Bioleaching of metals from WEEE shredding dust

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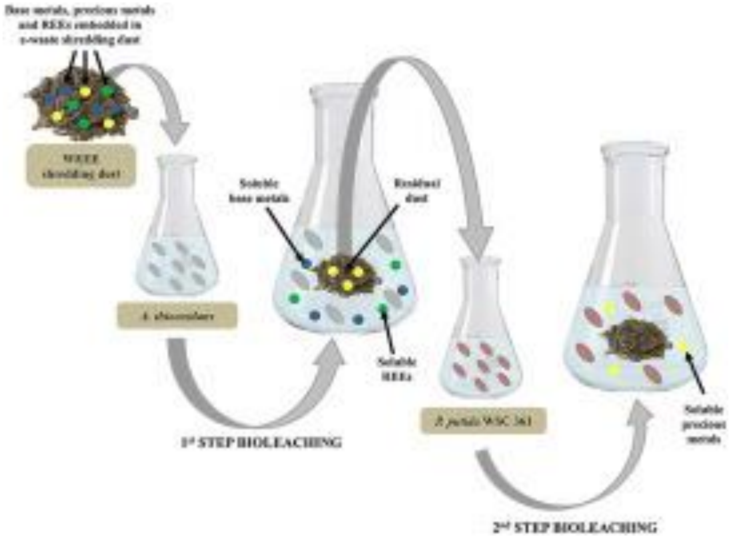
Highlights

- Valuable and critical metals can be bio-extracted from WEEE shredding dust.
- Bioleaching by *Acidithiobacilli* provided high REE extraction yields from WEEE dust.
- Au leaching by biogenic cyanide was less effective than REE leaching by acidophiles.

Abstract

A bioleaching process developed in two separate steps was investigated for the recovery of base metals, precious metals and rare earth elementsfrom dusts generated by Waste Electrical and Electronic Equipment (WEEE) shredding. In the first step, base metals were almost completely leached from the dust in 8 days by *Acidithiobacillus thiooxidans* (DSM 9463) that lowered the pH of the leaching solution from 3.5 to 1.0. During this step, cerium, europium and neodymium were mobilized at high percentages (>99%), whereas lanthanum and yttrium reached an extraction yield of 80%. In the second step, the cyanide producing *Pseudomonas putida* WSC361 mobilized 48% of gold within 3 h from the *A. thiooxidans* leached shredding dust. This work demonstrated the potential application of biohydrometallurgy for resource recovery from WEEE shredding dust, destined to landfill disposal, and its effectiveness in the extraction of valuable substances, including elements at high supply risk as rare earths.

Graphical abstract



Keywords

Waste electrical and electronic equipment
Secondary source
Rare earth elements
Precious metals
Recovery
Circular economy

1. Introduction

As metals are largely used for manufacturing electronic products, the electrical and electronic equipment reaching the end-of-life, known as Waste Electrical and Electronic Equipment (WEEE) or e-waste, is regarded as a potential secondary source of these elements (Hageluken, 2006, Lee and Pandey, 2012, Tuncuk et al., 2012). The recycling of metals from WEEE represents an important opportunity to feed resources back to the economy, lowering the environmental impacts as well as the energy consumption associated with raw material supply, as promoted by the circular economy concept (Hageluken, 2006). In this view, WEEE recycling plays a key role as this waste stream has a fast growth rate per year (Robinson, 2009) and moreover contains strategic elements, such as precious metals and rare earth elements (REEs) (Schüler et al., 2011). Mining REEs from WEEE is particularly attractive since these elements have been listed by the European Commission as the most critical raw materials at supply risk due to their growing demand by several process industries and their scarce worldwide production, mainly restricted to a single country, i.e. China (Binnemans et al., 2013, European Commission, 2014, European Commission, 2010). Furthermore, the recycling of WEEE can bring benefits not only in terms of raw resource supply, but it can also contribute to the reduction of potential environmental pollution and human health risks related to the release of the hazardous substances present in WEEE as a result of improper management practices (Zeng et al., 2017).

Mechanical, pyrometallurgical and hydrometallurgical processes are used for the recovery of metals from WEEE (Cui and Zhang, 2008, Khaliq et al., 2014, Priya and Hait, 2017). Besides these conventional techniques, emerging technologies in the field of electrochemistry, supercritical fluids, mechanochemistry and ionic liquids are receiving increased attention (Tan and Li, 2015, Wang et al., 2017). Over the recycling chain, mechanical treatments are employed in the early stage as pre-processing, aiming at separating the larger valuable and hazardous components and upgrading the metal fraction, which is further routed to end-refining processes (Cui and Forssberg, 2003). Pyrometallurgy and hydrometallurgy are metallurgical techniquescoming from the mineral sector, employed as end-processes for the recovery of the metal of interest (Cui and Zhang, 2008). These processes are, however, not yet effectively targeted on the extraction of REEs, whose recycling rates are still low (Binnemans et al., 2013). During the mechanical treatment, REEs as well as large amounts of precious metals end up in the output fractions as dust or ferrous materials, usually not involved in the metallurgical refining process (Bachér and Kaartinen, 2016, Tsamis and Coyne, 2015). REEs are also easily lost during pyrometallurgical processes, since these elements are generally entrapped in the slag phase due to their strong affinity with oxygen (Binnemans et al., 2013, Haque et al., 2014). Conversely, hydrometallurgy has been considered as an effective method for the recovery of REEs, although its industrial application is still limited (Tunsu et al., 2015).

Current metallurgical treatments have, moreover, several impacts on the environment due to the generation of secondary pollutants. The necessity to set environmentally sound treatments, increasing the sustainability of recycling processes (Canal Marques et al., 2013), has thus moved the research interest towards biohydrometallurgy as a promising technique for metal recovery from WEEE (Cui and Zhang, 2008, Khaliq et al., 2014, Priya and Hait, 2017). Biohydrometallurgy exploits either the ability of microorganisms to solubilize the metals contained in the solid matrix (bioleaching) or the capacity of microbial biomass to sorb metals from the aqueous solution (biosorption) (Erüst et al., 2013, Hoque and Philip, 2011, Olson et al., 2003, Zhuang et al., 2015). The feasibility of biohydrometallurgy has been demonstrated for the extraction of metals from ores (Bosecker, 1997, Brierley and Brierley, 2013, Brierley and Brierley, 2001, Watling, 2016) and electronic waste (Beolchini et al., 2012, Cui and Zhang, 2008, Ilyas and Lee, 2014, Işıldar et al., 2016) as well as for the removal of metals from aqueous solutions (Andrès et al., 2003, Vijayaraghavan and Yun, 2008, Wang and Chen, 2009, Won et al., 2014). In this regard, several autotrophic and heterotrophic bacteria as well as fungi have been involved in WEEE bioleaching studies (Madrigal-Arias et al., 2015, Rozas et al., 2017, Zhang and Xu, 2016) which, however, mainly focused on the extraction of single metals, particularly copper and gold, from printed circuit boards (PCBs). Despite the increasing interest in biotechnology, the potential application of this technique needs to be further explored, especially for the recovery of critical metals from WEEE as the REE-microbe interactions are not clearly understood and the bio-recovery of REEs from secondary sources, including WEEE, is poorly documented (Barmettler et al., 2016, Ilyas et al., 2017).

