ESTIMATION OF MINERALOGICAL, MORPHOLOGICAL, AND MAGNETIC PROPERTIES OF THE MINERAL SEDIMENTS OF GUANO CAVES IN THE KARST AREA

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ABSTRACT

Guano sediments can record environmental changes in caves and be investigated through the magnetic mineral properties contained therein. The properties of magnetic guano depend on the type, concentration, shape and size of the magnetic mineral grains. These properties can provide clues to the cave-forming environment, both lithogenic and anthropogenic influences. Therefore, this study aims to analyse the magnetic properties, mineralogy, and magnetic mineral morphology of guano sediments in caves in the karst region of Maros, Indonesia, especially Bat caves and Dream caves. Guano samples were taken according to the condition of the cave floor. Magnetic susceptibility measurements are used to analyse the magnetic properties of guano sediments. X - Ray Diffraction (XRD) and Scanning Electron Microscopy - Energy Dispersive Spectroscopy (SEM - EDS) measurements were used to analyze the mineralogy and morphology of the magnetic mineral guano sediments, respectively. The results show that the magnetic susceptibility value at low frequencies ranges from (0.072 - 1.476) x 10^{-6} m^3 kg^{-1} for Bat cave guano. The χ_{LF} value of Dream cave guano ranged from (2.867 - 85.507) x 10^{-6} m^3 kg^{-1}. The χ_{FD} (%) of Bat cave guano samples (2.78 - 8.70) %, while χ_{FD} (%) of Dream cave guano samples (0 - 4.18) %. The analysis of magnetic susceptibility, mineralogy, and magnetic mineral morphology showed that the guano samples from both caves tended to be affected by the ferrimagnetic mineral of the iron-titanium oxide group, namely magnetite (Fe_3O_4). The grain shapes of the guano samples from the two caves are similar; they are oval and irregular, and there are also fractures and heterogeneous grain distribution. Thus, the magnetic mineral sources in Bat cave and Dream cave guano tend to come from magnetic minerals controlled by lithogenic and anthropogenic components.

Keywords: guano, magnetic susceptibility, mineralogy, morphology, magnetic mineral.

INTRODUCTION

The cave environment is closely related to the natural conditions around the cave. As a result of the precipitation process of cave - forming rocks, namely carbonate rocks ($CaCO_3$), cave ornaments such as stalactites and stalagmites are formed [1]. In addition, $CaCO_3$ precipitation is associated with cave sediments such as guano deposited on the cave floor. Guano sediments settle for thousands of years to record environmental changes in the cave [2]. Environmental changes can be investigated through magnetic minerals contained in guano sediments. The magnetic minerals contained in guano sediments can come from materials transported by water, wind, cave animals, and even human activities in the cave. The transported magnetic minerals are associated with guano sediments in the cave [3].

Rock magnetism is a method that is magnetically sensitive to minerals whose behaviour is controlled by changes in their environment [4]. The behaviour of magnetic minerals, both in mineralogy, concentration and morphology, can respond to changes in the environment that occur. Therefore, magnetic mineral parameters can be used as a proxy indicator to determine the process of changing the cave environment. Besides that, other advantages of the magnetic method are; 1) measurement can be performed on all materials; 2) measurement is safe, simple, fast and non - destructive; 3) measurement can be performed in the laboratory as well as in situ; 4) measurement can complement many other types of environmental analysis, 5) offers a wide choice cost - effective in measurement and analysis [4]. This method has been used to study the chronology of environmental changes [5].

One of the rock magnetism parameters most commonly used is the magnetic susceptibility parameter (χ_{LF}), which is the ratio or ratio between the magnetization obtained by a sample with a weak magnetic field given to see the magnetic moment response in sediments [4, 6]. The magnetic susceptibility parameter is a good proxy indicator to determine the concentration and type of magnetic minerals, grain size and status of the magnetic domains, and their shape, which is influenced by the forming environment [4]. However, this method has not been widely used to identify and assess environmental changes in karst caves.

