INDONESIAN MINING JOURNAL Vol. 21, No. 2, October 2018 : 99 - 112

THE NATURES OF ZINC SULFIDE CONCENTRATES AND ITS BEHAVIOR AFTER ROASTING PROCESS

KARAKTER KONSENTRAT SENG SULFIDA DAN PERUBAHANNYA SETELAH PROSES PEMANGGANGAN

DESSY AMALIA[1], TATANG WAHYUDI[1] and YUHELDA DAHLAN[2]
[1] R&D Centre for Mineral and Coal Technology
Jalan Jenderal Sudirman 623 Bandung 40211
Ph. (+6222) 6030483, Fax. (+6222) 6003373
e-mail: dessyamalia79@gmail.com
[2] Pusat Survei Geologi, Badan Geologi
Jalan Ponegoro No. 57 Bandung

ABSTRACT

The sample used for this study was a sulfide flotation concentrate that came from PT Lumbung Mineral Sentosa. The phase changes that occur in the particles of zinc sulfide concentrate during roasting in a muffle furnace were investigated using light microscopy, X-ray diffraction (XRD), and Scanning Electron Microscopy (SEM) with Energy Dispersive Spectroscopy (EDS) while its chemical composition was analyzed using atomic absorption spectroscopy (AAS) and X-ray fluorescence (XRF) methods. Characterization also employed DTA-TGA instrument to provide data on the transformations that have occurred while the TGA data presents information about physical and chemical phenomena. Mineral composition of the sample included sphalerite, galena, chalcopyrite, and pyrite. Sphalerite was the most dominant one. Roasting sphalerite samples was intended to release the sulfur from its sulfide, Such a release was made easier for further treatment of the zinc such as a leaching process. Roasting temperature varied from 200-1,100°C. Sphalerite starts to change when the temperature was raised to 650°C performing the formation of zincite, franklinite, quartz, and sphalerite as well. This composition did not change although the temperature increased to 1,100°C. The Zn content within the original sample was 59.00% and then increased with the increasing of temperature but the improved in line with the increased temperature. The highest Zn was 78.98% achieved at 1,100°C.

Keywords: concentrate, zinc sulfide, sphalerite, galena, chalcopyrite

ABSTRAK

Percontoh yang digunakan pada studi ini adalah konsentrat sulfida dari PT Lumbung Mineral Sentosa. Konsentrat berasal dari flotasi mineral seng sulfida dengan komposisi mineral sfalerit, galena, kalkopirit dan pirit. Pemanggangan sfalerit ditujukan untuk melepaskan belerang dari bentuk sulfidanya agar logam seng yang terkandung di dalamnya mudah untuk diproses selanjutnya, misalnya dengan pelindian. Variasi temperature untuk pemanggangan divariasikan antara 200 - 1.100 °C. Perubahan sfalerit jadi mineral lain terjadi pada suhu 650 °C dengan terbentuknya zingsit, franklinit dan sfalerit. Komposisi ini tetap tidak berubah walaupun temperatur dinaikkan sampai 1.100 °C. Kandungan seng pada percontoh asli adalah 59,00% kemudian bertambah seiring dengan meningkatnya temperatur. Kandungan seng tertinggi adalah 79,98% yang dicapai pada saat suhu 1.100 °C.

Kata kunci: konsentrat, seng sulfida, sfalerit, galena, kalkopirit

Received: 05 February 2018, first revision: 14 May 2018, second revision: 17 July 2018, accepted: 24 October 2018.
DOI: 10.30556/imj.Vol21.No2.2018.698

INTRODUCTION

In nature, zinc can be found in sulfides, oxides, biotite and ferromagnesian minerals such as olivine and orthopyroxene (Stanton, 1994). The element comprises an atomic number of 30 as the first element within Group 12th of the periodic table. Its application includes galvanizing (55%), alloys (21%), brass and bronze (16%), miscellaneous (8%). Galvanizing is coating the iron or steel to protect them against corrosion (McLeod, 2017). Zhang *et al.* (2014) states that the element was applied in automotive, construction, marine, and battery as well as light industries. Moreover, the metal is more reactive than the iron (BBC, 2016).

Zinc is usually produced from sulfides (sphalerite – ZnS), oxide-carbonate zinc ores and secondary materials such as zinc ash, zinc dross, flue dust of the electric arc furnace, leach residues (Baba and Adekola, 2011). The primary product of most zinc companies is slab zinc, which is produced in 5 grades: special high-grade, high-grade, intermediate, brass special, and prime western. The zinc ores typically may contain from 3 to 11 percent of zinc, along with cadmium, copper, lead, silver, and iron. Beneficiation or the concentration of the zinc in the recovered ore is accomplished at or near the mine by crushing, grinding, and flotation process. More than 85% of sulfidic zinc production in the world are using roasting-leaching-electro-winning routes (de Souza, Pina and Leão, 2007; de Souza *et al.*, 2009). Processing the element from its host materials includes froth flotation, roasting, and electro-winning. However, there are difficulties in processing the zinc from its sulfide minerals due to its passivation characters on the mineral surface. The materials require a preliminary process to eliminate such a passivation.

Almost 80% of the processed sulfide comes through the pyrometallurgical method and the rest is from hydrometallurgical one (Filippou, 2004). Pyrometallurgy is a branch of extractive metallurgy that deals with the extraction of minerals from ore by treating them with heat. It consists of the thermal treatment of minerals and metallurgical ores and concentrates to bring about physical and chemical transformations in the materials to enable the recovery of valuable metals. The process includes calcination, roasting, and smelting. Roasting of sulfide ores and concentrates is often as the first step in the production of metals or chemicals (Figure 1). This process consists of thermal gas-solid reactions, which can include oxidation, reduction, chlorination, sulfation, and pyro-hydrolysis. In roasting, the ore or ore concentrate is treated with very hot air. This process is generally applied to sulfide minerals. During roasting, the sulfide is converted to an oxide and sulfur is released as sulfur dioxide, a gas. For the ores that consist of Cu_2S (chalcocite) and ZnS (sphalerite), balanced equations for the roasting are:

$$2\ Cu_2S + 3O_2 \rightarrow 2\ Cu_2O + 2\ SO_2$$
$$2\ ZnS + 3\ O_2 \rightarrow 2\ ZnO + 2\ SO_2$$

This process would release large amounts of acidic, metallic, and other toxic compounds. The oxygen required for the roasting reactions is supplied to the muffle furnace as atmospheric air. With the addition of air, the metal sulfides are transformed into solid oxides and gaseous sulfur dioxide at temperatures of 600 – 1,000°C (Runkel and Sturm, 2009). Roasting depends on the following factors: time, temperature, availability of O_2 or air and physical condition, while the selection criteria of the roasting process include:

- the physical condition of furnace smelting product. The product should be fine and porous;
- the chemical composition of the product, for copper – retain some sulfur and for lead and zinc – the complete elimination of sulfur.

