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Identification of Clay Element Content in Fabric Dyeing Process Using X-Ray Fluorescence Spectrometry

Reni Fitria Nengsih, Hamdi Rifai✉, Letmi Dwiridal, Akmam, Ahmad Fauzi, and Yusrizal

Department of Physics, Faculty of Mathematics and Natural Sciences, Padang State University, Jl. Prof. Dr. Hamka, Padang 25131, Indonesia

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ABSTRACT

Clay in West Sumatra has been used as a natural dye for batik, so the batik is known as clay batik. The dyeing of the cloth is done by boiling and soaking the clay together with the cloth for 10 days. The color of the fabric is caused by certain elements present in the clay. Currently, no one has investigated the changes in elemental content due to the dyeing process. This research aims to investigate the changes in the concentration of the elements that affect the color of the fabric. Samples were taken from 4 areas, namely Solok, Sijunjung, Lima Puluh Kota, and Pesisir Selatan with a total of 8 samples, of which 4 samples were measured before the staining process and 4 more samples after the staining process. Changes in the elemental content of clay were investigated using *X-Ray Fluorescence* (XRF). The results of the study showed changes in the content of the dominant element, namely Al, Si, and Fe contained in samples CL-PYKLN 2A and CL-TJG GDG 210422. While the samples CL-PSB-SJJ 210421 and CL-SPPS 1 210314 the change in the concentration of the elemental content was not too significant. The elements that cause colored cloth are all elements in clay, but the dominant element in coloring cloth is Fe.

✉ Corresponding author:
Department of Physics, Padang State University
E-mail: rifai.hamdi@fmipa.unp.ac.id

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INTRODUCTION

Land Clay is a brownish white soil with relatively low hardness, containing alumina, silica, magnesium oxide, and iron (Nisah, 2017). The arrangement and composition of different chemical elements, such as iron (Fe) contained in clay can form a magnetic mineral. Magnetic minerals have high magnetic properties (Nasution et al., 2013), such as ferromagnetic minerals. Examples of ferromagnetic minerals are magnetite (Fe_3O_4) with the same content as $Fe_3O_4 \rightarrow Fe_2^{+3}Fe^{+2}O_4^{-2}$, hematite ($\alpha$-$Fe_2O_3$), and maghemite ($\gamma$-$Fe_2O_3$) with the equation for the content of γ-$Fe_2O_3 \rightarrow Fe_2^{+3}O_3^{-2}$. The content of the element iron (Fe) in the form of hematite gives a red color, magnetite e I give brown color, and ilmenite gives a yellow color (Sartohadi et al., 2014), geothite gives a brown color (Holilullah et al., 2015). The colors in this soil occur due to the presence of iron oxide and organic elements, which will usually burn brownish yellow, red-brown, rust color, or dark brown, depending on the iron oxide and impurities contained (Hastutiningrum, 2013). Clay can be developed as an industrial material with high economic value (Nurkhusna, 2020), which can be used as a natural dye for clay batik (Octaviana, 2019). Clay batik is a typical batik originating from West Sumatra (Putri & Midawati, 2020) .

Many types of research on clay batik have been carried out, starting from the history of clay batik, motif designs, and techniques of clay batik as natural dyes. So far there has been no study on changes in the content of clay elements due to the fabric coloring process. The resulting color depends on the mineral or element content in the clay (Holilullah et al., 2015). Identification of clay elements needs to be known in the process of dyeing fabrics to determine the elements that cause color in fabrics. To determine the elemental content before and after the fabric dyeing process was investigated using *X-Ray Fluorescence Spectrometry*. XRF (*X-Ray Fluorescence Spectrometry*) is a tool that can be used to determine the elements of the material. XRF spectrometry utilizes X-rays emitted by the material which are then captured by the detector to analyze the elemental content in the material.

