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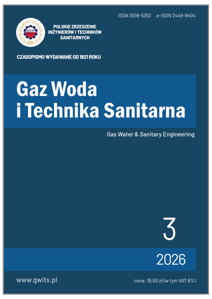

Nickel recovery potential from thermally treated waste printed circuit boards in hydrometallurgical processes

Potencjał odzysku niklu z termicznie przetworzonych odpadowych płytek drukowanych w procesach hydrometalurgicznych

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Abstract:
The growth of waste electrical and electronic equipment has intensified the search for effective recovery technologies of valuable metals from printed circuit boards (PCBs). In this study, the hydrometallurgical behavior and recovery potential of nickel from thermally treated PCB were investigated. The PCB waste was subjected to steam gasification as a pretreatment step, followed by mechanical separation and subsequent leaching using different acidic media, including 2 M sulfuric acid, aqua regia, and dilute acids assisted by ultrasonic treatment. The results indicate that thermal pretreatment improves the accessibility of nickel-bearing phases and promotes their dissolution during hydrometallurgical processing. Leaching efficiency depended on the leaching agent and particle size. Ultrasound-assisted leaching with dilute acids further enhanced nickel extraction.

Keywords: PCB, recycling, steam gasification, circular economy

Streszczenie:
Wzrost ilości zużytego sprzętu elektrycznego i elektronicznego sprzyja rozwojowi technologii odzysku metali z odpadowych płytek obwodów drukowanych (PCB). W pracy oceniono potencjał hydrometalurgicznego odzysku niklu z materiału PCB poddanego wstępnej obróbce termicznej metodą zgazowania parą wodną. Po separacji mechanicznej na frakcje ziarnowe przeprowadzono ługowanie w różnych środowiskach kwaśnych, w tym w 2 M H_2SO_4, wodzie królewskiej oraz rozcieńczonych kwasach wspomaganych ultradźwiękami. Wyniki wskazują, że obróbka termiczna zwiększa dostępność faz niklonośnych i sprzyja ich rozpuszczaniu. Efektywność ługowania zależała od rodzaju czynnika ługującego oraz wielkości ziaren. Zastosowanie ultradźwięków dodatkowo intensyfikowało proces ługowania.

Słowa kluczowe: PCB, recykling, zgazowanie parą wodną, gospodarka o obiegu zamkniętym

1. Introduction

The dynamic growth of the global economy has led to a substantial increase in the production of electrical and electronic equipment. Both technological innovation and market expansion accelerate the replacement cycles of electrical and electronic equipment (EEE), resulting in the large-scale generation of waste electrical and electronic equipment (WEEE).

One of the core component of WEEE – waste printed circuit boards (WPCBs), is classified as hazardous waste. The core layers of PCBs are conventionally composed of fiber-reinforced laminates impregnated with flame-retardant resins containing bromine and chlorine. The presence of organic fractions in the laminate, as well as its advantageous properties such as high resistance to environmental conditions and mechanical strength, become major drawbacks during disposal and treatment processes. The wide range of applications, mass production, and extensive use of fiber-reinforced laminates generate a growing stream of this type of waste, which, after the end of its service life, is often disposed of in an unchanged form in landfills. As materials that are resistant to degradation, they pose a long-term environmental challenge. On the other hand, the copper content in PCB lami-

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nates, as well as the presence of glass fibers and precious metals (Au, Ag, Pd) in finished printed circuit boards makes them economically attractive from a resource recovery perspective [1]. In addition to copper and precious metals, PCBs also contain base and accompanying metals such as tin, lead, aluminum, and nickel. Nickel is widely used in electronics as a component of protective coatings, intermediate layers in electroplated connections, and structural elements of connectors [2].

Beyond its functional role in electronic components, nickel is a transition metal of high industrial relevance, primarily consumed in the production of stainless steel, high-performance alloys, electroplated coatings, and battery materials. Approximately 70% of global nickel production is directed toward stainless steel manufacturing, while additional applications include chemical components and industrial catalysts [3]. Owing to its strategic importance for modern industry and energy transition goals, nickel is increasingly considered in European assessments of critical and strategic raw materials, particularly in battery-grade quality, which is essential for electromobility and energy storage technologies [4].

In the context of electronic waste recycling, such as waste printed circuit boards, nickel present in ionic form in hydrometallurgical process solutions represents a potentially valuable secondary raw material. Although nickel concentrations in these solutions are typically lower than those found in primary ores, its recovery may contribute to closing material loops, reducing dependence on primary resources, and mitigating the environmental burden associated with the extraction and processing of critical metals [5].

