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Feasibilities for hydrometallurgical recovery of precious metals from waste printed circuit boards in Lithuania

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Given the current trends in the WPCBs generation and precious metal value in the market, it can be concluded that recovery of precious metals from WPCBs is beneficial both economically and environmentally. Leaching by thiourea demonstrated the feasibility of Au, Cu, Ag and Pd recovery. Also the importance of pre-treatment was confirmed. The metal leaching rate for preliminarily cut and thermally treated WPCBs was 18–100% higher in comparison with that of untreated WPCBs. It can be explained by the removal of a protective soldermask layer, also by partial change of the mass and chemical structure of the WPCB matrix. After the thermal treatment, WPCBs become more crumble, they can decrease consumption of reagents and facilitate cutting. Also it was confirmed that the metal content in the old (age >20 years) WPCBs is significantly higher than in the currently generated WPCBs what is caused by the development of PCB production technology.

Keywords: leaching efficiency, recovery limits, precious metals recovery, thiourea leaching, waste printed circuit boards

INTRODUCTION

Printed circuit boards (PCBs) (Fig. 1) are an important component of electrical and electronic equipment (EEE), sales and production of which increase every year due to the constantly growing demand for EEE, therefore it can be assumed that amounts of WPCBs increase respectively. According to Ghosh et al. (2015), the global PCB production trend remains relative steady in the period of 1980–2016 – from ~7000 to ~75000 millions of USD [1].

Fast-paced development of industry with its huge metal consumption has started to give certain concerns to the global community since metals are non-renewable and finite resources which can eventually become depleted and scarce. For example, only automotive catalyst production consumes more than 50% of worldwide mined platinum (Pt), lead (Pb) and rhodium (Rh). About 3% of gold (Au) and silver (Ag), 13% of palladium (Pd) and 15% of cobalt (Co) are used for mobile phones and personal computer production. Such consumption causes a need for recovery and secondary use of these metals [2, 3]. Thus metal recovery, especially precious metals recovery, is a driving factor for WPCBs recycling. It is assumed that 1 t of this waste contains 300–500 g of potentially extractable Au and other precious metals. Notably, in the same time only about 5 g of Au can be extracted from 1 t of ore [4]. For this reason, recovery of metals from WPCBs is attractive both economically and environmentally [5].

Normally PCB consists of the following components: composite material formed from epoxy resin and woven fiberglass layers, conductive copper tracks, a soldermask (a thin layer of a lacquer-like polymer with considerable chemical and thermal resistance); also PCB can contain nickel (Ni), iron (Fe), aluminum (Al) and a certain amount of precious metals such as Au and Ag. Tin (Sn)-lead (Pb) solder is usually used to attach mounted components (capacitors, resistors, etc.) to PCB [5]. In regard to metals inside PCB, approximately 10–20% of PCB weight is Cu that forms electrical connections between different components of PCB, and Pb-Sn solder alloys usually contribute to 4–6% of PCB weight. Mounted components may contain a variety

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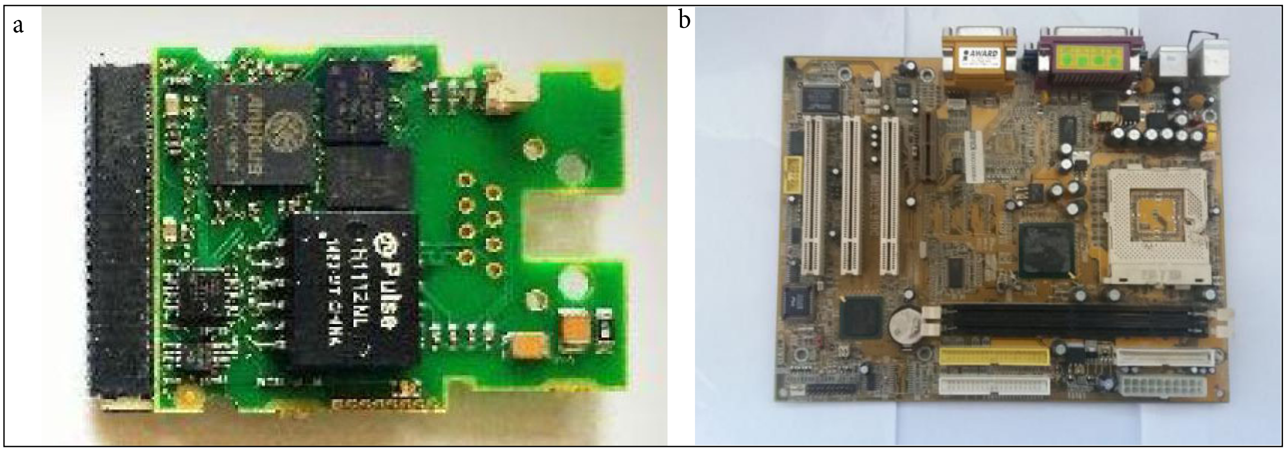