Processing sulfide ores by smelting process seemingly easy to be conducted as the sulfur element will be oxidized to sulfur dioxide (SO_2) gas. If its production is abundant, such the gas can be used for another purpose such as for sulfuric acid making. However; if the amount of the produced gas is relatively scarce, the SO_2 cannot be used for sulfuric acid making and requires a special handling. If not well handling, it will provide negative impacts on human health. Apart from the SO_2 handling, the energy required for smelting the process will also demands a high cost as well. High-energy consumption can be handled if the content of the precious metal is enough. The fact that at current condition and in the future, the metal content within the ores is getting lesser or lower will require efficient processing and energy saving. Therefore, efficient processing and reasonable energy consumption are important factors that should be considered.

The objective of this study is converting the sulfide minerals into the oxide ones in terms of comprehending the optimum condition of ZnS pretreatment for making zincite (ZnO) through the pyro-hydrometallurgy process. Many sulfide minerals contain other components such as arsenic that is released into the environment when roasted. Roasting helps to concentrate the zinc by reducing the unwanted element(s). Once concentrated, the zinc ore is transferred to smelters or leaching reactors for the production of zinc or zinc oxide.

METHODOLOGY

The ore is heated in a regular supply of air in a furnace at a temperature below the melting point of the metal. Its temperature is around 600 - 1,000°C which is generally the most suitable range to process the metal-bearing sulfide ore such as sphalerite, pyrite, and Galena. but it depends on the process and mineralogical requirements (Runkel and Sturm, 2009). Regardless of the purpose, a plant for roasting the metal-bearing sulfide consists of mainly three plant sections: roasting, gas cleaning, and sulfuric acid. With the addition of being transformed into solid oxides and gaseous sulfur dioxide at temperatures such temperatures.

The roasted Zn materials were then leached using 5-M sulfuric acid with percent solid of 25%. The purpose is to evaluate the effect of roasting on the material if leached using a reagent. Other variables are 90°C of reaction temperature, 3-hours of leaching and 400-RPM of stirring speed. Samples for the experiments include the materials that have been roasted at 500, 900 and 1,000 °C. The process employs a three neck rounded flask along with the condenser as shown in Figure 2.

Representative samples were partially batch roasted in a muffle furnace. The material used in this study came from PT Lumbung Mineral Sentosa-Bogor, West Java as sulfide flotation concentrates (Figure 3a). The company specializes its project on Galena – a Pb-bearing sulfide mineral, however, the sulfide concentrates does not only contain lead mineral but also other sulfide minerals such as sphalerite (Zn-bearing sulfides) and chalcopyrite (Cu-bearing sulfides). Naturally, the sample was mined in a massive feature as shown in Figure 3b but then crushed and ground to a certain size (fine sand) for flotation purpose.

At Research and Development Centre for Mineral and Coal Technology, the flotation concentrates were characterized for their mineralogical natures prior and after roasting process by optical microscope, X-ray diffraction (XRD) and scanning electron-energy dispersive spectroscopy (SEM-EDS) methods while its chemical composition was analyzed using atomic absorption spectroscopy (AAS) and X-ray fluorescence (XRF) methods. Characterization also employed thermal analysis.

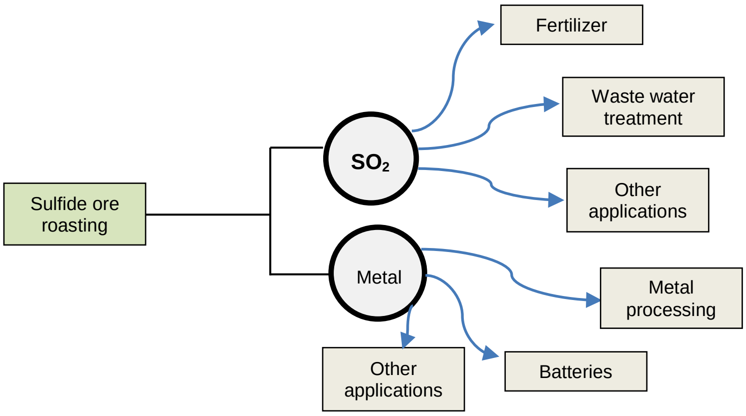


Figure 1. Roasting process for sulfide ore

Figure 2. Instrument setting for leaching experiment of the roasting zinc sulfide

a

b

Figure 3. Sulfide concentrates from PT Lumbung Mineral Sentosa (a) and the original sample of sulfide mineral prior to crushing and concentrating (b)

For roasting section, the samples were proceeded in a furnace applying various temperatures, i.e. 200, 300, 400, 500, 550, 600, 650, 850, 1000 and 1100 °C. The roasting time for each temperature lasted for two hours.

RESULT AND DISCUSSIONS

The chemical composition of the sulfides prior roasting is shown in Table 1. Of the nine detected elements, zinc is the most widely composition of 59.0%. Lead as the part of galena is only 0.99% while the percentages of copper and iron as chalcopyrite and pyrite components are 0.77 and 2.48% respectively. Molybdenum as molybdenite component is 0.14% while antimony and cobalt are less than 0.001 %. Zinc increases significantly when the sulfides were roasted using a series of temperature, from 200 to 1,100 °C (Table 2) while sulfur decreases considerably in line with the increasing of temperature. The higher the roasting temperature, the bigger the zinc content but the lesser the sulfur substance. This is due to the sulfur vaporized to the air as SO_2, while zinc was deposited in-situ.

Table 1. Chemical composition of sulfide concentrate

Elements	Amounts (%)
Pb	0.99
Zn	59.0
Cu	0.77
Fe	2,48
S	32.3
Sb	< 0.001
Mo	0.14
Co	< 0.001

Table 2. Chemical composition of roasted sulfides

Oxides/elements (%)	Roasting Temperature (°C) 200	300	400	500	550	650	850	1000	1100
Zn	57.52	58.04	58.29	58.89	64.11	73.37	76.34	77.07	78.98
S total	38.05	37.60	37.38	36.68	29.75	20.77	17.72	17.38	16.19
Fe total	1.38	1.37	1.39	1.49	1.78	1.90	1.93	1.97	2.04
Pb	0.91	0.93	0.94	0.96	1.23	1.33	1.10	0.97	0.10
Cu	0.53	0.53	0.51	0.52	0.65	0.73	0.77	0.81	0.82
SiO_2	0.74	0.68	0.66	0.65	1.17	0.75	0.76	0.76	1.00
Al_2O_3	0.14	0.13	0.12	0.11	0.31	0.17	0.18	0.12	0.14

Mineralogy characteristics of the sulfide concentrate from PT Lumbung Mineral Sentosa at normal temperature were identified by XRD at room temperature. The XRD result of the original sample detected four minerals. The major phases in the concentrates are sphalerite, galena, chalcopyrite pyrite and quartz (Figure 4). There is a possibility of other minerals such as quartz but it is in very low quantity. The phases were then changed when the samples were roasted using a series of temperature, from 200 through 1,100°C. No mineral transformation occurred when the sample was roasted at 200, 300, 400, and 500 °C. The available minerals are still the same as those at room temperature. Yet the mineral transformation starts happening when the temperature increases to 550 up to 1.100°C. The available within such temperatures are sphalerite, zincite, franklinite, and quartz. Table 3 illustrates XRD testing results of the roasted sulfides. It is seen that the quartz starts being detected when the temperature reached 550 °C. Such a mineral was detected at a temperature below 550 °C. It is assumed that the increasing of temperature to 550 °C, the entrapped quartz particles within the sulfides is released. Zincite (ZnO) and franklinite ($ZnFe_2O_4$) were then occurred at 550°C along with sphalerite and quartz. Both minerals are supposed to occurring due to a lot of zinc elements from previous sulfides was released and then reacted with iron and oxygen to form other zinc oxide minerals.

