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PART A

APPLIED
AND NATURAL
SCIENCES

PROQRES
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2ND ISSUE

COMPOSITION ANALYSIS OF TITANOMAGNETITE AND ILMENITE FROM IRON SAND USING NEUTRON ACTIVATION ANALYSIS AS PRELIMINARY STUDY FOR PRODUCING IRON OXIDE AND TITANIUM DIOXIDE

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ABSTRACT

Identification and composition analysis of iron sand from local resources have been performed. Iron sand samples retrieved from Banten, Indonesia was milled for 10 hours at room temperature by using high energy milling (HEM). The powder sample was separated by its type using magnetic separator. They are found two type of iron sand, titanomagnetite and ilmenite types. The elementary analysis results using neutron activation analysis (NAA) method is obtained that the iron sand contained the majority of iron (Fe) and titanium (Ti). The refinement results of X-ray diffraction pattern and NAA shows that the ilmenite type consists of 75.60 wt% ilmenite phase ($Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$), and 24.40 wt% hematite phase (-Fe_2O_3). The titanomagnetite type consists of 72.37 wt% titanomagnetite phase ($Fe_{2.54}Ti_{0.30}M^*_{0.15}O_4$), 11.67 wt% ilmenite phase ($Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$), and 15.96 wt% hematite phase (-Fe_2O_3). From this study it has been successfully understood the identification and analysis of iron sand composition from Banten, Indonesia, is able to be used as a reference of further processing both for laboratory and industrial scale.

Key words: iron sand, local resources, milling, separator, neutron activation analysis, element, composition phase

1. INTRODUCTION

One of the potential local natural resources in Indonesia is iron sand. It is found more than 2 billion tons and scattered along the beach on the island of Java, Sumatra, West Nusa Tenggara, and still has not been processed yet in an integrated way until now [1-2]. Iron sand is a source of raw materials that can be processed into semi-finished industry raw materials to substitute the imported materials [3]. The iron sand has potentially as raw material sources for magnets industry of ferrite-based [4-5].

Iron sand or called as titan iron ores formed due to sedimentation processes in chemical and physical from andesitic up to basalytic rocks. The process continued to physical process, through the destruction of rocks by water currents, washing repeatedly, displacement due to waves currents, and precipitate along the coast containing iron rich (Fe). The content of iron in iron sand in each area will vary depending on several factors, such as the formation of deposits of iron sands, temperature, and topography factors (slope) which plays a central role as the accumulation of iron sand. This iron sand has a number of relatively high iron content but generally carry a number of relatively high titanium content as well which sometimes explored to produce titanium concentrate in addition to used for iron ore [6]. According to the elemental analysis results showed that the elements content in the iron sand is very complex. Some of the elements contained in the iron sand is most likely not stand alone but linked together to form specific compounds it requires the process technology to separate those compounds [7]. However, prior to processing, preliminary identification of iron sand is necessary to know the content of elements and compounds.

The elements in the iron sand can be identified using a nuclear technology, neutron activation analysis (NAA) [8]. NAA is quantitative and qualitative method of high efficiency for the precise determination of a number of main-components and trace elements in different types of samples. NAA works based on the nuclear reaction between neutrons and target nuclei. It is a useful method for the simultaneous determination of about 25-30 major, minor and trace elements of geological, environmental, biological samples in ppb-ppm range without or with chemical separation. Although new analytical methods, such as (Inductively Coupled Plasma Atomic Absorption Spectroscopy ICP-AAS, Inductively Coupled Plasma Mass Spectrometry ICP-MS, X-ray fluorescence XRF, Energy Dispersive Spectroscopy EDS, etc.), can also be widely applied in anaiytical chemistry, NAA is still competitive and superior in many areas. The indisputable advantage of the method is its sensitivity and accuracy, especially in respect of some trace elements. The method is able to determine elements simultaneously without chemical separation.

The measurement with NAA combined with the phase analysis using neutron diffraction or X-ray diffraction has been obtained the phase composition of iron sand samples. So it will be known that each element of a stand-alone form its oxide or mutually bonded to each other to form a particular compound. These results will determine the next process step for each element separation. For example, if the results of the phase composition analysis showed that the presence of iron oxides and titanium dioxide is separate, then the separation can be easily done by using a magnetic separator or floatation technique. However, if the elements of iron and titanium bonded to each other to form specific compounds, the separation process each element is not simple anymore but must be carried out with extraction process using chemical separation and based on the phase composition data.

