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INFLUENCE OF APPLIED MINERALOGY IN DEVELOPING AN OPTIMAL HYDROMETALLURGICAL PROCESSING ROUTE FOR COMPLEX SULPHIDE ORES

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The selection of an optimum technical and economic processing and extraction route for minerals and metals requires complete knowledge of the ore, especially its chemical and mineralogical compositions, relative amounts, and grain size distribution. Thus, to obtain optimum results during base metals recovery from complex sulphide ore, processing must start from a sound and complete mineralogical study. This work therefore investigates the influence of applied

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mineralogy in the selection of the optimal route for processing and extracting base metals from complex sulphides, by studying the size, mineralogy, and elemental distribution of Ishiagu bulk complex sulphide ore. Bulk complex sulphide ore from Ishiagu, in Eboyin State, in the southeastern part of Nigeria, was sequentially crushed in a jaw crusher and a cone crusher, and ground in a rod mill. The results obtained during laboratory sieve analysis were used to evaluate its size distribution. Identification of mineral phases distributed within the sizes was determined by microscopy, using a scanning electron microscope coupled with energy dispersed x-ray analyzer to produce backscattered images. The elemental distribution was determined by the optical emission spectrometry using x-ray fluorescence and inductively coupled plasma-optical emission spectrometer. Results obtained showed variations in elemental and mineralogical composition within the different sizes. The concentrations of zinc, copper, and iron reduce as particle size decreases, while silicon, sulphur, and lead contents increase. The overall results obtained were used as a basis for predicting parameters for which optimal hydrometallurgical recovery of the constituent's metals could be achieved.

Keywords: complex sulphide ores, applied mineralogy, minerals processing, base metal extraction

Complex sulphide ores are extremely complicated mineralogical associations, basically of chalcopyrite, galena, and sphalerite finely disseminated in a matrix of pyrite (Gomez et al. 1997), usually composed from the minerals pyrite (FeS_2) and arsenopyrite ($FeAsS_2$). They are generally made up of fine intergrown minerals, in which precious metals such as gold and silver often occur as interlocked refractory and finely disseminated metals in them. They usually occur as hydrothermal deposit; as epigenetic—skarn and/or vein, and syngenetic—volcanic massive sulphide (VMS) or sedimentary massive sulphide (Sedex).

Whatever the form in which the different elements occur, they usually are very difficult to process (Deveci et al. 2004; Rubio and Frutos 2002). This may be due to the close similarities in their mineralogical properties, which hinders their suitability for conventional methods of processing. Due to their poor electromagnetic properties, they are unsuitable for the magnetic method of separation, while the closeness in their specific gravities limits their suitability for the gravity concentration and heavy medium separation methods. Moreover, the metal values in the sulphide minerals are prevented from being chemically

modified by reagents during flotation and are hindered from chemical attack during leaching. In the hydrometallurgical process for treating and extracting metals from these ores, it is observed that sulphide ores do not allow the recovery of metal by direct chemical leaching (Dutrizac 1989; Hiskey and Wadsworth 1975) because the sulphides are insoluble in nearly all reagents. For the metal content to be leached either through the chemical or even the bioprocess, the reagent/organism must come into direct contact with metal atoms or metal-containing compounds within the mineral ore.

An approach to achieving this is to thoroughly liberate all the mineral phases in order to enable them to be exposed to chemical attack. A limitation to grinding many sulphide ores is that the ore cannot practically be ground down fine enough to expose the metals. For instance, chalcopyrite and sphalerite are frequently intergrown, with micro-size grains of 10–20 μm being dispersed within the pyrite (Gomez et al. 1999). Therefore, due to these specific mineralogical characteristics, it is necessary to finely grind and concentrate the ore prior to the solubilization of the valuable metals (Barbery et al. 1980). However, the crushing and grinding of ore is a significant capital and operational cost in many mineral-processing plants. According to Bilgili and Scarlett (2005), size reduction is an expensive and energy-inefficient process, however operated. Considering these factors, a small gain in comminution efficiency can have a large impact on the operating cost of a plant, while conserving resources as well (Fuerstenau et al. 1999). Hence, it is important to fully determine the comminution parameters that are relevant to the crushing and milling of an ore to enable complete plant design to take place.