Figure 5 shows the minerals occurred during roasting at a temperature of 400°C to 1.100°C. It seems that the higher temperature provided a chance to initiate other oxide mineral development, such as franklinite and zincite. Both minerals started occurring at 550 °C and continued to the temperature of 1,100 °C. It is showed that some sulfidic sulfur has vapored to SO_2 gas but it is not completed because some zinc sulfide (sphalerite) still exists.

The phase changes that occur in the particles were investigated using light microscopy and SEM-EDS.

Optical microscopy test confirms the XRD test. The samples consist of sulfide minerals, i.e. sphalerite, galena, chalcopyrite, and pyrite as well. Most of the sphalerite particles are liberated (Figure 6). Similar to sphalerite, other sulfide minerals either chalcopyrite, pyrite or galena were also performed as the liberated minerals.

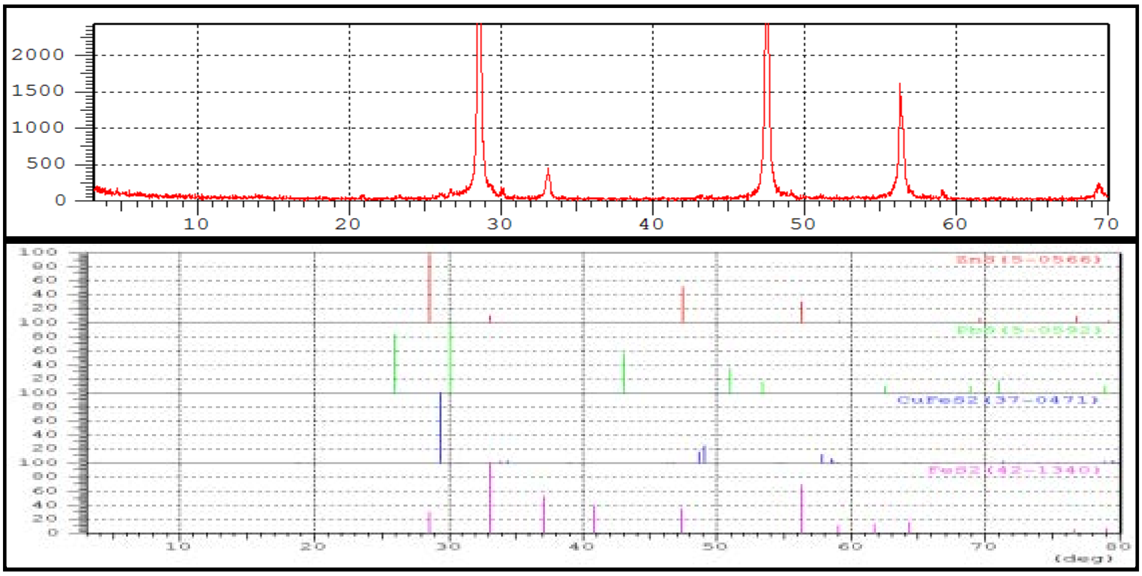


Figure 4.Result of XRD analysis for sulfide concentrate of PT Lumbung Mineral Sentosa showing sphalerite (ZnS), galena (PbS), chalcopyrite ($CuFeS_2$), quartz (SiO_2) and pyrite (FeS_2)

Table 3. Mineral composition of roasted concentrate at various temperature

Temperature (°C)	Mineral composition
200	Sphalerite (ZnS), galena (PbS), chalcopyrite ($CuFeS_2$), pyrite (FeS_2), quartz (SiO_2)
500	Sphalerite (ZnS), galena (PbS), chalcopyrite ($CuFeS_2$), pyrite (FeS_2), quartz (SiO_2)
550	Sphalerite (ZnS), zincite (ZnO), franklinite ($ZnFe_2O_4$), quartz (SiO_2)
650	Sphalerite (ZnS), zincite (ZnO), franklinite ($ZnFe_2O_4$), quartz (SiO_2)
850	Sphalerite (ZnS), zincite (ZnO), franklinite ($ZnFe_2O_4$), quartz (SiO_2)
1,000	Sphalerite (ZnS), zincite (ZnO), franklinite($ZnFe_2O_4$), quartz (SiO_2)
1,100	Sphalerite (ZnS), zincite (ZnO), franklinite ($ZnFe_2O_4$), quartz (SiO_2)