Fig. 1. Waste Printed Circuit Board (WPCB) sample: <1 year old (a); >20 years old (b)

of metals, including gallium(Ga), indium(In), titanium (Ti), silicon (Si), germanium (Ge), arsenic (As), antimony (Sb), selenium (Se), tellurium (Te), tantalum (Ta), etc. Pt can commonly be found in transducer switches or sensors [1].

WEEE can be recycled both by manual dismantling and by implementation of the more advanced mechanical, chemical and thermal treatment technologies. These technologies aim towards rational combining of economic profitability and environment-friendly operation [3]. WPCB recycling can be divided into the two main stages – the pretreatment stage for preparation of waste and the post-treatment stage for extraction of target materials. The pre-treatment can include dismantling (with removal of hazardous components like batteries, capacitors, etc. from the recycling process) [1, 6, 7] and shreddingx[1, 8].

Post-treatment methods are divided into physical (magnetic, electric conductivity, gravitational) [1, 8–13] and chemical (pyrometallurgical [1, 5], biometallurgical [13–15], hydrometallurgical) methods. Although being effective, pyrometallurgical methods require a substantial amount of investment; also their drawbacks are emissions of toxic dioxins, furans, bigger energy consumption and lower selectivity in comparison to hydrometallurgy [16, 17]. Biometallurgy is successfully used for minerals refining (recovery), however, biometallurgical technologies have not yet reached a sufficient advance regarding precious metal recovery from WPCBs [18]. Therefore, hydrometallurgical recovery of metals became attractive from an economic and environmental point of view.

Since precious metals under normal conditions are not reactive, high oxidation potential is needed for hydrometallurgical recovery. In order to improve the selectivity for precious metals recovery it is necessary to minimize the content of impurities and remove other non-precious metals. For this purpose, WPCBs are exposed to acids for non-precious metals leaching. As it was mentioned above, Cu contributes to a significant share of PCB weight therefore Cu recovery is prioritized above the extraction of other non-precious metals. Leaching of Cu by sulfuric acid with oxidizers (oxygen, iron chloride, dichromate, chlorine or hydrogen peroxide) was investigated in detail by many researchers. It was established that hydrogen peroxide has a high oxidation potential, therefore it is a suitable oxidation agent in the leaching process. More than 95% leaching efficiency for Cu can be achieved by using a sulphuric acid and hydrogen peroxide mixture [1, 19, 20].

Secondly, Au and Ag leaching is usually performed after Cu removal. Cyanide, aqua regia, thiosulfate, thiourea and other leaching agents can be used for this procedure. Au can be dissolved by the means of aqua regia which is traditionally used for this purpose, however, this solvent is a strong oxidizer and may cause corrosion of reactor equipment, therefore it is used only in laboratories. In addition, waste acid generates difficult to treat effluents [19]. During the past century cyanide was used for precious metals leaching from ore due to its good efficiency and relatively low cost. However, in this case effluents are highly contaminated [1]. Thiosulfate is one of the available alternatives for leaching of precious metals. Thiosulfate provides >95% Au dissolution, but one of the largest disadvantage is a high consumption of reagents, also a quite low reaction rate [1].

