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Determination structure and crystallite size of pumice magnetic minerals from Ngarai Sianok using X-Ray Diffraction (XRD)

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Abstract: The structure and size of crystal are are important aspects of magnetic minerals pumice from Ngarai Sianok to understand the geological processes of the area. Volcanic rocks in Ngarai Sianok contain magnetic minerals with unknown crystal structures. The crystal structure and size in pumice are analyzed using X-Ray Diffraction (XRD), where the type of magnetic mineral and crystal structure are determined by comparing the data measurement with the mineral database, while the crystal size can be determined using the Scherrer equation. Magnetic minerals in pumice from Ngarai Sianok are magnetite (Fe_3O_4) with a cubic mineral structure, ilmenite mineral ($FeTiO_3$) with a hexagonal structure, and hematite mineral (Fe_2O_3) with a rhombohedral structure. The crystal size of magnetic minerals vary. The lowest is at an angle of 18.25 with a FWHM value of 0.1279 which results in a crystal size of 62.8995 nm. The highest crystal size is at an angle of 35.46 with a FWHM value of 0.0624, resulting in a crystal size of 133.6609 nm. FWHM value is inversely proportional to the size of the crystal, meaning that the size of the crystal formed is getting smaller with increasing FWHM. The average size of pumice crystals formed in Ngarai Sianok was calculated to be 102.6392 nm, the small crystal size indicates the rapid cooling of magma, usually occurring during explosive eruptions.

Keywords: Crystal Structure, Crystallite Size, Magnetic Minerals, Pumice, X-Ray Diffraction

1. Introduction

Pumice is a type of igneous rock [1] originating from volcanic activity that goes through a natural cooling and deposition process underground for many years [2]. Compared to other rocks, pumice has an extremely low specific gravity; the composition and structure of the rock's creation influence pumice's density. Pumice does not sink in water because of its vesicular (hollow) structure and abundance of cells [3]. The elemental composition of pumice in general is

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such as aluminium (Al), potassium (K), silica (Si), calcium (ca), iron (Fe), sodium (Na), magnesium (Mg) [4]. Europium (Eu), yttrium (Y), neodymium (Nd) and cerium (Ce) [5]. Pumice contains the minerals tridymite, cristobalite, obsidian, feldspar, pumice also has an abundance of magnetic minerals such as, ilmenite ($FeTiO_3$), magnetite (FeO_{34}) [6], hematite (α-Fe_2O_3), maghemite (γ-Fe_2O_3) [7]. The abundance of minerals in pumice can be used as selling points such as the minerals tridymite, cristobalite, obsidian, and feldspar contained in pumice are used as raw materials for making glass, mortar [8]. Understanding the geological process of the Ngarai Sianok requires fieldwork to identify the magnetite minerals in the corresponding pumice region.

Magnetic minerals are natural materials that contain magnetic minerals. The most powerful magnetism is found in ferromagnetic materials, which are diamagnetic, paramagnetic, and occasionally diamagnetic minerals found in nature [9]. The categories iron hydroxides, iron sulfides, and iron titanium oxides are commonly used to categorize ferromagnetic materials. Goethite (FeOOH) is iron hydroxide; phyrhotite (Fe_7S_8) and greigite (Fe_3S_4) are iron sulfides; and magnetite (Fe_3O_4), hematite (Fe_2O_3), and maghemite (Fe_2O_3) are titanium oxide minerals [10]. Identification of magnetic minerals can be done using the X-Ray Diffraction (XRD) Method. Magnetic minerals are characterized by being composed of atoms and lattices that form a crystal structure; the crystal structure of these minerals largely determines their magnetic properties. Magnetite has a cubic structure that allows magnetic interactions between iron ions, while hematite has a distinct hexagonal structure, resulting in weaker magnetic properties [11], the method used to determine the structure and size of magnetic mineral crystals is X-Ray diffraction (XRD).

