilmenite gives a yellow color[6].

Magnetic minerals are minerals that have high magnetic properties and can be utilized optimally and have high economic value. The method used to obtain the type of magnetic mineral is XRD (X-Ray Diffraction). The XRD (X-Ray Diffraction) method produces diffraction patterns which are then used to determine the crystal structure, chemical composition, crystal size based on the diffraction pattern, and identify the types of magnetic minerals in clay.

Based on the research that has been done, it can be seen that clay has a type of magnetic mineral, but in West Sumatra it is not yet known the type of magnetic clay mineral and changes in the magnetic mineral lattice due to the dyeing process of batik cloth.. By using the XRD method, it is hoped that the types of magnetic minerals contained in the clay can be obtained and can see changes in the magnetic mineral latticedue to the dyeing process of batik cloth. Therefore, it is necessary to do research on "Identification of Changes in Clay Magnetic Mineral Lattice Due to the Dyeing Process on Batik Fabrics Using X-Ray Diffraction".

METHODS

Sampling was carried out in two areas of West Sumatra, namely Pesisir Selatan Sijunjung in April 2021. The following sampling locations in this study can be seen in Figure 1.

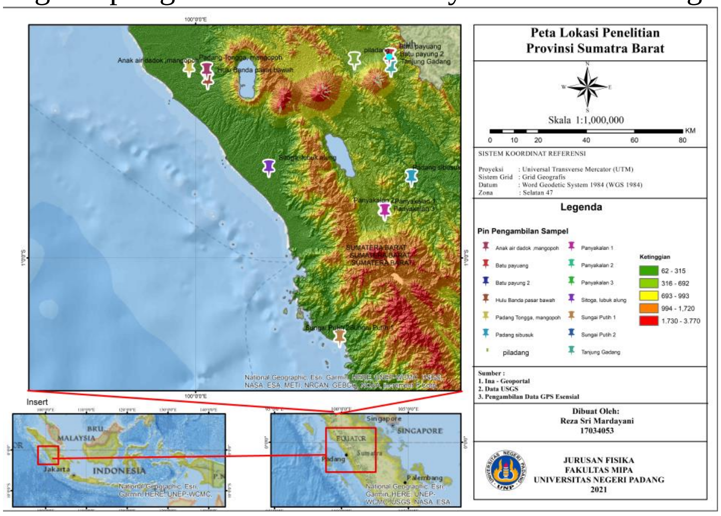


Figure 1. Locations of sampling on a map

Sampling

Sampling is done by prepare tools and materials such as GPS, shovels, and plastic and scrape the sampling point first to take pure clay that has not been contaminated. after that, measurecoordinates for each sampling where the sample is taken 4 points.Then the sample is put into a plastic that has been given a name.

Sample Preparation

a. Determination of the type of magnetic mineral
   Before determining the type of mineral using X-Ray Diffraction (XRD), extraction is carried out first. The clay sample was weighed as much as 100 grams to be extracted using strong and weak magnetic magnets as much as 20 times, then the sample from the extract was then put into a holder to measure the mass and then tested using XRD.
b. Coloring
   Wet clay is dried directly using sunlight. Next, the clay is ground using a mortar and sieved using a sieve. Then the coloring process is carried out.

Determination of Mineral Type

The first X-Ray Diffraction measurement step is the sample is placed on the sample stage, when the computer is measuring, a graph will appear that describes the peaks of the sample being measured. This graph shows the relationship between 2 theta and intensity. Furthermore, the data is processed using HighScore Plus software. This measurement is carried out before and after the dyeing process

Fabric Dyeing

The first step is staining the fabric first. The clay sample is cooked with a weight of 1 kg and 2000 ml of water. The type of fabric used is sutera cloth, dobby cloth, primissima cloth, and prima cloth cut into 20x20 cm sizes. Then wetted by soaking (so that the pores of the fabric can be opened). As on, the cloth that has been moistened is put into the clay which is cooked for one hour with stirring. Then the cloth is left for 10 days by turning it back and forth every day. The cloth is then washed with water until it is clean. After that the cloth is dried in the sun without being exposed to the sun.

