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PARAMETERS THAT AFFECT THE DISSOLUTION OF INDONESIAN GALENA CONCENTRATE IN FLUOROSILICIC ACID AND HYDROGEN PEROXIDE

PARAMETER-PARAMETER YANG MEMENGARUHI PELARUTAN KONSENTRAT GALENA INDONESIA DALAM LARUTAN ASAM FLUOROSILIKAT DAN HIDROGEN PEROKSIDA

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ABSTRACT

Pyrometallurgical process still dominates the extraction of galena concentrates. The process used to extract the lead includes reduction smelting in a blast furnace, air flash smelting (Boliden process), oxygen flash smelting (Kivcet, Boliden Kaldo, Outokumpu), air-slag bath smelting (Isasmelt) and oxygen-slag bath smelting (QSL). However, those generate dust, SO_2 gas and volatile Pb liquid. As a result, such processes are ineffective to treat the complex sulfides and low-grade flotation con concentrates. Referring to the lack of high-grade lead ore the lead pyrometallurgical is a problem in the future. In addition, the environmental regulation becomes very strict lately. Those pushes the metallurgist to seek the alternative process that are environmentally friendly and able to treat the low-grade concentrates. Lead extraction through hydrometallurgical process is considered to be safer as the process do not produce dust, SO_2 gas and lead vapor.Researches for lead extraction through hydrometallurgical routes have been performed using various leaching agents such as acetic acid, ferric methanesulfonate, ferric chloride, ferric fluorosilicate and nitric acid with hydrogen peroxide and ferric ion as the oxidants. So far, no lead plant operates hydrometallurgically in an industrial scale. Fluorosilicic acid has a potential to be used as the leaching reagent for concentrating the lead because of high lead solubility in this solution and cheaper price of the reagent in compared to sulfamate and fluoroborate solutions. This research used galena concentrates from a mining area in Bogor, Indonesia, fluorosilicic acid and hydrogen peroxide as the oxidants. The highest Pb extraction percentage of 99.26% was achieved from the leaching experiment using 3.44 M of H_2SiF_6 and 9.79 M of H_2O_2, at 97^oC and concentrate particle size distribution of -100+150 mesh after 135 minutes. The XRD analysis of the leaching residue with no oxidant showed the presence of galena, sphalerite and chalcopyrite, while the residue of the leaching with oxidant showed anglesite ($PbSO_4$), galena, sphalerite, sulphur and pyrite. Lead extractions were increased by the increase of temperature and concentration of fluorosilicic acid. The best solid percentage that gave the highest lead extraction percentage was 12%. Variations of rotation speeds at the range of 300-700 rpm did not significantly influence lead extraction percentage. However, the particle size distribution that resulted in the best extraction percentage of lead is 100+150#, at which the finer particle size of the concentrate give a lower extraction percentage of the lead due to $PbSO_4$ precipitation.