The aim of this study was to identify and analyze the chemical and phase composition of iron sand from Banten, Indonesia. It was expected to obtain the compound contents of the iron sand, that to be used as a reference of further processing both for laboratory and industrial scale.

2. MATERIALS AND METHODS

Iron sand samples retrieved from Banten, Indonesia, was cleaned from non magnetic materials using magnetic separation with high magnetic field. The iron sand was milled using a high-energy milling (HEM) of SPEX 8000 type. HEM was set at normal conditions with the speed of 1400 rpm, run time of 90 minute, and rest time of 30 minutes. The iron sand was milled for 10 hours at room temperature to obtain fine particles in order to facilitate further characterization and processing. The next step was to separate the iron sand post grinding by using a magnetic separator with low magnetic field. From this separation, it was obtained 2 types of iron sand, namely type-1 (iron sand does not stick to the magnet) and type-2 (Iron sand stuck to the magnet). Furthermore, the iron sand powders are respectively called by ISMS-1 and ISMS-2 samples for type-1 and type-2.

The elemental composition analysis was performed using NAA. Iron sand sample ISMS-1 and ISMS-2 weighed in vial LDPE (low density polyethylene) were using a microbalance with a weight of 20-50 gr. The standard reference material of Buffalo river sediment from NIST is used as internal quality control and as flux monitor used Al-0.1% Au from the Institute for Reference Materials and Measurement (IRMM). The samples, SRM and flux monitor put together into irradiation capsules made of polyethylene or aluminium. The irradiation have been done at thermal neutron flux of about 10^{13} n.cm^{-2}.s^{-1} in the irradiation facility of Multi Purpose Reactor GA. Siwabessy in Serpong. The counting of irradiated samples have been performed by a high resolution HPGe (ε =15 %, FWHM=1.89 keV on 1.33 MeV) detector from Cannbera coupled to multichannel analyzer. The data have been analyzed by GENIE 2000 and k0-IAEA software. The Scheme of irradiation, decay and counting time for analysis of iron sand samples are listed in Table 1.

Table 1. Scheme of irradiation, decay and counting time for analysis of iron sand samples

Facilities	T_i	T_d	T_c	Radionuclides
Rabbit System	15 s	3-5 m	200 s	^{28}Al, ^{57}Mg, ^{56}Mn, ^{51}Ti, ^{52}V
	10 m	1-2 d	1800 s	^{24}Na, ^{42}K, ^{76}As, ^{140}La, ^{239}U, ^{72}Ga, ^{187}W
	1 h	2 w	7200 s	^{51}Cr, ^{60}Co, ^{141}Ce, ^{152}Eu, ^{59}Fe, ^{124}Sb, ^{46}Sc, ^{65}Zn, ^{182}Ta, ^{181}Hf, ^{169}Yb

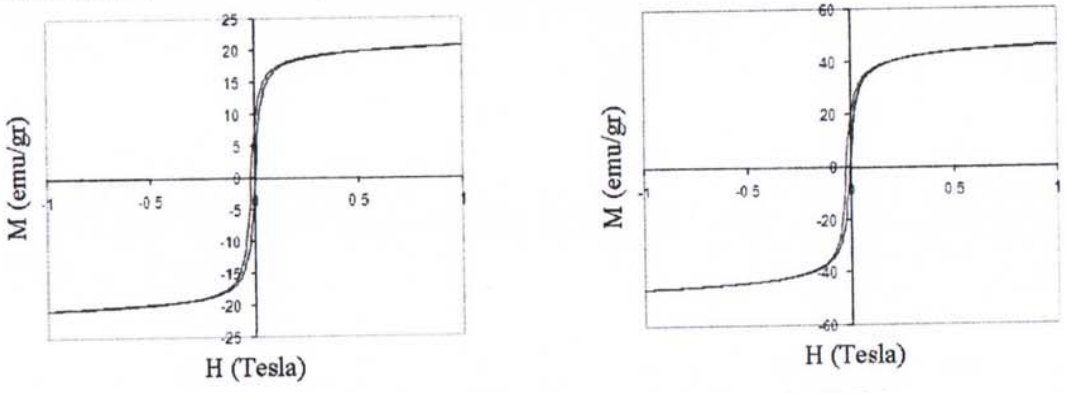
Annotation: T_i (irradiation time), T_d (decay time), T_c (counting time), s (seconds), m (minutes), h (hours), d (days), and w (weeks).

