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The effect of flotation and parameters for bioleaching of printed circuit boards

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ABSTRACT

Waste electrical and electronic equipment (WEEE) is currently one of the fastest growing waste streams in the world. Typical for WEEE is the high content of valuable and precious metals, as well as harmful contaminants like halogens, flame retardant chemicals and plastics. Currently, WEEE treatment and metal recovery methods are imperfect, polluting and energy intensive. In this paper, novel treatment possibilities are outlined for printed circuit boards (PCB) utilizing both the flotation separation technique and acid bioleaching. Flotation, conducted after crushing and sieving of PCB, produced two fractions: metal-rich concentrate, which is more suitable for pyrometallurgical treatment than untreated PCB, and metal-poor froth suitable for acid bioleaching. It was seen that especially low pH (1.6), high initial Fe^{2+} concentration (7.8 g/l) and low PCB froth concentration in the bioleaching solution (50 g/l) were beneficial for the rapid and selective dissolution of copper. With these parameters, 99% of copper was solubilized from PCB froth in bioreactor treatment, with Cu (6.8 g/l) and Fe (7.0 g/l) being the only major metallic elements in bioleaching solution.

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1. Introduction

The growing standard of living, the rapid development of information and communication technologies and lowered product prices have shortened the lifespan of consumer electronics (Tanskanen, 2013). This has led to an increase in the generation of waste electrical and electronic equipment (WEEE), which is currently one of the fastest growing waste streams in the world with an annual growth rate of 3–5% (Drechsel, 2006) and global production of 20–50 million tons (UNEP, 2013). When treating or utilizing WEEE, printed circuit boards (PCB) play an important role, due to their presence in almost every modern electronic device. Typically PCBs contain 40% metals, 30% organics and 30% ceramics. In addition, plenty of brominated flame retardant chemicals are found. (Luda, 2011) These characteristics stress the importance of seeking efficient recycling, utilization and treatment methods for both WEEE and PCBs.

Currently the main industrial-scale treatment and metal recovery method for PCBs is the pyrometallurgical process where PCBs are mixed with metal concentrates and smelted to recover copper and precious metals (Cui and Zhang, 2008). These processes are energy intensive and may cause the formation of dioxins and other gaseous emissions due to existing flame retardant chemicals (Luda, 2011). In addition, an accumulation of impurities like Sb, Bi and As may occur, which disrupts refining process (Samuelsson and Björkman, 2014). Furthermore, some valuable elements or compounds are lost in these processes (UNEP, 2013).

Bioleaching, which is stated to be a low-cost and "low-tech" method with low hazardous emissions, may offer also new possibilities for recovering metals from WEEE and PCBs (Lee and Pandey, 2012). The autotrophic bioleaching bacteria, like *Acidithiobacillus thiooxidans* and *At. ferrooxidans* are well known for their ability to oxidize reduced sulphur compounds or elemental sulphur to produce sulphuric acid; in addition *At. ferrooxidans* and *Leptospirillum ferrooxidans* are known to oxidize iron from a ferrous to ferric state (Sand et al., 2001; Watling, 2006). However, due to the fact that WEEE and PCBs are very unconventional materials for autotrophic micro-organisms (e.g. no source for mineral Fe–S-substrates, but high occurrence of potential inhibitors), bioleaching has also been studied with heterotrophic bacteria and fungi, and cyanogenic bacteria (Brandl et al., 2001; Chi et al., 2011; Cui and Zhang, 2008; Lee and Pandey, 2012). Nevertheless, in recent studies it has been shown that also autotrophic bioleaching is clearly possible, and due to microbiologically produced sulphuric acid and ferric iron attack, copper yields especially can be significant (Liang et al., 2013; Liang et al., 2010; Xiang et al., 2010; Yang et al., 2009; Zhu et al., 2011).

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Froth flotation, which is a separation technology widely applied, e.g. in the mining industry, is a very interesting method also for PCB treatment in order to separate valuable metals from plastics and other non-metallic substances. In the flotation process, the solid material to be treated must be crushed to liberate its compounds. Then, the crushed material is mixed with liquid to produce slurry which is agitated and gas is introduced to the system. Gas bubbles attach on the surfaces of hydrophobic particles and lift them up to form a froth layer which is collected. Material that is not floated remains in solution during agitation and can be collected later from the bottom of the floatation apparatus (Heiskanen, 2014; Yarar, 2000).

This paper presents an experimental study conducted to determine whether froth flotation and autotrophic acid bioleaching could be utilized in the treatment and metal recovery process for PCBs. The role of flotation was to separate plastics and some harmful elements from metals, to produce improved quality metal concentrate for pyrometallurgical processes. It was estimated that improved quality feed for pyrometallurgical plants would increase their WEEE treatment capacity, but also decrease emissions and maintenance costs (Luda, 2011; Samuelsson and Björkman, 2014). However, PCB froth, a "waste" fraction produced by flotation, may still contain residual metals and other harmful elements in quantities that may prevent further utilization or disposal strategies according to legislation. Therefore, autotrophic acid bioleaching was utilized to remove residual metals, especially copper, from the froth.

