

MIRL Report No. 84

**FERRIC CHLORIDE LEACHING OF THE
DELTA SULFIDE ORES AND
GOLD EXTRACTION FROM
THE LEACHING RESIDUE**

By

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Library of Congress Cataloging in Publication Data

Library of Congress Catalog Card Number 88-061592
ISBN 0-911043-08-X

May, 1988

Published by

Mineral Industry Research Laboratory
210 O'Neill Research Laboratory
University of Alaska Fairbanks
Fairbanks, Alaska 99775-1180

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INTRODUCTION

Conventional differential and bulk flotation processes have difficulties in achieving high recoveries with acceptable grades for zinc, lead and copper from the complex sulfide ores found at Tok, Alaska¹. Furthermore, gold and silver, which account for a significant fraction of total value of the ores, are distributed evenly in the flotation tailings and concentrate. Therefore, processing both flotation tailings and concentrate would be necessary to obtain high recoveries of gold and silver.

A mineralogical study revealed that the economic sulfide minerals are interstitially associated with a large preponderance of pyrite. The economic sulfide minerals are 10 to 40 microns in size. These mineralogical facts explain the difficulties encountered in the flotation process.

A hydrometallurgical method involving ferric chloride leaching and subsequent steps to recover lead, zinc, silver and copper from the leach liquor has been studied at the Mineral Industry Research Laboratory, University of Alaska Fairbanks for the treatment of Delta ores. This alternative is attractive for processing complex sulfide ores which conventional flotation and smelting cannot handle. In addition, the liberation of sulfur in the environmentally acceptable elemental form, rather than as sulfur dioxide, may prove a major advantage of this hydrometallurgical method because of stringent environmental regulations.

As early as 1923, ferric chloride leaching was applied to one sulfide mineral² and was recognized as a highly efficient process. Chloride leaching has become more practical during the past decade, as corrosion problems of concentrated chloride solution have been partially overcome by the advent of modern materials of construction.

The United States Bureau of Mines has conducted extensive tests on ferric chloride leaching of chalcopyrite concentrates^{3,4} and galena concentrates^{5,6}. A hydrometallurgical test involving ferric chloride leaching of galena concentrates, purification of the resulting solution and direct electrolysis of the purified lead chloride solution was performed in the Minemet Research Laboratory⁷.

Lucas and Shimano⁸ have investigated two-stage ferric chloride leaching of a pyritic Zn-Pb-Cu-Ag bulk concentrate and found that more than 92% of zinc, lead, copper and silver could be extracted in 7.5 hours. The primary leaching reactions of sulfide minerals have been proposed as:



Many other chloride-based hydrometallurgical processes such as: Clear, Cymet, Elken and Phelps Dodge, have been developed to treat copper or copper-zinc-lead concentrates on pilot-plant scale. But a direct ferric chloride leaching process for complex sulfide ores has yet to be developed.

The proposed chloride leaching process for the treatment of Delta complex sulfide ores is shown in Figure 1. In the proposed flowsheet, the ores are treated by single and two stage ferric chloride leaching to dissolve zinc, lead, copper and silver. The leach residue is treated for gold recovery while the leach liquor will be directed to lead, copper, silver and zinc recovery steps. Lead chloride will be crystallized first followed by a solvent extraction and electrolysis step to produce metallic copper. Cementation, by adding zinc powder, is then applied to the liquor to precipitate silver. Zinc production is by solvent extraction and electrolysis. The leach solution is regenerated with chlorine gas produced in the electrolysis steps. If a large amount of iron coprecipitates with silver, a hot cementation with metallic lead can be applied prior to the lead chloride crystallization step in order to precipitate both silver and copper. Zinc cementation would then be removed from the process.

The results of a study on chloride leaching and gold recovery from the leaching residue are presented in this report.

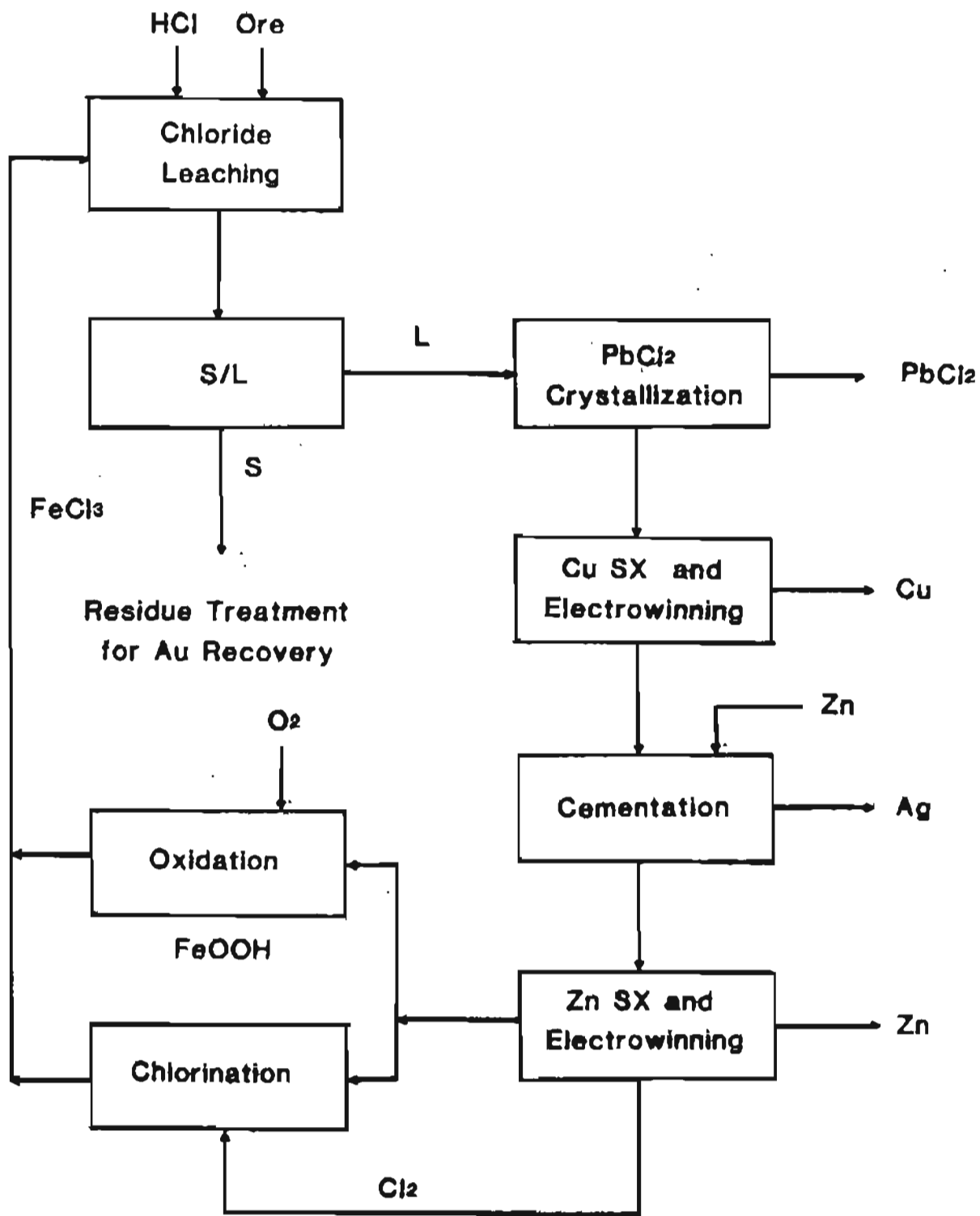


Figure 1. Proposed chloride leach process for treatment of Delta complex sulfide ores.

EXPERIMENTAL WORK

Ores

Six samples of ore were collected from different locations in the Delta Massive sulfide deposit in 1985. These samples were named LPU, LPL, DDS, TRIO, DDN5 and DDN6. Their elemental compositions were presented in the first stage report.