The qualitative and quantitative phase has carried out by using the X-ray diffractometer, type of PW1710 Philips with cooper anode tube. The measurement has running with the current of 30 mA and voltage of 40 kV. The wavelength of radiation (CuKα) was 1.5406 Å. The range of diffraction angles was from 20° to 80° with a step size of 0.02°. The data was analyzed by GSAS program (Rietveld code). The pseudo-Voigt function was used in the refinement of diffraction line profiles [9,10]. The magnetic properties were measured by vibrating sample magnetometer, Oxford VSM1.2H Instrument. The all characterizations were carried out at the Center for Science and Technology of Advanced Materials (PSTBM), National Nuclear Energy Agency (BATAN).

3. RESULTS AND DISCUSSION

3.1. Magnetic Properties

In Figure 1, the hysteresis curves of ISMS-1 and ISMS-2 samples are compared. All of samples showed ferromagnetic behavior. It indicates the presence of remanence and coersivity field in each respective magnetization.



(a) ISMS-1 (b) ISMS-2

Fig. 1. *The hysteresis curves of the samples*

The saturation value of magnetization from ISMS-1 and ISMS-2 samples are ~ 20 emu/gr and ~ 40 emu/gr, respectively. It is suspected that ISMS-1 and ISMS-2 samples are respectively weak and strong soft magnetic phases. These results confirmed with the identification of the samples using magnetic separator as results the ISMS-1 sample does not stick to the magnet compared with ISMS-2 sample that stuck to the magnet. However, VSM is not able to identify the phases of the sample ISMS-1 and ISMS-2.

3.2. Analysis of the Composition

Results of the internal quality control of NAA method by using the NIST SRM 2704 Bufallo river sediment is presented in Figure 2.

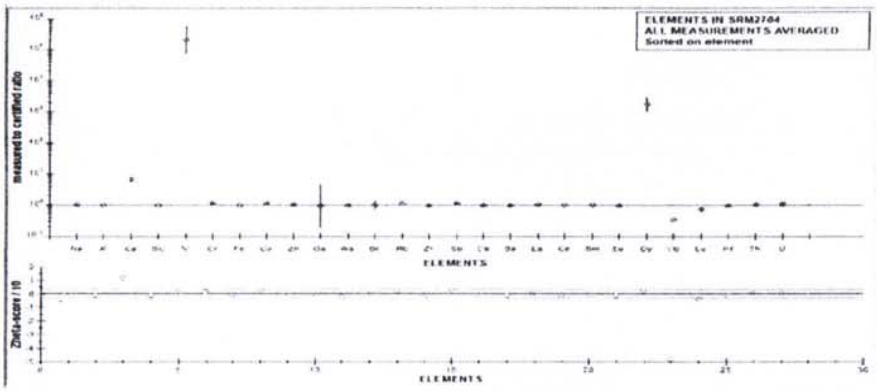


Fig. 2. *The results of the quality control method using NIST SRM 2704 river sediment Bufallo [11]*

Results data of the measurement of Fe, Co, As, Sb, Cr and Zn were compared to the value of the certificate which is to 1, almost all value located on one line and the Z-score within the range of ± 3. This means that the data analysis by k0-AANI method is valid [11].

Figure 3 shows the gamma-ray spectrum of NAA measurement result on ISMS-1 sample.