Several studies have been conducted on the characterization of magnetic minerals in cave guano. Studies on the susceptibility of magnetic and heavy metals to guano in the Mampu and Bubau caves in South Sulawesi have been reported by analysing magnetic grains and magnetic mineral sources [3]. Element such as Fe and Ni which are part of ferromagnetic elements, paramagnetic (K, Mg, Mn, Ti, Al, and Ca) and diamagnetic (Pb, Cu, and Zn). Ca is a high concentration element with percentage 54.822 % in Mampu cave and 33.294 % in Bubau cave. While Pb is a low concentration element found in the Mampu cave with percentage 0.004 % and 0.002 % Bubau cave. Concentration S is not present in the Mampu cave than Bubau cave with percentage 3.372 %. Concentration of Fe in the Mampu cave is greater (7.45 %) than Bubau cave (3.379 %). A mixture of superparamagnetic (SP) and stable single domains (SSD) magnetic grains dominates the size of the magnetic grains in the two caves.

Also, the magnetic minerals contained in cave guano are thought to be of lithogenic and anthropogenic origin. In addition, a pollution study identifies a correlation between magnetic parameters and the heavy metal (with density more 5 g m^{-3}) content of Solek cave in West Sumatra [7]. Magnetic susceptibility variations are not only controlled by the concentration of heavy metal Fe and magnetic grain size but also paramagnetic element such as Ti, Mg, Al, K and Ca. The magnetic grain size is dominated by multi-domain magnetic grains. The correlation between magnetic susceptibility and heavy metal content is weak, so the presence of authigenic minerals is suspected.

Studies on the relationship between magnetic susceptibility and heavy metals have also been studied in Bat cave Guano, South Sulawesi [8]. This cave is in the karst area of Maros. Heavy metals (Fe, Cu, Zn, Zr, Nb) affect guano's magnetic mineral content. The heavy metal in guano is indicated as material carrying magnetic properties in the cave. Fe dominated the heavy metal content of the guano sample. The concentration of Fe in all samples showed low concentrations, thus causing low magnetic susceptibility. The concentration of Fe becomes the controller of the magnitude of the magnetic susceptibility in a sample. Meanwhile, Fe controls the size of the magnetic susceptibility value. Thus, magnetic susceptibility is a proxy indicator of heavy metals [9 - 11]. The high abundance of magnetic minerals is thought to originate from anthropogenic material, and sediments that contain much anthropogenic material have a high heavy metal content associated with a high abundance of magnetic minerals [9, 12].

Research conducted in the bat cave in the Maros Karst area focused on the correlation of magnetic parameters and heavy metal content in guano. Therefore, this study aims to analyse the magnetic properties, mineralogy, and morphology of magnetic minerals in guano sediments in caves in the karst area of Maros, Indonesia. The study compared the guano in the Bat cave with the Dream cave. Magnetic mineral characterization is widely used for studies of environmental changes such as rivers [13 - 17], lakes [18 - 20], TPA [21, 22], as well as urban areas [23 - 26]. Magnetic properties are a proxy indicator of environmental changes [27] and a key parameter to indicate the conditions of magnetic phase formation and processes such as diagenesis and weathering [28].

Magnetic susceptibility parameters are supported by analysis of mineralogy and morphology of magnetic minerals. The magnetic properties of guano can be determined based on its magnetic susceptibility value. At the same time, the mineralogy and morphology are analysed based on the shape of the magnetic grains and the types of magnetic minerals found in the guano. By carrying out the magnetic mineral characterization of guano, it is hoped that it can be used to monitor the environmental conditions of caves in the Maros Karst area.

EXPERIMENTAL

Guano samples were taken from two caves in the Maros Karst Area, namely Bat cave and Dream cave. These two caves are in separate karst cluster areas. The first location, Bat cave, is in the Rammang - Rammang Karst Area in Salenrang Village, Bontoa District, Maros Regency, South Sulawesi. This karst area is 42.30 km north of Makassar City. Bat cave has a wide mouth of ± 10 m, a width of ± 25 m, a height of ± 50 m, and a length of ± 30 m that can be reached. The Rammang - Rammang Karst area is not included in the Bantimurung Bulusaraung National Park zone but is in the economic development zone. As a result, this karst area has overlapping functions, namely as an economic developer and a conservation area.

Meanwhile, Dream cave's second location is in Bantimurung Bulusaraung National Park. Cave of Dreams is in Bantimurung Hamlet, Jenetaesa Village, Simbang District, Maros Regency, South Sulawesi. This cave is a type of horizontal cave with a length of ± 1,415 m and penetrates a karst hill. This cave has a depth of about ± 48 m. This cave is included in the Maros - Pangkep Karst Area and one of 34 caves in the Bantimurung Nature Reserve area of the Bantimurung - Bulusaraung National Park, right above the Bantimurung Waterfall. The path leading to the Dream cave is quite steep and is made of cement concrete with a length of 900 m.