A sample of LP ore was collected in August, 1986. The sample was representative of the outcrop that was drilled and blasted a year earlier. A total of 1150 lbs of sample was brought to the laboratory. The sample was crushed in stages to 4 mesh. Further size reduction was carried out in a 8" dia x 7" ball mill. After a series of grinding tests a 7 1/2 minute grinding was selected. A 4.5 kg charge was used at 60% pulp density. About 170 kg of sample was ground in the ball mill and the product was deslimed in a 4" cyclone with a cut point at approximately 15 microns for gangue minerals. The cyclone overflow was flocculated, filtered and dried. The cyclone underflow was dried and sized using a sieve shaker down to 400 mesh. The minus 400 mesh material was further sized at 20u sieve. These sized products were used in the leaching experiments. The concentrations of metals in various size fractions of the LP-86 ore sample are presented in Table 1.

The sized products were pelletized in epoxy for microscopic studies. Mineralogical composition was determined by point counting. A minimum of 1000 points were counted for each size fraction. Liberation study was conducted by grain counting of free and locked particles. Table 2 shows mineralogical composition of various size fractions.

Table 1. Concentration of Metals in the Different Sizes of LP-86 Ore.

| Ore Size Mesh | Zn % | Pb % | Cu % | Au ppm | Ag ppm | Sb ppm |
|------------------|---------|---------|---------|-----------|-----------|-----------|
| +30 | 4.90 | 2.96 | 0.20 | 2.10 | 67.3 | 355 |
| 200 x 270 | 5.10 | 2.84 | 0.19 | 1.70 | 64.2 | 375 |
| 325 x 400 | 5.90 | 2.76 | 0.21 | 1.90 | 69.1 | 470 |
| 400 x 20 | 6.80 | 3.52 | 0.28 | 2.10 | 86.8 | 630 |

Table 2. Mineralogical Analysis of LP-86 Ore.

| Size, Mesh | Mineral Composition Wt % | | | | | | | Total |
|---------------|--------------------------|--------|-------------------|----------------|-------------------|--------|--------------------|-------|
| | Sphal- erite | Galena | Chalco- pyrite | Tenna- nite | Arseno- pyrite | Pyrite | Gangue Minerals | |
| 70 x 100 | 10.6 | 3.6 | 0.8 | 1.3 | <0.1 | 62.1 | 21.6 | 100.0 |
| 100 x 140 | 11.0 | 2.4 | 0.6 | 0.5 | 1.6 | 63.1 | 20.8 | 100.0 |
| 140 x 200 | 9.5 | 2.9 | 0.6 | 0.1 | 1.3 | 63.6 | 22.1 | 100.0 |
| 200 x 270 | 10.9 | 2.0 | 0.3 | <0.1 | 0.6 | 63.6 | 22.6 | 100.0 |
| 270 x 325 | 11.4 | 2.5 | 0.6 | 0.3 | 0.7 | 68.0 | 16.5 | 100.0 |
| 325 x 400 | 11.3 | 2.4 | 0.6 | 0.7 | 1.1 | 69.9 | 14.0 | 100.0 |
| 400 x 20u | 11.3 | 2.4 | 0.6 | 0.4 | 0.7 | 72.1 | 12.5 | 100.0 |

This mineralogical study revealed that pyrite comprises at least 60% by weight in every size fraction of ore particles. Sphalerite and galena are the predominant zinc and lead minerals respectively; chalcopyrite the major copper mineral and tennantite the major silver mineral. The gangue minerals consist mainly of quartz and dolomite.

Table 3 shows volume percent free grains of minerals as well as grains containing 2, 3, 4, or 5 minerals. Table 4 shows liberation of each mineral by size. It shows at 100 x 140 mesh there was no free sphalerite. At 400 x 20 u there was only 39.6% free sphalerite. Even at 20 u x 10 u only 78.3 percent of the sphalerite is liberated. Galena is only 52% liberated even at 20 u x 10 u size. The only mineral that is totally liberated at 400 mesh and finer is arsenopyrite. This study shows that total liberation of valuable minerals is not possible until a 10 micron or finer is reached.

Table 3. Distribution of Locked and Free Grains in Ball Milled LP-86 Ore.

| Size, Mesh | Volume Percent | | | | | | | | | | |
|---------------|---|-----|------|------|------------------|--------|-------------------|-----------------|-------------------|--------|--------------------|
| | Locked grains - No. of minerals in a grain | | | | Liberated grains | | | | | | |
| | 5 | 4 | 3 | 2 | Sphal- erite | Galena | Chalco- pyrite | Tennan- tite | Arseno- pyrite | Pyrite | Gangue Minerals |
| 70 x 100 | 0.9 | 7.9 | 42.0 | 29.8 | 0.5 | 0.1 | 0 | 0 | 0 | 9.9 | 8.9 |
| 100 x 140 | 1.0 | 9.1 | 34.7 | 32.3 | 0 | 0 | 0.3 | 0 | 0.1 | 12.3 | 10.2 |
| 140 x 200 | | 4.3 | 23.4 | 39.8 | 1.7 | 0.1 | 0 | 0.1 | 0 | 17.8 | 12.8 |
| 200 x 270 | 0.1 | 2.4 | 13.8 | 38.6 | 2.7 | 0.2 | 0.1 | 0.1 | 0.2 | 23.8 | 18.0 |
| 270 x 325 | | 1.0 | 10.2 | 38.2 | 4.8 | 0.5 | 0.2 | 0.1 | 0.2 | 29.3 | 15.5 |
| 325 x 400 | | 0.2 | 3.4 | 29.8 | 5.5 | 0.3 | 0.2 | 0.3 | 0.2 | 41.4 | 18.7 |
| 400 x 20u | | 0 | 2.8 | 24.1 | 7.4 | 0.6 | 0.4 | 0.1 | 0.5 | 51.6 | 12.5 |
| 20u x 10u | | 0 | 1.4 | 13.8 | 11.9 | 1.3 | 0.7 | 0.2 | 0.4 | 56.6 | 13.7 |

**Table 4. Liberation Study of LP-86 Ore.
% Mineral Liberated in Each Size Fraction***

| Size, Mesh | Sphal- erite | Galena | Chalco- pyrite | Tennan- tite | Arseno- pyrite | Pyrite | Gangue Minerals |
|---------------|-----------------|--------|-------------------|-----------------|-------------------|--------|--------------------|
| 70 x 100 | 4.6 | 5.0 | 0 | 0 | 0 | 19.3 | 2.6 |
| 100 x 140 | 0 | 0 | 50 | 0 | 9 | 23.6 | 30.8 |
| 140 x 200 | 11.5 | 6.3 | 0 | 100 | 0 | 34.1 | 36.6 |
| 200 x 270 | 24.3 | 18.2 | 33 | 100 | 50 | 46.0 | 50.8 |
| 270 x 325 | 39.3 | 35.7 | 33 | 33 | 40 | 50.6 | 57.4 |
| 325 x 400 | 44.7 | 21.4 | 33 | 50 | 25 | 68.1 | 79.6 |
| 400 x 20u | 59.6 | 42.9 | 66 | 25 | 100 | 81.3 | 59.0 |
| 20u x 10u | 78.3 | 52.0 | 58.3 | 50 | 100 | 89.0 | 87.5 |

* concentration of the minerals was determined by point count whereas concentration of free minerals was determined by grain count.

Leaching Procedure

Bench scale leaching tests were performed in a one-liter glass reaction vessel with a removable cover. The whole assembly consisted of a four-necked pyrex flask, a Friedrich's condenser, a sampling device and an agitator. The leach slurry in the reaction vessel was maintained at $\pm 1^\circ\text{C}$ of desired temperature in a constant temperature jar bath manufactured by Blue M Electric Company. Water is used as the medium in the bath when the desired system temperature is lower than 100°C . Prestone antifreeze is used when the temperature is greater than 100°C .