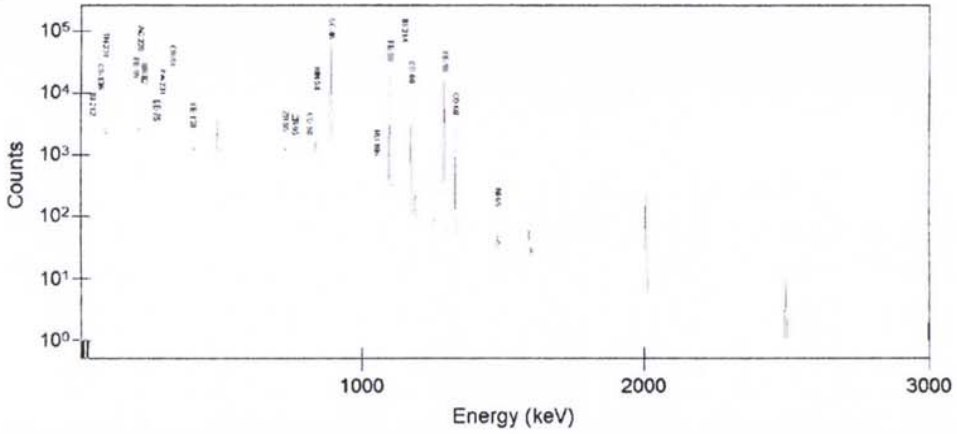


Fig. 3. *The gamma-ray spectrum from ISMS-1 sample showing several short-lived elements where irradiated for 2 hours, decayed for 15 days, and counted for 1.800 seconds using HPGe detector.*

Figure 4 shows the gamma-ray spectrum of NAA measurement result on the ISMS-2 sample.

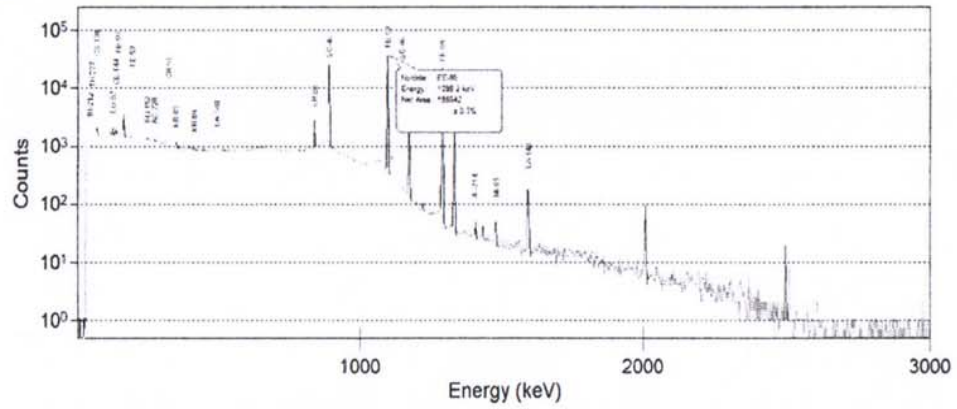


Fig. 4. *The gamma-ray spectrum from ISMS-2 sample showing several elements where irradiated for 2 hours, decayed for 15 days, and counted for 1.800 seconds using HPGe detector.*

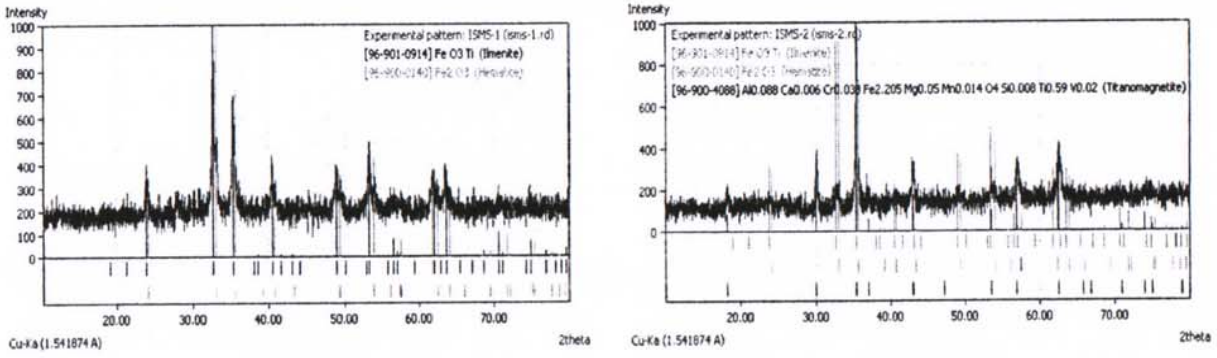
The spectrum of gamma energy as Figure 3 and Figure 4 show that the dominant elements are iron (Fe-59) at 1099.25 keV, and titanium (Ti-51) at 320.08 keV, respectively. This means that the sample is rich in content of iron and titanium. The detail concentration of elements content in the sample is given in Table 2.