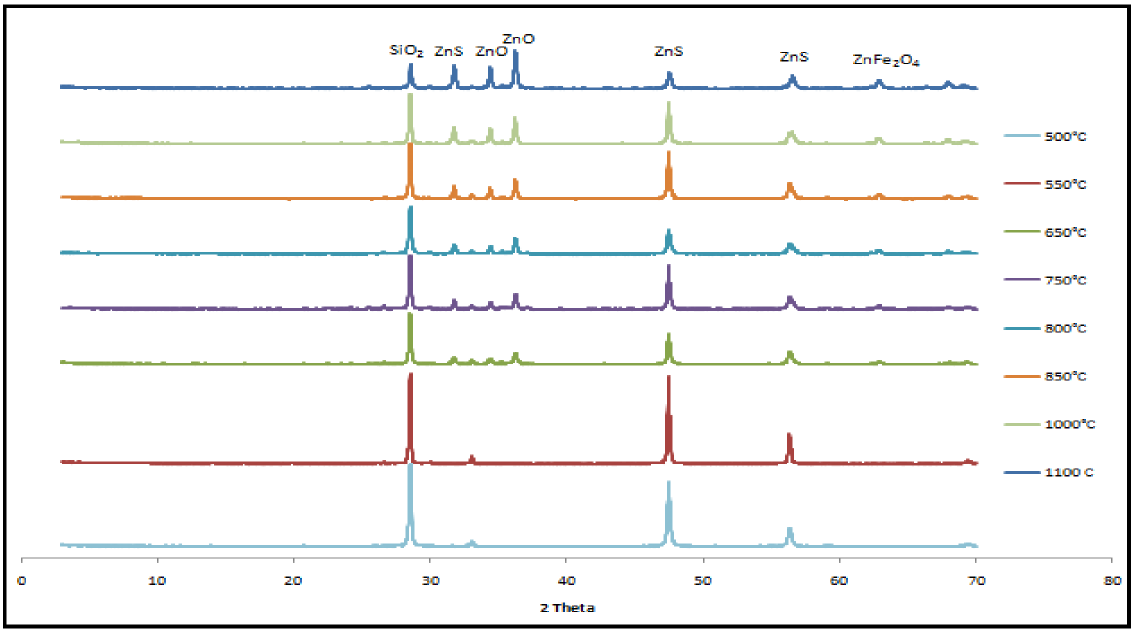


Figure 5. Results of XRD tests for the roasted sulfide

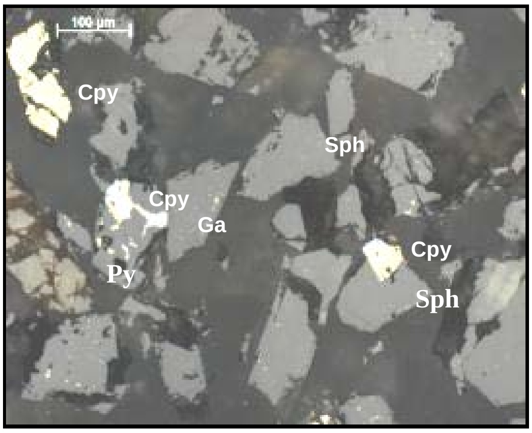
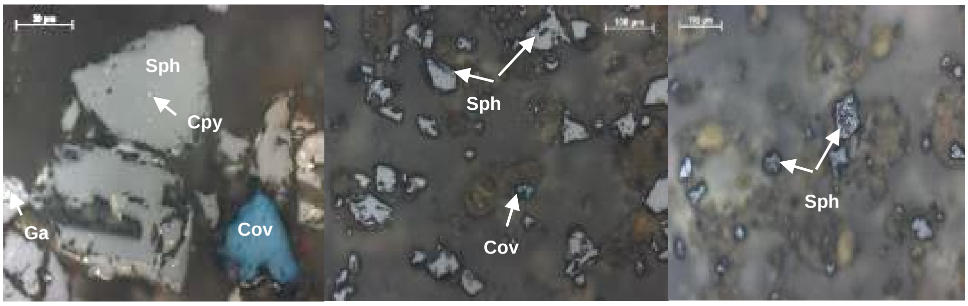


Figure 6. Microscopy analysis of the sulfide sample from PT Lumbung Mineral Sentosa. Sphalerite as the dominant sulfide mineral that normally encapsulated other sulfides
(Sph= sphalerite, Ga= Galium and Cpy = chalcopyrite)

However, some of them are included within sphalerite. Of the four sulfide minerals, sphalerite is seemed as the dominant one as shown in Figure 6. Microscopy analysis indicates that sphalerite mostly encapsulated other sulfides, either chalcopyrite, galena or pyrite. Yet the sulfides also showed another locked structure, which is distributed along the fine structure. Liberating the encapsulated sulfides seemed easy. Roasting such sulfides would liberate the locked particles.

Roasting the sulfide concentrates developed positively (Figure 7). During the roasting, the sulfide was converted to an oxide as either other minerals or elements, and sulfur was released as sulfur dioxide gas. Some were remained in its nature phases. Figure 7a shows that roasting the sulfides at 550°C obtained sphalerite (Sph) as a single granule or a bound particle with another phase notably galena (Ga). The sphalerite was also found to entrap the chalcopyrite (Cp). Covellite (Co) is also served as a single crystal that performs a blue color. Increasing the temperature to 850°C results in the particle of sulfide minerals occurs (mostly) as individual particles. The bound phases are mostly released. The mineral phases include predominantly sphalerite and rarely covellite as a single crystal (Figure 7b). The sphalerite is partly changed into semi-metal material when the roasting temperature is changed to 1,100°C (Figure 7c).

Mineralogical characters were also evaluated through SEM-EDS analysis using X-ray mapping method. Different from results of XRD and microscopy analyses, SEM-EDS analysis by X-ray mapping method was only detected four elements, i.e. sodium (Na), iron (Fe), zinc (Zn) and sulfur (S) as shown in Figure 8. Sodium might be came from sodium sulfide (Na_2S) or more commonly as hydrate $Na_2S{\cdot}9H_2O$. The material is colorless water-soluble salts that provide a strongly alkaline solution. When exposed to moist air, sodium sulfide releases hydrogen sulfide that smells like rotten eggs. Sodium sulfide is normally utilized in water treatment as an oxygen scavenger agent and also as a metals precipitant, in ore flotation, oil recovery, making dyes, and detergent. Referring to the mapping figure, sodium seems dominant compared to zinc and iron. Its performance is similar to thallium and molybdenum elements. In nature, thallium sulfide (Tl_2S) is found as carlinite which has the distinction of being the only sulfide mineral of thallium that does not contain at least two metals. Tl_2S was used in some of the earliest photo-electric detectors. Molybdenum along with sulfur is the element of molybdenite (MoS_2). The mineral is commonly used as dry lubricant and catalyst for desulfurization in petrochemistry. Zinc element may come from zinc sulfide or known as sphalerite. It performs black appearance, however, pure sphalerite is actually white. The black performance occurred due to various impurities. Compared to sodium, thallium, and molybdenum; zinc in the analyzed area is not so dominant. Iron within the detected area is supposed to be come from pyrite (FeS_2). When roasting occurred, the sulfides started to decompose. Roasting has driven off the zinc, lead, copper, and iron from the original sphalerite, galena, chalcopyrite, and pyrite, leaving behind granular textures of undecomposed sulfide. Prior roasting, the sulfides performed mostly angular to sub-angular textures (Figure 8) but then changed to subrounded – sub-angular ones as shown in Figure 9. Sodium, zinc, copper, molybdenum, thallium, and sulfur are available within a roasted sample of 550°C.Its quantity is 3.93, 40.13, 1.07, 11.9, 9.09 and 5.37% respectively.



a b c

Figure 7. Mineral phases of sulfide concentrate, roasted at 550°C (a), 850°C (b) and 1,100°C (c).
(Sph= sphlerite, Ga= Galium and Cpy = chalcopyrite,.s = sulfur)

