



Research article

Selective separation of Cu, Ni and Ag from printed circuit board waste using an environmentally safe technique



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ABSTRACT

Printed circuit boards (PCBs) make up a large part of e-waste and include high concentrations of high-value metals. Therefore, the recovery of these metals is interesting from both the environmental and economic points of view. Here, the extraction/separation of copper, nickel and silver from PCB leachate was studied using an aqueous two-phase system (ATPS) formed by triblock copolymers with an electrolyte and water, which is in compliance with the principles of green chemistry. The best conditions for the selective extraction consisted of 1-(2-pyridylazo)-2-naphthol (3.5 mmol kg^{-1}) at pH = 6.0 in 6 sequential steps for the Cu(II), dimethylglyoxime ($5.00 \text{ mmol kg}^{-1}$) at pH = 9.0 for the Ni(II) and thiocyanate ($5.20 \text{ mmol kg}^{-1}$) at pH = 9.0 for the Ag(I). These conditions were applied sequentially for extraction of Cu, Ni and Ag from the PCB leachate, obtaining high separation factor (S) values between the analyte and the metallic concomitants ($S_{\text{Cu,Ni}} = 1,460$, $S_{\text{Cu,Fe}} = 15,500$, $S_{\text{Cu,Ag}} = 15,900$, $S_{\text{Ni,Fe}} = 32,700$, $S_{\text{Ni,Ag}} = 34,700$ and $S_{\text{Ag,Fe}} = 4800$). The maximum extraction percentages (% E) for Cu, Ni and Ag were 99.9%, 99.9% and 99.8%, respectively. After the extraction, a single step stripping process was performed, resulting in more than 82% of the ion available in a clean lower phase. For the first time, an ATPS has been used for sequential extraction of several metal analytes from a real sample.

1. Introduction

New electronic products come on the market every year due to rapid technological evolution, which not only amplifies the applications and uses of such equipment but also makes them obsolete very quickly. The accelerated obsolescence of these products has been highlighted as an increasingly significant problem. In both developed and developing countries, these materials represent the fastest-growing type of solid waste in the world (Arshadi and Mousavi, 2014). This equipment consists in part of printed circuit boards (PCBs), whose essential function is the mechanical support of the components and the electrical connectivity of the circuits. PCBs account for approximately 30% of all electronic scrap generated, with 28–32% of the content being metal (10–20% copper, 15% lead, 13% nickel, and 0.3–0.4% precious metals such as silver, gold and platinum) (Sarvar et al., 2015).

As government regulations are not strictly enforced, electronic equipment is often disposed of in landfills, where it contaminates the soil and groundwater with heavy metals (Canal Marques et al., 2013).

The effects of these contaminations on the environment are serious (Anderson et al., 2017; Yang et al., 2017; Zongo et al., 2017) because these elements tend to accumulate in the tissues of organisms, including humans, causing substantial disorders such as damage to the blood composition and the central nervous system (Abukhadra et al., 2018). In addition to the environmental and legal issues, the recovery of precious metals from PCB residues has become of economic interest because the metals represent more than 95% of the total value of the board. Moreover, their concentrations in the residues are up to 10 times higher than in raw ores; accordingly, this type of recovery has been called urban mining (Ghosh et al., 2015).

The processes for recycling PCBs can be mechanical (comminution, classification and separation), biological (Rodrigues et al., 2015) or chemical (pyrometallurgy, hydrometallurgy, electrometallurgy and biometallurgy) (Birloaga et al., 2014; Birloaga and Vegliò, 2016). The chemical processes allow a refinement of the process, so they are widely used, especially hydrometallurgy.

The hydrometallurgical process is characterized by obtaining metals

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with high purity. For this, the techniques used in sequential stages include leaching, separation of the metal species of interest (primarily solvent extraction, SE) and electrolysis. This process has several advantages: efficient separation of major scrap components, reduction of processing costs (low energy consumption and recycling of chemical reagents) and applications to complex and low-metal samples (Birloaga et al., 2013; de Lemos et al., 2012).

The most critical step of the hydrometallurgical route is SE, which is used for the separation, purification or preconcentration of a given analyte present in an aqueous phase that is in contact with an organic phase. However, the organic solvents used are toxic and flammable and form stable emulsions, which makes the phase separation process slow. Therefore, solvent extraction does not fit within the principles of sustainable chemistry (Armenta et al., 2015; Rodrigues et al., 2013). One promising alternative to replace SE, based on the principles of green chemistry, is aqueous two-phase systems (ATPS). These systems are composed primarily of water, and their other components can be non-toxic, non-flammable and biodegradable, which makes them an environmentally safe extraction system. In addition, their constituents are commercially accessible and inexpensive (de Oliveira et al., 2017; Santos et al., 2015).

An ATPS may be formed by mixing aqueous solutions of polymer and salt. After a brief time period, the phase separation occurs spontaneously. The phase split results in a polymer-enriched top phase (TP) and a salt-enriched bottom phase (BP). Additionally, these systems present high water content in both phases (Sosa et al., 2017). The overall composition of the ATPS as well as the composition of each phase is known through the respective phase diagrams (de Barros et al., 2016; Hamta and Dehghani, 2017). These systems have already been applied successfully in the separation of bioparticles (de Barros et al., 2016), dyes (Borges et al., 2016) and other analytes (Delgado et al., 2017; Murari et al., 2017). Furthermore, ATPS have been efficient in the extraction/separation of metal ions from both synthetic and real samples (de Lemos et al., 2012, 2013; de Oliveira et al., 2017).

