

RECOVERY OF METALS FROM PRINTED CIRCUIT BOARDS BY MEANS OF ELECTROSTATIC SEPARATION

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Abstract:

Without the use of appropriate recycling technologies, the growing amount of electronic waste in the world can be a threat to the development of new technologies, and in the case of improper waste management, may have a negative impact on the environment. This is due to the fact that this waste contains large amounts of valuable metals and toxic polymers. Therefore, it should be recycled in accordance with the assumptions of the circular economy. The methods of mechanical recovery of metals from electronic waste, including printed circuits, may be widely used in the future by waste management companies as well as metal production and processing companies. That is why, a well-known and easily applicable electrostatic separation (ES) method was used to recover metals from printed circuit boards. The grain class of 0.32 - 0.10 mm, obtained after grinding the boards, was fed to a separator. Feed and separation products were analyzed by means of ICP-AES, SEM/EDS and XRD. The concentrate yield obtained after electrostatic separation amounted to 32.3% of the feed. Its density was 11.1 g/cc. Out of the 91.44% elements identified in the concentrate, over 90% were metals. XRD, SEM observations and EDS analysis confirmed the presence of non-metallic materials in the concentrate. This relatively high content of impurities indicates the need to grind printed circuit board into grain classes smaller than 0.32-0.10 mm.

Key words: *electrostatic separation, metals recovery, PCB, SEM, XRD*

INTRODUCTION

The production of Waste Electrical and Electronic Equipment (WEEE) is growing at an alarming rate. In 2016, 44.7 million metric tonnes of WEEE were generated, but is expected to increase to 55 million metric tonnes by 2021 [5, 25]. People can process them, degrading the environment to a greater or lesser extent [24]. Effective management of WEEE has become a global problem, because in the event of improper management and recycling, they can have a significantly impact on the environment.

Considering environmental protection, depleting of metal deposits and economic benefit, environmentally friendly and high-efficiency methods of recovering metals from printed circuit boards (PCB) should be sought. Basically, the methods of recovering metals from PCB are divided into physical and chemical [15]. Since chemical methods usually have a negative impact on the environment, the authors of the study focused on one of the physical methods, i.e. electrostatic separation (ES) [15, 23, 30].

The aim of the article was to assess the efficiency of metal recovery from PCB using ES. The article contains the results of the tests on the recovery of metals from grinded PCB with a grain size of 0.1-0.32 mm, using an ES.

In order to obtain accurate test results and eliminate potential measurement errors, the following analysis methods were used: X-ray Powder Diffraction (XRD), Scanning Electron Microscopy (SEM) with the Energy Dispersive Spectroscopy (EDS) system and Inductively Coupled Plasma atomic emission spectroscopy (ICP-AES). As a result of the tests, non-metallic and metallic parts were separated from PCB.

LITERATURE REVIEW

The basic element of the construction of most WEEE are PCB which contain about 70% of non-metallic parts, such as fiberglass, epoxy resin, polyester, woven glass, as well as 30% of metallic parts [2]. It is difficult to determine the type and amount of metals in PCB. It can be estimated that a PCB contains about 16% Cu, 3% Fe, 3% Sn, 2% Pb, 1% Zn 0.05% Au, 0.03% Ag, 0.01% Pd and others metals such as Cr, Na, Cd, Mo, Ti, Co [26, 27].

In ES, grains placed in an electric field are separated as a result of differences in the ability to accumulate electric charges on grain surfaces [9]. The scheme of the electrostatic drum separator used in the study is shown in Fig. 1.

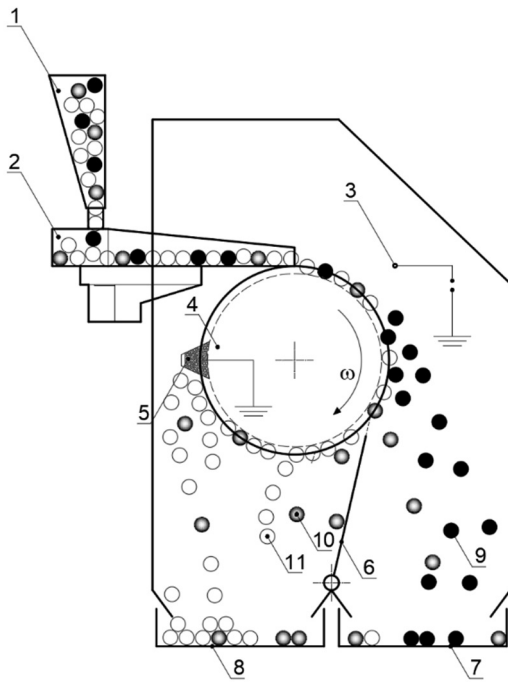


Fig. 1 Scheme of electrostatic drum separator:
 1 – feed container, 2 – vibrating feeder, 3 – electrode, 4 – drum, 5 – brush, 6 – partition, 7 – conductors container (concentrate), 8 – non-conductors container (waste), 9 – grains with good electrical conductivity, 10 – complex grains folded with metals and non-metals, 11 – grains with weak electrical conductivity