Guano samples were taken on the surface of the cave floor and randomly adjusted to the conditions of each cave floor, both Bat cave and Dream cave. Coordinate/position data for each sampling point was measured using GPS. 30 guano samples from Bat cave and Dream cave were prepared to measure magnetic susceptibility. The dried samples were then crushed and sieved using a 100 - mesh sieve. Then, the sample is inserted into the cylinder holder to measure magnetic susceptibility. This measurement uses the Bartington Susceptibility Meter with the MS2B sensor, operating at a low frequency of 470 Hz and a high frequency of 4700 Hz. The measurement results obtained high - frequency magnetic susceptibility and low - frequency magnetic susceptibility [29, 30]. The percentage difference between low - frequency and high - frequency magnetic susceptibility is called frequency-dependent magnetic susceptibility [29, 30]. Magnetic susceptibility depends on the frequency of interpreting the superparamagnetic mineral content in the sample [17, 31].

Furthermore, Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS) measure the selected sample with the highest magnetic susceptibility value. For this purpose, two samples of Bat cave guano and two of Dream cave guano were extracted using a bar magnet. The SEM - EDS test uses the JCM - 600 PLUS type SEM tool. The SEM tool uses the JED - 230 Analysis Station Plus for EDS analysis. The JED - 2300 Analysis Station Plus is equipped with JEOL's DrySD™ (Dry Silicon Drift Detector), a high - speed analyser, and analytical software specially designed for JEOL electron microscopes. SEM - EDS measurements to identify magnetic mineral morphology and elemental composition in guano.

To complete the SEM - EDS testing, X - Ray Diffraction (XRD) testing was also carried out. Two samples of Bat cave guano were measured using a Rigaku MiniFlex II type XRD apparatus. The measurement results were analysed using the PDXL2 software [32, 33]. Meanwhile, two samples of Dream cave guano were measured using a Shimadzu XRD - 7000 Maxima type XRD tool. The measurement results were analysed by Match 3 software. Mineralogical analysis of the sediment samples was identified through XRD measurements.

RESULTS AND DISCUSSION

Magnetic properties

The results of magnetic susceptibility measurements (Table 1) show that the range of χ_{LF} values of Bat cave guano samples is (0.072 - 1.476) x 10^{-6} m^3 kg^{-1}, with an average of 0.7392 x 10^{-6} m^3 kg^{-1}. The range of χ_{LF} values for Dream cave guano samples is (2.867 - 85.507) x 10^{-6} m^3 kg^{-1}, with an average of 16.417 x 10^{-6} m^3 kg^{-1}. Meanwhile, the range of χ_{HF} values for Bat cave guano samples was (0.07 - 1.355) x 10^{-6} m^3 kg^{-1}, with an average of 0.6942 x 10^{-6} m^3 kg^{-1}. The range of χ_{HF} values for Dream cave guano is (2.815 - 85.041) x 10^{-6} m^3 kg^{-1}, with an average of 16.176 x 10^{-6} m^3 kg^{-1}. From the two χ_{LF} and χ_{HF} measurements, the range of χ_{FD} values (%) for Bat cave guano samples was 2.78 - 8.70 %, with an average of 5.64 %. The range of χ_{FD} values (%) for Dream cave guano samples is 0 - 4.18 %, with an average of 1.9 %.

The magnetic mineral susceptibility (χ_{LF}) values of guano in both Bat cave and Dream cave were higher than 10 x 10^{-8} m^3 kg^{-1}. It shows that all guano samples contain magnetic minerals, which are dominated by ferrimagnetic mineral groups because, in general, the χ_{LF} value is higher than 10 x 10^{-8} m^3 kg^{-1}, which indicates that the samples are controlled by ferrimagnetic minerals. It is in line with the literature review, which states that cave guano found the presence of ferrimagnetic minerals [7, 34].

The χ_{LF} value of the Dream cave guano sample was more significant than the χ_{LF} of the Bat cave guano sample. It is influenced by the character of Dream cave, which includes horizontal caves and penetrates karst hills, while Bat cave does not penetrate karst hills. As a result, the wind that blows into the cave and carries magnetic minerals will eventually associate with the soil in the Dream cave with a greater chance than in the Bat cave. In addition, the Dream cave is more frequent with human activity than the Bat cave. Thus, it indicates that the ferrimagnetic mineral content in the Dream cave is greater than that of the Bat cave.

