Scanning electron microscope (SEM) imaging and analysis of magnetic minerals of lake Diatas peatland section DD REP B 693

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Abstract. Peatlands are formed from the accumulation of organic matter that originate from weathering vegetation for a long time. Minerals within peatlands can come from volcanic ash and consist of several types of minerals, one of which is magnetic minerals. This research aims to look the morphology, elemental composition and type of magnetic minerals found in the Lake Diatas peatland in Solok Regency, Indonesia because magnetic minerals have different morphologies depending on the source. The selected samples were from core DD REP B 693, which had a high magnetic susceptibility value. The magnetic minerals in the peatland were imaged and identified using Scanning Electron Microscope (SEM), which was equipped with Energy Dispersive Spectrometer (EDS). The results of morphological analysis showed that peatland had oval-shaped minerals with fractures and are multidomainal. The Back Scattered Electron (BSE) images show that the minerals have a bright colored surface that indicates the presence of a high amount of Fe. The EDS results showed that magnetic minerals from the peatland predominately contain the elements Fe, Ti, Si, and O. Based on the relative proportions of these elements, the magnetic minerals that can be formed in peatland are Magnetite (Fe₃O₄) and Ilmenite (FeTiO₃).

1. Introduction
Volcanic ash and sand are pyroclastic material originating from volcanoes which are sprayed during eruptions [1]. The erupted materials have a range of sizes, can travel far and forms well-preserved tephra layers in quiet [2]. Large materials will fall 5-7 km from the crater, while smaller ones will fall at a distance of tens of kilometers after transportation by wind [3]. Material carried by the wind will be deposited directly from the center of the eruption and can also be transported through transport media in the form of water where during the transport process material mixing will occur and will be deposited in various places. One place that might be a place for deposition of volcanic ash and sand is peatland, which is land formed from the long-term accumulation of organic matter from weathering vegetation [4]. These peatlands contain several types of minerals and one of the minerals that may be present on peatlands is magnetic minerals.

We can infer existence of magnetic minerals by using rock magnetization methods are generally effective, efficient, and affordable [5]. For example, the abundance of magnetic minerals in a material
can be determined by measuring the value of magnetic susceptibility using the rock magnetization method. The value of the susceptibility of magnetic minerals in peat soils with topographic differences has been analyzed by Rothwell (2007) and it was found that peatland adjacent to the edge of the gutter has a high concentration of magnetic minerals [6]. In addition, magnetic minerals can also be identified by looking at the morphology and elemental composition [5]. This is done because magnetic minerals have diverse morphologies depending on the source [7].

Morphology is of the shape and size of a grain. Magnetic grains with generally round shapes indicates that the mineral comes from an anthropogenic process. Whereas oval-shaped minerals with and fractures indicate that the mineral has been eroded from its source rock and redeposited [7]. The morphology can be observed using Scanning Electron Microscope (SEM), which utilizes the interaction of high energy electrons with the surface of the mineral grains. The SEM method has been used by Sari (2014) to identify magnetic minerals in guano, which are oval shaped and have many fractures on the surface. The top of these guano grains is bright, and the underside of the grain is dark [8]. However, the observation of magnetic minerals from peatlands using SEM has never been done. Therefore, research was conducted on the magnetic mineral content of the peatland in the Lake Diatas area of Solok Regency to see what kind of magnetic minerals are present and determine their origin by looking at the morphology and chemical composition.

2. Methods
This research was conducted on peat samples of the Lake Diatas peat land in Solok Regency, West Sumatera, Indonesia are show in Figure 1.

![Figure 1. Map of Sampling Location](image-url)
In Figure 1 shows sampling in position 1°4’19.93”S and 100°46’13.53”E. Sampling was carried out in March 2018 using a peat corer with a 0.5 m long container on the bottom. A 7-meter long core was retrieved from the peatland, placed in a PVC pipe and taken to the Geophysical Laboratory of FMIPA UNP. The sample was then measured using the MS2C Magnetic Susceptibility Meter every 1 cm depth. A high susceptibility value was obtained at a depth of 693 cm, indicating a high concentration of magnetic minerals. After that, a sample of DD REP B 693 is taken and inserted into the holder to be measured using the MS2B Magnetic Susceptibility Meter.