The material analyzed can be in the form of determining the concentration of elements present in solid, powder, or liquid samples that can be dried first. Elemental analysis was carried out both qualitatively and quantitatively. Qualitative analysis analyzes the types of elements contained in the material and quantitative analysis is carried out to determine the concentration of elements in the material (Munasir et al., 2012). The advantages of the XRF method are that it is easy to carry out measurements and can detect various elements. From the studies that have been carried out, no one has studied the content of clay elements in the fabric coloring process. Therefore, it is necessary to study the identification of changes in the content of the magnetic mineral-forming elements of clay before and after the dyeing process investigated using *X-Ray Fluorescence Spectrometry*.

METHOD

Sampling locations in West Sumatra are Solok, Sijunjung, Lima Puluh Kota, and Pesisir Selatan. Sample preparation, data collection, and sample measurement were carried out at the Geophysics Laboratory, Department of Physics and Chemistry Laboratory, FMIPA, Padang State University. The following sampling locations in this study can be seen in Figure 1.

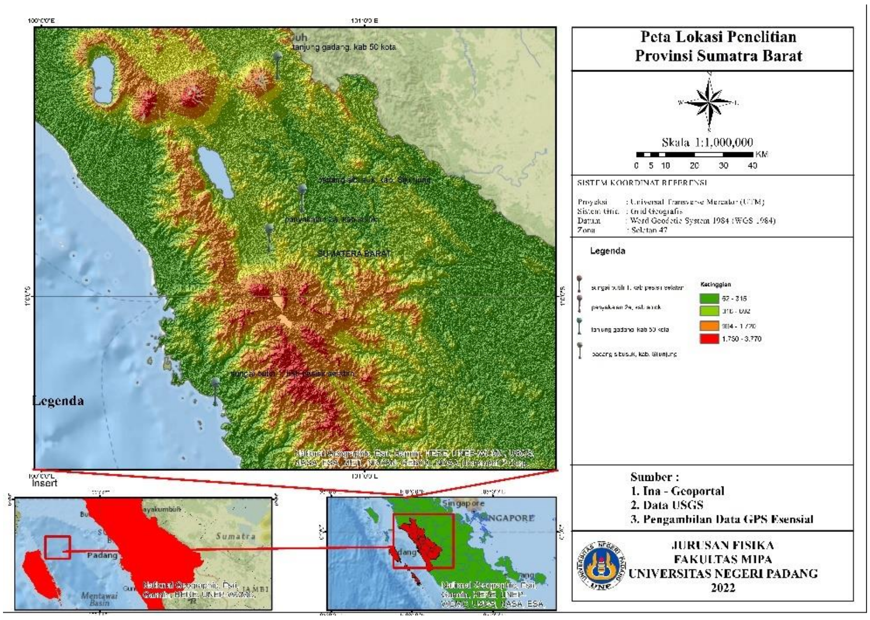


Figure 1. Sampling location map

Research procedure

This research consisted of sampling, sample preparation, sample measurement, and making samples of batik cloth coloring.

Sampling

The sampling procedure first prepares tools and materials such as GPS, plastic, hoes, and shovels. Determine the coordinates of the sampling location. Scrape the surface of the clay to make it easier to take pure clay so it doesn't become contaminated. Put the sample into the sample plastic and label it.

Sample Preparation

Before determining the composition of the clay element using *X-Ray Fluorescence* (XRF), the existing sample preparation was carried out first. The steps taken in sample preparation are as follows: The clay is dried in the sun to dry. Smooth the sample using a mortar. Put the sample that has been in the form of fine grains into the holder and give it a name using label paper.

XRF Measurement

The XRF measurement step prepares the sample. Characterization of samples with *X-Ray Fluorescence* (XRF) test equipment serves to determine the variation of chemical elements and the percentage of elements contained in the sample. Directing the XRF test equipment towards the surface of the sample and then firing X-rays. The data appears on the program monitor screen by displaying the composition in detail.

