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Geochemical Studies of Claystone Based on Analysis of Scanning Electron Microscope (SEM), Talangsawah, Merapi District and Surroundings of Lahat Regency, South Sumatra

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Abstract. Claystone formation is related to its geological condition, which is where the claystone is formed. The claystone environment is very influential on the composition that supports the formation of the claystone. The fine-sized claystone in the petrography is very difficult to determine its composition so by using the Scanning Electron Microscope (SEM-EDX) analysis it will be known the claystone environment. The purpose of this study was to determine the claystone environment based on its chemical composition. The SEM-EDX analysis showed that five samples were analyzed, claystone samples (F-1, F-2, F-3 and F-4) consisting of calcite, halite, and siderite minerals characterizing marine environment deposits, and claystone samples (F-5) consists of siderite, kaolinite, and pyrite minerals characterizing subaerial environmental deposits in the abandoned channel.

1 Introduction

Claystone is a clastic sedimentary rock that has clay grain size, including granules that have a diameter of less than 1 or 2 microns and are predominantly arranged by silica [1]. Because of its very fine grain size, it is difficult to describe claystones either megascopically or microscopically, so chemical analysis is important information to determine claystone composition. Claystone formation is related to its geological condition, which is where the claystone is formed. The claystone environment is very influential on the composition that supports the formation of the claystone. The claystone environment will give an overview of the process or geological conditions of the area studied. It is important to know the environment and claystone composition. Claystones formed in different regions have different physical features [2]. Claystones formed in the sea generally have thick coatings, contain deep sea fossils, or animals that live in shallow seas which then sink after death [3].

Based on [4] some index minerals can be used in defining the environment of a deposition process, which can be seen from the content of certain chemical elements. From the results of SEM analysis, the results are in the form of surface morphology of samples that can display the crystalline form of a mineral and EDX analysis produces a percentage of the chemical elements contained in the sample,



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which serves to determine the mineral composition contained in rocks. So that the authors make an interpretation of the depositional environment of the three samples based on data from SEM and EDX analysis.

According to [5] the most common clay minerals are illite, kaolinite and chlorite. Whereas smectite (montmorillonite and saponite) is a mineral that is rarely present. These minerals consist of elements of silicon, aluminum, oxygen and hydrogen. The analysis method of Scanning Electron Microscope (SEM) is carried out for special research studies. The analysis was conducted on fine-grained rock samples, in the study area was claystone. One method used in understanding claystone based on the composition of its constituent minerals is Scanning Electron Microscope (SEM) and Energy Dispersive X-Ray (EDX) analysis. In the SEM analysis method using an electron beam whose wavelength is shorter than light waves, so that it can display a higher magnifying resolution than a light microscope. The SEM analysis method is able to display sample surface information, which serves to determine minerals based on the shape of the crystal. Accompanied by EDX analysis method that produces a percentage of the presence of elements contained in the sample.

Scanning Electron Microscope (SEM) analysis method on fine grained rock samples, where in the study area was claystone with determination of claystone constituent minerals [6]. SEM analysis was carried out on claystone due to the very fine size of clay minerals making observations in polarization microscopes in petrographic analysis displaying poor resolution. So it is difficult to determine the type of mineral. Using the SEM analysis method, observations will display a three-dimensional formation on granules, especially on very fine grain sizes such as clay to facilitate the determination of the types of constituent minerals. In addition, another advantage of SEM observation is displaying a larger resolution with an enlargement range between $10 \times$ to $20,000 \times$. In identifying minerals in SEM analysis, they are in the form of surface appearance / sample morphology.

2. Methodhs

SEM analysis utilizes interactions between source electrons and sample electrons, resulting in emission of electrons or photons. SEM samples will be placed in the sample room. Furthermore, the sample will be fired with a beam of high-energy electrons, then there will be an interaction between the source electrons and the sample electrons. The results of electron interactions will be recorded by a detector which results in the display of the sample surface morphology on the observation screen. In SEM-EDX analysis the picture of the analysis results was taken using the Tescan Vega3 tool. While the EDX analysis process occurs during the process of firing electrons at a certain point. Interactions between electrons can cause emissions from X-rays that display a graph of the presence of elements contained in the sample. Scheme of working principle of electron microscope to display SEM-EDX analysis data. (Figure. 1)