The increase in the complexity during the processing of complex sulphide ore and their cost implications has put a formidable challenge to the process engineer in designing suitable recovering routes and the operation of metallurgical plants. These complexities therefore necessitate a detailed mineralogical characterization of such ores in determining an optimal processing route for its constituent minerals and metals. Detailed mineralogical analysis plays an important role in overcoming incorrect assumptions that may have disastrous consequences during process design. Mineralogical characterization provides a sound background in understanding the behavior of the minerals during beneficiation and the designing of optimal beneficiation routes. It also provides insights and information on the type, nature, and amount of minerals

and elements present within the ore, and the mineralogical reason(s) governing the metal-recovery processes.

This process of applying mineralogical information to understanding and solving problems encountered during the processing of ores and concentrates is referred to as applied mineralogy (Petruk 2000). It involves characterizing minerals and interpreting the data with respect to mineral processing (Xiao and Laplane 2004). This data guides the process engineer in the determination of the optimal processing and extraction route. This article therefore studies the size and the mineralogical and elemental distribution of Ishiagu bulk complex sulphide ore and interprets the mineralogical data to predict parameters for which optimal hydrometallurgical recoveries of base metals could be obtained from the ore.

MATERIALS AND METHODS

Complex sulphide ore obtained from Ishiagu in Ebonyin state, Nigeria, was used for this study. Morphological and qualitative analyses of the bulk ore were performed using scanning electron microscopy (SEM) equipped with energy dispersive X-ray (EDX) spectrum (SEM-EDX technique). The ore was stage crushed in the laboratory in a jaw crusher and later by a cone crusher. Ore grinding was carried out in a rod mill at varying rod mill parameters. Ten rods of 460 g each were used for the grinding. The grinding time and the amount of feed ore varied, as shown in Table 1. Size analysis of the ground products from each of the varied rod mill parameters were carried out to determine the quality of each grinding parameter using the sieve analysis method in a laboratory test sieve. After the sieve analysis for the different milling routes, the same

Table 1. Sizes reduction processes

	Milling parameters		
Milling route	No. of rods	Amounts of ore (g)	Time (s)
A	10	1000	5
B	10	1000	10
C	10	1000	15
D	10	2000	15
E	10	500	15

amount of ore samples of the size fractions from each of the grinding routes were mixed together. Ten grams each of the homogenized particles of sizes of –53, 53, 75, and 106 μm, were mixed together, further ground to a powder, and 2 g of the homogenized sample was subjected to elemental analysis to give the composition of the bulk ore total. Identification of mineral distribution within the sizes was determined by x-ray diffractometry using Philips PW 1830 x-ray diffractometer with a Cu-anode. Mineral liberation pattern within the sizes was determined using the SEM model JEOL 840, combined with EDX analysis to produce backscattered images (BSI). Quantitative analysis of the elemental distribution was determined by x-ray fluorescence with Magi ″X Pro XRF spectrometer at 4 KV, using IQ^{+} "Standard less" analysis and optical emission spectrometry using the inductively coupled plasma—optical emission spectrometer (ICP-OES) model SPECTRO CIRO.

RESULTS AND DISCUSSION

Mineral Phases, Morphology, and Liberation

Major mineral phases identified through the x-ray diffraction include siderite, sphalerite, galena, quartz, and traces of chalcopyrite. The sphalerite in the ore occurs as sphalerite ferrous while the chalcopyrite occurred as a chalcopyrite group mineral with chemical formula Cu_2MnSnS_4. Rietveld analysis indicates that the ore contains 42% siderite, 35% sphalerite, 11% galena, and 8% quartz. The percentage composition for the chalcopyrite group mineral could not be accurately determined by the Rietveld analysis. This might be due to the fact that the relative amount is less than 2% (Kile and Eberl 2000).

Figure 1 shows different morphologies of the bulk complex ore examined directly without polishing by SEM. The galena-rich phase shows the characteristic galena cleavage and the box-shaped polyhedral habit, with small grains. These grains with the characteristic cleavage and crystal habit might have an effect on strength and could therefore enhance easy breakage during comminution. The fine-grained massive morphologies of the sphalerite-rich phases with flat surfaces and their distinct crystallographic planes shown in Figure 1E and 1F also determine and indicate their flattened characteristics during breakage.

