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Evaluation of oxidic by-products as Neutralizing agents in Biooxidation of a Refractory Gold concentrate and their influence on Gold extraction through Cyanidation

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Abstract

The neutralization cost in bioleaching operations is one of the biggest operation costs and therefore the aim of the present study has been to replace the generally used lime/limestone with industrial oxidic by-products. A comparative study on the potential use of some selected industrial by-products as neutralizing agents during biooxidation and their influence on subsequent gold recovery was carried out with reference to a commercial grade $Ca(OH)_2$. The by-products used comprised of an electric arc furnace slag (EAF slag), and a slag from ladle refining (Ladle slag) both from scrap based steel production, an EAF dust and a lime sludge from paper and pulp industry (Mesa lime). Continuous biooxidation of a refractory gold concentrate was performed in single stage reactor at a retention time of 56 h with a mixed mesophilic culture. Biooxidation results as well as gold recoveries were good for all by-products investigated and similar to the results obtained with the slaked lime reference. However, cyanide consumption was elevated in the experiments with steel slags and the EAF dust partly because of a higher content of S° in the bioresidues in these experiments. It is however expected, that in a bioleaching operation with several reactors in series, that sulfur oxidation would be more complete, thereby possibly decreasing cyanide consumption.

Keywords: EAF dust, Mesa lime, Ladle slag, EAF slag, neutralization, biooxidation, cyanidation, microorganisms.

Introduction

Biomining is today a well-established technology which is used for extraction of metal values from sulfide ores and concentrates using microorganisms. Biomining is carried out either in stirred tanks or as heap leaching operations. Continuous stirred tank biooxidation is mainly applied as a pre-treatment step for the recovery of Au and Ag from refractory gold concentrates but is also applied in one plant for the recovery of Co from a cobaltiferrous pyrite, whereas heap bioleaching is used for Cu recovery from low-grade secondary copper sulfides (CuS and Cu_2S)[1-3]. Competing technologies for pre-treatment of refractory gold concentrates for Au and Ag recovery by cyanide leaching are roasting and pressure leaching where the latter is the dominating technology[3]. Biooxidation has proven to be economically competitive as well as environmentally friendly compared to the conventional methods and is therefore gaining market shares[5-7]. Gold field's proprietary BIOX™ technology and Canadian-based BacTech Mining Company's BACOX process are widely used today for bioprocessing of refractory gold concentrates[6]. In the biomining technology autotrophic and acidophilic mesophilic and moderate thermophilic microorganisms are utilized. Biooxidation plants for treatment of refractory gold concentrates have reported higher cyanide consumption compared to when roasting and pressure leaching techniques are used which influences the operation costs negatively[8]. Biooxidation of refractory gold ores or concentrates is normally an acid producing process (equation 1 and 2).

$$2FeAsS+Fe_2(SO_4)_3+6O_2+4H_2O \rightarrow 4FeSO_4+2H_3AsO_4+H_2SO_4 \quad (1)$$

$$FeS_2 + Fe_2(SO_4)_3 + 3O_2 + 2H_2O \rightarrow 3FeSO_4 + 2H_2SO_4 \quad (2)$$

The acid produced is generally neutralized by using limestone as neutralizing agent to the desired pH level of 1-2. The residue obtained after S/L separation is cyanide leached to recover Au and Ag, while the leach liquor is neutralized to precipitate Fe/As and other elements. The cost of neutralization in biooxidation plants is known to be the second highest operational cost, for which cheaper neutralizing agents like dolomite, ankerite or calcrete (a low-grade limestone) are used at various plants to improve the process economy[9]. The aim of the present investigation has been to study the possibilities to use oxidic by-products like slag, dust generated in the steel industry and lime sludge from the paper and pulp industry as neutralizing agents during biooxidation of a refractory gold concentrate. Eventual toxic effects of the by-products on the microorganisms, biooxidation efficiency together with Au and Ag recoveries after cyanide leaching were evaluated.