At the start of each test, lixiviant was added to the reaction vessel and was allowed to reach the thermal equilibrium within the bath. A weighed charge of the solid sample was loaded into the reaction vessel. The slurry temperature would rise as much as 2°C because of the heat evolved by the leaching reactions. However, the slurry temperature returned to the desired temperature rapidly.

At the end of each test, the slurry was filtered immediately using a vacuum filter. In a two-stage leaching test, a fresh ore sample was loaded for first stage leaching and the leach residue of first stage was used to charge the second stage. Newly prepared lixiviant was used in the second stage while the resultant leach liquor from the second stage was used as the lixiviant in the first stage.

During the test, solution samples were taken periodically from the reaction vessel. The soluble zinc, lead, copper, silver, calcium and total iron were determined by atomic adsorption spectroscopy. Iron(II) concentration was determined by titration with standard KMnO_4 solution. Soluble sulfate concentration was gravimetrically determined with BaCl_2 solution. Metal values in the solid were determined by aqua regia digestion followed by atomic adsorption spectroscopy. Elemental sulfur in the solid was determined using xylene dissolution complimented by evaporation.

Gold Dissolution Tests

Gold dissolution from the leach residue by cyanide or thiourea solution at atmospheric pressure was conducted in a reaction vessel similar to the one described above. An autoclave was used for the pressure leaching of gold. A solid/liquid ratio of 50 g/300 ml was applied to all the gold dissolution tests. Gold in solution was determined by extraction using a mixture of MIBK and Aliquat 336 followed by atomic adsorption spectroscopy. Free cyanide was electrochemically determined using a pH/mV meter coupled to free cyanide and reference electrodes.

RESULTS AND DISCUSSION

During leach tests involving the sulfide ore, solid/liquid ratio, particle size, ferric ion concentration and temperature were considered as variables. Cyanide or thiourea concentration, particle size and oxygen pressure were considered as variables in gold dissolution for the resulting chloride leach residue of the sulfide ore.

Leaching of Sulfide Ore

Sufficient agitation (400 rpm) for proper suspension of the slurry was applied in all tests.

- (i) **FeCl₃ concentration** - The stoichiometric amount of FeCl_3 required for dissolving sphalerite, galena, chalcopyrite and pyrrhotite is approximately 0.3 mol/l based on the solid/liquid ratio of 50 g/500 ml. A larger stoichiometric amount was expected when partial dissolutions of pyrite and arsenopyrite were taken into consideration. The experimental results of lead, zinc, copper and silver leaching with ferric chloride concentrations of 0.3 mol/l to 1.0 mol/l are shown in Figures 2 and 3. Under these conditions, no significant effect of FeCl_3 concentration on metals extraction was observed from FeCl_3 concentrations higher than 0.5 mol/l. When a higher solid/liquid ratio is applied, the ferric chloride concentration would be adjusted accordingly.

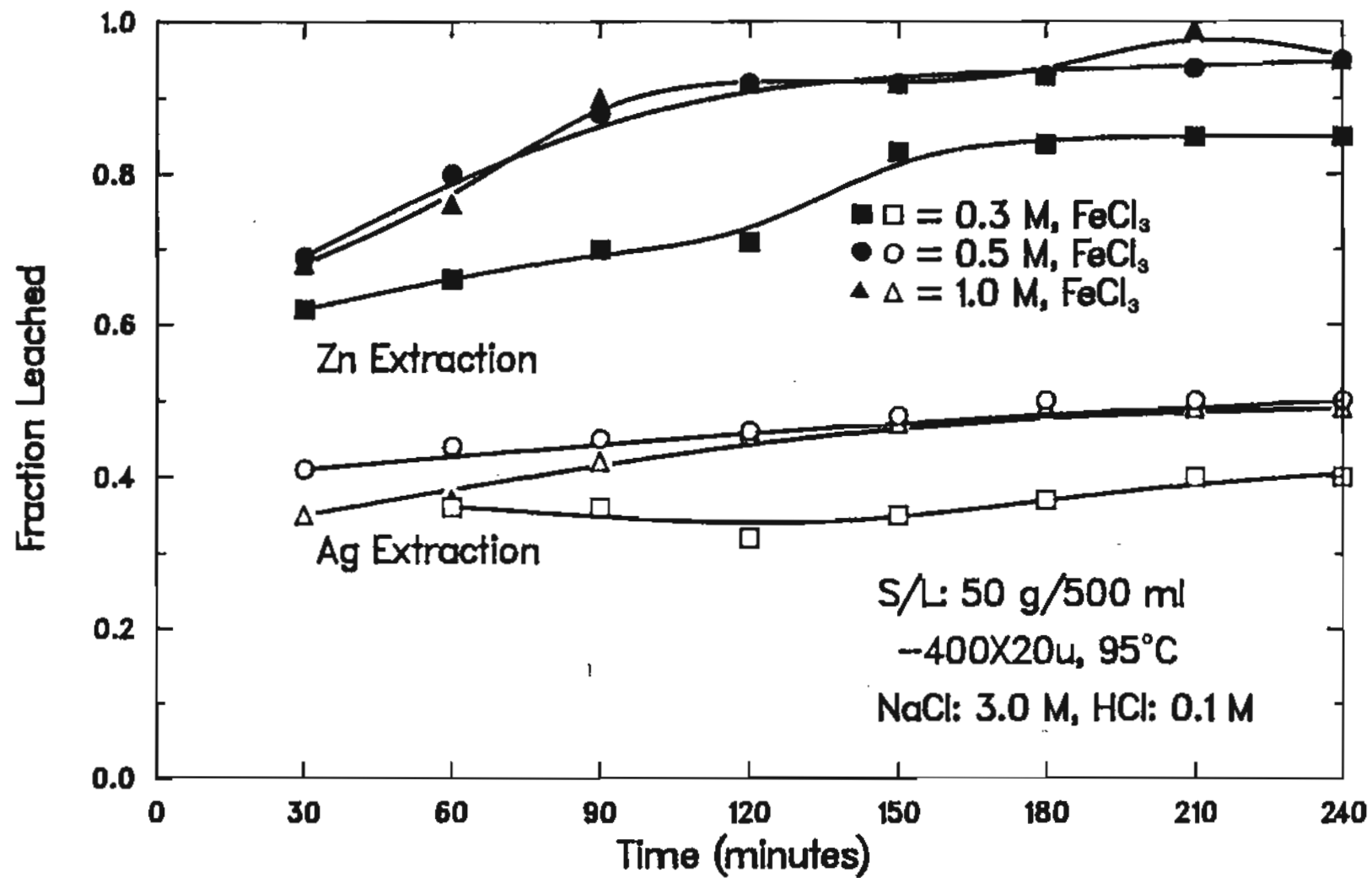


Figure 2. Effect of FeCl₃ concentration on the extraction of Zn and Ag from LP-86 ore.

