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ORIGINAL PAPER

Sampling and digestion of waste mobile phones printed circuit boards for Cu, Pb, Ni, and Zn determination

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Abstract

Sampling is the most important procedure in arriving at the value of an electronic scrap. Inappropriate sampling makes other procedures, including assaying, valueless, as the sample must really represent the electronic scrap from which it was taken. The aim of this study was to develop the procedure to investigate the composition of heterogeneous printed circuit boards (PCBs) material and to show its applicability as well as its related limitations. The contents of Cu, Pb, Ni, and Zn in PCBs from wasted mobile phones were determined. Mixtures of aqua regia, hydrogen peroxide and hydrofluoric acid were applied to sample digestion. Open and closed vessel wet digestion with or without heating and microwave-assisted one was realized. For these purposes different sample amounts (0.2, 0.4, and 0.5 g) were applied and on the basis of calculated relative standard deviation values (RSD), the precision of repeated the most effective digestion was evaluated. The microwave-assisted wet digestion and mixture of aqua regia and hydrogen peroxide was found as the most effective one of the compared mixtures and applied conditions. A sample amount of 0.5 g was found as the most suitable. RSD values confirm the importance of electronic waste material sample preparation.

Keywords Waste printed circuit boards · Material processing · Metals · Acid digestion · Microwave digestion

Introduction

In recent years growing attention is given to possibilities of reutilization of electronic waste material (Cui and Zhang 2008). Due to its composition, electronic waste is a promising source of metals with high industrial importance and rare elements (Zhan et al. 2015), respectively. On the other hand, effort to reuse the material itself can be also seen (Xu et al. 2014).

PCBs are the basic component of all electrical and electronic equipment in which both valuable and potentially hazardous materials and metals are concentrated. As part of mobile phones, PCBs from discarded equipment belong to the category No. 3 ''IT and telecommunications equipment'' (Directive 2002/96/EC, Annex 1). The EU calls for an increased recycling rate for this type of waste, for both, environmental protection and resource conservation (Osibanjo and Nnorom 2008). This is due to the growing demand for metals. The electrical and electronic equipment industry is a large consumer of especially precious and less common metals, but also some basic metals (Oguchi et al. 2012). Along with this there is growing consumption of mobile phones [they have only a 2-year life span (Robinson 2009)], or other electronic household products. Many of these are discarded before the loss of functionality.

The research on the treatment of mobile phones has focused on the characterization of different types of mobile phones to identify their material composition, valuable metal contents as well as impurity levels (Chancerel and Rotter 2009; Palmieri et al. 2014). Mobile phones contain toxic elements, such as lead, mercury, chromium, nickel, beryllium, antimony, and arsenic as well as valuable metals, such as gold, silver, palladium and platinum. The material composition is very variable, depending on the type of mobile phone. Another determining factor is the year of production. Material composition of mobile phones is the reaction of producers to the EU´s legislative requirements; e.g., the content of lead is considerably reduced (Holgersson et al. 2017) and the same situation is elsewhere in the world (Chen et al. 2016). From an analogy between natural resource development

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and metal recovery from waste electrical and electronic equipment (WEEE), it was pointed out that not only metal concentration but also the total amount of metals contained in WEEE should be considered in the characterization of WEEE as secondary metal resources (Oguchi et al. 2011). Similarly, the total amount of contained metals should also be considered when discussing the importance of managing toxic metals contained in WEEE (Oguchi et al. 2013).

The method of sampling and sample processing directly and firstly affects the accuracy and precision of the material composition survey and the related determination of the precious and non-ferrous metals content of the PCBs material. One of the theories on sampling for chemical analysis, that takes into account both the technical and statistical aspects of sampling, has been developed by Pierre Gy. Pierre Gy's theory of sampling is currently a widely applied and taught theory in the field of mining and minerals engineering. Gy's theory of sampling deals with many issues related to sampling and a main part of Gy's theory consists of the prediction, estimation or minimization of the variance of the ''Fundamental Sampling Error'', denoted here by Var(FSE). Var(FSE) can be regarded as the relative variance of the sampling error that is obtained under thorough mixing of the population prior to sampling.

$$\mathrm{Var}_{\mathrm{Gy}}(\mathrm{FSE}) = CD^3 / M_{\mathrm{sample}}, \tag{1}$$

where $\mathrm{Var}_{\mathrm{Gy}}(\mathrm{FSE})$ is the prediction made by Gy's theory of sampling for Var(FSE), C is sampling constant for the material being sampled (g $\mathrm{cm}^{-3)}$, D is typical particle size (cm), and M_{sample} is the mass (or weight) of a sample (g) (Gy 2004; Minkkinen 2004; Geelhoed 2011).