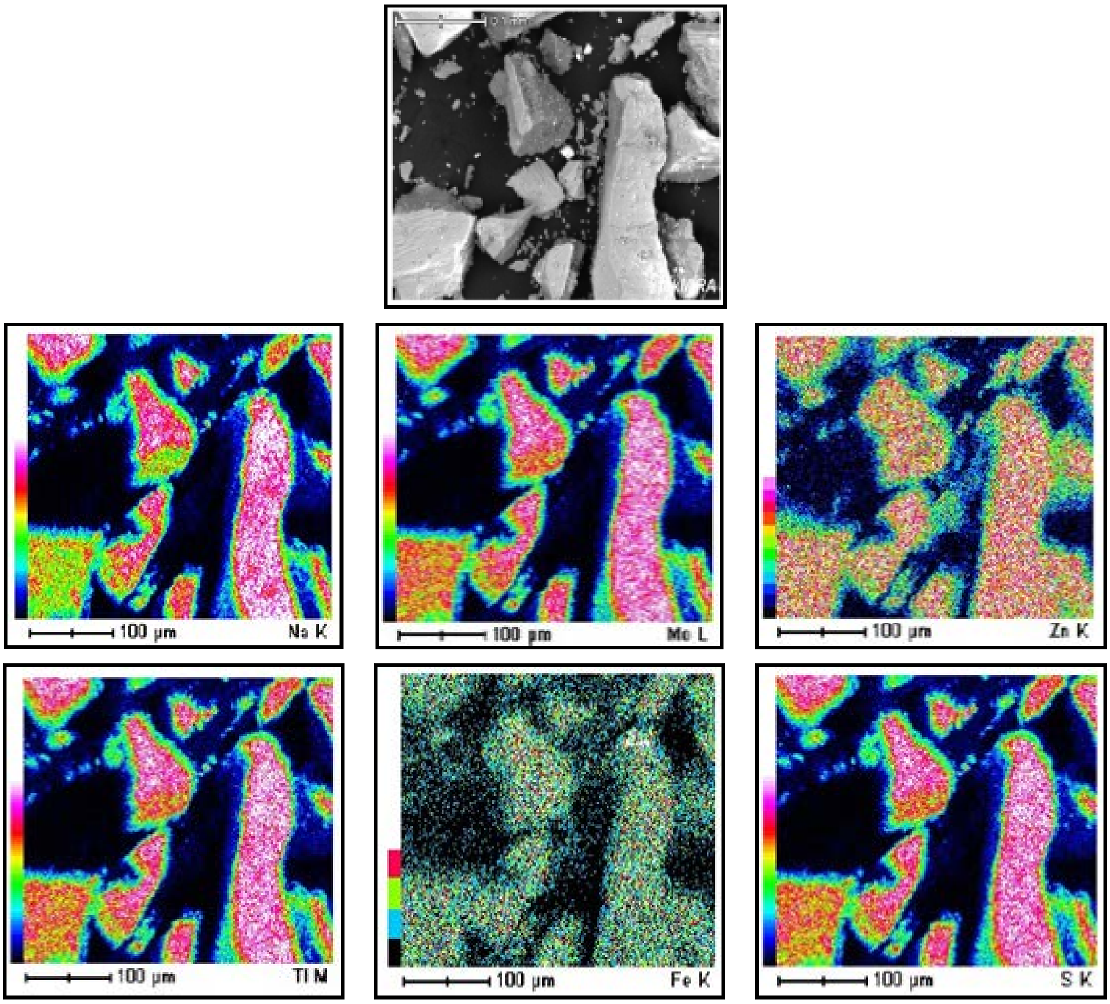


Figure 8. X-ray mapping method of SEM-EDS analysis conducted on sulfide concentrate from PT Lumbung Mineral Sentosa

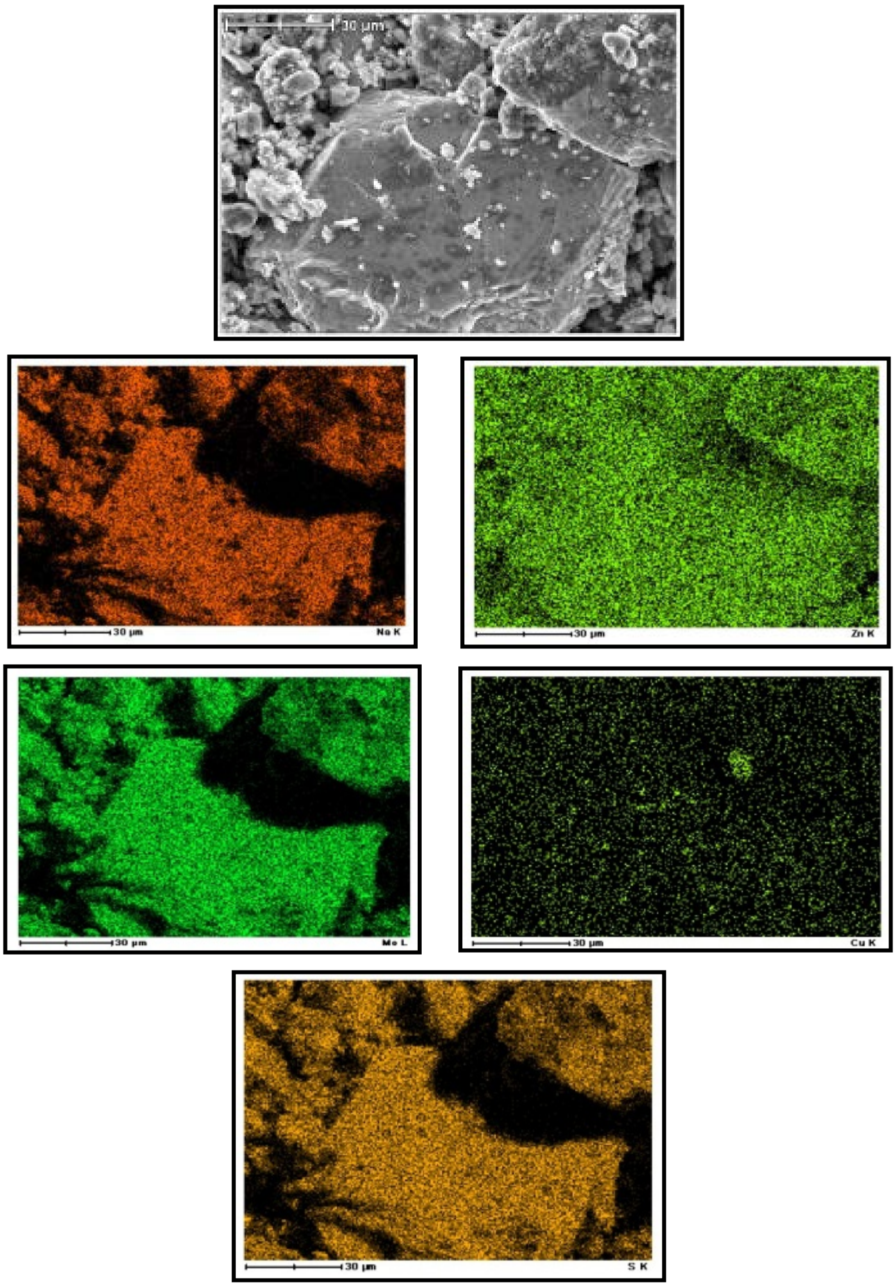


Figure 9. Result of SEM-EDS test on 550°C-roasted zinc concentrate showing sodium, zinc, molybdenum, copper, and sulfur as the detected elements