Recently, much attention was paid to thiourea leaching due to the lower cost, higher reaction rate, lower environmental impact and reduced toxicity compared to precious metals leaching by cyanide [19]. Thiourea forms a cationic complex with Au in the acid media and can dissolve up to 99% of Au [1]. It is important to mention that thiourea leaching requires the presence of an oxidizer (like hydrogen peroxide, oxygen, ozone, ferric ions, etc.) in the media in order to achieve favourable reaction conditions. Ferric ions (Fe^{3+}) are the most effective oxidizer for this type of process according to the available research data [20]. Jing-Ying et al. (2012) investigated the influence of the set of parameters on thiourea leaching of Au and Ag from WPCBs extracted from mobile phones. 90% Au and 50% Ag dissolution rate was achieved with 24 g/l thiourea and 0.6% Fe^{3+} concentrations at pH 1, temperature 298 K and reaction time 2 h [1]. Birloaga and Veglio (2016) achieved 69% Au recovery rate by thiourea leaching after removing 75% of Cu. During the experiment a solution of 20 g/l thiourea, 6 g/l Fe^{3+} and 10 g/l sulfuric acid at 600 rpm mixing was used [20].

Given these points, the goal of this article was to select and prove the most environment-friendly, effective and safe method for Cu, Ag, Au and Pd recovery from WPCBs as well as to estimate metal recovery feasibility from WPCBs of different age in Lithuania.

EXPERIMENTAL

Initial treatment of samples

WPCBs for the current experiment were collected from obsolete personal computers provided by Kaunas University of Technology. The collected WPCBs were manually dismantled in order to separate mounted components. The bare WPCBs were divided into two large groups with both of the groups containing samples of >20 years and <1 year of age: the WPCBs from the first group were cut into ~20 mm pieces using plyers, while the samples from the second group were left in their initial condition (uncut).

At the second step, the cut and uncut WPCBs samples of >20 years and <1 year of age were placed in a porcelain crucible and treated thermally in an electric furnace for 30 min at 500 °C temperature. The temperature was selected according to the previous scientific research data for metals recovery from WPBCs (21). After the thermal treatment, the samples were taken from the furnace and left for 2 h for cooling until the room temperature. The cooled samples were weighed by analytical balance weights (accuracy ±0.1 g). After the initial treatment of the samples hydrometallurgical leaching of precious metals was performed.

Aqua regia leaching of precious metals

Leaching of precious metals from the selected samples by aqua regia (nitric acid (HNO_3) and hydrochloric acid (HCl) ratio 1:3) was performed according to the following technique. The samples were submerged into 50 ml of aqua regia with a solid/liquid ratio of 1:10 (w/v) and stirred in a shaker for 1 h at 200 rpm.

After the aqua regia treatment, the liquid fraction was separated from the solid fraction by centrifugation. At the next step, Au, Pd, Ag and Cu concentrations were measured by the atomic absorption spectroscopy (AAS) method. The flowsheet for the experiment is shown in Fig. 2.

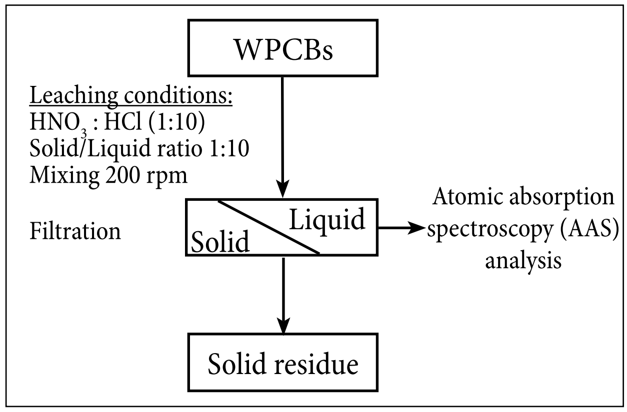

Fig. 2. Flowsheet for aqua regia leaching of precious metals from waste printed circuit boards