This study focused on the recovery of base metals, precious metals and REEs from WEEE shredding dust through biohydrometallurgical processes. The work aimed to investigate (i) the application of bioleaching to dusts originating from a WEEE mechanical treatment facility and (ii) its effectiveness for the recovery of different metals contained in WEEE. To this end, the bioleaching experiments were carried out in two separate steps in order to assess if a selective recovery of the metals could be achieved. The first step aimed for the extraction of base metals and REEs exploiting acidophilic bacteria (*Acidithiobacillus thiooxidans),* whereas the second stage was performed for precious metal mobilization by cyanogenic bacteria (*Pseudomonasputida* WSC361). Moreover, wider technical considerations on the development of biohydrometallurgical treatments and on the management of WEEE shredding dust in a circular economy view have been presented.

2. Materials and methods

2.1. Material characterization

A full scale plant located in Southern Italy and providing the mechanical treatment of small electronic equipment, information technology (IT) and consumer appliances was chosen for this study (Cesaro et al., 2017). Over the treatment line, representative samples of dust materials resulting from the shredding of WEEE, namely WEEE shredding dusts, were collected.

The carbon content of the sampled dust was determined by means of an elemental analyser (OEA Flash 2000; Thermo Finningan). For metal analysis, samples were digested using the standard aqua regia extraction procedure (ISO 11466:1995). Thereafter, the concentrations of dissolved base metals (aluminium, cadmium, copper, iron, nickel, lead and zinc), precious metals (gold, silver, palladium, platinum) and REEs (cerium, europium, lanthanum, neodymium and yttrium) were analysed by means of inductively coupled plasma-optical emission spectroscopy (ICP-OES, Thermo iCap 6000 series).

The particle size distribution was obtained by a sieve analysis, considering the following size fractions: <0.5 mm, 0.5–1 mm, 1–2 mm, >2 mm. Mineral phase analysis was performed using a X-ray powder diffractometer (XRD, Bruker D8 advance). The following conditions were applied: Cu Kα radiation, 35 keV accelerating voltage, 40 mA current, 10–80° scanning range and 0.5 s/step (0.0296°/step) scan speed.

2.2. Microorganisms and growth conditions

Bioleaching was performed using the acidophilic and cyanogenic bacterial strains as described by Işıldar et al. (2016). *A. thiooxidans* (DSM 9463) was purchased from the Leibniz Institute (DSMZ), Braunschweig (Germany), whereas *P. putida* WSC361 was kindly provided by Peter Bakker from Utrecht University (the Netherlands).

A. thiooxidans was grown in a mineral medium containing (g/L): $(NH_4)_2SO_4$ (2.0), $MgSO_4 \cdot 7H_2O$ (0.25), K_2HPO_4 (0.1), KCl (0.1) and S^0(5.0). The pH was adjusted to 3.5 using H_2SO_4. Cultures were inoculated with 10% (*v/v*) inoculum size in 100 mL growth medium and incubated at 30 °C and 150 rpm for 10 days prior to the bioleaching experiments. The pH and oxidation reduction potential (ORP) were periodically recorded by Ag/AgCl reference electrodes (SenTix 21, WTW, Germany and QR481X, Qis, the Netherlands) for monitoring indirectly the bacterial growth.

A. thiooxidans cells were enumerated using the spread plate method (Starosvetsky et al., 2013). *Thiobacillus* agar, containing 0.4 g $(NH_4)_2SO_4$, 0.5 g $MgSO_4 \cdot 7H_2O$, 0.25 g $CaCl_2$, 4 g KH_2PO_4, 0.01 g $FeSO_4$, 5 g $Na_2S_2O_3$, 12.5 g agar in 1 L distilled water, was used. Samples were serially diluted up to 10^{-7} using a saline solution (0.85% NaCl) and plated on agar. The Petri dishes were then incubated for 7–10 days at 31 (±0.5) °C.

P. putida was grown in nutrient broth containing (g/L): meat extract (1.0), yeast extract (2.0), peptone (2.0) and NaCl (5.0). The pH was adjusted to 7.3 using NaOH. The cultures were inoculated with 1% (*v/v*) in 150 mL growth medium supplemented with 10 g/L of glycine (Işıldar et al., 2016) and incubated at 30 °C and 150 rpm for 18 h prior to the bioleaching experiments. The bacterial growth was monitored by the measurement of both pH and optical density (OD) at 600 nm using a Perkin Elmer Lambda 20 Spectrophotometer.

The ability of *P. putida* to produce CN^- was checked by a modified colorimetric method (Işıldar et al., 2016, Ruan et al., 2014). *P. putida* was plated on nutrient agar supplemented with glycine. A sterile filter paper soaked in a solution of 0.5% picric acid and 2% sodium carbonate was fixed to the inner side of the Petri dish lid. The dishes were covered with paraffin film and incubated at 30 °C (Işıldar et al., 2016). CN^- production induces a colour reaction on the filter paper which turns from yellow to red, proportionally to the CN^-concentration (Ruan et al., 2014).

2.3. Bioleaching experiments

As WEEE can inhibit bacterial growth due to the presence of hazardous components (Liang et al., 2010), a pre-growth strategy was followed to reduce the toxic effects of the source material: microorganisms were grown in the absence of dust (Brandl et al., 2001, Işıldar et al., 2016). The latter was subsequently added to the inoculated medium when the optimum bioleaching conditions were reached.

2.3.1. Bioleaching by *A. thiooxidans*

Bioleaching experiments involving acidophilic bacteria were carried out with three different concentrations of WEEE shredding dust (0.5%, 1% and 2% *w/v*), previously dried at 80 °C overnight (Brandl et al., 2001), to 100 mL of bioleaching medium containing active growing cultures of *A. thiooxidans* ($2.1 \pm 0.3 \cdot 10^7$ CFU/mL). The flasks were agitated on an orbital shaker (Brunswick Innova 2000; USA), set to 150 rpm, in a temperature controlled room at 30 °C for 8 days. During the bioleaching tests, samples were taken at different time intervals (2, 4, 6 and 8 days) and analysed for their metal content. The bioleaching process was monitored by pH and ORP, denoting indirectly the status of the bacterial growth (Liang et al., 2010). Each test was performed in duplicate and control experiments, namely negative controls that contained non-inoculated bioleaching medium, were also tested.

2.3.2. Bioleaching by *P. putida*

The solid residue from the first bioleaching step was collected, washed twice with distilled water, dried and used for the second bioleaching step involving the CN^- producing bacteria. 1% (*w/v*) of dust was added to an active *Pseudomonas* culture ($OD_{600} = 0.9$) and the bioleaching experiments were carried out at 30 °C and 150 rpm for 30 h. Samples were taken at different intervals (3, 8, 20, 24 and 30 h). pH was measured to indirectly monitor the bioleaching process. All the experiments were performed in duplicate and control experiments (negative controls) were run as well.

The results of the bioleaching tests carried out with the cyanogenic bacteria were compared with those obtained from chemical leaching assays using a KCN solution at 5, 10, 25 mg/L CN^- and a 1% (*w/v*) pulp density. The operating conditions of temperature, agitation rate, and leaching time were set to ambient temperature (21 ± 1 °C), 150 rpm, and 30 h, respectively. Prior to the CN^- chemical leaching, a H_2SO_4leaching step was carried out for removing the base metals as described by Birloaga et al. (2013).

