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Recovery of Metal Values from Car-Radiator Scrap

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ABSTRACT

Large quantities of car-radiator tubes (brass alloy coated with lead-tin layer) are rejected due to manufacturing failures. Metal values were successfully separated from scrap containing 55% copper, 25.5% zinc, 13.2% lead, and 6.3% tin. The scrap was completely leached with hot HCl solution containing HNO_3 as an oxidant. Upon cooling, about 61% of the lead content was precipitated as lead chloride. Tin was separated as hydrated tin oxide at pH 2.6. Copper was then separated by solvent extraction with an aromatic oxime (Acorga PT-5050Z) in kerosene and stripped with 3 M H_2SO_4($M = mol/dm^{-3}$). Zinc was recovered as carbonate at pH 6.5, and the remaining lead was precipitated as hydroxide at pH 9.0.

Complete leaching was achieved using 2.7 M HCl (equivalent to 1.35 stoichiometry) and 0.96 M HNO_3 at 80°C for 1 h. A synthetic solution containing 300 ppm of each metal was first tested. Copper was almost completely extracted with 10% Acorga PT-5050Z from an acidic solution of $pH > 1$ for 20 min, leaving almost all of the zinc and lead in the aqueous solution. Copper was then separated from the real leach solution (containing in g/L: 36 Cu, 16.2 Zn, and 1.2 Pb) applying three extraction stages with O : A of 4 : 1.

Key Words. Car-radiator scrap; Acid leaching; Recovery; Solvent extraction; Acorga PT-5050Z; Copper; Zinc; Lead; Tin

INTRODUCTION

Continuous depletion of primary metal resources has resulted in the treatment of complex and secondary resources. Scraps, for instance, are significant contributors to the metal supply. Car-radiator tubes are made of brass alloy

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coated with a lead-tin layer, and part of the product is rejected due to manufacturing failures. This results in the accumulation of large quantities of scrap. About 30 tons of scrap accumulate annually in just one Egyptian company (El-Maady Co. for Eng. Industries, Cairo), and are sold at low prices. However, each metal contained in this scrap (Cu, Zn, Pb, and Sn) has important industrial applications. Recovery of these metals by pyrometallurgical treatment causes pollution problems due to hazardous lead and zinc dusts, and results in considerable loss of metals.

Different hydrometallurgical techniques for recovery of copper, zinc, lead, and tin from wastes of brass foundries have been reported. Nesbitt and Xue (1) developed a process using various leachants, including sulfuric acid, ammonia, hydrochloric acid, cyanide, and acetic acid. Sulfuric acid containing copper sulfate with dissolved oxygen was the most successful leachant for copper. Guy et al. (2) used cupric chloride leaching; copper and zinc form soluble salts, while lead forms an insoluble salt. Rabah (3) showed that adding an oxidizing agent such as hydrogen peroxide assists the ammoniacal leaching of copper and zinc. Preliminary experiments for leaching the present scrap showed that a hydrochloric acid solution in the presence of an oxidizing agent, such as nitric acid, is a favorable leachant in terms of high dissolution rate of metals and fast separation of pure lead chloride upon cooling. This technique was practiced in this work where the different parameters were systematically studied to reach complete dissolution of metals.

Separation of copper from other metals in leach solutions could be achieved either by traditional precipitation and filtration, or by solvent extraction and ion-exchange techniques. Solvent extraction followed by electrowinning (SX/EW) produces extra-pure copper for electrical uses. The SX/EW process is now common in the copper industry, where more than 1.5 million tons of copper are produced annually by this process. The SX/EW process also continues to grow in importance as variable sources of copper are leached (4). Recovery of copper from HCl solution and its transfer into H_2SO_4 solution were reported using extractant mixtures such as (trioctylamine + LIX-54) (5), and (Alamine 336 + LIX 54) (6, 7). Kumar et al. (8) used LIX 84 for separation of Cu and Zn from sulfate solution in the presence of gangue metals such as Fe and Mn. Copper was recovered from motor scrap in a process that consists of Cu(II)amine leaching, solvent extraction with LIX 84, and electrowinning of Cu from the stripping solution (9, 10). Ritcey et al. (11) studied the separation of copper and zinc from chloride liquors using Acorga P5300 for extraction of Cu and TBP for extracting Zn. PT-5050Z (Acorga product) has the advantages of strong extraction of copper from relatively low pH solutions and good stripping with strong acids (12). A sample of Acorga PT-5050Z was received from Zeneca Specialities, and preliminary experiments showed a good selectivity for copper over zinc and lead at low pH. This extractant therefore seemed to be favorable for the present task of separation.

Separation of zinc and tin from aqueous solutions was reported elsewhere. Zinc was recovered from solution either as zinc sulfate by crystallization from the purified sulfate solution (13–16), or as zinc carbonate at pH 6.5 by addition of sodium carbonate (17–19).

