A CHARACTERISTIC STUDY OF POPAY ZIRCON SAND USED FOR CERAMICS, REFRACTORY AND FOUNDRY RAW MATERIALS

STUDI KARAKTERISTIK PASIR ZIRKON POPAY SEBAGAI BAHAN BAKU KERAMIK, REFRAKTORI DAN PASIR CETAK

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ABSTRACT

The objective of this study is characterizing the zircon sand from Popay of Nanga Pinoh District, West Kalimantan as well as its performance when separated using physical method, i.e., tabling and magnetic separator in terms of obtaining zircon concentrate for making zircon flour. The satisfied requirement of zircon flour will be used for ceramics, refractory and foundry raw materials. Tabling followed by magnetic separator of Popay zircon sand increase zircon content from 43.54 to 65.50%. The content increases to 66.11% when reversing the process, namely started with magnetic separator and then tabling. Mineralogical analysis using optical microscope detected six minerals available within zircon sands. Those are zircon, ilmenite, magnetite, hematite, rutile and quartz while XRD analysis only identified five minerals. Hematite was not distinguished within Popay samples. Chemical analysis of the samples shows that the ZrO_2 content within zircon flour is bigger than 65%. Such a figure is categorized as premium class for zircon flour to be used for refractory, ceramics and foundry.

Keywords: Popay zircon, tabling and magnetic separator, optical microscope and XRD analysis.

ABSTRAK

Tujuan penelitian ini adalah melakukan karakterisasi pasir zirkon Popay yang berasal dari Kecamatan Nanga Pinoh, Kalimantan Barat dan kinerjanya ketika diolah dengan meja goyang dan pemisah magnet. Pengolahan menggunakan kedua alat tersebut adalah untuk memperoleh tepung zirkon yang dapat memenuhi spesifikasi bahan baku untuk keramik, refraktori dan pengecoran. Pengolahan menggunakan meja goyang dan diikuti oleh pemisah magnet menunjukkan kenaikan kadar zirkon dari 43,54 menjadi 65,50% sedangkan bila proses dibalik, yaitu pemisah magnet terlebih dahulu dan kemudian meja goyang menghasilkan kadar zirkon sampai 66,11%. Komposisi mineralogi dari pengujian mikroskop optik maupun XRD hampir sama, yaitu zirkon, ilmenit, magnetit, rutil and kuarsa. Hematit hanya terdeteksi dari pengujia mikroskop optik. Hasil analisis kimia menunjukkan kandungan ZrO_2 dalam tepung zirkon >65% dan angka tersebut menyatakan bahwa tepung zirkon Popay hasil olah sudah termasuk kelas premium yang dapat digunakan untuk refraktori, keramik dan Pasir Cetak.

Kata kunci: zirkon Popay; meja goyang dan pemisah magnet; analisis mikroskop optik dan XRD.

INTRODUCTION

Zircon concentrates, derived from zircon sand, can be processed into several zircon-based materials such as zircon opacifier ($ZrSiO_4$), zirconium oxychloride or zirconyl chloride or ZOC - $ZrOCl_2.8H_2O$, zirconium sulphate or ZOS - $Zr(SO_4)_2.4H_2O$, zirconium basic sulphate or ZBS - $Zr_5O_8(SO_4)_2.xH_2O$ or $Zr_5O_7(SO_4)_3.xH_2O$, zirconium basic carbonate

or ZBC - ($ZrOCO_3.xH_2O$), zirconium hydroxide or ZOH - ($Zr(OH)_4.xH_2O$), zirconium oxide or zirconia (ZrO2) and zirconium metal (Zr). The need of zircon increases from year to year. Dealing with zircon prospect, a market study shows that world consumption on zirconium products will sharply increase through 2020. Such consumption is not proportionate to the current zircon smelter. As a result new smelter plants need to be developed.

Though reserve of Indonesian zircon sand has not yet been recognized, its potential is assumed significant enough. Such a mineral is distributed in North Sumatera, Riau, Bangka, and Belitung and also in West-, Central- and East Kalimantan. The sand is normally associated with tin and gold ores as an accessory mineral. Zircon mineral retains $ZrSiO_4$ formula or known as zirconium silicate and such a mineral is the most commercially used. Zircon resources in Indonesia is around 1.000.000 tons (Eddy, 2006), however, the material has not yet been beneficiated optimally while other minerals that occurs with zircon has optimally been processed. As a by-product mineral from either gold or tin ore processing, zircon needs to be treated to get its added value (Herman, 2007).

Zircon is used for various purposes, as either metal or non metal minerals. Raja et al. (2007) stated that in the world market, the usage of zircon is mostly for foundry, refractory, ceramics and glass as well as zirconium element. However, zircon raw materials should satisfy the specification prior to processing it to be another material such as for ceramics. Enhanced material and glaze properties are two characters of zircon used in ceramics industry (Jiandong Zhang et al. 2012). Zirconium oxide (known as zirconia), partially stabilized zirconium (PSZ) and micronized zircon are raw materials for glass and ceramics industries. For engineering and electrical ceramic making, the PSZ has been developed into (Yamagata et al., 2008):
- those with CaO and MgO components that provide ceramics with better strength and endurance compared to the conventional one (SiC, Si_3 and Al_2O_3). The material has mostly been employed for nozzles, chemical pump and machine components;
- those with Y_2O_3 component. The produced ceramics are more stable and

retain low conductivity and longer durability. Such ceramics are good for a component of adiabatic diesel machine such as cylinder liner, piston head and valve. PSZ utility is also used for electrode making to control the ratio between fuel and oxide in the machine. Those PSZ functions relate to environment problems, fuel savings and machine efficiency.

Enamel frit is one of zircon flour usages. The word "frit" comes from Italian language. It refers to a ceramic composition that has been fused in a special fusing oven, quenched to form a glass and granulated. Figure 1 shows enamel frit from zirconia used for ceramics. As an enamel frit, zircon flour is employed to line the metal either steel or cast iron. Zircon flour in ceramics white and color serves as opacifier glazes due to its high refraction index. It replaces Sn-oxide to yield high quality of ceramics, notably the ceramics for table ware and tile ceramics (Poernomo, 2012).

Figure 1. Zirconia enamel frit, a material made by fusing powdered zirconia between 750 and 850 ^{o}C (www.alibaba.com).

Commercial AZS (alumina-zirconia-silica) refractory and zirconia mullite are a good alternative as refractory materials for the glass industry as they can support the aggressive conditions during liquid processing of glass. The materials are also used for lining steel smelters. Both AZS and zirconia mullite retain excellent properties in neutrality and endurance on a sudden heat. The zirconia mullite is used in a bar or nodule form and arranged regularly. Zirconia

refractory can be used through 3.600^{o}F as a ladle brick (Harben, 1995).

Zircon is a proper material for foundry due to its several good properties. Such properties include:
- very high heat conductor. As a result, it has a faster cooling process compared to quartz-based foundry;
- not reactive to other metals;
- clean-surface, spherical form. This character is appropriate for all binder type;
- less binder material;
- stable grain size though heat escalation occur;
- neutral pH.