Three-stage thiourea leaching of precious metals

As it is shown in Fig. 3, during the first leaching step, 50 ml of 2 M sulfuric acid (H_2SO_4) were mixed with 12 ml of hydrogen peroxide (H_2O_2) (35%). The WPCB samples (sample weight 5–10 g) of the four following types were exposed to this reagent: cut and non-thermally treated samples, cut and thermally treated samples, uncut and non-thermally treated samples, uncut and thermally treated samples. The samples were stirred in the shaker at 200 rpm (temperature 298 ± 2 K) for 3 h; after this solid and liquid phases were separated by filtering. Cu, Au, Ag and Pd concentrations in the filtrate were measured by the AAS method. In the second step, the separated solid residue was additionally treated by the same reagent in the 250 ml conical flask and stirred again at 200 rpm and temperature 298 ± 2 K.

Cu and Ag oxidation is performed as follows:

$$Cu^{o} + H_2O_2 + H_2SO_4 \rightarrow Cu^{2+} + SO_4^{2-} + 2H_2O, \quad (1a)$$

$$2Ag^{o} + H_2O_2 + H_2SO_4 \rightarrow 2Ag^{+} + SO_4^{2-} + 2H_2O. \quad (1b)$$

For the third leaching step, 80 ml of 20 g/l thiourea, 6 g/l $Fe_2(SO_4)_3$, 10 g/l H_2SO_4 solution was prepared. Residues after the second leaching step were submerged into the solution in a 250 ml conical flask and the mixture was stirred for 3 h at 200 rpm and 298 ± 2 K temperature. Oxidation reactions that took place are shown below (Eqs. 2, 3):

$$Au^{o} + 2CS(NH_2)_2 + Fe^{3+} \leftrightarrow Au[CS(NH_2)_2]_2 + Fe^{2+}, \quad (2)$$

$$Ag^{o} + 3CS(NH_2)_2 + Fe^{3+} \leftrightarrow Ag[CS(NH_2)_2]_3 + Fe^{2+}. \quad (3)$$

After 3 h solid and liquid phases were separated by filtration; Cu, Au, Ag and Pd concentrations in the filtrate were again measured by the AAS method. During the dissolution Au and Ag form complexes with thiourea and the Fe^{3+} ion is reduced to Fe^{2+}. Also thiourea is easily oxidized to formamidine disulfide:

$$2CS(NH_2)_2 + 2Fe^{3+} \rightarrow CS(N_2H_3)_2 + 2Fe^{2+} + 2H^{+}. \quad (4)$$

Formamide disulfide is not stable in the acid media and splits into cyanamide and elemental sulphur:

$$CS(N_2H_3)_2 \rightarrow CS(NH_2)_2 + NH_2CN + S. \quad (5)$$

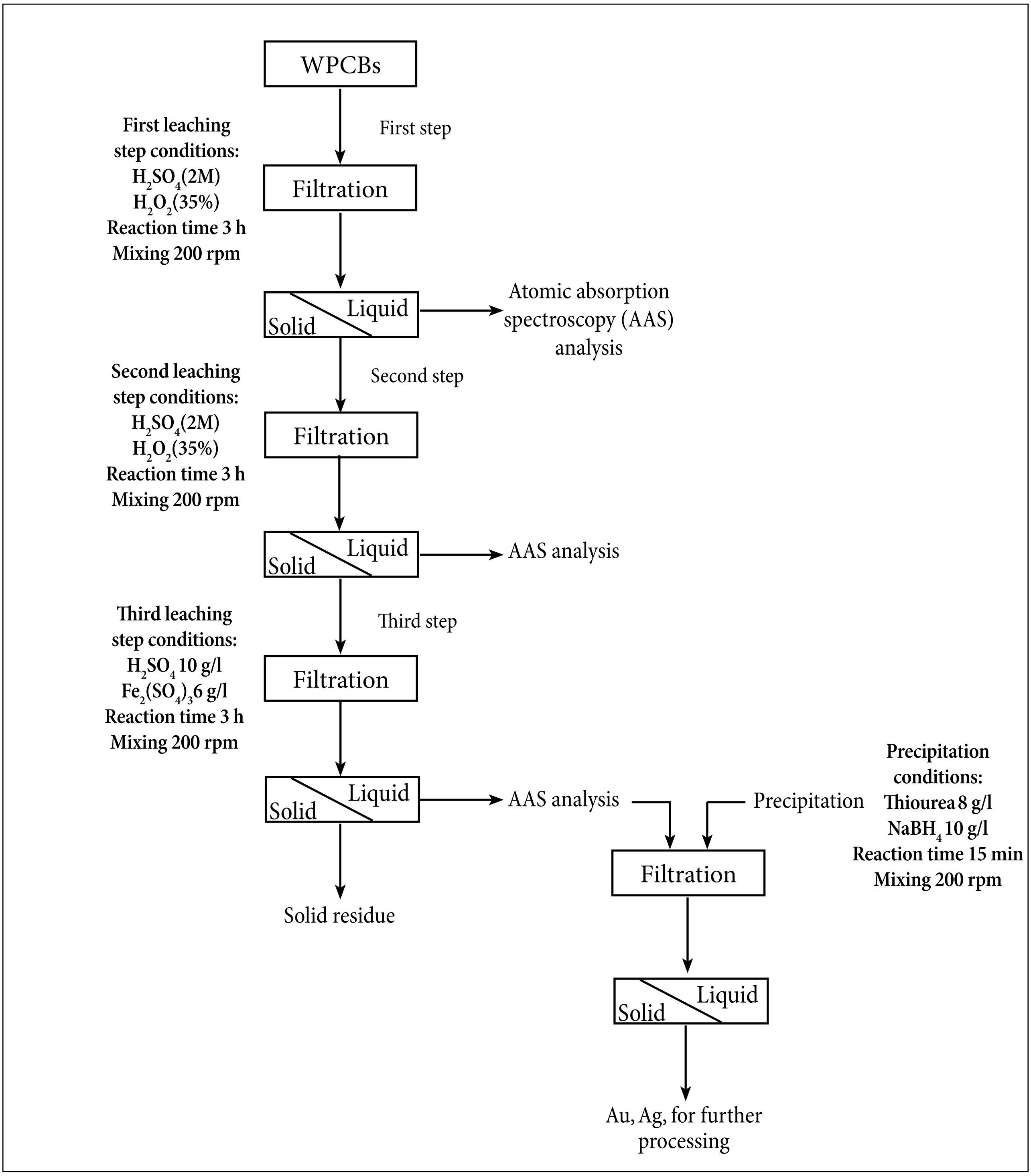

Fig. 3. Flowsheet for three-stage thiourea leaching of precious metals from waste printed circuit boards

Therefore it is important to avoid thiourea oxidation by a careful selection of the amount of an oxidizer. Ag and Au were precipitated from the thiourea solution by 50 ml of 8 g/l sodium borohydride ($NaBH_4$):

$$7Au[CS(NH_2)_2]_2 + BH_4 + 10OH^- \leftrightarrow$$

$$BO_3^{3-} + 7H_2O + 7Au + 14CS(NH_2)_2, \tag{6}$$

$$7Ag[CS(NH_2)_2]_3 + BH_4 + 10OH^- \leftrightarrow$$

$$BO_3^{3-} + 7H_2O + 7Ag + 21CS(NH_2)_2. \tag{7}$$

Precipitation was facilitated by mixing performed for 15 min at 298 ± 2 K. After the consequent filtration Au and Ag concentrations were measured by the AAS method.

RESULTS AND DISCUSSION

Aqua regia leaching of precious metals

The results for Cu, Ag, Au and Pd leaching by aqua regia are presented in Table 1. The concentrations of metals obtained during the experiment were recalculated to grams of metals per tonne of WPCBs. The extraction rate obtained by using aqua regia was considered to be 100% since this method was formerly proved by Veit et al. (2014) during the aqua regia leaching experiments [11]. Thus, the aqua regia leaching method was used as a reference method in order to estimate the efficiency of the thiourea leaching method.