Pure lead chloride is easily crystallized by cooling the chloride leach solution, whereas tin was recovered from solution at pH 2.4–2.8 by treatment with NaOH to produce hydrated tin oxide (20).

The aim of this work is to recover valuable salts of Cu, Zn, Pb, and Sn from the studied brass-alloy scrap applying an acid-leaching technique. A process flow sheet was used to keep track of dissolution and separation of metal contents. The studied parameters included stages of leaching and solvent extraction where the known chemistry was directly applied in other stages.

EXPERIMENTAL

Materials

A 50-kg sample of car-radiator scrap was obtained from El-Maady Co. for Eng. Industries, Cairo, Egypt. The sample was cut into pieces of about 20 × 13 × 1 mM each. Pure-grade ammonium hydroxide, sodium carbonate, and sodium hydroxide were used for precipitation of tin, zinc, and lead, respectively. Copper and zinc stock solutions were prepared by dissolving pure metals in 1 : 1 HNO_3 and 1 : 1 HCl, respectively, where lead solution was prepared by dissolving $Pb(NO_3)_2$ in 1% HNO_3. In liquid–liquid extraction, the pH in aqueous solutions was adjusted with 0.1 M sodium acetate/acetic acid or 0.1 M ammonium hydroxide/ammonium chloride buffer solutions. Acorga PT-5050Z (Zeneca Specialties) was diluted with pure kerosene and used as an extractant, and A. R. grade H_2SO_4 was diluted to 3 M and used as a stripping solution for copper.

Techniques

Leaching experiments were performed in a 500 mL cylindrical glass reaction vessel immersed in a thermostatically controlled water bath. Samples of 5-g scrap were magnetically mixed with acid solutions composed of 70 mL HCl and 5 mL HNO_3. Concentrations of metal ions in aqueous phases were determined by atomic absorption spectrometer (Perkin-Elmer Model 3100) and the recovery percentage of the metal in solution was calculated according to the equation.

$$\text{Recovery } \% = (W_p/W_i) \times 100$$

where W_p and W_i are weights of a metal in the output and input materials, respectively.

Solvent extraction experiments were carried out first using a synthetic aqueous solution containing only 300 ppm each copper, zinc, and lead to obtain the optimum conditions for metals separation. An aqueous solution containing metal ions was put in a glass tube, and an equal volume of Acorga