The mineral phases identified by SEM with EDX analysis through BSI are as shown in Figure 2. The ore is typically made up of fine to coarse grains intergrowths of the constituent crystalline phases both at

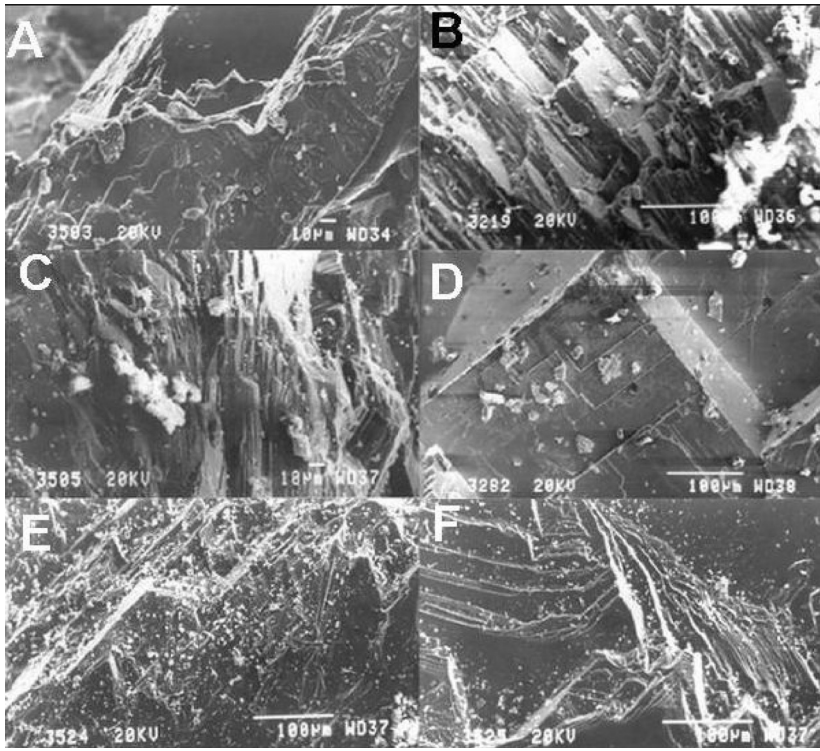

Figure 1. SEM micrographs of the bulk ore showing different mineral morphologies. (A) growth morphology of siderite; (B and C) growth morphologies of complex phases of galena and quartz grains; (D) galena-rich phase with its characteristic cleavage and box-shaped polyhedral habit, with some small grains; (E and F) fine-grained massive morphologies of the sphalerite-rich phases with flat surfaces and their distinct crystallographic planes.

the interstitials and the boundaries. Although there are several intergrowths among the mineral phases, the fine to coarse structure of the phases is expected to ease the liberation of the constituent mineral phases. The major mineral phases are constituted essentially by siderite, sphalerite, galena, quartz, and chalcopyrite group minerals. Other minerals occur as trace minerals and were not discriminated by image analysis, similar to the analysis by Kahn et al. (2002). The mineral liberation characteristics of the ore, as shown in Figure 3, revealed that the liberation of the minerals is good. Although the ore showed mineral intergrown nature, it could be understood that the intergrowths are less