Research on χ_{LF} in guano samples was also found in the χ_{LF} of Mampu cave which was more significant than the χ_{LF} of Bubau cave due to increased Fe concentration resulting in increased χ_{LF} [35]. In addition, the same thing was shown through a study of χ_{LF} guano in Solek cave, West Sumatra, where χ_{LF} was controlled by the concentration of iron oxide and magnetic grain size [7]. The χ_{FD} (%) value of all Bat cave guano samples and some Dream cave guano samples was 2.0-10.0 %, included in the medium χ_{FD} (%) category. This sample contains a mixture of fine and coarse superparamagnetic grains with grains < 0.005 μm in size. Meanwhile, some of the Dream cave guano samples have a χ_{FD} (%) value of 0 - 2.0 %, included in the low χ_{FD} (%) category. This sample has non - superparamagnetic grains with a grain size of > 0.03 μm.

Superparamagnetic grains are minerals with excellent grain sizes (smaller than ~ 0.03 μm) and have magnetic behaviour that shows changes over time, as in Bat cave and Dream cave guano samples. Naturally occurring (lithogenic) magnetic grains are mostly superparamagnetic grains. In sediments that are polluted by (anthropogenic) pollutants, low χ_{FD} (%) values (1 - 4 %) are often found, while sediments that undergo natural or lithogenic processes have higher χ_{FD} (%) values (> 10 %) [36].

Table 1. Descriptive statistics of the magnetic susceptibility parameters of guano samples from Bat cave and Dream cave (n = 30).

Cave	Parameter	Descriptive of statistics			
		Range	Mean	Median	SD
Bat Cave	χ_{LF} (x 10^{-6} m^3 kg^{-1})[a]	0.072-1.476	0.7392	0.7015	0.2671
	χ_{HF} (x 10^{-6} m^3 kg^{-1})[a]	0.07-1.355	0.6942	0.6660	0.2413
	χ_{FD} (%)[a]	2.78-8.70	5.64	5.55	1.48
Dream Cave	χ_{LF} (x 10^{-6} m^3 kg^{-1})	2.867-85.507	16.417	11.63	16.634
	χ_{HF} (x 10^{-6} m^3 kg^{-1})	2.815-85.041	16.176	11.4015	16.571
	χ_{FD} (%)	0-4.18	1.9	1.76	1.02

[a]This data has been published [8].

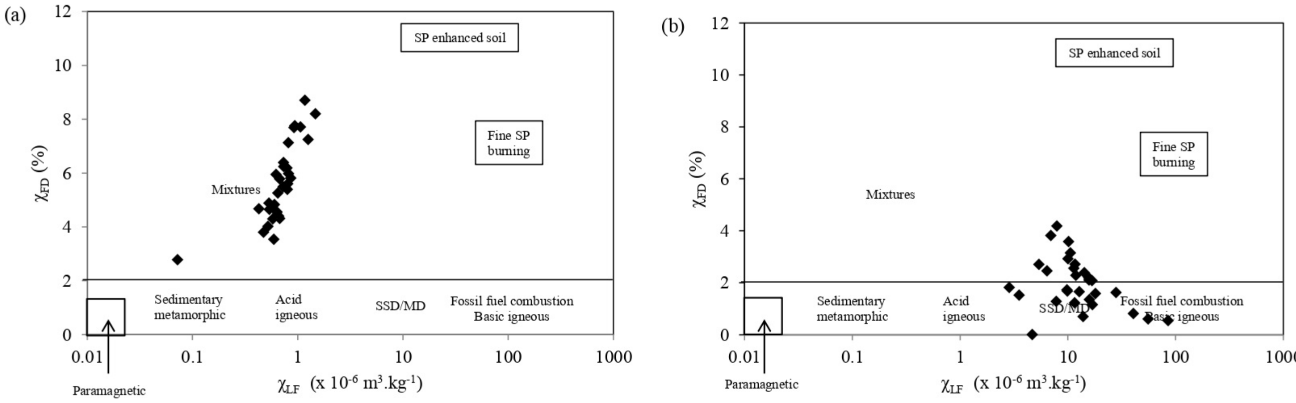

Fig. 1 $\chi_{LF} - \chi_{FD}$ (%) diagrams of guano samples (a) Bat cave and (b) Dream cave.

