"Babeș - Bolyai" University of Cluj-Napoca
Faculty of Chemistry and Chemical Engineering
Doctoral School of Chemical Engineering

ELECTROCHEMICAL RECOVERY OF BASE METALS FROM WASTE PRINTED CIRCUIT BOARDS

SUMMARY OF THE DOCTORAL THESIS

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Cluj-Napoca
2025

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List of abbreviations used

AS	Sensor area
BTG	Sandstone type slip
BTF	Faience type slip
BTFA	White faience slip
CAP	Galvanostatic chronoamperometry
CE	Counter electrode
CoA	Anode compartment
CoC	Cathode compartment
CPG	Galvanostatic chronopotentiometry
CV	Vitreous carbon
DEEE	Waste electrical and electronic equipment
DPCI	Waste printed circuit board
DPCID	Disassembled waste printed circuit board
EED	Electrodeposition efficiency
EDR	Rotating disc electrode
EL	Working electrode
FN	Glazed Earthenware
FNZ	Fe-Ni-Zn
FS	Faience S
FTE	Faience type Swiss
GGTS	Glazed Stoneware type S
ICP	Inductively coupled plasma
ICP-AES	Inductively coupled plasma atomic emission spectrometry
MB	Base metals
MC	Ceramic membrane
MCO	Horizontal ceramic membrane
MCP	Porous ceramic membrane
MP-EDR	Raw material -EDR
MP-P	Raw material -powder
MRC	Certified reference materials
MSA	Anion exchange membrane
MSC	Cation exchange membrane
p.a.	Analytical purity
P1.1/P1.2	Peristaltic pumps
PB	Biological processes
PBC	Printed circuit board
PDPCIDM	Dry ground uncoated WPCB powder
PE	Electrochemical processes
PH	Hydrometallurgical processes
PP	Pyrometallurgical processes
PR	Real evidence
R	Tank
RA	Anolyte tank
RC	Catholyte tank
RC	Chemical reactor
RCT	Drum chemical reactor

RDH	Hydrogen discharge reaction
RE	Electrochemical reactor
REC	Compartmentalized electrochemical reactor
RECFP	Filter press type electrochemical reactor
RECT	Tubular electrochemical reactor
Ref.	Reference electrodes
RETC	Compartmented tank type electrochemical reactor
RL	Leaching reactor
SAACG	Graphite furnace atomic absorption spectrometry
SAAF	Flame atomic absorption spectrometry
SB	Basic solution
SES	The supporting electrolyte solution
SLC	Concentrated bleach solution
SLICu	Cu-enriched SL
SS	Stainless Steel 304
TRM	Metal recovery technologies
V	Valve
VHBCP	Hydrodynamic voltammetry with cyclic potential sweep
VHBLP	Hydrodynamic voltammetry with linear potential sweep
VL	Linear voltammetry
VOC	Cyclic voltammetry

List of symbols used

E	Medium potential, [V]
E_{CE}	Counter electrode potential, [V]
E_{EL}	The potential applied to the working electrode, [V]
$E_{ÎNT}$	The potential for return, [V/ER]
r_F	Faradaic efficiency, [%]
G	Ceramic membrane thickness, [cm]
i	Current density, [A]
I_{EL}	Current through the working electrode, [A]
I_L	Current limit, [A]
i_L	Limiting current density, [A]
k_M	Mass transport coefficient, [m/s]
Me	Metal concentration, [g/L]
Me_I	Initial metal concentration, [g/L]
Me_F	Final metal concentration, [g/L]
m_D	Deposit mass, [g]
n	Valence of ions,
ORP	Oxidation-reduction potential, [V]
ORP_M	Medium oxidation-reduction potential, [V]
$PR_{HBr,A}$	Average productivity, [kg/(m^2*h)]
R	Resistance, [Ω]
$r_{F,A}$	Faradaic efficiency on the anode, [%]
$r_{F,C}$	Faradaic efficiency on the cathode, [%]
s	pH sensor sensitivity, [mV/dec.]
T	Temperature sensor, [°C]
U_B	The voltage at the reactor terminals, [V]
W_S	Specific energy consumption, [kWh/kg]
$W_{S,A}$	Specific energy consumption per anolyte, [kWh/kg]
$W_{S,C}$	Specific energy consumption per catholyte, [kWh/t]
ϕ_{EXT}	Outer diameter, [m]
ϕ_{INT}	Inner diameter, [m]
ω	Rotation speed, [rpm]
σ	Conductivity passing through the membrane, [$\Omega^{-1}cm^{-1}$]

Introduction

Industrial and urban development at increasingly accelerated rates and, with it, population growth, rising levels of civilization and meeting increasing consumer needs have led to the advanced depletion of natural sources and reserves of raw materials, energy, fuels and materials.

In the current global context, where environmental problems are increasingly acute, the issue of waste electrical and electronic equipment (WEEE) is of increasing interest, both for local and central authorities, and for ordinary citizens. The damage to the environment and, implicitly, to the health of the population make WEEE management an issue of major interest. Thus, solving this problem as efficiently as possible should become a priority for any civilized state. The interest is not only due to these environmental problems, those related to the depletion of natural mineral resources, but also to the possibility of recovering large sums of money from the recovery of WEEE.