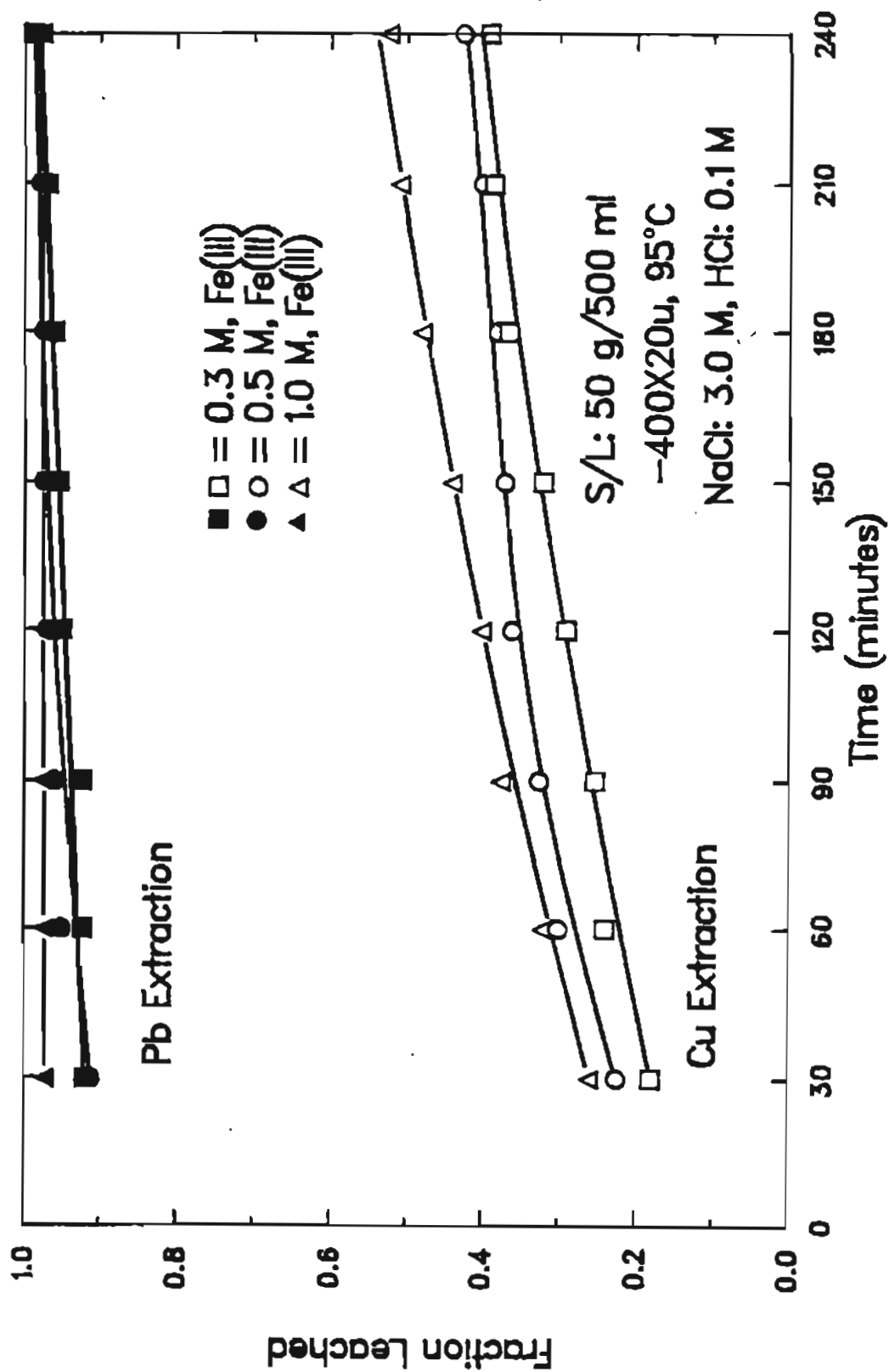


Figure 3. Effect of FeCl₃ concentration on the extraction of Pb and Cu from LP-86 ore.

- (ii) Effect of solid/liquid ratio- Various solid/liquid ratios were tested while the FeCl₃/ore ratio was kept constant. The effect of the solid/liquid ratio on leaching at 90°C is shown in Figure 4. Particle interference at higher solid/liquid ratios hindered the dissolution rate of sphalerite significantly while only minor changes in the leaching rates of lead, silver and copper were observed.
- (iii) Effect of particle size- To study the effect of particle size, two size fractions of the ore were leached. Figure 5 indicates that faster dissolution of zinc, silver and copper was observed for finer particle sizes. Dissolution of lead was too rapid to be differentiated between these two samples.
- (iv) Effect of temperature- It is important to note that zinc is the most valuable component in the Delta ores and it is desirable to recover at least 90% of the zinc values. Acceptable zinc recoveries using a single-stage chloride leach do not seem attainable without very fine grinding and/or excessive retention time. Hence, two-stage leaching at 105°C was tested to improve the zinc extraction. The results, shown in Figure 6, indicate that at least 90% of the zinc can be extracted from LP-86 ore ground to minus 200 mesh and leached in two stages at 105°C with a solid/liquid ratio of 150 g/500 ml.

Silver Extraction and Microprobe Examination of its Occurrence

Silver extraction is low even in saturated NaCl solution (2 mol/l), as shown in Figure 7. Electron microprobe analysis was conducted for the minerals in LP-86. The results are presented in Table 5. They represent the average of several determinations on a single polished section. Arsenopyrite and tennantite data include the determinations made on polished sections of leach residue and subsequent cyanidation residue. Significant in these findings is that tennantite contains 0.75% silver and this mineral is unaffected during ferric chloride leaching as well as cyanidation.

Table 5. Electron Microprobe Analysis of Minerals in LP-86 ore.

| | % by Weight | | | | | | | | Total |
|--------------|-------------|-------|-------|-------|-------|-------|-------|------|--------|
| | Zn | Pb | Cu | S | Fe | As | Sb | Ag | |
| Sphalerite | 53.37 | -- | 1.16 | 33.71 | 6.74 | 0.52 | 0.05 | -- | 99.55 |
| Galena | 1.55 | 84.64 | 0.24 | 14.67 | 1.97 | 0.30 | 0.03 | 0.07 | 103.47 |
| Chalcopyrite | 0.31 | -- | 34.14 | 34.95 | 29.70 | -- | -- | 0.02 | 99.12 |
| Tennantite | 3.39 | -- | 41.54 | 27.97 | 5.08 | 15.50 | 5.85 | 0.75 | 100.08 |
| Arsenopyrite | 1.09 | -- | 0.03 | 21.01 | 35.69 | 42.04 | 0.18 | -- | 100.04 |
| Pyrite | 0.42 | -- | 0.06 | 52.69 | 46.33 | 0.39 | -- | -- | 99.89 |
| Bourmonite | 0.34 | 42.32 | 13.04 | 19.93 | 0.18 | 0.03 | 24.24 | 0.04 | 100.12 |

Oxidation of the silver-containing minerals may be needed for more rapid and complete silver extraction. Roasting the ore prior to leaching is one possibility. The ore was roasted at 750°C for 15 minutes before the leaching was conducted. About 75% of silver can be extracted in the first two hours of leaching with roasting compared to 45% of silver extraction without roasting (Figure 8).

Two-stage leaching was also applied to DDN-5 and TRIO ore samples. The results shown in Figures 9 and 10 indicate that zinc and lead extraction is greater than 90% for both samples while silver extraction is low on the TRIO ore sample.

