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#### SCIENTIFIC PAPER

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## THE CHARACTERISATION OF MAGNETIC MATERIALS EXTRACTED FROM ACEH IRON SAND

#### Article Highlights

- Iron sand was taken from four coastal places: Mon Klayu, Mantak Tari, Lam Panah and Syiah Kuala
- 100 g of the iron sand was separated by an external magnet and washed with distilled water
- The iron oxide found in Aceh iron sand was predominantly magnetite ( $\text{Fe}_3\text{O}_4$ ) or ilmenite ( $(\text{Fe}, \text{Ti}_3)\text{O}_4$ )

#### Abstract

*This study aims to identify the content and particle shape, and to determine the type, of iron oxide in the magnetic material from Aceh iron sand. The samples were taken from four seacoast locations, namely Mon Klayu, Mantak Tari, Lam Panah and Syiah Kuala beaches. The magnetic materials are separated from the iron sand using an external magnet, washed out with distilled water and dried at 80 °C. The materials were characterized by XRD and SEM. The results show that the magnetic materials sampled from Aceh iron sand contain 88.93, 96.3, 92 and 85% of Fe, respectively. The iron oxide found in Aceh iron sand was predominantly magnetite ( $\text{Fe}_3\text{O}_4$ ) or ilmenite ( $(\text{Fe}, \text{Ti}_3)\text{O}_4$ ); however, there are also other oxide impurities, such as silica and chromium oxides. The identified minerals were classified as lithogenous sedimentary volcanic minerals, which have a black colour and spherical shape. Related to that, the sampling locations of these minerals were types of volcanic minerals and lithogenous sediments, since the position of the coast locations is adjacent to Burni Telong, Peut Sago and Seulawah volcanos, and the minerals are carried by the stream to the beaches through the river flow, which further supports the assumption of their volcanic origins.*

*Keywords:* iron sand, magnetic materials, iron oxide, Aceh.

Along with the development of technology, magnetic materials are currently used not only as raw materials for steel making, but also in various fields such as electrochemical, catalytic and medical sciences [1,2]. The development of the magnet industry critically depends on the use of magnetic material components [3]. The currently used magnetic materials include iron oxide materials, such as hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ), maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ), ilmenite ( $(\text{Fe}, \text{Ti}_3)\text{O}_3$ )

and magnetite ( $\text{Fe}_3\text{O}_4$ ), and sulfide iron materials such as pyrrhotite ( $\text{Fe}_7\text{S}_8$ ) and greigite ( $\text{Fe}_3\text{S}_4$ ) [4]. The research on the use of iron oxide materials has been carried out in various fields [5].

Maghemite iron oxide is used as a supercapacitor material electrode [6] while magnetites are widely used as drug delivery agents to transport drugs to certain parts of the human body and as contrast agents in magnetic resonance imaging (MRI) [5]. The magnetic nanoparticle materials modified with polymers were used to adsorb metal ions such as Cd(II), Zn(II), Pb(II) and Cu(II) [3]. These three magnetic materials are usually synthesized in the laboratory using the co-precipitation method [7]. The precursors in the synthesis of magnetic materials are usually  $\text{FeCl}_{36}\cdot\text{H}_2\text{O}$ ,  $\text{FeCl}_{24}\cdot\text{H}_2\text{O}$ ,  $\text{FeSO}_{47}\cdot\text{H}_2\text{O}$  or  $\text{Fe}(\text{NO}_3)_{39}\cdot\text{H}_2\text{O}$  [8-10]. However, these precursors are