X-Ray Diffraction (XRD) is used to determine the size and structure of the crystals found in pumice. X-rays can determine the composition of unknown minerals by using the X-ray diffraction method [12]. The crystal structure can be determined by comparing the data obtained from crystallography to a mineral database, whereas the crystal size can be determined using a scherrer. The method of crystal size also has drawbacks, smaller crystal can reveal the superparamagnetic effect, in which magnetism appears only in external medan magnets and vanishes when medan is exhausted [13]. Numerous materials, such as minerals, polymers, plastics, metals, semiconductors, and ceramics, can be examined using this technique [14]. The tool used in X-Ray Diffractometer analysis is called an X-Ray Diffractometer. Its working principle is based on a series of atoms, which, when heated to a certain temperature, will produce X-rays in a vacuum. Subsequently, the atoms will be broken down into individual particles called cathodes, which will produce electrons. Finally, the electrons will pass through the anodes. Electrons with extremely fast response times are directed towards airborne anodes [15].

Previous studies utilizing X-Ray Diffraction include the magnetic mineral characterization of the iron sand of Pasia Nan Tigo Beach Padang using X-ray Diffraction (XRD) [16] and the characterization of magnetic mineral types of cave sediments in Liang Cave, Manggarai, and Nusa Tenggara Timur using the X-Ray Diffraction method [12]. The structure and crystal size of magnetic minerals have not yet been established, yet, by the pumice from Ngarai Sianok. As a result, research utilizing X-Ray Diffraction is required to ascertain the structure and crystal size of magneric minerals in pumice from Ngarai Sianok.

2. Materials and Method

This research is descriptive research; to determine the structure and crystallite size of magnetic minerals using an X-Ray Diffractometer. Pumice collected in 2023 by the magnetist team serves as the sample. The sampling is in West Sumatra's Ngarai Sianok (Figure1).