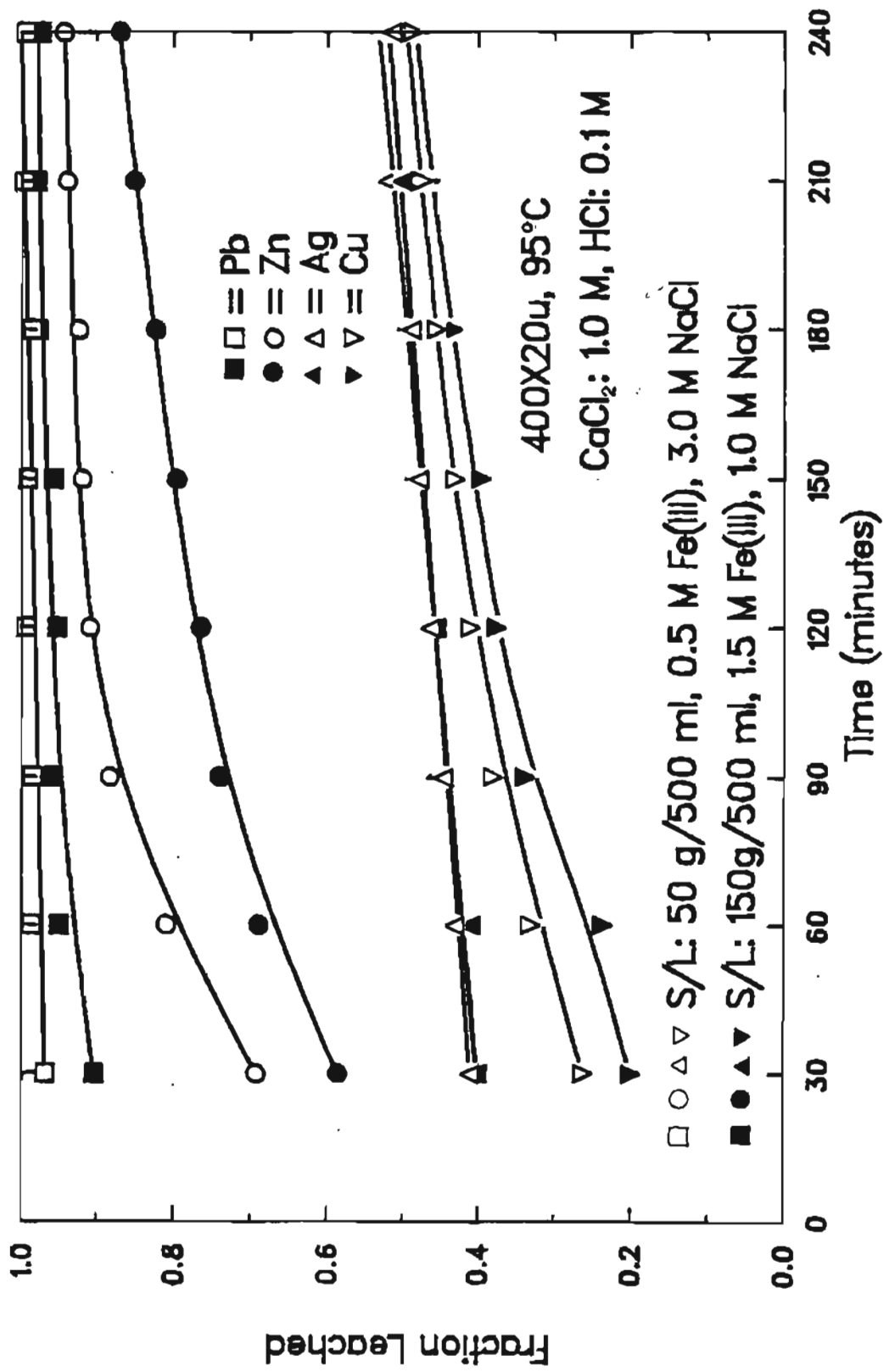


Figure 4. Effect of solid/liquid ratio on metal extraction of LP-86 ore.

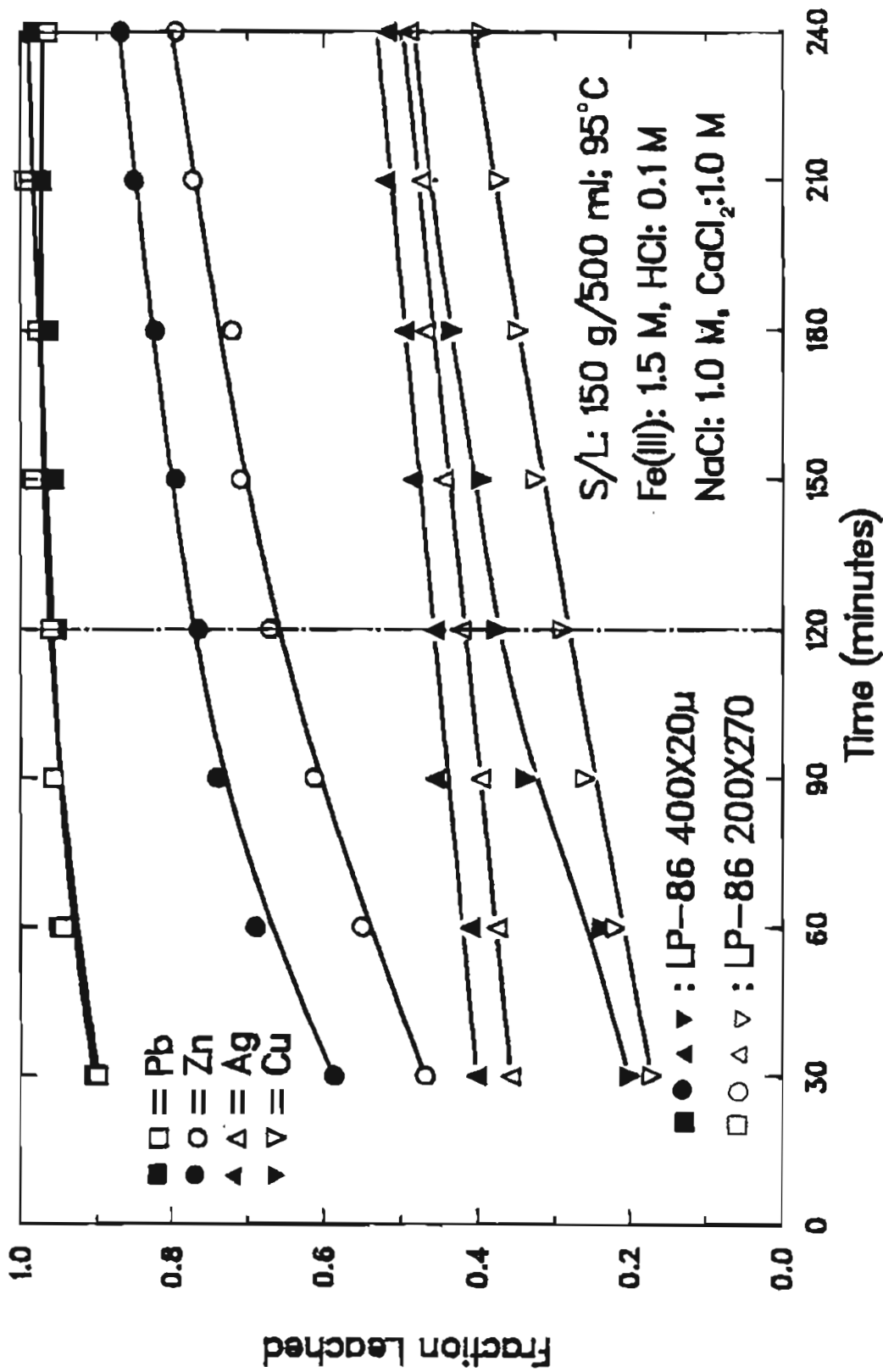


Figure 5. Effect of particle size on metal extraction of LP-86 ore.