2.3.3. Sample analysis and leaching ability determination

The leaching ability of base metals, precious metals and REEs was evaluated in each leaching step by means of a metal analysis using inductively coupled plasma optical emission spectrometry (ICP-OES, Thermo iCap 6000 series). Samples collected from the biological leaching experiments were centrifuged at 12,000×g for 10 min, filtered using 0.45 µm cellulose membrane filters (Whatman), acidified and properly diluted prior to the ICP-OES analysis. Samples collected during the chemical CN^- leaching step were filtered via a 0.45 µm cellulose membrane (Whatman), acidified and diluted for the ICP-OES analysis. Concentrated HNO_3 at 2% was used for base metal and REE analysis, whereas 5% HCl was used for precious metal analysis.

The leaching ability (L_i) of the metal *i* was calculated by considering the mass of the sample used for the experiment (M), the volume of the leaching solution (V) and the concentration of the selected metal *i* (C_i) detected in the leachate, as given in Equation (1) (Savvilotidou et al., 2015):

$$L_i = \frac{C_i \cdot V}{M} \qquad (1)$$

where:

L_i = leaching ability for metal *i* (mg/g)
C_i = concentration of metal *i* in the leachate (mg/L)
M = mass of the dust sample (g)
V = volume of the leaching solution (mL)

The extraction or recovery efficiency (R_i) for the metal of interest was thus obtained referring the leaching ability of the selected metal to its initial concentration, as reported in Equation (2) (Chen et al., 2015):

$$R_i = \frac{L_i}{C_{in,i}} \quad (2)$$

where:

R_i = recovery efficiency of metal *i* (%)

L_i = leaching ability for metal *i* (mg/g)
$C_{in,i}$ = concentration of metal *i* in the source dust (mg/kg)

3. Results

3.1. WEEE shredding dust characterization

The average composition of the WEEE shredding dust in terms of its carbon and metal content is given in Table 1. Besides 18% wt. of organic carbon, significant amounts of base metals were also present in the dust samples. Approximately 30 g of aluminium, 30 g of copper and 20 g of lead were present in 1 kg of dust sample. Iron was present in relatively high concentrations (16 g per kg of dust). Trace concentrations of gold and palladium were found, together with 270 mg of silver per kg of dust. Several mg of REEs per kg of dust were also recorded.

Table 1. Average composition of dust samples.

Element	Unit	Concentration
Ag	mg/kg	271 ± 85
Al	mg/kg	31797 ± 1476
Au	mg/kg	8.7 ± 0.6
Cd	mg/kg	236 ± 36
Ce	mg/kg	72 ± 1.1
Cu	mg/kg	33501 ± 4063
Eu	mg/kg	2 ± 0.2
Fe	mg/kg	16003 ± 3890
La	mg/kg	87 ± 11.6
Nd	mg/kg	88 ± 1.9
Ni	mg/kg	1294 ± 154
Pb	mg/kg	22100 ± 2689
Pd	mg/kg	23 ± 5.6
Pt	mg/kg	<1.3
Y	mg/kg	42 ± 8.0
Zn	mg/kg	8202 ± 597
Total organic carbon (TOC)	%wt.	18.7 ± 0.9

The particle size distribution of the dust is reported in Table 2. Particles with a diameter smaller than 0.5 mm made up around 90% by weight of the dust samples. The remaining portion, representing 10% of the total weight, consisted of fluffy material and aluminium foils. For a more detailed characterization, a mineral phase analysis of the dust was performed. Several peaks were recorded in the XRD pattern (Fig. 1) indicating the presence of different crystalline phase substances in the matrix. The peaks marked in Fig. 1 with the same letters were assigned to substances with a similar crystal form. Silicon dioxide, metallic copper, metallic aluminium, metallic iron as well as metallic lead were the main mineral phases detected. REE-bearing compounds were also present. While the pattern suggested the presence of base metals in their elemental form, REEs were found to be mainly present as oxides.

Table 2. Particle size distribution of the sampled dust.

Fraction (mm)	% *w/w*	Description
<0.5	92.6	Homogeneous dust
0.5–1	2.2	Fluffy material
1–2	0.6	Fluffy material
>2	4.6	Al-coated capacitor foils

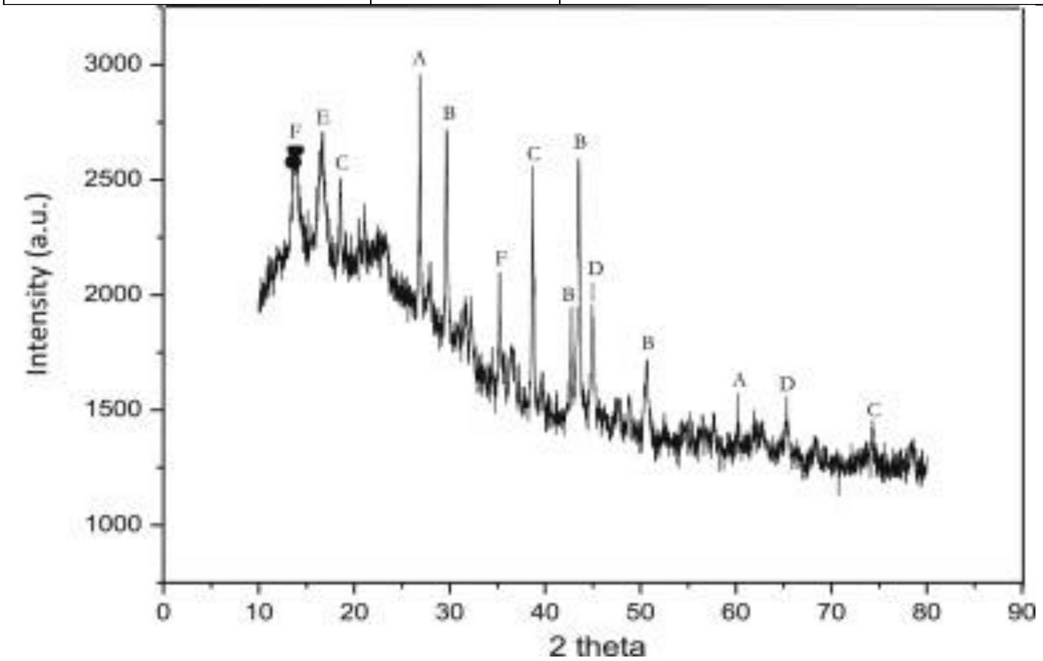


Fig. 1. XRD pattern of WEEE shredding dust: SiO_2 (A), metallic copper (B), metallic aluminium (C), metallic iron (D), metallic lead (E) and Ce-La-Nd-Y-bearing minerals (F).

3.2. Bioleaching using acidophiles

Prior to the bioleaching experiments, *A. thiooxidans* was grown in the absence of dust for 10 days in order to reach the optimum conditions for bioleaching, thereby avoiding the inhibition of bacterial growth due to the toxic nature of WEEE (Brandl et al., 2001, Işıldar et al., 2016). Fig. 2 shows the pH and ORP profiles during the bioleaching experiments using *A. thiooxidans* at different dust concentrations.