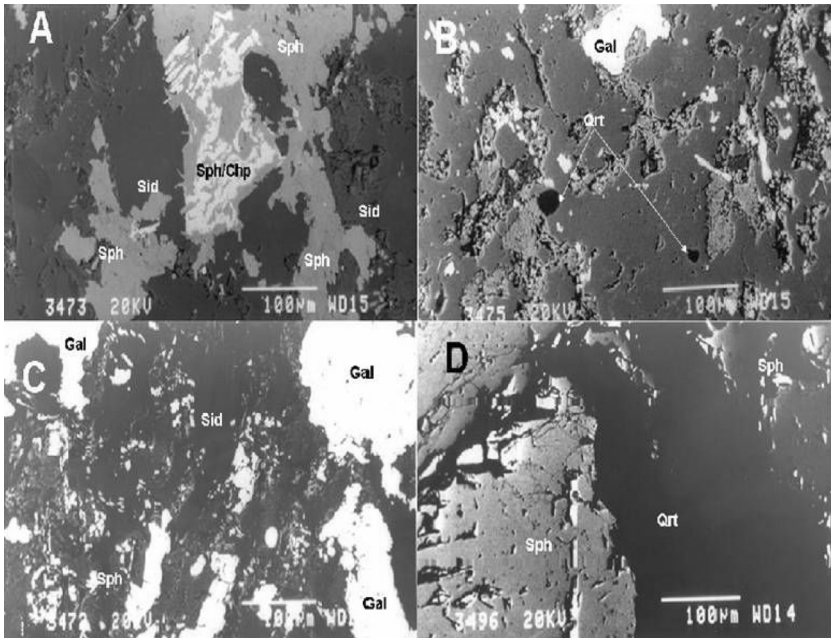

Figure 2. SEM/BSI micrographs of the bulk ore after polishing, showing the morphologies and fine-to-coarse grains intergrowths of the constituent crystalline phases; sphalerite (sph), galena (gal), siderite (sid), and quartz (qrt). (A) Intergrowths of chalcopyrite group mineral (lighter gray) with sphalerite (light gray), and the intergrown of quartz (black) with siderite (dark gray); (B) very complex intergrowth nature of the ore with grain boundaries of sphalerite, galena (white), and quartz intergrown together with siderite; (C) fine-grained and complex intergrowths of siderite, sphalerite, and galena; (D) fine intergrowths at the interstitials and boundaries of sphalerite and quartz.

complex and the grain boundaries have little interpenetrations. The liberation might also have been promoted by the differences and combination of both transgranular and intergranular fracturing liberation, which might cause grains to be released easily.

Size-Reduction Characteristics

The effectiveness of practically all mineral processing and extraction processes is a function of the size of the particle treated. Because of the comparatively high cost of size reduction (Benzer 2005) and the difficulties associated with separating minerals when over- or under-liberation occurs, it is essential that the correct amount of size reduction

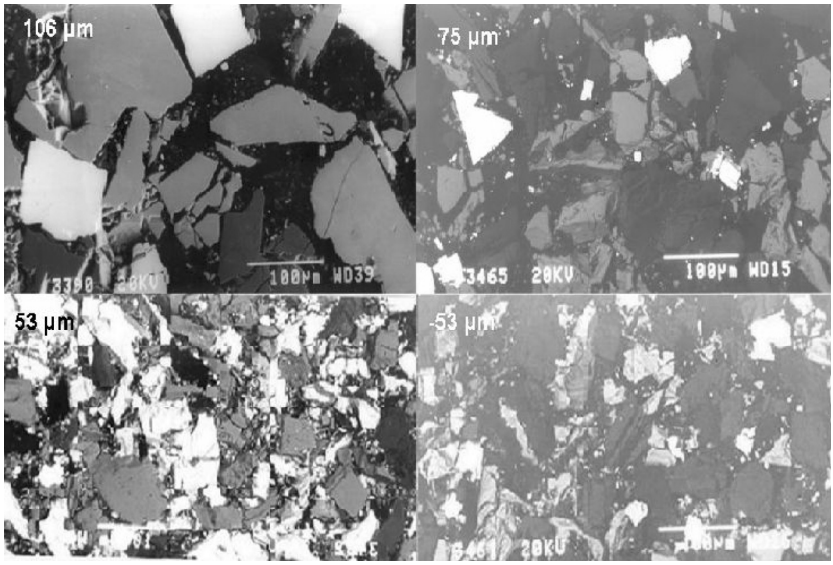

Figure 3. SEM/BSI micrographs of particle sizes of 106, 75, 53, and −53 µm showing the mineral Liberation pattern.

be achieved. To this end, size analysis was carried out on the products of all the rod mill parameters (amount of feed, no amount of rod and milling time) varied to determine the optimal size reduction (grinding) process. The results of the size analysis, as presented in Figure 4A and 4B, show that, at longer grinding times, more particles of lower particle sizes were obtained. This implies that grinding time has a positive effect on size reduction. It was also observed that the amount of feed ore has a negative affect on size reduction of the ore, as revealed by more particles of a larger size at higher amounts of feed ore. Results also show that the particles were mostly distributed in the size range of +106 µm–150 µm, with the exception of grinding 500 g at 15 min. These observations therefore present options for which the selection of rod mill parameters for grinding the ore could be easily estimated to obtain any desired particle size and to reduce excessive grinding. There also was a general change in the linearity of the particle breakage in all the milling routes. These might be attributed to changes in the heterogeneity of the particles being ground, which could be due to conditions in which the larger particles are ground preferentially or the larger particles become protected after time (Fuerstenau et al. 2003).