Placing the grain that has accumulated electric charge in the electric field induces the electric field force. The value of the resultant force depends on the value of the electric field force in which the grain is located. The surface electric charge is generated on the surface of any material, and depends on time and the type of material. Materials with high electrical conductivity (metals) quickly get rid of the accumulated electrical charge [9]. However, the electrostatic force is not the only one acting on the grain during the separation process. There are also (in the electrostatic drum separator): gravity force, image forces and centrifugal force. The resultant force acting on well-conductive grains is directed outwards, contrary to grains with low conductivity (non-metals) [1].

Consequently, the performance of the electrostatic drum separator is mainly dependent on the electrical conductivity of the grain, as well as the grain size and its density [9]. Electrical conductivity of selected metals, the values of electrical resistance of plastic materials, and their densities are shown in Table 1

Based on the experimental research carried out by the authors of the paper and the literature review, it can be concluded that purity of the concentrate is most impacted by the size of grain. According to Niu et al, Dascalescu et al. and Hogzhou, changes in parameters such as voltage and rotational speed do not significantly affect the purity of the concentrate [4, 18, 19]. That is why the choice of the method and device for crushing PCB is very important.

According to the authors, Kozłowski et al. and Franke and Suponik, grinding can be carried out in a knife mill [6, 11].

Table 1
Densities and electrical properties of selected metals and plastics

	Material		Density,	Electrical conductivity,
			g/cc	$10^6 \Omega^{-1} \text{m}^{-1}$
Metals	Gold	Au	19.30	44.35
	Lead	Pb	11.30	4.74
	Silver	Ag	10.50	61.84
	Copper	Cu	8.96	58.41
	Iron	Fe	7.87	10.13
	Silicone	Si	2.33	0.04
Plastics	Material		Density,	Electrical resistivity,
			g/cc	$10^6 \Omega \text{m}$
	Fiberglass reinforced plastics	FRP	1.80-2.00	10^6
	Polyesters	PET vs. PBT	1.31-1.39	$1-1.4 \times 10^7$
	Polypropylene	PP	0.90	10^9

Source: [3, 21, 28].

METHODS

Preparation for electrostatic separation

PCB from personal computers, hard disks, graphic cards and RAMs were used in this study. The way of preparing and grinding electronic waste is presented in the paper written by Franke and Suponik [6]. The knife mill manufactured by TESTCHEM was used to grind the PCB. The rotation speed of mill was 2815 rpm. The blades used were made of hardened steel and perforated sieve with a mesh size of 2 mm. Four grain classes were obtained from the grinded material: 2.00-0.56 mm, 0.56-0.32 mm, 0.32-0.10 mm and < 0.10 mm. The grain class of 0.32-0.10 mm was 40% of the total. This was a feed for the electrostatic separator. Results for the grain class of 0.56-0.32 were presented in the paper by Franke and Suponik [6]. So far, remaining grain classes have not been tested for the following reasons: in the grain class of 2.00-0.56 mm there were significant connections of metals with non-metals parts that reduce the purity of the concentrate, while for grains lower than 0.1 mm, the damage of electrode triggered by high risk of spark discharge [16] can occurred. In addition, the aggregation effect may appear for this class, which may also affect the efficiency of separation [13, 14]. However, despite this, it is planned that the efficiency of electrostatic separation will be tested for grain size < 0.1 mm.

Electrostatic separation

The drum separator used in the study allows to change three operating parameters. As a result of the experimental research, the following parameters were used: shaft rotation speed 100 rpm, electrical voltage at the electrode 17 kV and distance of the electrode from the shaft 0.03 m.