Keywords: galena concentrate, leaching, fluorosilicic acid, extraction

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ABSTRAK

Hingga saat ini, proses ekstraksi timbal dari konsentrat galena masih didominasi oleh proses pirometalurgi. Proses pirometalurgi yang digunakan untuk mengekstrak timbal adalah peleburan reduksi di dalam tanur tiup, udara kilat (proses Boliden), oksigen kilat (Kivcet, Boliden Kaido, Outokumpu), udara-genangan terak (Isasmelt), dan oksigen-genangan terak (QSL). Semua proses menghasilkan debu, gas SO_2 dan uap Pb. Proses pirometalurgi tersebut tidak efektif untuk mengolah konsentrat sulfida kompleks dan kadar rendah hasil flotasi. Keterbatasan ini menjadi kendala penerapan jalur pirometalurgi untuk masa depan karena ketersediaan bijih timbal berkadar tinggi semakin sedikit. Selain itu, peraturan yang terkait dengan pencemaran lingkungan juga semakin ketat sehingga risiko emisi debu, Pb dan gas SO_2 akan menjadi tantangan berat penggunaan proses ekstraksi timbal dengan jalur pirometalurgi di masa depan. Untuk itu, perlu dicari alternatif lain proses ekstraksi timbal lain yang lebih ramah lingkungan dan mampu mengolah bijih dengan kadar rendah. Ekstraksi Pb dengan jalur hidrometalurgi dianggap memberikan solusi atas permasalahan-permasalahan tersebut di atas karena proses tersebut tidak menghasilkan debu, emisi gas SO_2 dan emisi uap timbal. Penelitian ekstraksi Pb dari konsentrat galena dengan jalur hidrometalurgi telah dilakukan dengan berbagai jenis reagen pelindi yang meliputi asam asetat, ferric methanesulfonate, feri klorida, feri fluorosilikat dan asam nitrat menggunakan oksidator hidrogen peroksida dan ion feri. Sayangnya, sampai saat ini, belum ada pabrik ekstraksi timbal secara hidrometalurgi yang beroperasi pada skala industri, kecuali hanya sebatas penelitian. Asam fluorosilikat berpotensi sebagai reagen pelindi untuk pelindian (pelarutan) konsentrat timbal karena kelarutan timbal dalam larutan ini sangat tinggi dan harganya lebih murah dibandingkan larutan sulfamat dan fluoroborat. Dalam penelitian ini, keefektifan pelindian timbal dari konsentrat galena, yang diperoleh dari Bogor, Indonesia, menggunakan asam fluorosilikat dan hidrogen peroksida sebagai oksidator dipelajari. Penelitian ini menentukan persen ekstraksi timbal tertinggi berdasarkan kondisi pelindian terbaik yang berhasil dicapai. Persen ekstraksi Pb tertinggi yaitu 99,26% diperoleh pada percobaan pelindian dengan konsentrasi H_2SiF_6 3,44 M dan konsentrasi H_2O_2 9,79 M pada temperatur 97^oC dan distribusi ukuran partikel konsentrat -100+150# setelah 135 menit. Analisis XRD pada residu pelindian tanpa penambahan oksidator (H_2O_2) menunjukkan keberadaan galena, sfalerit dan kalkopirit, sementara pada residu pelindian dengan oksidator teridentifikasi adanya anglesit ($PbSO_4$), galena, sfalerit, sulfur dan pirit. Persen ekstraksi Pb meningkat dengan naiknya temperatur dan konsentrasi asam fluorosilikat. Persen padatan terbaik yang memberikan persen ekstraksi Pb paling tinggi adalah 12%. Variasi kecepatan pengadukan dalam rentang 300-700 rpm tidak mempengaruhi persen ekstraksi Pb secara signifikan. Distribusi ukuran partikel konsentrat galena yang memberikan persen ekstraksi Pb paling tinggi adalah -100+150#, sedangkan pada ukuran partikel konsentrat lebih halus diperoleh persen ekstraksi Pb yang lebih rendah karena terbentuknya endapan $PbSO_4$.

Kata kunci: konsentrat galena, pelindian, asam fluorosilikat, ekstraksi

INTRODUCTION

According to Liu *et al.* (2018), the most important lead (Pb) mineral is galena (PbS). Yet, the extraction processes of lead from galena concentrate are still dominated by pyrometallurgical processes. The processes used for lead extraction include reduction smelting in a blast furnace, air flash smelting (Boliden process), oxygen flash smelting (Kivcet, Boliden Kaldo, Outokumpu), air-slag bath smelting (Isasmelt) and oxygen-slag bath smelting (QSL). These processes generate dust, SO_2 gas and volatile Pb liquid that result in ineffective to be used in treating complex sulfides and low-grade flotation concentrates. These limitations are a challenge to apply pyrometallurgical processes in the future due to the lack of high-grade lead ore. Meanwhile, the environmental regulation becomes strict related to the risks of dust, SO_2 and lead emissions from smelting processes in the future. Therefore, it is important to find the alternative processes which are more environmentally friendly and able to treat the low low-grade concentrate.