Nickel is recovered at various stages of industrial processing of both primary and secondary resources using integrated pyrometallurgical–hydrometallurgical routes. Such approaches are increasingly applied in large-scale recycling facilities treating complex waste streams, including end-of-life electronics. Industrial installations, such as those operated by Umicore and Aurubis (Kayser Recycling System, Lünen, Germany), process electronic waste through smelting followed by electrorefining and hydrometallurgical treatment. These processes enable the recovery of nickel alongside other valuable metals, primarily copper and selected precious metals. In the Aurubis KRS process, nickel is recovered from the electrolyte, typically in the form of nickel sulfate, allowing its further industrial utilization [6, 7]

In hydrometallurgical processes applied to electronic waste, including printed circuit boards, nickel is typically transferred into the liquid phase in the form of Ni^{2+} ions in acidic leaching solutions. The presence of nickel in post-process solutions represents both a technological challenge and a potential opportunity for its recovery as a secondary raw material. As highlighted in studies on non-ferrous metal hydrometallurgy, the effective management and separation of nickel ions from leachates is a key aspect in the design of efficient metallurgical process flowsheets [8, 9].

This paper presents the outcomes of collaborative research conducted with EKO PM Ltd., a company involved in the processing of various multi-material waste streams, in particular waste electrical and electronic equipment, using steam gasification combined with hydrometallurgical treatment routes [10].

The aim of this study was to discuss the potential for nickel recovery from thermally treated waste PCB material using selected hydrometallurgical processes, as well as to provide a preliminary assessment of the relevance of this phenomenon from the perspective of the design and operation of recycling installations treating waste streams containing this element.

2. Materials and methods

2.1. Research material

The research material consisted of waste type A printed circuit boards (PCBs), which were used in steam-atmosphere gasification experiments. The material was prepared by EKO PM Ltd. Steam gasification was carried out in accordance with the assumptions of the patented technology [11], using an integrated reactor equipped with a gas cooling and steam condensation zone. The main products of the process were a solid residue and a hydrogen-enriched synthesis gas. The solid product comprised metallic and mineral fractions. Gasification enabled the recovery of metallic and ceramic components in an essentially unchanged form, close to their original state. Following the gasification process, the resin-bonded layers of mineral fibres within the PCB laminate became loosened, which allowed for the mechanical separation of individual components and their subsequent processing by hydrometallurgical methods in order to obtain pure metals and their concentrates. Figure 1 illustrates a PCB after the gasification process.

Fig. 1. Waste printed circuit board (PCB) after gasification in a steam atmosphere [12]

Rys. 1. Odpadowa płyta obwodu drukowanego (PCB) po procesie zgazowania w atmosferze pary wodnej [12]

The solid residue remaining after the gasification process was subjected to grinding in a ball mill, followed by fractional separation into 18 fractions. Subsequently, taking into account the sample sizes, eight main fractions were distinguished. In the next stage, the physical properties of the material were considered, and further separation was performed within the range of the main fractions.

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2.2. Research methodology

Hydrometallurgical approach

First, the sample was rinsed with distilled water. This step was performed only for fraction No. 1 (<0.1 mm). The solid-to-liquid ratio was 1:5. The experiment was conducted for 1 h at the process temperature with a stirring speed of 800 rpm. After completion, the sample was pressure-filtered using a Büchner funnel.

In the next stage, acidic leaching with 2 M sulfuric acid (H_2SO_4) was carried out according to the procedure shown in Fig. 2. The following fractions were selected for this step: 1, 2, 3, 4, 5A, 5B, 5C, 6A, 6D, 6E, 7 and 8A. According to information provided by EKO PM Ltd., these fractions were characterized by the potentially highest metal content.

The subsequent stage involved leaching with aqua regia (AQ), prepared from concentrated hydrochloric acid (HCl) and nitric acid (HNO_3) in a volume ratio of 3:1. The same fractions as those used for sulfuric acid leaching were selected for aqua regia leaching. The procedure was analogous to that applied during sulfuric acid leaching (Fig. 2), with the only difference being the leaching agent.