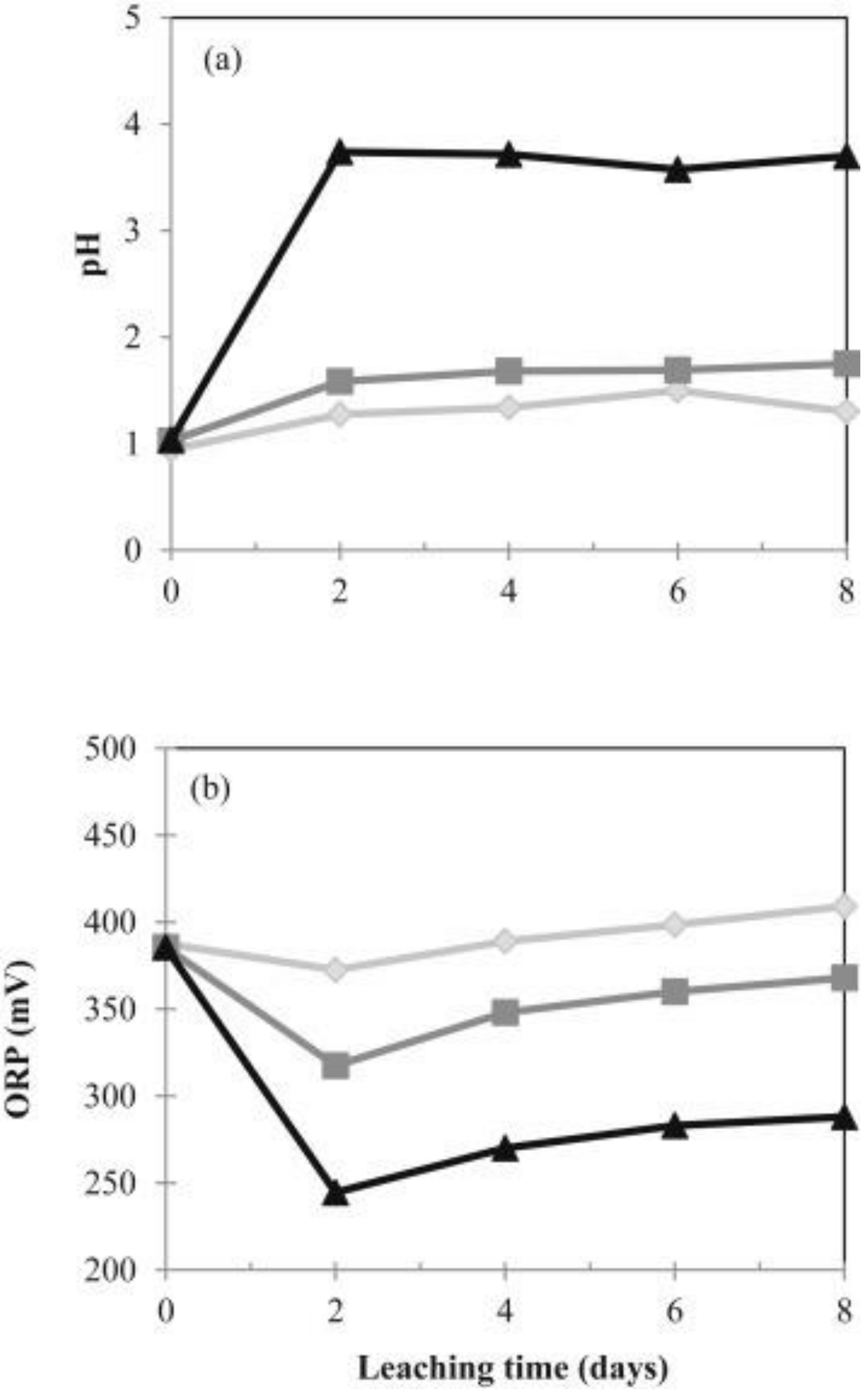


Fig. 2. Evolution of pH (a) and ORP (b) during *A. thiooxidans* bioleaching of WEEE shredding dust at different pulp densities: 0.5% (*w/v*) ◆, 1% (*w/v*) ■, 2% (*w/v*) ▲.

The bioleaching experiments began when the pH of the inoculum dropped to 1.0. The addition of WEEE shredding dust (t = 0) caused an increase in pH, because of the alkaline nature of the waste material (Brandl et al., 2001). A higher pH increase was recorded for the higher material load (2% pulp density). After a leaching period of 2 days, a steady pH profile was observed. Conversely, the addition of dust lowered the ORP of the solution. This reduction was more evident for the larger solid to liquid ratios (S/L) applied. After 2 days, the ORP profile increased again.

Different leaching efficiencies were observed among the investigated metals after 8 days of bioleaching (Fig. 3). Cadmium, nickel and zinc were completely extracted both at lower and higher pulp densities, whereas for aluminium, iron and copper the best leaching efficiencies were achieved at 0.5% pulp density. No lead was detected in the leaching solution. The bioleaching profiles of aluminium, copper and iron at the different pulp densities investigated are reported in Fig. 4. In control flasks containing non-inoculated samples, only low concentrations of copper were detected in the leaching medium, corresponding to a low copper mobilization (229–334 mg/kg) upon exposure to H_2SO_4 for pH adjustment, as also observed in other studies (Brandl et al., 2001, Işıldar et al., 2016).

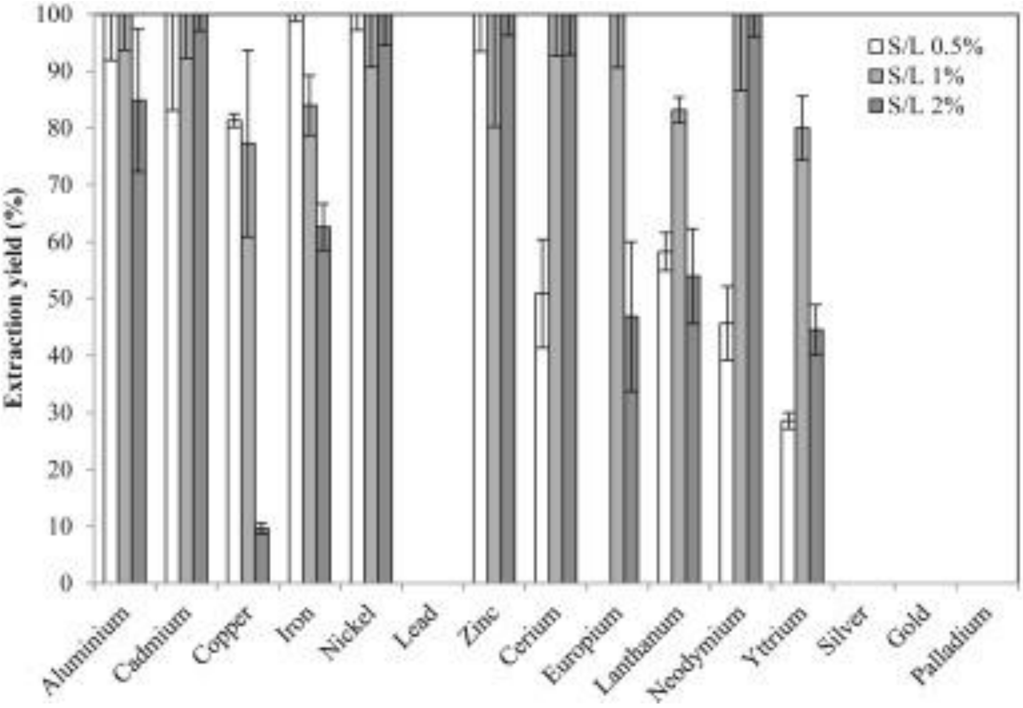


Fig. 3. Metal mobilization from WEEE shredding dust after 8 days of bioleaching using *A. thiooxidans* at different pulp densities.