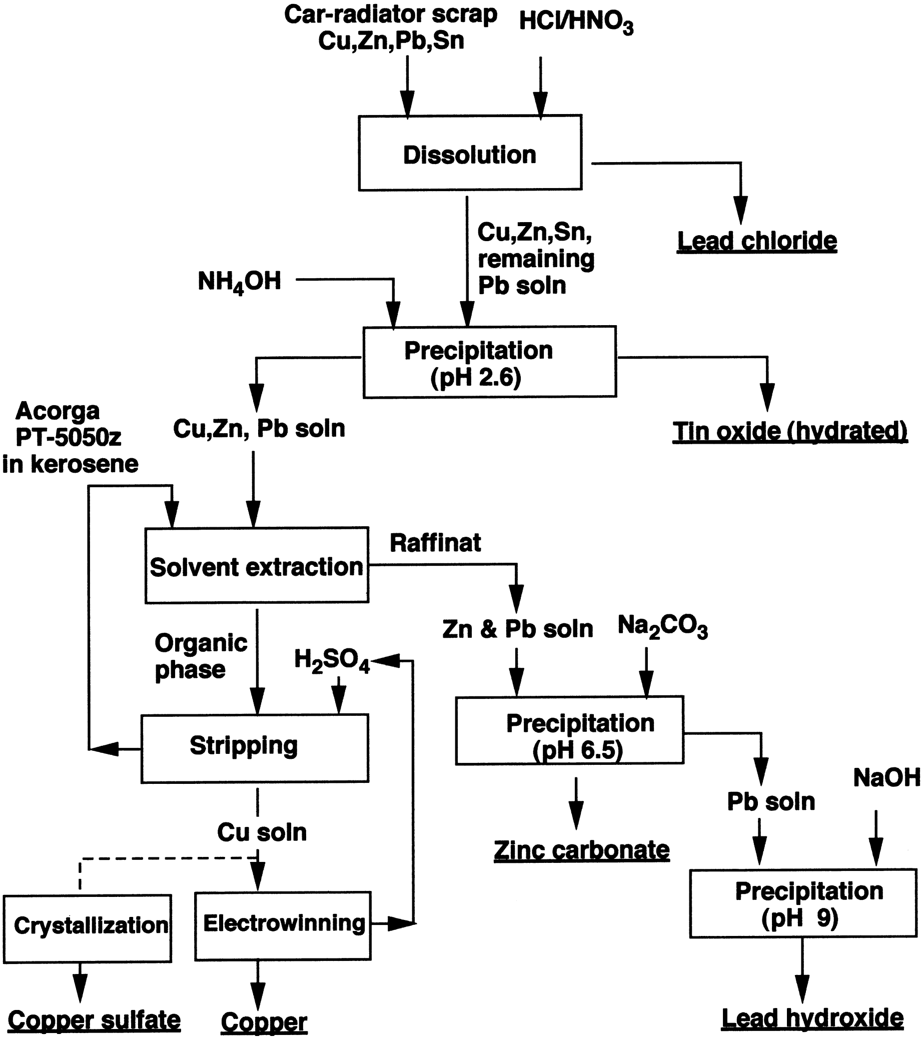

FIG. 1 Process flow sheet for recovery of copper, zinc, lead, and tin from car-radiator scrap.

PT-5050Z in kerosene was added. The two phases were horizontally shaken for the required time at 35°C using a thermostat shaker (GFL Model 1083). The equilibrium pH was measured by a bench-top pH meter (Orion model 420A). The extraction efficiency of a metal in the organic phase was calculated from the difference in metal concentrations in aqueous phase before and after extraction.

Process Flow Sheet

The sequence of operations for separation of Cu, Zn, Pb, and Sn from the studied scrap is shown in Fig. 1. At optimum conditions of HCl/HNO_3 leaching, about 61% of the lead content was precipitated as pure lead chloride upon cooling the solution to 10°C. Tin was recovered as hydrated tin oxide by adjusting solution pH at 2.6 using ammonia solution. Copper was extracted in organic phase containing PT-5050Z and then stripped by 3 M H_2SO_4 into aqueous solution. The strip solution can be subjected to crystallization to produce copper sulfate or electrowinning to produce highly pure copper metal. Zinc was separated from the raffinate (free copper solution) by adding Na_2CO_3 until pH 6.5 to precipitate zinc carbonate; the remaining lead was separated as hydroxide at pH 9 using NaOH.

RESULTS AND DISCUSSION

Leaching

Table 1 shows the chemical composition of the car-radiator scrap. As shown in Table 1, the major constituents are copper, 55%, and zinc, 25.5%, whereas the lesser constituents are lead, 13.2%, and tin, 6.3%. Dissolution of metals from the studied scrap was performed by applying an acid-leaching technique. Figures 2–5 illustrate the effect of HCl concentration on the leaching recovery of copper, zinc, tin, and lead, respectively, using different concentrations of HNO_3 at 80°C for 2 h at liquid : solid of 15 : 1. From Fig. 2, it is clear that with free nitric HCl solution, the percentage of recovery of copper is nearly zero at 2–5 M HCl and very slightly increased at higher acid concentrations. A small increase in recovery was observed after the addition

TABLE 1
Chemical Analysis of the Used Car-Radiator Scrap

Constituent	Copper	Zinc	Lead	Tin
Mass %	55	25.5	13.2	6.3

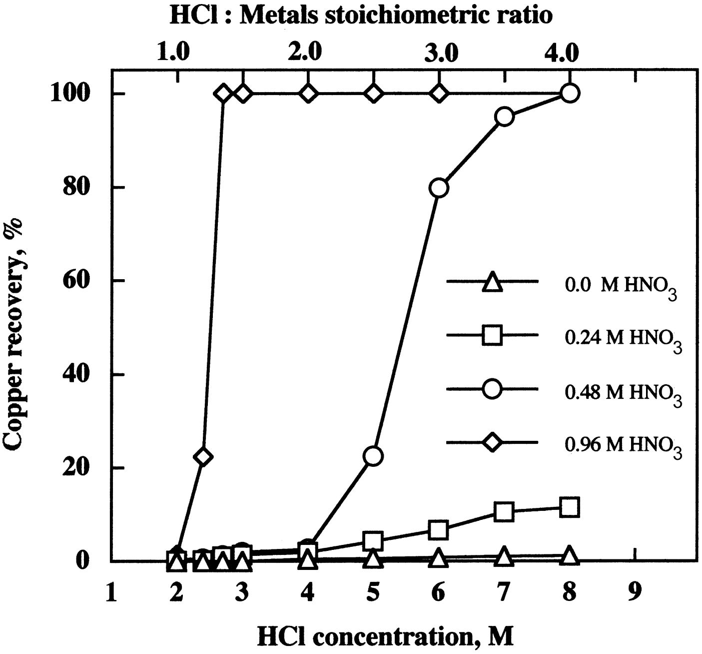

FIG. 2 Effect of acid concentration on the recovery of copper from scrap. Leaching time: 2 h, temp.: 80°C.