Then the sample was dried by leaving it at room temperature for few days so that the magnetic minerals could be easily extracted from the sample using a strong magnet coated with plastic. The results of this extraction were taken to the Asian School of the Environment Laboratory at Nanyang Technological University, Singapore to carry out further preparations. First the samples were washed in an ultrasonic cleaner, which is a sample washing device that uses ultrasonic waves and water, to separate clay and organic matter. During the ultrasonic washing, the magnetic minerals contained in the sample settled to the bottom of the beaker, and the clay and organic matter became suspended in the water. The clay and organic matter were then poured out with the water. The washed magnetic minerals were then dried in the oven for ±1 hour. Then each sample was poured onto double-sided tape through one of the 12 holes of a sample mount are shows in Figure 2.

![Figure 2. Sample Preparation Process](image)

In Figure 2 we can see there are 12 different samples in each hole. The process of measuring morphology and elemental composition was carried out using a JEOL JSM-7800F SEM equipped with BSE and EDS detectors. The samples were fixed onto sample holder located inside the SEM and loaded into the sample chamber. Once the scanning electron beam began sweeping across the specimen surface, the reflected BSE were collected by the detector, and an image of the sample surface morphology was displayed. The X-rays produced by the electron beam and sample interaction were collected by the EDS, and a spectrum of relative intensities of the elements was displayed. The EDS software was then used to calculate the elemental concentrations in weight percent from the spectra. The BSE images were used to characterize the structure and shape of the magnetic grains, while the EDS spectra and elemental concentrations were used to determine the minerals contained in the sample.

### 3. Results and discussion

The measurement results for DD REP B 693 peatland samples are focused on 2 measurement areas
3.1. Results of Measurement of DD REP B 693 (I)

BSE image of the surface morphology of grain 1 from DD REP B peatland sample 693 are show in Figure 3.

![Sample surface morphology DD REP B 693 (I)](image)

Figure 3. Sample surface morphology DD REP B 693 (I)

In Figure 3, it can be seen this mineral appears to have an oval-shaped surface and has fractured, it was evident that magnetic minerals in peatland were natural and not affected by anthropogenic influence [9][10]. The elemental concentrations were obtained in this mineral from several spots that have different colors, there are parts that are dark in color, and there is a brightly colored part, indicating a high iron content [8]. This grain is 480µm and is classified in the multidomain group because it has a size greater than 10µm [11]. The elements contained in each analytical spot of these grains can be seen from the measurement results of EDS as follows:

In area 1, the result of EDS Spectrum 145 (brightly colored parts) are show in Figure 4

![EDS result DD REP B 693 (I) spectrum 145](image)

Figure 4. EDS result DD REP B 693 (I) spectrum 145

Figure 4 above shows the EDS spectrum obtained from a bright part of the grain. There are several dominant elements, namely Fe, O, Ti, and Si and several other elements such as Al and Mg. The weight percentages of the different elements normalized to 100% total are shown in Table 1 below:
Table 1. Elements of DD REP B 693 (I) spectrum 145

| Elements          | % Weight |
|-------------------|----------|
| Oxygen (O)        | 26.58    |
| Natrium (Na)      | 0.03     |
| Magnesium (Mg)    | 0.1      |
| Aluminium (Al)    | 0.44     |
| Silika (Si)       | 2.55     |
| Kalium (K)        | 0.01     |
| Calcium (CA)      | 0.14     |
| Titanium (Ti)     | 8.01     |
| Chromium (Cr)     | 0.01     |
| Mangan (Mn)       | 0.04     |
| Besi (Fe)         | 61.87    |
| Nitrogen (Ni)     | 0.2      |

Table 1 shows the composition of the elements in the DD REP B 693 samples in the brightly colored section of the grain. This data indicates that this brightly colored part has the high Fe content compared to other elements. When viewed as a whole, the types of minerals that may be formed from these elements are Magnetite (Fe3O4), Hematite (Fe2O3), and Ilminite (FeTiO3), this mineral is almost the same as mineral soil around the Danau Diatas is included in the ferromagnetic and ferrimagnetic mineral groups which is dominated by mineral ilmenite [12].

In the second area the EDS Spectrum 158 (dark gray parts) are show in Figure 5

Figure 5 above shows the EDS results from parts of the grain that look dark gray. Here it is seen that in this spectrum there are several dominant elements namely Fe, O, Ti, and Si and several other minor elements such as Al and Mg. The weight percentages of the different elements normalized to 100% total are shown in Table 2 below:
Table 2. Elements of DD REP B 693 (I) spectrum 158

| Elements       | % Weight |
|----------------|----------|
| Oxygen (O)     | 31.21    |
| Natrium (Na)   | 0.51     |
| Magnesium (Mg) | 0.6      |
| Aluminium (Al) | 1.93     |
| Silika (Si)    | 9.92     |
| Fosfor (P)     | 0.06     |
| Sulfur (S)     | 0.1      |
| Kalium (K)     | 0.47     |
| Titanium (Ti)  | 3.9      |
| Mangan (Mn)    | 0.37     |
| Besi (Fe)      | 50.37    |
| Nitrogen (Ni)  | 0.57     |

