RECYCLING OF PRINTED CIRCUIT BOARDS BY ACID LEACHING

Magdalena LISIŃSKA, Mariola SATERNUS

Silesian University of Technology, Gliwice, Poland, EU, mariola.saternus@polsl.pl

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Abstract

Currently, an avalanche of technology development can be observed, which results in an increase in the production and sale of electrical and electronic devices. The lifetime of electronic components, which at the end of the 20th century was 4 to 6 years, was shortened to 2 years in the first decade of the 21st century, and even to 9 months in the case of mobile phones. Today, most electrical and electronic devices contain printed circuit boards (PCB). On the one hand, PCB with electronic components is a hazardous waste due to the content of toxic components, on the other hand, it is a source of many valuable elements, including precious metals, non-ferrous metals, rare earth metals, platinum group metals, as well as iron and silicon. The metals in PCBs can be present in much higher concentrations than in conventional ore deposits, making PCB waste a particularly interesting material for recycling. PCB recycling involves multiple steps using a combination of physical, chemical, thermal and metallurgical processes. The main methods of recovering metals from PCBs are pyrometallurgical and hydrometallurgical methods. Hydrometallurgical techniques largely involve acid leaching, which has many advantages including high leaching rate and fast kinetics, but is quite corrosive. In laboratory tests, mineral acids (H_2SO_4, HNO_3, HCl) as well as aqua regia are used for leaching, sometimes with oxidants. The article reviews interesting and effective methods of leaching printed circuit boards from used mobile phones in the context of recovering copper and other metals contained in this waste.

Keywords: Hydrometallurgy, PCB, acid leaching, metal recovery

1. INTRODUCTION

In today's rapidly developing world of technology, electrical and electronic equipment such as TV sets, computers, printers, mobile phones or laptops have a shorter lifespan compared to devices from previous years [1]. The lifetime of electronic components, which at the end of the 20th century was 4 to 6 years, was shortened to 2 years in the first decade of the 21st century [2]. There are many reasons for shortening the life of electrical and electronic devices, including: the ever-lower price of devices that have more and more new features, devices that are faster, changes in the lifestyle of people with modern equipment, and strong competition between companies that want to manufacture and sell the best products based on advanced technologies [3]. An example is a mobile phone called a smartphone, whose sales have increased more than 12-fold in the last 13 years. According to statistics [4], approximately 1.43 billion smartphones were sold worldwide in 2021, which is a significant increase compared to 680 million units sold in 2012.

The properties of electronic waste, as well as their diverse composition, make them a large part of the hazardous waste stream, which can pose a serious threat to human health and life, but also to the environment. A common element of electronic devices and the main carrier of most metals are printed circuit boards (PCB). A printed circuit board with electronic components, on the one hand, is a hazardous waste due to the content of toxic components (Cr, Pb, Be, Hg, Cd, Zn, Ni) [5], and on the other hand, a source of many valuable elements, including precious metals (e.g. Au, Ag, Pt, Cu), non-ferrous metals (Sn, Ni, Cr, Zn, Sb, In, Pb), rare earth metals (Ta, Ge), platinum group metals, as well as iron and silicon. The concentration of metals (Cu, Sn), and in particular precious metals such as Au, Ag, Pd and Pt, is much higher than their original resources (conventional ore deposits), which makes PCB waste a particularly interesting material for recycling [1-3,5].

2. METHODS OF PCB TREATMENT

Recycling of printed circuit boards involves many stages consisting of a combination of physical, chemical, thermal and metallurgical processes [6]. No single approach is sufficient for the successful recovery of metals, so it is preferable to carry out this process using a combination of different extraction methods. The main methods of recovering metals from PCBs are: pyrometallurgical method [3,7] and hydrometallurgical method [8-34]. Pyrometallurgy is a commonly used method of metal recovery, including incineration, smelting in a plasma furnace or shaft furnace, sintering, high-temperature melting of waste materials [7]. The advantage of the methods of pyrometallurgical recovery of metals from electronic waste is the speed of reaction due to high temperature, or the ease of separating valuable substances from waste. The disadvantage is the high level of energy demand, the loss of Fe and Al in the slag, the production of dioxins, furans and volatile metals that threaten the environment and human life and health [35]. Hydrometallurgy is more precise, predictable, easy to control, requires low capital costs, energy and has a lower environmental impact compared to the pyrometallurgical method. The disadvantage of this method is that it is slow, time-consuming, requires thorough leaching, uses a large amount of various types of chemicals, which is associated with a large amount of wastewater produced [1, 8-10, 13, 34]. The large number of advantages of the hydrometallurgical method encourages research into the recovery of metals from circuit boards printed in this way.