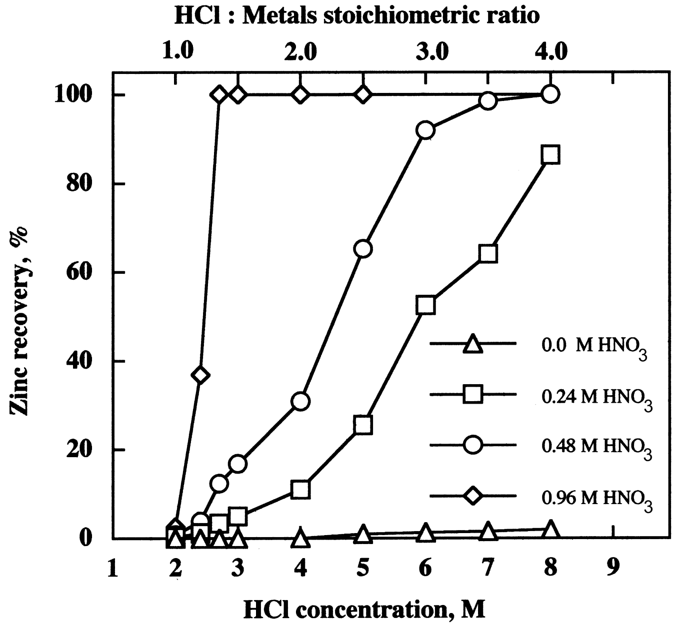

FIG. 3 Effect of acid concentration on the recovery of zinc from scrap. Leaching time: 2 h, temp.: 80°C.

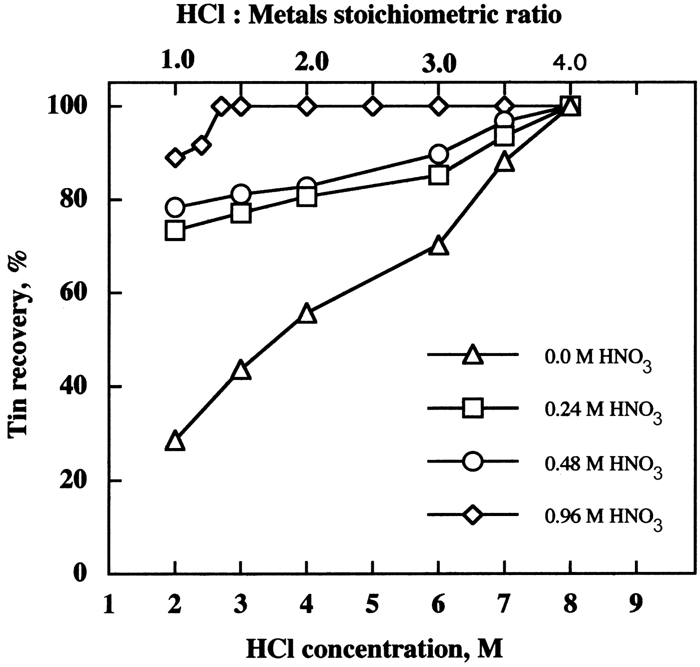

FIG. 4 Effect of acid concentration on the recovery of tin from scrap. Leaching time: 2 h, temp.: 80°C.

of 0.24 M HNO_3, reaching a maximum value of 10% at 8 M HCl. Higher HNO_3 concentrations greatly improved recovery. Almost complete recovery was achieved at 8 M HCl and 0.48 M HNO_3. Increasing nitric acid concentration to 0.96 M caused a sharp increase in recovery, reaching greater than 99% at only 2.7 M HCl. Figure 3 shows the efficiency of zinc recovery using the same experimental conditions. Similar trends were achieved except that at lower HNO_3 concentrations the percentage of recovery values of zinc are higher than those of copper, i.e., at 8 M HCl, about 82% of Zn was recovered, compared with 10% of Cu.

The low reactivity of copper with free nitric HCl solution can be attributed to the fact that copper cannot displace hydrogen in the acid solution due to its lower electronegativity ($E^\circ_{cu} = +0.34$). Nitric acid is added to oxidize copper first, then the oxide reacts with HCl.