As foundry sand, it is used to get molded products that have smooth surface. In addition to zircon material, the fused zirconia is continually employed for glass molding. Zircon sand is also beneficiated for abrasive material after the sand is processed into zirconia alumina (Priyono and Febrianto, 2012). Based on its zirconia percentage, the zircon-based abrasive material belongs to:
- AZ-abrasive that contains 25% zirconia. It is mostly applies to construction materials from metal such as steel billet, automotive etc.;
- NZ-abrasive that consists of 40% zirconia. The material includes E347 (bonded abrasive) and E349 (coated abrasive). The application of NZ-abrasive is mainly for a grinding wheel or a bit in rock-ornament cutting machine;
- sandblast replacing the quartz sand.
- Zircon consumption for various purposes is shown in Figure 2. Ceramics dominates such consumption.

METHODOLOGY

Material used in this study came from Popay of Nanga Pinoh, West Kalimantan. Characterization of the samples including sieving and chemical analyses as well as mineralogical examination was conducted using a series of sieve, AAS and XRF as well as XRD, optical microscope and SEM. Separating the zircon from its gangue, notably iron was used a magnetic separator.

Yet the material was concentrated by tabling procedure and then milled to get zircon flour (http://www.zirox.co.in/zircon_sand.htm accessed on 26 February 2015 at 2.00 pm). Figure 3 illustrates the flow chart for zircon study.

Tabling is conducted to separate light- and heavy minerals that based on their specific gravity (Falconer, 2003). The objective is to get zircon concentrate, middling and tailing materials. Condition for operating tabling includes feeding rate (250 g/minute), tabling slope (2.0^o), wash water (14 l/minute), % solid (15%) and length of stroke (0.75"). The table for processing zircon belongs to Wiffley type (Figure 4), however, the process will provide a good result if the processed particle size is less than 65 mesh or 0.25 mm and satisfies the prerequisite as follows:

$$\frac{\rho_{hvm.} - \rho_{w.}}{\rho_{lght.} - \rho_{w.}} \geq 2,5$$

where $\rho_{hvm.}$: density of heavy mineral, $\rho_{lght.}$: density of light mineral and $\rho_{w.}$: density of water.

Tabling performance is affected by riffle, deck coating material, head motion mechanism, feeding method, frequency. Slope, % solid, feed, wash water and the desired products are variables that can be adjusted any time when operating shaking table for processing the material.

Magnetic separation is accomplished to separate magnetic- and non-magnetic materials. Magnetite, ilmenite, garnet and monazite are magnetic minerals that are commonly associated within zircon sand while rutile, zircon and quartz belong to non-magnetic minerals available within the sand. The process of magnetic separation for this study consists of three steps. Step1 separates minerals that retain strong magnetic character such as magnetite (Fe_3O_4) from non-magnetic ones. Using magnetic separator in a dry condition (Figure 5), the used magnetic intensity for Step1 is 1.200 Gauss. The non-magnetic mineral from Step 1 serves as a feed for magnetic separation Step 2.
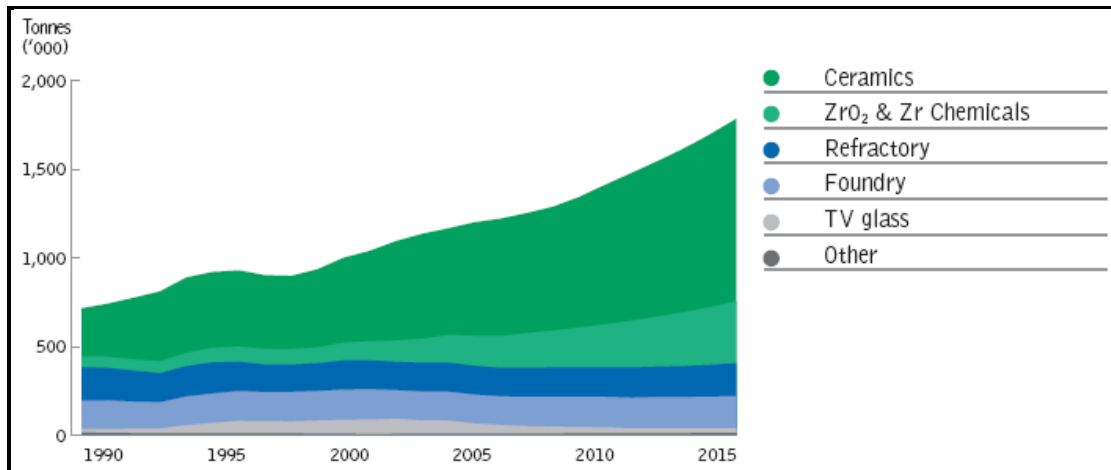
Figure 2. Zircon consumption for various purposes starts from 1990 to 2015. Ceramics dominates the consumption (Purnomo, 2012)
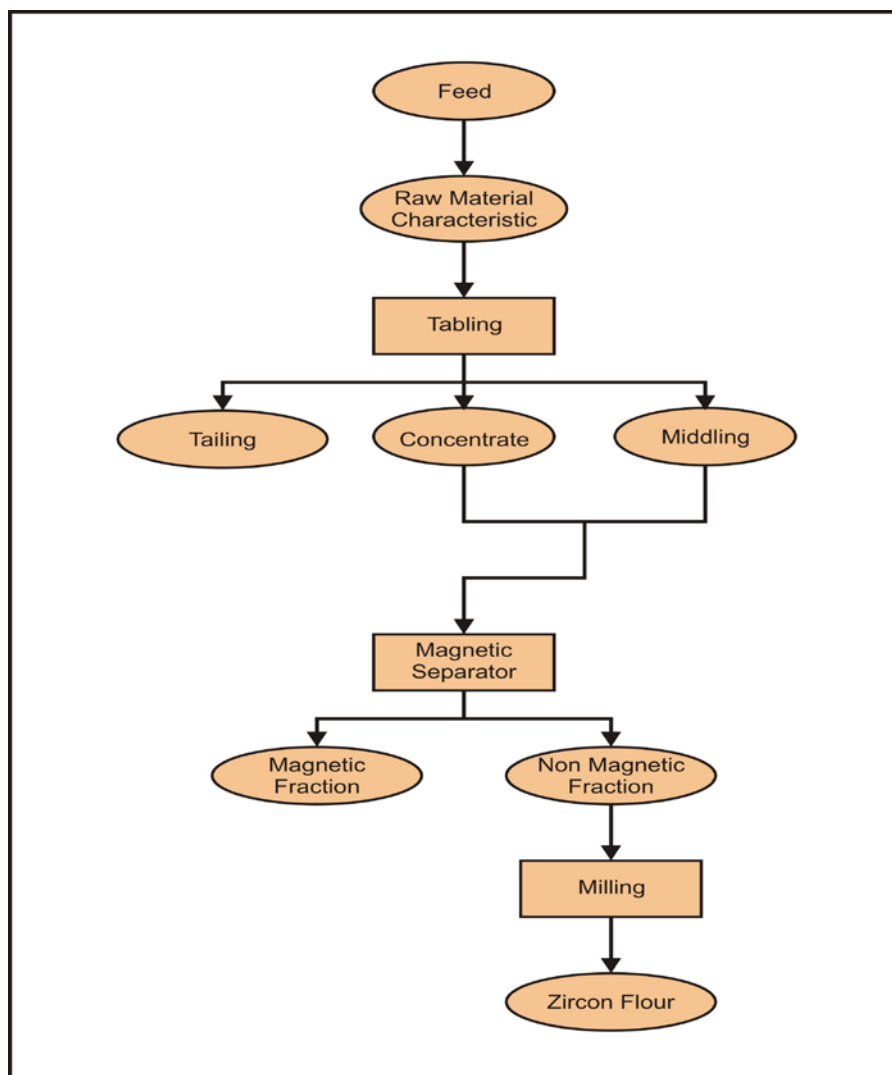

