

PRELIMINARY BIOLEACHING EXPERIMENT OF E-WASTE

Alexandra KUCMANOVÁ¹, Zuzana SANNY¹, Kristína GERULOVÁ¹,
Matej PAŠÁK¹, Imrich CZÉRE²

¹SLOVAK UNIVERSITY OF TECHNOLOGY IN BRATISLAVA
FACULTY OF MATERIALS SCIENCE AND TECHNOLOGY IN TRNAVA
INSTITUTE OF INTEGRATED SAFETY
ULICA JÁNA BOTTU 2781/25, 917 24 TRNAVA, SLOVAK REPUBLIC

²ŽELEZNIČNÉ OPRÁVOVNE A STROJÁRNE ZVOLEN, a.s.
MÔŤOVSKÁ CESTA 259/11, 96003 ZVOLEN, SLOVAK REPUBLIC

e-mail: alexandra.kucmanova@stuba.sk, zuzana.sanny@stuba.sk, kristina.gerulova@stuba.sk,
matej.pasak@stuba.sk, czere@zoszv.sk

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Abstract

*Nowadays, we can hardly imagine our life without the achievements of modern technology, such as refrigerators, televisions, computers, or smartphones. Producers are pushing consumers to buy newer, more modern appliances instead of repairing the original ones. However, all the devices represent a huge amount of waste. E-waste is one of the fastest growing types of waste, and only less than a half of it is recycled. Its disposal poses a great challenge for the population and constitutes a huge burden for the environment. In the research described in this article, we focused on individual techniques for metals recovery from e-waste. In the experimental part, we deal with the recycling of electronic waste by bioleaching, using microorganisms *Acidithiobacillus ferrooxidans* and *Acidithiobacillus thiooxidans*. Compared to conventional techniques, bioleaching is a more environmentally friendly and economical alternative of metals recovery from e-waste.*

Keywords

Bioleaching, electronic waste, microorganisms, printed circuit boards

INTRODUCTION

Technical innovation in the field of electrical and electronic equipment keeps growing rapidly. The industry produces huge amounts of new devices, using newer and better technologies. It forces consumers to change the equipment often. Individual repairs are no longer profitable, and the purchase of newer and more modern equipment becomes more economical. The electronic devices have a short replacement cycle resulting in the exponential growth of electronic waste (e-waste) [1]. As such, it has become one of the fastest-growing

types of waste. It poses a challenge for waste management, as many electronic products contain both, hazardous and valuable materials [2]. The European Commission has issued the 2002/96/EC Directive, known as the **WEEE (Waste Electrical and Electronic Equipment) Directive**, with effect from February 13, 2003, with the aim of achieving up to 70 – 80 % recovery rates of electrical and electronic equipment [3].

ELECTRONIC WASTE

E-waste is usually referred to as **WEEE (Waste Electrical and Electronic Equipment)**, electronic waste or e-scrap. It includes a wide range of products [4]. The **material composition of e-waste** depends on its origin, manufacturer, and age. It can be defined as a mixture of various metals, plastics and ceramics [5, 6]. **The printed circuit boards (PCBs)** are an important [7] and economically attractive component of WEEE [8]. PCBs contain about 28 – 30 % metallic portion (10 – 20 % Cu, 1 – 3 % Ni, 1 – 4 % Fe, 1 – 5 % Pb, and 0.3–0.4 % of precious metals like Au, Ag, and Pt). The remaining 70 % are non-metallic parts, such as polymers, ceramics, and plastics [9, 10]. Therefore, the PCBs are considered as secondary metal reserves [11]. The conventional methods available for metal removal from e-waste and PCBs waste are **pyrometallurgical** and **hydrometallurgical techniques** [12]. Pyrometallurgical processes use heat treatments like roasting, smelting and hydrometallurgical processes are based on chemical leaching by acids/alkalis [13]. The recycling of e-waste is necessary for both, reducing environmental pollution, saving natural ores and protecting human health [9]. **Bio-hydrometallurgy** or **bio-metallurgy** is a biological method for metal recovery. It is based on the same mechanism of hydrometallurgical process and uses microorganisms for production of reagents to extract metals [14, 15] from primary (natural ores) or secondary resources (e-waste, i.e., PCBs) [11, 16, 17]. The recovery of metals via bio-metallurgy includes five different mechanisms - biotransformation, biosorption, bioaccumulation, bioleaching, biomineralization [16].