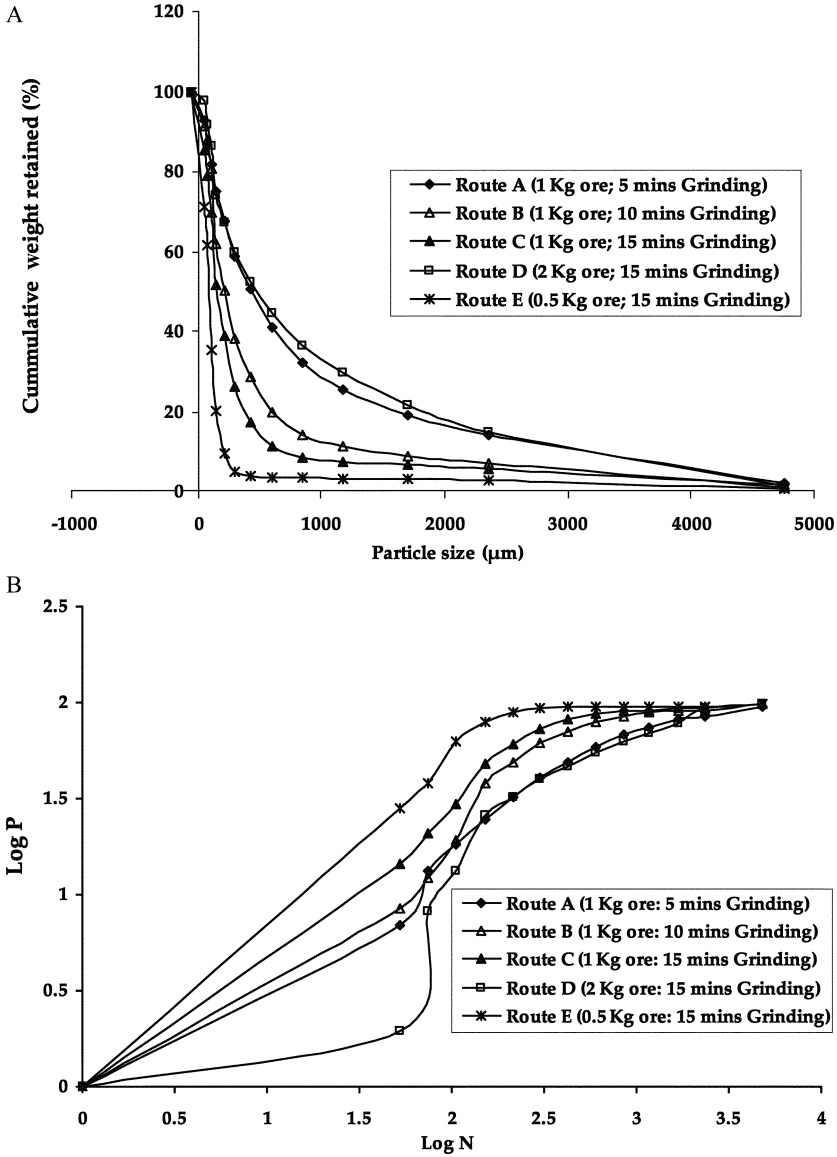

Figure 4. (A) cummulative weight retained (%) against particle size (μm) and (B) gate Gaudin–Schuhmann size distribution of the grinding routes.

Particle-size analysis is of great importance in the design of an optimal process for mineral processing and extraction, and several studies have been carried out to determine its effects on mineral recovery (Deveci 2004; Hossain et al. 2004; Ozcan et al. 2000). It is observed, however, that particle size alone may not reveal enough information about a process to allow process optimization (Barrett 2003). Although particle size analysis provides information on measuring the extent of the liberation of the value minerals from the gangue at various particle sizes and determines the optimum size of feed to the process for maximum efficiency as well as the size range at which any losses are occurring in the plant so that they may be reduced, it does not provide detailed information on the distribution of the constituent minerals and elements within the various size ranges to ascertain the degree of the reduction of specified minerals. While they are useful for characterizing and determining efficiency, they offer little information about the breakage characteristics of the material within the comminution system (Fuerstenau et al. 1999).