Referring to a hydrometallurgy route, Pb extraction is considered to be the way in solving those problems because it does not produce SO_2 gas and lead fume (Golpayegani and Abdollahadeh, 2017). In hydrometallurgical lead extraction, sulphur is separated from the Pb as the elemental sulphur (S^0) and removed by filtration. It is necessary to oxidize the solution in the sulfide so that the sulphur will be converted into the elemental sulphur (Uçar, 2009). According to Lee, Wethington and Cole (1986), leaching of galena concentrate at low temperature using fluorosilicic acid

(H_2SiF_6) and hydrogen peroxide as an oxidant can reduce the lead emission, eliminate the SO_2 emission and produce elemental sulphur which is easy to remove. The elemental sulphur performance is solid and porous and does not hinder galena dissolution. This a by product that can be sold (Nnanwube and Onukwuli, 2018).

Selecting the leaching agent in lead extraction galena concentrate is determined by the advantages and the weaknesses of the agent itself. Acetic acid has relatively low conductivity that can reduce the effectiveness of the dissolved Pb recovery. Fluoroboric acid tends to release the fluoride ions which produce corrosive lead fluoride precipitate ('Afif, 2015). Ferric chloride has a limitation in which the resulting lead chloride has further to be removed by electrolysis in fused salt. This is clearly more expensive than electrowinning if fluorosilicic acid is used (Wu *et al.*, 2014). Therefore, the utilization of fluorosilicic acid is promising because the Pb dissolution in fluorosilicic acid is quite high and its lower price compared to sulphamate and fluoboric's. In this research, the effective of Pb extraction from Indonesian galena concentrate by leaching in H_2SiF_6 solution using H_2O_2 as oxidant was studied.

METHODOLOGY

The galena concentrate sample was received from a flotation plant of galena in Bogor, West Java Province of Indonesia. Sample preparation and sampling was then carried out to prepare representative sample for particle size distribution, chemical and mineralogical analyses and for leaching test works. Particle size distribution of the galena concentrate is presented in Table 1. It was found that the size of concentrate particle mostly has particle size distributions of -100+150 mesh (42.87%) and -150+200 mesh (38.86%). Chemical composition of the sample was measured by Atomic Absorption Spectrophotometer (AAS) of AA 240 FS type, while the chemical composition of the sample with various particle size fraction was also determined by X-Ray Fluoroscence (XRF) of Shimadzu. Results of chemical composition analysis of the samples and chemical composition of the concentrates per fraction are given in Table 2 and 3, respectively. The major metal elements present in the lead concentrate are lead (66.6%), zinc (7.38%), iron (2.79%) and copper (0.84%) with total sulfur of 17.62%. Mineral identification was conducted by X-Ray Diffraction (XRD) analysis using XRD-7000 Maxima type from Shimadzu. XRD spectrum of the galena sample is presented in Figure 1. It was identified that the major minerals in the galena concentrate sample are galena (PbS), sphalerite (ZnS) and chalcopyrite ($CuFeS_2$).

Table 1. Particle size distribution of the galena concentrates

Size	Weight [g]	Weight [%]
+100 #	355.75	7.07
-100 + 150 #	2,158.00	42.87
-150 + 200 #	1,956.00	38.86
-200 + 325 #	258.35	5.13
-325 + 400 #	65.93	1.31
-400 #	239.48	4.76
TOTAL	5,033.51	100.00

Table 2. Chemical composition of the galena concentrate sample (as-received sample)