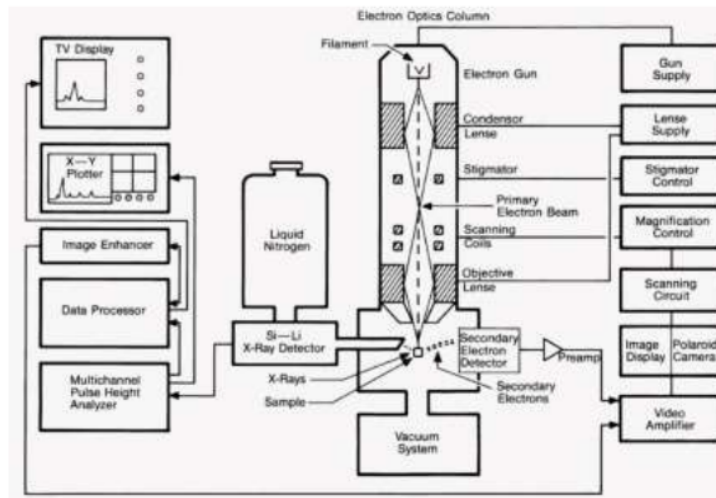


Figure.1 Scheme of the working principle of electron microscopy to display SEM-EDX analysis data [1]

The sample used came from the TalangSawah area, Merapi and surrounding districts of Lahat, South Sumatra. The rock sampling method can be done by taking samples from outcrops on the cliffs or on the river walls. Rock sampling techniques are carried out with rock samples on the surface. Rock samples can be taken directly from the location of observation of rock outcrops found in the field in a condition that is not weathered (fresh).

The sample requirements for SEM analysis are solids in the form of fine grains and in a dry state. The sample size needed is small so that it is enough to be inserted into the SEM sample room. The initial stage of sample preparation for SEM analysis is the destruction of samples to form fine grains. In addition, the analysis using Energy Dispersive X-ray (EDX) techniques was also carried out to obtain quantitative results of the chemical elements contained in the sample which made it easier to find out the constituent minerals of the rock. (Figure.2)

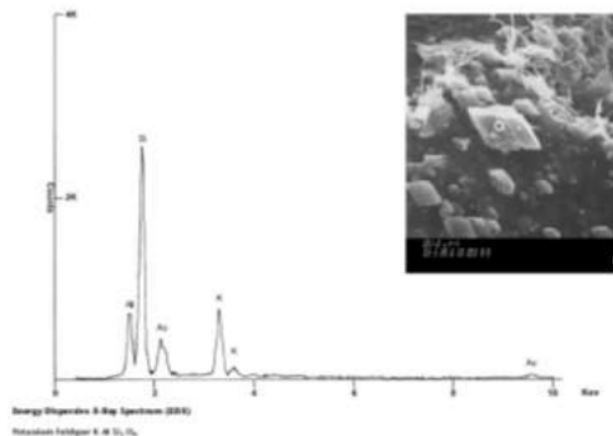


Figure.2. The image identifies the graph from EDX analysis and adjusts the crystal shape of the sample surface image on the results of SEM analysis [5].

3. Results and Discussion

The results of the SEM-EDX analysis of the sample were distinguished by looking at the morphology of the rock-forming minerals and the percentage of the presence of chemical elements. The presence of a group of silicate minerals is determined based on the presence of elements of silicon (Si) with the presence of quartz minerals in the detrital form of SEM with the presence of Si and O elements, aluminum (Al), and potassium (K). (Table 1.) (Figure 3)

Table 1. Comparison of the presence of elements in each claystone sample

Elements	% claystone F-1	% claystone F-2	% claystone F-3	% claystone F-4	% claystone F-5
O	53,84	52,51	51,94	54,44	56,45
Si	16,26	22,21	26,67	19,91	22,22
Al	11,20	11,71	12,69	12,11	12,32
C	10,60	10,08	6,22	8,89	5,76
Ca	4,54	0,39	0,35	0,23	0,00
K	1,5	1,16	0,63	1,72	0,95
Fe	1,06	1,09	0,86	2,50	1,12
Na	0,90	0,27	0,07	0,16	0,06
S	0,14	-	-	0,06	1,12
Mg	-	0,59	0,58	-	-

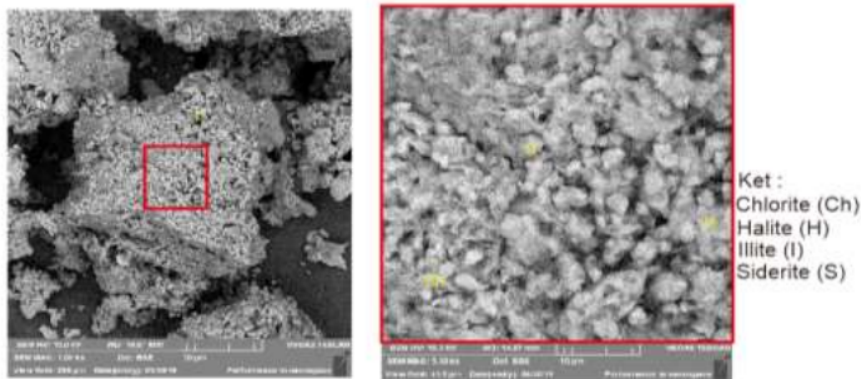


Figure 3. The morphology of the SEM-EDX analysis in claystone samples was determined based on its crystalline shape with 1,000 × (left) observation magnification and 5,000 × magnification (right)