Due to the diversity, complexity and often varying metal content, the sampling and assaying processes, as the critical steps in the proper material assessment are being continuously innovated by recyclers in close collaboration with the suppliers. However, most of the technologies applied are developed "in-house".

After their collection, the appliances are usually pretreated with unit processes such as dismantling, crushing, sieving, magnetic, eddy current, sensor-based and density separation to produce fractions like PCBs, plastics, and ferrous metals (Cui and Forssberg 2003; Luda 2011). The problem comes with determination of interesting metal contents because there is no unique procedure for PCBs treatment either for recycling or for chemical analysis. Various approaches can be found, such as single acid digestion with aqua regia, where material digestion runs for duration of, e.g., 1 h (Zhou et al. 2016), 24 h (Yamane et al. 2011) or 72 h (Maragkos et al. 2013). We can also meet with combination of HNO_3 and H_2O_2 (Chen et al. 2016), aqua regia, alkaline solution or HCl digestion (Oguchi et al. 2013), hot aqua regia, HNO_3 + HF digestion followed by microwave heating and Na_2O_2 fusion in combination with HCl dissolution of residues (Ogunniyi et al. 2009) or digestion in two steps with conc. HNO_3, HBF_4 solution and H_2O_2 followed by conc. HCl (Ernst et al. 2003). Besides the single or few steps digestion, pyrolysis is another procedure used for PCBs material disruption. Temperatures of 800 or 850 °C are applied and for residues treatment acid digestion alone or in combination with ashing is used (Hall and Williams 2007, Vats and Singh 2015). As mentioned, there is still some solid residue, in many cases not negligible amount, with potential content of determined metals (Santos et al. 2010, 2011; Nnorom and Osibanjo 2009) as electronic waste is a heterogeneous mixture of metals, plastics, glass, and ceramics and it is difficult to attain complete material digestion.

Since there is no standard reference material available for printed circuit boards and the composition is variable, it is very difficult to verify the determined metal content in the material. In this work, different techniques for PCBs digestion have been explored to get, as close as possible, to the complete digestion of the material, in order to obtain reliable concentration data that can be used to recover valuable metals.

Experimental

Sampling procedure of PCBs of mobile phones

Processing material consisted of discarded mobile phones with different years of production and from different manufacturers. About 80 kg of collected mobile phones were used for further processing. Batteries and plastic covers from mobile phones were hand dismantled. The remaining printed circuit boards of mobile phones (Fig. 1) were used for the experiment. Their total weight was 2 kg. The crushing operation was carried out in the crusher hammer (type SK 600) to the fraction ($d \leq 8$ mm) (Fig. 2). Crushed printed circuit boards were homogenized. A sample of 1 kg was obtained by hand quartering, which was then divided three times by

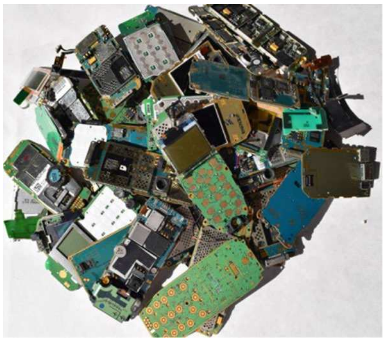

Fig. 1 PCBs of mobile phones

Fig. 2 Crushed PCBs of mobile phones—8 mm

the mechanical divider. The magnetic separation was carried out to remove the magnetic fraction from the sample. Eight representative samples were milled separately in a hammer mill (type VEBER SPO) to the fraction ($d \leq 1$ mm). The obtained product, the fraction ($d \leq 1$ mm) was analyzed. 1 g of the test sample was taken from each sample for the chemical analysis. The scheme of representative sample preparation is shown in Fig. 3.