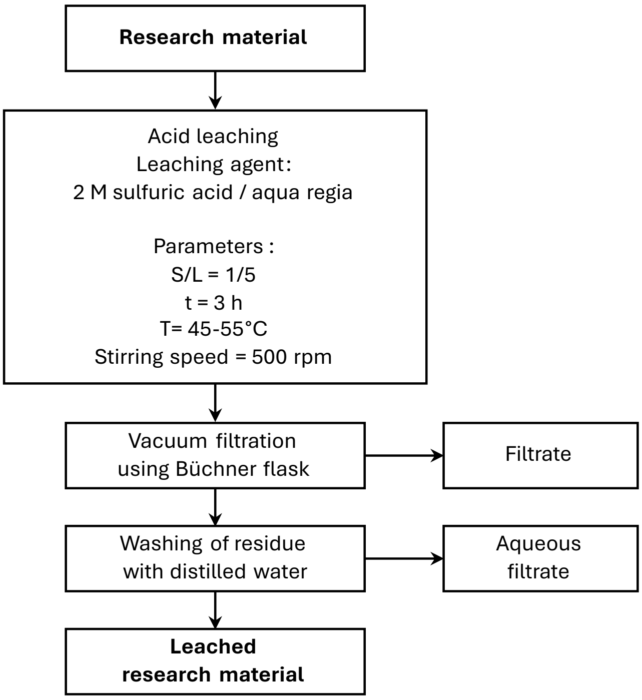

Fig. 2. Scheme of the leaching procedure using 2 M sulfuric acid (H_2SO_4), own study [12]

Rys. 2. Schemat procedury ługowania 2M kwasem siarkowym (VI), opracowanie własne [12]

Additionally, a portion of the samples was subjected to ultrasound-assisted leaching in order to intensify the contact between the solid phase and the solution and to accelerate dissolution processes. The leaching process in the ultrasonic bath was carried out using less concentrated acids, namely 0.1 M sulfuric acid (H_2SO_4) and 0.1 M nitric acid (HNO_3). This process was applied to fractions that had not been included in the acidic leaching stage. These fractions were excluded mainly due to their larger particle size and low specific surface area (rods, sheets, screws). The fractions subjected to this process were 6B, 6C, 7, 8, 8B and 8C. The process was conducted according to the procedure shown in Fig. 3.

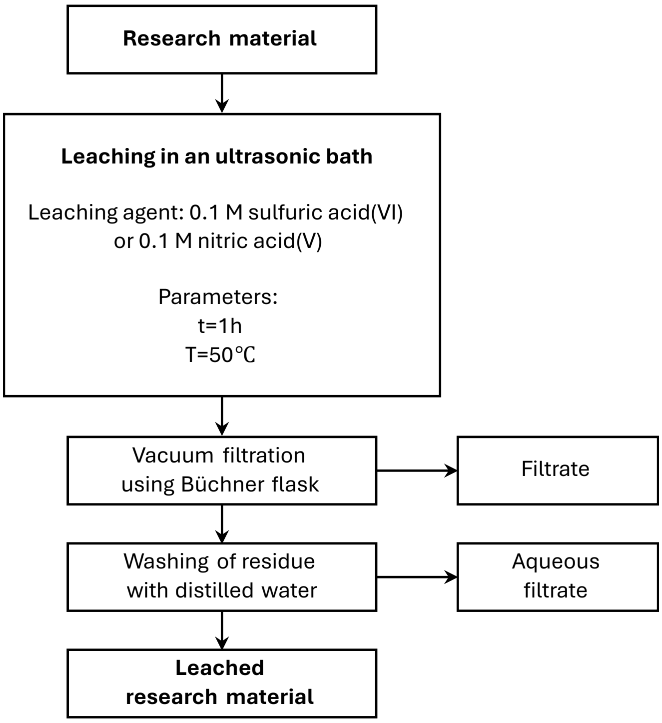

Fig. 3. Flowchart of ultrasounf-assisted leaching procedure own study [12]

Rys. 3. Schemat procedury ługowania wspomaganego ultradźwiękami), opracowanie własne [12]

2.3. Analytical method

Gravimetric analysis

After rinsing with water and after each leaching stage, i.e. acidic leaching with 2 M sulfuric acid (H_2SO_4), aqua regia leaching, and ultrasonic bath leaching using 0.1 M sulfuric acid (H_2SO_4) and 0.1 M nitric acid (HNO_3), the solid residues were transferred to pre-weighed crystallizing dishes and dried at 105°C for 24 h. The crystallizing dishes containing the material were weighed on an analytical balance after mass stabilization. In this way, mass losses after each leaching stage were determined.

Qualitative and quantitative chemical analysis

In order to determine nickel concentrations in the leachates, inductively coupled plasma optical emission spectrometry (ICP-OES) was applied. The ICP-OES analysis of metal contents was performed for all filtrates, including both aqueous rinses and leachates obtained after each leaching stage, at the GEO-3EM Hydrometallurgical Processes Laboratory of Wroclaw University of Science and Technology. The analytical results for individual samples were obtained in mg/L and subsequently recalculated to g/t of sample based on the known volume of the analyzed solution and the initial mass of the sample.