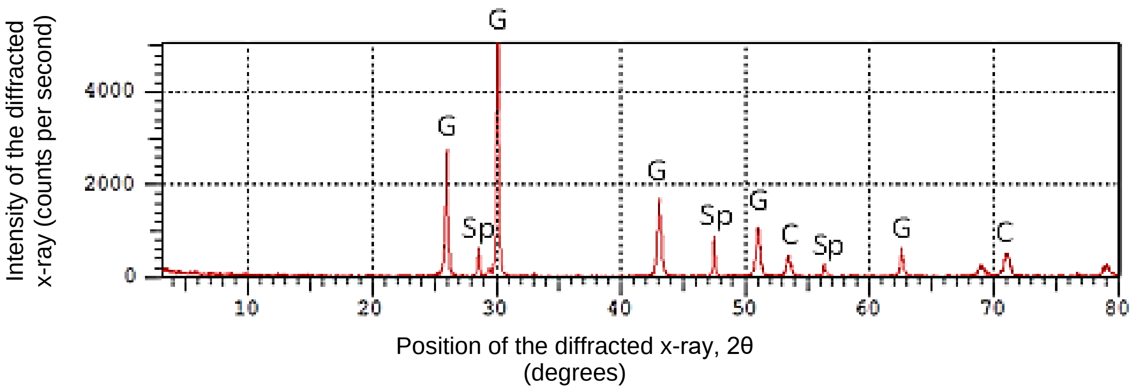
Element	Content (%)
Pb	66.6
Zn	7.38
Cu	0.84
Fe	2.79
S Total	17.62
Sb	<0.001
Mo	0.12
Co	<0.001

Concentrate milling and sieving were conducted to obtain leaching sample with particle size distributions of -100+150, -150+200, -200+325 and -325+400 mesh. The reactor for the leaching test was made from Teflon having a volume of 1 L and equipped by a condenser to maintain solution volume at a high temperature by condensating the vaporized water. Solution heating was done by MR Hei-Tec type of heater from Heidoph which was integrated with a mechanical stirrer. A series of leaching test works were carried out to study the effects of temperature, stirring speed, slurry density, acid concentration and particle size distribution of the lead extraction. Fluorosilicic acid with certain concentration, constant hydrogen peroxide of 9.8 M and 70 g of galena concentrate were prepared for every leaching test. In investigating the effect of one variable on the lead extraction, the other variables were kept constant. Prior to addition of the galena sample, one-fourth of the required H_2O_2 was added to the H_2SiF_6 solution. The rest of H_2O_2 was added periodically every 10 minutes during the leaching test to avoid the lack of dissolved oxygen due to H_2O_2 evaporation. Slurry samples were taken from the reactor at 5, 15, 25, 35, 75, 135 and 150 minutes after the start of leaching and then vacuum-filtered to separate the filtrate and residue. The filtrate was analyzed by AAS for measuring dissolved Pb and calculation of extracted lead percentages.

Table 3. Chemical composition of the galena concentrate sample (per fraction)

Component	Composition (%)			
	-100+150 mesh	-150+200 mesh	-200+325 mesh	-325 mesh
Pb	60.800	72.530	71.730	73.350
Zn	13.340	9.300	9.970	9.400
Cu	0.840	0.410	0.400	0.650
Fe	2.520	0.990	0.850	0.740
S	20.840	14.450	14.980	13.520
Ni	0.003	0.003	0.004	0.003
Co	0.028	0.028	0.029	0.029
Cd	0.092	0.081	0.084	0.081
Si	0.056	0.233	0.247	0.247
Al	0.013	0.005	nd*)	0.042
Ti	0.003	nd*)	0.001	0.009
Ca	nd*)	nd*)	nd*)	nd*)
K	nd*)	nd*)	nd*)	nd*)
Mn	0.020	0.017	0.017	0.018
Mg	0.039	0.027	0.019	0.035

nd*): not detected



G : Galena
Sp : Sphalerite
C : Chalcopyrite

Figure 1. XRD spectrum of galena concentrate sample

RESULT AND DISCUSSIONS

Pb extraction percentage was determined in this experiment by a formula:

$$\text{Pb extraction percentage} = \frac{\text{the concentration of Pb dissolved } \left(\frac{mg}{L}\right) \text{x } V_{solution}(\text{mL})}{\text{the percentage of Pb in the feed x feed weight (g) x 1000}} \text{ x } 100\% \qquad (1)$$