Table 2 shows the composition of the dark gray part of the grain in the DD REP B 693 samples. This data indicates that this dark gray part has a lower Fe content compared to brightly colored minerals in Spectrum 145. However, the content of Oxygen and Silica in this dark gray part is higher. When viewed as a whole, the types of minerals that may be formed from these elements are Magnetite (Fe3O4), Hematite (Fe2O3), Ilminite (FeTiO3) and Quartz (SiO2).

3.2. Results of Measurement of DD REP B 693 (II)

BSE image displaying the surface morphology of grain 2 of DD REP B peatland sample 693 are show in Figure 6.

In Figure 6, we can be seen that this mineral shows a brightly colored surface and there are some parts that are slightly dark in color to black. The brightly colored part is indicated to have a high iron content. This grain is 250µm in size and belongs to the multidomain group. This mineral appears to have an uneven surface and looks still covered by non-magnetic minerals and is round in shape, and its
angular surface has fractures. To indicate the elements contained in each spectrum of these grains can be seen from the measurement results of EDS including:

In the first area the EDS Spectrum 261 (brightly colored parts) are show in Figure 7

Figure 7. EDS result DD REP B 693 (II) spectrum 261

Figure 7 above shows the EDS spectrum from a part that looks bright. Here it is seen that in this spectrum there are several dominant elements namely Fe, O, Ti, and Si and several other elements such as Al and Mg. The weight percentages of the different elements normalized to 100% total are shown in Table 3 below:

Table 3. Elements of DD REP B 693 (II) spectrum 261

| Elements          | % Weight |
|------------------|----------|
| Oxygen (O)       | 26.91    |
| Natrium (Na)     | 0.07     |
| Magnesium (Mg)   | 0.04     |
| Aluminium (Al)   | 0.35     |
| Silika (Si)      | 3.77     |
| Sulfur (S)       | 0.04     |
| Kalium (K)       | 0.18     |
| Calcium (CA)     | 0.02     |
| Titanium (Ti)    | 6.49     |
| Chromium (Cr)    | 0.1      |
| Mangan (Mn)      | 0.34     |
| Besi (Fe)        | 61.5     |
| Nitrogen (N)     | 0.17     |

Table 3 shows the composition of the elements in the DD REP B 693 samples in the brightly colored section of the grain. This data indicates that this brightly colored part has the high Fe content compared to other elements. When viewed as a whole, the types of minerals that may be formed from this element are Magnetite (Fe3O4), Hematite (Fe2O3), and Ilminite (FeTiO3).

In the second area the EDS Spectrum 273 (dark gray parts) are show in Figure 8.
Figure 8. EDS result DD REP B 693 (II) spectrum 273

Figure 8 above shows the appearance of EDS in parts that look dark gray. Here it is seen that in this spectrum there are several dominant elements, namely O, Fe, Ti, and Si and several other elements such as Al and K. The weight percentages of the different elements normalized to 100% total are shown in Table 4 below:

Table 4. Elements of DD REP B 693 (II) spectrum 273

| Elements     | % Weight |
|--------------|----------|
| Oxygen (O)   | 38.99    |
| Natrium (Na) | 0.29     |
| Magnesium (Mg)| 0.74    |
| Aluminium (Al)| 1.87    |
| Silika (Si)  | 21.67    |
| Sulfur (S)   | 0.06     |
| Kalium (K)   | 2.11     |
| Calcium (CA) | 0.8      |
| Titanium (Ti)| 3.74     |
| Chromium (Cr)| 1.13     |
| Besi (Fe)    | 28.21    |
| Nitrogen (Ni)| 0.37     |

Table 4 shows the composition of the dark gray part of the grain in the DD REP B 693 samples. This data indicates that this dark gray part has a much lower Fe content compared to brightly colored minerals in Spectrum 261. However, the content of Oxygen and Silica in this dark gray part is higher. When viewed as a whole, the types of minerals that may be formed from this element are Hematite (Fe2O3) and Quartz (SiO2).