3. Results and discussion

Mechanical processing

The results of the particle size fractionation are presented in Table 1. The results of mechanical size-based separation of waste PCB material after the gasification process indicate a dominant contribution of the fine-grained fraction (0.1 mm), which accounted for nearly half of the total material mass (46.8%). Such a high proportion of this fraction can be attributed to the

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preliminary thermal treatment, which led to the loosening of the laminate structure and increased material brittleness, thereby promoting intensive fragmentation. This observation confirms that gasification in a steam atmosphere effectively removes the polymeric fraction of the PCB laminate, facilitating its subsequent mechanical separation.

In this context, fractions with a significant mass contribution are expected to exhibit enhanced nickel leaching due to their increased specific surface area and improved accessibility of reactive surfaces for leaching agents. This is consistent with literature reports indicating the influence of particle size reduction and contact surface area on the kinetics of metal extraction from PCB waste [13]. Fine fractions may contain fragmented nickel-rich electroplated coatings as well as small particles of nickel-based alloys with other metals formed as a result of mechanical disintegration.

In contrast, coarse fractions (>5 mm), consisting of larger fragments of structural components, connectors, and pins, may locally exhibit elevated nickel contents; however, due to their lower specific surface area, their direct processing by hydrometallurgical methods may result in reduced leaching kinetics. In industrial practice, such fractions may require additional comminution or preliminary physical separation to increase the efficiency of nickel transfer into leaching solutions.

The contribution of intermediate fractions (0.1–5 mm) reflects the considerable morphological heterogeneity of the post-process material and the non-uniform distribution of metallic and mineral phases. From the perspective of designing a technological flowsheet for nickel recovery, differentiated treatment of individual size fractions appears justified, whereby fine fractions are directed directly to hydrometallurgical leaching, while coarser fractions are subjected to further mechanical processing or selective metal separation.

Table 1. Fractional division of the research material, own study based on the work [12]

Tabela 1. Podział materiału badawczego na frakcje, opracowanie własne, według [12]

Sample number	Fraction [mm]	Mass [g]	Percentage share [%]
1	<0.1	1159.86	46.8
2	0.1–0.25	141.83	5.4
3	0.25–0.5	100.21	3.1
4	0.5–1	117.95	4.0
5A	1–2	68.82	3.6
5B	1–2	9.27	0.2
5C	1–2	28.78	1.1
6A	2–5	133.34	5.8
6B	2–5	33.26	1.3
6C	2–5	71.71	3.0
6D	2–5	21.44	0.7
6E	2–5	66.22	2.8
7	5–10	187.74	5.8
8	>10	200.14	6.3
8A	>10	62.28	2.6
8B	>10	24.10	0.9
8C	>10	137.30	6.5

Qualitative and quantitative chemical analysis

The results obtained after leaching with 2 M sulfuric acid and aqua regia are presented in Table 2, whereas Table 3 summarizes the ICP-OES results obtained after leaching with less concentrated acids (0.1 M HNO_3 and 0.1 M H_2SO_4) assisted by ultrasonic treatment. The obtained data indicate that the efficiency of nickel transfer into the leaching solution is strongly dependent on the applied leaching agent and the particle size of the post-reaction PCB material. For fine-grained fractions subjected to sequential leaching, the use of 2 M H_2SO_4 enabled partial dissolution of nickel. The measured nickel concentrations in the solutions showed significant variation between individual samples and ranged from approximately 65 g/t to over 3200 g/t, depending on the sample. This variability can be attributed to the heterogeneous distribution of electroplated coatings and nickel-containing alloys within the PCB laminate, which is commonly reported in the literature on electronic waste [4, 17]. In addition, the observed behavior is consistent with the known resistance of nickel to dissolution in non-oxidizing mineral acids under moderate temperature and redox conditions in hydrometallurgical systems [9].

Much higher nickel concentrations were observed after secondary leaching of the same fractions with aqua regia. The measured nickel contents ranged from approximately 1200 g/t to over 35,000 g/t. These results indicate that a significant portion of nickel remains bound in forms that are poorly soluble in sulfate media. Such forms include Ni–Cu and Ni–Fe alloys, as well as passivated metallic layers, which are not effectively dissolved in 2 M H_2SO_4. Due to its strong oxidizing properties and its ability to break down alloy structures, aqua regia enables a much more complete transfer of nickel into the liquid phase. This confirms its high efficiency as a reference leaching agent in metal balance studies [7, 15]. However, such an aggressive leaching environment is less suitable for industrial application because of safety concerns, equipment corrosion, and the complexity of solution treatment.