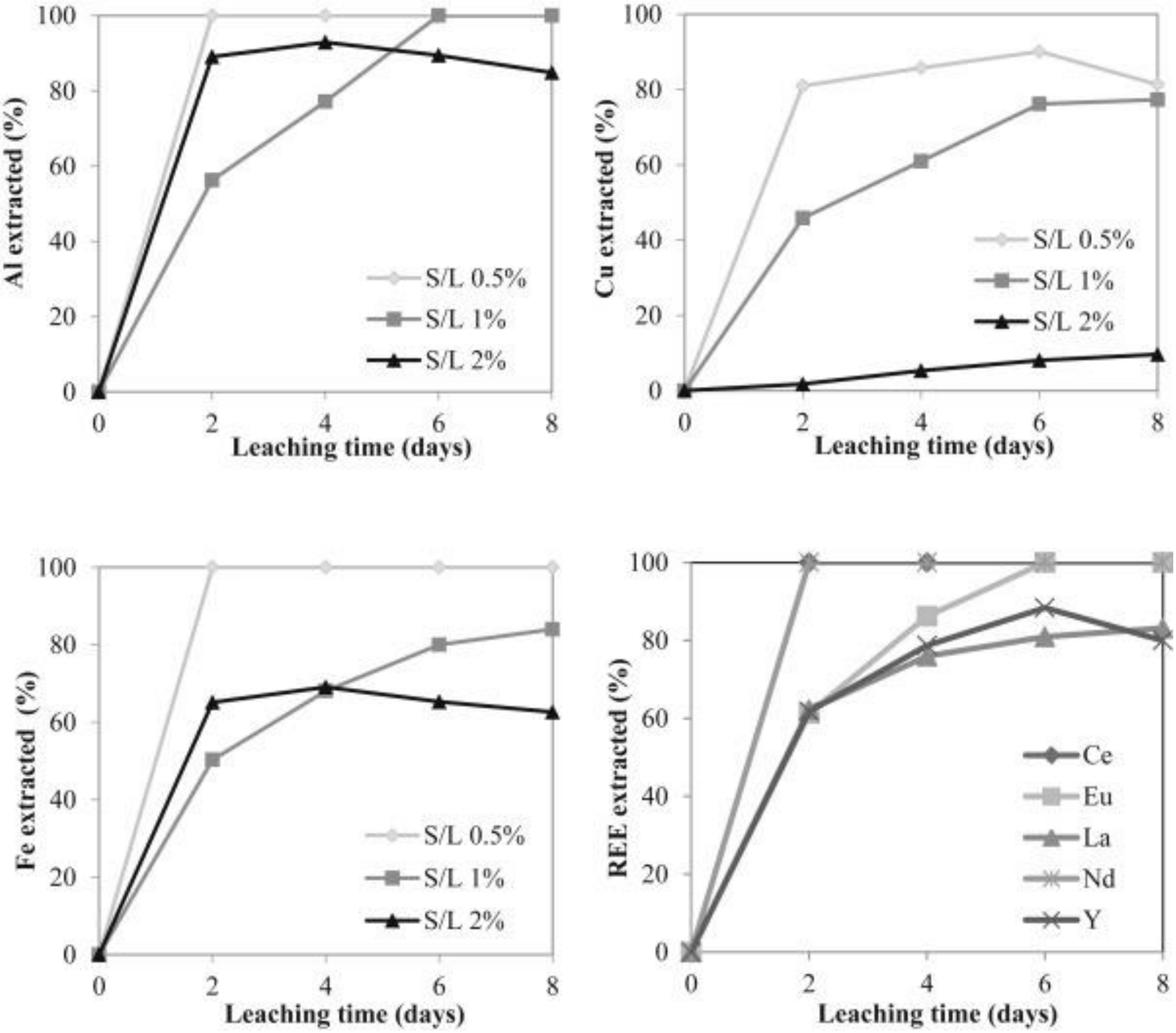


Fig. 4. Bioleaching profile of selected metals from WEEE shredding dust by *A. thiooxidans* as a function of time.

REEs were leached from the dust at the best efficiencies in the bioleaching experiments with 1% pulp density. High extraction yields (>99%) were recorded for cerium, europium and neodymium. Relative lower efficiencies were obtained for lanthanum and yttrium: approximately 80% of these elements contained in the source material were extracted. Fig. 4 shows the bioleaching profile of REEs over time at 1% pulp density. After 2 days, cerium and neodymium were completely extracted, whereas the remaining rare earths were leached at higher efficiencies only after 8 days of incubation. REEs were not found in the control samples, confirming that the metal mobilization was a result of the pH decrease induced by the microbial activity. Precious metals were not detected in the *A. thiooxidans* leaching solutions (Fig. 3).

3.3. Bioleaching using cyanogenic bacteria

The dust collected from the first bioleaching step was subjected to a second step involving *P. putida* WSC 361. Prior to the bioleaching experiments, *P. putida* WSC 361 was grown in the absence of dust, following a similar pre-growth strategy applied for the first stage using *A. thiooxidans*. As cyanogenic bacteria produce CN^- in the late exponential or early stationary phase (16–20 h incubation) (Işıldar et al., 2016, Natarajan and Ting, 2015), the dust was added to the inoculum 18 h after inoculation when maximum CN^- production was expected. As reported by Işıldar et al. (2016), the CN^- production is approximately 20 mg/L at this incubation time. Indeed, the cyanogenic production by *P. putida* WSC 361 was confirmed by the colour reaction of the filter papers soaked in a picric acid solution, which turned from yellow to an intense red colour after 18 h incubation (Fig. 5).

Fig. 5. Colour reaction confirming the CN^- production by *P. putida* WSC 361. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

During the bioleaching experiments, only gold was detected among the precious metals, while the process showed no release of silver or palladium from the *A. thiooxidans* pre-treated dust. The maximum gold extraction (48 ± 2%) was achieved in 3 h, thereafter the concentration of gold in solution decreased (Fig. 6a). Furthermore, around 15% of the residual copper from the first leaching step was detected in the *P. putida* WSC 361 leaching solution (data not shown), whereas REEs were not found in the bioleaching solution. No metals were detected in the control experiments, confirming that the biological mobilization of gold was solely due to the activity of the cyanogenic *Pseudomonas* sp.