Based on engineering sciences, a system of technical norms and standards has been developed that regulate the recovery and recycling of WEEE, but a series of conditions imposed by technical standards and legislative norms have to some extent discouraged the reuse and recovery of this waste.

On the other hand, technological progress and the exacerbated consumerism of the population have led to an explosive development of the production of "smart" equipment and gadgets, stimulated by fierce competition in the field. These new devices incorporate state-of-the-art technologies, increasingly sophisticated, but which no longer have time to prove their usefulness, correct functioning and, above all, reliability, significantly contributing to the increase in the quantities of WEEE produced.

Regardless of the generation of equipment that is transformed into WEEE, they invariably include in their structure, printed circuit boards (PCB), which are transformed into PCB waste (WPCB). The composition of WPCB includes a series of recyclable materials such as metals: copper, tin, lead, iron, nickel, zinc and others. DPCI also contains recyclable plastics. By recovering them (metals, plastics), they can be reintroduced into the production process. Since metal ore resources are limited today, WEEE are considered “surface mines” to partially cover the demand on the world market.

1. Bibliographic study

The evolution of total WEEE production will lead to an increase of 30% in the coming years [1], as a result of advances in materials technology, manufacturing processes, rapid market penetration and planned obsolescence. However, a significant portion of WEEE generated globally is not recycled [2, 3, 4]. They contain a variety of metals, among which the lanthanides (La, Ce, Pr, Nd, Gd, Dy), precious metals (Au, Ag, Pd, Pt) stand out [5], hazardous metals (Hg) [6], metals with high economic value (Cu, Sn, Pb, Ni, Fe, Zn, Al), but also plastics [7, 8, 9].

Printed circuit boards (PCBs) that are an integral part of electronic products are manufactured in large volumes worldwide [10]. The concentrations of precious metals contained in waste PCBs are over 10 times higher than those in the richest ores, so that PCIs have also been called "urban mineral resources". In Figure 1.3. on the Periodic Table of the Elements, the base metals (green) and noble metals (red) most frequently present in PCBs are marked. [11, 12].

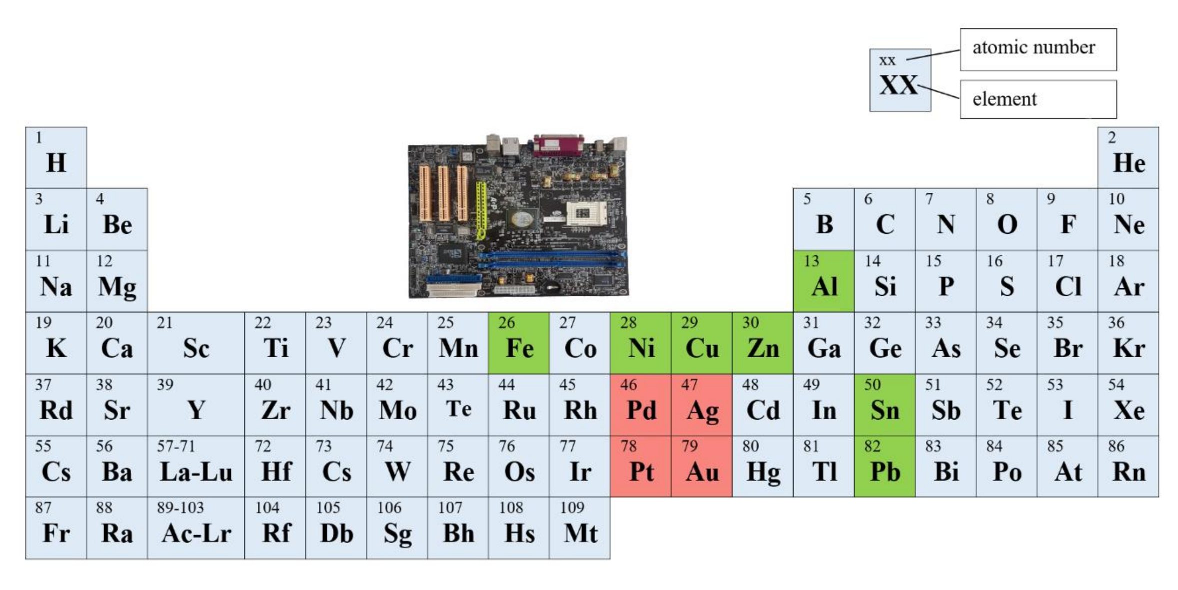


Figure 1.3. Base and noble metals of greatest interest in printed circuit boards.

PCBs represents approximately 3% of the total volume of WEEE and contains approximately 30% metals and 70% non-metals [13, 14].

2. Recycling materials from WPCB

The lifespan of electrical and electronic equipment, including PCBs, is relatively short and continuously decreasing, as a result of rapid changes in characteristics and performance [15, 16].