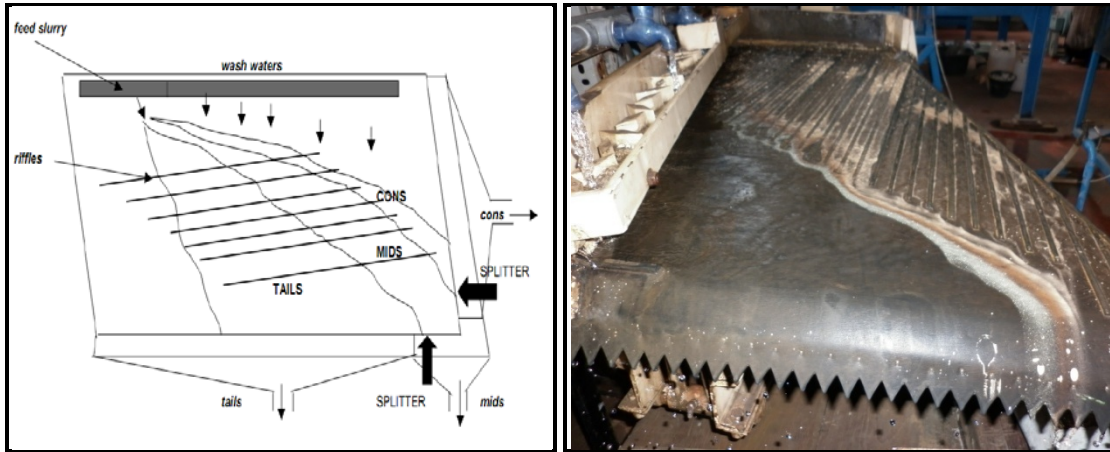Figure 3. A flow chart showing the activities of zircon flour making

Figure 4. Wiffley Table used for processing zircon materials at laboratory scale
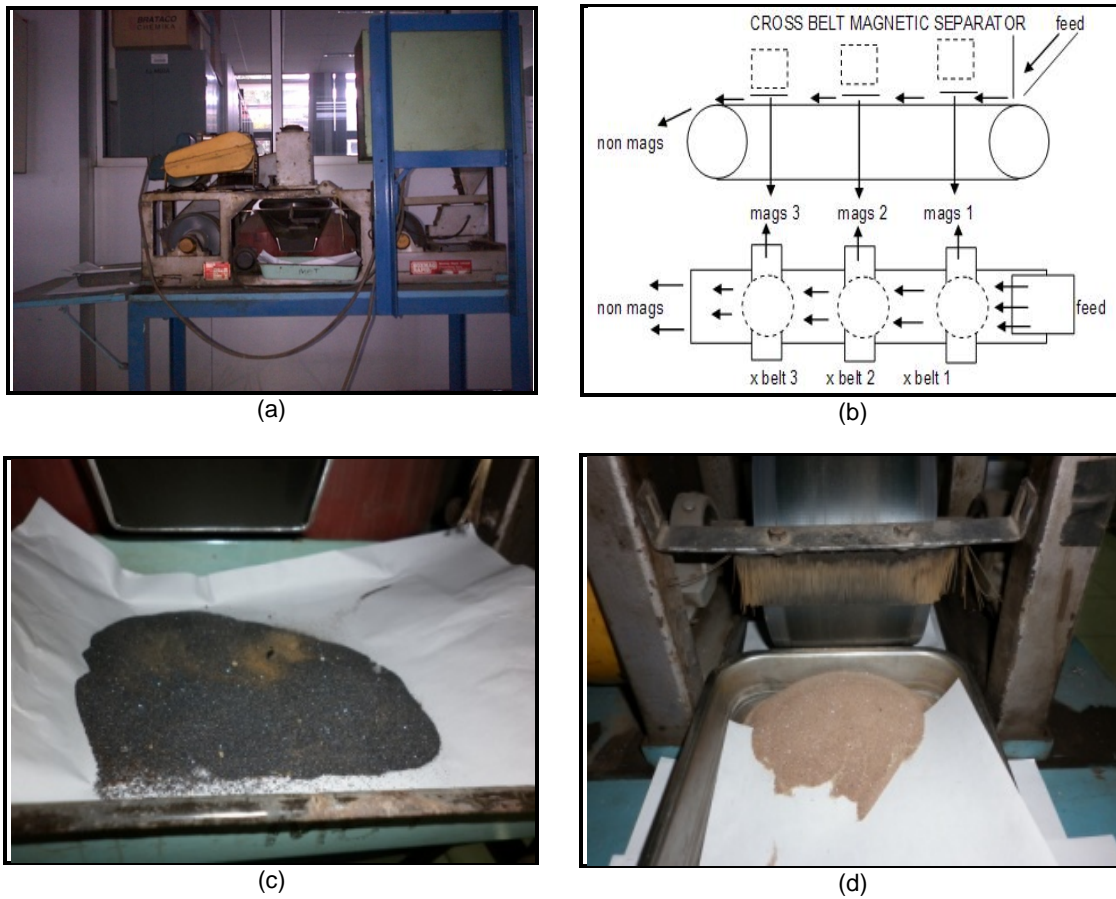

(a)

(b)

(c)

(d)

Figure 5. Magnetic separator (a) used for separating the zircon from its association; scheme of the applied instrument (b); magnetic and non magnetic fractions results from magnetic separator (c) and (d).

This step utilizes magnetic intensity of 9.000 Gauss and intends to get less magnetic minerals, such as ilmenite ($FeTiO_3$) as well as non-magnetic mineral, namely zircon and rutile. Utilizing step 2-tailing as the feed and magnetic intensity of 13.000 Gauss, Step 3 intends to increase the available zircon content within feed by splitting weak, magnetic minerals and non-magnetic ones.

RESULTS AND DISCUSSION

Zircon sand for this study was sampled from Popay Block of Nanga Pinoh District, West Kalimantan. Sampling area belonged to Selatan Jaya Melawi, Ltd. Sampling method included test pitting and trenching systems. The sampled material was then sun-dried and packed in a 30-kg sack to be sent to Bandung for further processing. Figure 6 shows sampling activity at Nanga Pinoh.