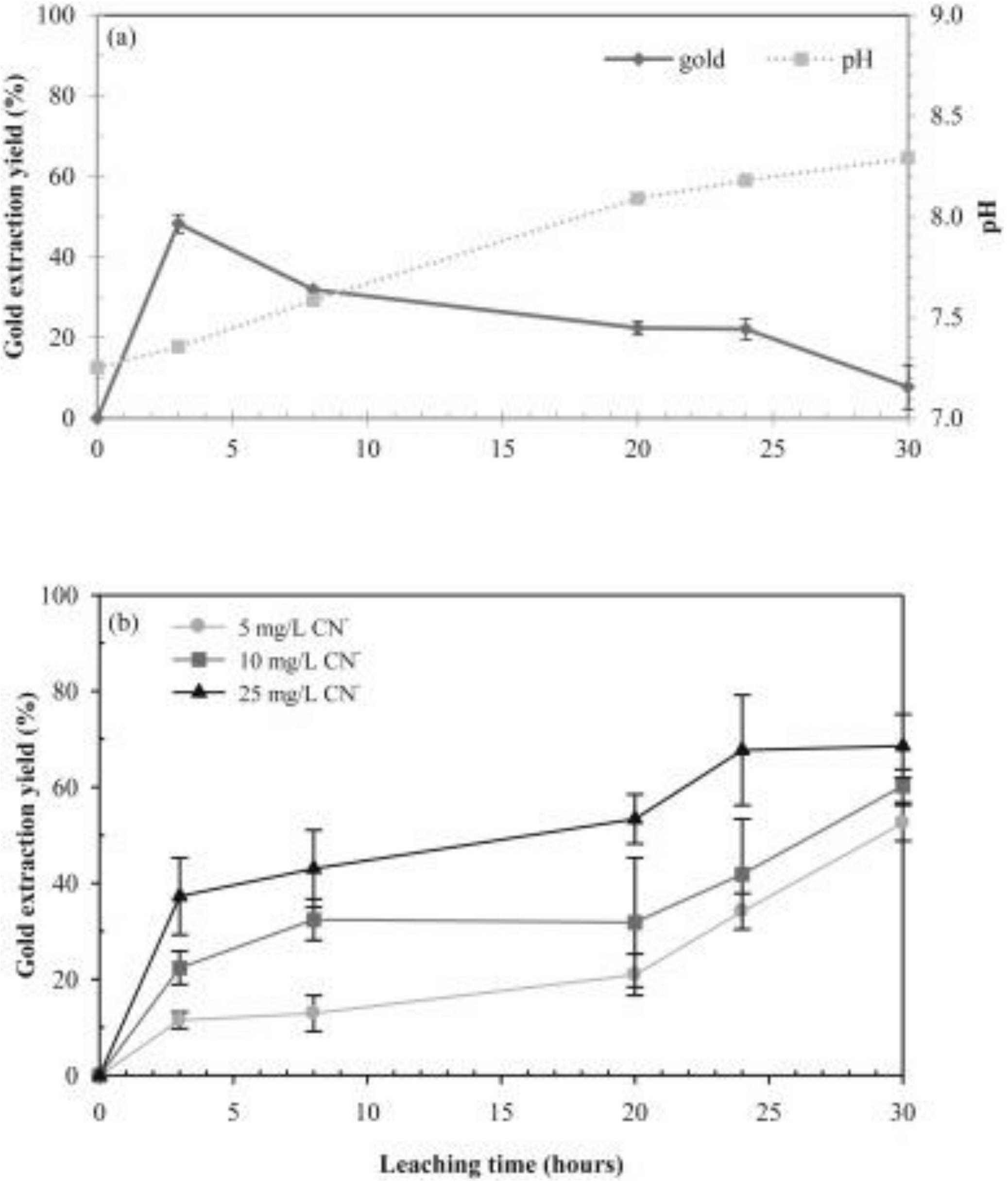


Fig. 6. Extraction yields of gold over time during *P. putida* WSC 361 bioleaching (a) at 1% pulp density and chemical CN^- leaching (b) at different CN^- concentrations.

A chemical leaching experiment using KCN was performed to compare the results obtained from bioleaching experiments. Fig. 6b shows the gold mobilization at different CN^- concentrations. During the leaching experiments, the pH of the solution was around 12.0. The gold extraction via chemical leaching showed an increasing profile over time. Higher CN^- concentrations resulted in higher gold mobilization: at 25 mg/L of CN^- approximately 70% of gold was leached from the WEEE shredder dust after 30 h. Moreover, the chemical process was found to be effective also for the mobilization of silver. More than 95% of the silver contained in the dust material was extracted, even at a CN^- concentration of 5 mg/L. Conversely, no palladium was detected in the chemical CN^- leaching solution.

4. Discussion

4.1. WEEE shredding dust characterization

Due to the action of the shredding process, considerable amounts of metals were removed along with the powdery particles collected from the processing line under investigation (Table 1). Copper, aluminium and iron were the metals present at higher concentrations in WEEE shredding dust, as they are the prevalent materials contained in WEEE (Widmer et al., 2005). Significant amounts of lead and relatively high concentrations of precious metals and REEs were present in the dust samples as well. The occurrence of lead raises concern in view of environmental pollution but, on the other hand, the presence of both valuable and critical metals in this fraction attracts great interest as it can represent a potential secondary source of these materials (Wang et al., 2015). However, the simultaneous presence of such a wide range of multi-metals poses challenges for their selective extraction, which is highly influenced by the chemical speciation of the metals present in the solid matrix (Tunsu et al., 2015).

As highlighted by the particle size characterization (Table 2), the dust fraction was primarily composed of tiny particles. A small portion was made of small pieces of plastic and foils coated with aluminium, which was the main metal found in this matrix as demonstrated from the metal characterization results (Table 1). The obtained dust size classification was roughly consistent with the characterization of fine-grained electronic scrap reported by Brandl et al. (2001).

The XRD analysis reported in Fig. 1 confirmed the presence of several metallic substances, accounting for the complex composition of the dust matrix. Moreover, the background noise pointed out the existence of amorphous materials related to plastic matter, as confirmed by the organic content detected in the dust samples. Due to the presence of this organic fraction as well as the relative high iron concentrations, the analysis of the XRD pattern is not easy as these conditions widen the background profile of the diffraction (Wang et al., 2015). The mineral phase analysis confirmed the presence of base metals in their elemental form, while the occurrence of REEs in their oxide form suggested the potential extraction of these critical materials using mild acid solutions (Han et al., 2014).

4.2. Extraction of base metals and rare earth elements by acidophilic leaching

Due to their ability to leach metals from sulfidic ores (Brandl, 2001), *Acidithiobacilli*, including both iron and sulphur oxidizers as *A. ferroxidans* and *A. thiooxidans*, respectively, have been recently considered for the extraction of metals from WEEE (Ilyas and Lee, 2014). Compared to *A. ferroxidans*, the research on *A. thiooxidans* is less widespread (Travisany et al., 2014). In this study, *A. thiooxidans* was chosen for its well-known capacity in mobilizing base metals (Hong and Valix, 2014, Kumar and Nagendran, 2007, Travisany et al., 2014) and for testing this strain with regard to the extraction of REEs. As WEEE is a non-sulphidic material, the bioleaching process is recognized to be mainly dominated by an indirect leaching mechanism involving the biogenic formation of a leaching agent (Hong and Valix, 2014, Zhu et al., 2011). In this case, the role of *A. thiooxidans* during the bioleaching process is to catalyse the oxidation of elemental sulphur(S^0), externally added as it is not present in WEEE, to H_2SO_4. The biogenic H_2SO_4 is, in turn, responsible for the solubilisation of the metals contained in WEEE as a result of the following equations (Bosecker, 1997):(3)(4)where M stands for the metal. During the bioleaching process, the H^+ ions produced via the biogenic oxidation of S° in H_2SO_4 were balanced by the H^+ ions consumed through the oxidation of metals as shown by the steady profile of the pH after 2 days incubation (Fig. 2). The consequent increasing profile of the redox potential confirmed the H_2SO_4 formation (Liang et al., 2010).