Product analysis

The feed and products obtained from ES were digested and the concentrations of the elements were measured with the JY 2000 spectrometer (by Yobin-Yvon) using the ICP-AES method. The source of induction was a plasma torch coupled with a frequency generator of 40.68 MHz. Furthermore in the feed, concentrate and waste phase composition have been determined on the basis of the X-ray diffraction measurements, performed with the Panalytical X'Pert Pro MPD diffractometer, utilizing filtered radiation of a copper-anode lamp ($\lambda_{K\alpha}$ 0.154 nm). The diffraction lines were recorded in the Bragg-Brentano geometry, using the step-scanning method by means of a PIXcell 3D detector on the diffracted beam axis, in the angle range from 20-95° [20] (1 step 0.05°, count time per step 120 s). The diffractograms obtained were analyzed with the use of Panalytical High Score Plus software with the PAN-ICSD database.

The morphology of the feed and products from ES, as well as the chemical composition in microareas, were analyzed by means of the Zeiss Supra 35 high resolution electron microscope, equipped with EDAX EDS chemical analysis system.

RESULTS AND DISCUSSION

As a result of ES, the grinded PCB with grain size of 0.32-0.10 mm were separated into concentrate and waste. The concentrate was about 1/3 of the mass of the tested sample (Table 2), what confirms the average metal content in PCB ranging from 20% to 40%, assessed by authors such as Kumar et al., Bizzo et al., Burat et al. and Wu et al. [8, 17, 26, 27]. The waste was 2/3 of the mass. A high concentrate density of about 11 g/cc indicates high separation efficiency, while waste density of 3 g/cc may indicate the penetration of metal parts into the waste. The analysis of the ferromagnetic content shows that the waste did not contain ferromagnetic parts, in contrast to the concentrate, which had the ferromagnetic content of 0.3% (see Table 2).

Table 2
The results of ES

Product	Density of product, g/cc	Yield of product, %	Content of ferromagnetics in product, %
Feed	5.4	-	0
Waste	3.0	67.7	0
Concentrate	11.1	32.3	0.3

The results of measurements carried out in the ICP-AES of the feed, concentrate and waste products are presented in Table 3.

Out of the 91.44% elements identified in the concentrate, over 90% were metals. Si and Br content was over 8%. They form a lead-barium borosilicate glass on PCB. This relatively high content of impurities indicates that PCB needs to be ground into grain classes smaller than 0.32-0.10 mm. In this way, metals would be free of impurities. These elements were probably mechanically bonded to metals.

Table 3
Elemental concentrations in the feed and in ES products: A – this study, B – study by Guo et al. for a similar grain class [10], “-” no data

Element	Content of the element [%] in					
	Feed		Concentrate		Waste	
	A	B	A	B	A	B
Al	3.33	1.51	1.89	2.63	0	0.93
Si	15.6	-	5.15	-	0.0989	-
K	0.0589	-	0.00980	-	0	-
Ca	8.99	-	1.11	-	0.0095	-
Mg	0.0045	-	0.00890	1.23	0.00055	0.28
Mn	0.0355	-	0.10	-	0	-
Fe	0.3821	1.38	0.93	3.74	0	0.19
Ni	0.185	0.28	0.85	0.75	0	0.039
Cu	19.5	27.08	59.70	72.81	1.22	3.99
Zn	0.25	0.79	1.09	2.12	0	0.11
Br	13.8	-	2.98	-	0.00055	-
Ag	0.1415	0.0019	0.4996	-	0	-
Au	0.0019	0.0069	0.0101	-	0	-
Sn	2.38	3.23	7.83	9.63	0.0045	0.01
Ba	2.2	-	1.27	-	0.0075	-
Pb	1.95	2.44	8.00	9.63	0	0.12
Totality based on this study (A)	68.81		91.44		1.34	
Totality based on study by Guo et al. (B)		36.72		99.99		5.65

An example of connection of metal parts with plastics is shown in Fig. 4, while Table 4 presents the results of the chemical analysis. On the other hand, the non-metallic elements could have penetrated into the concentrate as a result of imperfections in the separation process. This issue should be checked in further studies. A similar problem concerned waste. Over 1% of copper was found in this group of products. Probably, the reason for contamination by copper was the layered construction of the PCB. According to Tatariants et al. and LaDou, some very thin elements consists of several layers, and the segments responsible for connecting them together are often made of copper [12, 20]. It can be assumed that, if the PCB were grinded to smaller fractions, this element would not penetrate into non-metals.

Guo et al. [10] (see Table 3) received a cleaner concentrate from the ES of a similar grain class. But in their analyzes, they did not take into account such elements as Si, Ca, Br, Ba neither in feed nor in the product of ES.