$$2HNO_3 \xrightarrow{\text{Heat}} 2NO_2 + H_2O + O \qquad (1)$$

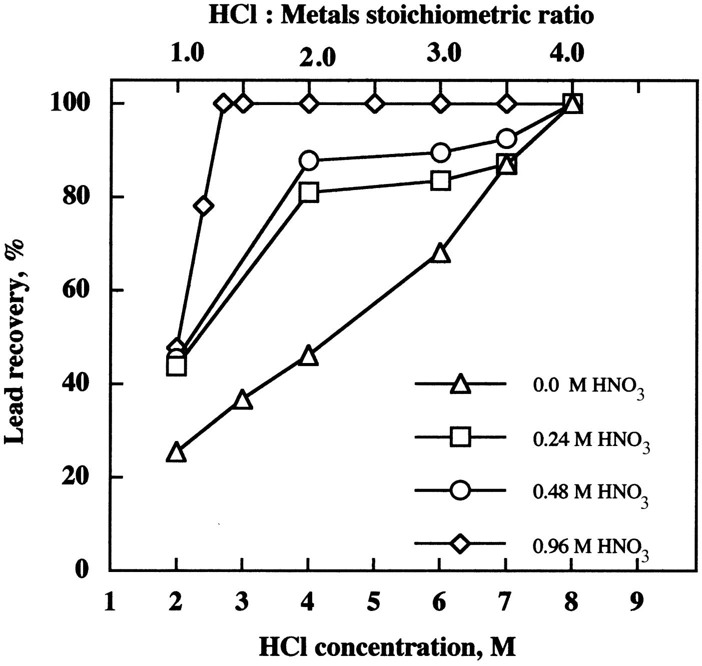

FIG. 5 Effect of acid concentration on the recovery of lead from scrap. Leaching time: 2 h, temp.: 80°C.

$$2Cu + O \rightarrow Cu_2O \qquad (2)$$

$$Cu_2O + O \rightarrow 2CuO \qquad (3)$$

$$CuO + 2HCl \rightarrow CuCl_2 + H_2O \qquad (4)$$

On the other hand, in spite of the higher electronegativity of zinc ($E^\circ_{zn} = -0.76$), the evolved nascent hydrogen in the displacement reaction with zinc is adsorbed on the reacting surface and constitutes a barrier for progressive acid attack. The rate of zinc–acid reaction gradually decreases unless excess heat energy or an oxidizing agent is supplied to overcome the higher hydrogen overvoltage. Nitric acid enhances zinc dissolution via the oxidation effect of hydrogen gas and consequently the gaseous barrier is removed. The oxidation effect increases with increasing the concentration of HNO_3, which leads to accomplishment of the leaching reactions with lower HCl concentration.

Figures 4 and 5 show the recovery efficiency of tin and lead, respectively. Tin and lead could be recovered completely using only hydrochloric acid. The metal recovery was directly proportional to the acid concentration, reaching greater than 99% using 8 M HCl. However, the addition of HNO_3 improved the recovery with lower HCl concentrations. The optimum acids concentrations are 2.7 M HCl (equivalent to 1.35 HCl: metals stoichiometric ratio) and 0.96 M HNO_3. The trends of tin and lead recoveries being affected by acid concentration are close to each other, which is possibly attributed to their close values of standard potentials ($E^\circ_{Sn} = -0.137$, $E^\circ_{Pb} = -0.126$).

The effect of liquid : solid ratio (L : S) on the recovery of Cu, Zn, and Sn at the optimum acids concentrations was studied and the results are presented in Fig. 6. The recovery percentage of Cu, Zn, and Sn increased gradually with increasing acid volume, where complete recovery was achieved using L : S of 15 mL/g. At lower acid volumes the recovery percentage decreased in the order Zn > Cu > Sn. The recovery of lead is not given in Fig. 6 due to the precipitation inconveniences. A complete recovery of Pb was attained at L : S of 15 mL/g. However, at lower L : S, the scrap was partially dissoluted. The insoluble residue of scrap was contaminated with the pro-

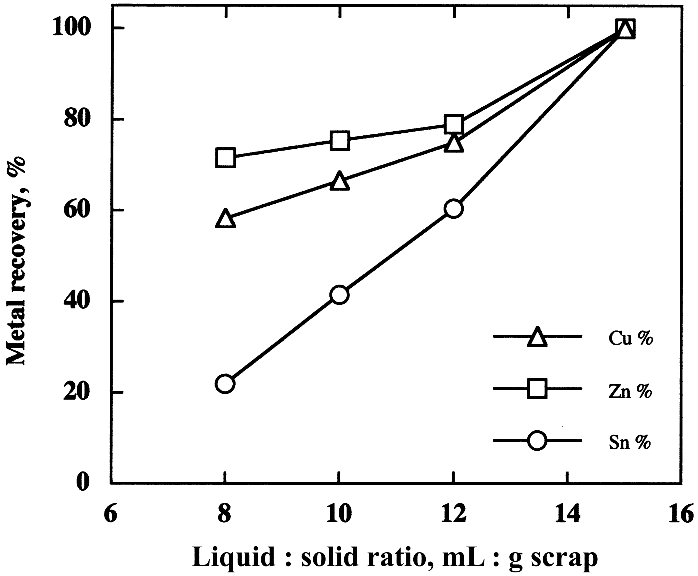

FIG. 6 Effect of liquid: solid ratio on recovery of copper, zinc, and tin from brass scrap. [HCl]: 2.7 M, [HNO_3]: 0.96 M. Leaching time: 2 h, temp.: 80°C.

duced precipitate of lead chloride, which started to appear at <80°C due to saturation.