Color Resistance Testing on Fabric

At the time of testing the dried cloth was cut to a size of 5 cm x 5 cm.Put 2 ml of liquid detergent into 1000 ml of water and 2 g of powdered detergent into 1000 ml of water in different basins. The cloth was soaked for 5 minutes and washed manually for 2 minutes at 29ºC. When finished, the cloth is washed and dried without being exposed to direct sunlight for 1 hour, then the color changes on the cloth after washing are seen.

RESULTS AND DISCUSSION

The results of the measurement of clay extraction with X-Ray Diffractometer in the form of diffraction intensity with a diffraction angle (2θ). The measurement results are obtained in the form of a graphic called a diffractogram. Analysis of the diffractogram was carried out to obtain the type of mineral and crystal system. The mineral type is shown by comparing the angle of diffraction (2θ) and relative intensity (Ir) of the measurement result with the database mineral. The diffractogram was processed using Highscore Plus Software. Measurement data were obtained before and after the dyeing process on the fabric.

1. Data on the measurement results of B-CL-SPPS-210314-2 and S-CL-SPPS-210314-2 samples.

Data from the measurement of clay samples from Pesisir Selatan District using XRD obtained diffractograms before and after fabric coloring can be seen in Tables 1 and 2. Table 1 shows the results of XRD measurements on clay samples before the dyeing process on the fabric. The type of magnetic mineral formed at diffraction angles (2θ) 32.2331º, 54.7944º and 77.3336º is Maghemite with a Tetragonal crystal system and its lattice parameters a=b is 8.3400 , c is

8.3220 . In addition, other types of minerals at diffraction angles (2θ) 26.7211º, 39.7014º, 54.7944º and 77.3336º are Quartz. Thus the diffractogram analysis shows that the type of magnetic mineral present in the clay before staining is Maghemite (γ-Fe_2O_3). And the non-magnetic mineral is Quartz (SiO_2).

Table 1. Comparison of measurement data with mineral databases before dyeing on fabric

Measurement result data		Mineral database		Mineral type	Crystal System
2θ(0)	Ir(%)	2θ(0)	Ir(%)		
26.7211	63.90	26,642	100.0	Quartz	Hexagonal
32.2331	100.0	32.181	0.7	Maghemite	Tetragonal
39.7014	39.43	39,470	9.3	Quartz	Hexagonal
54.7944	18.14	55.008	0.1	Maghemite	Tetragonal
		55,330	2.5	Quartz	Hexagonal
77.3336	0.71	77.579	0.1	Maghemite	Tetragonal
		77.681	2.2	Quartz	Hexagonal

Table 2. Comparison of measurement data with mineral database after dyeing on fabric

Measurement result data		Mineral database		Mineral type	Crystal System
2θ(°)	Ir(%)	2θ(°)	Ir(%)		
20.9224	29.80	20,848	14.6	Quartz	Hexagonal
26.6781	100%	26,624	100%	Quartz	Hexagonal
32.3872	15.03	32.205	2.0	Maghemite	Tetragonal
45.7127	11.34	45,770	3.1	Quartz	Hexagonal
50.1568	32.05	50.169	1.6	Maghemite	Tetragonal
		50.109	1.6	Quartz	Hexagonal
59.9928	18.95	59,777	0.2	Maghemite	Tetragonal
		59,926	6.3	Quartz	Hexagonal
68.0466	15.25	68,368	0.3	Maghemite	Tetragonal
		68.096	6.3	Quartz	Hexagonal

The diffractogram analysis of the measurement results with the mineral database after staining on the fabric (Table 2) shows that the presence of magnetic minerals in the sample with diffraction angles (2θ) 32.3872º, 50.1568º, 59.9928º and 68.0466º, namely Maghemite with a Tetragonal crystal system and its lattice parameters. a=b is 8.3400Å, c is 8.3220Å. In addition, other types of minerals at diffraction angles (2θ) 20.9224º, 26.6781º, 45.7127º, 50.1568º, 59.9928º and 68.0466º are Quartz. Thus the diffractogram analysis shows that the type of mineral contained in the clay after staining is Maghemite (γ-Fe_2O_3). And the non-magnetic mineral is Quartz (SiO_2). Based on tables 1 and 2, you can see the results of XRD measurements in the form of a diffractogram as shown in Figure 2.