The creation of a semiproduct chamber in the electrostatic separator can improve the efficiency of metal recovery. Metals mechanically bonded to plastics or glass can be found in this product. They could be ground again to separate metals from plastics. Then this product could be separated again.

The concentrate contained the following valuable metals: Cu, Pb, Sn, Al, Zn, Ni, Ag, Au. The amount of the metals identified depends on the date of production, the manufacturer or the quality of the PCB and the type of the components used [22]. As provided by Bizzo et al., over the years PCB have had various metal contents i.e. Cu 12-28%,

Al 1.7-7%, Pb 1-3%, Zn 0.08-2.7%, Ag 79-3300 ppm, Au 29-11200 ppm [27].

To determine the morphology of the feed and products obtained from the ES, SEM observations and chemical analysis in micro-regions, by means of energy-dispersive X-ray spectroscopy (EDS) were performed. Imaging of the tested samples using the backscatter electron detection technique (QBSD) (Fig. 2 and 3), allowed to investigate the morphology.

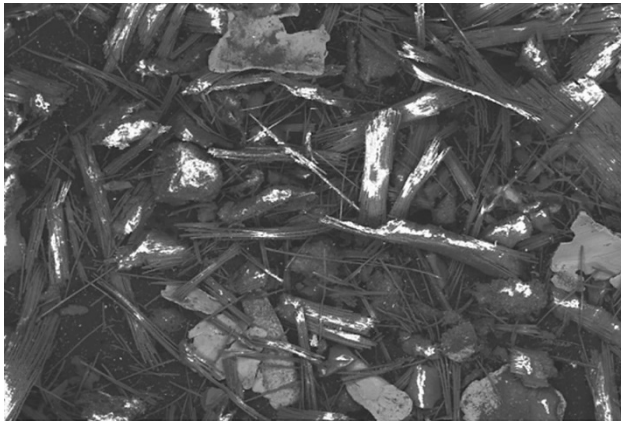


Fig. 2 Image of the feed (QBSD SEM)

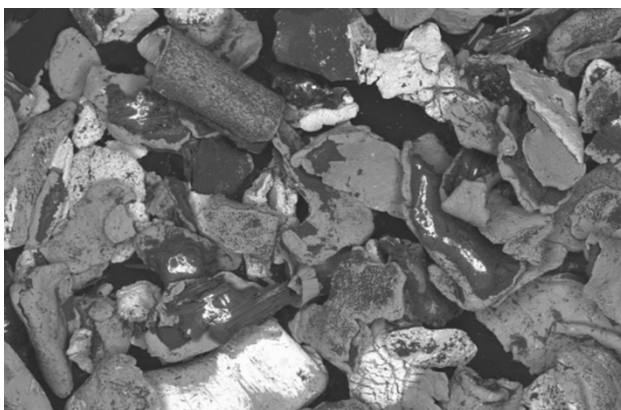


Fig. 3 Image of the concentrate from ES (QBSD SEM)

The contrast obtained in these pictures is a result of differences in the chemical composition. The areas containing elements with a high atomic number are clearly brighter compared to the areas consisting of lower Z-number elements. In the tested feed sample (Fig. 2), both metallic particles of various shapes and dimensions mostly in the range of 100 to 400 μm , as well as many fragments of non-metallic fibers and particles, were observed. In many cases, these non-metallic particles are bonded with metal, which may be due to the PCB production process, in which thin films of good electrical conductivity metals (mainly Cu and Sn, Au, Ag, Pt) are applied on a glass fiber and epoxy laminate [7, 29, 31]. This may create difficulties in the ES process, leading to "contamination" of the metallic product with non-metallic particles.

The SEM analysis of the concentrate (Fig. 3 and 4) showed the presence of mainly metal particles with a small amount of non-metallic materials, such as glass fiber, polymers, and ceramics, which were not separated from the metallic particles in the milling process. These metal particles with various geometry and dimensions approx. 300-400 μm (a few particles of the order of 800 μm were also observed) were characterized by different chemical composition, even within one particle, which was demonstrated by means of the chemical composition analysis in micro-areas (Fig. 4 and Tab. 4).