Evaluating the results obtained in the current paper, we can see that the Au and Cu content is notably higher in the WPCBs of higher age (>20 years), being 749 g and 509 kg of Au and Cu, respectively. Additionally, old WPCBs were found to have such valuable metal as Pd, containing 1064 g of Pd per tonne of WPCBs. Regarding the new WPCBs (age <1 year), they were found to have a significantly lower content of metals with Pd completely absent. Leaching was able to provide recovery of only 43 g of Au and 117 kg of Cu per tonne of WPCBs. It is important to mention that aqua regia is not effective for Ag dissolution because its usage leads to the formation of an insoluble compound AgCl. As it will be shown, a significantly higher Ag dissolution rate can be achieved by H_2SO_4 and thiourea leaching.

H_2SO_4 and thiourea leaching of precious metals

The results of Cu, Ag, Au and Pd leaching by a multistage selective method are presented in Table 2.

Leaching of copper by H_2SO_4–H_2O_2 mixture

Comparing the results from Tables 1 and 2 we can see that during the first leaching step ~85% of the total amount of Cu from the samples of types 1, 2 and 4 was successfully dissolved. However, for the samples of type 3 this amount was only 26%. It can be explained by the presence of a modern soldermask on the newer PCBs which has higher chemical resistance than older counterparts and needs thermal treatment for decomposition. In the second step the last 5% of totally leachable Cu from the samples of types 1, 2 and 4 was dissolved. For the samples of type 3 the dissolved amount of

Table 1. **Results of aqua regia leaching of Cu, Au, Ag, Pd from WPCBs**

Sample types	Cu (kg/t WPCBs)	Au (g/t WPCBs)	Pd (g/t WPCBs)	Ag (g/t WPCBs)
1	509.03	748.71	1063.83	91.35
2	423.87	1077.59	862.93	74.4
3	73.09	33.28	–	4.76
4	117.06	43.11	–	5.73

1 is cut and thermally treated WPCBs (age >20 years), 2 is cut only WPCBs (age >20 years),
3 is non-treated WPCBs (age <1 year), 4 is cut and thermally treated WPCBs (age <1 year).

Table 2. **Results of three-stage H_2SO_4 leaching and thiourea leaching of Cu, Au, Ag, Pd from WPCBs**

Sample types	Cu (kg/t WPCBs)	Au (g/t WPCBs)	Pd (g/t WPCBs)	Ag (g/t WPCBs)
First step (H_2SO_4 (2M); H_2O_2 (35%))				
1	448.75	–	–	1060.07
2	377.3	–	-	4275.86
3	18.74	–	–	–
4	93.77	–	–	–
Second step (H_2SO_4 (2M); H_2O_2 (35%))				
1	21.59	–	–	11750
2	21.94	–	–	9900
3	53.61	–	–	–
4	4.95	–	–	–
Third step (20 g/l thiourea; 6 g/l $Fe_2(SO_4)_3$, 10 g/l H_2SO_4)				
1	–	732.60	–	275.86
2	–	899.33	–	138.27
3	–	13.82	–	79.86
4	–	27.24	–	140.47

1 is cut and thermally treated WPCBs (age >20 years), 2 is cut only WPCBs (age >20 years), 3 is non-treated WPCBs (age <1 year), 4 is cut and thermally treated WPCBs (age <1 year).

copper was 73%. In the third step <1% of Cu was dissolved, therefore these data were not taken into account for the further evaluation.

Leaching of silver by H_2SO_4–H_2O_2 mixture

For the old WPCBs (age >20 years) Ag extraction was detected during the all leaching steps. The highest Ag dissolution rate was observed for the old WPCB samples of types 1 and 2 during the second step. On the other hand, Ag from the new WPCBs turned on to be dissolvable only by thiourea during the third step.

Thiourea leaching of gold and silver

It was established that 733 g Au and 276 g Ag can be leached from 1 t of cut and thermally treated WPCBs (age >20 years). Further it was discovered that 899 g Au and 138 g Ag can be leached from 1 t of cut and thermally untreated old WPCBs (age >20 years). Regarding the new (age <1 year) WPCBs, it was found that 14 g Au and 80 g Ag can be leached from 1 t of uncut waste; for the cut and thermally treated samples this value was bigger – 27 g of Au and 140 g of Ag. A bigger amount of leached Au for the untreated old samples may have been due to their different chemical composition and formation of leaching-hindering substances from the organic part of the WPCBs. Thus, this issue requires additional investigation in following research papers.