The objective of sample preparation is to obtain representative samples in both particular quantity and size. Sample was then sun-dried to get homogeny dry samples and then divided into several sample bags for laboratory analyses and processing experiments. Laboratory analyses for chemical composition of the zircon sand was accomplished by AAS and XRF. The former was focused to the main composition available within the sample while the later tried to search whether zircon samples also comprised rare earth elements. Table 1 shows results of chemical analyses of zircon samples using AAS and XRF. Chemical analyses by both AAS and XRF showed that the ZrO_2 content within samples is bigger than 40% while the impurities (SiO_2 and TiO_2) are bigger than 20 and 10%. Iron impurity detected by AAS is quite high (8.03%), however, the detected iron came from XRF analysis is less than 1 %. The fact

that such iron detection by XRF instrument in the form of iron oxides (not the total iron) is supposed to be the cause of low iron in XRF analysis. Of the 17-REEs, only yttrium (Y) was found within Popay zircon sand. Its quantity was quite significant, namely around 0.29% or 2.900 ppm. However, no other REEs were found within the sand. Normally, to evaluate the existence of REEs within samples entirely, the zircon sands were better subjected to ICP-OES analysis to assess REE abundance compared to XRF analyses (Routray and Rao, 2011). However, such an instrument at Chemistry Laboratory of RDCMCT had not yet been operated due to some reasons. Scarcity of REEs within samples was supposed to be inhomogeneity of the sample that occurred during sampling in the field.

Zircon flour industry requires the ZrO_2 content within the zircon sand >65%. However, the ZrO_2 contents, analyzed by AAS and XRF, are only 43.54 and 44.05%%. It means that the Popay zircon sand is unqualified for zircon flour making as the requirement for making the flour, the sand should have ZrO_2 content above 65%. To fulfill such a requirement, the zircon sand needs to be improved by separating the ZrO_2 from its impurities using magnetic separator and tabling. No radioactive minerals detected by XRF instrument (Pownceby et al., 2015).

(a)   (b)

Figure 6. Sampling activity for zircon sand at Nanga Pinoh; Channeling procedure to get zircon sand (a) and pre-sieving to separate coarse and fine sand (b)

Table 1. Chemical composition of the zircon samples, result from AAS (a) and XRF (b) analyses

Chemical composition of the zircon sand, detected by AAS	
Detected elements	Quantity(%)
ZrO_2	43.54
SiO_2	23.90
$Fe_{(total)}$	8.03
TiO_2	11.72
Chemical composition of the zircon sand, detected by XRF	
Detected elements	Quantity(%)
ZrO_2	44.05
SiO_2	21.89
HfO_2	0.88
Al_2O_3	0.25
Fe_2O_3	0.60
Fe_3O_4	0.33
CaO	0.14
K_2O	0.028
TiO_2	10.10
P_2O_5	0.07
Y_2O_3	0.29
LOI	9.55

Sieving analysis was conducted to evaluate size distribution of the sample. Sieving equipments were set into fraction of 25, 60, 80,120 and 200 meshes. The results showed that Popay zircon sand was dominated by coarse to medium size fractions, namely fractions of 25, 60, 80, 120 meshes (Table 2). The weight percent of such fractions is between 10.20 – 35.66% and fraction of -80+120 acquired the highest percentage, namely 35.56%. The finest fractions (-120+200 and -200 meshes) provided the least weight percentages, namely 1.11 - 1.20%. The fact that sampling the sand was accomplished on the surface was supposed as the cause of low quantity at such sizes. As young alluvial, the zircon silicate was still restrained to a surface. Zircon was dominant at size -60+80 mesh performing quantity of 85.04 while size +25 and -25+60 meshes retained quantities of 76.00 and 72.15%. Its content decreased at -80+120, 120+200 and -200 mesh fractions namely around 3.46, 12.30 and 10.30%. Referring to sieving analysis result, the highest weight percent (35.66%) occurred at fraction of -80+120 mesh though its ZrO_2 content was 3.46%. Fraction of -60+80 mesh obtained the highest ZrO_2 content but its percent weight was less than that of -80+120 mesh. Such a condition

had actually satisfied the requirement for gravity concentration but the processed employed the fraction of -80+120 mesh as the feed in consideration that the related feed would provide more ZrO_2 content due to its bigger weight percent.

Quartz and iron minerals were separated by tabling and magnetic separator equipment respectively. SiO_2 as a gangue mineral was available in all fractions but fraction -80+120 mesh retained the biggest quantity, namely 68.70%. The finest fraction (- 200 mesh) preserved a quantity of 2.01%. The quantity of another gangue mineral such as iron mineral, varied from one fraction to another though its variation was only 0.13 - 1.60%. Titanium oxide that was supposed to be come from rutile was also detected within zircon samples. Its percentage was around 0.88 - 5.97%. Fraction of -25+60 mesh performed smallest content, namely 0.88% and fraction of -120+200 mesh had the highest quantity (5.97%). Of all fractions, the highest TiO_2 content belonged to fraction of -80+120 mesh, namely 5.97%.

Mineralogical analysis of Popay zircon of Nanga Pinoh, West Kalimantan was accomplished using optical microscope,

scanning electron microscope and XRD instruments. The analysis was also intended to evaluate mineral liberation performance within samples. Therefore, the samples were sieved and divided into five fractions, namely +25, 25+60, -60+80,-80+120 and -120+200 meshes. At least, 6 minerals were identified within the samples (Figure 7). Those were zircon, quartz, magnetite, hematite, ilmenite and rutile. Of the six-detected minerals, zircon, magnetite and quartz were the most dominant minerals. Their existence were found within every fraction except for quartz at fraction -80+120 mesh, however, it did not mean that the quartz did not exist within such a fraction. Referring to sieving analysis as shown in Table 2, quartz was the dominant one. It is supposed that when making a specimen for optical microscope test, only a few quartz grains were taken and the grain was not detected by the microscope. Popay zircon was liberated at all fractions, from +25 to -60+80 meshes (Table 2). Almost 97.08 to 99.90% minerals were liberated. Referring to such a condition, it seemed that the zircon mineral was easy to separate by gravity concentration and magnetic separator. This suggestion was supported by the fact that of all fractions, fraction -60+80 mesh had the most zircon quantity (around 85.05%) and less impurities even magnetite was absent within such a fraction.
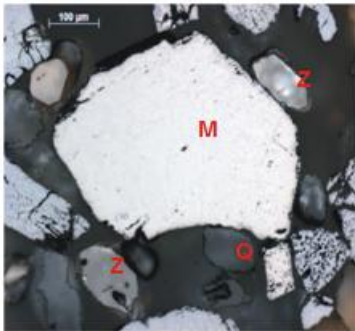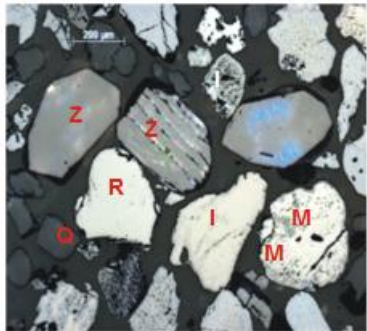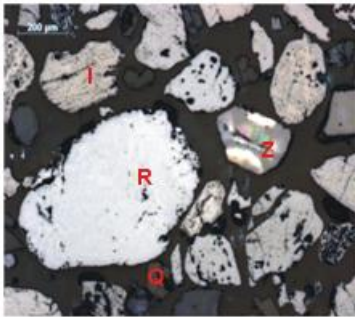
Fraction (mesh)	Detected mineral	Fraction (mesh)	Detected mineral
+25		-60+80	
-25+60		-80+120	
-120+200			