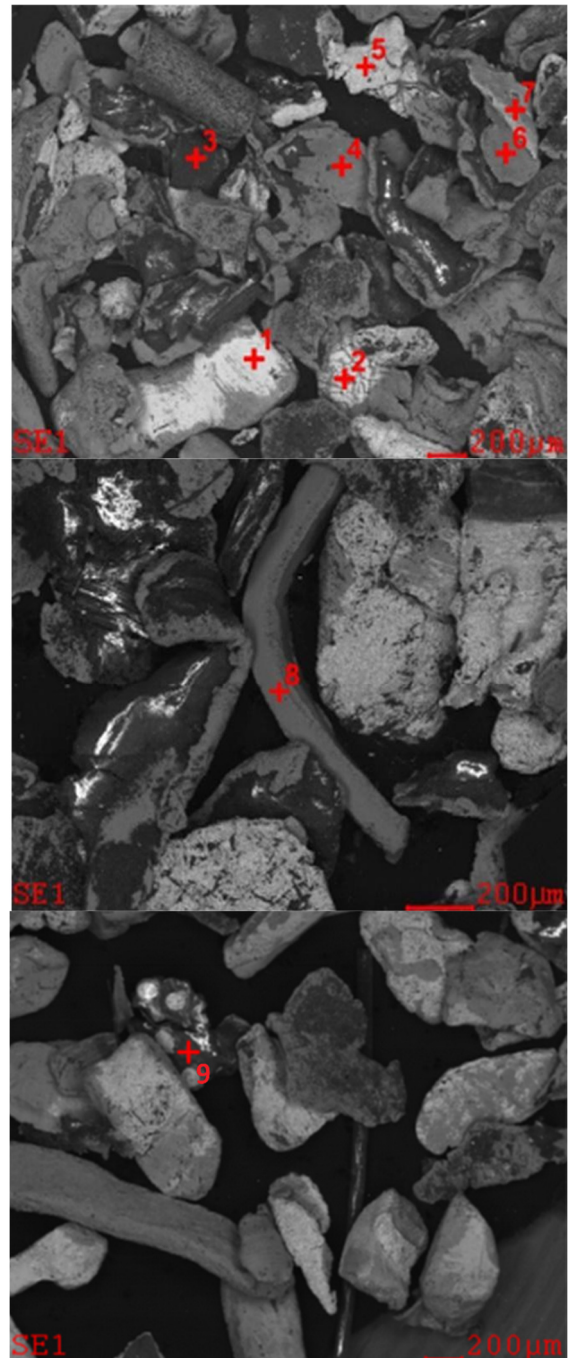


Fig. 4 Images of the concentrate obtained from ES with marked points of chemical microanalysis

Table 4
Results of chemical composition microanalysis for points shown in Figure 4

Element	Point of analysis/Concentration [% at.]								
	1	2	3	4	5	6	7	8	9
Cu	9.71	49.43	38.47	83.39	88.37	38.62	-	-	3.84
Sn	16.76	0.73	3.04	-	-	-	100	-	1.07
Ni	5.19	30.20	-	-	-	2.71	-	37.59	-
Au	68.35	2.65	-	-	-	-	-	-	-
O	-	5.25	30.41	11.66	-	25.23	-	-	38.19
Al	-	7.05	23.81	3.56	-	29.28	-	-	22.33
Si	-	2.69	4.27	1.39	-	0.7	-	2.24	26.55
Pb	-	2.01	-	-	11.63	-	-	-	-
Ti	-	-	-	-	-	1.56	-	-	0.38
P	-	-	-	-	-	0.81	-	-	-
K	-	-	-	-	-	0.5	-	-	-
Mo	-	-	-	-	-	-	-	0.72	1.28
Ag	-	-	-	-	-	-	-	1.45	-
Mn	-	-	-	-	-	-	-	0.67	-
Fe	-	-	-	-	-	-	-	57.35	-
Br	-	-	-	-	-	-	-	-	5.28

The results of the XRD (qualitative phase analysis) of the feed and concentrate and waste products are presented in Fig. 5. For the feed sample, diffraction lines from metallic phases (Cu, Sn, Pb, CuSn) and oxides phases SiO_2 and BaO were recorded. The same phases were indicated in the waste sample, while the intensity of lines obtained from metallic phases significantly decreased, which indicates a much lower volume share of these phases. It can be assumed, that these are mainly the residues of small metal fragments which, combined with larger non-metallic particles of PCB, got into the waste during the separation process. On the diffractogram obtained from the concentrate sample, only the diffraction lines from Cu, Sn, Pb, CuSn metallic phases were identified. However, the presence of other metallic phases in a lower volume share being under detection limit cannot be excluded, as well as with this method it is difficult to identify the small amounts of amorphous phases (polymers, glass).