Palladium leaching

The chosen technique (H_2SO_4 and thiourea leaching) turned out to be not able to leach Pd from the given WPCB samples.

Gold and silver precipitation

After the third leaching step, precious metals were precipitated by sodium borohydride ($NaBH_4$). The precipitate was separated by filtration and the filtrate was sent to AAS analysis. The analysis detected Cu traces and no presence of Au or Ag in the liquid phase, therefore it can be stated that the efficiency of precious metals precipitation by $NaBH_4$ reaches 100%.

Waste pre-treatment influence on leaching

According to the obtained results it can be concluded that pre-treatment considerably enhances precious metals leaching from WPCBs. The pre-treatment applied in the current paper resulted in the dissolution rate increased by 18–36% for Cu, 100% for Au and 76% for Ag.

Analysis of feasibilities for precious metals recovery from WPCBs

According to the data of the Lithuanian Environmental Protection Agency, during the 2014, 31.5 thousand tons of (EEE) were supplied for the internal Lithuanian market and 22.9 thousand tons of WEEE were collected, i.e. the collection rate was 72.9% (see Table 3) [22].

Based on the current world trends we can state the evidence of growing EEE demand and corresponding WEEE generation in Lithuania. The common share of WPCBs in the WEEE flow is 1.71% [23]. Thus during 2014, 392.62 t of WPCBs could have been collected from collection points and landfills in Lithuania after WEEE dismantling. According to the data of the Lithuanian Statistical department, the population of Lithuania was 2.943 millions, therefore in 2014 the possible collection of WPCBs could have been 0.133 kg per capita.

Given the fact that shares of old and new WPCBs are not known, the ranged values for valuable metals recovery were determined. Based on the research results for the three-stage leaching technique, feasible recovery ranges for Au, Cu and Ag were presented in Table 4. Pd recovery feasibility was not evaluated since the investigated technology of leaching by H_2SO_4 and thiourea was not effective for Pd.

Table 4. **Feasibilities for annual recovery of Cu, Ag and Au from WPCBs in Lithuania**

Metal	Cu, t	Ag, t	Au, t
Minimum	28.41	0.03	0.005
Maximum	184.67	5.62	0.35

SUMMARY AND CONCLUSIONS

1. Given the current trends in the WPCBs generation and precious metal value in the market, it can be concluded that recovery of precious metals from WPCBs is beneficial both economically and environmentally. After the literature analysis and evaluation of Cu, Au, Ag and Pd recovery described in these sources, leaching by the thiourea solution was selected. This decision was motivated by its sufficiently high leaching efficiency and low environmental impact in comparison to the cyanide and thiosulfate metal dissolution techniques. Thus thiourea can be used for precious metals recovery from WPCBs as an alternative to cyanide.

2. The performed experiments confirmed the feasibility of Au, Cu, Ag and Pd recovery. Also the importance of pre-treatment was confirmed. The metal leaching rate for

Table 3. **EEE surplus in the market and WEEE collection in Lithuania**

EEE surplus in internal market		WEEE collection			
Year	Amount supplied to the internal market, t	WEEE collected in Lithuania, t			
		Household WEEE	Non-household WEEE	Totally collected	Collection rate, %
2014	31515.88	21978.35	981.9	22960.25	72.9
2013	27548.14	15566.76	762.02	16328.78	59.3
2012	28336.94	13375.81	883.25	14259.06	50.3

preliminarily cut and thermally treated WPCBs was 18–100% higher in comparison with that of untreated WPCBs. It can be explained by the removal of a protective solder-mask layer, also by partial change of the mass and chemical structure of the WPCB matrix. After the thermal treatment, WPCBs become more crumble, they can decrease consumption of reagents and facilitate cutting. Also it was confirmed that the metal content in the old (age >20 years) WPCBs is significantly higher than in the currently generated WPCBs what is caused by the development of PCB production technology. The old WPCBs contain 17 times more Au, 4 times more Cu and 2 times more Ag in comparison to the new WPCBs which already do not contain Pd.