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relatively expensive and produce new waste. Therefore, a more effective, efficient, and environment-friendly source of magnetic materials is required to replace these precursors. The source of magnetic materials can be replaced with magnetic materials derived from natural iron sand. Iron sand is generally composed of iron oxide, namely magnetite ( $\text{Fe}_3\text{O}_4$ ), hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ), and maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ), and several other oxide compounds such as  $\text{Al}_2\text{O}_3$ ,  $\text{MgO}$ , and  $\text{SiO}_2$  [11]. Iron sand in Indonesia is found mostly in coastal locations, such as the coast of West Sumatra, the southern coast of Kebumen and the north coast of Java Island. Aceh has several regions with excellent potential minerals, such as Mon Klayu, Mantak Tari, Lam Panah and the Syiah Kuala coast. Potential minerals found in the area include magnesite, chromite, iron, quartz, limestone, sandstone and clay stone [12]. Therefore, it is necessary to identify iron sand materials from different areas, especially the province of Aceh, and to determine its iron content. An iron ore obtained from iron sand is usually mixed with soil, so an appropriate method is required to purify the iron sand. On a laboratory scale, the iron sand magnetic material is prepared manually using an external magnet [13], but it still contains several impurities [10]. Rianna *et al.* separated the magnetic phase by magnetic separation process using a permanent magnet, and showed that it mainly contains  $\text{Fe}_3\text{O}_4$  phase [14].

The sand can be classified by its physical properties or by the constituent mineral content. The classification based on the physical properties of the sand can be distinguished based on the shape, size, color and density of the sand. Three sub-quantities were used, where each quantity describes the shape at different scales [15]. These terms are morphology/form, roundness, and surface [16]. It is shown in Figure 1 how the scale terms are defined, while the graphic scale to illustrate the quantitative measure is shown in Figure 2 [17]. It is important to highlight that any comparing chart that describes particle properties

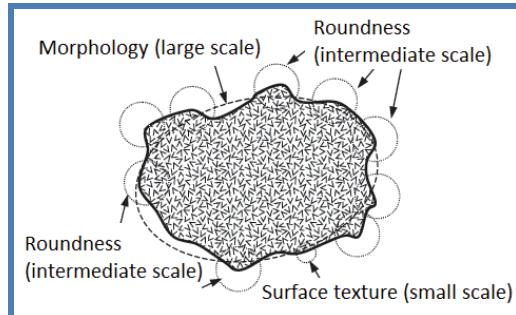


Figure 1. The shape-describing sub-quantities.

has a high degree of subjectivity. The classification can also be done according to differences of the chemical constituents of the sand. One method used to analyze mineral content in the sand is X-ray diffraction (XRD), which calculates the mineral percentages in the specific sample [18].

Therefore, the magnetic material has to be purified to remove impurities that cannot be pulled out by external magnets. In this study, the separation of the magnetic material from the iron sand was performed using external magnets; the magnetic material was prepared by washing out with distilled water before further characterization, identification of magnetic material content and determination of iron oxide types in the magnetic material of Aceh iron sand. This research can provide an alternative use of iron sand, supporting the national steel industry.

## EXPERIMENTAL

The samples of the iron sand originating from the coasts of Mon Klayu, Mantak Tari, Lam Panah and Syiah Kuala Beach were used for the preparation of magnetic materials, and the sampling coordinates and locations are given below, with a geographical representation in Figure 3.

Mon Klayu: latitude  $5^{\circ}16'18.14''\text{N}$ , longitude  $96^{\circ}51'48.90''\text{E}$

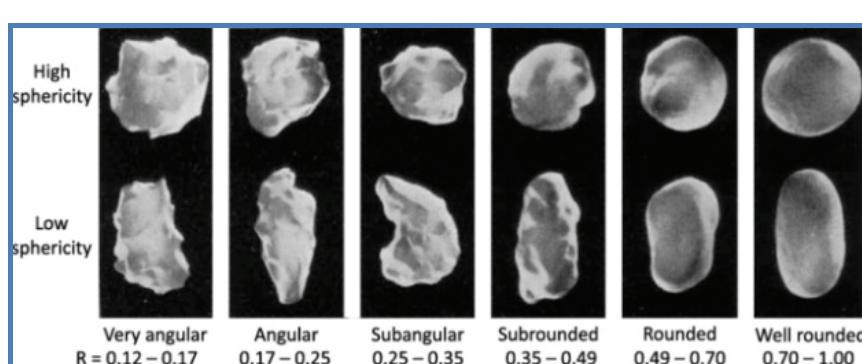


Figure 2. A roundness qualitative scale [16].