Table 2. The result of elementer analysis of the ISMS-1 and ISMS-2 samples

No.	Element	Content (□g/g)			
		ISMS-1		ISMS-2	
		Concentration	Uncertainty	Concentration	Uncertainty
1.	Fe	369,435.83	61.50	585,223.43	103.85
2.	Ti	166,045.86	9,460.53	70,051.89	4,250.25
		M* (impurity)			
No.	M* (impurity)	Content (□g/g)			
		ISMS-1		ISMS-2	
		Concentration	Uncertainty	Concentration	Uncertainty
1.	Na	286.17	4.81	258.60	4.27
2.	Sc	78.08	0.74	33.96	0.43
3.	Cr	1,231.90	3.73	685.89	3.29
4.	Co	100.22	0.98	150.98	1.52
5.	Zn	292.27	3.42	696.64	4.67
6.	Ga	19.82	0.88	53.97	1.41
7.	As	4.08	0.38	6.65	0.52
8.	La	5.26	0.12	11.24	0.20
9.	Sm	1.34	0.04	1.88	0.04
10.	Eu	2.29	0.23	0.50	0.05
11.	Ho	0.56	0.14	0.45	0.11
12.	Yb	2.29	0.23	1.11	0.22
13.	Hf	19.01	0.55	1.87	0.35
14.	W	3.91	0.30	1.17	0.25
15.	Pt	256.83	23.36	87.13	17.83
16.	U	0.52	0.17	0.97	0.14
17.	Sb	-	-	0.74	0.15
18.	Th	0.94	0.33	1.67	0.19
19.	Ce	-	-	43.66	3.18
20.	Mg	8,855.94	1,142.74	3,890.12	1,207.50
21.	Mn	4,970.22	171.64	4,161.70	149.28
22.	V	1,560.68	49.70	2,758.38	87.13
23.	Al	7,376.73	262.52	14,131.33	568.53
24.	Ta	8.27	0.34	-	-

According to the results of the elementary analysis the element contents of the iron sand sample is also very complex. In the Table 2 displays that the content of iron and titanium ISMS-1 sample are respectively around 36.94 and 16.61 wt%. It means that this ISMS-1 sample is grouped in the kind of low of iron content and rich of titanium. While the content of iron and titanium in the ISMS-2 sample are respectively around 58.52 and 7.01 wt%, it is grouped in the kind of rich of iron. These results indicate that the iron sand has economically potential to be processed further. However some of the elements contained in the iron sand is most likely not stand alone but linked together to form specific compounds.

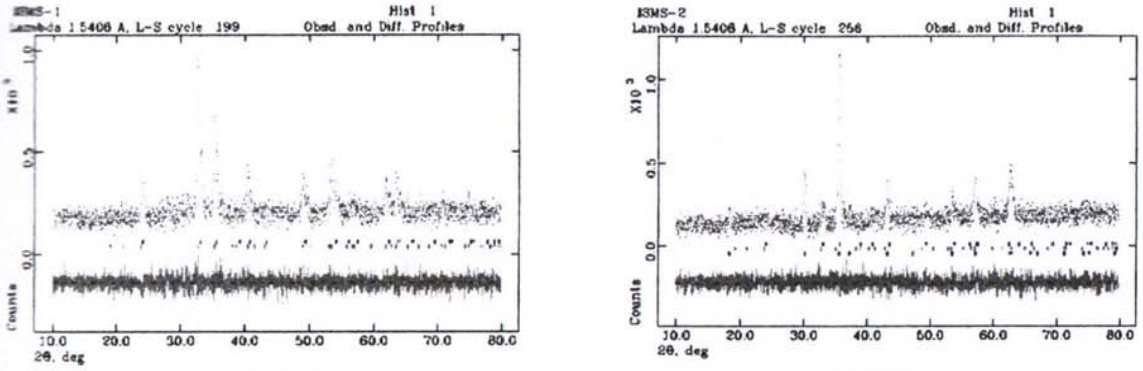
The identification results of measurements by X-ray diffraction on the ISMS-1 and ISMS-2 samples are shown in Figure 5. According to the Hanawalt table showed that the samples of ISMS-1 and ISMS-2 can be identified as multi phases.