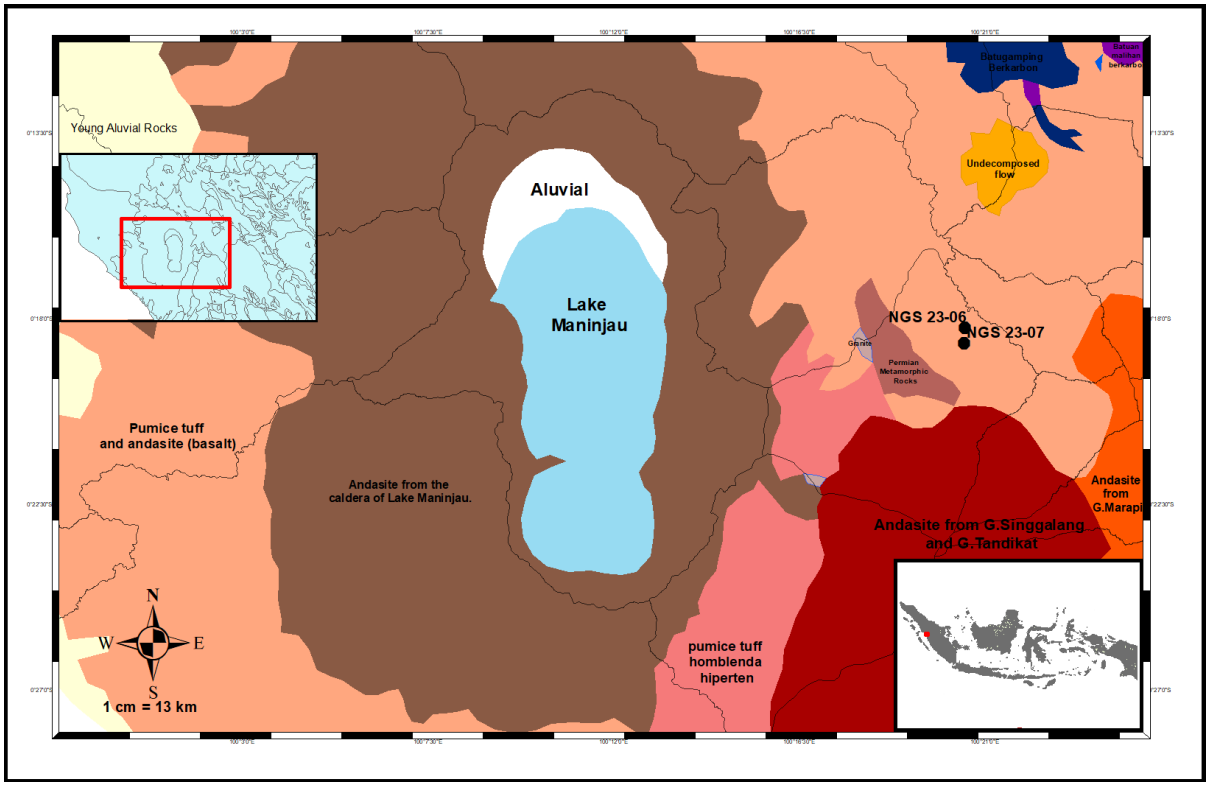

Figure 1. Sampling Location Map

Sampling locations map the distribution and types of rocks exposed in the area around Lake Maninjau, where the area is dominated by igneous rocks formed due to magma fluids that undergo a freezing process; this liquid comes out of the bowels of the earth or magma that is still in the earth [17]. Along with volcanic rocks, the region surrounding Lake Maninjau also contains sedimentary rocks, such as alluvial rocks, that were created by erosion and river deposits. Ngarai Sianok is home to a variety of igneous rocks surrounding Lake Maninjau, including andesite, tuff, malihan rock, limestone, granite, and pumice. Ngarai Sianok is located at coordinates S 00018.601' E 100020.506' and S 00018.220 E 100020.529', a global position system was used to establish the sampling area's coordinates (GPS). The sample site is approximately two hours and seventeen minutes away from Padang city. The geography of Sianok Gorge is varied, featuring a steep valley (ravine) that borders Bukittinggi City, IV Koto District, Agam Regency, West Sumatra. Ngarai Sianok presents intriguing research opportunities due to its distinct geographical and geological features. Weather and climate, which can have an impact on geological conditions, can play a significant role in this investigation. Samples were taken in 2 areas in Ngarai Sianok with sample names NGS 23-06 and NGS 23-07. Taken atop a cliff ± 18 meters high with 3 meters of vegetation at the top, sample NGS 23-06 was collected. NGS 23- 07 was taken on a cliff that was 6 meters high and had 1 meter of vegetation at its peak. Following sample collection, the material was prepared by washing, drying, and then mashing using a mortar before being placed within the holder. The Material Physics Laboratory, Department of Physics, Faculty

of Mathematics and Natural Sciences, Padang State University, was the site of this sample preparation. The device that uses X-rays to view the crystal size, structure, and kind of mineral An X-Ray Diffractometer, the working principle is to direct X-rays at the sample, which will then be reflected or refracted by the atoms in the crystal (Figure 2).

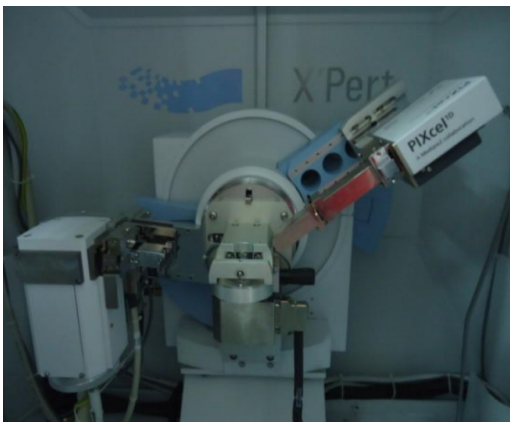

Figure 2. X-Ray Diffractometer

A graph displaying the type of mineral, its crystal structure, and its size is produced using the X-Ray Diffractometer tool. A crystal is a solid that has a natural surface plane around it. Its constituent atoms are arranged regularly and repetitively in a structure known as a polyhedron [18]. There are various varieties of mineral crystal formations, which include: An isometric system is one in which all three dimensions of the crystal are the same size. Cubic crystal systems are another name for isometric crystal systems [19]. Three mutually perpendicular axes, two of which are equal in length, make up a rhombohedral crystal system. Four axes make up the trigonal crystal system, three of which have equal lengths. A crystal system with four axes is called hexagonal; one with three mutually perpendicular axes is called orthorhombic. A triclinic crystal structure is made up of three axes that create an oblique angle and have varying axis lengths. A monoclinic crystal system is one in which the three axes are perpendicular to the two axes and the two axes create an oblique angle [20].