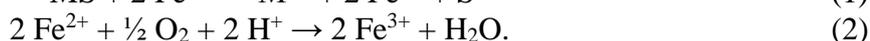
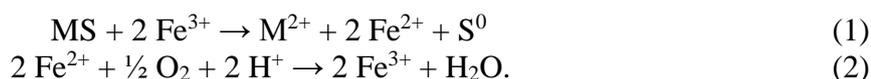
BIOLEACHING

Bioleaching, also known as microbial leaching [18], is a specialized bio-hydrometallurgical process which utilizes either metabolic activities or products of microorganisms [17] under soft operating conditions, i.e. low temperatures and a mild pHs [15]. Bioleaching methods are based on the application of the sulphur compounds oxidizing bacteria and microorganisms. The most commonly used microorganisms in bioleaching are **iron- and sulphur-oxidizing chemolithotrophic bacteria** (*Acidithiobacillus ferrooxidans*, *A. thiooxidans*, *Leptospirillum ferrooxidans*, *Ferrobacillus*), **cyanogenic bacteria** (*Chromobacterium violaceum*), **moderately thermophilic bacteria** (*Sulfobacillus thermosulfidooxidans*, *Thermoplasma acidophilum*, *S. thermosulfidooxidans*), **heterotrophic bacteria** (*Sulfolobus*, *Bacillus*, *Pseudomonas*) and **microscopic fungi** (*Aspergillus niger*, *A. ficuum* and *Penicillium simplicissimum*) [13, 14, 18, 21, 22, 37, 38, 39, 40, 41].

The bacteria can produce organic acids and complexing agents. The bioleaching has been successfully applied to remove heavy elements from sludge, galvanic slag, electronic scrap and combustion waste [19, 20, 21]. The commonly used commercial bioleaching methods to recover metals from various minerals and ores in an industrial scale include heap bioleaching, dump bioleaching and stirred reactor. Among those operations, the heap bioleaching is currently the most successful technology for the recovery of base metals, which is due to its low cost, simple operation and high yield [22].

Bioleaching can be divided into two main pathways: direct and indirect. The **direct pathway** is achieved by direct electron transfer from mineral sulphides to microorganism cells (e.g. *A. ferrooxidans*) [23] at the inoculation stage [24], without the formation of ferrous sulphate. The cells must be tightly attached to the mineral surface. The adhesion of

microorganisms to the substrate occurs first through the Van der Waals-type forces. The interaction between microorganisms and the mineral is also enhanced by the formation of extracellular compounds produced by bacteria, so-called exopolysaccharides which mainly consist of sugars, lipids, and proteins. Adherent bacteria begin to reproduce and form micro-colonies. The colonies cover the material and form a multilayer film – a biofilm [25, 26, 27]. In the **indirect pathway**, the microorganisms are not directly involved in the mineralization of metals, but they generate solid oxidizing agents [24]. Indirect bioleaching includes three primary methods: C-mediated, S-mediated and Fe-mediated. The **C-mediated** bioleaching is achieved through the reaction between e-waste and microbial metabolites (e.g., cyanide, citrate and oxalate) produced by microorganisms through carbon metabolism [28]. The **S-mediated** bioleaching can be achieved through the oxidation of reduced sulphur substances by acidophilic sulphur-oxidizing autotrophs or sulphur- and iron-oxidizing autotrophs to produce H₂SO₄ resulting in the acidification of metals from e-waste [27]. In **Fe-mediated** bioleaching, the oxidation of the reduced metal is caused by ferric ions (Fe³⁺) produced by microbial oxidation of the ferrous ion (Fe²⁺) compounds present in the mineral. Ferric ion (Fe³⁺) acts as an oxidizing agent and can oxidize metal sulphides. It is then chemically reduced to ferrous ion which can be microbially oxidized to the ferric ions again. In that case, iron has a role of an electron carrier [23]. The chemical transformations can be represented by the following simplified equations [5]:



From an industrial point of view, the indirect leaching is more beneficial. Starting the leaching process independently of the abiotic effects offers more flexibility in optimizing the process of metal extraction [25]. Some authors reported that high amounts of different metals [29][30] and organic components present in PCBs have a toxic effect on bioleaching bacteria, thus reducing the leaching efficiency [14]. On the other side, different authors [31, 32] state that *A. ferrooxidans* is a bacteria resistant to high amount of toxins to a certain extent, and can be used for recovery of heavy metals (such as Ni, Cd, Co) from exhausted batteries.