Particle-size analysis

The sieving analysis was carried out to evaluate the size distribution. 100 g of sample obtained through quartering of crushed printed circuit board was used for particle-size analysis by a vibration sieve shaker using 1.00; 0.71; 0.40; 0.20; 0.125; 0.063 mm openings. The results are expressed by cumulative weight percentage. The size distribution of PCBs of mobile phones after grinding is shown in Fig. 4. In order to determine the particle morphology of treated mobile phones, the sample was subjected to observation under a digital microscope Dino-Lite ProAM413T. The magnification used during the observation was 20. As can be seen in Fig. 5, the sample ($d \leq 1$ mm) was significantly heterogeneous.

Digestion procedures

The samples were weighed on a METTLER AE 240 analytical balance. Analytical grade purity commercial nitric acid (67–69%), hydrochloric acid (36%), hydrofluoric acid (38–40%) and hydrogen peroxide (30%) was used for sample digestion. For microwave-assisted wet digestion,

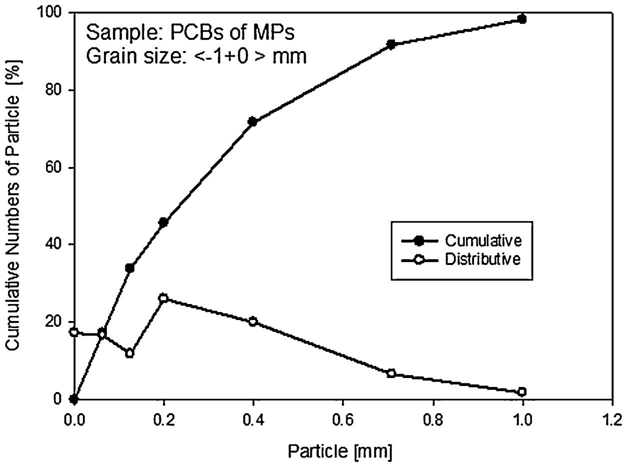

Fig. 4 Granulometric distribution ($d \leq 1$ mm)

Fig. 3 Scheme of representative sample preparation (Laubertová et al. 2016)

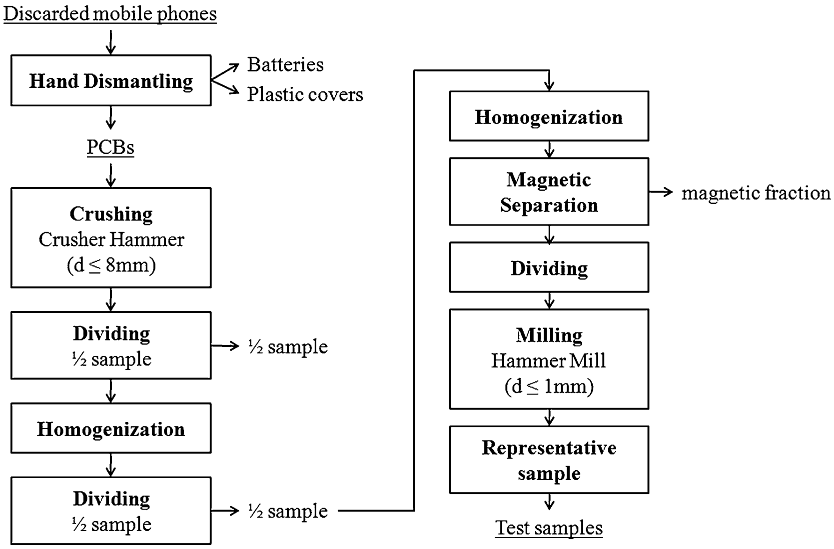

Fig. 5 Particle fraction ($d \leq 1$ mm) (20 × zoom)

the commercial microwave-digestion system ETHOS One (Milestone srl., Italy) was used. Concentrations of Cu, Pb, Zn, and Ni were determined in solutions after sample digestion in tested mixtures by high-resolution continuum source atomic absorption spectrometry (HR CS AAS) using contrAA 700 Spectrometer (Analytik Jena, Germany).