Magnetic minerals are among the several minerals that can be observed through the use of the X-Ray Diffraction method. Magnetic minerals are found in natural materials and are referred to as such. Ferromagnetic materials, which are present in a variety of paramagnetic and diamagnetic minerals found in nature, have the strongest magnetism [9]. Magnetic minerals come in several varieties, such as iron oxide, iron sulfide, and iron hydroxide. X-rays that fall in the wavelength range of 0.5 to 2.5 nm comprise the bulk of the content of XRD [21]. Bragg's law equation can be used to determine the mineral structure.

$$n\lambda = 2\, d \sin\theta \quad (1)$$

Where n is a multiple, λ is the wavelength, and d is the distance between fields [22]. he value of the widening of the diffraction half peak curve (FWHM) after choosing the subsequent peak can be obtained by applying the Scherrer equation (2) to calculate the crystal's size.

$$L = \frac{K\lambda}{\beta . cos\theta} \quad (2)$$

The Full Width at Halff Maximum (FWHM) is denoted by β, the Scherrer constant (k = 0.9), and the x-ray wavelength used is cathode co (λ = 1,790 nm). The most intense diffraction peak with a divergence angle 2θ larger than 300 was the one we chose. By averaging the size values of each crystal plane, the projected crystal size value is determined [23].

3. Results and Discussion

X-Ray Diffractometer was used in conjunction with X-Ray Diffraction analysis yields output from the device in the form of a graph of diffraction peaks based on the measurement results. To determine the relative intensity, the peak at the diffraction angle was compared with a significant intensity. The atoms or ions that are present and distributed in the material's unit cell determine whether a number of peaks produced have high or low relative intensities. A mineral database was then checked with the study's findings [24]. Figure 3 displays the peaks that were produced during the measurement of sample NGS 23-06.

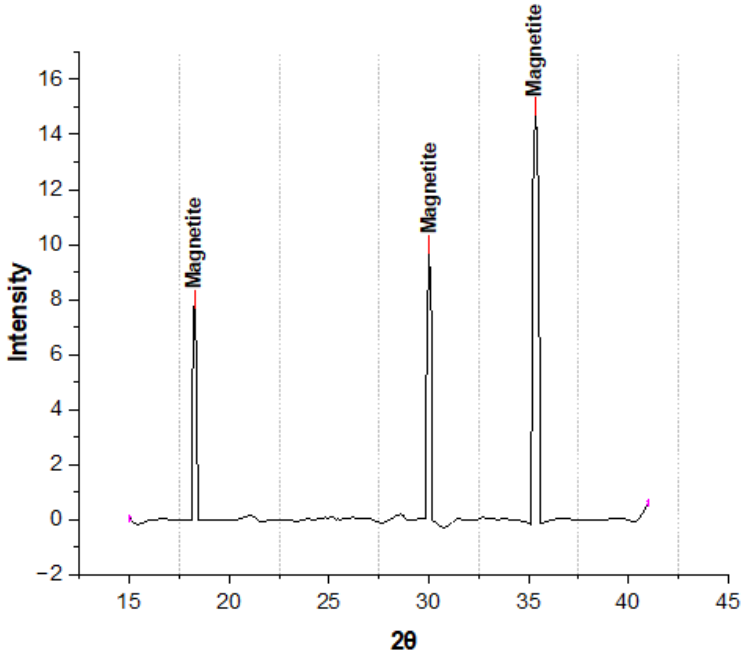

Figure. 3. Sample measurements (NGS 23-06)

The type of magnetic mineral obtained from the Ngarai Sianok pumice (Fig 3) is the mineral magnetite, this mineral belongs to the iron oxide mineral group with the mineral structure obtained is cubic (cube), the same results are also in pumice from Mount Ontake, Japan which has a magnetite structure [25], pumice samples from Mount Etna, Italy also have a magnetite crystal structure [11]. The measurement results of NGS 23-06 found can be listed in Table 1.

Table 1. Comparison of measurement data with NGS 23-06 mineral database

Data measurement		Database Minerals		Type Minerals	Structure Crystal
2θ(⁰)	I_r (%)	2θ(⁰)	I_r (%)		
18,25	8	18,25	10,3	Magnetite	Cubic
30,02	10	30,01	29,2	Magnetite	Cubic
35,46	15	35,46	100	Magnetite	Cubic

The next sample with the name of sample NGS 23-07 and the peaks resulting from the measurement of sample NGS 23-07 can be seen in Figure 4.