Elemental Distribution and Quantitative Analysis

In order to fully understand minerals' behavior during comminution from mineralogical point of view, minerals and elemental distribution among five different particles sizes were analyzed. Mineral identification (Figure 3) was also carried out, which were similar to those by Zapata-Massot et al. (2004) in which SEM was used to acquire images of the particles and particles in various families were classified. Table 2 show the XRF and ICP-OES results for the distribution of elements within

Table 2. Percentage composition and distribution of the elements

	X-ray fluorescence spectroscopy (XRF)						Optical emission spectrometry (ICP-OES)					
Sample	O	Zn	Fe	S	Pb	Si	Mn	Cu	Mg	Ca	Al	Cd
Bulk ore total	31.33	25.53	12.59	13.82	9.78	4.79	0.94	0.45	0.48	0.37	0.27	0.091
Size 106μm	33.33	26.63	11.98	13.55	9.02	3.02	0.89	0.52	0.44	0.34	0.15	0.099
Size 75μm	32.71	25.81	12.23	13.67	9.45	3.10	0.90	0.47	0.45	0.35	0.22	0.091
Size 53μm	32.61	25.05	12.96	13.91	10.15	4.23	0.91	0.42	0.52	0.37	0.28	0.088
Size −53μm	33.01	24.51	13.11	14.03	10.41	5.51	0.97	0.39	0.53	0.38	0.39	0.088

the sizes. The amounts of zinc, copper, and cadmium are reduced as the particle size decreases while lead, iron, sulphur, and silica concentrations increase.

From the results, it can be deduced that minerals' response to breaking is largely determined by their mineralogical properties. It is largely determined by the Bond work index (Casali et al. 2001) and its concept has been used to evaluate the efficiency of a grinding circuit (Rowland 1973). It is a function of a mineral's hardness, tenacity, cleavage, parting, and fracture. These are in turn dependent on the atomic structure of that mineral, which is controlled by the type of bonding and the distance between the atoms. Cleavage takes place parallel to atomic planes where the bonding in the crystal is weak, while parting results when minerals break along planes of structural weakness. Though fracture is controlled by crystal structure, it is an unpredictable form of breaking of a mineral that does not follow a crystallographic direction and occurs because there is equal bonding in all directions.

Sphalerite has a dodecahedral cleavage but fractures on uneven flat surfaces; hence, during grinding, sphalerite fractures only along the flat surface, resulting in flat particles that pose difficulties in reduction even at higher grinding. Galena has a good cleavage and fracture, and therefore easily breaks along its cleavages, into cubes. This ability to break along its cleavages with its soft and brittle properties makes its size-reduction easy. The increase in silicon content at particles of lower sizes could be traced to the mineralogical properties of quartz. Quartz is hard and very brittle, and therefore breaks easily into fine particles due to its good fracture, even though it has no cleavage. An increase in the amount of sulphur at lower particle sizes could be attributed to the increase in the degree of deformation, which caused some structural changes and transformations. This was also observed by Hu et al. (2004), where greater amount of new lattice defects were produced on the surface of mechanically activated pyrite because of the formation of elemental sulphur during mechanical activation and the extremely incomplete and cleavages.

Predictions for Process Selection and Design

The mineralogical differences among the constituent's minerals within the different sizes contribute to variation in their recoveries. This was observed by Harbort et al. (1999), who found that a higher degree of

grinding influenced to a greater extent the recovery of galena and increased the rejection of sphalerite and pyrites. The size analysis of the products from the five grinding routes gave clear information on determining the choice of rod milling parameters for obtaining a specified particle size.

Figure 5 presents variable choices that a mineral processor can make in deciding the process for obtaining a particular amount of size(s), while the elemental distribution analysis shown in Table 2 determines relative amounts of a particular element(s) within the size(s). Higher amounts of particle sizes of -53, 53 and $+75\,\mu m$ could be obtained through Process E, while Process B gave the highest amounts of particles in the size range of $+106\,\mu m$. The elemental distribution (Table 2) gives the mineralogical basis for the recovery at any given particle size and is useful in ascertaining the relative recovery of specified metal. Acar et al. (2005) adopted this principle and used the assays for respective size fractions to calculate the quantitative data, which was compared with the bulk sample (composite) assay. The type and amount of mineral present in an ore determines the reactivity and recovery process, especially during hydrometallurgical processing, and therefore governs the overall design