Material and Methods

Refractory gold concentrate: The refractory gold concentrate used originated from the Petiknäs North mine of Boliden Mineral AB. The concentrate contained 10.8 g Au/ton, 91 g Ag/ton, 34.9% Fe, 39.9% S, 10.2 % As and 5% S (table 1). The relative mineralogy was calculated based on the elemental composition of the concentrate resulted in 55.2% FeS_2, 22.2% FeAsS, 2.0% $CuFeS_2$ and 1.6% FeS. X-Ray diffraction (XRD) studies on the concentrate revealed the presence of FeS_2, FeAsS, ZnS, FeS and SiO_2. Previous studies have shown that the majority of the gold and silver is present in arsenopyrite and a minor part in pyrite.

Neutralizing agents: The slags used were an electric arc furnace slag (EAF slag) and a slag from ladle refining (Ladle slag) both originating from scrap based steel production. Both slags were disintegrating meaning that they upon cooling formed a powder. Moreover, a dust from the gas cleaning system of an electric arc furnace (EAF dust) and a lime sludge produced in paper and pulp industry (Mesa lime) were studied while calcium hydroxide ($Ca(OH)_2$) was used as reference material. To increase reactivity the slags and the EAF dust were ground in a ring mill. The particle size analysis of the final grind size via laser classification (Cilas 1064 liquid) expressed as 80% passing (d_{80}) were 4.8 µm for EAF dust, 15 µm for EAF slag, 18 µm for Ladle slag, 20 µm for Mesa lime and 8.5 µm for $Ca(OH)_2$[10] . The chemical composition of the by-products is given in table 1. As can be seen, the Ca content in the by-products varied from 13% for the Ladle slag up to 38% in the Mesa lime with 54% in the reference $Ca(OH)_2$. The Si content in the steel slags was 6-7% whereas the Si content in the other materials was in the range 0.1-1.4%. Some outstanding analysis are the high Al content in the Ladle slag (13.4%) and the high Zn content in the EAF dust (26.5%). The by-products used are well characterized which has been described in an earlier work[10] and their mineralogy together with $Ca(OH)_2$ is given in table 2. Chloride is known to be toxic for bioleaching microorganisms and therefore a pre-leaching with water was done on the EAF dust to reduce its relatively high chloride content. The pre-leaching step removed 91% of the chloride in the dust.

Table-1
Elemental composition of feed materials and bioresidues

		Si	Al	Ca	Fe	Mg	Mn	As	Pb	S	Zn	F	Au	Ag
		%	%	%	%	%	%	%	%	%	%	%	g/ton	g/ton
Concentrate	Refractory gold	5.0	0.8	0.7	34.9	0.6	0.03	10.2	0.5	39.9	3.4	NA	10.8	91
Neutralizing agent	$Ca(OH)_2$	0.1	0.0	53.6	0.0	0.4	0.01	0.0	0.0	0.02	0.0	0.0	-	-
	Mesa lime	0.1	0.1	38.2	0.1	1.0	0.14	0.0	0.0	0.03	0.0	0.0	-	-
	EAF dust	1.4	0.4	13.3	22.4	1.0	2.9	0.0	0.7	0.50	26.5	0.1	-	-
	Ladle slag	5.9	13.4	29.9	2.4	8.6	0.2	0.0	0.0	0.34	0.05	0.5	-	-
	EAF slag	7.0	3.0	31.2	19.8	1.9	2.3	0.0	0.0	0.06	0.01	0.01	-	-
Bioresidue	$Ca(OH)_2$	7.5	1.1	8.2	17.8	0.7	0.02	2.0	0.2	32.1	1.9	-	-	-
	Mesa lime	8.5	1.3	9.2	15.3	0.8	0.01	1.4	0.2	29.7	1.4	-	-	-
	EAF dust	8.0	1.3	5.5	22.8	0.9	0.68	1.4	0.6	25.6	3.4	-	-	-
	Ladle slag	10.1	1.6	7.0	16.0	1.0	0.0	1.5	0.4	27.9	1.5	-	-	-
	EAF slag	7.0	1.0	8.7	18.5	1.1	0.6	1.6	0.3	19.3	1.5	-	-	-