Table 2. Nickel content results for different leaching agents [12]

Tabela 2. Wyniki zawartości niklu dla różnych czynników ługujących [12]

Sample number	Ni		
	Aqua	2M H_2SO_4	AQ
	[g/t]		
1W	<LOD	n.a.	n.a.
1	n.a.	3230	4486
2	n.a.	1603	7838
3	n.a.	209.2	8668
4	n.a.	96.61	7591
5A	n.a.	70.37	4804
5B	n.a.	1670	35276
5C	n.a.	66.99	6381
6A	n.a.	64.91	1996
6D	n.a.	76.58	2588
6E	n.a.	66.62	1233
7	n.a.	119.0	1879
8A	n.a.	150.7	539.7

n.a. – not analysed

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Table 3. Nickel content after ultrasound-assisted leaching [2]
Tabela 3. Wyniki zawartości niklu po procesach ługowania wspomaganych ultradźwiękami [2]

Sample number	Ni	
	Ultrasonic bath leaching	
	0.1M H_2SO_4	0.1M HNO_3
	g/t	
6B	494	301
6C	193	324
7	30	89
8	299	242
8B	252	349
8C	38	3

The application of ultrasound in combination with less concentrated acid solutions increased the efficiency of nickel transfer into the liquid phase. This effect is attributed to enhanced mass transport, improved surface wetting, and easier penetration of the leaching solution into the porous structure of the thermally treated material, in agreement with previous studies on ultrasound-assisted hydrometallurgical processes [16, 17].

A comparison of both leaching agents does not indicate a clear advantage of either sulfuric or nitric acid. Higher nickel concentrations were observed in H_2SO_4 for some samples (e.g., 6B and 8), whereas HNO_3 was more effective for others (e.g., 6C, 7, and 8B). The lack of a consistent trend suggests that leaching efficiency is mainly controlled by the local mineralogy and the form of nickel in a given fraction, including electroplated coatings and Ni–Cu or Ni–Fe alloys [13, 14].

Relatively low nickel concentrations, particularly in samples below 50 g/t, may reflect either a low nickel content in the investigated fractions or limited leaching kinetics associated with compact structures and low specific surface area.

The presence of nickel in post-leaching solutions has important technological implications. On the one hand, it indicates the potential for secondary recovery of nickel as an accompanying metal in PCB recycling processes, in line with the principles of the circular economy and the growing need to diversify sources of technologically important metals [18]. On the other hand, nickel ions represent an additional burden for solution treatment systems, as they require effective removal and lead to the generation of secondary residues [8].

The obtained results confirm that the design and operation of electronic waste recycling installations should consider not only major metals but also accompanying metals such as nickel, which can significantly affect process balances and solution treatment requirements. An integrated approach combining pyrometallurgical and hydrometallurgical stages enables the potential utilization of nickel-bearing streams as secondary raw materials in modern metal recycling systems.

6. Conclusions

The conducted study demonstrated that nickel, although not being the primary target of metal recovery from waste printed circuit boards, is transferred to hydrometallurgical solutions to a significant extent after the thermal treatment of PCB material by steam gasification. The degree of nickel dissolution was strongly dependent on the applied leaching agent and process conditions. More aggressive leaching environments, particularly aqua regia, promoted enhanced dissolution of nickel-containing phases present in the PCB structure, while sulfuric acid enabled only partial nickel extraction.

Thermal pre-treatment of PCB material resulted in a pronounced loosening of the laminate structure and an increase in material brittleness, which improved the accessibility of metallic phases for hydrometallurgical reagents. This effect was particularly evident for fine-grained fractions, characterized by a higher specific surface area and more intensive nickel transfer into the liquid phase. Ultrasound-assisted leaching further intensified nickel dissolution when using dilute acids, confirming the beneficial role of physical process intensification methods in enhancing mass transfer and leaching kinetics.

From a technological perspective, the presence of nickel ions in post-leaching solutions represents both an opportunity and a challenge. On the one hand, nickel-bearing process streams constitute a potential secondary raw material that could be integrated into circular economy-oriented recycling schemes. On the other hand, dissolved nickel increases the load on wastewater treatment systems, contributing to the formation of metal hydroxide sludges and generating additional secondary residues requiring further management.

The results obtained in this study indicate that accompanying non-ferrous metals such as nickel should be systematically considered in process mass balances and in the design of hydrometallurgical circuits for WEEE recycling. Even when nickel is not the primary recovery target, its behavior in process solutions significantly affects the technological performance, environmental footprint, and overall sustainability of integrated pyrometallurgical–hydrometallurgical recycling installations.

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