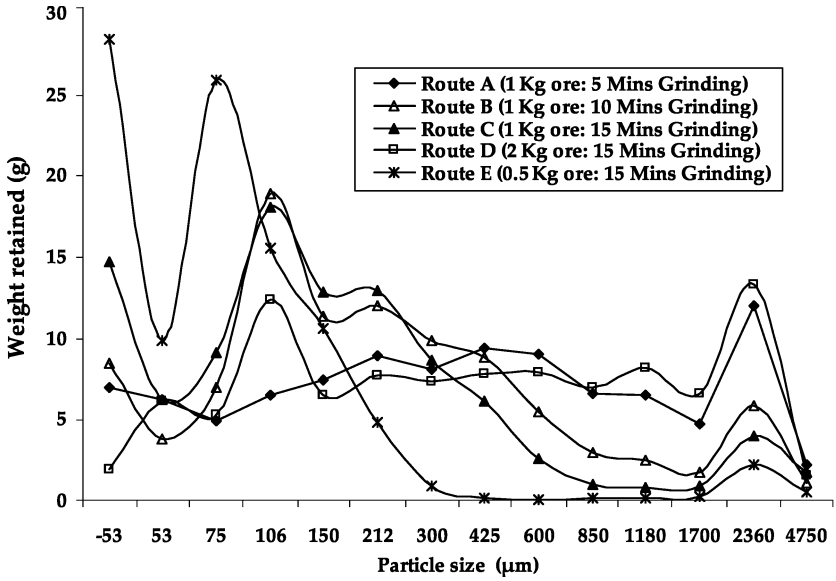

Figure 5. Weight retained (g) against particle size (μm).

process. For example, many sulfide minerals, including pyrite and marcasite (FeS), pyrrhotite ($Fe_{1-x}S$), chalcopyrite (CuFeS), and enargite (CuAsS), generate acid when they interact with oxygenated water, while other sulfide minerals, such as sphalerite (ZnS) and galena (PbS), generally do not produce acid when oxygen is the oxidant (Plumlee and Nash 1995).

The data in Table 2 can be used to determine the relative percentages of metal recovered at a given particle size. Since there is variation in the amounts of each element within the particle sizes, estimation of percentage metal recovered would be a quotient of the percentage of such metal within a particular particle size. The effects of associated metal(s) within a particular particle size on the recovery of metal can also be predicted at a glance. For instance, it is expected that the dissolutions of copper and zinc are highest where the amount of iron is widely distributed. This is due to the fact that the presence of iron has positive effects on their recoveries (Rodriguez et al. 2003a, 2003b, 2003c). According to Zielinski et al. (2000), this increase is believed to result from the autocatalytic action of the dissolved iron rather than from the distortion of the sphalerite lattice. Moreover, sulfide-rich mineral assemblages with high percentages of iron sulfide or sulfide minerals having iron as a constituent (such as chalcopyrite or iron-rich sphalerite) will generate significantly more acidic water than sphalerite- and galena-rich assemblages that lack iron sulfide minerals (Plumlee and Nash 1995). On the other hand, the presence of silica is expected to affect the dissolution process, as it has been observed to cause a shift in iron mobilization (Davis et al. 2001). High levels of silica alter the mineralogy of ferrous oxidation products in natural systems, thereby leading to the precipitation of more stable solid precipitate (Mayer and Jarrell 1996). Phoenix et al. (2003) noted that iron may precipitate in the presence of silica as either an amorphous $Fe(OH)_{3(s)}$ phase or as a poorly ordered hydrous iron-silicate precipitates, e.g., $((Fe,Mn)SiO_3, Fe_2^{3+}Si_2O_7 \cdot 2(H_2O))$ and $((Fe,Mg)_3Si_4O_{10}(OH)_2)$. The precipitate form therefore alters the reactivity and redox behavior of ferrous ions. Rushing (2002) confirmed that increased silica content decreases the rate of oxidation of Fe^{2+} to Fe^{3+}. Moreover, silica has negative effects on solution purification process, especially during the roast–leach–electrowining zinc extraction process (DiFeo et al. 2001). In this case, such concentrates should be pretreated to eliminate or reduce the silica content. In achieving this, additional treatment and process cost is incurred (Lewis and Streets 1978).

CONCLUSION

This study has clearly shown the role played by applied mineralogy in determining an optimal hydrometallurgical recovery process for base metals from complex sulphide ores. It also showed that the choice of a grinding process is largely determined by the mineralogical properties of the ore. The results obtained for the minerals and elemental distribution among the various particle sizes gave clear information on which mineral process an engineer could base his choice of processing route. In conclusion, when critical decisions are being made on recovery processes, attention should not be solely based on particle sizes, but more on the distribution of the constituent minerals and elements within the sizes.

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