There are also external factors affecting the successful bioleaching and metabolic activity of microorganisms. Those include, for example pH, concentration of Fe²⁺, qualitative and quantitative composition of E-waste, the type of grinding material, grinding temperature and grinding time [33, 34], particle size, surface area of bioleached material, prior adaptation of the microorganisms, oxidation – reduction potential (ORP), available O₂ and CO₂, amount of nutrients and the form in which the metals are present (oxides, sulphides, metallic form) [26].

pH of the leaching medium influences both, leaching biology and chemical reactions. The pH of the medium has a direct relationship with bacterial growth, and it regulates the metal solubilisation. A majority of metals are recovered at acidic pH of 2.0 – 2.5. A higher pH decreases the bacteria-mediated oxidation of Fe²⁺ to Fe³⁺. A very low pH slows down the leaching so as the high pH has an inhibitory effect on the bacterial growth, leading to the low metal solubilisation [13].

Another factor significantly affecting the efficiency of bioleaching and relating to pH is **oxidation reduction potential (ORP)**. ORP is a parameter indicating the Fe³⁺/Fe²⁺ ratio in the system with a higher concentration of Fe²⁺ [8, 11]. There is an indirect relationship between pH and ORP. When pH increases, ORP decreases, and vice versa. This has been proven by many researchers [9, 33].

The optimal bioleaching **temperature** depends on the growth requirements of different microorganisms. Most mesophilic acidophiles mobilize metals at the optimal temperature range of 25 - 30 °C, while thermophiles leach at the optimal temperature of 40 - 45 °C. Therefore, the

temperature must be compatible with the active growth and metabolism of the selected bacteria involved in the biological leaching process in order to effectively solubilize the metal [13].

Another important factor affecting successful bioleaching is an **appropriate nutrient requirement of the leaching medium**. It affects the metal dissolution by arresting the cell growth. The rate of bioleaching of the iron and sulphur-oxidizing bacteria varies with the concentration of Fe^{2+} and S^0 . An increase of the Fe^{2+} concentration in nutrient medium causes precipitation of the Fe^{3+} -forming **jarosite** which inhibits the bioleaching process [13]. Nazari et al. examined the effect of ferrous iron concentration in the growth medium (9K), pH, and temperature of jarosite formation in the presence of *Acidithiobacillus ferrooxidans*. They found that the highest amount of jarosite was formed at a ferrous sulphate concentration of 50 g L^{-1} at $32 \text{ }^\circ\text{C}$ and an initial pH of 2.2 [35]. Jin-yan et al. studied the effect of *Thiobacillus ferrooxidans* on the oxidation of metal sulphide and the formation of jarosite. The minimal jarosite precipitates (3.73 g L^{-1}) and the good growing activity of bacteria was at the initial pH of 1.6 – 1.7 with the Fe^{2+} concentration of $9 - 10 \text{ g L}^{-1}$, and the applied potential of -0.5 V for 7 hours [36]. To assess the success of bioleaching, it is important to measure the Fe^{3+} and Fe^{2+} concentrations [31].

The aim of our experiment was to perform the bioleaching of selected E-waste using acidophilic bacterial strains, and to verify the ability of bacterial strains to speed up the dissolution of metals into solution.