SEM-EDS analysis was also conducted to the roasted sample. The examined sample was the 500°C-roasted sample - the material in which the sphalerite started decomposing to other materials as seen in Table 3 and Figure 4. The oxidation of iron-bearing sphalerite occurs in three stages. The first involves the selective diffusion of most of the iron to the particle surface resulting in the formation of an iron oxide shell enclosing a largely unreacted zinc sulfide kernel. In the second stage, this kernel is oxidized to form a solid solution of zinc oxide and iron oxide. The iron is initially present in the ferrous state but, with the disappearance of the sulfide kernel, is oxidized to ferric iron. In the final stage, this dissolved iron oxide and the iron oxide shell react with the surrounding zinc oxide to form the refractory spinel zinc ferrite. There are five detected elements available within the 500°C-roasted zinc sample. Those are sodium (Na), zinc (Zn), molybdenum (Mo), copper (Cu) and sulfur (S) as shown in Figure 9. Although the XRD test detected the zinc ferrite or known as franklinite, iron (Fe) was not detected in X-ray mapping test. Quantitatively, (as per EDX spectrum reading), the amount of iron is lower than others as seen in Table 4 and its peak also does not show a sharp point (Figure 10). Both are supposed to be the reason why such the element cannot be mapped. Although the quantity of copper is smaller than that of the iron and mapped as a noise, its relative bigger energy (the keV as shown in Table 4) makes the copper can be mapped although just a small spot. Sodium is supposed to be the element of sodium sulfide. It seems such a material is still available in the roasted material. Zinc occurs as the element of sphalerite, zincite, and franklinite. Franklinite is a potential as zinc resources to be recovered. However, the formation of zinc ferrite during the roasting of zinc concentrates requires substantial additional processing to recover the zinc from this compound by leaching and to eliminate the iron from the leachate. The processes which the iron undergoes during its eventual transformation into ferrite have been clarified by examination of the phases and the morphology of partially roasted marmatite sphalerite particles (Zn, Fe)S from this compound by leaching and to eliminate the iron from the leachate.

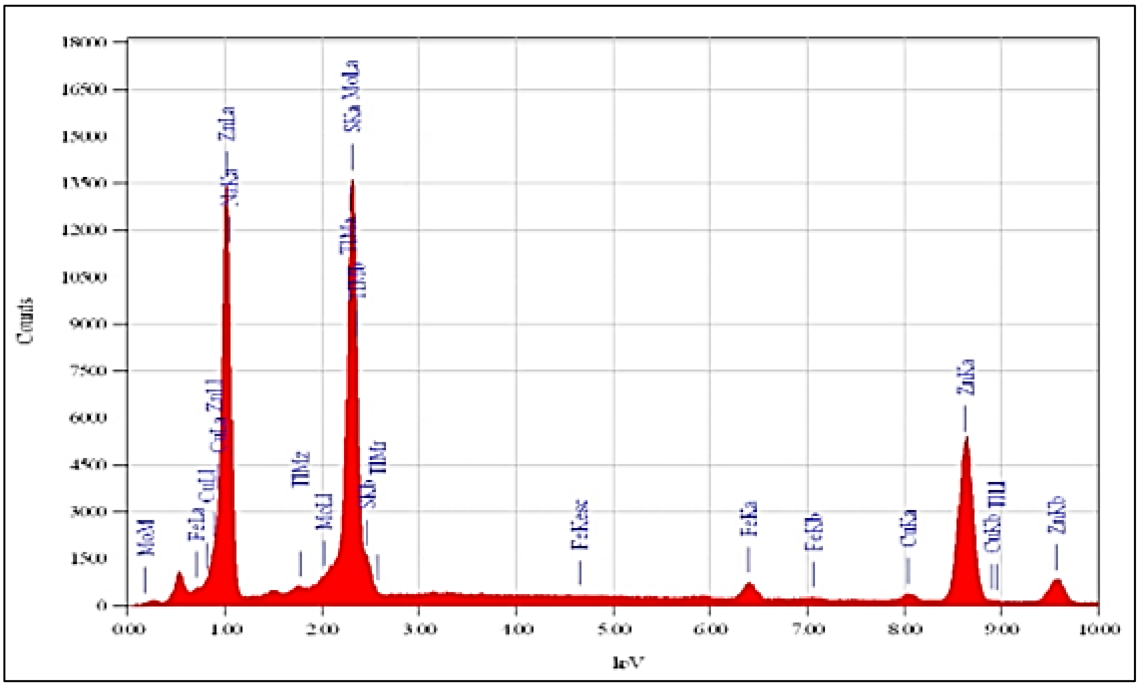


Figure 10. Spectrum test of 500°C-roasted zinc sulfide sample showing almost flat of the iron peak

Table 4. Quantitative analysis of 500°C-roasted zinc sample showing 7 detected elements.

Element	(keV)	Mass%	Error%	At%	Compound	Mass%	Cation	K
O		26.96						
Na K	1.041	3.93	0.16	8.07	Na_2O	5.29	2.43	2.6808
S K	2.307	5.37	0.25	15.84	SO_3	13.41	2.39	7.6952
Fe K	6.398	1.55	0.22	2.62	FeO	1.99	0.40	2.4449
Cu K	8.040	1.07	0.44	1.59	CuO	1.34	0.24	1.5952
Zn K	8.630	40.13	0.56	58.04	ZnO	49.95	8.74	59.9701
Mo L	2.293	11.90	0.53	11.73	MoO_3	17.86	1.77	13.6207
Tl M	2.267	9.09	0.28	2.10	Ti_2O_3	10.16	0.63	11.9931
Total		100.00		100.00		100.00	16.60	

SEM-EDS analysis were also conducted to 1,100°C-roasted zinc sulfide to examine element distribution and their performance after roasting at such a temperature. The mapped elements in this sample include zinc, molybdenum, copper, thallium, sodium, and sulfur (Figure 11). Zinc element in this sample shows disentangled performance compared to the same element within 500°C-roasted zinc sulfide sample. It seems that such an element starts being the metal although the sulfur is still detected. Its quantity increases from 40.13% in 500 °C-roasted zinc sulfide sample to 45.59% in 1,100°C-roasted zinc sulfide sample.

In terms of investigating the thermal effect on the weight change of the sample during heating, a TGA was also conducted on the zinc sulfide samples. Yet, preparing the samples was a significant effect in obtaining good data. Maximizing the surface area of the sample in a TGA pan improved resolution and reproducibility of weight loss by the temperatures. Typically 10-20 mg of the sample is preferred in most applications. Whereas, the sample with volatiles of 50-100mg is considered as an adequate one. In such an analysis, the sample was heated in a given environment (Air, N_2, CO_2, He, Ar, etc.) at a controlled rate. The temperature was increased at a constant rate for a known initial weight of the substance and the changes in weights were recorded as a function of temperature at a different time interval (Hutabarat *et al.*, 2015).

The thermal analysis of this study used the TGA method. It measured the amount and the rate of weight change of a material with respect to temperature or time in controlled environments. The TGA diagram below shows the thermal analysis of Zn concentrate that commenced from room temperature to 1000°C (Figure 12). Water vaporization began around 100°C followed by weight loss. Further vaporization of moisture accomplished at 250°C with smaller weight loss than the previous one. The downward trend of weight reduction starts at 500°C lead to SO_2^-off gas production. The weight dropped significantly to 80 mg which means approximately 13% of the weight was decreased when it held for 2 hours at 1000°C of heating (Boyanov and Peltekov, 2013).

