

Preliminary Study of Hydrometallurgical Extraction of Silver from Selected E-Waste

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Abstract

Electronic relays (ER) are a type of switch which are an essential part of electronic devices used to open or close circuits by using electronic components without any mechanical operation. After decommissioning, it can be considered as an important source of base and precious metals with high economic value. The annual growth rate of about 4-5 % in 2016 is one of the fastest growing waste stream, and only about 20 % of e-waste is recycled. After the use-phase, electronic devices become electronic waste (e-waste); consequently, it is important to consider e-waste as a secondary supply for the recovery of precious metals. In this study, a simple hydrometallurgical recovery method for silver (Ag) extracting from used electronic relays was performed. The silver extractions consisted of six stages: disassembling of relays, removal of base metals, leaching in nitric acid, precipitation, conversion silver oxide and melting. Measurements of extracted precious metals were carried out by scanning electron microscope (SEM) and EDS (Energy Dispersive Spectrum) analysis. The purity rate of the final deposit was 94.9 % on the surface of the sample, and the final silver recovery yielded 0.44 % of the raw material. During the second stage of the experiment, mixed types of electronic relays were used to compare the yields. Electronic relays can be considered as an important source of base and precious metals with high economic value. The final yield of the mixed electronic relays reached up to 0.54 % of Ag with the purity of over 95 %.

Keywords

E-waste, EDS analysis, Hydrometallurgy, Recycling, Silver extraction



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Introduction

E-waste has been a major segment of the waste produced in the past decades (Widmer et al., 2005) (Behnamfard et al., 2013). In recent years a huge growth in the use of many information and communication technology products has been observed. In the European Union, approximately 8 million tonnes of e-waste are generated every year with an annual increase of 3 – 5 % (Drechsel, 2006) while approximately 20 – 50 million metric tonnes of e-waste are generated worldwide (Petraniková, 2008). There are also growing concerns about the e-waste generated in developed countries due to the lack of infrastructure for environmentally sound management of e-waste. The reasons for decreasing life span of electrical and electronic devices are as follows (Akcil et al., 2015, Ivanova et al., 2018):

- Incoming of highly advanced and technically skilled devices/equipment at a lower price and more features.
- Rapid growth in the lifestyle of human beings with modern facilities having user-friendly electrical and electronic equipment.
- Stiff competition amongst individuals to use and small enterprises and industries to produce and sell the best products made on advanced technologies.

WEEE (Waste of electric–electronic equipment) contains a variety (>1000) of organic and inorganic substances with its composition depending largely on the type, manufacturer and age of the equipment (Table 1). WEEE can contain up to 61% metals and 21% plastics (Widmer et al., 2005, Biały et al., 2019, Biały et al., 2019a). Polyethylene, polypropylene, polyesters and polycarbonates are typical plastic components (Gramatyka et al., 2007, Sviatskii et al., 2020). Many of the materials such as chlorinated and brominated substances, toxic metals, photoactive and biologically active materials, acids, plastics and plastic additives present in WEEE are highly toxic.

Table 1. Material composition of WEEE (Widmer et al., 2005)

Material	Content [%]
Metals	
Iron and steel	47.9
Copper	7.0
Aluminium	4.7
Non-ferrous	1.0
Total	60.6
Plastics	
Flame retarded plastics	5.3
Non-flame retarded plastics	15.3
Total plastics	20.6
Glass	5.4
Rubber	0.9
Wood and plywood	2.6
Ceramic	2.0
Printed circuit boards	3.1
Other	4.6

E-waste encompasses valuable metals, alongside numerous dangerous materials. An enormous number of dangerous metals (Cd, Hg, Pb, Cr) from e-waste may contribute to increasing the toxicity levels of the ecosystem (Qu et al., 2019). E-waste material in the environment may increase the exposure risk of hazardous materials. Serious pollution of groundwater and human health could be associated with these hazardous materials. One of the important routes to enter toxic chemicals from e-waste to the human body is the soil-crop-food pathway. Toxicity, negative environmental impact, as well as financial reimbursements from e-waste, are necessitated the need for metal recovery from e-waste. The utilization of e-waste could be a potential secondary source of precious and base metals (Otto et al., 2018). Without knowing the hurtful impact, E-waste has been discarded in the open wellsprings of water bodies, the agricultural land, and open landfills by unconscious social people. For the open disposal of the e-waste containing toxic substances in water bodies and landfills pollutes the groundwater (Romaric et al., 2019), as shown in Fig. 1.