MATERIALS AND METHODOLOGY

Adapted bacterial strains

Based on literature [30, 42, 43], we performed a two-step bioleaching. For the bioleaching experiments, an adapted culture of *A. ferrooxidans* and a nonadapted culture of *A. thiooxidans* were used. The adaptation process was taken 5 days in the presence of the same electronic waste sample as it was used in the experiment. Bacteria were predominantly cultivated in a sterile 9K culture medium which contained ferrous sulphate (44.22 g L^{-1} , as the energy source), $(\text{NH}_4)_2\text{SO}_4$ (3.0 g L^{-1}), MgSO_4 (0.5 g L^{-1}), K_2HPO_4 (0.5 g L^{-1}), KCl (0.1 g L^{-1}) and $\text{Ca}(\text{NO}_3)_2$ (0.01 g L^{-1}). Both cultures of nonadapted microorganisms were obtained from the Department of Mineral Biotechnology of the Slovak Academy of Sciences in Košice.

Experimental design of bioleaching

The electronic waste samples used in the experiment are shown in Figure 1. The samples were pre-treated in a saturated solution of NaOH and KOH to remove protective coating. In total, 400 mL of 9K culture medium with the inoculum (mixed bacteria of *A. ferrooxidans* and *A. thiooxidans* 1:1) were transferred into a 500 mL Erlenmeyer flask and cultivated under **constant temperature** (30 ± 1) $^\circ\text{C}$. The inoculum was used at three different concentrations (10 %, 15 % and 25 % (vol.)). The control sample did not contain any inoculum. The **pH** during the bioleaching or the adaptation (measured with WTW Multi 340i) was maintained in the range of 1.8–2.0 by the addition of H_2SO_4 . An **oxidation reduction potential (ORP)** was measured by a maintenance-free SenTix®PtR redox combination electrode (WTW Multi 340i). The suspension was stirred constantly at 160 r min^{-1} and continuously aerated. The **aeration** was provided by an ALITA Air Pump AL-6SA using a glass pipette. Prior to injection, the air was purified on a Millex-FG 50mm PTFE air filter with a pore size of $2 \mu\text{m}$. The aeration was used only in the bioleaching experiment itself. The electronic waste samples were analysed under a microscope at baseline and after 0, 48, 96 and 120 hours. The total experiment duration was 120 hours.

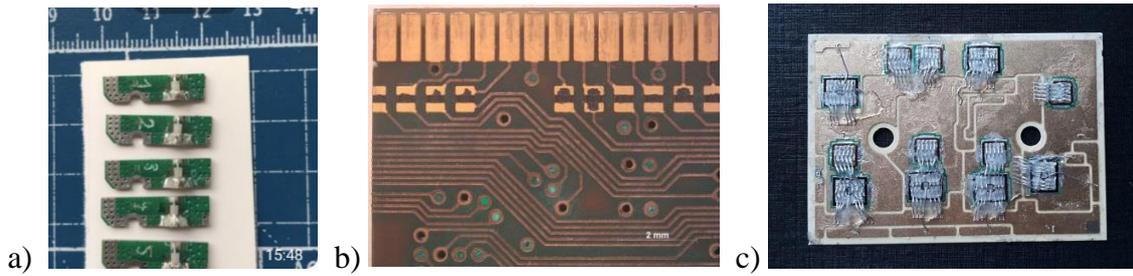


Figure 1 Samples (a, b, c) of untreated electronic waste used in the experiment of bioleaching

RESULTS AND DISCUSSION

Figures 2-4 document the e-waste after bioleaching process with and without the presence of bacteria (control). Cross-section of the e-waste samples is provided in Figure 2, and top views are given in Figures 3 (an overview) and 4 (detail). As can be seen, the bioleaching in the presence of bacteria was faster; it helped to transfer the metals from electronic waste into solution more efficiently compared to the control sample.