3. During the leaching experiments performed by aqua regia and H_2SO_4/thiourea 98% Au (samples age >20 years), 63% Au (samples age <1 year), 92.5% Cu (samples age >20 years) and 84.4% Cu (samples age <1 year) leaching efficiency was achieved. The obtained leaching efficiency for Au is not high enough, therefore additional investigations having an aim to increase the extraction rate are needed. According to the experimental results, the recovery of 140 g Ag from 1 t of the new WPCBs is possible, however, it is impossible to determine Ag leaching efficiency because the insoluble AgCl forms during the reaction of Ag with aqua regia. Evident differences between the WPCB matrix structures were found during the extraction – the three-stage leaching method had the highest extraction rate of Ag for old WPCBs achieved by H_2SO_4 and H_2O_2 leaching steps while for the new WPCBs Ag was leached only by thiourea. Therefore further investigations in order to study the influence of the WPCB matrix structure on the metal recovery process are currently performed. These researches are also supported by Kaunas University of Technology.

4. If the investigated methods for Au, Cu and Ag recovery from WPCBs were implemented in Lithuania, the following annual metals recovery rates would be possible: Cu – from 28 to 185 t, Ag – from 0.03 to 5.6 t, Au – from 0.005 to 0.35 t.

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HIDROMETALURGINIO TAURIŲJŲ METALŲ IŠGAVIMO IŠ SPAUSDINTINIŲ PLOKŠČIŲ ATLIEKŲ GALIMYBĖS LIETUVOJE

Santrauka

Spartų elektros ir elektronikos įrangos atliekų (EEĮA) augimą lemia intensyvi šios įrangos rinkos plėtra ir trumpas produkto gyvavimo ciklas. Kaip atskiras EEĮA tipas spausdintinių plokščių atliekos (SPA) išsiskiria sudėtimi. SPA sudėtyje gali būti tokių metalų kaip auksas (Au), varis (Cu), sidabras (Ag) ir paladis (Pd). Todėl SPA tampa vertingos kaip potenciali žaliava išgaunant tauriuosius metalus (TM).

Šiame straipsnyje nagrinėjamos Cu, Au, Ag, Pd hidrometalurginio išgavimo iš SPA galimybės palyginant jų išplovimą tiriamuoju ir kontroliniu metodais. Taikant tiriamąjį metodą buvo naudojamas trijų pakopų procesas. Pirmoje ir antroje pakopose išplovimui naudoti 2 M H_2SO_4 ir H_2O_2 (35 %). Trečiojoje metalai išplauti 20 g/l tiokarbamido ir 6 g/l $Fe_2(SO_4)_3$ tirpalais. Taikant kontrolinį metodą pasitelktas karališkasis vanduo.

Tiriamuoju metodu smulkintiems ir termiškai apdorotiems SPA bandiniams nustatytas toks TM išplovimo efektyvumas: 98 % Au (bandinių amžius >20 metų), 63 % Au (bandinių amžius <1 metai), 92,5 % Cu (bandinių amžius >20 metų) ir 84,4 % Cu (bandinių amžius <1 metai). Mechaniškai smulkintų ir termiškai apdorotų SPA metalų išplovimų efektyvumas padidėja taip: 18 % vario Cu (bandinių amžius >20 metų) ir 36 % vario Cu (bandinių amžius <1 metai); 100 % aukso Au (bandinių amžius <1 metai) ir 76 % sidabro Ag (bandinių amžius <1 metai). Taip pat nustatyta, kad, lyginant su 1 metų SPA, 20 metų SPA yra 17 kartų daugiau aukso, 4 kartus daugiau vario ir 2 kartus daugiau sidabro. Naujose SPA nebuvo rasta paladžio, tačiau nustatyta, kad iš 1 tonos senų SPA galima gauti 1 064 g paladžio.