(a) ISMS-1 (b) ISMS-2

Fig. 5. *The result of identification of X-ray diffraction pattern of iron sand samples*

The phase identification refers to the research results of Downs (ICDD 99-100-0951) [12], Yamanaka (ICDD 96-901-0914) [13], and Blake (ICDD 96-900-0140) [14] for the phase of titanomagnetite, ilmenite, and hematite, respectively. Since the Hanawalt table can be used to qualitative analysis only, the GSAS software is used for quantitative analysis. Figure 6 shows the refinement result of X-ray diffraction pattern of ISMS-1 and ISMS-2 samples.



(a) ISMS-1 (b)ISMS-2

Fig. 6. *Refinement result of X-ray diffraction pattern of iron sand samples*

The refinement result has produced very good quality of fitting with very small R factor. Factor R is a criteria factor of fit. Goodness of fit (S or χ^2 (chi-squared)) is very small. According to Izumi that S values is allowed a maximum of 1.3 [10]. While the parameters structure of the refinement results are showed in Table 3 and Table 4 for ISMS-1 and ISMS-2 samples, respectively.

Table 3. Structure parameter, factor R and *goodness of fit* (χ^2) of ISMS-1 sample

Ilmenite phase		
Space group: R -3 (148), Crystal system : Hexagonal Lattice parameter: $a = b = 5.072(1)$ Å, $c = 13.978(4)$ Å, $\alpha = \beta = 90°$ and $\gamma = 120°$, $V = 311.4(1)$ Å^3 and $\rho = 4.854$ gr.cm^{-3}		
Hematite phase		
Space group: R -3 c (167), Crystal system : Hexagonal Lattice parameter: $a = b = 5.037(2)$ Å, $c = 13.772(9)$ Å, $\alpha = \beta = 90°$ and $\gamma = 120°$, $V = 302.6(3)$ Å^3 and $\rho = 5.257$ gr.cm^{-3}		
Factor R	*w*Rp = 14.31 Rp = 11.22	χ^2 (chi-squared) = 1.245

Table 4. Structure parameter, factor R and *goodness of fit* (χ^2) of ISMS-2 sample

Titanomagnetite phase		
Space group: F d -3 m (227), Crystal system : Cubic Lattice parameter: $a = b = c = 8.376(2)$ Å, $\alpha = \beta = \gamma = 90°$; $V = 587.6(5)$ Å^3, and $\rho = 5.045$ gr.cm^{-3}		
Ilmenite phase		
Space group: R -3 (148), Crystal system : Hexagonal Lattice parameter: $a = b = 5.116(8)$ Å, $c = 13.873(2)$ Å, $\alpha = \beta = 90°$ and $\gamma = 120°$, $V = 314.5(1)$ Å^3 and $\rho = 4.807$ gr.cm^{-3}		
Hematite phase		
Space group: R -3 c (167), Crystal system : Hexagonal Lattice parameter: $a = b = 5.029(3)$ Å, $c = 13.827(2)$ Å, $\alpha = \beta = 90°$ and $\gamma = 120°$, $V = 302.9(7)$ Å^3 and $\rho = 5.252$ gr.cm^{-3}		
Factor R	*w*Rp = 19.56 Rp = 15.09	χ^2 (chi-squared) = 1.296

Based on the refinement result of X-ray diffraction pattern is obtained mass fraction of phase contents as shown in Table 5 for ISMS-1 and ISMS-2 samples.