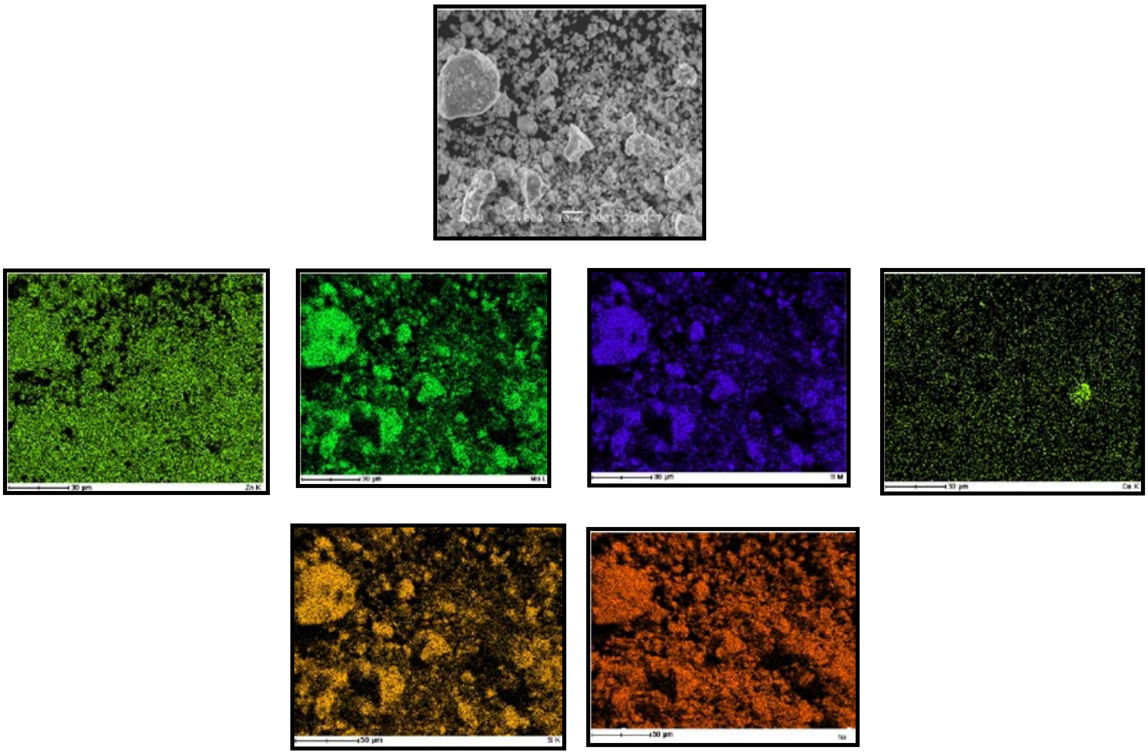


Figure11. SEM-EDS test on 1,100°C- zinc sulfide sample showing disentangled Zn element

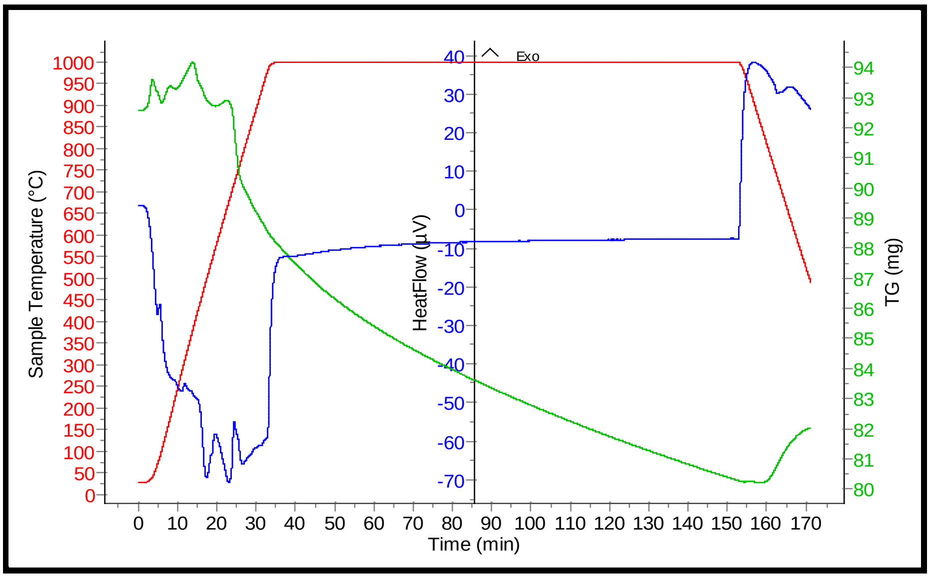


Figure 12. Result of TG analysis for zinc sulfide concentrates

Figure 13 shows weight development of the roasted materials. In such a roasting process, the oxidation temperature depends on the grain size, the heating rate, and the amount of sample. A plateau condition occurs at a temperature between 200-500°C. The roasted mass (the ZnS) is essentially constant or there is no change in mass. As the temperature increases, the zinc weight also improves from more or less 60% at a temperature of 200°C to around 80% at a temperature of 1,100°C. Such the weight start increases at a temperature of 500°C. Different from the zinc element, there is a large mass loss followed by a mass plateau and is formed when evaporation of volatile products during drying takes place. The sulfur starts decreasing at the same temperature of which the zinc weight starts increasing. When the temperature extends to 500°C, the zinc sulfide (ZnS) starts decomposing into ZnO and SO_2 (Földvári, 2011).

The experiment results show that leaching the 550°C-roasted sample provided percent extraction of 17.41%. The dissolved zinc is 27.802 g/l (Table 5). The 900°C-roasted sample yields the extracted zinc of 47.48%. Such a figure is the same as 102.724 g/l of dissolved zinc. Using the 1,100°C-roasted sample and similar leaching condition with two other experiments, the dissolved zinc increased almost 20% bigger than that of the 900°C-roasted sample. The amount of sulfide within the 900°C-roasted sample and the 1,100°C-roasted sample seem much reduced compared to that in 550°C-roasted sample. As a result, the zinc dissolution works better. The sulfide in the 550°C-roasted samples causes a passivation phenomenon on the sample surfaces and the remaining zinc is difficult to get a contact with the sulfuric acid (Khattar, Dudeja and Arora, 2011).

The fact that the original and roasted samples contain other elements instead of Zn seems also one of the factors that causes low recovery of zinc. The liberated elements within roasted samples improved in line with the increased temperature. Such the elements are supposed to be the preg-robber solution in the leaching experiments. Referring to mineralogical aspects, microscope analysis shows the higher the temperature the lesser the available minerals within the samples. The 550°C-roasted sample, for example, has several minerals similar to the original, unroasted-sample such as sphalerite, chalcopyrite, galena, and covellite (Figure 7a). The Increasing of the roasting temperature results in decreasing of the mineral types within samples (Figure7b and 7c). However, the morphology of some available minerals, examined by SEM-EDS, exhibits a massive surface that can inhibits the leaching solution to reach the Zn element. If the surface has the roughness and porosity as shown in Figure 9 and 11, the leaching process is supposed to have a progressive increase in the Zn recovery (de Souza *et al.*, 2009).