Making batik fabric coloring samples

The first step is to make a sample of the color of the clay batik cloth and cook it weighing 1 kg with 2000 ml of water. The fabrics used are silk, dobi, primissima, and prima fabrics, which are cut into 15 x 15 cm sizes. The cloth is soaked with water (so that the fabric pores can be opened). Clay mixed with water, then filtered. The filtered soil is put into a pot that has been filled with water. Put the soaked cloth in the pan. Cook for 30 minutes (occasionally remove the cloth so that the color is even). After cooking the cloth is cooled and soaked for 10 days (the cloth must be removed every day) in a closed basin. The fabric is locked so the color can last a long time. The cloth is cleaned with water until it is clean. The final stage of the fabric is dried in the sun without being exposed to direct sunlight.

RESULTS AND DISCUSSION

The combined results of the measurement of elemental content using XRF from 4 samples of clay before and 4 samples after the dyeing process on the fabric, where for the Solok area with the sample code CL-PYKLN 2A 210421, the Sijunjung area with the sample code CL-PSB-SJJ 210421, the Lima Puluh area. The city with the sample code CL-TJG GDG 210422, and the Pesisir Selatan area with the sample code CL-SPPS 1 210314 can be seen in Table 1.

Table 1. Elemental content using XRF

No	Element	Element											
		CL-PYKLN 2A 210421			CL-PSB-SJJ 210421			CL-TJG GDG 210422			CL-SPPS 1 210314		
		Before Coloring (%)	After Coloring (%)	Δ	Before Coloring (%)	After Coloring (%)	Δ	Before Coloring (%)	After Coloring (%)	Δ	Before Coloring (%)	After Coloring (%)	Δ
1	Al	17.352	26.605	-9.253	14.239	13.949	0.29	18.468	5.196	13.272	17.362	17.284	0.078
2	Si	51.98	40.328	11.652	62.946	62.155	0.791	41.125	14.795	26.33	41.457	40.569	0.888
3	P	2.432	1.088	1.344	3.726	3.524	0.202	5.076	3.162	1.914	2.793	2.789	0.004
4	K	3.604	2.269	1.335	2.879	2.712	0.167	5.367	4.199	1.168	2.938	2.918	0.02
5	Ca	0.872	0.308	0.564	1.206	1.109	0.097	2.754	0.991	1.763	0.964	0.956	0.008
6	Ti	2.197	1.241	0.956	2.27	2.249	0.021	3.357	2.128	1.229	3.281	3.227	0.054
7	V	0.079	0.041	0.038	0.048	0.047	0.001	0.138	0.074	0.064	0.085	0.081	0.004
8	Cr	0.144	0.1	0.044	0.044	0.026	0.018	0.039	0.022	0.017	0	0	0
9	Mn	0.015	0.011	0.004	0.008	0.007	0.001	0.072	0.036	0.036	0.051	0.047	0.004
10	Fe	31.261	15.43	15.831	12.639	11.814	0.825	60.401	28.281	32.12	30.292	29.57	0.722
11	Ni	0.082	0.037	0.045	0.012	0.01	0.002	0.025	0.005	0.02	0.007	0.002	0.002
12	Zn	0.039	0.014	0.025	0.023	0.022	0.001	0.165	0.054	0.111	0.076	0.076	0
13	Ga	0.02	0.008	0.012	0.013	0.011	0.002	0.049	0.017	0.032	0.021	0.021	0
14	As	0.024	0.007	0.017	0.007	0.007	0	0.014	0.003	0.011	0.003	0.003	0
15	Rb	0.051	0.018	0.033	0.044	0.039	0.005	0.105	0.035	0.07	0.036	0.036	0
16	Sr	0.038	0.014	0.024	0.024	0.022	0.002	0.029	0.01	0.019	0.036	0.036	0
17	Y	0.02	0.007	0.013	0.02	0.019	0.001	0.038	0.013	0.025	0.019	0.019	0
18	Zr	0.149	0.056	0.093	0.261	0.253	0.008	0.435	0.145	0.29	0.192	0.19	0.002
19	Ag	1.127	0.687	0.44	0.867	0.597	0.27	1.619	1.076	0.543	1.125	1.085	0.04
20	Eu	0.107	0.054	0.053	0.042	0.042	0	0.186	0.103	0.083	0.111	0.108	0.003
21	Yb	0	0	0	0.007	0.001	0.006	0.004	0.003	0.001	0	0	0
22	Re	0.002	0.001	0.001	0.001	0.001	0	0	0	0	0.001	0.001	0
23	Pb	0.026	0.013	0.013	0.012	0.011	0.001	0.064	0.026	0.038	0.026	0.026	0
24	Cu	0.03	0.011	0.019	0.009	0	0.009	0.069	0.023	0.046	0.029	0.027	0.002