Table 5. The mass fraction of phases contents on the ISMS-1 sample

No.	Mineral name	Phase	Mass fraction (%)	
			ISMS-1	ISMS-2
1.	Ilmenite	$(Fe,Ti,M^*)_2O_3$	75.60 ± 0.03	11.67 ± 0.01
2.	Hematite	α-Fe_2O_3	24.40 ± 0.06	15.96 ± 0.01
3.	Titanomagnetite	$(Fe,Ti,M^*)_3O_4$	-	72.37 ± 0.04

Table 5 indicated that the phases contained in the ISMS-1 sample are titanomagnetite, ilmenite, and hematite phases. While the phases contained in the ISMS-2 sample are only ilmenite, and hematite phases. Since an impurity elements M* presents from iron sand, then it requires further confirmation in connection with the analysis of the composition of the iron sand. Therefore, it is necessary to analyze its composition by using neutron activation analysis. In accordance to the calculation results of the mass fraction and element content is obtained the result of chemical element for ilmenite ($(Fe_p,Ti_q,M^*_r)_2O_3$) and titanomagnetite ($(Fe_x,Ti_y,M^*_z)_3O_4$) as shown in Table 6.

Table 6 shows the chemical element for ilmenite and titanomagnetite phases in the samples.

Table 6. The chemical element for ilmenite and titanomagnetite phases in the samples

Phase	Atomic composition (at.%)						Chemical element
	p	*q*	*r*	*x*	*y*	*z*	
$(Fe_p,Ti_q,M^*_r)_2O_3$	0.36	0.35	0.29	-	-	-	$Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$
$(Fe_x,Ti_y,M^*_z)_3O_4$	-	-	-	0.85	0.10	0.05	$Fe_{2.54}Ti_{0.30}M^*_{0.15}O_4$

The result of calculation showed that the ilmenite phase has the empirical composition of $Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$ and the titanomagnetite phase has the empirical composition of $Fe_{2.54}Ti_{0.30}M^*_{0.15}O_4$.

The results of the phase composition analysis showed that the presence of iron and titanium are separately, in other hand the elements of iron and titanium bonded to each other to form specific compounds, namely the ilmenite phase ($Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$) and the titanomagnetite phase ($Fe_{2.54}Ti_{0.30}M^*_{0.15}O_4$). So the separation of the phases can not be easily done by using a magnetic separator or floatation technique, but must be carried out with the extraction process using chemical separation.

4. CONCLUSIONS

This study has successfully understood identification and analysis of the composition of iron sand from Banten, Indonesia. Neutron Activation Analysis is quantitative and qualitative method of high efficiency for the precise determination of a number of main-components and trace elements in different types of samples. The method is also able to determine elements simultaneously without chemical separation. The refinement results of x-ray diffraction pattern and NAA shows that the iron sand consists of two type, namely, titanomagnetite and ilmenite types. The ilmenite type consists of two phase, namely 75.60 wt% of ilmenite ($Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$), and 24.40 wt% of hematite (α-Fe_2O_3) phases. The titanomagnetite type consists of three phase, namely 72.37 wt% of titanomagnetite ($Fe_{2.54}Ti_{0.30}M^*_{0.15}O_4$), 11.67 wt% of ilmenite ($Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$), and 15.96 wt% of hematite (α-Fe_2O_3) phases. $Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$ phase has a structure of hexagonal (R -3 c) with lattice parameters $a = b = 5.116(8)$ Å, $c = 13.873(2)$ Å, $\alpha = \beta = 90^\circ$ and $\gamma = 120^\circ$, V = 314.5(1) Å^3 and $\rho = 4.807$ gr.cm^{-3}, while the titanomagnetite phase has the empirical composition of $Fe_{2.54}Ti_{0.30}M^*_{0.15}O_4$. $Fe_{0.72}Ti_{0.70}M^*_{0.58}O_3$ phase has a structure of cubic (F d -3 m) with lattice parameters $a = b = 5.116(8)$ Å, $c = 13.873(2)$ Å, $\alpha = \beta = 90^\circ$ and $\gamma = 120^\circ$, V = 314.5(1) Å^3 and $\rho = 4.807$ gr.cm^{-3}. The phase composition analysis showed that the presence of iron and titanium bonded to each other to form specific compounds, so that the separation of the phases can not be easily done by using a magnetic separator or floatation technique, but must be carried out with the extraction process using chemical separation.

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