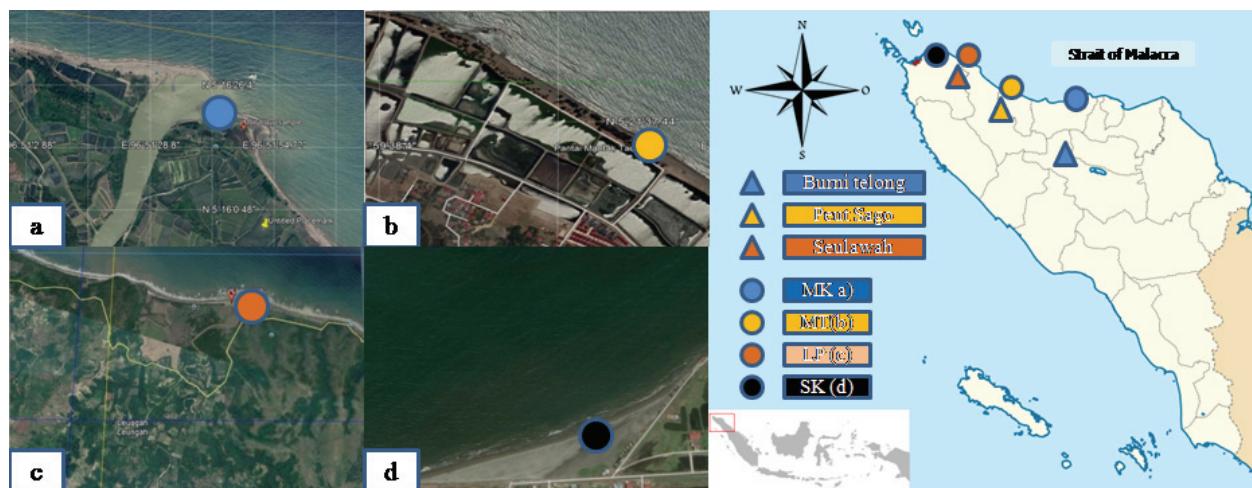


Figure 3. The location and coordinates of sampling: a) Mon Klayu (MK) beach; b) Mantak Tari (MT) beach; c) Lam Panah (LP) beach; d) Syiah Kuala beach (SK).

MantakTari: latitude 5°21'35.21"N, longitude 95°59'57.48"E

Lam Panah: latitude 5°34'42.22"N, longitude 95°43'3.72"E

Syiahkuala: latitude 5°35'37.94"N, longitude 95°19'47.18"E

100 g of the iron sand was separated by an external magnet and afterwards washed with distilled water. The washing procedure was repeated three times. The magnetic solids were dried in an oven at a temperature of 70 °C for 24 h. The obtained iron sand powder was characterized by X-ray diffractometry (Shimadzu XD610, and data processing software HighScore Plus) and scanning electron microscopy SEM equipped with EDS analysis (SEM JSM-6510LA, JEOL) in the laboratory of the National Nuclear Energy Agency of Indonesia (BATAN). The additional characteristic observation of the sand shape was conducted using an optical microscope. The shape of each sample was then compared with the image of the sand form in Figure 2.

## RESULTS AND DISCUSSION

### The Mineral content

The iron sand of Mon Klayu (MK) beach, Mantak Tari (MT), Lam Panah (LP) and Syiah Kuala (SK) before magnetic separation showed a grayish black color. After the magnetic separation, the black magnetic concentrate was obtained. The black color visually implies that iron oxide in the magnetic material is predominantly composed of the magnetite type [19]. However, further characterization is needed to support the identification of iron oxide types and the content of the iron sand magnetic material. The content of

the iron sand magnetic material and the amount of impurities that cannot be separated by the external magnet were determined by EDS analysis coupled with SEM characterization of the samples (Figure 4). The results of the characterization of iron sand magnetic materials are shown in Table 1.