According to Anugrah, Mubarok and Amalia (2017), adding the H_2O_2 and increasing the leaching temperature enhanced lead extraction percentage. In this test works, the Pb extraction could reach a maximum level to 99.26% in 135 minutes then decrease to 94.95% in 150 minutes. This was obtained by a leaching test using 3.44 M of H_2SiF_6 (3 times stoichiometry), 9.80 M of H_2O_2, concentrate particle size -100+150 mesh (0.1 to 0.149 mm), slurry density of 12% and temperature of 97°C. Profiles of Pb extraction as a function of temperature and particle size distribution, in the absence and the presence of H_2O_2 are presented in Figure 2a and 2b, respectively. Dissolution of the lead from galena in fluorosilicic acid at the presence of hydrogen peroxide is as follows (Lee, Wethington and Cole, 1986):

$$PbS_{(s)} + H_2SiF_{6(aq)} + H_2O_{2(aq)} \rightarrow PbSiF_{6(aq)} + S^o + 2H_2O_{(aq)} \quad \text{... (2)}$$

In addition, parts of sphalerite and pyrite in the concentrate will be also dissolved through the following reactions:

$$ZnS_{(s)} + H_2SiF_{6(aq)} + H_2O_{2(aq)} \rightarrow ZnSiF_{6(aq)} + S^o + 2H_2O_{(aq)} \quad \text{...(3)}$$

$$FeS_{2(s)} + H_2SiF_{6(aq)} + H_2O_{2(aq)} \rightarrow FeSiF_{6(aq)} + 2S^o + 2H_2O_{(aq)} \quad (4)$$

Sulfur elemental was detected by XRD and SEM-EDX analyses of the leaching residue. SEM fotomicrograph that shows the presence of sulfur elemental in the leach residue is illustrated in Figure 3. In the absence of H_2O_2, the highest Pb extraction was only 58.3%, obtained at similar condition. At this condition, oxidation of sulfur from sulfide minerals relies on the dissolved oxygen concentration in the water used in leaching solution.

The decrease of lead extraction by the increase of temperature from 85 to 97 °C was observed for the leaching tests using H_2O_2 at particle size of -150+200, -200+325 and -325 + 400 meshes. This occurred due to the precipitation of the dissolved Pb in the form of anglesite ($PbSO_4$). At finer particle size of the concentrate, sulfur oxidizes to sulfate faster and resulted in precipitation of $PbSO_4$ rather than formation of soluble $PbSiF_6$. The formation of anglesite was confirmed by XRD analysis at which it detected the presence of anglesite peaks along with galena, sphalerite, sulfur and pyrite as can be seen in Figure 4. Without addition of H_2O_2, there was no anglesite ($PbSO_4$) formation presumably due to the lack of sulfate anions generated by oxidation of sulfur in sulfide minerals, in the absence of the oxidant. Anglesite formation can also be minimized by using complexing agents (Zárate-Gutiérrez, Lapidus and Morales, 2010).