Figure 7. Minerals available within Popay zircon sand of Nanga Pinoh: zircon (Z), magnetite (M), quartz (Q), ilmenite (I), rutile (R) and hematite (H)

Table 2. Result of sieving analysis for Popay zircon sand, performing the quantities of each fraction and mineral distribution

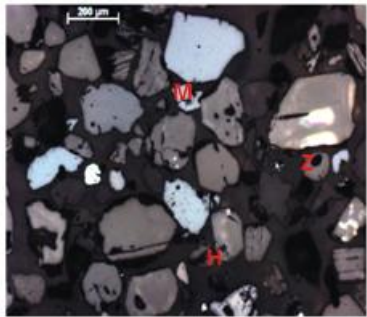| Fraction (mesh)	Quantity (%)	Mineral composition (%)						Liberation degree (%)
		Zircon	Ilmenite	Magnetite	Hematite	Rutile	Quartz	
25	10.20	76.00	5.45	1.85	0.90	1.80	10.10	97.08
35	18.45	72.15	4.60	1.50	0.35	0.46	8.52	98.04
20	33.38	85.04	0.87	nd	0.40	0.50	0.50	99.00
40	35.66	3.46	0.75	0.22	0.71	1.92	60.40	99.01
80	1.11	12.30	1.10	0.81	0.28	nd	40.62	99.90
-200	1.20	10.30	1.00	0.50	0.35	nd	35.50	99.50
Total	100	-	-	-	-	-	-	-

The purpose of tabling is separating the heavy and light minerals based on their density differences (Wills and Munn, 2006). Tabling produces concentrate, middling and tailing while magnetic separator splits the minerals based on their magnetic properties and yields magnetic and non magnetic materials. Tabling had been conducted to Popay zircon sand using tabling equipment Wilfley type. Of the three-tabling products, around 80.77% belonged to middling material that contained 44.50% ZrO_2, 12.81% went to concentrate that performed 38.88% ZrO_2 and the rest (6.42%) went to tailing that comprised 40.81% ZrO_2. The feed used for magnetic separator was the mixture between concentrate (4.68%) and middling (88.90%) from tabling process. Total weight of the mixture was 93.58% and its chemical composition consisted of ZrO_2 = 43.73%. Fe $_{total}$ = 7.92 %. TiO_2= 10.85 % and SiO_2 = 24.00%. Recovery calculation suggested that ZrO_2 = 93.98%. TiO_2 = 93.99% and concentration ratio = 0.93.

When conducting magnetic separation at Step1, the process used magnetic intensity of 1.200 Gauss. The result showed that magnetic concentrate of the process retained composition of 16.11% ZrO_2, 29.93% Fe total, 25.68% TiO_2 and 8.73% SiO_2 while the non magnetic fraction comprised 45.18% ZrO_2, 6.76% Fe total, 10.07% TiO_2 and 24.81% SiO_2. Table 3 illustrated the recovery of the process at Step1. Non magnetic fraction possessed the highest ZrO_2 recovery performing the content of 29.93% but recoveries of TiO_2 and Fe were also relatively high though their content was around 25.93 and 25.68%. This fact seemed a motivation

to re-process the non magnetic fraction from Step1 using magnetic separator with magnetic intensity of 9.000 Gauss. The results detected that the magnetic fraction belonged to ilmenite concentrates and retained weight of 22.70%. The fraction consisted of 19.48% ZrO_2, 22.30% Fe total and 35.19% TiO_2 as well as 9.69% SiO_2. The recovery of ilmenite from magnetic fraction was 89.23% while the ZrO_2 was 11.01%. The non magnetic fraction recovered 10.07% TiO_2 and 88.99% ZrO_2.

Table 3. The achieved recoveries of magnetic separation Step 1, using magnetic intensity of 1.200 Gauss.

Recovery of	Magnetic fraction (%)	Non magnetic fraction (%)
TiO_2	11.83	88.17
Fe	18.89	81.11
ZrO_2	1.84	98.16

A magnetic separation was also completed to the non magnetic fraction from Step2 as the feed. In terms of evaluating how many zircon concentrates would be gained from this step (known as Step 3), the applied magnetic intensity within the process was 13.000 Gauss. Table 4 summarized all activities from tabling to the last magnetic separation. The yielded weight of non magnetic fraction in Step 3 was 35.76%. It consisted of ZrO_2 (65.50%), Fe total (0.08%), TiO_2 (1.85%) and SiO_2 (33,13%). Step 3 showed the recovery of magnetic fraction as many as 31.74% for TiO_2 and 44.46% for ZrO_2 while the non magnetic

fraction of Stepperformed TiO_2 recovery of 68.41% and ZrO_2 recovery of 65.53%. Referring to the requirement for zircon flour making that should meet the demand of >65% , the ZrO_2 from non magnetic fraction at Step3 had been satisfied such a requirement (Serena Righi et al, 2005).

Three procedures were conducted to magnetic fraction of Step1, 2 and 3 as well as non magnetic fraction of Step3. Those were XRD, chemical and optical microscope analyses. The purpose of this step was to understand the associated minerals within the zircon sand as well as its composition. The results were illustrated in Table 5, 6 and 7. XRD analysis identified five minerals

available within the analyzed samples (Table 5). Those are magnetite, ilmenite, zircon and rutile. Based on XRD diffractogram, zircon was the dominant mineral at either magnetic or non magnetic fractions.

Analyzing four feed samples by AAS showed that ZrO_2 is the dominant oxide within non magnetic sample (Table 6). Its quantity was 65.50%; however, the ZrO_2 content was insignificant within samples Magnetic 1200 G and Magnetic 9000 G. This condition was similar to SiO_2 content at which the Magnetic 1200 G and Magnetic 9000 G retained minor quantities but relatively significant ones in Magnetic 13000 G and Non- magnetic samples.