Open acid digestion at laboratory temperature (conditions 1)

At first, three samples of 0.4 g were weighed. Then open acid digestion at laboratory temperature was realized with three reagent mixtures: A. 12 cm^3 HCl + 4 cm^3 HNO_3 + 4 cm^3 H_2O_2; B. 12 cm^3 HCl + 4 cm^3 HNO_3; C. 12 cm^3 HCl + 4 cm^3 HNO_3 + 4 cm^3 HF. After 24-h digestion, mixtures were filtered and in final solutions the contents of Cu, Pb, Zn, and Ni were determined.

Open acid digestion with heating (conditions 2)

Open acid digestion with heating of two mixtures containing 0.4 g of sample and appropriate volume of reagent mixtures (A. 12 cm^3 HCl + 4 cm^3 HNO_3 + 4 cm^3 H_2O_2; B. 12 cm^3 HCl + 4 cm^3 HNO_3) in laboratory beaker was realized. After complete evaporation of solutions, 10 cm^3 of 1% HCl was added to the solid residue. Finally, after filtration the obtained solutions were analyzed for Cu, Pb, Zn, and Ni content.

Closed acid digestion in closed vessels at laboratory temperature (conditions 3)

Closed acid digestion in closed vessels for 0.4 g of sample and appropriate volume of reagent mixtures (A. 12 cm^3 HCl + 4 cm^3 HNO_3 + 4 cm^3 H_2O_2; B. 12 cm^3 HCl + 4 cm^3 HNO_3; C. 12 cm^3 HCl + 4 cm^3 HNO_3 + 4 cm^3 HF) was carried out at laboratory temperature for 24 h. After filtration of the obtained solutions, determination of chosen elements content in final solutions was performed.

Microwave-assisted wet digestion (conditions 4)

For microwave-assisted wet digestion 0.2 g of sample was taken and appropriate amounts of reagents was added (A. 6 cm^3 HCl + 2 cm^3 HNO_3 + 2 cm^3 H_2O_2; B. 6 cm^3 HCl + 2 cm^3 HNO_3; C. 6 cm^3 HCl + 2 cm^3 HNO_3 + 2 cm^3 HF). Digestion was realized in a microwave device programmed to 220 °C, 45 bar pressure and 1000 W power with 20 min digestion time. After 20 min of cooling step, when the vessels became colder the solutions were filtered to obtain final solutions for elements content determination.

Microwave-assisted wet digestion of different sample amounts

To find out the most suitable amount of sample for microwave-assisted wet digestion with aqua regia and hydrogen peroxide, three different sample amounts (0.2, 0.4, and 0.5 g) were digested with appropriate amounts of reagent mixtures (for 0.2 g: 6 cm^3 HCl + 2 cm^3 HNO_3 + 2 cm^3 H_2O_2, for 0.4 g: 12 cm^3 HCl + 4 cm^3 HNO_3 + 4 cm^3 H_2O_2, for 0.5 g: 15 cm^3 HCl + 5 cm^3 HNO_3 + 5 cm^3 H_2O_2) and the obtained solutions were analyzed for elements content determination.

Evaluation of repeated microwave-assisted wet digestion precision

According to previous results, 0.5 g of sample was found out as the most suitable amount for the microwave-assisted wet digestion with aqua regia and hydrogen peroxide. To evaluate precision of applied procedure, 10 repeated digestions were realized and subsequently RSD values were calculated.

Results and discussion

With the aim of evaluating the efficiency of different sample digestion processes, their verification by total content determination of selected metals was realized. Digestion mixture "C" was not applied for open acid digestion with heating (conditions 2) due to the technical limitations. The results of these experiments are shown in Table 1.