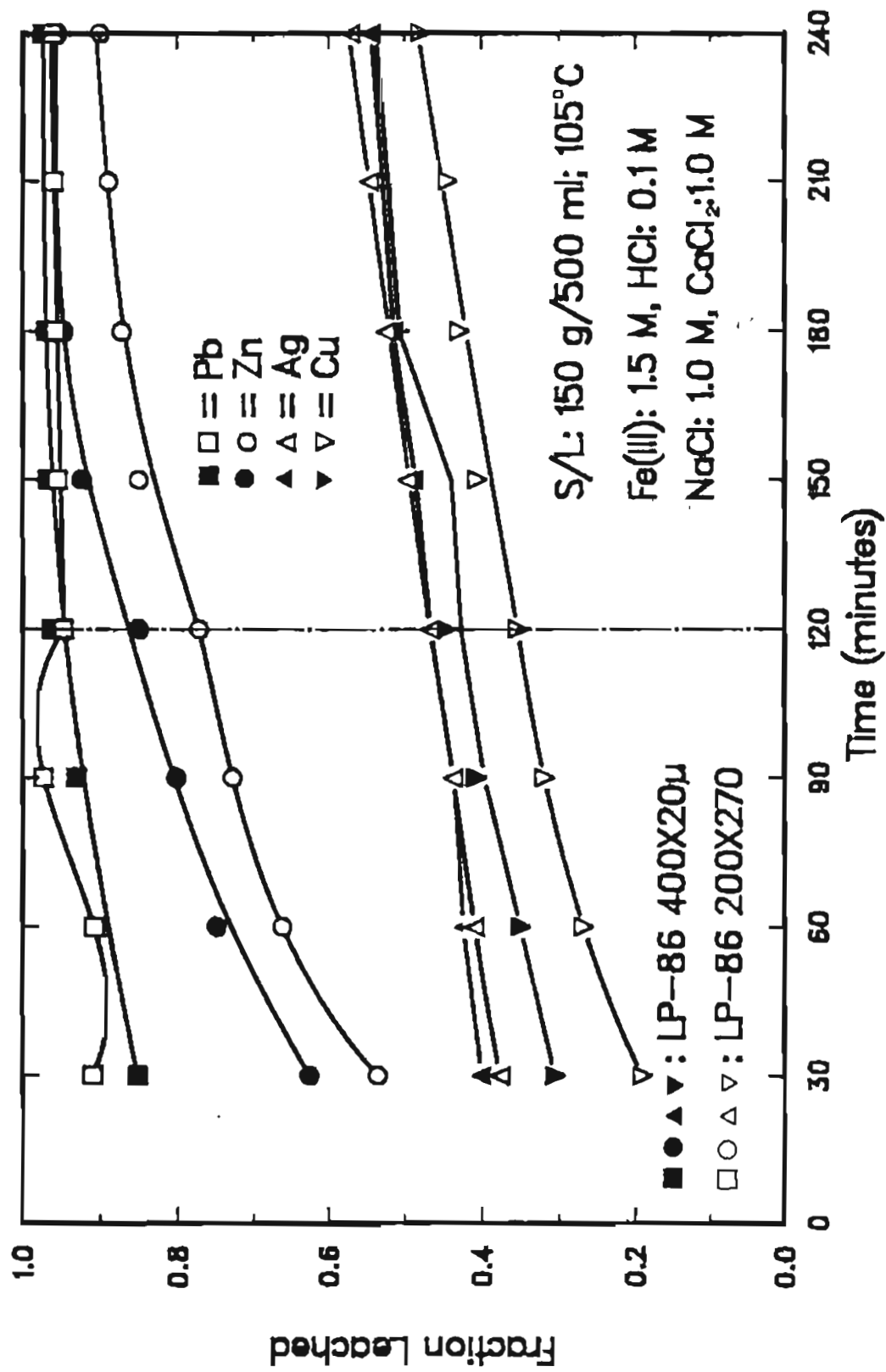


Figure 6. Metal extraction by two-stage leaching at 105°C.

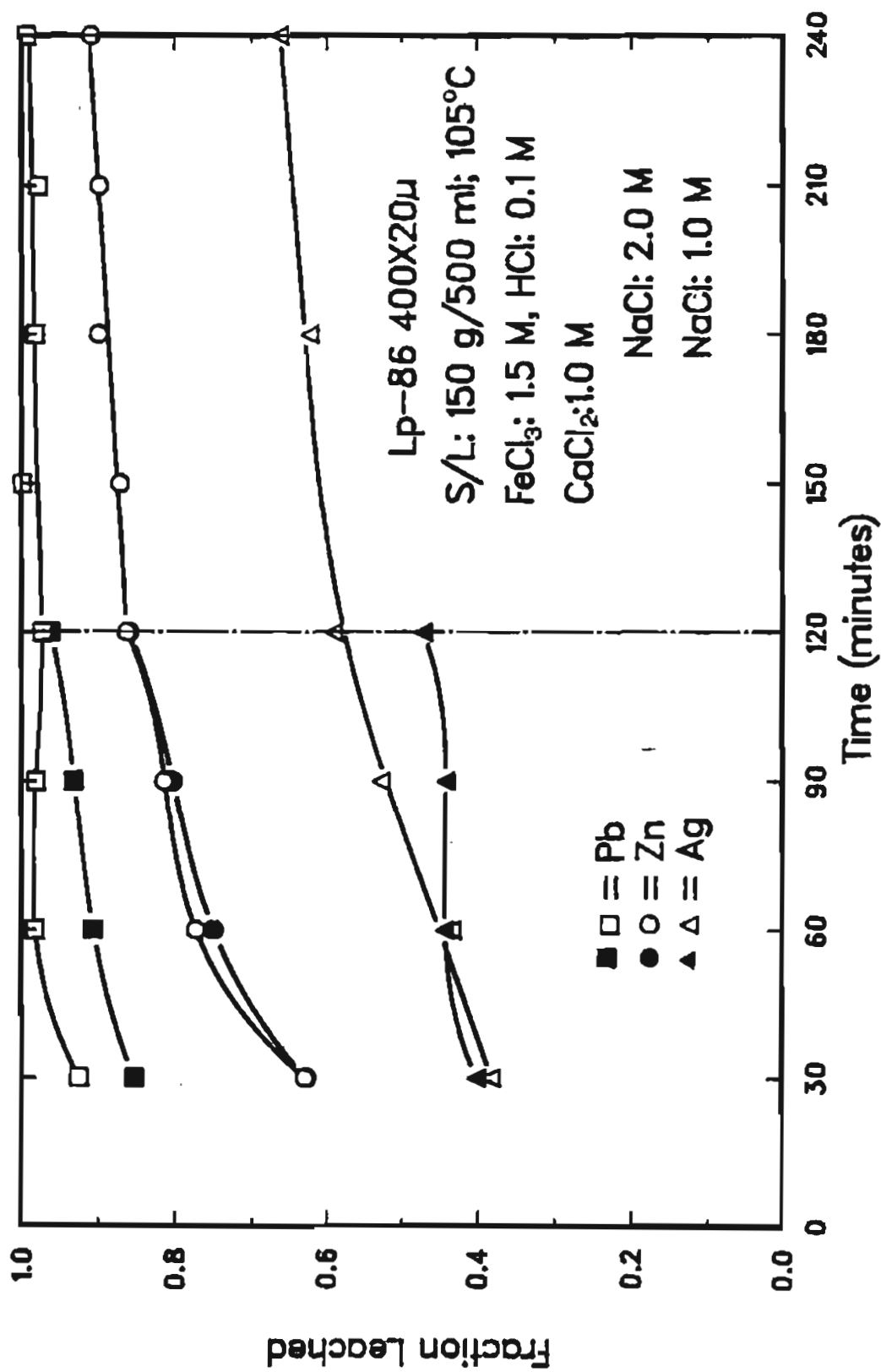


Figure 7. Effect of NaCl concentration on metal extraction of LP-86 ore.