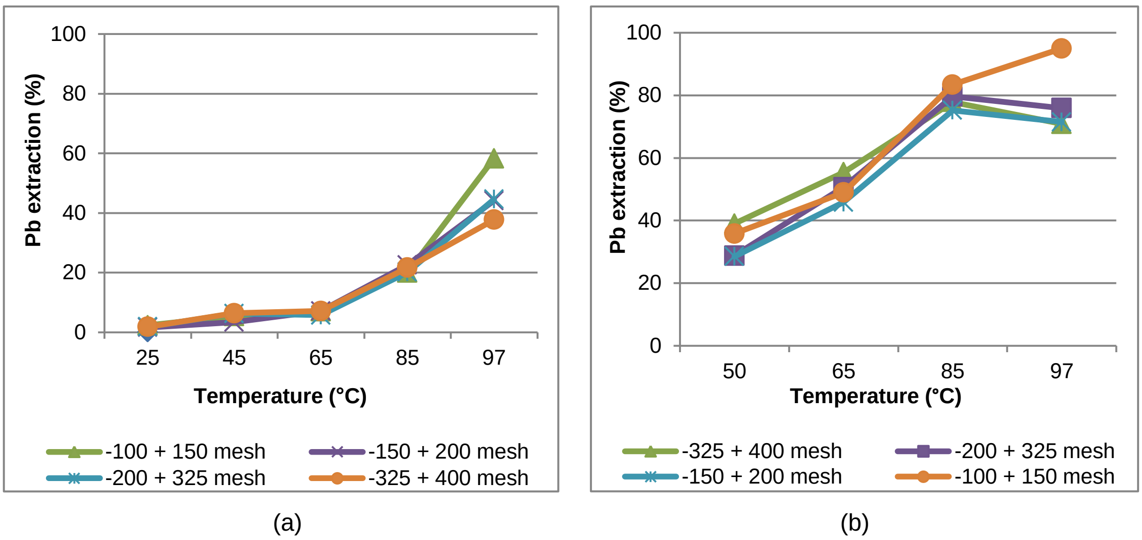
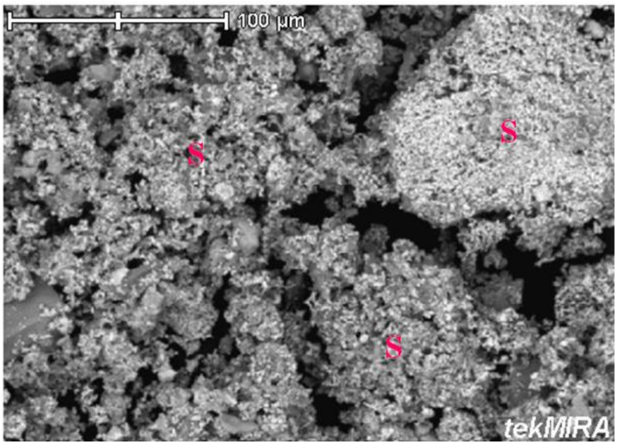
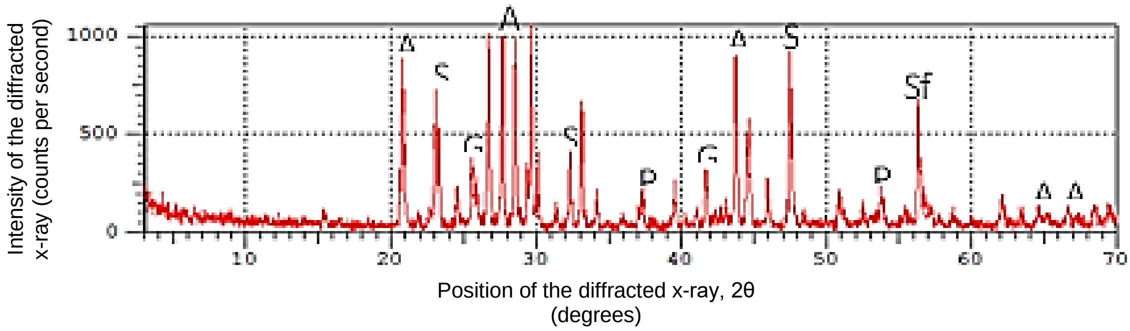


Figure 2. Profiles of Pb extraction at various temperatures and particle size distributions (3.44 M of H_2SiF_6, 300 rpm of stirring speed) (a) without H_2O_2 (b) with addition of 9.8 M of H_2O_2



S: Sulphur

Figure 3. SEM fotomicrograph shows the presence of elemental sulfur in the leaching residue (3.44 M of H_2SiF_6, 300 rpm of stirring speed, 12% of slurry density, temperature of 97 ^oC, particle size of -100+150 mesh)



A: Anglesite ($PbSO_4$), G: Galena, Sp: Sphalerite, S: Sulfur, P: Pyrite

Figure 4. XRD spectrum of leaching residue (3.44 M of H_2SiF_6, 9.80 M of H_2O_2, 300 rpm of stirring speed, 12% of slurry density, 97 ^oC of temperature, -100+150 mesh of particle size)

Effect of Stirring Speed

Sufficient stirring speed is required for a good contact between galena mineral and H_2SiF_6 as well as H_2O_2 (Anugrah, Mubarok and Amalia, 2017). In case of the leaching rate is controlled by the mass transfer, a stronger agitation at higher stirring speeds would reduce the thickness of diffusion layer on the surface of minerals that can enhance the leaching rate. The experimental results showed that the stirring speed did not affect significantly the Pb extraction percentage (Figure 5). The stirring speed only slightly influenced the lead extraction for the leaching of the coarse particle (-100+150 mesh), though it was only obvious on the increase of stirring speed from 100 to 300 rpm, while stirring speed variations in the range of 300-700 rpm did not give remarkable effect on the extracted Pb.