Hydrometallurgical techniques largely involve acid leaching, which is the most common leaching method today and has many advantages including high leaching rate and fast kinetics, but is quite corrosive. Other reagents used are thiourea, thiosulfate, cyanides [36]. Cyanide leaching, which was once standard in gold mining, is being phased out due to its high toxicity. Thiourea and thiosulfate are the least dangerous leaching methods, but they are not as economically viable [34]. Thiourea has poor stability, thiosulfate has slow kinetics, and both require large amounts of reagent. One of the hydrometallurgical techniques is also bioleaching, which is becoming an interesting and promising option [37]. The same is true for organic acids. However, methods using these factors are under development or at an early stage of research. After leaching, various ways of removing metals from solutions are used - these include cementation, solvent extraction and electrolysis.

In the literature, you can find many results describing the recovery of metals from electronic waste, e.g. by acid leaching, using mineral acids such as H_2SO_4, HNO_3, HCl, as well as aqua regia. Additionally, oxidants O_2, O_3, Cl_2, H_2O_2, $Fe_2(SO_4)_3$, $FeCl_3$, NaCl, $CuSO_4$ were used. **Table 1** presents a summary of selected results of leaching with hydrochloric, nitric (V) and sulfuric (VI) acids by the most experienced researchers, process parameters (fineness/fraction size, solid to liquid fraction ratio, applied rotation, temperature, leaching time) and copper recovery and other metals contained in this waste.

Table 1 Summary of research on the recovery of metals from PCBs using HNO_3, HCl, H_2SO_4

Literature	Leaching agent	Process parameters	Temperature (K)	Time (min)	Metal recovery (wt%)
		Leaching with HNO_3			
[2]	1-6M HNO_3	PCS 2.5 mm^2, S/L=1 g/0.03 dm^3	296-353	360	99% Cu, 70% Sn, 99% Pb
[8]	1-4M HNO_3; 0.5-1.5M H_2SO_4; 4-16% H_2O_2	PCS 2-4 mm, S/L=25g/125 dm^3, 200-800 rpm	P: 383 303-363	P: 120 300	96% Cu HNO_3 75% CuH_2SO_4+H_2O_2
[9]	2-5M HNO_3	250 µm, 0.2 dm^3, 2-10% w/v, 350 rpm	303-343	120	99% Cu, 68% Ag
[10]	1M HNO_3	PCS 10x20x2 mm, S/L=20 g/0.1 dm^3	353	360-1140	95% Cu, 99% Ni, 98% Au
[11]	1-5M HNO_3, H_2SO_4, HCl	-	P: 1163 298-373	P:180 90	100% Cu, 100% Sn, 100% Pb
[12]	0.2M HNO_3 3.5M HCl	S/L=1 g/0.1 dm^3 (Pb, HNO_3); S/L=1 g/0.02 dm^3 (Sn, HCl)	363	45(Pb) 120(Sn)	100% Pb, 98% Sn