The χ_{LF} - χ_{FD} (%) relationship can help distinguish between grain size and domain status and provide information regarding the classification of magnetic properties and their sources [37]. Fig. 1a, based on the χ_{LF} - χ_{FD} (%) diagram, shows that the Bat cave guano samples are thought to have come from the grains of the bedrock forming the cave, namely carbonate rocks [33, 34]. The same applies to the Dream cave guano sample (Fig. 1b).

However, some of the Dream cave guano samples have undergone changes. It is because the Dream cave is a cave that is more frequently visited by tourists, which allows the transportation of minerals from the outside environment into the cave so that samples can record pollutant-carrying magnetic minerals. Magnetic mineral transportation can be through the media of wind, water, or human activity. Combustion magnetic minerals will have a grain shape different from the natural magnetic mineral grain shape. The shape of magnetic minerals (morphology) will be known by SEM measurements [14, 38].

Mineralogy

X - Ray Diffraction measurement to identify the type of magnetic minerals in Bat cave and Dream cave guano samples. Fig. 2 shows the XRD diffractogram of the T29 guano Bat cave sample, while Fig. 3 shows the XRD diffractogram of the I-5 guano sample of Dream cave. The peaks on the diffractogram indicate the minerals in the guano sample. The results showed that the Bat cave T29 guano sample contained the minerals calcium indium (Ca_8In_3), calcium aluminium antimonide ($Ca_{14}AlSb_{11}$), and magnetite (Fe_3O_4). Meanwhile, the

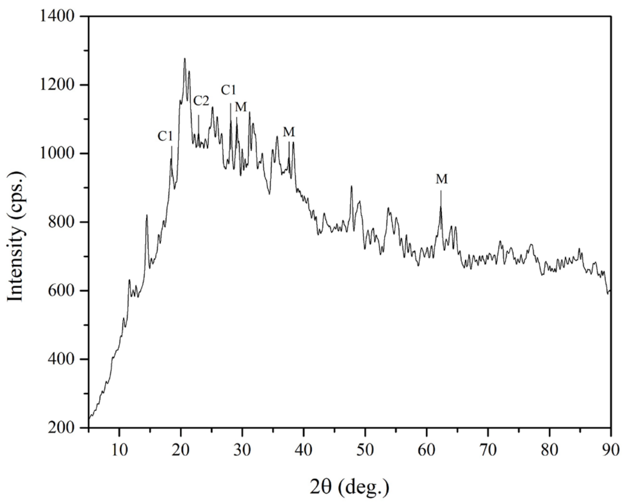

Fig. 2 XRD diffractogram of guano T29 Bat cave sample where C1 = calcium indium (Ca_8In_3), M = magnetite (Fe_3O_4), C2 = calcium aluminium antimonide ($Ca_{14}AlSb_{11}$) [8].

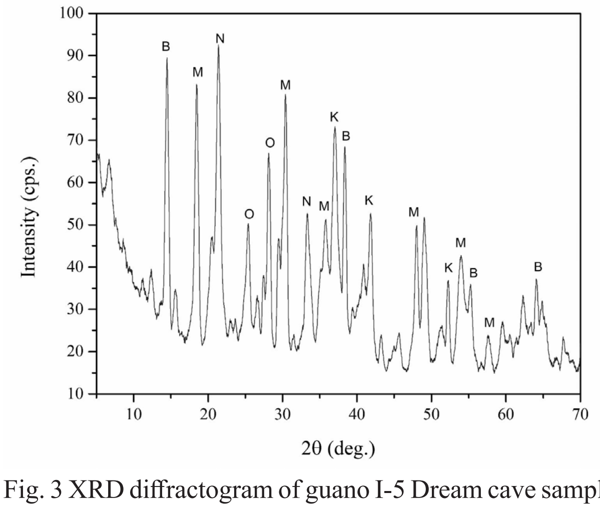

Fig. 3 XRD diffractogram of guano I-5 Dream cave sample where B=Boehmite ($AlHO_2$), M = magnetite (Fe_3O_4), N = natrolite ($Al_2Na_2O_{10}Si_3$), O = orthoclase ($AlKO_8Si_3$), K = kaolinite ($Al_2H_4O_9Si_2$).

mineral content in the I-5 guano sample is boehmite ($AlHO_2$), magnetite (Fe_3O_4), natrolite ($Al_2Na_2O_{10}Si_3$), orthoclase ($AlKO_8Si_3$), kaolinite ($Al_2H_4O_9Si_2$).