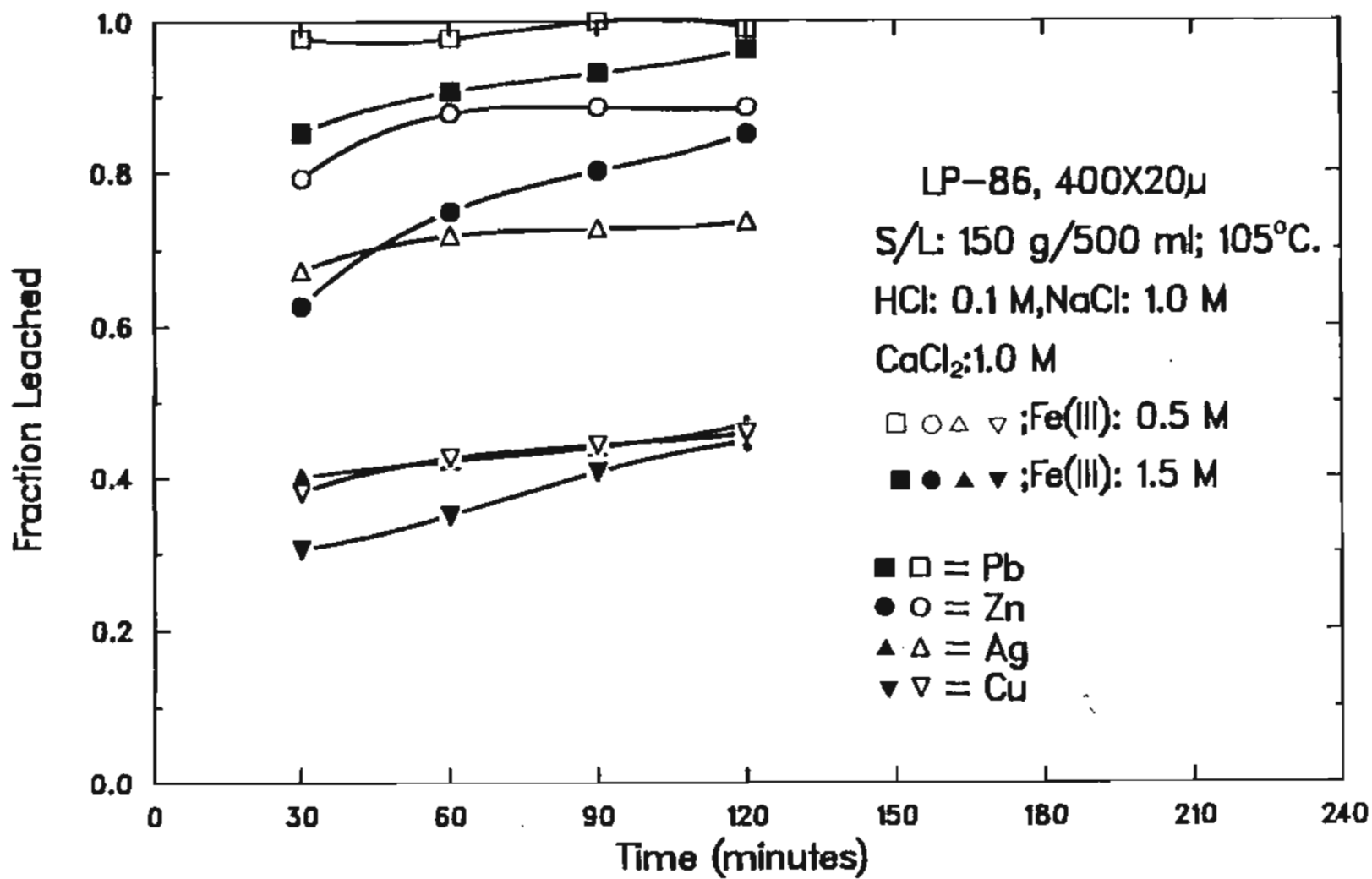


Figure 8. Comparison of metal extractions with and without roasting.

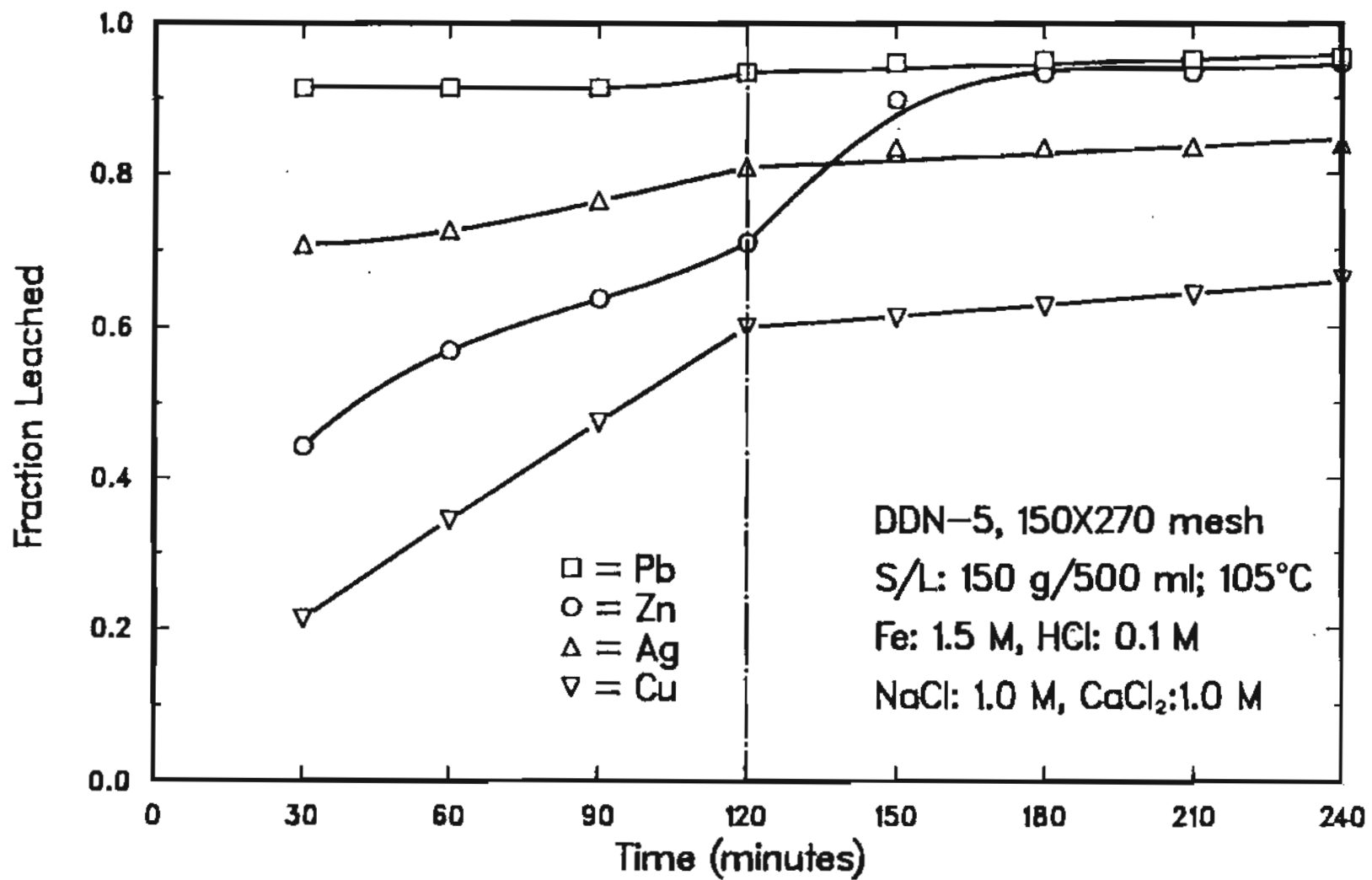


Figure 9. Metal extraction of DDN-5 ore.