Table 4.   Result of zircon sand separation using tabling and magnetic separator

Feed materials and Products	% weight	Composition (%)				Distribution (%)			
		ZrO_2	Fe_{total}	TiO_2	SiO_2	ZrO_2	Fe_{total}	TiO_2	SiO_2
Tabling									
Feed (Original zircon sand sample)	100.00	43.54	8.03	11.00	23.90	100	100	100	100
Concentrate	12.81	38.88	4.85	19.99	21.07	11.44	7.74	23.27	11.29
Middling	80.77	44.50	8.41	9.40	24.47	82.54	84.56	69.00	82.70
Tailing	6.42	40.81	9.63	13.24	22.42	6.02	7.70	7.73	6.02
Mag. sep. I (1,200 G)									
Feed (mixture of concentrate-middling from tabling)	93.58	43.73	7.92	10.85	24.00	93.98	92.30	92.27	93.99
Magnetic I	4.68	16.11	29.93	25.68	8.73	1.73	17.44	10.92	1.71
Non magnetic I	88.90	45.18	6.76	10.07	24.81	92.25	74.86	81.35	92.28
Mag. sep. II (9,000 G)									
Feed (non magnetic I)	88.90	45.18	6.76	10.07	24.81	92.25	74.86	81.35	92.28
Magnetic II	22.70	19.48	22.30	35.19	9.69	10.16	63.03	72.59	9.20
Non magnetic II	66.20	54.00	1.43	1.46	29.99	82.10	11.83	8.77	83.08
Mag. sep. III (13,000 G)									
Feed (non magnetic II)	66.20	54.00	1.43	1.46	29.99	82.10	11.83	8.77	83.08
Magnetic III	30.44	52.20	3.02	1.00	26.30	36.50	11.47	2.76	33.50
Non magnetic III	35.76	65.50	0.08	1.85	33.13	53.80	0.36	6.00	49.58

Table 5. Mineralogy of magnetic separator feed analyzed by XRD

Sample code	Identified mineral
Magnetic 1200 G	Magnetite, ilmenite, zircon
Magnetic 9000 G	Ilmenite, rutile, zircon, quartz
Magnetic 13000 G	Magnetite, Ilmenite, Zircon
Non- magnetic	Zircon, quartz

Table 6. Chemical composition of magnetic separator feed analyzed by AAS

Feed type	Chemical composition (%)			
	ZrO_2	Fe_{total}	TiO_2	SiO_2
Magnetic 1200 G	16.11	29.93	25.68	8.73
Magnetic 9000 G	19.48	22.30	35.19	9.69
Magnetic 13000 G	52.20	3.02	1.00	26.30
Non magnetic	65.50	0.08	1.85	33.13

Minerals identified by optical microscope include zircon, ilmenite, rutile, pyrite, cassiterite, hematite, magnetite-hematite, limonite, tourmaline and quartz (Table 7). The availability of such minerals within each sample is not the same. For example; rutile, pyrite, tourmaline and quartz are absent in magnetic fraction of 1200 G. Of the five mentioned minerals, only pyrite is available in magnetic fraction of 9000 G others are not. Quartz occurred in magnetic fraction of 13000 G and non-magnetic fraction of 13000 G. It can be seen that the higher the magnetic intensity for separating process the lesser the magnetic minerals such as hematite, magnetite-hematite and limonite. Though ilmenite, pyrite and hematite-magnetite belong to magnetic minerals, their presence in Non magnetic fraction of 13000 G is not quite understandable. Such minerals are supposed to be attached to the non magnetic one and then pass to the non magnetic part. Lessening the magnetic minerals in non magnetic section can be accomplished by size reduction of the feed to free the attached minerals.

Previous process to concentrate the sand into magnetic and non magnetic minerals was conducted by tabling and then magnetic separation. In terms to evaluate the result, another concentration was also accomplished but in this step, magnetic separation was applied as the first step and then continued by tabling procedure. Tabling was applied to non magnetic fraction of 13000 G as the feed. The result is shown in Table 8. The ZrO_2 content within the feed (non magnetic fraction of 13000 G) is 64.52%. After tabling, the content is a little bit higher. It increases to 66.11% while its middling is 63.71%.

In terms gaining the zircon flour, milling was conducted to the product of zircon concentration using magnetic fraction of 13000 G as the feed. The used feed was 200 g and its composition comprised 65.50 %, ZrO_2, 0.08 % Fe_{total}, 1.85 % TiO_2 and 33.13% SiO_2. Milling employed ceramic ball mill and 325-mesh sieve to get the product Figure 8. Prior to milling, the color of the zircon sand was light brown but the changed to grayish white after milling. It can be seen from Figure 9 that the longer the milling time the lesser the obtained product for size +325 mesh but the bigger the quantity for size -325 mesh. Zircon recovery for size -325 mesh significantly increases during 184-minute milling time although the final result is only 50.49% for-325 mesh and 49.51% for +325 mesh. Referring to such a result, it is concluded that the milling time needs to be enhanced to 5 - 6 hours.

To maximize milling recovery, another milling process was conducted by changing the milling condition. Previously, the weight ratio of ceramic ball to zircon sample was determined 20 : 1 but the ratio for maximizing milling recovery employed three

ratios, namely 5:1, 10:1 and 20:1. Other condition is fixed milling time (3 hours) and weight of ceramic ball (2.000 g). The result is shown in Table 9 and Figure 10. Of 418 g zircon sample when milled using condition ratio of 5:1 only yields 37.32% zircon materials of -325 mesh while the retained one is 62.68%. Changing the ratio to 10:1 and 20:1 result in increasing the recovery of -325 mesh size although the used milled sample is less than previous milling process. High recovery (89.09%) is achieved at ratio of 20:1, 3-hour milling time and 2.000-g weight of milling media.

Table 7. Optical microscope analysis of material from magnetic separation process

Mineral	Mineral composition in			
	Magnetic fraction of 1200 G	Magnetic fraction of 9000 G	Magnetic fraction of 13000 G	Non magnetic fraction of 13000 G
	(wt. %)			
Zircon	22.98	33.68	97.57	94.33
Ilmenite	74.48	62.59	-	0.77
Rutile	-	-	0.56	2.68
Pyrite	-	0.49	0.29	0.77
Cassiterite	0.68	0.68	0.83	0.36
Hematite	0.49	1.00	-	-
Magnetite-Hematite	1.00	1.51	-	0.26
Limonite	0.37	-	-	-
Tourmaline	-	-	-	0.33
Quartz	-	-	0.47	0.54

Table 8. Result of separating the Popay zircon sand by magnetic separator and tabling

