



Contents lists available at ScienceDirect

Waste Management

journal homepage: www.elsevier.com/locate/wasman

Copper leaching from electronic waste for the improvement of gold recycling

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ARTICLE INFO

Article history:

Received 8 December 2015

Revised 4 March 2016

Accepted 4 March 2016

Available online xxxxx

Keywords:

Leaching

Copper

Gold

Electronic waste

ABSTRACT

Gold recovery from electronic waste material with high copper content was investigated at ambient conditions. A chemical preliminary treatment was found necessary to remove the large quantities of copper before the precious metal can be extracted. For this purpose inorganic acids (HCl, HNO₃ and H₂SO₄) and two organic substances EDTA and citrate, were tested. The effect of auxiliary oxidants such as air, ozone and peroxide hydroxide was studied. In pretreatments with peroxide and HCl or citrate, copper extractions greater than 90% were achieved. In the second leaching stage for gold recovery, the solid residue of the copper extraction was contacted with thiourea solutions, resulting in greater than 90% gold removal after only one hour of reaction.

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1. Introduction

In recent years, two simultaneous and contradictory tendencies have occurred world-wide: the first is a renewed interest in environmental protection seeking environmental sustainability, while the second is an exponential increase in the acquisition of electronic devices. Generally, these devices are manufactured using a variety of new materials, rare earths elements, alloys and/or precious metals. Some components, if not disposed of or recycled properly, can generate toxic substances, such as BFR's, Sb, Ni, and Pb, causing environmental problems (Kiddee et al., 2013).

The solution to this paradox, at least partially, involves recovering these metallic values from electronic residues, in this study called EW-PCB (acronym for Electronic Waste from Printed Circuit Boards). A variety of methodologies have been employed to recover especially the precious metals, included in the waste material generated by the constant renovation of electronic devices. The electronic waste usually consists of extremely irregular mixtures of several types of materials, which include polymers, metals and fiberglass.

The electronic components or printed circuits consist of in a polymeric substrate upon which copper is deposited. The majority include a very thin precious metal coating, in order to increase the

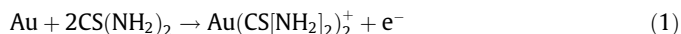
service life (from corrosion and wear) of the elements, such as contactors. In general, the preferred metal for this purpose is gold. Some studies have demonstrated that, in addition to copper and gold, a common electronic card can contain up to 15 additional metals, either in elemental form or as alloys (Cui and Zhang, 2008; Tuncuk et al., 2012; Yang et al., 2011). It is important to note that most of the residues generated by this type of components contain polymers, which can generate an enormous environmental impact, if they are processed by smelting (Guo et al., 2015).

Different process routes for the gold recovery from electronic waste have been reported (Behnamfard et al., 2013; Birloaga et al., 2014a,b). These treatments include total fusion stage, followed by leaching and recovery of metal values (Cui and Zhang, 2008; Guo et al., 2015; Tuncuk et al., 2012). Other authors employ ionic liquids, mixed with acids, to leach base metals (Chen et al., 2015). Precious metals aqueous processing includes the leaching stage with solutions of thiosulfate, thiocyanate, thiourea, cyanidation or total digestion by aqua regia (Akcil et al., 2015; Syed, 2012). Of these, thiourea has several advantages over the others, the most important of which are the high processing rate and the capacity to recycle the solution once the metal values are stripped or precipitated (Poisot-Díaz et al., 2008). In order to design an eco-friendly procedure for recycling the metallic content of electronic waste, specifically for gold, the traditional methods (with aqua regia or cyanidation) should not be considered (Akcil et al., 2015).

The anodic reaction (Eq. (1)) between metallic gold and thiourea can be expressed as follows:

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However, this methodology presents the disadvantage that copper is a metal that consumes and degrades thiourea (Behnamfar et al., 2013; Birloaga et al., 2014a,b; Syed, 2012). For this reason, a preliminary treatment is required to remove the majority of copper present in the EW-PCB, before leaching with thiourea (or other agents). In this manner, the metallurgical extraction of noble metals can be effectively accomplished.

For copper extraction from EW-PCB, leaching procedures based on the use of inorganic acids have shown acceptable results. However, in most cases, elevated acid concentrations (up to 2.5 M), considerable quantities of oxidants (up to 15% by volume) and process temperatures above 70 °C were necessary (Calgaro et al., 2015; Ghosh et al., 2015; Silvas et al., 2015). In general, these procedures do not take into account the large amount of liquid and solid waste generated and its management. The present study proposes to demonstrate that less contaminating aqueous processes at more moderate operating conditions, with recovery efficiencies similar to the more traditional methods, can be developed. The principal advantages sought after are the following: low toxicity, possibility of solution re-utilization and ease of management. The principal objective of the study consists on the selective extraction of the majority of copper from the EW-PCB residues, through the use of both conventional inorganic acids as well as carboxylates such as EDTA (diaminoethanotetraacetic acid) and citrate (trisodium 2-hydroxypropane-1,2,3-tricarboxylate), focusing on the improvement of the subsequent precious metals extraction in a thiourea leach of the pretreated materials. The influence of the inorganic acid concentration, in combination, with different oxidizing agents (air, hydrogen peroxide and ozone), on the copper leach will be presented. For the carboxylate systems, the effect of solution pH and reagent concentration will be analyzed. Finally, results of the gold extraction with the thiourea solution will be compared for the most successful pretreatment residues.