Based on the XRD analysis of the guano samples, it was found that there was the presence of ferrimagnetic minerals, especially the iron-titanium oxide group, namely magnetite (Fe_3O_4). Even though it contains a small amount, the properties of the magnetite mineral have magnetizing properties that are 1000 times more magnetic than paramagnetic minerals, especially diamagnetic minerals [8]. The mineral content in the Bat cave guano sample was dominated by minerals thought to have come from the environment outside the cave [33]. During the rainy season, water enters the cave through the media and flows through the pores of the cave until it flows down the cave's walls, then reaches the cave's floor. It shows that Bat cave is a natural cave [34].

Morphology of magnetic mineral

SEM-EDS measurements in this study can provide information on magnetic mineral morphology, grain size, and elemental composition obtained from scanning electron microscopy analysis of selected samples. Fig. 4 shows the SEM image of Bat cave guano samples using

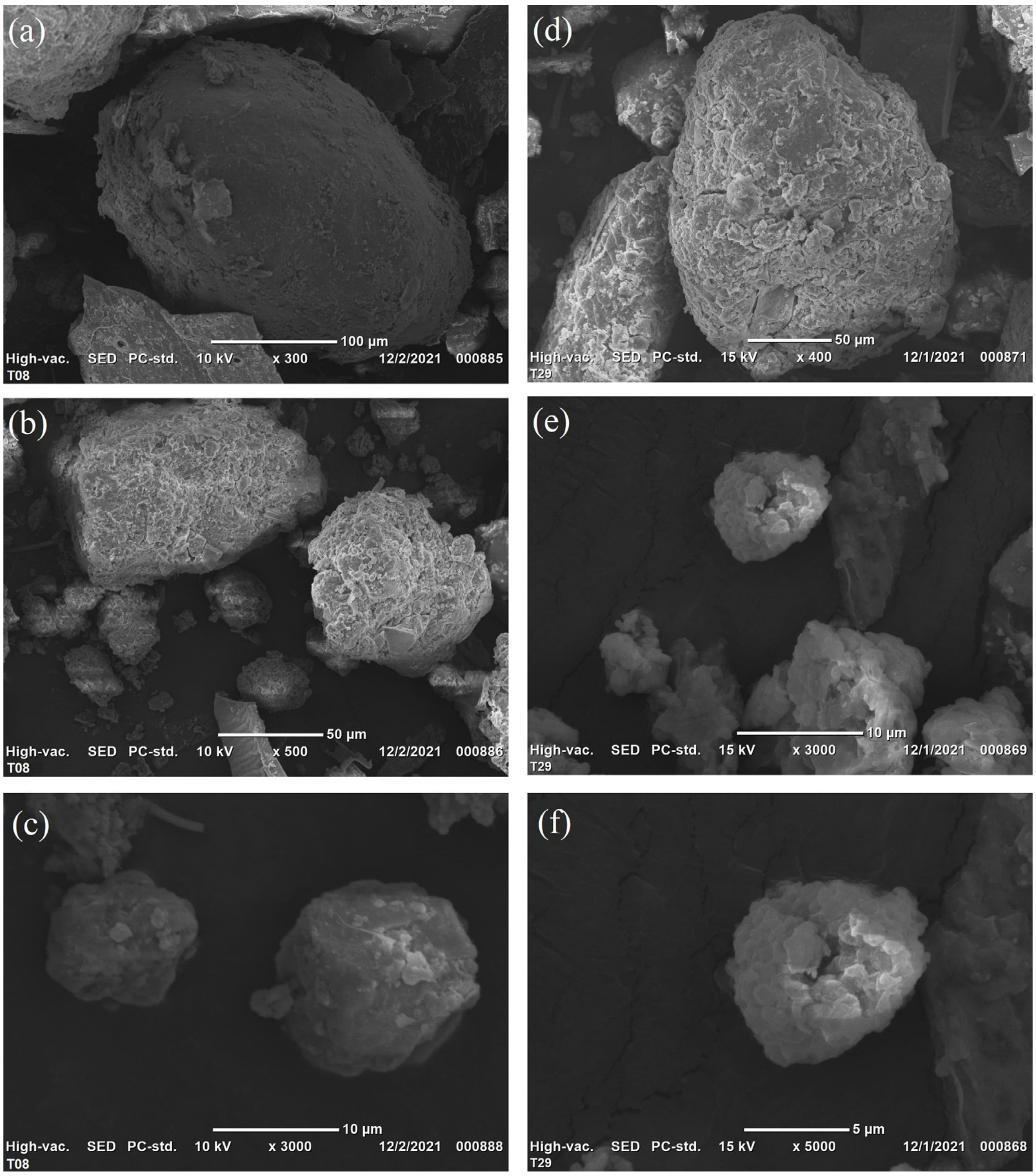

Fig. 4 SEM images of Bat cave guano samples (a-c) T08 and (d-f) T29.