Figure 2 shows the types of magnetic minerals found in the clay before (a) and after (b) the dyeing process on the fabric. The number of diffraction peaks resulting from measurements before fabric coloring is 5 peaks while after fabric coloring is 7 peaks.

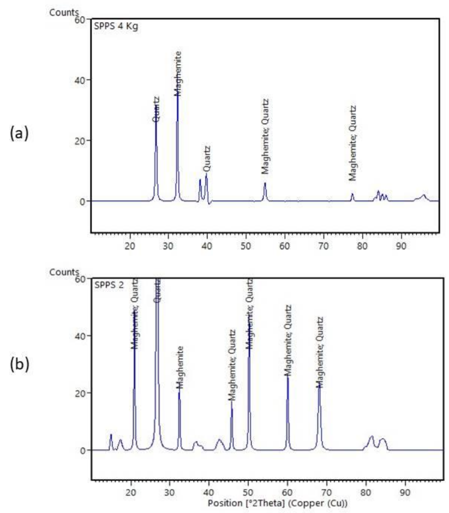


Figure 2. The results of X-Ray Diffraction measurements of clay samples from Sijunjung Regency before (a) and after (b) staining on fabrics

2. Measurement results B-CL-PSBSJJ-210421 and S-CL-PSBSJJ-210421.

The data from the measurement of clay samples in Sijunjung Regency using XRD obtained a diffractogram before and after fabric coloring can be seen in Table 3 and 4. The results of XRD measurements on the sample before staining on the fabric (Table 3) show that there are types of magnetic minerals in the sample with diffraction angles (2θ) 29.9650º, 32.2312º 35.5901º, 43.2212º, 53.8280º, 57.1998º, 62.8135º, 76.5398 , 87.0449º and 89.8772º is Maghemite with crystal system Tetragonal and the lattice parameters a=b is 8.3400 , c is 8.3220. In addition, other types of minerals at diffraction angles (2θ) 26.8591º, 39.4892º, 57.1998º and 87.0449º are Quartz. Thus the diffractogram analysis shows that the type of magnetic mineral present in the clay before staining is Maghemite (γ-Fe_2O_3), and the non-magnetic mineral is Quartz (SiO_2).

Table 3. Comparison of measurement data with mineral databases before dyeing on fabric

Measurement result data		Mineral database		Mineral type	Crystal system
2θ(0)	Ir(%)	2θ(0)	Ir(%)		
26.8591	11.50	26,624	100	Quartz	Hexagonal
29.9650	6.87	30,287	15.9	Maghemite	Tetragonal
32.2312	17.93	32.205	2.0	Maghemite	Tetragonal
35.5901	41.22	35,684	100.0	Maghemite	Tetragonal
39.4892	9.22	39,441	8.3	Quartz	Hexagonal
43.2212	15.82	43,363	17.7	Maghemite	Tetragonal
53.8280	5.13	53,827	11.9	Maghemite	Tetragonal
57.1998	17.82	57,366	23.7	Maghemite	Tetragonal
		57.201	0.2	Quartz	Hexagonal
62.8135	15.85	62,997	21.1	Maghemite	Tetragonal
76.5398	4.69	76.589	0.6	Maghemite	Tetragonal
87.0449	2.77	87,463	2.4	Maghemite	Tetragonal
		87.015	0.0	Quartz	Hexagonal
89.8772	0.00	89.402	0.3	Maghemite	Tetragonal

Table 4. Comparison of measurement data with mineral database after dyeing on fabric