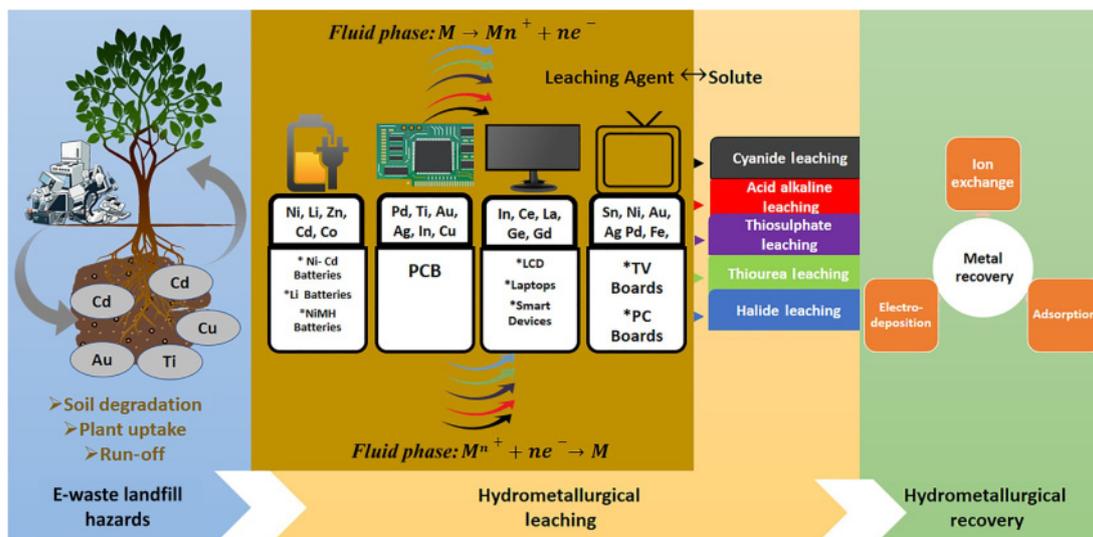


Fig. 1. E-waste from landfills to resource recovery (Ashiq et al., 2019)

E-waste from such equipment contains many toxic elements such as lead, mercury, cadmium, nickel, chromium, etc., which has an adverse impact on our environment. Moreover, e-waste also contains many valuable metals, such as gold, silver, platinum, and palladium (Tripathi et al., 2012). The proper management of discarded electronic devices is an emerging issue for solid waste professionals throughout the world because of the large growth of the waste stream, and the content of toxic metals in them, most notably heavy metals such as lead (Jang and Townsend, 2006). Harmful effects of various metals present in the electronic waste on human health are summarized in Table 2.

Table 2. Hazardous substances and their possible adverse effects (Kaya, 2016)