Table-2
Mineralogical phases identified in feed materials and bioresidues

Material		Mineralogical phases
Concentrate	Refractory gold	pyrite (FeS_2), sphalerite (ZnS), quartz (SiO_2), arsenopyrite ($FeAsS/FeS_2.FeAs_2$)
Neutralizing agent	$Ca(OH)_2$	portlandite ($Ca(OH)_2$)
	Mesa lime	calcite ($CaCO_3$)
	EAF dust	calcium zinc hydroxide hydrate ($Ca(Zn(OH)_3)_2{\cdot}2H_2O$), franklinite ($ZnFe_2O_4$), zincite (ZnO), magnetite ($FeFe_2O_4$)
	Ladle slag	calcium silicate (Ca_2SiO_4), gehlenite ($Ca_2Al_2SiO_7$), mayenite ($Ca_{12}Al_{14}O_{33}$), periclase (MgO)
	EAF slag	calcium iron oxide ($CaFe_2O_4$), calcium magnesium aluminum iron silicate ($Ca_2Mg_{0.2}AlFe_{0.6}Si_{0.2}O_5$), calcium silicate ($Ca_2SiO_4$), wuestite (FeO)
Bioresidue	$Ca(OH)_2$	gypsum ($CaSO_4{\cdot}2H_2O$), pyrite (FeS_2), quartz (SiO_2)
	Mesa lime	pyrite (FeS_2), quartz (SiO_2), gypsum ($CaSO_4{\cdot}2H_2O$)
	EAF dust	pyrite (FeS_2), Quartz (SiO_2), franklinite ($ZnFe_2O_4$), bassanite ($CaSO_4{\cdot}0.5H_2O$)
	Ladle slag	bassanite ($CaSO_4{\cdot}0.5H_2O$), gypsum ($CaSO_4{\cdot}2H_2O$), pyrite (FeS_2), quartz (SiO_2)
	EAF slag	bassanite ($CaSO_4{\cdot}0.5H_2O$), pyrite (FeS_2), quartz (SiO_2)

Microorganisms: The microbial inoculum used was a mixed mesophilic culture of acidophiles comprising of both iron and sulfur oxidizing bacteria and together with some archaea. *Leptospirillum ferriphilum* was found to be the dominant species followed by *Acidithiobacillus caldus* and similar number of *Acidithiobacillus thiooxidans*, *Sulphobacillus sp.* and *Ferroplasma sp.* determined by Q-PCR at Bioclear B.V., Netherlands[11-12].The microbial culture was maintained in a continuous culture at a dilution rate of 0.021 h^{-1} in a modified 9K nutrient medium ($(NH_4)_2SO_4$, 3.0 g/L; KCl, 0.1 g/L; K_2HPO_4, 0.5 g/L; $MgSO_4 \cdot 7H_2O$, 0.5 g/L; $Ca(NO_3)_2 \cdot 4H_2O$, 0.01 g/L) supplemented with 4.5 g/L Fe^{2+} and 2 mM potassium tetrathionate[13]. Temperature and pH in the culture were maintained at 37 ºC and 1.50 ± 0.05, respectively. The redox potential in the culture was 740 ± 5 mV versus the Ag/AgCl reference electrode.

Analytical techniques: The redox potential was measured with a platinum electrode against the Ag, AgCl reference electrode and dissolved oxygen (D.O.) was measured with a Lange LDO^{TM}/sc100. The Fe, As, Zn concentrations were analyzed by atomic absorption spectroscopy (AAS). The elemental composition of feed materials, residues and leachate was analyzed by combinations of Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) / Quadrupole Mass Spectrometry (ICP-QMS) / Sector Field Mass Spectrometry (ICP-SFMS). The mineralogy of solid samples was determined by X-Ray powder diffraction (XRD) on a Siemens D5000 automatic diffractometer with Cu Kα radiation of 40 kV and 30 mA with a sample rotation of 30 rpm. The diffraction patterns were measured in the 2ɵ range of 10°-90°, while the crystalline phases were identified by the Joint Committee for Powder Diffraction Standards (JCPDS) database of the instrument.