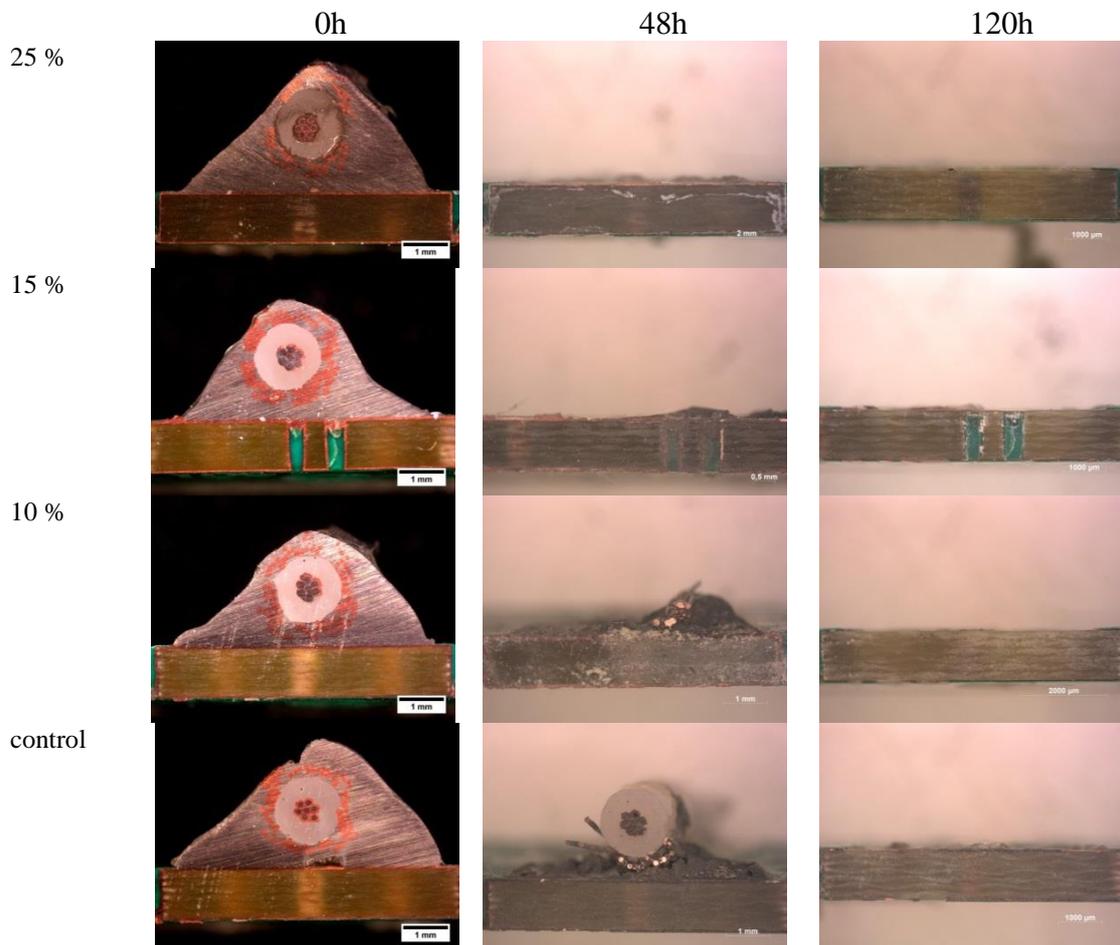


Figure 2 Samples (a) of E-waste treated by different amounts