Substances	Occurrence in WEEE	Possible adverse effects
Lead (Pb)	CRT screens, batteries, PCBs	Vomiting, diarrhea, convulsions, coma or even death, appetite loss, abdominal pain, constipation, fatigue, sleeplessness, irritability, and headache
Mercury (Hg)	Fluorescent lamps, some alkaline batteries, switches	Brain and liver damage
Chromium VI (Cr ⁶⁺)	Data tapes, floppy-discs	Irritating to eyes, skin and mucous membranes, DNA
Barium (Ba)	Getters in CRT	Brain swelling, muscle weakness, damage to the heart, liver, and spleen
Cadmium (Cd)	NiCd batteries, fluorescent layer (CRT screens), printer inks and toners	Symptoms of poisoning (weakness, fever, headache, chills, sweating and muscle pain), lung cancer and kidney damage
Arsenic (As)	Gallium arsenide in light-emitting diodes (LED)	Skin diseases, decrease nerve conduction velocity, lung cancer
Americium (Am)	Smoke detectors	Radioactive element
Antimony (Sb)	Flame retardants in plastics	Carcinogenic potential
Chlorofluoro carbon (CFC)	Cooling units, insulation foams	The deleterious effect on the ozone layer, increased incidence of skin cancer and/or genetic damages
Polychlorinated biphenyls (PCB)	Condensers, transformers	Cancer, effects on the immune systems, reproductive system, nervous system, endocrine system, and other health effects
PBDEs, PBBs	Flame retardants in plastics	Hormonal effects, under thermal treatment possible formation of dioxins and furans

Currently, e-waste recycling focuses mainly on mechanical approaches, pyro-metallurgy, bio-metallurgy, and hydro-metallurgy (Hsu et al., 2019; Liu et al., 2019). The reason for preference of hydro-metallurgy over pyro-metallurgy is because of low or no gas emission compared to pyro process which releases toxic gases (dioxins/furans) and volatile metals, dust, Cl₂, Br₂, SO₂ and CO₂ together with others Pb, Hg, Cr⁶⁺, Cd, flame retardants. No dust or low dust generation, low energy consumption, high recovery rate, no slag generation except few plastics, and easy working conditions (Ni et al., 2013; Tue et al., 2013; Zhang et al., 2012). According to these studies (Andrews et al., 2000; Cui and Zhang, 2008), hydro-metallurgy could be preferred over pyro-metallurgy for the recovery of precious metals such as gold, silver, and platinum.

Table 3. Development of global silver demand by sectors (2011 - 2019) (The Silver Institute and The Materials Focus, 2020)

Metric t	2011	2012	2013	2014	2015	2016	2017	2018	2019
Industrial	15804	14012	14332	13984	14189	15250	16087	15909	15891
...of which photovoltaics	2127	1711	1571	1505	1683	2914	3166	2877	3070
Photography	1916	1633	1425	1356	1281	1176	1092	1064	1048
Jewellery	5045	4952	5819	6018	6302	5885	6106	6317	6261
Silverware	1291	1247	1421	1630	1760	1627	1795	2034	1860
Net physical investment	8460	7490	9334	8790	9654	6653	4858	5154	5788
TOTAL	32515	30590	33246	31775	33187	30963	30005	30739	30848

Table 4 shows the development in global silver supply by the sources During the last decade (2011 – 2019). Global recycling edged higher last year (2019), up 1.3 % to 5,284t. Volumes from industrial end-uses, the biggest source of scrap, rose 2 % to the highest level this decade (The Silver Institute, 2020).

Table 4. Development of global silver supply by sources (2011 - 2019) (The Silver Institute and The Materials Focus, 2020)

Metric t	2011	2012	2013	2014	2015	2016	2017	2018	2019
Mine production	23642	24656	26136	27293	27772	27753	26855	26369	26018
Recycling	7244	6718	5994	5440	5179	5113	5216	5216	5284
Net hedging supply	370	-	-	333	68	-	-	-	488
TOTAL	31405	31489	32183	33100	33050	32901	32105	31626	31822

Spent electronic equipment consists of several components in the form of metals and multicomponent elements. The base metals include iron, aluminum, nickel, zinc, selenium, indium, and gallium. The noble metals can be divided into copper, palladium, or gold, silver. Hazardous substances that can be found in spent electronic equipment include mercury, beryllium, lead, arsenic, cadmium, antimony and plastics, glass, and ceramics. Depending on many factors, such as the age of the device, manufacturer, or the type of equipment, the content of the individual electronic component in the waste is mixed (Fornalczyk et al., 2013). Spent electronic equipment consists of several components in the form of metals and multicomponent elements. The base metals include iron, aluminum, nickel, zinc, selenium, indium, and gallium. The noble metals can be divided into copper, palladium, or gold, silver. Hazardous substances that can be found in spent electronic equipment include mercury, beryllium, lead, arsenic, cadmium, antimony, and plastics (Fornalczyk et al., 2013). Table 2 shows the selected material composition of electronic devices. A decisive impact on the value of electronic scrap has the content of precious metals, although iron and plastic are dominant components, and a seemingly small content of precious metals in different electronic devices (<0.5 %).