Biooxidation and Cyanidation: The biooxidation experiments were conducted in a single stage 5 L baffled bioreactor at a temperature of 36-37 ºC. Air enriched with 2-3% (v/v) CO_2 was added under the stirrer at a flow rate of 5 L/min and the D.O. level in the reactor was 3-5 mg/L in the experiments. Each experiment started in batch mode with 1-2 % solids (w/w) and 20% inoculum (v/v) until a redox potential of 600 mV was reached. At that time continuous pumping of a feed pulp containing 10%-solids (w/w) started with gradual increase of the flow-rate to attain the desired retention time of 55±1 hours. Both the feed and discharge from the reactor was pumped intermittently with peristaltic pumps 1-2 times per hour. To ensure a constant working volume in the reactor approximately 10% of the oxidized pulp was discharged via over-flow. When active biooxidation was obtained and the pH decreased from the desired level of 1.5 ± 0.1 addition of neutralizing agents was done. Steady state was considered to be achieved after undisturbed operation of 11-12 days, i.e. 5 times the retention time. During steady state the oxidized pulp was collected and filtered and the residues were dried and stored for subsequent cyanide leaching tests. Finally, the biooxidation experiments were terminated by harvesting the whole content of the reactor. S/L separation was achieved via vacuum filtration and the residues were thoroughly washed with acidified (H_2SO_4) deionized water. Leachates and dried residues were then sent for analysis. Sulfide mineral oxidation was calculated based on the particular elemental content in the feed and residue. Regarding oxidation of Fe care was taken to include the part that had been leached and later precipitated as $Fe(OH)_3$ and $XFe_3(SO_4)_2(OH)_6$ $X^+ = K^+, Na^+, H_3O^+, NH_4$ by dissolving the residues in 6 M HCl. The Fe content originating from the by-products that dissolved at pH 1.5 determined in a previous study[14] was also taken into account in the calculations.

The dried residues obtained were gently ground in a mortar and divided prior to cyanide leaching. The cyanidation tests were carried out in glass vessels equipped with a motor driven stirrer at a pulp solid content of 25% (w/w). Due to the limited amount of bioresidues available the residues were diluted with inert silica sand at a ratio of (1:4), 100 g residue to 400 g silica sand. A free cyanide concentration of 0.03-0.1% NaCN (v/v), determined through a titrimetric method with $AgNO_3$ as titrant and rhodanine as indicator, was maintained during the experiments. Quick lime was added, whenever required to maintain the required pH of 10-11. Aeration was achieved by the vortex action of the agitators. Duration of the cyanide leaching experiments varied between 52-75 hours. The leach liquors were analyzed for SCN^- content by UV-VIS spectrophotometry through a colorimetric method, while the leach residues were analyzed for Ag and Au by the fire assay method.

Results and Discussion

Biooxidation: The pH was adjusted twice daily to the desired level of 1.5 by additions of the different neutralizing agents. Due to the irregular additions and high microbial activity pH during nights decreased down to 1.3. Depending on mineralogy of the different neutralizing agents used various reactions were responsible for neutralization of the acid produced during biooxidation, some examples are given below:

$Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 + 2H_2O$ (slaked lime) (3)

$CaCO_3 + H_2SO_4 \rightarrow CaSO_4 + H_2O + CO_2$ (mesa lime) (4)

$ZnO + H_2SO_4 \rightarrow ZnSO_4 + H_2O$ (EAF dust) (5)

$Ca_2SiO_4 + 2H_2SO_4 \rightarrow 2CaSO_4 + 2H_2O + SiO_2$
(ladle slag/EAF slag) (6)

Due to the limited solubility of Ca^{2+} ions in SO_4^{2-} environment $CaSO_4$ precipitates were found in all bioleached residues either as gypsum or bassanite table 2. Silicates present in some of the by-products also have neutralizing capacity as is exemplified with Ca_2SiO_4 in equation 6. Some of the elements found in silicates and oxides are soluble under these conditions and therefore reports into the leaching solution, examples are Fe, Mg, Al and Zn. This is seen in the leach liquor from the experiment where Ladle slag was used for neutralization, where elevated levels of Al and Mg was obtained due to the dissolution of the $Ca_2Al_2SiO_7$ and $Ca_{12}Al_{14}O_{33}$ together with MgO contained in that slag (tables 2 and 3).