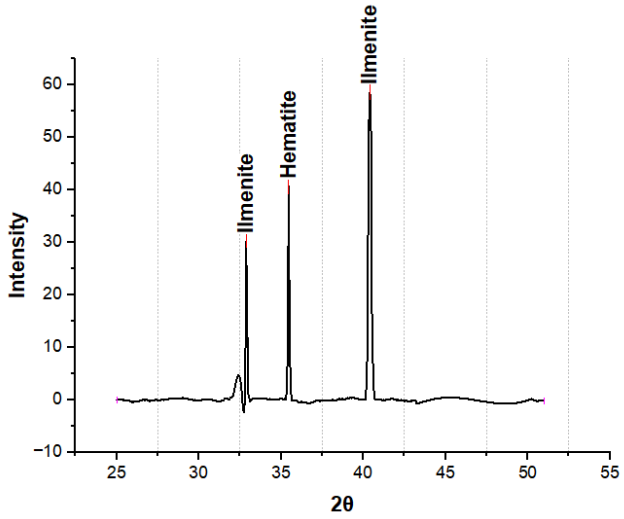

Figure 4. Sample measurements (NGS 23-07)

The magnetic mineral type of pumice NGS 23-07 (Figure 4) is ilmenite mineral with the mineral structure obtained hexagonal and hematite mineral with the mineral structure obtained is rhombohedral, ilmenite and hematite minerals belong to the iron oxide mineral group, The same results can be seen in pumice from Mount Ontake, Guatemala which has an ilmenite structure [26], pumice samples from Mount Fuji, Japan with a hematite crystal structure [27], and pumice samples from Mount St. Helens, USA with a hematite crystal structure. Helens, USA with hematite crystal structure [28], pumice samples from Mount Vesuvius, Italy with hematite crystal structure [29]. The measurement results of NGS 23-07 can be written in Table 2.

Table 2. Measurement data comparison with NGS 23-07 mineral database

Data measurement		Database Minerals		Type Minerals	Structure Crystal
2θ(⁰)	I_r (%)	2θ(⁰)	I_r (%)		
32,88	30,29	32,90	100	Ilmenite	Heksagonal
35,46	40,61	35,46	100	Hematite	Rhombohedral
40,41	58,56	40,41	50	Ilmenite	Heksagonal

From the results of the calculation of the XRD data of the Ngarai Sianok pumice, determine the crystal size of the pumice using the Scherrer equation in equation (2). obtained in the following Table 3.

Table 3. Crystal size on Ngarai Sianok pumice stone

2θ	FWHM	Crystal size
18,25	0,1279	62,8995
35,46	0,0936	89,1072
40,41	0,0936	90,4403
32,88	0,0767	107,9180
30,02	0,0624	131,8095
35,46	0,0624	133,6609
Average crystal size		102,6392

The measurement results of the Ngarai sianok sample showed that the lowest value of the crystal size was at an angle of 18.25 and an FWHM value of 0.1279 so that a crystal size of 62.8995 nm was obtained, and the highest value of the crystal size was at an angle of 35.65 and an FWHM value of 0.0624 so that a crystal size of 133.6609 nanometer was obtained. The calculation results are similar to pumice from Mount Tambora, Indonesia, with crystal sizes varying from 9.000 nm with FWHM 0.16° to 12.000 nm with FWHM 0.15° [30]. The obtained data indicate that there is an inverse relationship between the size of the crystal and the FWHM value. The FWHM value increases with decreasing crystal size and vice versa. Based on calculations, the average crystal size of pumice from Ngarai Sianok is 102.6392 nanometer. The small crystal size indicates the rapid cooling of magma, usually occurring during explosive eruptions. Rapidly cooling magma does not allow enough time for minerals to form large crystals. This often occurs in areas with intense volcanic activity, indicating a dynamic and rapidly changing geological environment.

4. Conclusion

The pumice from Ngarai Sianok is composed of three types of minerals: mineral magnetite with a crystal structure that is derived from a crystal structure that is derived from a crystal structure that is hexagonal; ilmenite minerals with crystal structures obtained, namely hexagonal; and hematite minerals with Rhombohendra crystal structures, with an average crystal size obtained of 102.6392 nanometer, the small crystal size indicates the rapid cooling of magma, usually occurring during explosive eruptions.

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