Table 5. Composition of metals from different e-waste samples (Cui and Zhang, 2008)

E-waste	Weight [%]					Weight [ppm]		
	Fe	Cu	Al	Pb	Ni	Ag	Au	Pd
TV board scrap	28	10	10	1.0	0.3	280	20	10
PC board scrap	7	20	5	1.5	1	1000	250	110
Mobile phone scrap	5	13	1	0.3	0.1	1380	350	210
Portable audio scrap	23	21	1	0.14	0.03	150	10	4
DVD player scrap	62	5	2	0.3	0.05	115	15	4
Calculator scrap	4	3	5	0.1	0.5	260	50	5
PC mainboard scrap	4.5	14.3	2.8	2.5	1.1	639	566	124
Printed circuit boards	12	10	7	1.2	0.85	280	110	-
Printed circuit boards	5.3	26.8	1.9	-	0.47	3300	80	-

Although iron and plastic are dominant components, in terms of weight, a seemingly small content of precious metals in different electronic devices (<0.5 %), constitutes about the electronic scrap value (Table 4) (Fornalczyk and Saturnus, 2013). Analysing only computer equipment and mobile phones, this share is 3 % of the world's production for Ag, 4 % for Au, and 16 % for Pd. In 2019 the global silver demand was 30848 metric tonnes (Table 3). The highest demand is in long-termed achieved in the industry, which also includes the electronics and IT sector.

More than ten times higher purity of precious metals in waste printed circuit boards compared to the rich ore content attract the attention to extract noble metal from e-waste. Hence, the extraction of noble metals (Au, Pt, Pd, Ta, Te, Ge, Se) from e-waste should be given major priorities. However, the realization of recycling should be the basis of maximum recovery and minimum negative impact on the environment (Islam et al., 2020). This paper discusses the extraction of silver from used electronic relays via a simple hydrometallurgical process. The objective was to determine the relative amount of Ag recoverable from this type of waste.

Material and Methods

Chemical leaching (Fig. 2) involves leaching either by using acid or ligand supported complexation. Chemical leaching of metals from E-waste can also be done by utilizing various inorganic-acids.

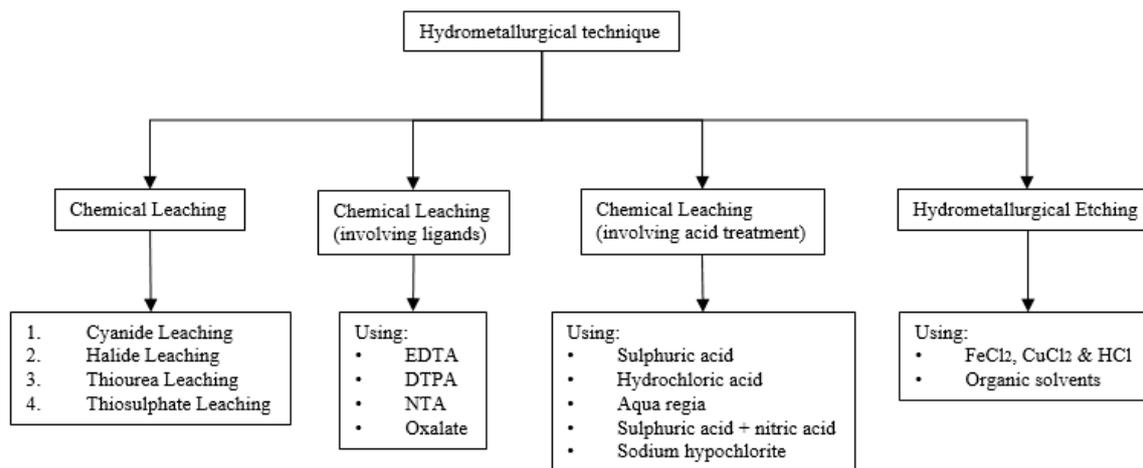


Fig. 2. Types of hydrometallurgical techniques

In this paper, two sets of electrical relays were used to extract silver by hydrometallurgical acidic techniques. The difference in the two sets was in the used precipitation of the silver from the pregnant leach solution.