In the leachate from the EAF dust experiment an increased level of Mn is seen. The concentration of Mn in the EAF dust was 2.9% (table 1), despite this; minerals containing Mn were not detected in the dust. In EAF dust Mn is present to a smaller extent as MnO, which is soluble under the conditions employed, while the major part usually is found as a solid solution in spinels like franklinite ($ZnFe_2O_4$) with the general formula $(Mn_xZn_yFe_{1-x-y})Fe_2O_4$ and is therefore difficult to detect by XRD[15]. The EAF dust had a high concentration of Zn (27%) of which a large part was in the form of soluble ZnO and the remaining in insoluble $ZnFe_2O_4$. The concentration of zinc at steady state operation was 9.5 g/L (table 3) which shows that in a bioleaching process for Zn recovery from ZnS, EAF dust would be an excellent choice for neutralization, since it in addition also enhances the Zn tenor in the leachate. However, it should be realized that elements like Al, Mn and Zn, unless they are meant to be recovered, have to be neutralized in a later stage before the final effluent is released into the environment. This implies that the savings obtained in neutralization costs during biooxidation, when by-products containing soluble and unwanted elements are used, is lost during effluent treatment since then slaked lime has to be used for their precipitation.

In figure 1 the trend in iron concentration and redox potential during the duration of the experiments is given. Slight variations in both Fe concentrations and also in redox potentials (figure 1) are to a large extent due to the irregular additions (twice daily) of neutralizing agents. Fe concentrations in the final leachate collected varied from 25 up to 37 g/L while As concentrations ranged between 11 and 13 g/L.

It is interesting to note that the highest redox potentials were obtained in the experiments using slags and EAF dust for neutralization with values ranging between 623-632 mV while the redox potential in the experiments with Mesa lime and $Ca(OH)_2$ both had a redox potential of 604 mV (table 4). The high redox potentials obtained proves that the by-products used did not impose toxic effects on the microorganisms used, at least this is true for the iron oxidizing bacteria. Fluoride is also an element known to be highly toxic for the microorganisms used in bioleaching despite this the relatively high F content in the EAF slag (0.5%) did not cause any negative effect on the microbes. The most probable reason for this is that the Al ions released into the leachate formed complexes with the F ions and thereby efficiently reduced the F toxicity which has been shown to be effective in previous work[16-17].

Table-3
Elemental composition (g/L) of final bioleach liquors

	Si	Al	Ca	Fe	K	Mg	Mn	Na	As	Cr	Cu	Pb	Zn	SO_4^2
$Ca(OH)_2$	0.1	0.0	0.7	25.0	0.3	0.2	0.0	0.0	10.9	0.00	0.3	0.5	2.1	53.3
Ladle slag	0.5	2.4	0.6	36.8	0.1	1.7	0.1	0.1	13.2	0.01	0.4	0.5	3.3	96.2
EAF slag	1.4	1.0	0.7	35.0	<0.1	0.2	0.1	0.1	12.5	0.04	0.3	0.3	3.0	79.4
Mesa lime	0.1	0.0	0.6	27.5	0.4	0.3	0.1	0.2	11.0	0.00	0.3	0.4	2.4	59.9
EAF dust	0.3	0.1	0.6	36.3	0.2	0.3	0.4	0.1	13.0	0.07	0.3	0.5	9.5	84.5