Measurement result data		Mineral database		Mineral type	Crystal system
2θ(0)	Ir(%)	2θ(0)	Ir(%)		
26.7749	100.00	26,647	14.3	Quartz	Hexagonal
30.3429	25.18	30,321	33.6	Maghemite	Tetragonal
35.6229	65.56	35,684	100.0	Maghemite	Tetragonal
39,6109	15.05	39,478	9.0	Quartz	Hexagonal
43.2798	12.60	43,363	17.7	Maghemite	Tetragonal
50.2566	30.45	50.103	1.6	Maghemite	Tetragonal
		50,634	0.4	Quartz	Hexagonal
53.5621	30.45	53,827	11.9	Maghemite	Tetragonal
57.0255	41.72	57.252	0.2	Quartz	Hexagonal
		57,366	23.7	Maghemite	Tetragonal
60.0700	21.52	60,800	1.3	Maghemite	Tetragonal
		59,980	7.6	Quartz	Hexagonal
63.0106	28.95	63.073	41.3	Maghemite	Tetragonal
68.3036	21.26	68,368	0.3	Maghemite	Tetragonal
		68.337	6.4	Quartz	Hexagonal
80.0379	9.81	80,579	0.3	Maghemite	Tetragonal
		80.072	1.3	Quartz	Hexagonal
89.7829	12.37	89,610	0.4	Maghemite	Tetragonal

Analysis of the diffractogram measurement results with the mineral database after staining on the fabric (Table 4) shows that there are types of magnetic minerals in the sample with diffraction angles (2θ) 30.3429º, 35.6229º, 43.2798º, 50.2566º, 53.5621º, 57.0255º, 60.0700º , 63.0106º 68.3036º, 80.0379º and 89.7829º is Maghemite with crystal system Tetragonal and the lattice parameters a=b is 8.3400 , c is 8.3220.In addition, other types of minerals indiffraction angles (2θ) 26.7749º, 39.6109º, 50.2566º, 57.0255º, 60.0700º, 68.3036º and 80.0379º are Quartz. Thus the diffractogram analysis shows that the type of magnetic mineral contained in the clay after staining is Maghemite (γ-Fe2O3), and the non-magnetic mineral is Quartz (SiO2). Based on tables 7 and 8, you can see the results of XRD measurements in the form of a diffractogram as shown in Figure 3.

Figure 3 shows the types of magnetic minerals present in the clay before (a) and after (b) the dyeing process on the fabric. The number of diffraction peaks resulting from measurements before fabric dyeing was 12 peaks, while after fabric dyeing there were 13 peaks.

Based on the measurement results, it was found that there were differences in the number of peaks before and after dyeing the fabricbecause the amount of initial mineral lattice contained in the clay is less than the mineral lattice after the coloring process or vice versa is also due tothe presence of a damaged or reduced crystal lattice. The difference in the number of magnetic mineral diffraction peaks is influenced by the concentration of the type of magnetic mineral in a material[16] and crystals that diffract X-rays with a crystal lattice[17]. The diffraction pattern generated from the reflected rays will be captured by the detector and translated as a diffraction peak where the more crystal planes in the sample, the stronger the refractive intensity, which states that an X-ray beam scattered in a certain direction will produce a diffraction peak[18]. So, if there is a peak on the diffractogram then diffraction has occurred and if there is no peak on the diffractogram then there is no diffraction.