Due to the quantities of metals that DPCI presents, their recycling can be considered the best solution both in terms of environmental impact and economic point of view [17, 18, 19, 20].

As a rule, for higher separation efficiencies, more complex technologies are applied, consisting of a succession of several operations: DPCI grinding; magnetic separation of ferrous components from the ground mixtures; gravitational separation; electrostatic separation and magnetic separation of non-magnetic metals [21].

Pyrometallurgical Process (PP) technologies used for ore processing are also used in the recycling of WEEE [22] including DPCI which are burned in a furnace to remove plastics, and the metals are found in a solid material in the form of predominantly metal oxides.

Hydrometallurgical processes (HP) have shown extensive achievements in the recovery of metals from WPCB [23], especially base metals (BM): Cu Fe, Ni, Zn, Sn and Pb [24]. HPs are easy to control, environmentally friendly, involve low capital cost and provide a more advanced recovery of metals compared to other processes [25, 26].

Biological Processes (BP) use microorganisms, such as bacteria and fungi, to extract and recover metals from various aqueous solutions resulting from the solubilization of WEEE [27, 28]. Microorganisms have the ability to bind metal ions present in the external environment to the cell surface or to transport them into the cell. BP are still poorly known and applied. Bioleaching has been successfully applied for the recovery of precious metals and copper from ores [29].

Since the research in this doctoral thesis is focused on electrochemical methods involved in recycling of DPCI, the specific aspects of **Electrochemical Processes (EP)** are further developed. EPs are also increasingly involved in: environmental pollution prevention, pollutant monitoring, waste recycling, energy conversion and storage [30, 31, 32, 33, 34, 35, 36, 37, 38]. Due to the high performance of technologies based on combined processes, the research carried out within the framework of this doctoral thesis was oriented in this direction.

3. Conclusions of the bibliographic study

Based on literature data, the global production of WEEE has reached tens of millions of tons. It is estimated that approximately 35% of WEEE is collected from the EU. Due to the high concentrations of metals in this waste, they are considered "urban mines" becoming a secondary source of (metallic) raw materials. For these reasons, PP, HP, BP are today present in WEEE recycling technologies, often being preferred hybrid technologies by combining the above and involving EP.

II. Personal contributions

The bibliographical documentation presented in the first part of the thesis was carried out with the aim of establishing original research directions.

It is worth noting that this original research was part of a complex project called: Innovative technologies for advanced recovery of materials from waste IT and telecommunications equipment. (PN-III-P1-1.2-PCCDI-2017-0652, Financing contract nr. 84PCCDI / 2018), coordinated by the Technical University of Cluj-Napoca (**UTCN**) having partners: Babeș Bolyai University (**UBB**), INCDO-INOE 2000 Branch Research Institute for Analytical Instrumentation (**IC-INOE**), "December 1, 1918" University of Alba-Iulia (**UAB**), Gheorghe-Asachi Technical University of Iași (**UTIASI**) and the National Research Institute for Chemistry and Petrochemistry (**ICECHIM**).

Specifically, the doctoral thesis contains the results obtained by us within the UBB Component Project: Efficient and environmentally friendly electrochemical and chemical technologies for advanced recovery of materials from printed circuit board waste (WPCB), originating from IT and telecommunications equipment.

The main objectives of the original research developed further were:

- Establishing the conditions for solubilizing metals on WPCB and the methods for regenerating leaching solutions in parallel with the galvanostatic electrodeposition of solubilized metals;
- Modeling enhanced mass transport in a tubular electrochemical reactor;
- Selective recovery of Cu, Sn and Pb in potentiostatic regime from solutions resulting from the solubilization of metals on WPCB;
- Optimization of preparation and electrochemical regeneration of leaching solutions;
- Selective recovery of Fe, Ni and Zn from the resulting solutions after the prior electrochemical extraction of Cu, Sn and Pb;

- Optimization of the metal solubilization process in WPCB by on-site generation of HBr.

All these objectives would allow the development of an ecological and original technology for recycling Cu, Sn, Pb, Fe, Ni and Zn metals from IT and telecommunications equipment waste and its integration into a circular economy and ensuring sustainable development.

4. General experimental conditions

For the experiments and the interpretation of the obtained data, appropriate experimental stands equipped with commercially purchased or manufactured equipment were designed. The main equipment used are: Potentiostat / Galvanostat DXC236, Potentiostat / Galvanostat DXC240, Consort C863 multi-parameter analyzers, electrochemical sensors (pH for ORP and reference electrodes), AVANTA PM atomic absorption spectrometer, pumps and computers equipped with data acquisition boards. Details are presented at the beginning of each chapter (see. Chap. 5-12).

Experimental techniques used are: Electrochemical techniques based on voltammetry measurements (linear and cyclic voltammetry, hydrodynamic voltammetry with linear or cyclic potential sweep) or electrolysis in potentiostatic and galvanostatic regime. Quantitative determinations of the different species were performed by atomic absorption spectrometry (in flame, in graphite furnace or with inductively coupled plasma). Electrochemical analysis and monitoring techniques included: pH-metry, measurement of potentials reported to a reference electrode and conductometry. Aqueous solutions were obtained from case to case using distilled water and a.p. or chemically pure reagents.