The first set consisted of seven commercially used electrical relays (Allen-Bradley 700-HA33Z2-3) containing nine silver contacts made of silver, copper, nickel, and aluminum. The weight of the whole relay was 86.74 grams. The experimental procedure consisted of six main steps. All of the used chemicals were purchased from CentralChem s.r.o. The used chemicals were of analytical grade. After collecting the raw material, the relays were manually disassembled, and the silver-containing contacts were collected in a beaker. To remove all of the base and redundant metals except silver, the contacts were treated by concentrated hydrochloride acid + 35 % hydrogen peroxide (100 ml + 5 ml). The beaker was placed on a heating plate to accelerate the reaction of metals removal. Chemicals were added and heated until no visible signs of reaction were observed, and only silvery metal was present. The contacts were then washed three times with distilled water, and in the next step, concentrated nitric acid with distilled water (50 ml + 50 ml) were added to the beaker with the contacts. After two hours, the contacts were completely dissolved in the solution in the form of silver(I) nitrate (AgNO_3). To precipitate the silver(I) nitrate from the solution, a concentrated hydrochloric acid was used to produce AgCl (silver(I) chloride). After three washings with boiling distilled water, sodium hydroxide (NaOH) was added to convert AgCl to silver(II) oxide (Ag_2O). As a final step, the silver oxide dust was placed into a melting dish and melted at a temperature of $980\text{ }^\circ\text{C}$ to form a single bead of silver using a muffle furnace. Analysis of the metal was performed using a scanning electron microscope (SEM) and energy dispersive spectrum (EDS) analysis. The main individual steps and corresponding reactions of the procedure are shown in Table 6.

Table 6. Main steps of the silver extraction from electrical relays (1st set)

NR.	OPERATION	MAIN REACTION
1.	Disassembly of relays	–
2.	Removal of redundant metals by hydrochloride acid + hydrogen peroxide	$\text{H}_2\text{O}_2 + \text{Cu} + 2\text{HCl} \rightarrow \text{CuCl}_2 + 2\text{H}_2\text{O}$ $\text{H}_2\text{O}_2 + \text{Ni} + 2\text{HCl} \rightarrow \text{NiCl}_2 + 2\text{H}_2\text{O}$ $3\text{H}_2\text{O}_2 + 2\text{Al} + 6\text{HCl} \rightarrow 2\text{AlCl}_3 + 6\text{H}_2\text{O}$
3.	Dissolving in nitric acid	$\text{Ag} + 2\text{HNO}_3 \rightarrow \text{AgNO}_3 + \text{NO}_2 + \text{H}_2\text{O}$
4.	Precipitation with hydrochloric acid	$\text{AgNO}_3 + \text{HCl} \rightarrow \text{AgCl} + \text{HNO}_3$
5.	Conversion with sodium hydroxide	$2\text{AgCl} + 2\text{NaOH} \rightarrow \text{Ag}_2\text{O} + \text{H}_2\text{O} + 2\text{NaCl}$
6.	Melting	$2\text{Ag}_2\text{O} \xrightarrow{t^\circ\text{C}} 4\text{Ag} + \text{O}_2$