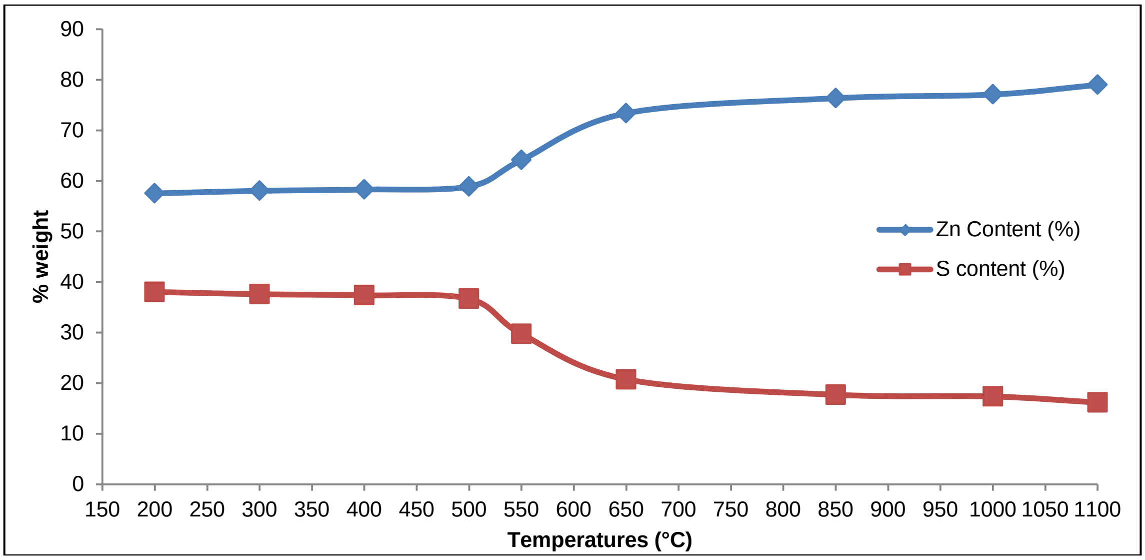


Figure 13. Development of zinc and sulfur weights during roasting.

Table 5. Results of leaching experiment using 5-M sulfuric acid

Sample codes	Dissolved Zn (g/L)	% extraction
550 °C-roasted sample	27.802	17.41
900 °C-roasted sample	102.724	47.48
1,100 °C-roasted sample	119.771	53.32

CONCLUSIONS AND SUGGESTIONS

Mineral composition of the sample includes sphalerite, galena, chalcopyrite, and pyrite in which the sphalerite is dominant within samples. When roasted, sphalerite started decomposing at 550 °C performing zincite, franklinite, quartz, and sphalerite as well. Such a composition do not change although the temperature increases to 1,100 °C. The Zn content within the original sample was 59.00%, but improved in line with the increased temperature. The highest Zn was 78.98% achieved at 1,100 °C.

Roasting changes the mineral phases of the samples. Optical microscope analysis on 500 °C-roasted zinc sample show sphalerite, covellite, galena, and chalcopyrite. Increasing the temperature to 850 °C, the available sulfide are sphalerite and covellite. The sulfide then diminishes when the roasting temperature increases to 1,100 °C; only sphalerite is available.

The oxidation of iron-bearing sphalerite produces zinc ferrite material. Such a material can be beneficiated as a substitute for applications in temperatures above 350 °F (177 °C). When added to high corrosion-resistant coatings, the corrosion protection increases with an increase in the concentration of zinc ferrite.

It is suggested to conduct magnetically separation to the zinc sulfide concentrates to minimize the iron-bearing impurities and other undesirable elements that can be harmful to the leaching process. As a results, the overall zinc recovery could be increased by more than 78.98%.

ACKNOWLEDGEMENTS

The authors would like to acknowledge the Ministry of Energy and Mineral Resources for providing research fund. Thanks also to Hidayat – the technician of Pyrometallurgy Laboratory at the RDCMCT for his assistance during the laboratory works and PT Lumbung Mineral Sentosa for providing the zinc-bearing sulfide samples.

REFERENCES

Baba, A. A. and Adekola, F. A. (2011) “Beneficiation of a Nigerian sphalerite mineral: Solvent extraction of zinc by Cyanex®272 in hydrochloric acid,” *Hydrometallurgy*, 109(3–4), pp. 187–193. doi: 10.1016/j.hydromet.2011.06.004.

BBC (2016) *Reactivity series*, *www.bbc.co.uk*. Available at: http://www.bbc.co.uk/education (Accessed: February 18, 2016).

Boyanov, B. S. and Peltekov, A. B. (2013) “Study of zinc sulfide concentrates by DTA, TGA, and X-Ray analyses and their roasting in fluidized bed furnace,” *ISRN Industrial Engineering*, 2013, pp. 1–8. doi: 10.1155/2013/719759.

Filippou, D. (2004) “Innovative hydrometallurgical prcesses for the primary processing of zinc,” *Mineral Processing and Extractive Metallurgy Review*, 25(3), pp. 205–252. doi: 10.1080/08827500490441341.

Földvári, M. (2011) *Handbook of thermogravimetric system of minerals and its use in geological practice*. Budapest: Innova-Print Kft.

Hutabarat, I., Schnideritsch, H., Poscher, A., Sobotka, C. and Antrekowitsch, H. (2015) “Characterisation and thermal behaviour of Indonesian copper sulphides for leaching process,” in *Proceeding European Metallurgical Conference Volume 1*. Düsseldorf, Germany: Papierflieger, pp. 171–188.

Khattar, D., Dudeja, R. and Arora, K. K. (2011) “Chemical bond,” in *The Pearson Complete Guide to the All India Engineering Entrance Examination*. 4th Ed. Delhi, India: Dorling Kindersley Pvt. Ltd., p. C-96-C-101.

McLeod, C. (2017) *Zinc applications and new demand sources*, *Zinc Investing News*. Available at: https://investingnews.com/daily/resource-investing/base-metals-investing/zinc-investing/zinc-applications-zinc-demand/.

Runkel, M. and Sturm, P. (2009) “Pyrite roasting, an alternative to sulphur burning,” *The Journal of The Southern African Institute of Mining and Metallurgy*, 109, pp. 491–496. Available at: http://www.scielo.org.za/scielo.php?script=sci_arttext&pid=S2225-62532009000800007.

de Souza, A. D., Pina, P. S. and Leão, V. A. (2007) “Bioleaching and chemical leaching as an integrated process in the zinc industry,” *Minerals Engineering*, 20(6), pp. 591–599. doi: 10.1016/j.mineng.2006.12.014.

de Souza, A. D., Pina, P. S., Santos, F. M. F., da Silva, C. A. and Leão, V. A. (2009) “Effect of iron in zinc silicate concentrate on leaching with sulphuric acid,” *Hydrometallurgy*, 95(3–4), pp. 207–214. doi: 10.1016/j.hydromet.2008.05.049.

Stanton, R. L. (1994) *Ore elements in arc lavas*. Clarendon Press.

Zhang, Y., Deng, J., Chen, J., Yu, R. and Xing, X. (2014) “The electrowinning of zinc from sodium hydroxide solutions,” *Hydrometallurgy*, 146, pp. 59–63. doi: 10.1016/j.hydromet.2014.03.006.