of inoculum

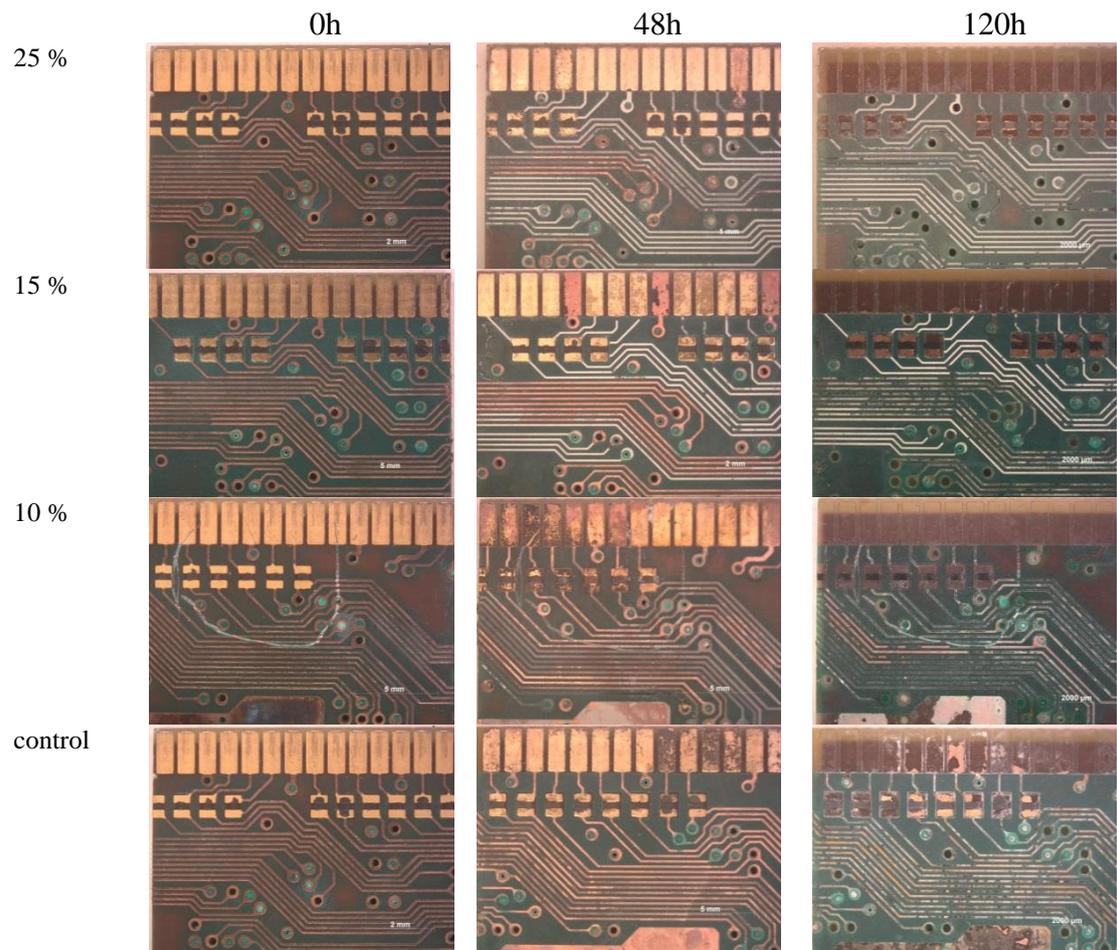
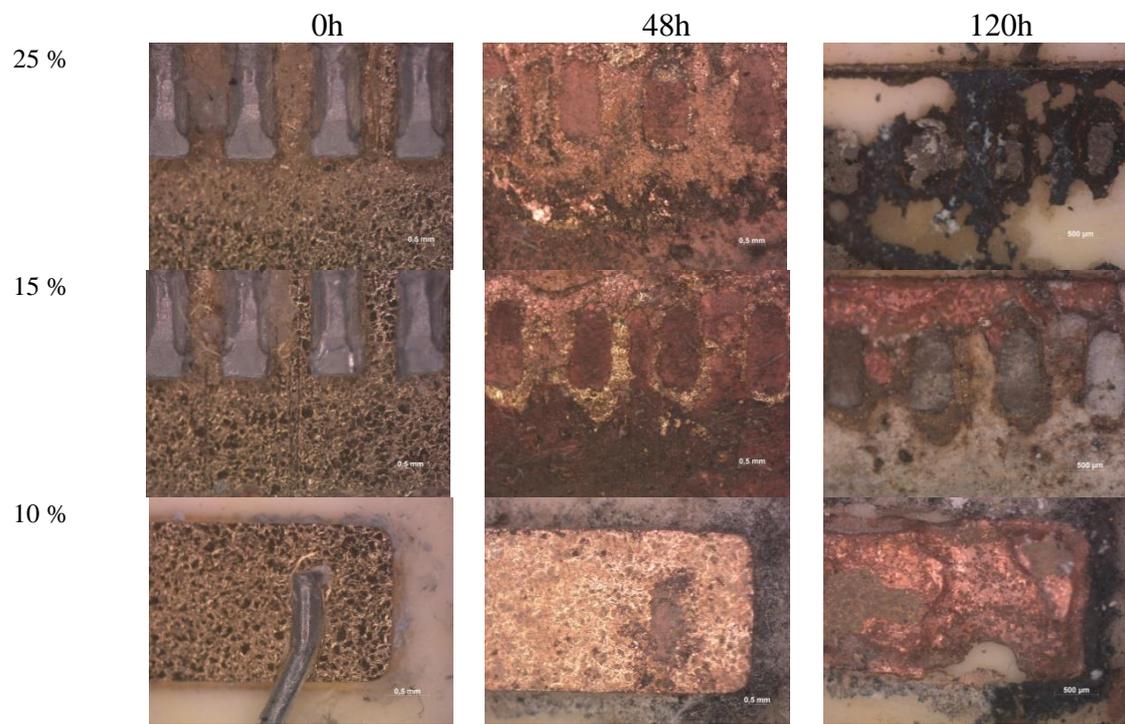