The effect of temperature on recovery is illustrated in Fig. 7. The metal recovery is directly proportional to the leaching temperature, reaching complete recovery at 80°C. At lower temperatures the recovery efficiency decreased in the order Sn > Cu > Zn. The retarding rate of zinc recovery in spite of its higher electronegativity may be due to hydrogen overpotential effect at the surface. The recovery of lead was nearly complete at ≥ 80°C, and precipitation took place at lower temperatures.

Table 2 gives the effect of HCl concentration on time of complete recovery of metals from the scrap at 80°C, and 0.96 M HNO_3. Upon using 2.7 M HCl, the required time for complete recovery was 60 min. Increasing the acid concentration up to 6 M reduced the leaching time to 25 min.

Liquid–Liquid Extraction

At optimum conditions, a selected organic reagent can react selectively with copper without interferences from other accompanying metals. PT-5050Z contains an active aromatic-oxime group and can form a stable complex with copper. This reagent behaves as an acid in the extraction of metals, thus the extraction is mainly dependent on the acidity of the aqueous solution.

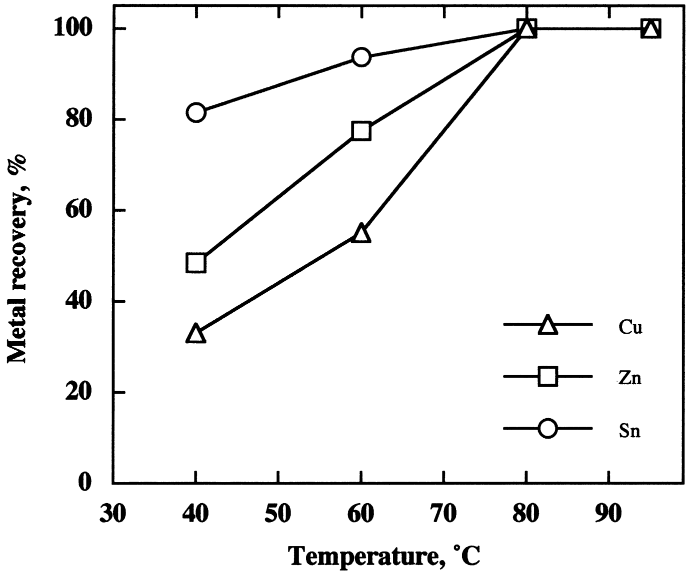

FIG. 7 Effect of temperature on recovery of copper, zinc, and tin from brass scrap. [HCl]: 2.7 M, [HNO_3]: 0.96 M. Leaching time: 2 h.

TABLE 2
Effect of HCl Concentration on Time of Complete Recovery of Metals from the Scrap. [HNO_3]: 0.96 M

HCl conc., M	2.7	3	4	6
Time, minutes	60	45	30	25

When the organic solution containing PT-5050Z comes in contact with an aqueous solution containing copper, the copper will be distributed between the organic and the aqueous phases. The transfer of copper from organic to aqueous phase is accompanied by the transfer of H^+ in the reverse direction. This is represented by Eq. 5

$$Cu_a^{++} + 2RH_o \rightleftharpoons R_2Cu_o + 2\,H_a^+ \tag{5}$$

where the subscripts *a* and *o* represent the aqueous and organic phases, respectively, and *RH* denotes the organic extractant, PT-5050Z.

Synthetic Solutions

Synthetic solutions containing 300 ppm each of Cu, Zn, and Pb were first tested to obtain the optimum conditions for separation that will be applied to the real leach solution. The extraction behavior of Cu, Zn, and Pb from their mixture solutions in different acid concentrations (from 5 M to 0.01 M HCl, and from pH 2 to 10) is shown in Fig. 8. The extraction percentage was plotted against −log HCl concentration and the pH. Copper extraction increased with the decrease in hydrogen-ion concentration, attaining complete extraction with 0.1 M HCl. Almost all zinc remained in the aqueous phase of strong acidity and the extraction proceeded above pH 5, where complete extraction was obtained in the alkaline region. Lead was not extracted from the tested solutions. The superior extraction of copper over zinc and lead at a wide range of pH (from 1 to 5) suggested the effective separation of copper from other metals in the leached scrap solutions.

The extraction separation of Cu, Zn, and Pb in 0.1 M HCl was performed using different concentrations of PT-5050Z in kerosene, and the results are presented in Fig. 9. Copper extraction rapidly increased with increasing the PT-5050Z concentration, whereas the increase in zinc extraction was slow. Lead remained in the aqueous phase along with the different concentrations of the extractant. Only 10% of PT-5050Z concentration was enough for complete extraction of copper without any significant extraction of zinc. Concentrations higher than 10% resulted in an increase in zinc extraction.