In the second stage of the research, ten electrical relays, each containing nine contacts made of silver, copper, nickel, and aluminum were used. The weight of the whole relay was 86.74 grams. The experimental procedure consisted of four main steps. All chemicals (analytical grade) were purchased from CentralChem s.r.o. Contacts were mechanically trimmed from relays, put in a beaker, and treated by a mixture of concentrated HCl and 35 % H₂O₂ (100 ml + 5 ml) to remove all the base metals. The solution was heated by a heating plate to increase the reaction speed. After the removal of all metals, the residual silver coatings were washed by distilled water. In the next step, concentrated HNO₃ with distilled water (ratio 1:1) was used to dissolve the silver. After two hours, all silver was completely dissolved, forming silver(I) nitrate (AgNO₃). In this case, pure metallic silver was obtained from the solution by cementing on a solid copper cylinder. Obtained silver was washed by hot distilled water three times and subsequently melted into one bead. The main individual steps and corresponding reactions of the procedure are shown in Table 7.

Table 7. Main steps and corresponding reactions used to extract silver from electrical relays (2nd set)

Nr.	Operation	Main reaction
1.	Mechanical separation of contacts	–
2.	Removal of redundant metals by HCl + 35 % H ₂ O ₂	$\text{H}_2\text{O}_2 + \text{Cu} + 2 \text{HCl} \rightarrow \text{CuCl}_2 + 2 \text{H}_2\text{O}$ $\text{H}_2\text{O}_2 + \text{Ni} + 2\text{HCl} \rightarrow \text{NiCl}_2 + 2\text{H}_2\text{O}$ $3 \text{H}_2\text{O}_2 + 2 \text{Al} + 6 \text{HCl} \rightarrow 2 \text{AlCl}_3 + 6 \text{H}_2\text{O}$
3.	Dissolving in nitric acid	$\text{Ag} + 2\text{HNO}_3 \rightarrow \text{AgNO}_3 + \text{NO}_2 + \text{H}_2\text{O}$
4.	Cementing on the copper cylinder	$\text{AgNO}_3 + \text{Cu} \rightarrow \text{Ag} + \text{CuNO}_3$

The summarised flowchart of the processes is shown in the Figure 4.