Open vessel digestion without heating (conditions 1) provided the most acceptable results for digestion using mixture of aqua regia and hydrogen peroxide (A). In all four cases of determined elements, the less suitable with less effectiveness seems to be aqua regia itself (B). The next step was the comparison of different reagent mixtures digestion efficiency under heating of digested sample (conditions 2). It was found that the efficiency of aqua regia (B) grows with

Table 1 Metal contents after classical and microwave-assisted wet digestion

Digestion mixture	Digestion conditions	Metal content/mg kg^{-1}			
		Cu	Ni	Pb	Zn
A (aqua regia + H_2O_2)	1	100585	257.500	12 817.5	12562.5
	2	79425.0	130.750	11315.0	12111.3
	3	105820	717.000	13824.5	19511.5
	4	119525	859.000	15355.0	22595.0
B (aqua regia)	1	89300.0	219.250	9722.50	11603.8
	2	104375	123.750	14962.5	14060.0
	3	92900.0	256.750	10640.0	12681.3
	4	93490.0	782.500	14587.5	18517.5
C (aqua regia + HF)	1	93100.0	266.000	14977.5	14900.0
	3	95625.0	223.000	14335.0	15712.5
	4	92375.0	561.000	10045.0	13266.0

higher temperature. On the other hand, heating the mixture of sample together with aqua regia and hydrogen peroxide (A) had the opposite impact, efficiency decreased. The only exception was observed in case of nickel, where can be seen that higher digestion efficiency belongs to aqua regia and hydrogen peroxide mixture (A). The closing of system (conditions 3) had positive impact on digestion efficiency. The most noticeable differences were observed in the case of mixture of aqua regia and hydrogen peroxide (A). In the closed system it was possible to notice a higher digestion effectivity. In the case of Cu, Zn, and Ni the most effective reagent mixture was “A” with the exception of Pb with the best results of determined concentration for reagent mixture “C” even though difference was minimum. In three of four determined elements it was confirmed that application of aqua regia itself for sample digestion without heating of reagent mixture and sample is least efficient. In the next step of experiments, the microwave-assisted wet digestion (conditions 4) was realized to compare each reagent mixture efficiency under that new conditions. It was found out that the most effective for sample digestion is mixture of aqua regia in combination with hydrogen peroxide (A). At the same time, it was confirmed that PCB material digestion using given the reagent mixture and closed system is more effective in comparison with open system. It was also found out that the less appropriate reagent mixture for digested type of sample under used conditions was mixture of aqua regia and hydrofluoric acid (C).

When results for sample digestion without the use of microwave heating (conditions 1–3) are compared with the one for microwave-assisted wet digestion (conditions 4), higher efficiency of the latter can be seen in the case of all determined elements (Fig. 6).

For the most effective digestion conditions and digestion mixture, the comparison of two different fractions of material (< 1 mm and < 8 mm) was realized. 0.2 g sample was used for microwave-assisted wet digestion with aqua regia and hydrogen peroxide. Comparison (Fig. 7) shows that double content of Cu and six times higher content of Ni was found for the rougher fraction. On the other hand, Pb content is ten times lower in comparison with the finer fraction. In the case of Zn, differences can be also observed, but they are not so significant. Finally, this comparison and the observed differences confirm the strong impact of grain size on metal determination, especially in the case of PCBs with such a heterogeneous character of matter.

Due to ensuring reliability of the results, with respect to the high heterogeneity of the material, three different sample amounts were tested, whereby the technical capabilities of microwave furnace vessels were taken into account. Obtained concentration values were calculated for 1 g of analyzed material. The results (Fig. 8) show that increasing amount of sample does not have always a direct effect on the metal concentration. Direct dependence can be observed for Cu and Ni. Zn has a markedly opposite trend, probably caused by material heterogeneity. In case of Pb no trend was observed, that can be considered as strong impact of PCBs material in the fraction < 1 mm. However, from the point of view of ensuring a reliable determination of the amount of metal in the sample, in the highest weight of material, the amount of metals is always the highest, with the exception of Zn.