SEM-EDS. Sample T08 is a sample in zone one or the front area of the cave. Meanwhile, the T29 sample is in zone two or an area inside the cave. Fig. 5 shows the SEM image of the Dream cave guano sample. Samples I-5 are sample at the mouth of the cave. This SEM-EDS measurement is also used to see the distribution of the dominant elements in the sample and the size of the magnetic grains, which can be used to determine the source of magnetic minerals [38, 39].

The SEM image results show that the magnetic mineral grains in the Bat cave and Dream cave guano samples are oval and have fractures on the surface. However, there are also irregular shapes and rough topography. Magnetic grain size distribution in guano samples varies > 10 µm. It indicates that the magnetic grain originates from the displacement that occurs through wind and water flow media. The magnetic grains are refined and have a small grain size in guano, indicating that the magnetic minerals are distributed into the cave through the wind medium. Coarse grains and large enough grain sizes can be said that magnetic minerals originate from the external environment and occur during the rainy season. Magnetic minerals through the water media are transported, flowing into

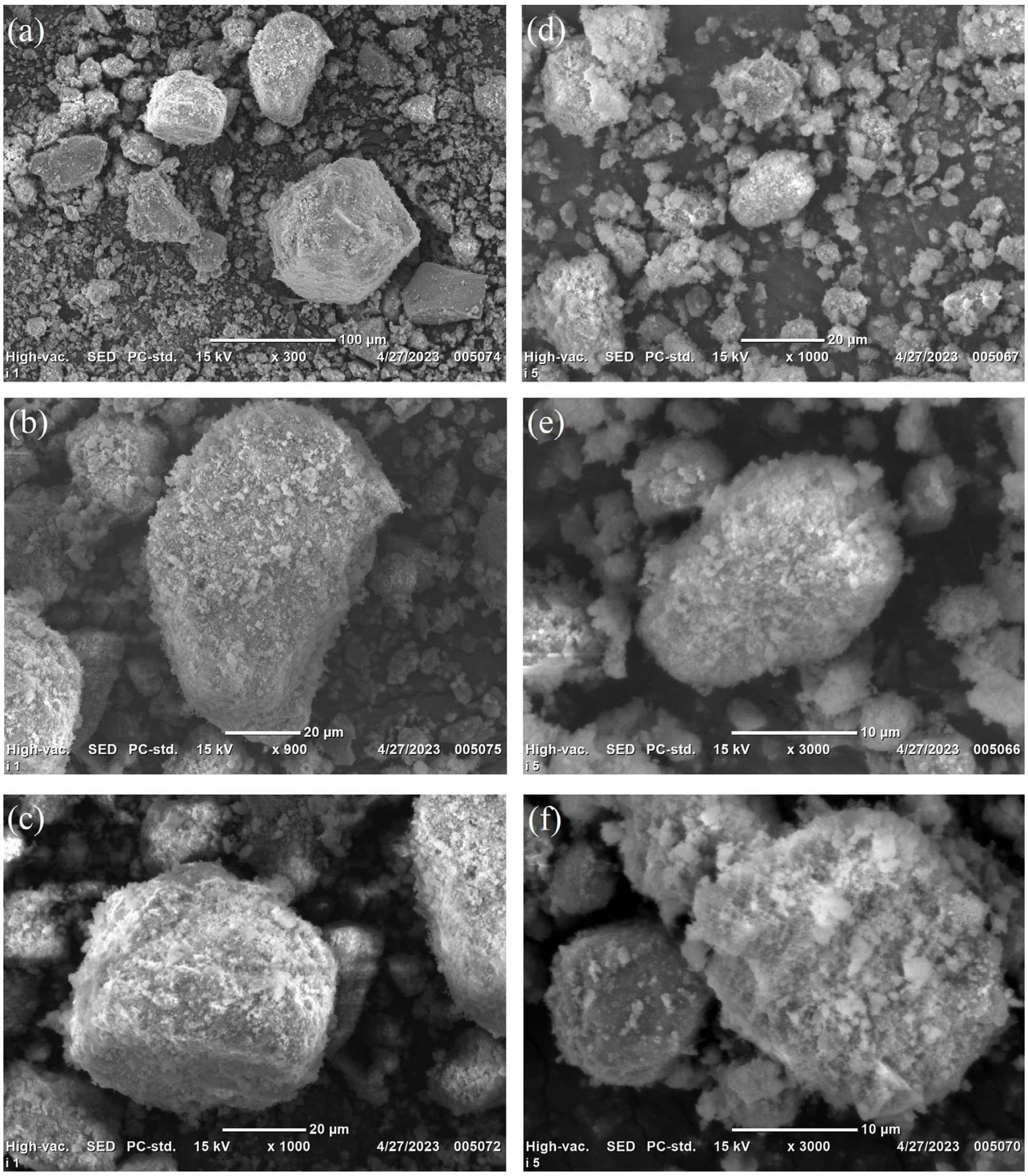

Fig. 5 SEM images of Dream cave guano samples (a-c) I-1 and (d-f) I-5.

the cave or dripping from the cave walls and stored for a long time. The SEM image results align with the χ_{FD} (%) interpretation which contains a mixture of fine and coarse magnetic grains for Bat Cave and some Dream cave guano samples. Furthermore, some of the Dream cave guano samples contained large magnetic grains. In addition, the SEM image results following the χ_{LF} - χ_{FD} (%) guano samples from Bat cave and Dream cave showed that the magnetic minerals in the guano come from natural processes (lithogenic) and human activities (anthropogenic) [40, 41].

SEM image also show that guano samples, which are light grey, belong to elements with high atomic numbers. In contrast, dark grey colours belong to elements with low atomic numbers. Elements with high atomic numbers, such as iron (Fe) shown in Table 2, were found in high concentrations in the Dream cave guano sample and vice versa in the Bat cave guano sample. Also, the Bat cave guano sample shows the presence of Ca and O elements, which are