[13]	2M HNO_3, 2M H_2SO_4, 2M HCl	Fr (4-0.045>mm), S/L=1/4, 400 rpm	333-353	360-1140	100% Sn, Ni, 96% Zn, 55% Cu
	2M H_2SO_4, 2M HCl, 30% H_2O_2, O_3	Fr (4-0.045>mm), S/L=1/4, 400 rpm	333-353	360-1140	100% Zn, Ni, Sn, 70% Al, 69% Fe, 45% Cu
[14]	2M HNO_3, H_2SO_4, HCl, 30% H_2O_2, O_3	Fr (4-0.045>mm), S/L=1/4, 400 rpm	298-353	240	100% Cu, Sn, 6.8% Au, 13% Ag
Leaching with HCl					
[15]	1M HCl	Fr 8-0, 8-3, 3-0 mm, S/L=3 g/0.4 dm^3	P:773-1173, L:353	P: 15-60, L: 180	98% Cu
[16]	1M HCl	PCS 40×40 mm, 150 rpm	298	1320	100% Cu, Zn, Sn, Ni, Pb, Fe
[17]	2M HCl	Fr 8-3, 3-0, 0.5-0, 0.24-0 mm	353	120	100% Sn, 0.5% Cu
	0.25-2M HCl	Fr: 8-0, 0.4 dm^3, 10,30,50 g, S/L40; 13; 8	293-353	120-360	100% Sn, 0.5% Cu
	0.25-2M HCl	Fr 8-0, 8-3, 3-0 mm, S/L=40	293-353	120-360	Sn 100%, Cu 4.5%
	2M HCl + O_2	Fr 8-0, 1-0, 0,6-0 mm	353	120	85% Cu, 82% Sn
[18]	1-4M HCl + Cl_2	Fr 1.4-3 mm, 600 ml, 10 g, S/L=17 g/dm^3, 400 rpm	298-323	240	71% Cu, 98% Zn, 96% Sn, 96% Pb
[19]	0.3M HCl + 0.3M $FeCl_3$, 2M H_2SO_4	S/L: 1/8, 30 rpm, 1.75 kWh/kg, 0,02 dm^3/min	298	240	99% Cu
[20]	0.5M HCl + 0.3M $FeCl_3$	PCS 40-100 cm^2,500 g, S/L=1/8, 30 rpm	298	1440	75% Cu, 99% Cu
[21]	0.05-0.8M HCl + 0-0.4M H_2O_2 0.5-0.8M NaCl	500 ml, 0.001-0.006 g/dm^3, 0-500 rpm	298-338	90	98% > Cu
[22]	2M HCl+O_3 2.1 g/h O_3	0.4 dm^3, 10; 30; 50 g (S/L = 40; 13; 8), O_3 (30 l/h O_2, 45 kPa)	298	180	50% Cu (8-0 mm),80% Cu (3-0 mm),100% Cu (8-3 mm, S/L= 8)
[23]	0.5-1M HCl, HNO_3, H_2SO_4; EDTA, citrate, O_2, 30% H_2O_2; thiourea	Fr ≥500 µm, S/L=20 g/dm^3, 125 rpm; air 1 dm^3/min, 175 and 1253 mg O_3/dm^3	298	60-1440	90% Cu, 90% Au
Leaching with H_2SO_4					
[24]	2M H_2SO_4, 3M HCl; 2M H_2SO_4 + 3M HCl; 3M HCl + 1M HNO_3	Fr<0,208 mm, S/L=50 g/500 ml	333	120	93% Cu, 98% Sn
[25]	1M H_2SO_4 (1)1M H_2SO_4+240 ml H_2O_2 (2)	Fr 1 mm, S/L=1/10	348	240	90% Al, 8.6% Sn, 40% Zn (1); 100% Cu, 60% Zn,10% Al (2)
[26]	1-2M H_2SO_4,10-20 ml 30% H_2O_2, thiourea	Fr<3 mm, S/L=1/10; S/L=1/50	303-323	180	76% Cu, 77% Fe, 26% Sn, 99% Zn, 69% Au
	2M H_2SO_4+30% H_2O_2	Fr < 3 mm, pH: 1,48, 0.1dm^3 H_2SO_4+0.02 dm^3 H_2O_2	323	180	46 % Cu, 5.7% Fe, 21% Sn, 51% Zn
	2M H_2SO_4+30% H_2O_2	Fr < 3.0 mm, pH: 1,52	323	180	11% Cu, 62% Fe, 26% Sn, 31% Zn
[27]	1.2-2 M H_2SO_4, H_2O_2, O_2	Fr<2 mm, S/L=25-150 g/dm^3	323-353	300	100% Cu
	2M H_2SO_4+30% H_2O_2 8M HNO_3	S/L=50-100 g/dm^3, 20-40 cm^3/h H_2O_2, 100-300 rpm (Cu), S/L= 100-500 g/dm^3, 300 rpm (Pb)	323-353	300 180	100% Cu (H_2SO_4+ H_2O_2), 100% Pb, 100% Ag (HNO_3)
[8]	1.2M H_2SO_4+10% H_2O_2	Fr 2-4 mm, 100 g/dm^3, 500 rpm	323	240	75% Cu
[28]	2M H_2SO_4+0.2M H_2O_2	Fr <1.0 mm, S/L=10 g/1 dm^3,150 rpm	358	480-720	100% Cu, Zn, 95% Fe, Ni, Al
[29]	0.45-1.6 M H_2SO_4, 0.8% H_2O_2	Fr < 3.35 mm, S/L=2 g/0.15 dm^3	341	240	98% Cu
[30]	2M H_2SO_4+5%, 30% H_2O_2	Fr < 2.0 mm, S/L=1/10, 200 rpm	298-303	180	90% Cu
[31]	2M H_2SO_4+35% H_2O_2 (1) 2MH_2SO_4+35% H_2O_2 (2)	Fr 300 µm, S/L=1/10, 0.1 dm^3+0.025 dm^3/12 g, 200 rpm	298	180	85% Cu (1), 14% Cu (2) 99% Cu whole