To assess the suitability of the sample material in terms of sample granularity and to verify the suitability of the applied digestion procedure, metals content determination was carried out in ten repeated microwave-assisted wet digestions of 0.5 g sample (fraction < 1 mm) with aqua regia and hydrogen peroxide. The results were processed by QC Expert 2.5™ software (Kupka 2002) in accordance with (Meloun and Militký 1998), whereby the obtained outlier values were excluded. A set of purified results was used to calculate average metal concentration c_{average}, standard deviation values s and RSD values for each determined metal (Table 2.). Except for Cu, RSD values were higher than 10%,

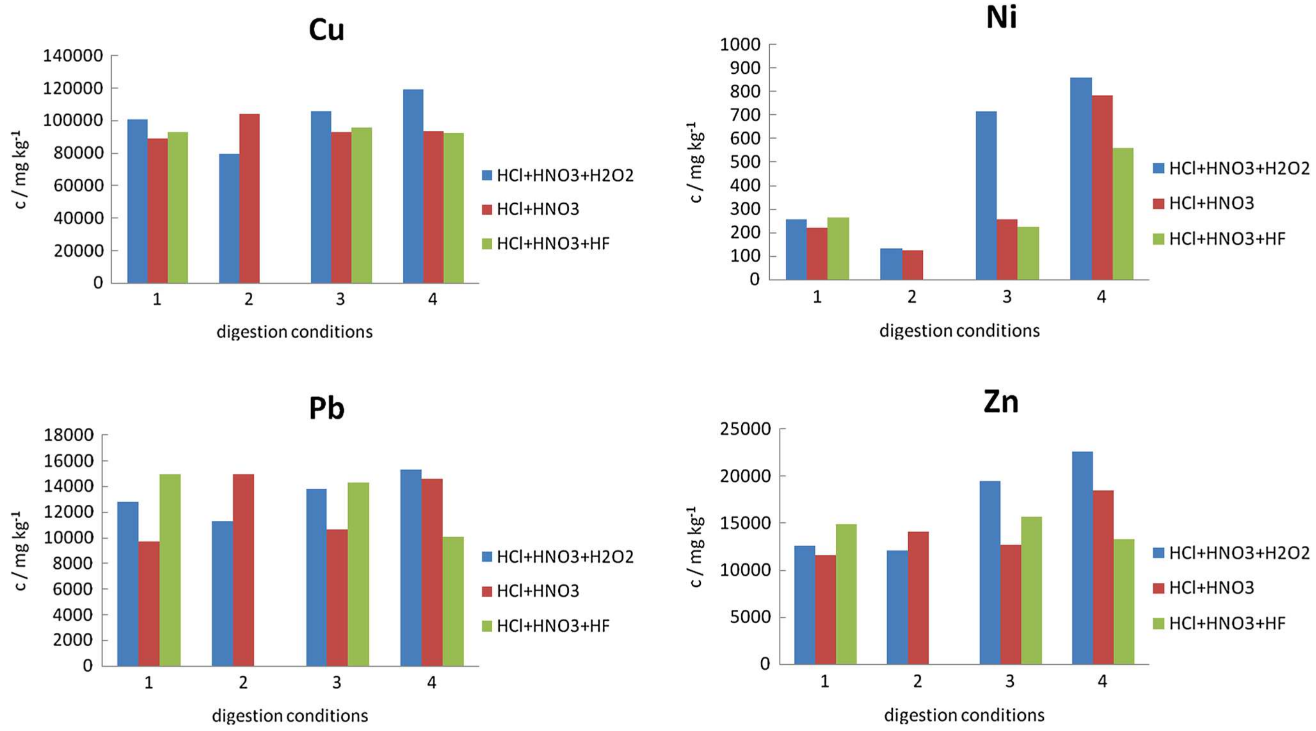

Fig. 6 The influence of digestion mixture and conditions on the determined metal content

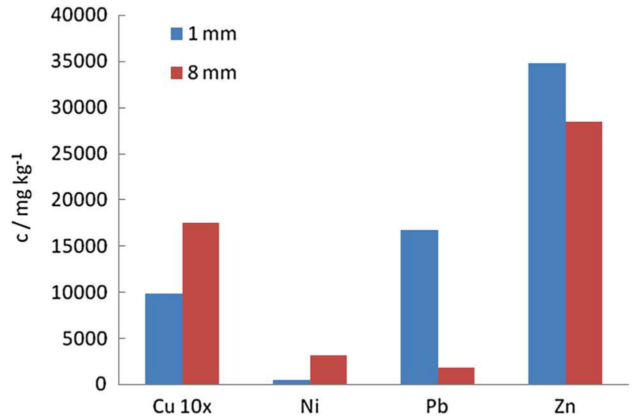

Fig. 7 Metal contents of two different fractions of PCBs

confirming the insufficient fineness of the digested sample and its heterogeneous character, which is not a good basis for objective determination of the material components.