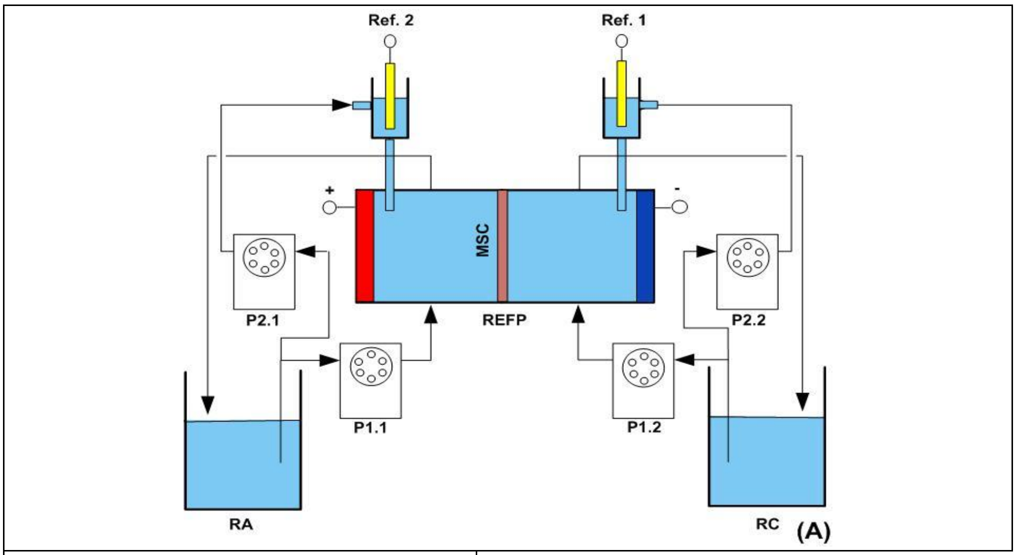
For the vast majority of the experiments carried out during the development of this doctoral thesis, the equipment control and experimental data acquisition were carried out by means of computers equipped with data acquisition boards produced by National Instruments (USA) and using the LabView 8.5 software developed by the same company. The experimental data processing was carried out using the ORIGIN 2018 program (OriginLab Corporation, USA) and Excel (Microsoft, USA).

5. Preliminary tests on the regeneration of the leach solution in parallel with MB leaching and galvanostatic electrodeposition of leached metals

Most of the previous studies conducted within the research team that targeted the electrochemical recovery of BM from WPCB [32 - 35] proposed and tested configurations that assumed that a single electrolyte would sequentially (in series) flow through → (i) the chemical leaching reactor → (ii) the cathodic compartment of the REC (for electro-extraction of leached metals) → (iii) the anodic compartment of the REC (for regeneration of the leaching solution).

In contrast to the previously mentioned approaches, during the current research, a different approach was decided, which involved the complete separation of the electrolyte flows from the anodic and cathodic circuit and the evaluation of the main performance parameters under the new conditions.

Before testing the REC – RCL tandem, we considered it imperative to evaluate the influence of the hydrodynamic regime and the potentials imposed on the electrodes on the current density resulting from REC. The schematic of the experimental stand used for this purpose is presented in Figure 5.1.A, along with a detailed photograph of the constructive elements of the REFP (see Figure 5.1.B) Micro Flow Cell® model (Electrocell, Denmark).



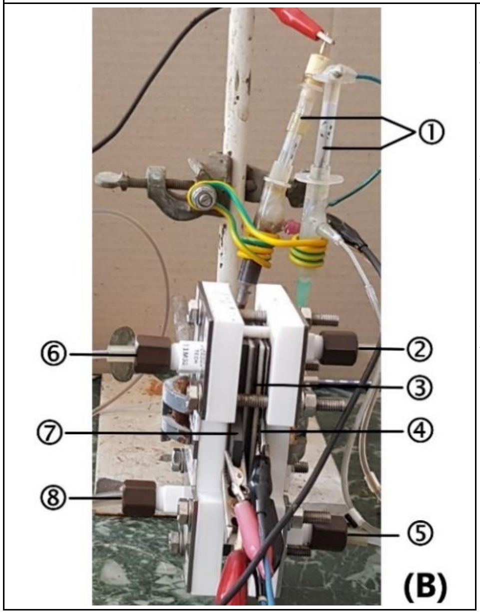
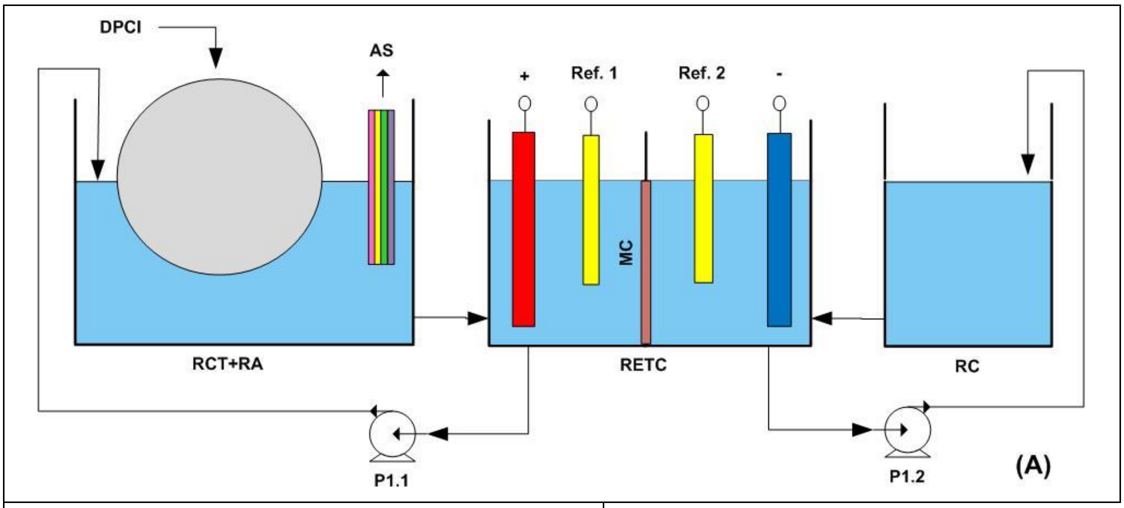