Based on Table 1, it is shown that the magnetic materials sampled out at Mon Klayu, Mantak Tari, Lam Panah and Syiah Kuala locations contain Fe as a major element with the average levels above 90%, and also other minor elements. The high level of Fe is caused by the preparation procedure, which was carried out using external magnets and the geographical conditions of the area from where the samples were taken. This is very important since the iron sand collected from Lampung, Indonesia consists of majority Fe around 65 wt.% and many additional elements as well [20].

SEM results show that the levels of elements C, Mg, Si, Ti, Al, V, S, Na, Cr in the investigated magnetic materials are quite large. This is in agreement with previous studies, where the magnetic iron sand retrieved from Banten, Indonesia showed the presence of many different impurities, like Ti, Al, Ce, Co, Cr, Eu, La, Mg, Mn, Na, Sc, Sm, Th, V, Yb and Zn [21]. The iron oxide from Aceh iron sand is predominantly composed of magnetite ( $Fe_3O_4$ ) or ilmenite ( $(Fe,Ti)_3O_4$ ) (Figure 5). The levels of these oxides are quite large since they also have magnetic properties. Therefore, if the magnetic material of iron sand is pulled out by an external magnet, the oxide is also attracted to an external magnet. In addition, the Aceh sampling area is also a region producing Cr mineral resources. The obtained magnetic material still contains these elements in sufficient quantities. The con-

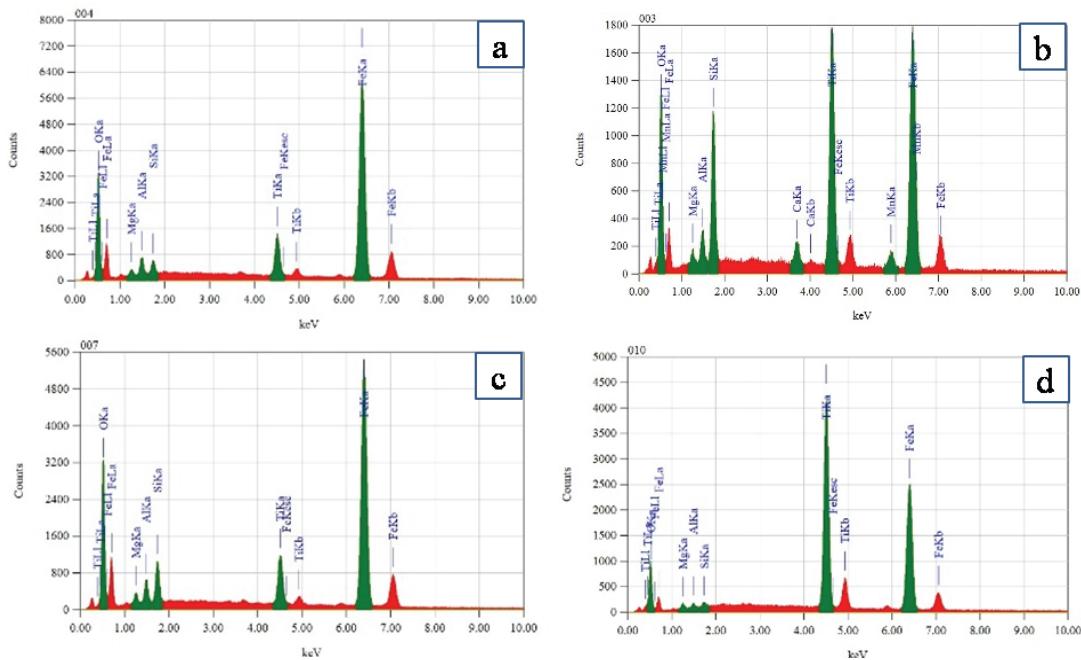


Figure 4. EDS spectra of iron sand samples from different regions: a) Mon Klayu; b) Mantak Tari; c) Lam Panah; d) Syiah Kuala beach.