| Feed materials and Products	% weight	Composition (%)				Distribution (%)			
		ZrO_2	Fe_{total}	TiO_2	SiO_2	ZrO_2	Fe_{total}	TiO_2	SiO_2
Mag-Sep I (1200 G)									
Feed (original zircon)	100.00	43.54	8.03	11.00	23.90	100	100	100	100
Magnetic I	4.58	15.21	42.82	2.87	20.98	1.60	24.42	1.20	4.02
Non magnetic I	95.42	44.90	6.36	11.39	24.04	98.40	75.58	98.80	95.98
Mag-Sep II (13000 G)									
Feed (non magnetic I)									
Magnetic II	33.26	8.23	17.18	30.58	9.16	6.29	71.16	92.47	12.75
Non magnetic II	62.16	64.52	0.57	1.12	32.00	92.11	4.41	6.33	83.23
Tabling									
Feed (non magnetic II)	62.16	64.52	0.57	1.12	32.00	92.11	4.41	6.33	83.23
Concentrate	36.82	66.11	0.22	0.42	31.80	55.91	1.01	1.41	48.99
Middling	24.41	63.71	1.05	0.87	31.80	35.72	3.19	1.93	32.48
Tailing	0.93	22.83	1.83	35.40	45.17	0.49	0.21	2.99	1.76
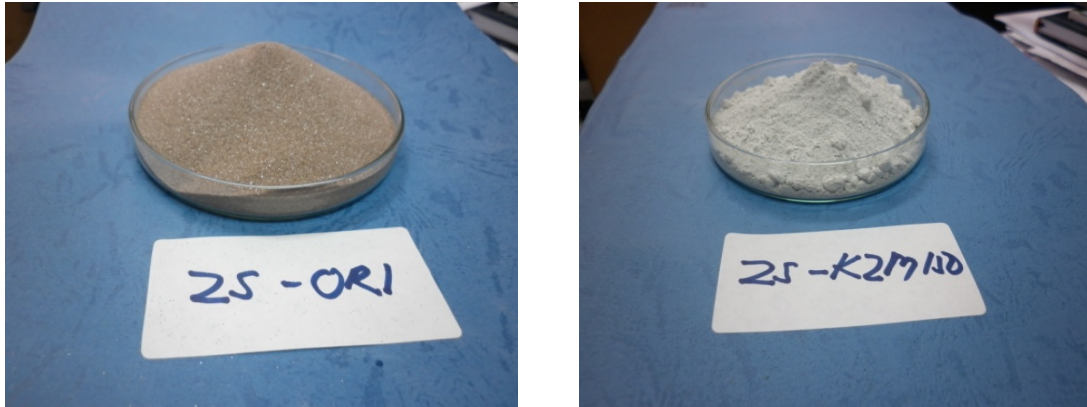
Figure 8.   Original zircon sand prior to milling performing light brown in color (left) and grayish white zircon flour size -325#
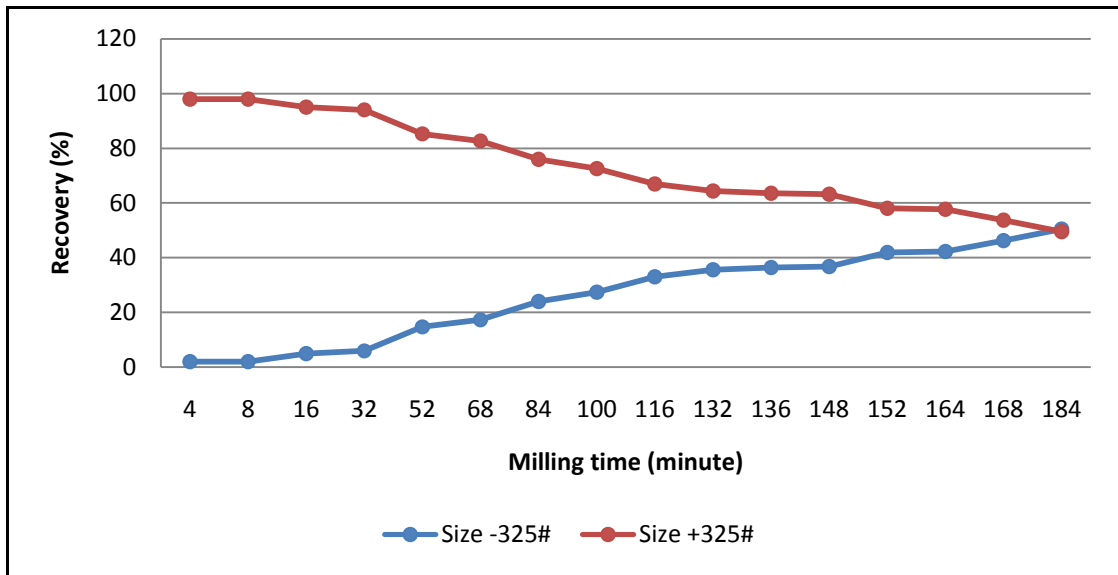

Figure 9.   Result of zircon sand milling. The weight of milling media and ratio of ceramic ball to zircon sample was made permanently, namely 2.000 g and 10 : 1

Table 9.   Result of zircon milling using several ratios and 3-hour milling time 2.000-g weight of milling media

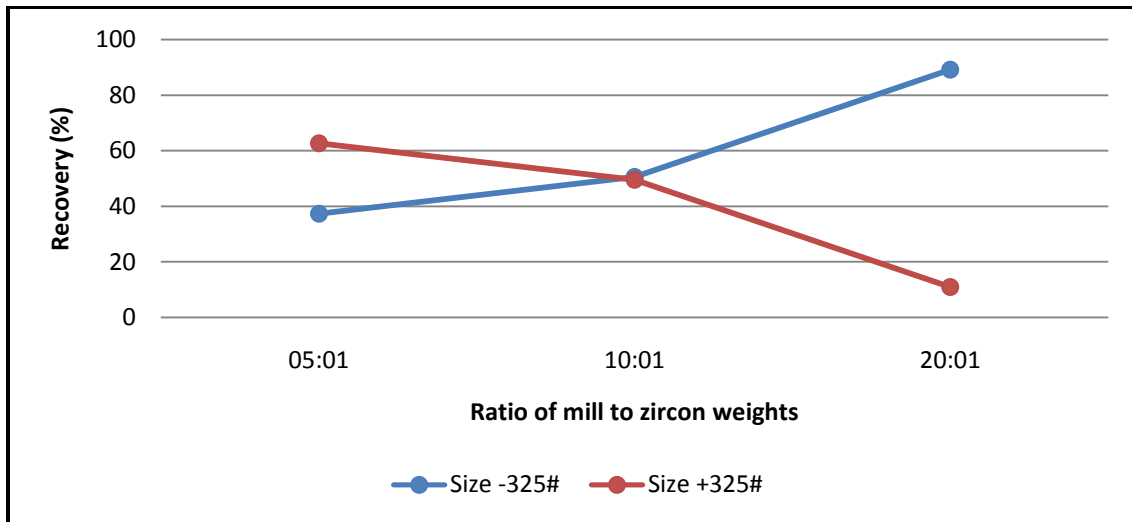| Ratio of milling media to zircon weights	Sample weight (g)	Fraction of			
		+325 #		-325 #	
		(g)	%	(g)	%
5:1	418	262	62,68	156	37,32
10:1	202	100	49,50	102	50,50
20:1	110	12	10,91	98	89,09

Figure 10.   Result of zircon sand milling. The process used three different weight ratios, namely 5:1, 10:1 and 20:1

The milling product was also analyzed by scanning electron microscope-energy dispersive spectroscopy (SEM-EDS analysis). The method used in this analysis was X-ray mapping. Photomicrograph of the sample (Figure 11) shows that zircon mineral still perform its tetragonal crystal structure. The detected elements within sample include Zr (45.11%), Si (18.26%) and Mg (0.72%). The presence of Mg is supposed to be one of zircon impurities that normally trigger the color to the mineral (Kaur and Pand, 2010). Other impurity elements such as iron, manganese, calcium, sodium, titanium that normally contribute several colors (clear white - greenish yellow, reddish brown, brownish yellow and dark) were not detected within this specimen. Titanium along with iron was detected from chemical analysis. SEM-EDS analysis also proved that the REEs were not available within Popay zircon sand. A research by Routray and Rao (2011) on characterization of detritus zircons of Orissa, India showed that the REEs were commonly found in the red sediment instead of the beach-dune sand. It seemed that the light color sand retained less REEs as the study sand from Popay. Scarcity of REEs within samples was

supposed to be sample inhomogeneity thatoccurred during sampling in the field or preparing SEM specimen for SEM-EDS analyses. Chemical analysis of the same sample also detected Zr and Si as the main elements within such a sample. Table 10 shows chemical composition of zircon flour size -325 meshes. The zircon has achieved high purity level (66.02%) - less than 1.38% from the pure zircon content (67.40%) while the detected impurity (TiO_2) is 1.51% (Lubbe et al, 2012). The fact that size for feeding the mill was -80+120 mesh is supposed to be the cause of the impurity existence as in such a fraction, titanium-bearing mineral - ilmenite or rutile - has not completely been liberated yet.