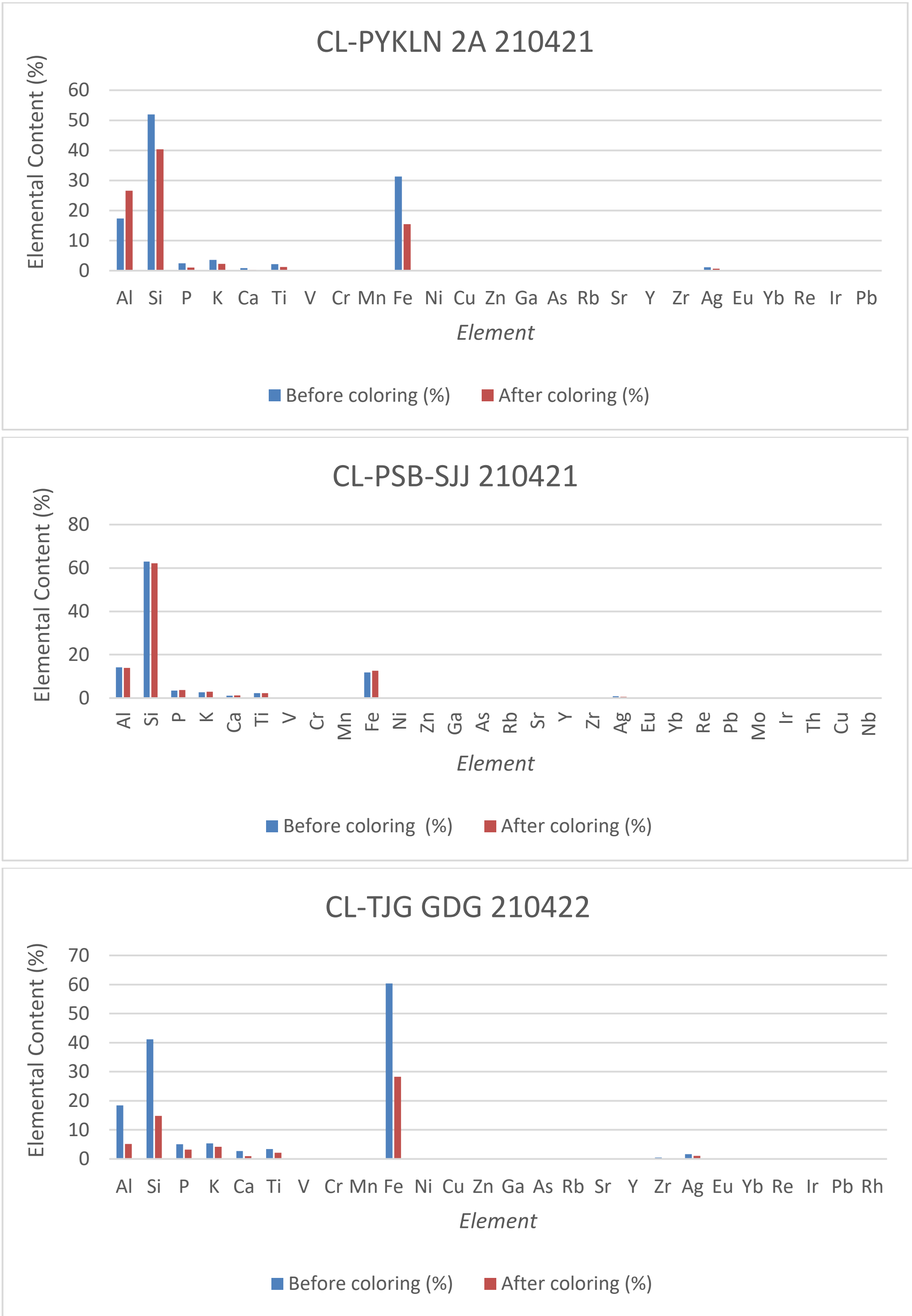
Table 1 shows that the element that experienced an increase after the coloring process was Aluminum (*Al)* as much as 9.253 contained in the sample with the code CL-PYKLN 2A 210421. While the elements *Fe, Si, P, Cl, K, Ca, Ti, V, Cr, Mn, Ni, Cu, Zn, Ga, As, Rb, Sr, Y, Zr, Ag, Eu, Yb, Re,* and *Pb* decreased after the staining process.