This work aimed to develop an approach for the sequential extraction/separation of copper, nickel and silver from leachates of printed circuit boards in aqueous two-phase systems. These analytes have high added value and are present in high concentrations in this electronic scrap.

2. Materials and methods

2.1. Materials

The copolymer poly(ethylene oxide)₁₃-poly(propylene oxide)₃₀-poly(ethylene oxide)₁₃ with an average molar mass of 2900 g mol⁻¹ and 40% ethylene oxide ((EO)₁₃(PO)₃₀(EO)₁₃), denoted L64, was purchased from Sigma Aldrich (Milwaukee, USA). Hydrogen peroxide (H₂O₂), magnesium sulfate (MgSO₄·7H₂O), sodium hydroxide (NaOH) and iron chloride (FeCl₃·6H₂O) were obtained from VETEC (Rio de Janeiro, Brazil). Nickel chloride (NiCl₂·6H₂O), copper sulfate (CuSO₄·5H₂O), and silver nitrate (AgNO₃) were purchased from ISOFAR (Duque de Caxias, Brazil). Sulfuric acid (H₂SO₄) and nitric acid (HNO₃) were obtained from Merck (Darmstadt, Germany). The extractants 1-(2-pyridylazo)-2-naphthol (PAN), potassium thiocyanate (KSCN) and dimethylglyoxime (DMG) were purchased from VETEC (Rio de Janeiro, Brazil), dithizone (Dz) was obtained from DINÂMICA (Diadema, Brasil) and 1-nitroso-2-naphthol (1N2N) was obtained from DIDÁTICA SP (Guarulhos, Brasil). All reagents were of analytical grade quality and were used as received, without further purification.

2.2. Sample preparation

The PCBs used in this work came from obsolete or defective personal computers. The PCBs were disassembled manually and all metal parts were removed. This material (23.458 g of sample) was leached

with 2 M sulfuric acid solution at a proportion of 1:10 (sample mass (g):volume of solution (mL)) and 50 mL of 30% (v/v) hydrogen peroxide solution at 80 °C for 180 min (Birloaga et al., 2014). The leachate was cooled, filtered and diluted in a 1.00 L volumetric flask. The amount of metals present in the leachate was quantified using flame atomic absorption spectrometry (FAAS) (Varian AA50). The instrumental conditions of the measurements in the FAAS are presented in Table S1. The pH value of the leachate was 1.03. Subsequent studies focused on the optimization of a sequential extraction of copper, nickel and silver from the PCB leachate.

2.3. Study of metal extraction

The ATPS used in the metal extractions was prepared by mixing stock solutions of polymer L64 (49.86% m/m), MgSO₄ electrolyte (17.86% m/m) and leachate, obtaining a system with 3.00 g of the TP and 3.00 g of the BP (Rodrigues et al., 2009). For all experiments, the pH of the water used in the preparation of the salt and polymer stock solutions was adjusted to either 3.00, 6.00 or 9.00. The PAN extractor was added in different concentrations (Table S2) to the polymer solution. Then, in a test tube, 3.00 g of the polymer stock solution containing the extractor, 2.50 g of the electrolytic stock solution of MgSO₄ and 0.50 g of the leachate were weighed. The tube was agitated (vortex for 1 min), and, after reaching an equilibrium thermodynamic state at 25 °C (controlled by a thermostatic bath), upper and lower phase aliquots were collected and diluted properly.

For the sequential extraction, 25.0 g of the polymer stock solution containing the PAN extractor (3.5 mmol kg⁻¹), 20.85 g of the electrolytic stock solution and 4.15 g of the leachate were added to a tube. The BP was removed and placed in contact with a new polymer stock solution containing the extractor. This procedure was repeated for a total of 6 iterations.

The metal concentrations were determined in the phases using the FAAS technique. The percentage of extraction (%E) of the metal ions was calculated through Equation (1).

$$\%E = \frac{nM_{TP}}{nM_t} \times 100 \quad (1)$$

where nM_{TP} is the mol of metal ions in the TP and nM_t the total mol of metal ions in the system. The standard curve was prepared using the matrix compatibilization principle.

To evaluate the ability of the system to separate the metal ions, the separation factor (S) was calculated. To calculate this parameter, equations (2) and (3) were used.

$$D_M = \frac{\%E}{100 - \%E} \quad (2)$$

$$S_{M,N} = \frac{D_M}{D_N} \quad (3)$$

where D_M is the distribution coefficient of the analyte, D_N is the distribution coefficient of the concomitant metal and $S_{M,N}$ is the separation factor between analyte and concomitant metal.

For the nickel and silver extraction studies, a synthetic solution containing Ni(II), Fe(III) and Ag(I) was prepared at the same concentrations of the metals and the pH of the original leachate. The methodology used was similar to that described for Cu(II). However, the extractors used for the Ni(II) studies were 1-nitroso-2-naphthol or dimethylglyoxime and those used for Ag(I) were dithizone or thiocyanate in different concentrations (Table S2). The scheme of the experimental procedure is showed in Fig. S1.

2.4. Sequential extraction from the real sample and stripping studies

The first six steps of the sequential extraction were performed as described in the previous section with the objective of completely

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