Conclusions

In order to ensure complete electronic waste material digestion and to determine metals content with appropriate accuracy, the comparison of different reagent mixtures for waste material digestion was realized. Three different types of reagent mixtures were compared, namely aqua regia, aqua regia in combination with hydrogen peroxide and aqua regia in combination with hydrofluoric acid. It was found out that the most effective mixture for PCBs material digestion is aqua regia in combination with hydrogen peroxide (the digestion mixture A). For all determined metals, the highest concentration values were measured for this digestion mixture. On the other hand, the digestion mixture of aqua regia with hydrofluoric acid (the digestion

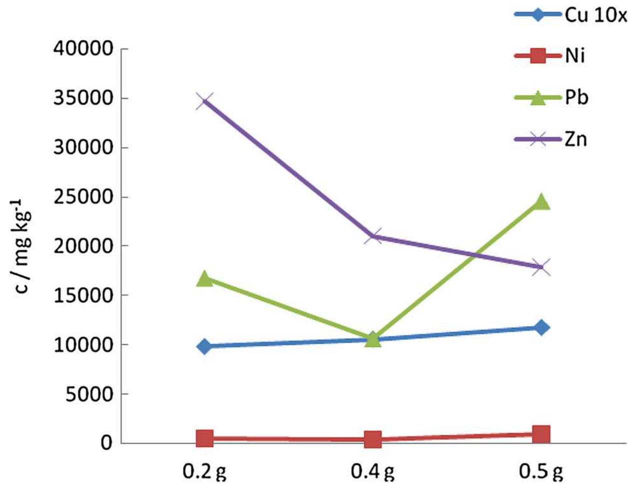

Fig. 8 The distribution of metals depending on the sample amount

Table 2 Evaluation of repeated microwave-assisted wet digestions precision

	Cu	Ni	Pb	Zn
Number of data	10	10	10	10
Number of outliers	1	2	1	2
c_{average}/mg kg^{-1}	138622	804.125	29666.7	14697.0
s	11872.26	105.05	5905.63	2016.16
RSD/ %	8.56	13.06	19.91	13.72

$c_{average}$, average metal concentration of repeated microwave-assisted wet digestions

mixture C) proved to be the least effective. In terms of conditions when the digestion mixture A and B (aqua regia with hydrogen peroxide vs. aqua regia) were compared, maximum metal content values were determined after microwave-assisted wet digestion (conditions 4) for all metals. Open acid digestion with heating (conditions 1) has proven unsuitable because of the lowest contents. The most significant differences in the efficiency of digestion can be observed in case of Cu, element with the highest content. With decreased content of the element in the PCBs the differences are smaller. The observed differences of determined metal contents depending on the digestion mixtures are related to the different forms of metal bonding in heterogeneous material of PCBs. The comparison of metal contents in two different fractions of material (< 1 and < 8 mm) showed strong grain size impact on metal content determination. Rougher fraction contained higher content of Cu and Ni in comparison with finer fraction, while Pb and Zn content in rougher fraction was lower. In case of Pb the differences observed are the most significant. Next, appropriate amount of sample was searched. The amounts of 0.2, 0.4, and 0.5 g were compared and the last one was found out as the most suitable. Finally, the values of relative standard deviations were evaluated in order to verify precision of digestion procedure. The obtained RSD values point to the insufficient homogeneity of digested material, in case of 1 mm fraction, in such a complicated type of sample as waste of electronic device is. The presented experiments are a pilot and the results obtained may serve as a guidance in further development of a suitable digestion method of printed circuit boards material for metal content determination with appropriate accuracy. Our aim was to provide useful information that can help in novel method development, which would be generally applicable in PCBs recycling and could help recyclers in solving problems connected with recovery of valuable metals from this kind of waste material.

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