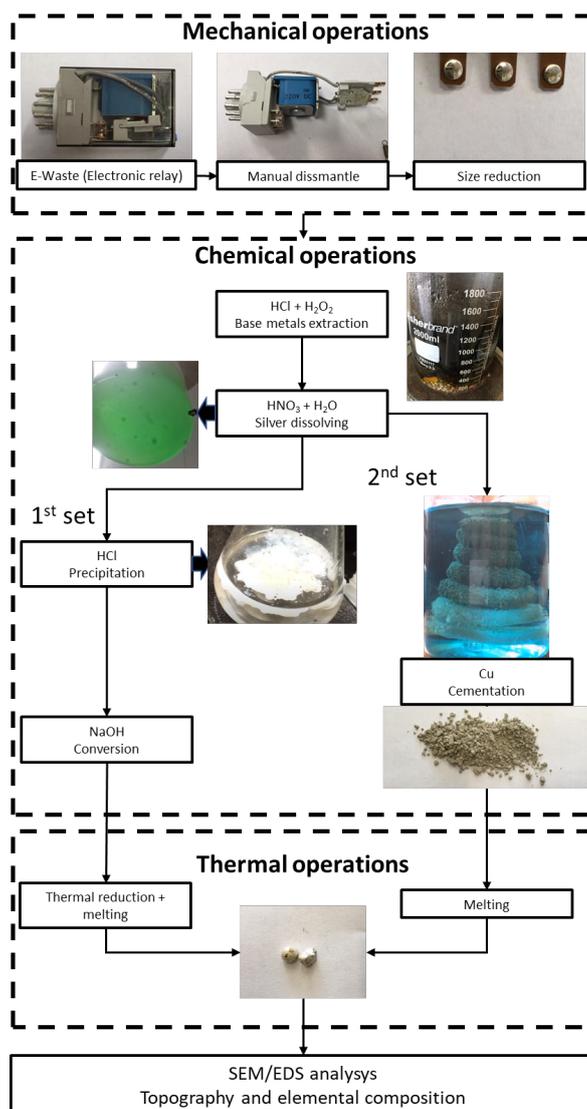


Fig. 3. Flowchart of the experiments

Results and Discussion

Researchers have indicated that effective recovery of precious metals from e-waste is feasible (Ficeriová et al., 2011, 2005; Ficeriová and Baláž, 2010). To illustrate, printed circuit board (PCB) of a PC can contain up to 20 % Cu and 250 g/ton Au, which are significantly high, i.e. 25–250-fold for gold and 20–40-fold for copper when compared with gold ores (1 – 10 g/ton Au) and copper ores (0.5–1 % Cu), respectively. Recycling turns WEEE into a secondary resource allowing the recovery and reuse of metals and non-metals contained and mitigating the environmental impact of WEEE (Cui and Zhang, 2008; Havlík et al., 2010; Yazici et al., 2010).

The recovery yield of silver obtained from the first set of electrical relays reached 0.44 %. In each of seven electrical relays, there are nine silver-containing contacts made of silver, copper, nickel, and aluminum. The total weight of one whole relay was 86.744 g (seven relays weight 607.21 g). After mechanical removal of the nine silver-bearing contacts, its weight was 2.892 g (63 pieces of contacts weight 182.196 g). After acid – peroxide bath, the contacts weighed 3.981 g. In total, 2.667 g of silver was extracted from the material by this process. Electrical relay mainly consists of circuitry, plastic, and base metals, which makes it easily recyclable e-waste. This kind of e-waste becomes a valuable material for further processing of basic mechanical separation. Therefore, an estimated concentration of silver in this type of relay is 4400 g/t.

The recovery yield of silver obtained from the second set of electrical relays reached 0.54 %. The total weight of ten relays was 921.35 g. The total weight of separated contacts (90 pieces) was approximately 25.4 g. In total, 4.79 g of silver was extracted from the material by this process, which is 5200 g/t (from the total weight).

Cui et al. (2008) determined the amount of Ag in different e-waste samples to be comparable or lower than in keyboards. Li et al. (2019), reported that CPU sockets also contain silver (431 g/t) with a lower yield. For the mining sector, the silver reserves are divided into known reserves and hidden reserves and stratified into four levels of ore quality: 1. Rich silver is labeled as extra high quality (10,000 – 6000 g/t), 2. High grade (1100 – 800 g/t), 3. Low grade (100 – 80 g/t) and 4. Ultra-low grade (below 10 – 8 g/t), which means this type of e-waste could be considered to be an extra high-quality source of silver (Sverdrup et al., 2014). As shown in Table 8, copper and precious metals contribute invariably and extensively to the economic potential of all WEEE.

Table 8. Composition of metals from different e-waste samples

Type of e-waste	Content (% or g/ton) and contribution to economic potential (%) (in brackets)								
	Fe (%)	Cu (%)	Al (%)	Pb (%)	Sn (%)	Ni (%)	Au (g/ton)	Ag (g/ton)	Pd (g/ton)
Price (\$/ton) ^a	525	9211	2298	242	25.900	24.180	4.9x10 ⁷	1.06x10 ⁶	2.68x10 ⁷
PC boards	7 (0)	20 (10)	5 (1)	1.5 (0)	2.9 (4)	1 (1)	250 (64)	1000 (5)	110 (15)
PC boards	2.1 (1)	18.5 (10)	1.3 (0)	2.7 (0)	4.9 (7)	0.4 (1)	86 (26)	694 (4)	309 (51)
TV boards ^b	0.04 (0)	9.2 (61)	0.75 (1)	0.003 (0)	0.72 (13)	0.01 (0)	3 (11)	86 (7)	3.7 (7)
TV boards	28 (5)	10 (28)	10 (7)	1 (1)	1.4 (10)	0.3 (2)	20 (30)	280 (9)	10 (8)
Mobile phones (1999)	5 (0)	13 (5)	1 (0)	0.3 (0)	0.5 (0)	0.1 (0)	350 (67)	1380 (6)	210 (22)
Typical ore grades	25	0.5	30	5	0.5	0.5	1	-	-