2. Material and methods

The study was performed using electronic waste with high copper contents. The electronic components were manually separated from each of the printed cards (EW-PCB), leaving only the substrate with traces of welding (Fig. 1). Once selected, a preliminary milling was performed and the EW-PCB was classified by particle size. Finally, a total digestion with aqua regia was carried out for each particle size and the elemental analysis determined by atomic absorption spectrometry (Varian SpectrAA 220fs).

The chemical pre-treatment for copper removal was accomplished using solutions formulated with analytical grade (J.T. Baker) inorganic acids and organic sodium salts (EDTA and citrate) at

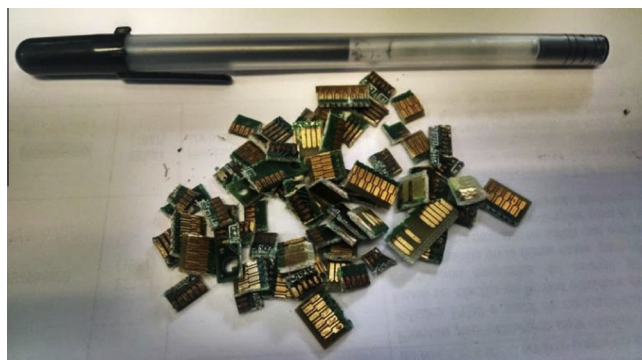


Fig. 1. Example of the unground EW-PCB material used in the experiment.

specific molar concentrations for each experiment and diluted with deionized water (millipore Milli-Q). Atmospheric air (without treatment) was supplied by an adjustable pump at a gas flow rate of 1 L/min. In those experiment requiring hydrogen peroxide, the dosification was practiced using the standard reagent grade (30% w/v, J.T. Baker). The ozone injected into the system was generated in-situ and sparged into the pretreatment solution/EW-PCB mixture. Medicinal grade oxygen (99%, Praxair) was passed through a UV lamp (185 nm) at a flowrate of 1 L/min to produce a constant oxygen-ozone supply, equivalent to 175 mg_{ozone}/L_{dissolution} min.

To determine the conventional behavior in each of the acids during leaching, treatments were performed with constant stirring for 24 h at a concentration of 1 M for each acid. Since the objective of this study consists of analyzing the behavior of inorganic acids in the presence of additional agents, many of leaching parameters, such as temperature and stirring velocity, were held constant.

All leaching experiments were performed in triplicate at 25 °C and ambient pressure, using a mechanical agitation of 125 rpm, with a constant solid-liquid ratio of 20 g EW-PCB per liter of leaching solution. In those tests where gas was sparged (either air or air/ozone mixtures), a Graham condenser, cooled with commercial ethylene glycol (30% by weight) at a temperature of 0 °C, was employed to avoid evaporation of the solution during the leaching process.

The leaching solutions were prepared with the required acid or reagent concentration. For the organic acid tests, the solution pH in each case was adjusted with H₂SO₄. Subsequently, the specific amount of solid (EW-PCB) was added corresponding to the volume of solution employed. In each case, the experiment was initiated upon the commencement of the gas sparge and/or the dosification of peroxide, after which time liquid samples were withdrawn at predetermined intervals. Upon termination of the experiment, the liquid-solid suspension was filtered and air dried. The quantification of metals (copper or gold) in the filtered leach liquor and in the aliquots taken during the process was performed using atomic absorption spectrometry (Varian SpectrAA 220fs). The experimental values reported, both in numerical and graphical form, correspond to the arithmetic averages of the results obtained in triplicate at the specific operating conditions; the error was also incorporated into the table and figures.

All thermodynamic data used in the document were obtained from the NIST database (Database 46–Vers. 8.0). The corresponding diagrams were designed using the MEDUSA© software (Making Equilibrium Diagrams Using Simple Algorithms) (Eriksson, 1979; NIST, 2004; Puigdomenech, 2004).

3. Results and discussion

3.1. Distribution of metal content

The EW-PCB solid samples were digested and analyzed to establish the total amount of metals for several size fractions, which in most cases exceeds 84% of the total weight of the material (Table 1). The preliminary selection by size indicated that gold, as

Table 1
Average chemical content for the principal metals in the EW-PCB with respect to particle size.

Particle size (μm)	≥500	500–400	400–177	<177
Cu (kg/t)	960.0 ± 0.2	898.9 ± 0.2	848.1 ± 0.1	851.1 ± 0.1
Au (g/t)	83 ± 2	48 ± 2	200 ± 1	108 ± 2
Ni (g/t)	169 ± 2	169 ± 2	183 ± 2	13 ± 2
Zn (kg/t)	1.13 ± 0.21	0.97 ± 0.16	1.63 ± 0.11	1.71 ± 0.16
Fe (kg/t)	2.05 ± 0.53	1.97 ± 0.76	10.87 ± 0.33	4.68 ± 0.47
Pb (kg/t)	2.23 ± 0.88	4.76 ± 0.95	1.48 ± 1.13	2.76 ± 1.07

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