Table 1. The elemental composition (%) of the magnetic materials determined by EDS coupled with SEM

Area	Element									
	Fe	C	Mg	Si	Ti	Al	V	S	Na	Cr
MK	88.93		0.93	1.39	6.65	2.09				
MT	96.3			1.67	0.54	1.22	0.27			
LP	92	2.7	0.54	2.5	0.74	0.94	0.2	0.13		0.12
SK	85	0.67	1.09	1.64	7.93	1.49			1.65	

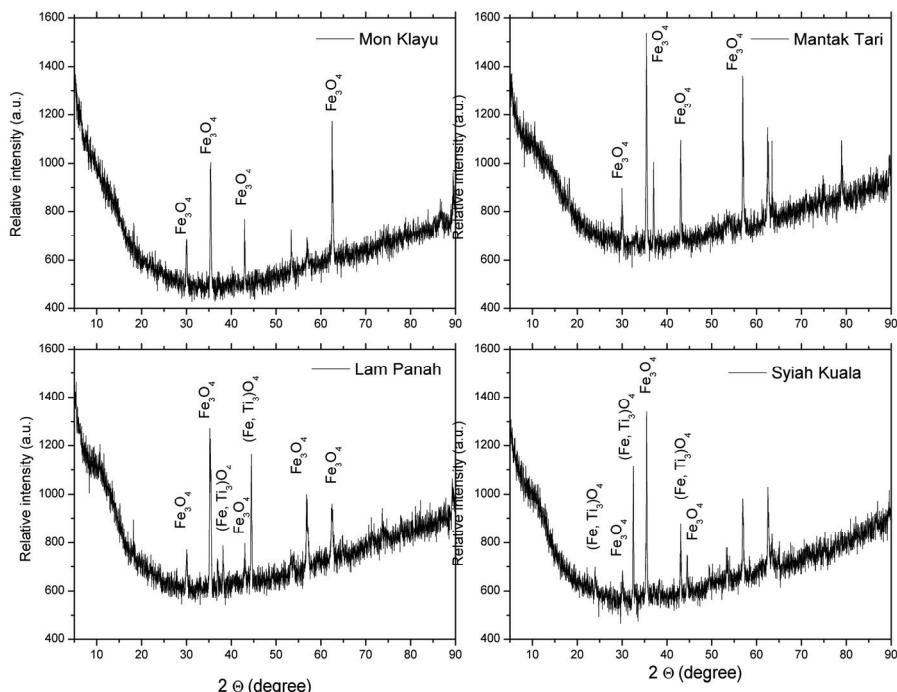


Figure 5. XRD patterns and related compositions of the ironsands: a) Mon Klayu and b) Mantak Tari; and the corresponding concentrates: c) Mon Klayu and d) Mantak Tari.

stuent elements of the magnetic material do not exist as chemical elements but rather in the form of oxides. Therefore, the magnetic material needs to be further characterized to find out the oxide phases present in the magnetic material. The mineral content affects the color of the iron sand [19]. The iron sand appears as black because of the dominant presence of cromite with a heterogeneous color layer structure: the layer contains a mixture of red, brown, white and solid black. This color identification can be seen in Table 2. The iron sand content is very dependent on the source of the local stone and environmental conditions. The bright white sand found on the beach is limestone or silica and some sands are rich in dark magmatic material so that the black appearance comes from volcanoes and oxides. In the black sand, the dominant minerals are magnetite ( $\text{Fe}_3\text{O}_4$ ), ilmenite ( $(\text{Fe},\text{Ti}_3)\text{O}_3$ ), diamond (C), or chromite ( $[\text{Mg},\text{Fe}]\text{Cr}_2\text{O}_4$ ). The darker the color of the sand, the higher the concentration of Fe phases or chromite is. This is interesting if compared to the iron sand extracted from Sarmi, Papua, Indonesia, region, which consists mainly of magnesium ferrite ( $\text{MgFe}_2\text{O}_4$ ) mineral, which has a high average magnetic susceptibility and proved to be useful for industrial applications [22].