^a Metal prices are from London Metal Office (LME) official prices for cash seller and settlement (December 14th, 2010)

^b Manufacturer waste without components

Analysis of the metal beads was performed using a scanning electron microscope (SEM) and energy dispersive spectrum (EDS) analysis (Figure 5). EDS (Energy Dispersive Spectrum) analysis was used to determine the chemical composition and concentration of individual elements. It was performed by the energy-dispersive X-ray spectroscopy analyzer, which was a part of the scanning electron microscope of JEOL JSM 7600 Ftype. The topography of silver beads was observed at an accelerating voltage of 20 kV, current 2 nA, and a working distance of approximately 15 mm. The chemical composition of silver bead was investigated by software INCA.

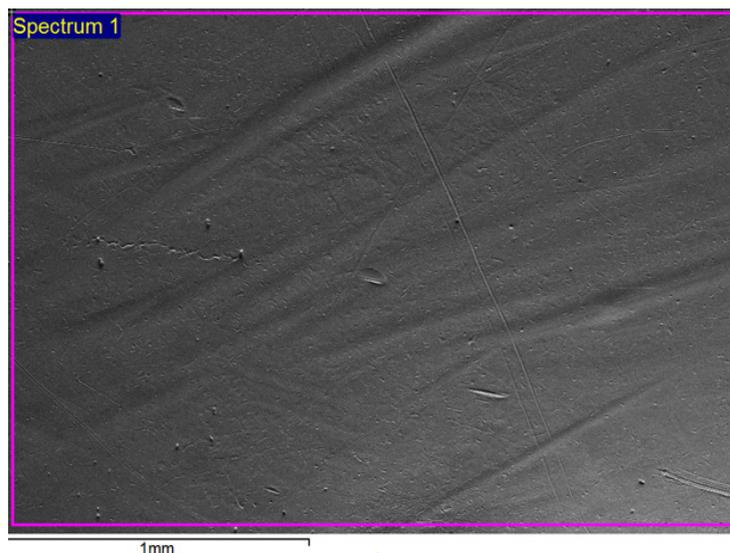


Fig. 4. SEM image of the resulting silver sample (1st set)

Table 9 shows the results of EDS analysis. Elemental analysis showed that the purity of obtained silver beads was 88.45 % (1st set) and 95.65 % (2nd set). Because only the surface of the sample is analyzed by this method, it is possible that the sample has a higher purity under the top layer. The presence of oxygen may be caused by the formation of silver oxide, which is produced under normal conditions when silver reacts with the oxygen present in the air. The presence of carbon may be explained by the impurities on the melting cupel from the previous melting. The magnesium also comes from the cupel, which is made of pure and compressed magnesium oxide (MgO).

Table 9. EDS analysis of the silver bead

	Element content (%)					
	C	O	Mg	Si	Ag	Total
Bead 1	4.53	6.43	0.32	0.27	88.45	100.00
Bead 2	3.78	0.34	0.23	-	95.65	100.00

The waste solution containing dissolved metals from the process was cemented according to the electronegativity series, and as a result, almost zero waste was produced during the whole extraction process.

Conclusion

At present mechanical and hydrometallurgical separation technologies has a relatively high recovery rate of precious metals, although these methods have not been adopted by countries with low GDP because of its complexity and high economic cost. Hence, recycling this kind of e-waste can both decrease the pressure on natural resources and reduce environmental contamination. This paper presented a simple silver extracting method from electrical relays and determined the quantity of recoverable precious metal. EDS analyses showed the purity of the obtained metal. The advantages of the presented hydrometallurgical method were its cost-effectiveness, environmental friendliness, and time-efficiency. We demonstrated that discarded electrical relays contain an appreciable quantity of silver with high economic potential. Using this method, it is possible to extract 4400 g/t –5200 g/t of silver from commercially used electrical relays (Allen-Bradley 700-HA33Z2-3) with 88.45 % purity (1st set) and 95.65 % purity (2nd set). The procedure should be further studied for different aspects and for different e-wastes to help with negative impacts on the environment.

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