Tabel 10. Chemical composition of zircon flour size -325 mesh analyzed by AAS.

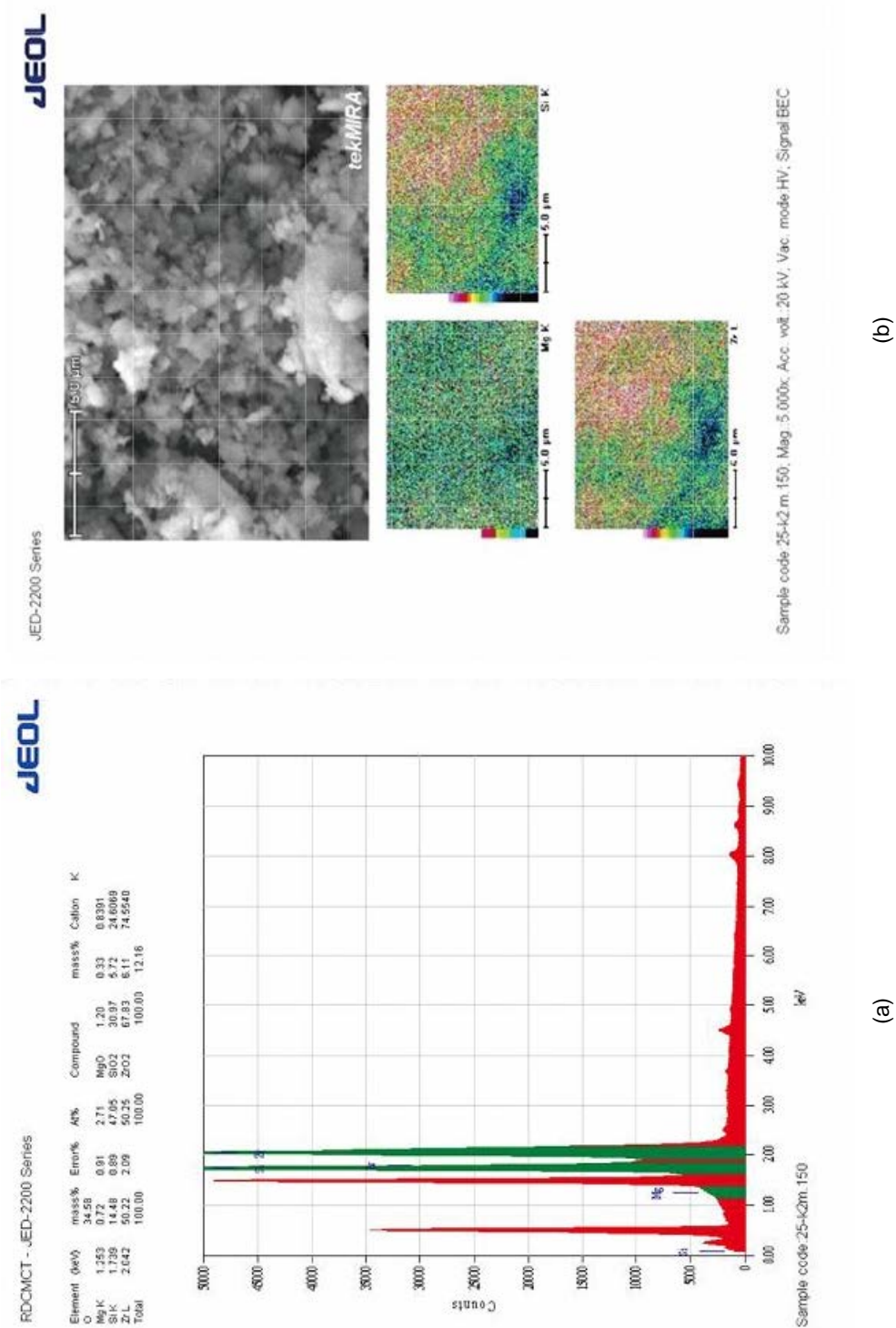| Detected materials	Quantity (%)
ZrO_2	66,02
SiO_2	35,4
Fe (total)	0,05
TiO_2	1,51

(b)

(a)

Figure 11. SEM-EDS analysis of the zircon flour showing tetragonal crystal structure (a).
Mapping of the specimen detected three elements, namely Zr, Si and Mg (a) and (b).
Mg is supposed to be one of zircon impurities that causes color to the mineral

CONCLUSION AND SUGGESTIONS

Conclusion

Using gravity concentration (tabling) and magnetic separator, the content of Popay zircon could be upgraded from 43.54 to 65.50%. However, the ZrO_2 content would be bigger than that of the previous one if the first process was magnetic separator and then followed by tabling. The content increased to 66.11%. Referring to the requirement for zircon flour making, zircon that has content bigger than 65% categorized as the premium one and could be applied for lining the furnace in steel and ceramics making and foundry as well.

The fact that the tabling slope was only 2 degrees instead of 3 degrees affected the yielded zircon. Using such a figure, the process was only capable to increase the content from 43.54 to 43.75% though its recovery was up to 93.58%. Popay zircon had also satisfied specification of enamel frit to be used in ceramics in terms of providing high quality ceramics. When in the flour form, the material also met with the specification for ceramics industries.

Suggestions

The fact that the processed zircon sand had varying size and resulted 3% of the material did not pass the 65-mesh sieve required addition process to separate the coarse and fine materials. If the addition process is conducted, this will provide effective results for tabling and magnetic separation procedures

Referring to the result of magnetic separator using Magnetic fraction of 1.200 G feed that performs high titanium oxide (25.68%), it is suggested using lower magnetic intensity than that of 1.200 G, namely between 800 and 1.000 Gauss. Using such figures, it is expected that the recovery of rutile (TiO_2) and other magnetic materials will increase.

When separating magnetic - non magnetic materials from Magnetic fraction of 9000 G as the feed, its concentrate was separated utilizing magnetic intensity of 13.000 Gauss. This step yielded non magnetic fraction containing ZrO_2 around 65.50% while the ZrO_2 in magnetic fraction was around 52.20%. The zircon content within magnetic fraction can be upgraded if re-processed using high tension separator (HTS) to get higher zircon as well as ilmenite and rutile concentrates.

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