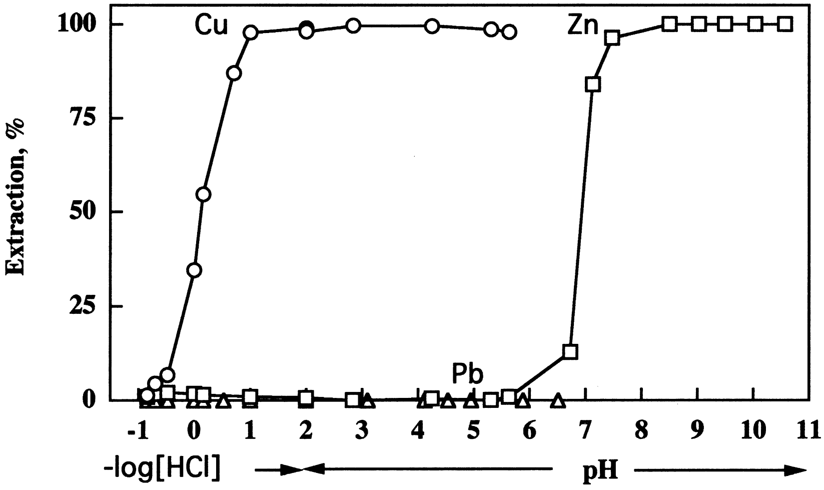

FIG. 8 Effect of acidity on solvent extraction of copper, zinc, and lead with PT-5050Z. Aqueous phase: 300 ppm each metal; organic phase: 10% PT-5050Z in kerosene; shaking time: 1 h.

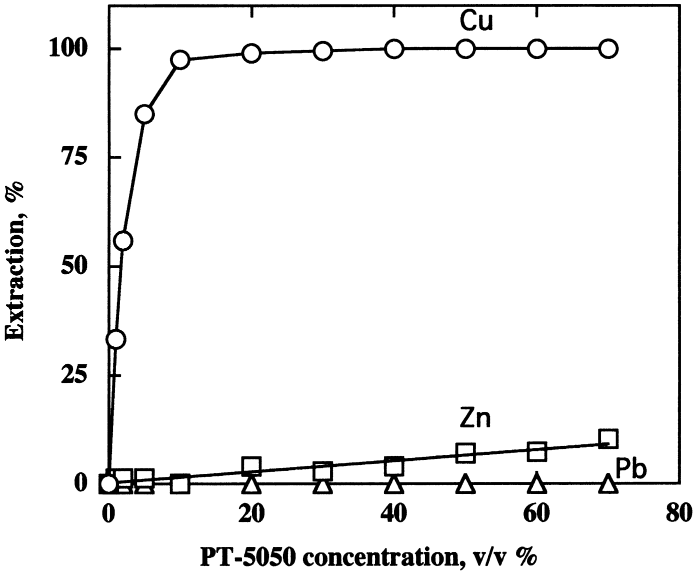

FIG. 9 Effect of PT-5050Z concentration on solvent extraction of copper, zinc, and lead. Aqueous phase: 300 ppm each metal in 0.1 M HCl; organic phase: PT-5050Z in kerosene; shaking time: 1 h.

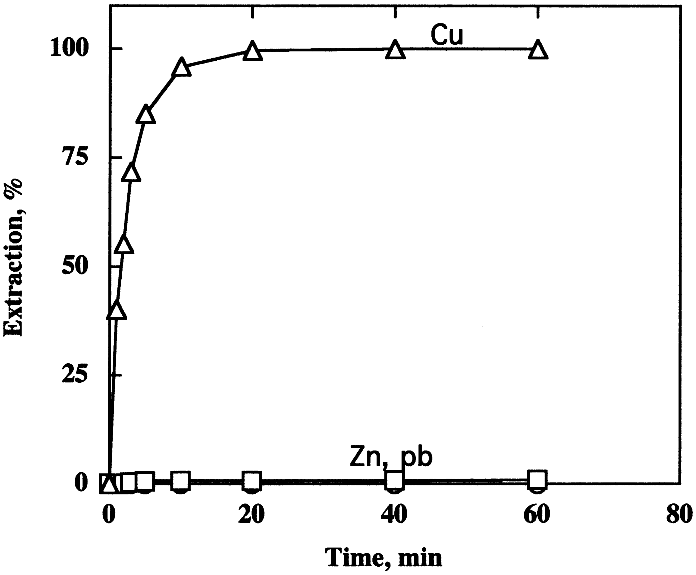

FIG. 10 Effect of shaking time on solvent extraction of copper, zinc, and lead. Aqueous phase: 300 ppm each metal in 0.1 M HCl; organic phase: 10% PT-5050Z in kerosene.