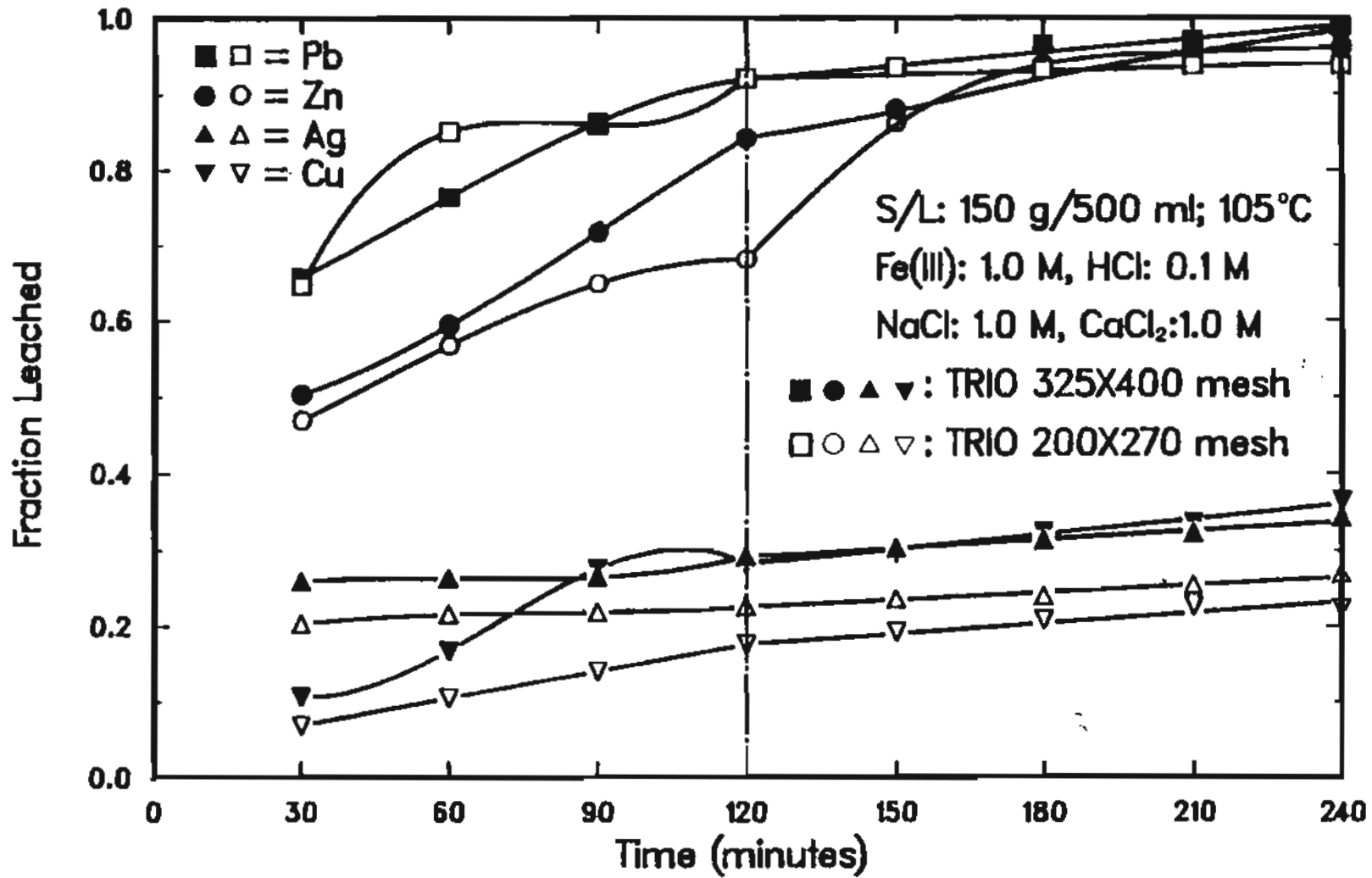


Figure 10. Metal extraction of TRIO ore.

Gold Dissolution Tests

The results of gold dissolution tests are listed in Table 6. All gold dissolution tests but one (test #1) were conducted using the FeCl_3 leaching residue as the solid feed. The high sulfide content in the ore might make direct cyanidation very difficult (test #1). Higher gold extraction can be obtained using higher cyanide and oxygen concentrations (tests #3, 4 and 5).

Increase in chloride leaching temperature to 105°C improved gold extraction in the cyanidation to 64% at 24 hours (test #5). Elemental extraction of chloride leaching residue prior to cyanidation could not improve gold extraction (tests #5 and 6) under these conditions tested. Roasting of the ore at 750°C for 15 minutes prior to the chloride leaching was able to increase gold extraction to 71% (test #7) while thiourea leaching (test #8) and cyanidation under high pressure (test #9) did not have positive effects on gold extraction. No significant increase in gold extraction was observed on a prolongation of retention time beyond 24 hours.

Free cyanide measurements of the end solution of tests #3 - 7 and #9 indicated that at least 30% of total cyanide was unreacted and remained free in the solution. The lower gold extraction, hence, cannot be attributed to the insufficiency of the reactant species.

Table 6. Experimental Results of Gold Dissolution Tests.

| Solid Feed | | Leaching Conditions | Au Dissolution Conditions | Percent Au | Reacted Ag |
|------------|--|--------------------------|--|--|------------|
| 1) | LP-86 400x20u | | NaCN: 0.015% CaO: 0.020% | 30 (18Hrs) | 2 |
| 2) | LP-86 400x20u with leaching | 95°C 4Hrs | " | 31 (24Hrs) | 10 |
| 3) | " | 95°C 8Hrs | NaCN: 1g/l, NaOH: 2g/l Air Bubbling | 53 (24Hrs) 47 (48Hrs) | 25 |
| 4) | " | 95°C 4Hrs | " | 51 (24Hrs) 58 (48Hrs) 60 (72Hrs) | |
| 5) | " | 105°C 4Hrs | " | 64 (24Hrs) 65 (47Hrs) | 17 |
| 6) | LP-86 400x20u with leaching & S extraction | " | " | 63 (48Hrs) | 10 |
| 7) | Roasting & leaching of LP-86 400x20u | " | " | 71 (16Hrs) 71 (24Hrs) | 9 |
| 8) | LP-86 400x20u | " | Fe^{3+} : 2g/l H_2SO_4 : 2g/l Thiourea: 2g/l | 50 (24Hrs) | 10 |
| 9) | " | " | NaCN: 1g/l NaOH: 2g/l O_2 : 150 psi | 57 (18Hrs) 58 (24Hrs) | |

An experiment was designed to test the readsorption of the dissolved gold by carbonaceous matters in leaching residue. A volume of 300 ml aurocyanide solution containing 0.30 ppm gold was purged by nitrogen gas for 30 minutes before 50 gm of chloride leaching residue was charged to the solution. The solution was agitated and nitrogen purging was continued. Gold analysis of the solution samples taken at 2, 4 and 6 hours showed no significant change of gold concentration in the solution. Therefore, the possibility of readsorption of gold by the carbonaceous matter in the leach residue was excluded.

Dissemination of very fine gold grains in the pyrite grains was suspected to be the factor which caused the low gold extraction. A microprobe was used to examine the ore and residue for the occurrence of gold. Unfortunately, gold could not be detected in the ore or the leach residue because of its low concentration.

SUMMARY

Over 95% of lead and 90% of zinc were extracted by a two-stage chloride leach of LP-86, TRIO and DDN-5 ore samples. The two-stage leach had a retention time of 4 hours, a temperature of 105°C, and used minus 200 mesh ore. Silver and copper extractions were below 60% for LP-86 and TRIO ore samples. 85% and 65% extractions for silver and copper respectively were achieved for DDN-5 ore sample. The difficulties with silver and copper extractions are probably attributable to their mineralogical occurrence.

About 64% gold extraction was obtained by conventional cyanidation of the chloride leach residue of LP-86 ore. Roasting the ore prior to the chloride leaching could increase the extractions of gold and silver drastically but roasting itself generates SO₂ and causes environmental problems.

ACKNOWLEDGMENTS

The authors wish to express their appreciation to Nerco Minerals Co. for their financial support and permission to publish this paper.

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