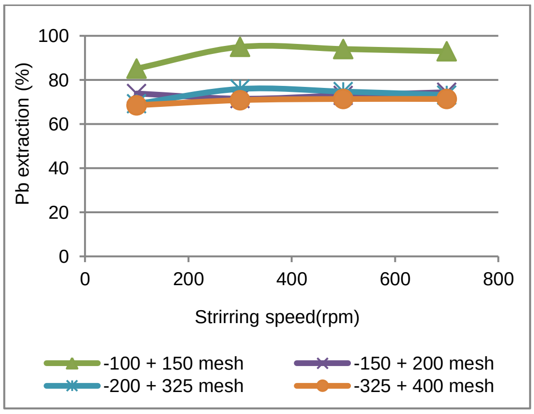


Figure 5. Profiles of Pb extraction percentage as a function of stirring speed at the various feed particle size (3.44 M of H_2SiF_6, 9.80 M of H_2O_2, 12% of slurry density, temperature of 97 ^oC)

Effect of Slurry Density (Solid Percentage)

Profiles of extracted Pb (%) as a function of time at various solid percentages are depicted in Figure 6. Extracted Pb increased by the decrease of slurry density and reached the best performance at 12% solid. The excessive solid percentage tends to reduce O_2 solubility required for sulfur in sulfide mineral oxidation and dissolution of lead (Jha *et al.*, 2012). Moreover, at lower solid percentage, slurry viscosity is lower and facilitating a better contact between leaching agent and the galena mineral and higher mobility of lead ions to move outward from interface of mineral-solution to the bulk of solution (Anugrah, Mubarok and Amalia, 2017).

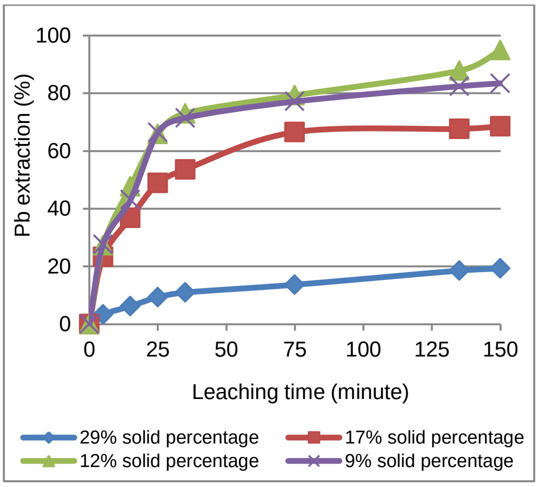


Figure 6. Profiles of extracted Pb (%) as a function of time at various solid percentages (3.44 M of H_2SiF_6, 9.8 M of H_2O_2, 300 rpm of stirring speed, temperature of 97 ^oC, particle size of -100+150 mesh)

Effect of Acid Concentration

Anugrah, Mubarok and Amalia (2017) showed that acid concentration affected the content of the extracted Pb. The leaching experiments to study the effect of the fluorosilicic acid concentration on Pb extraction were conducted at 12% of solid percentage and 9.8 M of H_2O_2, temperature of 97 ^oC, stirring speed of 300 rpm and particle size distribution of -100+150 mesh for 150 minutes. The fluorosilicic acid concentrations were varied at 0.5, 1, 2, 3 and 3.44 M.

Using a lower acid concentration, the extracted Pb was lower. The highest Pb extraction percentage (99.26%) was achieved in leaching experiment by 3.44 M of H_2SiF_6 concentration for 135 minutes. The effect of acid concentration on Pb extraction percentage is presented in Figure 7. Sufficient concentration of fluorosilicic acid is required to maintain solution at lower pH and stabilizes lead in its ionic state.