The histogram of the percentage of *elements* before and after the fabric coloring process for each sample can be seen in Figure 2.

(a)

(b)

(c)



(d)

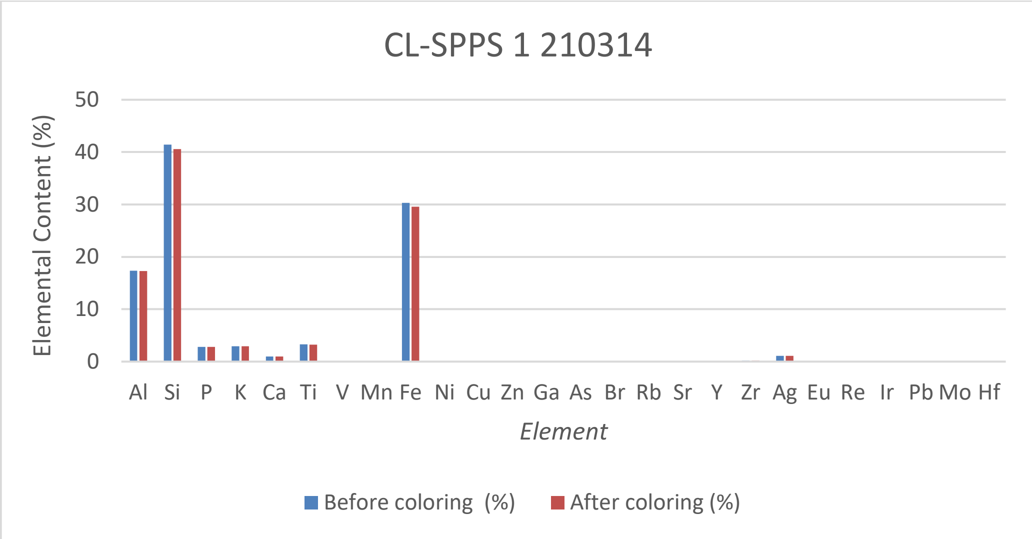


Figure 2. Histogram of clay element content from XRF measurements on samples (a) CL-PYKLN 2A 210421, (b) CL-PSB-SJJ 210421, (c) CL-TJG GDG 210422, (d) CL-SPPS 1 210314.