2. Materials and methods

2.1. PCB material, pre-treatment and flotation

Used mobile phones from 2000 to 2010 were collected and dismantled manually to obtain PCBs. As flotation typically requires feed material with a particle size of 10–300 μm (Heiskanen, 2014) the PCBs were crushed with a Rivakka hammer mill and sieved to a fraction with maximum grain size below 250 μm. The oversized fraction was further crushed with an Alpine hammer mill to a particle size below 250 μm in argon gas to prevent oxidation of the metals. The obtained PCB powder was then homogenized with a rotating divider and flotated with an Outotec Labcell flotation machine. Flotation was done without any collector chemicals as their task is to manipulate and increase the hydrophobicity of certain substances of a feed. As plastics are naturally hydrophobic, they tend to accumulate in the froth, causing separation from less hydrophobic substances, e.g. metals. (Heiskanen, 2014) Flotation produced two fractions from the crushed PCBs: metal-rich concentrate and metal-poor froth. It is noteworthy that the flotation parameters were not optimized in this study and therefore the separation rate for, e.g. copper, was still relatively poor.

2.2. Microorganisms and adaptation

The mixed acidophilic culture used, enriched from a sulphide ore mine site, as described earlier (Halinen et al., 2009), contained *At. ferrooxidans*, *At. thiooxidans/albertensis*, *At. caldus*, *L. ferrooxidans*, Sulfobacillus *thermosulfidooxidans*, *Sb. thermotolerans* and some members of *Alicyclobacillus* genus. The cultivation medium included mineral salts and trace elements (Table 1), 10 g/l S^0 and 4.5 g/l Fe^{2+} (as $FeSO_4 \cdot 7H_2O$), and was incubated in a rotary shaker (150 rpm, 28 °C). The temperature selection was due to the mesophilic members of the consortium, like *At. ferrooxidans* and *At. thiooxidans* (Rawlings, 2002). A new medium was prepared every 15 days and inoculated with 10% (v/v) of former cultivation.

Table 1
Mineral salts and trace elements in cultivation medium.

Chemical formula	mg/l	Chemical formula	mg/l
$(NH_4)_2SO_4$	2160	H_3BO_3	1.4
K_2HPO_4	36	$MnSO_4 \cdot H_2O$	1.4
$MgSO_4 \cdot 7H_2O$	360	$Na_2MoO_4 \cdot 2H_2O$	0.6
$Ca(NO_3)_2$	7.2	$CoCl_2 \cdot 6H_2O$	0.4
$FeCl_3 \cdot 6H_2O$	7.9	$ZnSO_4 \cdot 7H_2O$	0.6
$CuSO_4 \cdot 5H_2O$	0.4	Na_2SeO_4	0.07

It was decided that the acidophilic culture used should be adapted to tolerate the presence of PCBs. The adaptation experiments were conducted in 100 ml Erlenmeyer flasks including mineral salts and trace elements (Table 1), 10 g/l S^0 and 4.5 g/l Fe^{2+}. The flasks were inoculated with acidophilic culture (10%, v/v), followed by the addition of <250 μm particle-sized crushed PCBs (10–15 g/l). The pH was adjusted to 2.0 with strong H_2SO_4 as the acidophilic bacteria used, like *At. ferrooxidans*, *At. thiooxidans* and *L. ferrooxidans*, thrive at this pH (Rawlings, 2002; Watling, 2006). The cultivation flasks were incubated for 15 days in a rotary shaker (150 rpm, 28 °C). If a spontaneous decrease in pH and rise in redox potential was observed, new media with an increased amount of crushed PCB was prepared and inoculated (10%, v/v) with the former successful adaptation experiment. This was continued for 60 days.

2.3. Preliminary bioleaching experiments

After adaptation, both <250 μm particle-sized PCB powder and PCB froth were bioleached to see which fraction is more suitable for microorganisms. Preliminary bioleaching experiments were conducted in 100 ml Erlenmeyer flasks including mineral salts and trace elements (Table 1), 10 g/l S^0 and 4.5 g/l Fe^{2+}. The flasks were inoculated with adapted acidophilic culture (10%, v/v), followed by the addition of either <250 μm particle-sized crushed PCB (10 and 50 g/l), or PCB froth (10 and 50 g/l). The pH was adjusted to 2.0 with strong H_2SO_4, and the flasks were incubated for 15 days in a rotary shaker (150 rpm, 28 °C). Also abiotic H_2SO_4 leaching experiments were conducted.

2.4. Bioleaching parameters for PCB froth

The PCB froth was further studied to clarify how the initial Fe^{2+} concentration effects copper leaching. These experiments were conducted in 100 ml Erlenmeyer flasks including mineral salts and trace elements (Table 1) and 10 g/l S^0. The initial Fe^{2+} concentration of the experiments was 0.0, 4.5 and 9.0 g/l. The upper value was selected due to the experiments of Xiang et al. (2010) and Yang et al. (2009), suggesting that optimal initial Fe^{2+} concentration for copper leaching from PCBs would be somewhere between 6.7 and 12.0 g/l and higher concentrations would actually hamper the leaching efficiency. After the sulphur and ferrous iron additions the flasks were inoculated with adapted acidophilic culture (10%, v/v), followed by two-day pre-cultivation without PCB froth. After pre-cultivation 20 g/l of PCB froth was added. The pH was adjusted to 2.0 with strong H_2SO_4, and the flasks were incubated for 30 days in a rotary shaker (150 rpm, 28 °C). Also abiotic H_2SO_4 leaching experiments were conducted.

2.5. Scaled-up bioleaching of PCB froth

A scaled-up reactor experiment was conducted in three-step batch mode with a 3-l continuous stirred-tank reactor (CSTR) equipped with a top-entered agitator (propeller diameter 10 cm, 120 rpm) and aeration, supplied from the bottom of the reactor

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