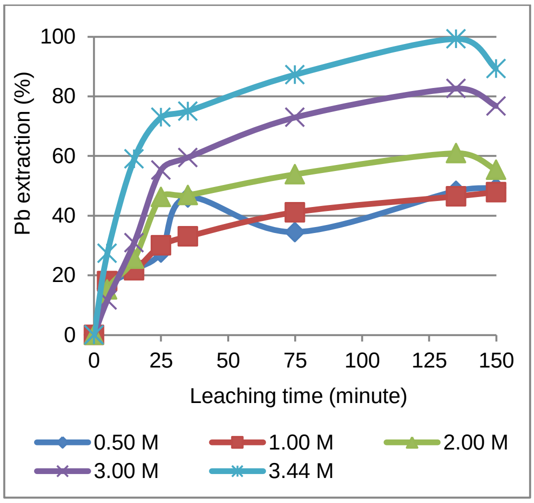


Figure 7. Profiles of extracted Pb as a function of time at various fluorosilicic acid concentration (300 rpm of stirring speed, temperature of 97 ^oC, particle size of -100+150 mesh)

Effect of Particle Size

Study the effect of particle size on Pb extraction percentage, the tests were carried out using 3.44 M of H_2SiF_6, 9.80 M of H_2O_2, 300 rpm of stirring speed at 97 ^oC for 150 minutes. The results revealed that the coarsest particle size of the concentrate (-100 + 150 mesh) gave the highest extraction percentage of Pb after 150 minutes (Figure 8). Leaching the finer concentrates with finer particle sizes (-325 + 400 mesh, -200+325 mesh and -150+200 mesh) provided a maximum Pb extraction after 75 minutes beyond which extracted Pb was slightly decreased. This phenomenon is associated with the anglesite ($PbSO_4$) precipitation as discussed earlier.

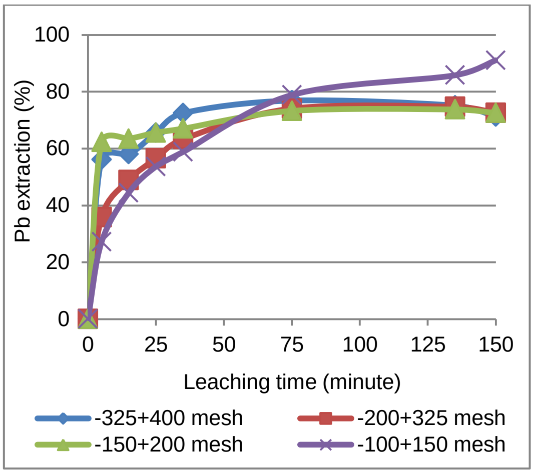


Figure 8. Profiles of the extracted Pb as a function of time at various particle size distribution of concentrate (3.44 M of H_2SiF_6, 9.80 M of H_2O_2, 300 rpm of stirring speed, temperature of 97 ^oC, 12% of slurry density)

CONCLUSIONS

The highest lead extraction of 99.26% was obtained by leaching the galena concentrate with particle size distribution of -100+150 mesh, using 3,44 M of H_2SiF_6 (3 times stoichiometry), 9.80 M of H_2O_2, 12% of slurry density at 97 ^oC after 135 minutes. At finer particle size distribution, higher temperature and longer periods of leaching, non-soluble anglesite ($PbSO_4$) tends to form that resulted in lower extracted lead. XRD analysis detected the presence of anglesite in the leaching residue along with the un-dissolved galena, sphalerite, pyrite and elemental sulfur. Variations of stirring speeds in the range of 300-700 rpm did not affect the Pb extraction in leaching testworks with 3.44 M of H_2SiF_6, 9.80 M of H_2O_2, 12% of slurry density at 97^oC after 150 minutes. At a constant H_2O_2 concentration of 9.80 M, the highest lead extraction after 150 minutes was obtained at slurry density of 12%.

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