Based on the morphological analysis of the DD REP B 693 samples above it is suspected that the magnetic minerals found on the peatland originated from the transport process and had eroded because the magnetic grains were more oval shaped and had many fractures [8]. This condition shows that this magnetic mineral has accumulated since quite a long time. Iron oxide magnetic minerals can move because of wind and water which will affect the shape of the magnetic mineral grains and as a guide to determine where the magnetic minerals come from. If the shape of the magnetic grain is smooth and has
a small grain size indicates that the mineral is in the peatland because it is carried away by the wind then settles. Whereas those with a large grain shape and large grain size can be said that the magnetic minerals are transported to peatland and stored for a long time [13].

The size of the grains in this sample of peatland belongs to the multidomain group, which is a large grain. The grains were carried to the peatland by erosion that occurred a long time ago because it is found at depths of more than 6 meters. The results of EDS analysis show that the magnetic minerals found in the peatland sample contain the following elements: Oxygen (O), Iron (Fe), Titanium (Ti), Silica (Si), Carbon (C), Nitrogen (N), and Bromine (Br). From these analyses, the magnetic grains are most likely Hematite (Fe2O3), Magnetite (Fe3O4), Ilmenite (FeTiO3) and Rutile (TiO2).

4. Conclusion
Based on the research that has been done, the results of observing samples of DD REP B 693 peatland provide information about the morphology and composition of elements that vary. From the EDS results, it can be concluded that peatlands contain four dominant elements, namely Fe, O, Ti and Si. While the types of minerals that can be formed are Magnetite (Fe3O4), Hematite (Fe2O3), and Ilmenite (FeTiO3) and Quartz (SiO2).

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References
[1] R. Rahayu, D. P. Ariyanto, K. Komariah, S. Hartati, J. Syamsiyah, and W. S. Dewi, “Dampak Erupsi Gunung Merapi Terhadap Lahan Dan Upaya-Upaya Pemulihannya,” Caraka Tani J. Sustain. Agric., vol. 29, no. 1, p. 61, 2014.
[2] C. B. De Maisonneuve et al., “Bathymetric survey of lakes Maninjau and Diatas (West Sumatra), and lake Kerinci (Jambi),” J. Phys. Conf. Ser., vol. 1185, no. 1, 2019.
[3] Sudaryo and Sutjipto, “Vulkanik Di Daerah Cangkringan Kabupaten Sleman,” no. November, pp. 715–722, 2009.
[4] A. Wibowo, “Peran Lahan Gambut Dalam Perubahan Iklim Global,” Tekno Hutan Tanam., vol. 2, no. 1, pp. 19–28, 2009.
[5] E. . Huliselan and S. Bijaksana, “Magnetic Properties as Proxy Indicators of Heavy Metals in Leachate: A Case Study from Jelekong Solid Waste Disposal Site, Bandung,” vol. 12, no. Figure 1, 2006.
[6] J. J. Rothwell and J. B. Lindsay, “Mapping contemporary magnetic mineral concentrations in peat soils using fine-resolution digital terrain data,” Catena, vol. 70, no. 3, pp. 465–474, 2007.
[7] E. Kristian and S. Bijaksana, “Identifikasi Mineral Magnetik pada Lindi (Leachate),” J. Geofis., vol. 2, no. 2004, pp. 8–13, 2007.
[8] T. A. Sari, Hamdi, and F. Mufit, “BAU-BAU KALIMANTAN TIMUR MENGGUNAKAN SCANNING ELECTRON MICROSCOPE (SEM),” vol. 1, no. April, pp. 97–104, 2014.
[9] E. K. Huliselan, S. Bijaksana, W. Srigutomo, and E. Kardena, “Scanning electron microscopy and magnetic characterization of iron oxides in solid waste landfill leachate,” J. Hazard. Mater., vol. 179, no. 1–3, pp. 701–708, 2010.
[10] R. Putra, H. Rifai, and C. M. Wurster, “Relationship between magnetic susceptibility and elemental composition of Guano from Solek Cave, West Sumatera,” J. Phys. Conf. Ser., vol. 1185, no. 1, 2019.
[11] B. R. F. Butler, “Paleomagnetism: magnetic domains to geologic terranes,” Choice Rev. Online, vol. 29, no. 10, pp. 29-5708-29–5708, 2013.

[12] R. N. Fajri, R. Putra, C. B. De Maisonneuve, A. Fauzi, Yohandri, and H. Rifai, “Analysis of magnetic properties rocks and soils around the Danau Diatas, West Sumatra,” J. Phys. Conf. Ser., vol. 1185, no. 1, 2019.

[13] H. Rifai, Erni, and M. Irvan, “Ekstraksi Magnetik pada Methanol-Soap Bathed Muds,” vol. 14, pp. 25–28, 2010.