the constituent elements of the karst mineral, namely calcite ($CaCO_3$). In addition, the presence of Si in the guano of the two caves indicated that the samples contained mineral quartz (SiO_2). In addition, it was indicated that the guano sample contained ferrimagnetic minerals such as magnetite, which was indicated by the presence of Fe and O. The SEM-EDS results strengthened the interpretation of magnetic susceptibility and XRD data which showed that the guano sample contained minerals belonging to the ferrimagnetic mineral category, namely magnetite (Fe_3O_4). Even though the presence of magnetic minerals is low in concentration, it provides excellent magnetization [42 - 44].

Table 2. Elemental composition of guano samples from Bat cave and Dream cave.

Element	Mass %			
	Bat cave		Dream cave	
	T08	T29	I-1	I-5
B	9.29	22.32	-	-
O	13.14	9.73	24.82	22.86
Al	6.85	3.27	30.59	22.63
Si	6.34	4.80	3.86	4.34
P	2.01	1.85	ND	3.43
Ca	19.59	20.24	-	-
Fe	6.42	6.53	40.72	46.74
Br	14.72	11.11	-	-
Rb	4.66	2.38	-	-
W	5.39	1.04	-	-

CONCLUSIONS

Characterization of magnetic minerals based on magnetic susceptibility measurements shows that there are natural (lithogenic) magnetite grains in Bat cave guano samples, most of which are superparamagnetic grains. Meanwhile, in the Dream cave guano samples, refined magnetite grains indicate the presence of anthropogenic material. These results are confirmed by morphological analysis of SEM - EDS imagery and mineralogical analysis based on XRD measurements. The magnetic minerals contained in the guano samples are ferrimagnetic; magnetite (Fe_3O_4) was found in the guano samples in both Bat cave and Dream cave. This mineral has an oval, irregular morphology and fractured surface. The distribution of magnetic grains is also heterogeneous. Thus, the magnetic mineral sources in Bat cave and Dream cave guano tend to come from magnetic minerals controlled by lithogenic and anthropogenic components.

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Authors' contribution: *V.A.T: Conceptualization, Methodology, Writing original draft; M.A.: Investigation, Data collection karst, Review; P.P.: Alaysis of XRD data; Review; S: Analysis of magnetic susceptibility data; Resourses; M.J.R.: Analysis of SEM-EDS data, Review, H.J.R. and A.M.R.: Editing, Visualization.*

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