Figure 3 Samples (b) of E-waste treated by different amounts of inoculum



control



Figure 4 Samples (c) of E-waste treated by different amounts of inoculum

Despite a regular measurement and adjustment to the desired value with sulfuric acid, **pH** slightly increased (Figure 5). The pH factor of solution is very important, as it usually controls the bacterial growth. According to Jin-yan et al. [36], pH affects the amount of jarosite precipitated and rate of bacterial growth. The bio-oxidation of Fe^{2+} could have consumed protons from the solution, which was accompanied by an increase of pH [44, 45]. The pH increase promoted a ferric ion formation and jarosite precipitation. It resulted in the inhibition of the bacteria-mediated iron oxidation owing to the formation of a precipitate layer around the bacterial cells [17].

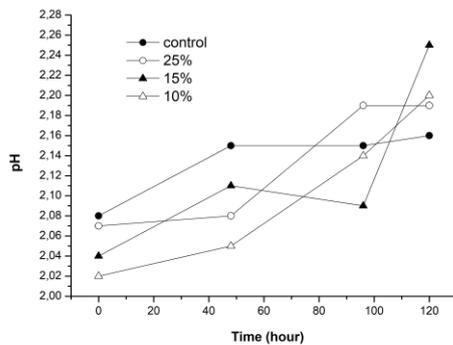


Figure 5 pH kinetics during the experiment

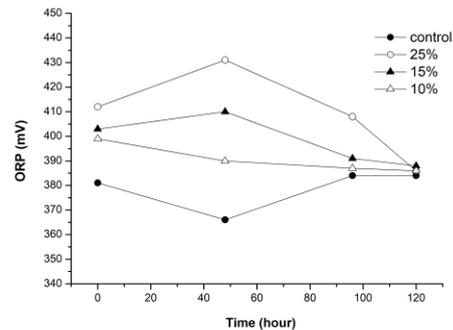


Figure 6 Oxidation reduction potential kinetics during the experiment

An increase of **ORP**, given in Figure 6, reflected the activity of microorganisms. It indicates that the leaching is promoted by bacteria and there was a bacterial oxidation and regeneration of Fe^{2+} to Fe^{3+} [34]. The ORP began to decrease in the samples with high concentration of inoculum after 48 hours. Similar results were also published in reference [9]. Willner [33] states that the decrease of ORP at the end of the process indicates a regressive phase of bioleaching and slows down the activity of bacteria in solutions.

After the inoculum adaptation process, it is recommended to centrifugate the bacteria from the cultivation solution. For better assessment of the conditions during bioleaching, it is also recommended to evaluate the level of Fe^{2+} and Fe^{3+} in the solution.

CONCLUSION

In the experiment described in this article, we investigated the possibility of e-waste recycling by bioleaching aided by *Acidithiobacillus ferrooxidans* and *Acidithiobacillus thiooxidans*. Bioleaching is a more environmentally friendly and economical alternative to metals recovery, compared to conventional techniques. We observed a higher rate of metal leaching into the solution in presence of inoculum. The leaching started with Al solder and continued with Cu substrate. An increase of ORP reflected the growth activity of microorganisms. It indicated that leaching is promoted by bacteria, and there was a bacterial

oxidation and regeneration of Fe^{2+} to Fe^{3+} . Determination of the exact metal concentrations in solution is a task for future experiments.

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ORCID

Alexandra Kucmanová	0000-0003-3089-7712
Zuzana Sanny	0000-0003-2079-4277
Kristína Gerulová	0000-0003-2588-8627
Matej Pašák	0000-0001-8342-3727