*Table 2. The identification of the minerals according to color*

Color	Type of Mineral/compound/element
Red	Hematite ( $\text{Fe}_2\text{O}_3$ ), cuprite ( $\text{CuO}_2$ ), or pyrope ( $\text{Mg},\text{Al}_2\text{SiO}_12$ )
Yellow	Marcasite ( $\text{FeS}$ ), sulfur (S), chalcopyrite ( $\text{CuFeS}_2$ ) or vanadium (V)
Black	Magnetite ( $\text{Fe}_3\text{O}_4$ ), ilmenite ( $(\text{Fe},\text{Ti}_3)\text{O}_3$ ), diamond (C), or chromite ( $[\text{Mg},\text{Fe}]\text{Cr}_2\text{O}_4$ )
Brown	$\text{TiO}_2$ compound or spinel ( $\text{MgAl}_2\text{O}_4$ )
White	$\text{SiO}_2$ compound or orthoclase ( $\text{KAlSi}_3\text{O}_8$ )
Light green	Uvarovite ( $\text{Ca}_3\text{Cr}_2\text{Si}_3\text{O}_12$ ) or olivine ( $[(\text{Mg},\text{Fe})_2\text{SiO}_4]$ )
Orange	Spessartine ( $\text{Mn}_3\text{Al}_2\text{Si}_3\text{O}_12$ )

Based on the mineral content, the particle size of the iron sand can be classified as being of fine grade. Armstrong suggested that high levels of  $\text{SiO}_2$  and Ca-oxide can be found in medium-sized sand, while in a fine sand, the mineral content is generally that of iron sand (Fe),  $\text{Al}_2\text{O}_3$  and Ti [23]. Related to this, it is suggested that these minerals belong to the type of volcanic minerals and lithogenous sediments because the position of the four beaches is adjacent to the Burni Telong, Peut Sago and Seulawah volcanos, and the minerals are carried by the stream to the coast by the river flow [24].

## Particle shape

It can be seen in Figure 6 that the grains of the iron sand have an irregular form. Sphericity is a measure that describes the tendency of a grain shape toward a spherical shape [25]. The high roundness value, or the round shape of the grains of sand, indicates that the grain has undergone a further transport process, which results in the fine sand grains. The sand or the particle/grain is shaped by abrasion during transportation, where collisions between particles or with bedrock exist, so that the shape changes from angled/pointed to rounded. The longer the distance traveled, the more complex and the more rounded particles form [26].

Based on the analysis of the sediment fraction, the average particle size of the sand is 0.14 mm, which means that Aceh beach sand can be categorized as fine. The visual inspection of the sample shows that the sand sample is in the coral form. The size of the sediment granules in the study location is inseparable from the surrounding environmental conditions, which favors the sediment formation. One of these conditions is the source of sedimentary components from the land, such as the process of abrasion or erosion, which are then carried by the river. The factor that influences the size of the sedimentation grain is the sediment material transport mechanism. This will determine the depositional variation that occurs, so the coarser particles are easier to be deposited than finer ones, since the transport mechanism is affected by hydro-oceanographic factors, such as water flow and tides [19].

## CONCLUSION

Depending on the locations, the extracted magnetic materials have different amounts of Fe-minerals: 88.93% (Mon Klayu), 96.3% (Mantak Tari), 92% (Lam Panah), and 85% of Fe (Syiah Kuala), as well as C, Mg, Si, Ti, Al, V, S, Na, Cr and other minor elements. Iron oxide in Aceh iron sand is predominantly in magnetite ( $\text{Fe}_3\text{O}_4$ ) or ilmenite ( $(\text{Fe},\text{Ti}_3)\text{O}_3$ ) form. The results of characterization show that the magnetic materials of the iron sand still contain oxide impurities, such as silica oxide and chromium oxide. The types of minerals identified were classified into volcanic mineral types from lithogenous sedimentary types, black in color and rounded in shape. This corroborates well with the position of the Mon Klayu, Mantak Tari, Lam Panah and Syiah Kuala beaches, which are adjacent to the Burni Telong, Peut Sago, and Seulawah volcanos, where the minerals are carried by stream to the beach by the river flow.