Extraction of Cu, Zn, and Pb with 10% PT-5050Z from 0.1M HCl was performed for different periods of time (Fig. 10). Complete extraction of Cu was achieved after 20 min, while extraction of Zn and Pb was very poor, even after 60 min. Stripping of copper from the resulting organic solution was tested using 3 M H_2SO_4. This relatively high acid concentration favors the transfer of copper to the aqueous phase due to the dissociation of the formed complex. The stripping was completed after only 5 min.

Real Leach Solution

From the above results it can be concluded that to separate copper effectively the acidic leach solution of brass scrap would be partially neutralized to pH ≥ 1 and <5. Ammonium hydroxide has been used to adjust the pH to about 2.6, at which Sn will be precipitated and separated by filtration. The solution is then subjected to solvent extraction of copper without further pH adjustment (see Fig. 1). This solution contains about 36 g/L Cu, 16 g/L Zn, and 1 g/L Pb. Several stages of extraction are required to separate all the copper content with 10% PT-5050Z.

Figure 11 shows the cumulative extraction percentage of copper in each stage with different organic : aqueous (O : A) phase ratios. The acidity of the

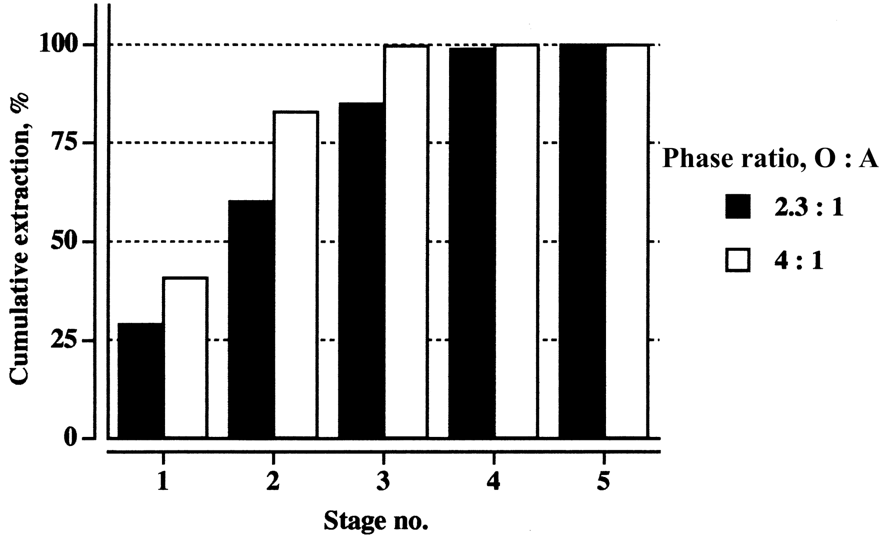

FIG. 11 Multistage extraction of copper from real leach solution. Aqueous phase: 36 g/L Cu, 16.2 g/L Zn, 1 g/L Pb, pH 2.6; organic phase: 10% PT-5050Z in kerosene; shaking time: 20 in.

aqueous solution increased with extraction due to the transfer of H^+ from organic to aqueous phase as indicated in Eq. (5). Thus, the pH of the aqueous solution was readjusted to pH 2.6 with NH_4OH to keep copper extraction high. About 99.98% of copper was extracted in the third stage using 4 : 1 (O : A), and almost all amounts of Zn and Pb remained in the aqueous solution. The stripping conditions of synthetic solutions were applied to release copper from the loaded organic phase. The resulting copper sulfate solution was freed from the metals of the scrap and could then be subjected to electrowinning to produce high-purity copper metal or crystallization.

The raffinate was then treated with alkalis to precipitate Zn and Pb, as shown in Fig. 1.

CONCLUSION

Brass-alloy scrap from car-radiator tubes could be completely leached with HCl solution in the presence of HNO_3 as an oxidant for subsequent recovery and separation of metal values. The proper leaching conditions were 2.7 M HCl and 0.96 M HNO_3 for 1 h at L : S of 15 mL/g scrap at 80°C.

Synthetic metal solutions showed that copper was selectively separated by solvent extraction using 10% PT-5050Z in kerosene at $pH \geq 1$ and <5, for 20 min, and stripped from the loaded organic phase using 3 M H_2SO_4. From the real leach solution, almost all copper was extracted applying three extraction stages with O : A of 4 : 1.

A process flow sheet was designed and used for separation of metal values from scrap. About 61% of the lead was separated as lead chloride by cooling the leaching solution to 10°C. Tin was separated as hydrated tin oxide by adding an ammonia solution until pH 2.6 was reached. Copper was separated from the resulting solution by solvent extraction. Zinc was separated from the raffinate as carbonate using Na_2CO_3 at pH 6.5. The remaining lead was separated as hydroxide at pH 9 using NaOH.

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