Pulp density plays a crucial role in the bioleaching process. Metals were extracted with more efficient yields at pulp densities lower than 2% (*w/v*) (Fig. 3). The bioleaching performance was inhibited at higher pulp densities, most likely due to either the toxic effects of both metallic and non-metallic compounds contained in WEEE on the acidophiles (Ilyas et al., 2007) or oxygen mass-transfer limitations (Zhu et al., 2011). The use of higher dust loads could be pursued as well, but a prolonged time for bacterial adaptation might be required (Brandl et al., 2001). However, the extraction of metals was significantly affected by the solid to liquid ratio at a pulp density of 2% (*w/v*). Comparison of the extraction efficiencies obtained at 0.5% (*w/v*) and 1% (*w/v*) pulp density shows that only a relative slight decrease in terms of iron and copper recovery was obtained after 8 days incubation for base metal bioleaching at a pulp density of 1% (*w/v*) (Fig. 3). For REE extraction, although the highest leaching is expected at the lowest pulp density, 1% (*w/v*) gave the best condition (Fig. 3), probably due to the low concentrations of REEs present in the shredding dust. However, as the pulp density compromises the industrial application of this process, 1% (*w/v*) can therefore be considered the optimum operating condition for metal extraction as also reported in other studies using PCBs as source material (Table 3).

Table 3. Process parameters and extraction efficiencies reported by previous bioleaching studies for base metals and rare earth mobilization.

Source matrix	**Microorganisms**	**Process parameters**	**Extraction efficiencies**	**References**
Dust from WEEE shredding processes	*A. ferrooxidans* + *A. thiooxidans*	pH: 2.1–0.9	Al: 90%	Brandl et al. (2001)
		T: 30 °C	Cu: 90%	
		S/L: 0.5–1% *w/v*	Ni: 90%	
		Stirring rate: 150 rpm	Zn: 90%	
		Leaching time: 10 d		
PCBs	*S. thermosulfidooxidans* + acidophilic isolate	pH: 2.0	Al: 79%	Ilyas et al. (2007)
		T: 45 °C	Cu: 89%	
		S/L: 1% *w/v*	Ni: 81%	
		Stirring rate: 180 rpm	Zn: 83%	
		Leaching time: 18 d		
PCBs	*A. ferrooxidans*	pH: 2.0	Cu: 100%	Yang et al. (2009)
		T: 30 °C		
		S/L: 2.5% *w/v*		
		Stirring rate: 165 rpm		
		Leaching time: 4 d		
PCBs	*A. ferrooxidans* + *A. thiooxidans*	pH: 1.5	Cu: 90%	Liang et al. (2010)
		T: 32 °C		
		S/L: 3% *w/v*		
		Stirring rate: 180 rpm		
		Leaching time: 10 d		
PCBs	Mixed Acidophilic bacteria	pH: 2.0	Al: 88.2%	Zhu et al. (2011)
		T: 30 °C	Cu: 96.8%	

Source matrix	**Microorganisms**	**Process parameters**	**Extraction efficiencies**	**References**
		S/L: 1.2% *w/v*	Zn: 91.6%	
		Stirring rate: 160 rpm		
		Leaching time: 1.8 d		
TV PCBs	*A. ferrooxidans* + *L. ferrooxidans* + *A. thiooxidans*	pH: 1.7	Cu: 89%	Bas et al. (2013)
		T: 35 °C		
		S/L: 1% *w/v*		
		Stirring rate: 175 rpm		
		Leaching time: 4.8 d		
PC PCBs	*A. ferrooxidans*	pH: 3.0	Cu: 100%	Arshadi and Mousavi (2014)
		T: 30 °C	Ni: 100%	
		S/L: 2% *w/v*		
		Stirring rate: 170 rpm		
		Leaching time: 0.8 d		
PCBs	*A. thiooxidans*	pH: 1.0	Cu: 60%	Hong and Valix (2014)
		T: 30 °C		
		S/L: 1% *w/v*		
		Leaching time: 5 d		
PC PCBs	*A. ferrooxidans*	pH: 2.2	Al: 75.4%	Yang et al. (2014)
		T: 30 °C	Cu: 96.8%	
		S/L: 1.5% *w/v*	Zn: 83.8%	
		Stirring rate: 170 rpm		
		Leaching time: 3 d		
PCBs	*A. ferrooxidans* + *A. thiooxidans*	pH: 1.5–2.2	Al: 20%	

Source matrix	**Microorganisms**	**Process parameters**	**Extraction efficiencies**	**References**
		S/L: 1% *w/v* Leaching time: 28 d	Cu: 100% Ni: 92% Zn: 89%	Mrazikova et al. (2015)
PCBs	*A. ferrooxidans* + *A. thiooxidans*	pH: 1.0–1.6 T: ambient S/L: 1% *w/v* Stirring rate: 150 rpm Leaching time: 7 d	Cu: 98%	Işıldar et al. (2016)
PCBs	Acidophilic bacteria from acid mine drainage	pH: 1.8 T: 30 °C S/L: 3.5% *w/v* Stirring rate: 170 rpm Leaching time: 15 d	Cu: 92%	Utimura et al. (2017)
CRT fluorescent powder	*A. ferrooxidans* + *A. thiooxidans* + *L. ferrooxidans*	T: 30 °C S/L: 10% *w/v* Stirring rate: 175 rpm Leaching time: 16 d	Y: 70%	Beolchini et al. (2012)
Red mud	*Penicillium tricolor*	pH: 2.0–2.5 T: 30 °C S/L: 2% *w/v*	La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Y, Sc: 36–78%	Qu and Lian (2013)
Coal ashes	Acidophilic chemolithotrophs	pH: 0.9–2.0 T: 45 °C	La: 59.5% Sc: 52%	

Source matrix	Microorganisms	Process parameters	Extraction efficiencies	References
		S/L: 10% *w/v*	Y: 52.6%	Muravyov et al. (2015)
		Stirring rate: 500 rpm		
		Leaching time: 10 d		
CRT fluorescent powder	*Gluconobacter oxydans*	T: 30 °C	Total REEs: 2%	Reed et al. (2016)
		S/L: 1.5% *w/v*		
		Stirring rate: 150 rpm		
		Leaching time: 1 d		

The selective mobilization of different metals strongly depends on their electrochemical interactions as the metal dissolution is an electrochemical process based on oxidation-reduction reactions. Therefore, metals with a lower standard electrode potential, such as aluminium and zinc, would be oxidized and preferentially dissolved compared to copper (Hong and Valix, 2014) as observed in the present study (Fig. 3). Moreover, metals such as aluminium and iron were already completely extracted after only 2 day at 0.5% (*w/v*), whereas copper required a prolonged time (Fig. 4). A similar trend in the extraction of copper was reported by Utimura et al. (2017) who performed bioleaching experiments with a mixed culture of acidophilic bacteria from an acid mine. The dissolution of metals is, furthermore, influenced by their solubility: this is the case of lead, which was not detected in the leaching solution as its sulphate salts are relatively insoluble and its precipitation as $PbSO_4$ is thus conceivable (Brandl et al., 2001, Ilyas et al., 2007). Similarly, precious metals were not solubilized by *A. thiooxidans*. Indeed, acidophiles are widely used in the bio-oxidation processes for gold mining operations as they are able to dissolve copper, but leave the valuable metals in the solid matrix for their subsequent recovery using different processes (Brandl, 2001, Natarajan and Ting, 2014, Pham and Ting, 2009).

In this study, *A. thiooxidans* was found to be able to effectively leach the REEs. Only limited information is currently available in the literature concerning REE-microbe interactions (Barmettler et al., 2016, Beolchini et al., 2012) and limited studies have been done on the bioleaching of REEs from secondary sources (Table 3). However, the general mechanisms of acidolysis, redoxolysis and complexolysis can be assumed to be also involved in the REE bioleaching process (Barmettler et al., 2016).