Figure 5.1. Schematic of the experimental stand for evaluating the influence of the hydrodynamic regime using REFP (B) and image of the essential elements of REFP model Micro Flow Cell® (B). A: MSC – Cation exchange membrane; P1, P2 – Peristaltic pumps; RA – Anolyte tank; RC – Catholyte tank; REFP – electrochemical filter press reactor. B: 1 – Reference electrodes; 2 – Anolyte outlet port; 3 – Cation exchange membrane; 4 – Stainless steel anode; 5 – Anolyte inlet port; 6 – Catholyte outlet port; 7 – Graphite plate cathode; 8 – Catholyte inlet port.

The experimental stand for preliminary tests of simultaneous leaching with regeneration of the leach solution and electrodeposition of metals used for these preliminary tests includes, as an essential element, as indicated in the diagram in Figure 5.2.A and the image in Figure 5.2.B, a polypropylene compartmentalized tank type REC (RETC) with a total volume of 1 L (7 in Figure 5.2.B).



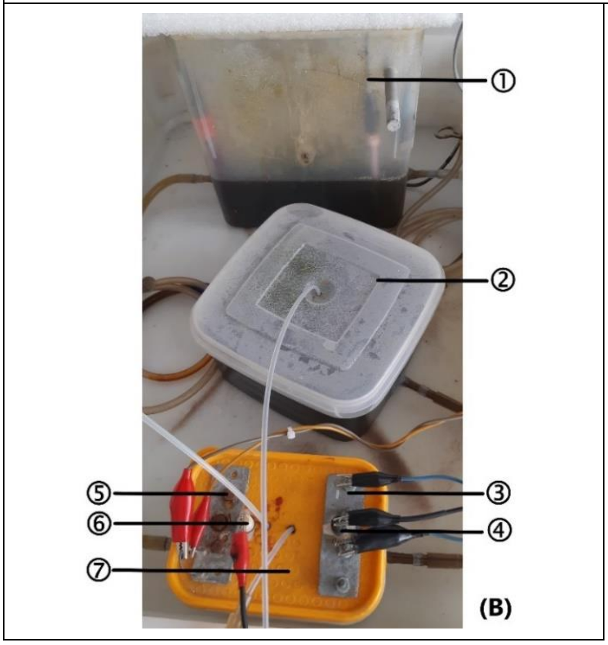


Figure 5.2. Scheme of the experimental stand intended for preliminary MB leaching tests, simultaneously with regeneration of the leaching solution and electrodeposition of leached MB (A) and partial image of the experimental stand (B): 1 – Chemical reactor with drum; 2 – Catholyte tank; 3 – Cathode assembly; 4, 6 – Reference electrodes; 5 – RETC anode; 7 – RETC body.

The decision to use the bromine-bromide system in the proposed process for the electrochemical recovery of all materials from DPCI is based on the fact that, in contact with a solution containing bromide ions, elemental bromine forms a soluble complex, according to the reaction:

$$Br_2 + Br^- \leftrightarrow [Br_3]^- \qquad (5.1)$$

In this way, the discomfort and dangers generated by the pronounced tendency of elemental bromine to vaporize are avoided. Furthermore, at the RCT level, elemental bromine or in the form of the $[Br_3]^-$ complex has the ability to oxidize most of the BM present in WPCB, according to the following general reactions:

$n/2Br_2 + Me \rightarrow Me^{n+}Br_n$ (5.2)

$n/2\ [Br_3]^- + Me \rightarrow Me^{n+}Br_n + n/2Br^-$ (5.3)

During the leaching process, as Fe^{3+} and Cu^{2+} ions appear in solution, they can oxidize more electronegative metals, including their own metal atoms, according to the following general reactions:

$n\ CuBr_2 + Me \rightarrow Me^{n+}Br_n + n\ CuBr$ (5.4)

$n\ FeBr_3 + Me \rightarrow Me^{n+}Br_n + n\ FeBr_2$ (5.5)

Even though CuBr is insoluble in simple aqueous solutions, in the presence of a significant concentration of Br^- ions, the Cu^+ ion is complexed, generating a series of soluble species, according to the general equation:

$CuBr + (m\text{-}1)Br^- \rightarrow [CuBr_m]^{1-m}(aq)$ (5.6)

According to the experimental arrangement described in Figure 5.2, in parallel with the leaching process, the resulting solution, enriched with BM ions dissolved in the RCT, is recirculated through the CoA of the RETC, where the electrochemical regeneration of the oxidative power of the leaching solution takes place thanks to the following processes:

$2\ Br^- - 2e^- \rightarrow Br_2(aq)$ (5.7)

$Fe^{2+} - e^- \rightarrow Fe^{3+}$ (5.8)

$[CuBr_m]^{1-m}(aq) - e^- \rightarrow [CuBr_m]^{2-m}(aq)$ (5.9)

where, depending on the oxidation state of the Cu ion and the concentration of bromide ions, m can take values between 2 and 4 [39]. In particular, considering the specific conditions in which the experiments were conducted ($[Br^-]$ of about 2.5 M), the cuprous ion is found predominantly in complexes with m = 2 and 3, and the cupric ion in the form of complexes with m = 3 and 4.

Exceptionally, if RETC is exploited inappropriately, at the CoA level the unwanted secondary process of water oxidation may start, according to the equation:

$2\ H_2O - 4\ e^- \rightarrow O_2\ (g) + 4\ H^+\ (aq)$ (5.10)

Under the conditions of maintaining a high concentration of Cu ions in the catholyte and if the RETC is operated adequately, in its CoC the electrodeposition process of metallic Cu takes place, predominantly, according to the reaction:

$[CuBr_m]^{1-m}(aq) + e^- \rightarrow Cu^0 + mBr^-$ (5.11)

If RETC is exploited inadequately (excessive current density) or the concentration of Cu ions is very low, electrodeposition of other leached MB is favored, according to the general reaction:

$$Me^{n+} + ne^{-} \rightarrow Me^{0} \qquad (5.12)$$

this unwanted secondary process leading to the impurity of the obtained Cu deposits.

The main average electrochemical parameters and the performance parameters of the overall process were evaluated, the results obtained for 4 experiments (Exp.05.01 ÷ Exp.05.04) being compared in Table 5.3, in which: the index "M" (e.g. $X_{Y, M}$) indicates the average value of the X_Y parameter; m_{DEP} represents the total mass of each cathodic deposit obtained and the indices "A" and "C" correspond to the performance parameters associated with the anodic and cathodic processes, respectively.

Table 5.3. Average values of electrochemical and performance parameters associated with the 4 preliminary experiments aimed at regeneration of the leaching solution in parallel with MB leaching and galvanostatic electrodeposition of leached metals.

Experiment	**Exp.05.01**	**Exp.05.02**	**Exp.05.03**	**Exp.05.04**
$I_{EL, M}$ (A)	2.00	1.99	2.99	4.00
m_{DEP} (g)	14.97	58.87	51.75	61.59
$i_{CE\ M}$ (A/dm^2)	2.04	2.03	3.05	4.08
$E_{EL, M}$ (V)	0.942	0.998	1.091	1.339
$E_{CE, M}$ (V)	-0.263	-0.285	-0.237	-0.429
$U_{B, M}$ (V)	3.768	3.765	5.255	8.525
pH_M	0.03	0.05	0.01	0.01
ORP_M (V)	0.427	0.403	0.392	0.475
$r_{F, A}$ (%)	272.0	92.9	96.3	59.7
$r_{F, C}$ (%)	72.5	95.8	87.6	32.5
$W_{S, A}$ (kWh/t)	148	385	518	1356
$W_{S, C}$ (kWh/kg)	2.47	1.66	2.53	11.07

The obtained results indicate that the similarity of the anolyte and catholyte allows their mutual replacement, a beneficial operation also to avoid the risk of anolyte saturation and catholyte depletion; and the total separation of the anodic electrolyte flow from the cathodic one and the imposition of reasonable operating parameters (i_{EL} = 100 ÷ 300 A/m^2) determines the obtaining of very advantageous values of faradaic efficiency and specific electrical energy consumption, significantly superior to the values obtained previously [40, 41].

6. Experimental measurements and modeling of enhanced mass transport on RET

The results that will be presented in this chapter aim to optimize the metal electrodeposition process by enhancing mass transport at the electrode surface. The most commonly used experimental technique for studying mass transport in electrochemical processes is EDR (see chapter 4).

Since the use of EDR for mass transport enhancement in RE in industrial facilities is not feasible, a new RE that provides enhanced mass transport, hereinafter referred to as the tubular electrochemical reactor (RET), was designed and used. Thus, the RET was tested with synthetic solutions in order to evaluate the feasibility of its use for copper electrodeposition from solutions resulting from WPCB processing.