Figure 2 shows that the results of clay measurements using *X-Ray Fluorescence* (XRF) change the concentration of the elemental content of each sample. The dominant elements that changed after the staining process were *Al*, *Si,* and *Fe* found in samples CL-PYKLN 2A 210421 (Figure (a)) and CL-TJG GDG 210422 (Figure (c)). While the samples CL-PSB-SJJ 210421 (Figure (b)) and CL-SPPS 1 210314 (Figure (d)) the change in the concentration of the elemental content was not too significant. The most common elements found in natural clay are Si, Al, Mg, and Fe (Boumaiza. 2020). Other clay-forming elements that change concentration are *P, Cl, K, Ca, Ti, V, Cr, Mn, Ni, Cu, Zn, Ga, As, Rb, Sr, Y, Zr, Ag, Eu, Yb, Re,* and *Pb*. This is following Putri's research (2017) that the dominant clay element content is *Si, Fe,* and *Al,* while the other elements are *K, Ca, Ti, P, Sr, Mn, Ba, Zr, Rb, Nb, Mo, Zn, Sn, In, Ru.*

Fe element from each sample decreased in the elemental content after staining. Clay with sample code CL-PYKLN 2A 210421 (Figure (a)) *Fe* content before coloring was (31.261). After being colored, the elemental content decreased to (15.43). Likewise, sample CL-TJG GDG 210422 (Figure (c)) decreased by (32.12), sample CL-PSB-SJJ 210421 (Figure (b)) decreased by (0.825), and sample CL-SPPS 1 210314 (Figure (d)) decreased by (0.722). This is a decrease due to the process of dyeing the fabric and soaking it for 10 days so that the elemental content moves to the fabric and produces a light brown color. Nurkhusna (2020) Clay contains *Fe* so that it can bring out colors that can be used as natural textile dyes. Based on the results of interviews conducted with a batik craftsman named Yusrizal on April 21, 2021, information was obtained that the cloth which was originally pure white, after being soaked in clay, will produce a basic colored cloth that tends to be cream or light brown. Natural dyes can be obtained by making an extraction which will be obtained by boiling the clay to be used, the purpose of making the extraction is to take the color pigments in the clay. Make clay extract for 4 pieces of cloth each measuring 15 x 15 cm. Requires 1 kg of clay and 2000 ml of water. In the opinion of Lemmens (1999:20), the extraction process is carried out by boiling the material with water as a solvent.

Al element in each sample experienced a change that occurred in the CL-PYKLN 2A 210421 sample (Figure (a)) increased by (-9.253), this was due to the pan used being an aluminum pan and the sample that was cooked for the first time was the CL-PYKLN sample. 2A 210421 (Figure (a)). The *Al* element in the pan moves to the ground, so it increases. The sample which was then cooked was the sample CL-TJG GDG 210422 (Figure (c)), where there was a decrease of (13.272). Likewise, the sample CL-PSB-SJJ 210421 (Figure (b)) was (0.29) and CL-SPPS 1 210314 (Figure (d)) was (0.078). Before the dyeing stage, the cloth is soaked for 30 minutes, after that the cloth is dipped in the extract solution while being turned over evenly and then aerated. Dyeing is done at least 5 to tens of repetitions until the resulting color is as desired. This is following the theory of Sunaryati (2000:40) which states that the natural color dyeing process can be carried out between 5 and 30 times according to the desired color density.

CONCLUSION

Based on the results of research on clay samples, conclusions can be drawn, namely: Results of clay measurements using *X-Ray Fluorescence* (XRF) changes in the concentration of the element content. The elements that decreased after the staining process were *Al*, *Si,* and *Fe* found in samples CL-PYKLN 2A and CL-TJG GDG 210422. While the samples CL-PSB-SJJ 210421 and CL-SPPS 1 210314 the change in the concentration of the elemental content was not too significant. Other elements that experience changes in the concentration of clay-forming elements are *P, Cl, K, Ca, Ti, V, Cr, Mn, Ni, Cu, Zn, Ga, As, Rb, Sr, Y, Zr, Ag, Eu, Yb, Re,* and *Pb*. The elements that cause colored cloth are all elements in clay, but the dominant element in coloring cloth is Fe.

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