The extraction efficiency shown for yttrium in this study was in good agreement with the investigation of Beolchini et al. (2012), who reported an yttrium mobilization of 70% from cathode ray tubefluorescent powders using a mixed culture of Fe/S oxidising bacteria such as *Acidithiobacillus ferrooxidans*, *Acidithiobacillus thiooxidans* and *Leptospirillum ferrooxidans*. The good REE leaching efficiencies obtained can be related to both the good solubility of REEs in H_2SO_4 media (Han et al., 2014) as well as their oxide form, confirmed by the mineral phase analysis (Fig. 1), which promotes their potential extraction (Jha et al., 2016, Tunsu et al., 2015).

4.3. Gold mobilization from WEEE shredding dust by cyanogenic bacteria

Although *Chromobacterium violaceum* is the most effective cyanogenic bacterium for gold bioleaching (Faramarzi et al., 2004, Natarajan and Ting, 2015, Pham and Ting, 2009, Pradhan and Kumar, 2012), some *Pseudomonas* species have been reported to produce biogenic cyanides, via the oxidative decarboxylation of glycine (Liang et al., 2014), which act in the leaching gold (Ilyas and Lee, 2014) as shown in Equation (5)(Priya and Hait, 2017):(5)

Pseudomonas strains can be easily employed in industry as they are commonly found in soil and water and have a strong adaptive ability (Ruan et al., 2014). Their use as single culture or in combination with *C. violaceum* for gold bioleaching from WEEE is well documented (Liu et al., 2016).

The ability of *P. putida* WSC361 in gold extraction from PCBs has been recently demonstrated (Işıldar et al., 2016). The extraction efficiency obtained in this study as a result of the biologically produced cyanides by *P. putida* WSC361 (Fig. 6a) is roughly comparable with the efficiency reported for the chemical process (Fig. 6b). Although a prolonged time revealed a more efficient extraction during the chemical process, the biological process is, however, desirable since bacteria decompose CN^- ions at a later stage making the process more environmental friendly.

As shown in Fig. 6a, the cyanogenic bioleaching process occurred at a pH of ∼7.0–8.0. These values are favourable conditions for the physiological growth of cyanogenic bacteria (Pham and Ting, 2009). However, during the bioleaching experiments the maximum pH was around 8.4 (Fig. 6a). As the CN^- concentration in solution is highly affected by pH, a partial loss of cyanides via volatilization could have occurred. Cyanides are completely present in solution as free ions only at pH 11.0, while at a pH less than 9.4, the amount of CN^- in the form of hydrocyanic acid, highly volatile, is predominant (Barnes et al., 2000).

The bioleaching profile showed a maximum gold extraction yield at 3 h (Fig. 6a), while with a prolonged leaching time the concentration of gold in solution decreased. This trend can be ascribed to either sorption onto biomass or to the biodegradation of the cyanide complexes by bacteria that use them as carbon or nitrogen source(Brandl et al., 2008). Although a small portion of REEs remained in the solid residue after the first leaching step, no REEs were detected in the leachate from the second (cyanogenic) step. This confirms that these transition elements cannot form stable complexes with cyanides (Barnes et al., 2000, Faramarzi et al., 2004), and CN^- leaching can thus not be considered for REE dissolution from WEEE.

As 15% of the residual copper was also detected during the experiment in the CN^- bioleaching solution, the extraction of gold as well as the mobilization of the other precious metals, such as silver and palladium, was likely limited by the reactivity of cyanide ions with copper. This confirms the necessity to remove copper prior to the cyanidation process (Birloaga et al., 2013, Ghosh et al., 2015, Zhang et al., 2012).

4.4. Technical implications

This study showed the feasibility of recovering valuable and critical metals from WEEE shredding dust through environmental friendly processes in the field of biohydrometallurgy. Bioleaching could offer several technical, economic and environmental advantages. As the agents responsible for the leaching action are produced biologically, the external supply of these reagents is not necessary (Beolchini et al., 2012) and the use of cheaper and recoverable sulphur forms as energy source can reduce the process costs (Pathak et al., 2009). The equipment maintenance is not as frequent as required for hydrometallurgical processes due to the low amount of corrosive acid produced. Moreover, the biological degradation of cyanide when cyanogenic bacteria are employed contributes to the reduction of the environmental impacts of the treatment. These aspects entail the promising application of biotechnology in the WEEE recycling system which is still characterized by weak performances in terms of environmental protection due a lack of environmental-sound technologies and facilities (Li et al., 2015). A cost-benefit analysis and a process scale-up evaluation of this novel technology need to be addressed in future studies. In this regard, life cycle assessment (LCA) is a useful tool whose application to the bioleaching process at an early stage (as the laboratory one) can guide further technological progresses providing a certain leeway for adjusting design developments (Villares et al., 2016).

Some aspects still hinder the implementation of biohydrometallurgy at the industrial scale. The literature studies on WEEE bioleaching are mainly limited to bench scale applications with only few attempts on column bioreactors at the laboratory scale (Chen et al., 2015, Ilyas et al., 2010, Lee and Pandey, 2012). The development of bioleaching processes is challenged by the complex and heterogeneous nature of WEEE, as this waste stream has different physical and chemical characteristics from other solid wastes. Moreover, due to the complexity of WEEE, the recovery of rare and precious metals is not achievable using a single technique but a combination of different processes are required (Wang et al., 2017). In this respect, bioleaching can be used as the downstream stage of an integrated treatment in which the upstream step is covered by a mechanical process (Awasthi et al., 2016). Moreover, integration and use of pre-treatments as mechanochemical technologies can enhance the leaching mechanism of metals from WEEE (Tan and Li, 2015).

The prolonged leaching time and the lower loading capacity restricted by the toxic nature of WEEE are the main limitations of the novel biohydrometallurgical process studied. The low treatment capacity is, nevertheless, not an obstacle for the application of bioleaching to WEEE shredding dust since this fraction is produced at a low mass percentage (Wang et al., 2015). Indeed, bioleaching processes are regarded as a promising technique for treating low grade materials at low costs (Beolchini et al., 2012). Moreover, the opportunity to treat WEEE shredding dust trough bioleaching is particularly relevant considering that this waste stream still represents a cost for the treatment plant, as it is currently sent to landfills for disposal, and that its composition includes valuable and critical materials (Bachér and Kaartinen, 2016, Wang et al., 2015). The presence of precious metals and REEs in WEEE dust generated during the shredding action of mechanical units makes this fraction particularly worthy for recovery as these elements or materials might be introduced back to the market following a management strategy based on a circular economyapproach. Furthermore, the treatment of WEEE shredding dust could contribute to the reduction of the toxic nature of this waste stream as the relevant portions of heavy metals that end up in the dust can be removed in the leaching processes, thus improving the quality of this matrix for its sustainable disposal or reuse.

5. Conclusions

This study investigated the potential application of bioleaching for the extraction of base metals, precious metals and REEs from WEEE shredding dust. Following a two-step bioleaching process, base metals and REEs were leached at high percentages in 8 days, whereas 48% of gold was mobilized in 3 h. The research findings suggest the biohydrometallurgical treatment of WEEE shredding dust, nowadays landfilled, for pursuing the recovery of materials at high supply risk, such as REEs, in order to avoid the losses of resources and limit the impacts on the environment as promoted by the circular economyconcept.

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