Mineral chlorite with the form of pseudo-hexagonal crystal and flake shape, characterized by the presence of the main elements that need to be considered consisting of Si, Al, and iron (Fe). Illite minerals with a pseudo-hexagonal crystalline form characterized as smooth elongated, based on the presence of the main elements that need to be considered consisting of elements Si, Al, and K. Halite minerals are crystalline cubic and interpreted based on the presence of sodium (Na) (Figure 30). Mineral calcite in the form of rhombic crystals which are interpreted based on the presence of calcium (Ca). Siderite minerals are rhombic crystals based on the interpretation of the presence of Fe, C, and O elements.

In claystone samples some index minerals can determine the depositional environment consisting of calcite, siderite. Mineral characterizing marine environments such as carbonate mineral groups such as the presence of calcite and siderite minerals. The content of element C forms inorganic carbon which produces carbonate (CO₃) as a result of the reaction between CO₂ and H₂O. As well as the presence of Ca elements in the EDX analysis (4.54% - 0.23%) with this percentage indicating the shell of marine micro-organisms, can react to minerals that are carbonate to form CaCO₃. However, the interpretation of absent mineral pyrite in this sample is because the formation of mineral pyrite is influenced by the sulfur content which involves the activity of sulfate reducing bacteria [4] Based on [2] in the formation of pyrite the content of S elements contained in the sample must be more than 1%. So that the results of interpretation of claystone depositional environments (F-1, F-2, F-3 and F-4) are based [10] are the type of deposition of the marine environment

F-4 claystone samples contained Fe of 2.5%. The high element content of Fe in the sample interpreted binds to the carbonate that forms siderite minerals and does not form mineral pyrite. F-5 claystone samples based on index minerals that can determine the depositional environment consist of kaolinite, siderite and pyrite minerals. The presence of the S element in the EDX analysis results in a greater percentage than the other samples indicates the influence of sea water during the deposition process. But the absence of the Ca element indicates that the depositional environment is not in the marine environment. Based on (Worden & Burley, 2003) (Figure 4) the results of the analysis of the content of elements contained in claystone samples in deposition interpretation are in the subaerial-wet condition depositional environment at the abandoned channel. The influence of sea water in the interpretation of the presence of S elements in the EDX sample F-5 analysis results, with a percentage of 1.12%. The

content of a high sulfur element of more than 1% will contain more inorganic sulfur content which is pyritic [6].

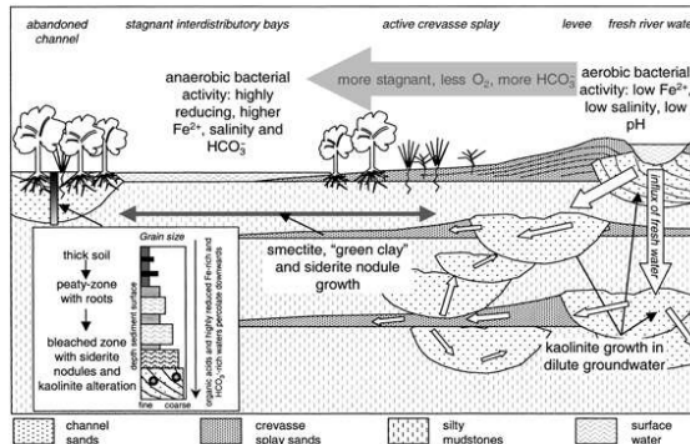


Figure 4. Subaerial depositional environment diagram based on wet and humid conditions

4. Conclusion

From the results of SEM-EDX analysis, the minerals that make up the F-1 sample consist of groups of silicate minerals, k-feldspar, chlorite, illite, halite, calcite, and siderite, in the deposition environment interpretation is a type of ocean deposition. Samples of F-2, F-3 and F-4, minerals that were present consisted of groups of silicate minerals, k-feldspar, chlorite, illite, halite, calcite and siderite with the interpretation of the depositional environment as a type of ocean deposition. Whereas for sample F-5 has darker and carbonic physical characteristics with its constituent minerals consisting of silicate minerals, kaolinite, siderite and pyrite in the deposition environment interpretation is a subaerial wet condition-abandoned channel deposition type.

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