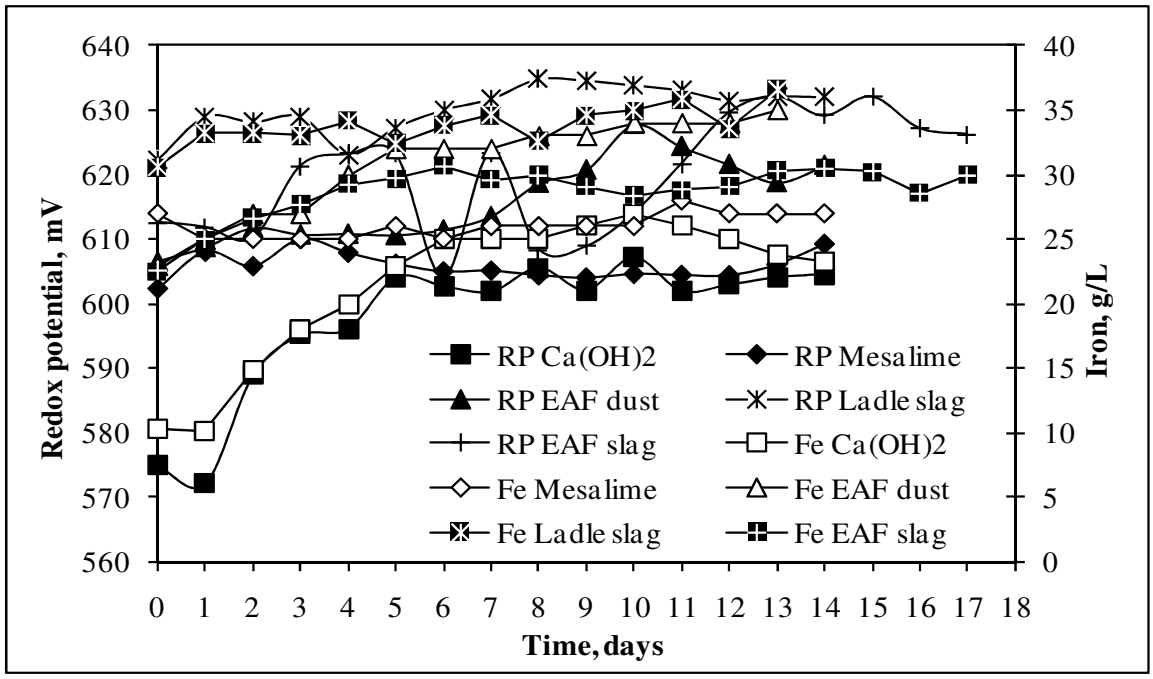

Figure-1
Redox potential (RP) and iron (Fe) concentration during biooxidation experiments

The neutralization potential of the by-products was determined from the amount required to maintain the pH at 1.5 during steady state operation. The EAF dust had the lowest neutralizing capacity, which required 296 kg per ton of concentrate fed to the bioreactor closely followed by the EAF dust with a requirement 267 kg/ton feed (table 4). The highest neutralizing capacity was obtained for the $Ca(OH)_2$ reference with 110 kg/ton feed needed, while the requirement for Mesa lime and Ladle slag was 141 kg/ton and 152 kg/ton, respectively (table 4). The amount of bioresidue generated was 1056 kg/ton concentrate for EAF slag and was the highest among all, while the ladle slag experiment generated the lowest amount, i.e. 687 kg/ton feed. The quantity of bioresidue produced was not in proportion to the amount of by-products added due to the presence of soluble elements in some of the by-products, as previously discussed. The big amount of bioresidue obtained in the EAF slag experiment is probably exaggerated as this slag generated fine clay-like particles that at times blocked the pumping tubes, resulting in accumulation of solids in the reactor and in addition also created filtration problems.

The oxidation of FeAsS in the experiments ranged from 83-90%, while the FeS_2 oxidation varied between 63-76% (table 5). Zn recoveries of 55-76% were lower than what normally is experienced but the reason is that part of ZnS in this concentrate is interlocked within the FeS_2 matrix[11].