[32]	2-2.5MH_2SO_4+20% H_2O_2 + CO_2	Fr 2 mm, S/L= 1/20, 600 rpm	308	60-240	90% Cu
[33]	0.5M H_2SO_4+0,5g/l Cu^{2+}+ O_2>2 g/l	Fr 1.0 mm	353	120	100% Cu
[13]	2M and 5M H_2SO_4, H_2O_2	PCS 20-40 mm, S/L=1/10, 340 rpm	298-343	360-4320	100% Fe, Zn,40% Al, 15% Cu
[34]	2M, 5M H_2SO_4, O_3	PCS 20-40 mm, S/L=1/10, 340 rpm	298-343	360-4320	100% Fe, Zn, Al, 26% Cu
Where: P - pretreatment, L - leaching, PCS - pieces, Fr - fraction, (1) - first stage, (2) - second stage.					

3. CONCLUSION

Laboratory tests in the field of hydrometallurgical processing of printed circuit boards have a wide spectrum of activity. Various leaching methods can be found in scientific publications, such as acid, alkaline, pressure and oxidative leaching. Individual metals cannot be obtained in one step, hence the authors of various works use multi-stage processing of these wastes, using a mixture of various acids and oxidant additives. In the first stages, it is possible to leach metals that do not require the use of aggressive reagents, e.g. zinc and tin. In the next stages, it will be mainly copper leaching with the addition of oxidants, and in the last stage it will be mainly precious metals. In particular, printed circuit boards from computers and mobile phones are processed, because the metal content is higher than in printed circuit boards from other electronic devices (televisions, monitors, etc.).

In most works, the authors use the pre-treatment of the material - mechanical-physical method (crushing and/or grinding) as the basic step before leaching. By comminuting the material, the access of the leaching agent to the surface of the material is increased. In particular, this concerns the release of metals such as copper, which is located between the layers of fiberglass. It is also important to choose the ratio of the solid to liquid fraction S/L. The use of elevated temperature in the tests resulted in an increase in the concentration of mainly Fe, Ni and Sn in the solution. The stirring speed showed a pronounced effect on metal recovery from PCB pieces.

Leaching precious metals from printed circuit boards is mainly thiosulfate or thiourea, while copper and other non-ferrous metals are leached mainly with acids (nitric acid, hydrochloric acid and sulfuric acid VI) with the use of oxidizing agents, e.g. such as hydrogen peroxide, Cu^{2+}, Cl_2, Fe^{3+} ions, air, oxygen or ozone. Recently, the influence of organic acids (e.g. citric acid, malic acid, lactic acid, oxalic acid, etc.) on the leaching of metals, which are more environmentally friendly, has also been studied. In general, the selection of a suitable leaching medium depends on the following factors: rapid and high solubility of the material in the leaching agent is desired, the leaching agent should preferably be cheap so that its losses do not reduce the economy of the process; a suitable leaching agent will extract only the desired component, it is desirable not to have a negative impact on the environment through the use of the leaching agent. Hydrogen peroxide as an oxidant is environmentally friendly as the end products of its decomposition are oxygen and water. Similarly, ozone, its use as an oxidant, can be a reasonable alternative, also providing environmental benefits in the leaching process, mainly due to the formation of oxygen as the only by-product of the reaction. The environmental impact of both the leaching agent and the products of the process must be considered when selecting.

Generally, the process of recovering metals from PCBs is a complex and complicated process, mainly due to the heterogeneity of the material and the possibility of individual elements influencing the chemical reactions between metals and acids, sometimes a given reaction is inhibited by the occurrence of coexisting reactions blocking, for example, further leaching of a given metal. Importantly, PCB leaching for metal recovery is only the first step, further study is required for the purification and recovery of metals from pregnant leaching liquids. In this case, the factors that affect the selection of an appropriate method are the leaching reagent system, concentration of metal(s), impurities and economy of the process.

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