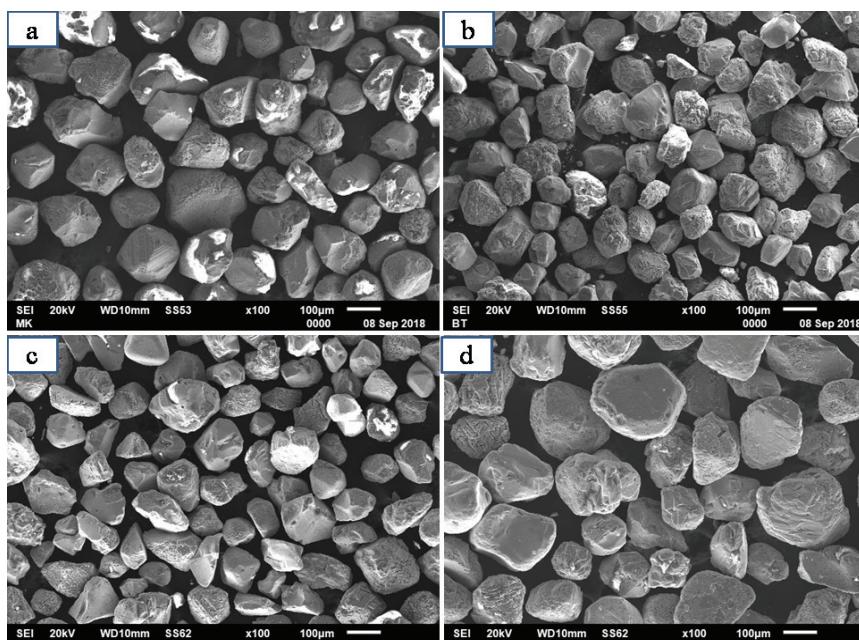


Figure 6. The SEM micrographs of particle shapes of different iron sands: a) Mon Klauy; b) Mantak Tari, c) Lam Panah and d) Syiah Kuala.

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NAUČNI RAD

## KARAKTERIZACIJA MAGNETNIH MATERIJALA IZDVOJENIH IZ PESKA SA SADRŽAJEM GVOŽĐA IZ ACEHA

Cilj ovog istraživanja je određivanje sadržaja i oblika čestica, kao i vrste gvožđe-oksida, u magnetnom materijalu peska iz provincije Aceh. Uzorci su uzeti sa četiri lokacije na obali mora, i to plaže Mon Klaiu, Mantak Tari, Lam Panah i Siiah Kuala. Magnetni materijali se izdvojeni iz peska spojnim magnetom, isprani destilovanom vodom i sušeni na 80 °C. Materijali su okarakterisani metodom XRD i SEM. Rezultati pokazuju da magnetni materijali sadrže 88,93, 96,3, 92 i 85 % Fe, redom. Gvožđe-oksid nađen u pesku iz Aceha je dominantno magnetit ( $Fe_3O_4$ ) ili ilmenit ( $Fe, Ti_3O_4$ ), ali postoje i drugi oksidi, kao što su oksidi silicijuma i hroma. Identifikovani minerali klasifikovani su kao litogeni sedimentni vulkanski minerali, koji imaju crnu boju i sferni oblik. S tim u vezi, lokacije sa kojih su uzeti uzorci ovih minerala bile vrsta vulkanskih minerala i litogenih sedimenata budući da je položaj priobalnih lokacija u blizini vulkana Burni Telong, Peut Sago i Seulawah, a minerali se donešeni do plaže rekom kroz, što dalje podržava pretpostavku o njihovom vulkanskom poreklu.

*Ključne reči:* pesak sa sadržajem gvožđa, magnetni materijali, gvožđe-oksid, Aceh.