It's well known that otherwise in a mixed sulfide system, the sulfide with higher electrochemical rest potential (FeS_2) acts as a cathode and the one with lower rest potential (ZnS, FeAsS) as anode resulting in faster dissolution due to galvanic interaction [18] . The extent of FeAsS oxidation observed was considered to be good for a single stage reactor operating at a retention time of 55 hours which of course could be further enhanced if done with normally used 2-3 stage reactor series with longer retention time. Regarding biooxidation performance Mesa lime, which essentially consists of calcite, could directly be used as a replacement for limestone in a full scale operation. The other by-products also performed well in biooxidation but since the microorganisms are autotrophic it would be better to mix these by-products with limestone in such proportions that the microbial CO_2 requirement is fulfilled. The S° content in the bioresidues were significantly higher for the slags and EAF dust compared to Mesa lime and slaked lime, this might be an indication that the S-oxidizers in the culture used might experience some difficulty when these materials were used.

Table-4
Summary of results during steady state operation (average over 5 days)

	$Ca(OH)_2$	**Mesa lime**	**EAF dust**	**Ladle slag**	**EAF slag**
Redox potential (mV, vs. Ag/AgCl)	604	604	623	632	629
By-product requirement (kg/ton feed)	110	141	296	152	267
Dry residues generated (kg/ton feed)	796	778	796	687	1056

Table-5
Summary of biooxidation and cyanidation results

Biooxidation					
Bioresidue origin	**Slaked lime**	**Mesa lime**	**EAF dust**	**Ladle slag**	**EAF slag**
Retention time (hours)	56	58	58	56	55
Pyrite oxidation (%)	67.8	73.6	63.4	76.1	71.4
Arsenopyrite oxidation (%)	85.4	89.8	89.2	89.8	83.1
Leaching yield zinc (%)	57.5	69.2	75.6	70.3	54.9
Bioresidue analysis					
S_{tot} (%)	32.1	29.7	25.6	27.9	19.3
S° (%)	1.74	1.97	3.33	3.33	2.60
Ratio bioresidue/feed (w/w)	0.79	0.78	0.80	0.69	1.06
S° (kg/ton feed)	13.9	15.3	26.5	22.9	27.5
Cyanidation					
Cyanidation time, hours	52	52	72	72	72
Cyanide leach residue grade, g Au/ton feed	1.19	1.01	1.59	1.37	1.48
Cyanide leach residue grade, g Ag/ton feed	24.7	13.2	29.5	38.5	35.9
Gold extraction (%)	88.9	90.6	86.9	87.3	86.3
Silver extraction (%)	72.9	85.5	69.5	57.7	60.5
NaCN consumption, kg/ton feed	10.9	11.4	15.9	20.0	31.5
SCN formation, kg/ton feed	4.10	4.34	5.82	9.38	5.82
NaCN losses as SCN, kg/ton feed	3.46	3.74	5.01	7.92	4.91
NaCN losses as SCN, % of total consumption.	31.9	31.8	31.5	39.5	15.6

Conclusion

It has been shown that all the oxidic by-products tested in this investigation effectively could be used as neutralizing agent during biooxidation of a refractory gold concentrate. Sulfide oxidation as well as final gold recovery after cyanide leaching on bioresidues was similar to what was obtained in the experiment with $Ca(OH)_2$ that served as reference material. The addition of by-products did not seem to have any negative impact on the Fe-oxidizing microbes since the redox potential was similar or higher when the by-products were introduced compared with the reference experiment. A higher content of S° in the bioresidues from experiments with steel slags and EAF dust might however be an indication that S-oxidizing microbes were slightly affected. On the negative side is a higher cyanide consumption which in the case of the slags were 2-3 times as high compared to what was obtained in the experiments with the $Ca(OH)_2$ (reference alkali) and Mesa lime from paper and pulp industry. It is believed that under more industrial like biooxidation conditions, i.e. with at least 3 reactors in series, that the S oxidation might be more efficient and that thereby the cyanide consumption could be decreased. However, further studies are needed in order to verify that.

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