The experimental stand scheme is shown in Figure 6.1. and includes the RET, the drum chemical reactor (RCT), the catholyte (RC) and anolyte (RA) tanks with the associated sensor systems (AS1) and (AS2) respectively and the recirculation pumps P1 and P2. Construction details of the RET are shown in Figure 6.2 (A-C). The RET dimensions are: total length 38.5 cm, the diameter of the outer polypropylene (PP) tube is 4.64 cm and of the inner tube 3.20 cm.

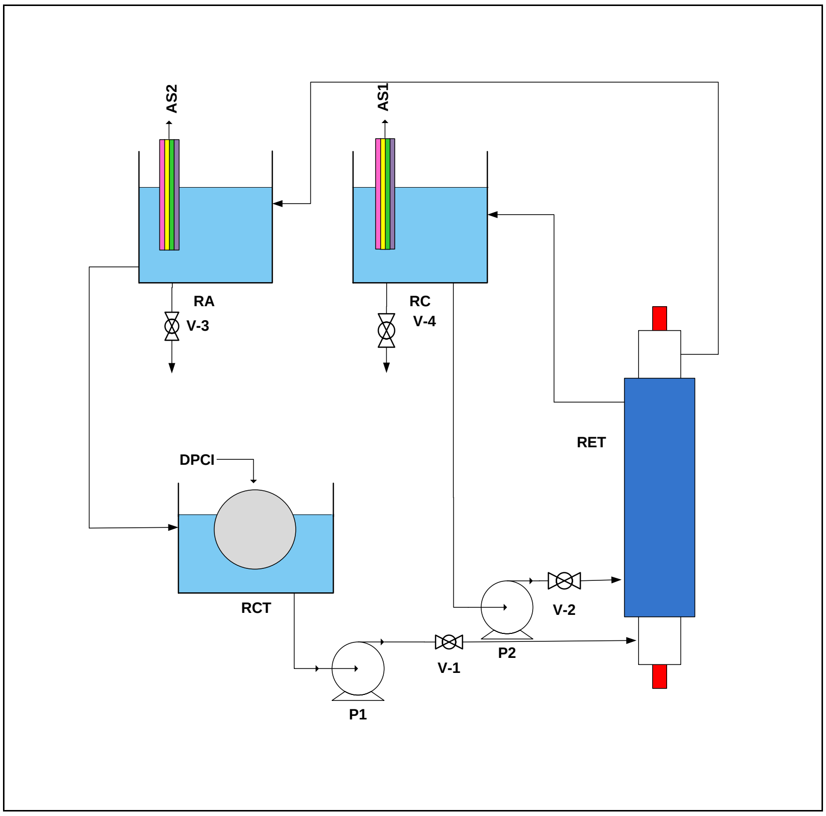


Figure 6.1. The facility designed for determining mass transport parameters in RET.

Strictly for the measurements related to mass transport in the RET, a non-compartmentalized configuration of the RET was used (Figure 6.3.) and the synthetic solution

(0.5 M $CuSO_4$ + 1 M H_2SO_4) identical to that used for the measurements on the EDR. This solution was recirculated from the electrolyte reservoir (RE). The experimental technique applied was voltammetry hydrodynamic cyclic potential sweep (VHBCP). We also specify that a copper foil was used in both the cathode and the anode fabrication, ensuring the reproducibility and accuracy of the measurements without the risk of modifying the concentration of Cu ions.