CONCLUSION

As a result of the research analysis, it can be concluded that the products obtained from the ES were contaminated. Based on the ICP analysis, approximately 91% of metals were identified in the concentrate. These were Cu, in the largest amount (ca. 60%), and then Pb, Sn, Si, Br, Al, Ba, Ca, Zn and small amounts of Fe, Ni, Ag, Mn, Au, K and Mg. It can be assumed that the maximum of 9% of the mass was contaminated. The EDS analysis, as well as the ICP-AES, confirmed appearance of these elements: Cu, Sn, Ni, Au, Al, Si, Pb, K, Ag, Mn, Fe and Br. Quantitative analysis was difficult to perform for both methods. The authors used a larger amount of material in ICP than in EDS, in which only microscopic survey was carried out. The XRD analysis revealed that the concentrate contained mainly Cu, Sn, Pb, CuSn metallic phases, as well as small amounts of oxides phases such as SiO_2 and BaO.

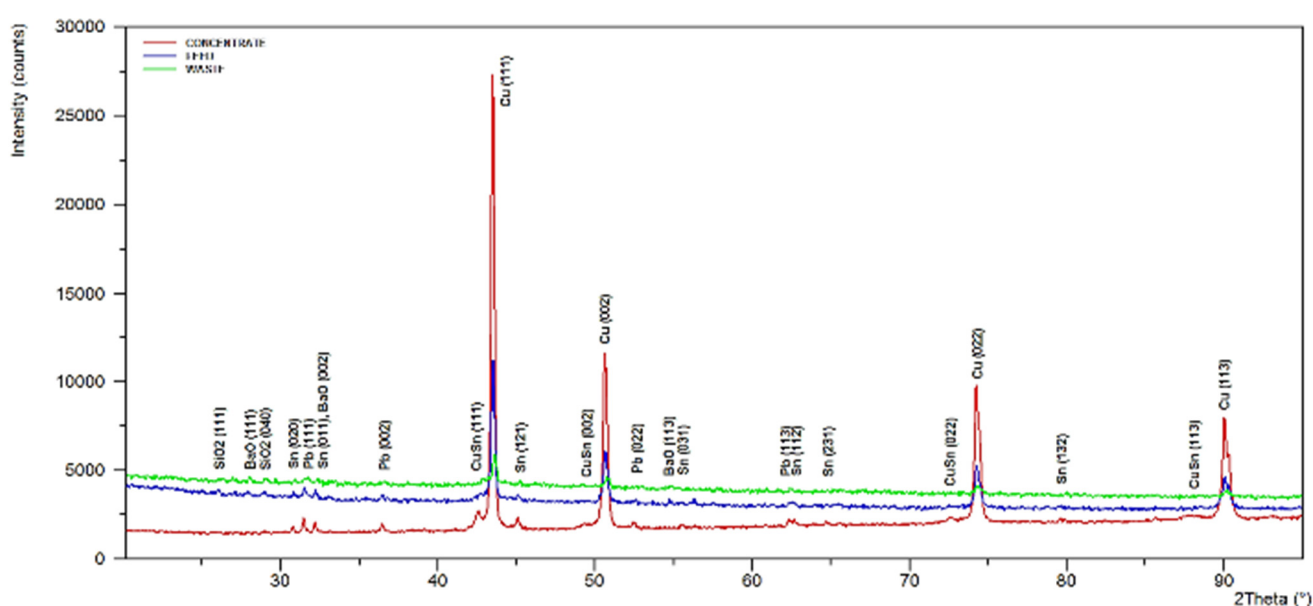


Fig. 5 X-ray diffraction patterns of feed (blue line), concentrate (red line) and waste (green line)

The SEM analysis of the concentrate showed the presence of mainly metal particles with a small amount of non-metallic materials, such as glass fiber, polymers, and ceramics, which were not separated from the metallic particles in the milling process. These metal particles, with various geometry and dimensions, were characterized by different chemical compositions, even within a single particle.

The analyzes of the waste indicated that the small amounts of metallic phases were in the waste sample. They were mainly Cu (ca. 1%) but also Ca, Mg, Sn, Ba in smaller quantities. Presumably, they were mainly the residues of small metal fragments which, combined with larger non-metallic particles of PCB, got into the waste during the separation process.

In conclusion, the results of the research confirmed that the efficiency of metal recovery for the grain class of 0.32-0.10 mm was still insufficient. It is reasonable to optimize the separation process for significantly smaller grains in subsequent works. Consideration should also be given to extending the separator with an additional receiver for semi products, i.e. for grains containing both metals and non-metals.

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