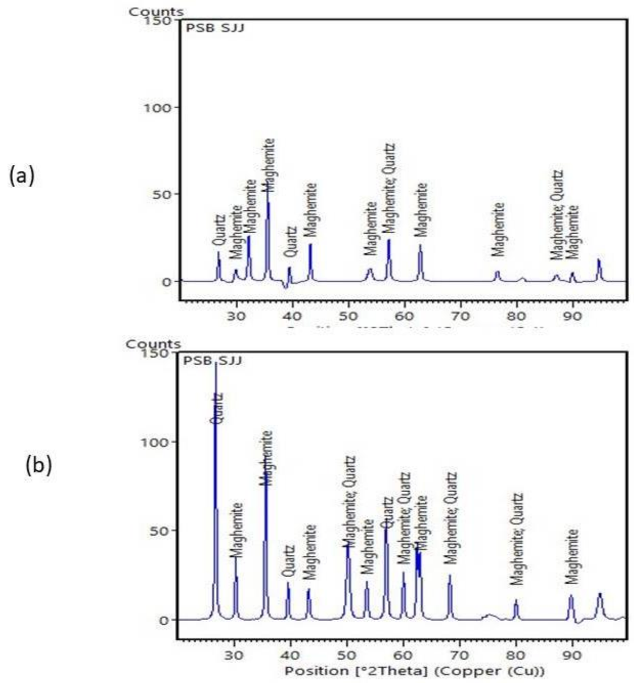


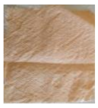
Figure 3. The results of X-Ray Diffraction measurements of clay samples from Sijunjung Regency before (a) and after (b) staining on fabrics

The resulting crystal system in the measurement of clay samples before and after dyeing the fabric is Tetragonal on Maghemite minerals. Mentioning the geometry of the crystal lattice will determine the direction of the beam reflected by the crystal[19]. Analysis of the types of magnetic minerals contained in the soil that dominates is Magnetite[20] and Hematite[21].

The color resistance test on the fabric was washed using liquid and powder detergents. The washing is done manually by soaking the cloth for 5 minutes and washing it for 2 minutes at a temperature of 29ºC. Then the cloth is rinsed with clean water and then dried in the sun without being exposed to direct sunlight for 1 hour at a temperature of 30ºC. The following is the difference in the color of the fabric after washing with liquid and powder detergent, which can be seen in Figure 4. Color resistance test This is done by observing the change or comparison of the original color on the fabric as (1) there is no change, (2) there is a slight change, (3) it has changed quite a bit, and (4) it has changed at all, due to the limitations of gray scale, colorimetry and spectrophotometry aids. to get the right research results.

Table 5 shows there is a sample of CL-SPPS-210314-2 does not change color with the original fabric after washing, on the contrary in the fabric sample CL-PSBSJJ-210421 experienced quite a change from the original color of the fabric or before washing. Changes in color or discoloration after washing are caused by the adhesion force of the clay in the dyeing process with the cloth and the temperature during cooking of the clay so that there are loose elements. Discoloration is caused by detergent content, which is an addictive substance in accordance with) theory which states that detergent is a cleaning agent containing surfactant compounds that can reduce the surface tension of water and soften the existing fat[22]. Other substances besides surfactants found in detergents include builders and additives such as fragrances, bleaches, solvents and other substances related to product commercialization[23].

Table 5. Color resistance test results on fabric

Sample name	Magnetic mineral type	Fabric type	Fabric color before washing	Fabric color after washing		Color resistance evaluation
				Liquid detergent (a)	Powdered detergent (b)	
CL-SPPS-210314-2	Maghemite	Dobby				a) 1 b) 1
		Silk				a) 1 b) 1
		prime				a) 1 b) 1
		Primis				a) 1 b) 1
CL-PSBSJJ-210421	Maghemite	Dobby				a) 3 b) 3
		Silk				a) 3 b) 3
		prime				a) 3 b) 3
		Primis				a) 3 b) 3

Fabric samples that did not change color after washing or were resistant had a peak number that changed or was not constant due to differences in the amount of the initial mineral lattice or before with the number of mineral lattices after the dyeing process. The types of magnetic minerals contained in the clay before and after the coloring process did not change or remain. Fabric samples that do not change color after pre-washing have a color on the fabric that is less intense or faded. While the fabric samples that experience fading or discoloration after being washed previously have a dense fabric color, this is caused by other minerals that are dominantly contained in the clay.

CONCLUSION

Based on the results of research on clay samples, it can be concluded that the results of the types of magnetic minerals contained in clay in West Sumatra through X-Ray Diffraction measurements before and after the coloring process, namely Maghemite (γ-Fe_2O_3). The clay in the Pesisir Selatan and Sijunjung areas has magnetic minerals. The test results obtained that the color resistance of the dobby, silk, prime and prime type fabrics experienced fading. In the CL-SPPS-210314-2 does not change color,sand on fabric samplesCL-PSBSJJ-210421 experienced quite a change from the original color of the fabric or before washing.

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