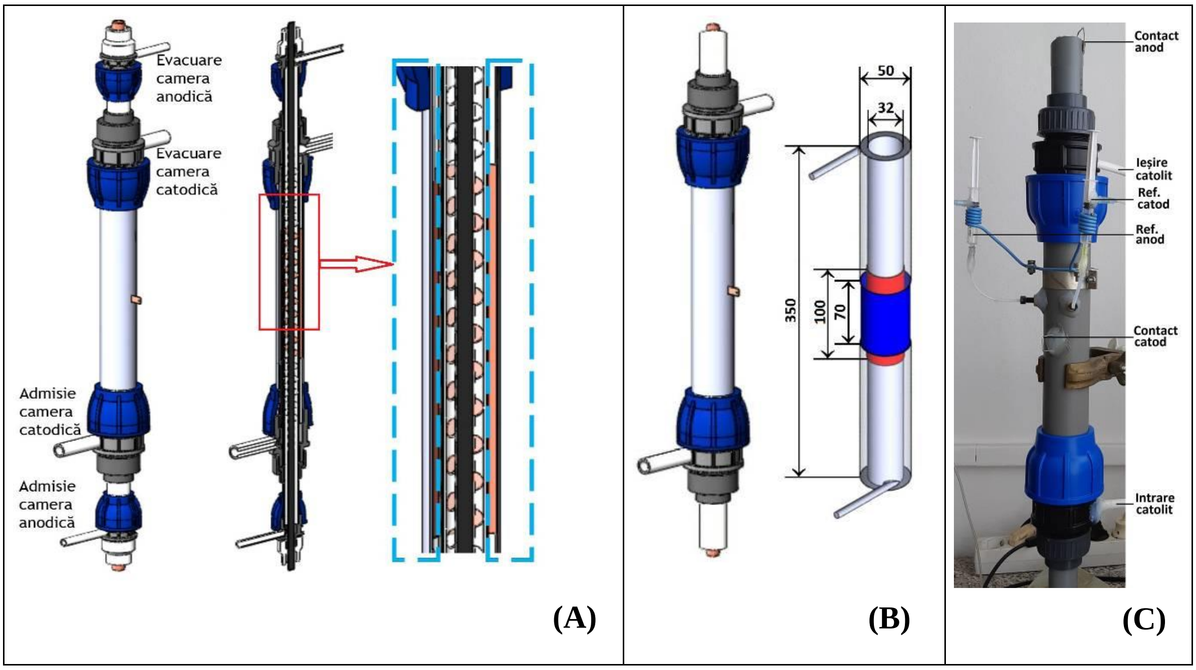


Figure 6.2. Construction details of the RET. (A) – Cross-section of the RET; (B) – Simplified RET scheme; (C) – Image of the tubular electrochemical reactor.

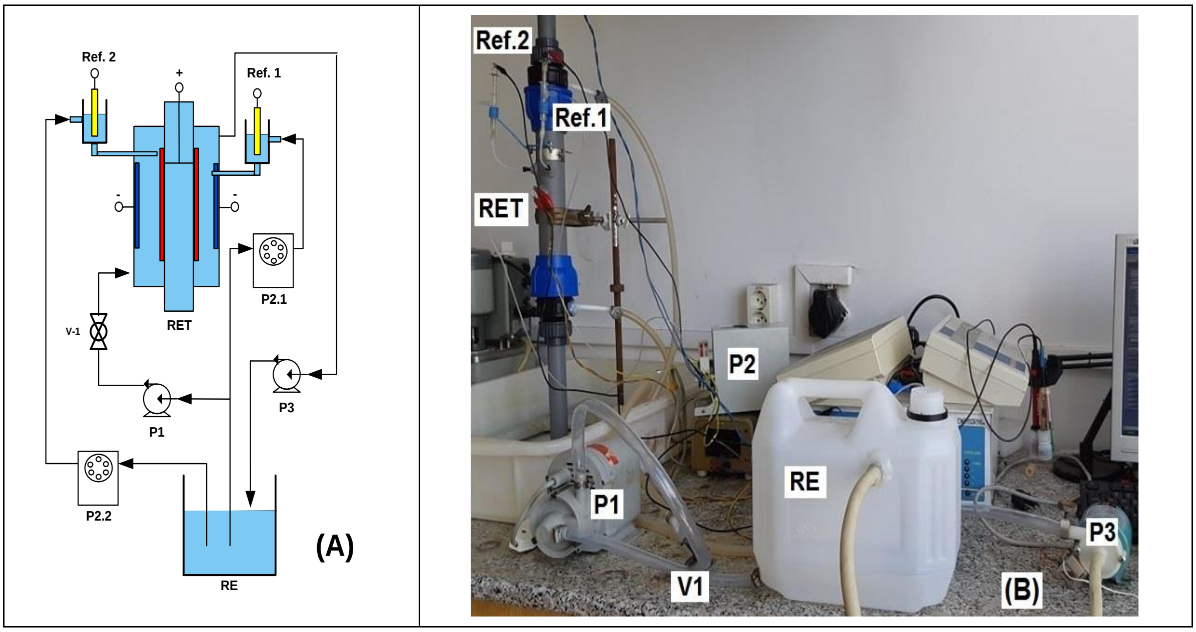


Figure 6.3. Schematic of the RET experimental setup (A) and an image of the experimental setup (B).

Based on all the experimental results presented previously, in collaboration with the Technical University of Cluj Napoca, the modeling and optimization process of the RET was started. The modeling of the electrolyte flow profile through the RET led to the conclusion that for the specified RET dimensions (see Figure 6.2) and electrolyte flow rates between 1 and 3.75 L/min (0.06 ÷ 0.225 m3/h), the flow profile at the level of the electrodes arranged at the middle of the reactor height is of the piston type, with a negligible tangential component [42, 43]. For this reason, for a solution containing less than 10 g/L Cu, even if the height of the cathode is only 7 cm, a significant change in the concentration of Cu ions occurs along it, as seen in Figure 6.8., and, implicitly, a significant increase in the thickness of the diffusion layer (δ).

With this new set of dimensional data, we returned to the modeling and optimization process of the RET in terms of the electrolyte flow rate in the cathode compartment. After several numerical simulations, we concluded that, using an electrolyte flow rate of 1500 L/h (25 L/min) and a dissolved Cu ion concentration at the RET inlet of 410 mol/m^3 (26 g/L or 0.41 M), Cu electrodeposition is performed uniformly and selectively, without significant differences in current density along the electrode height.

Research related to the interpolar separator aimed to create a material with appropriate properties and especially high electrical conductivity.

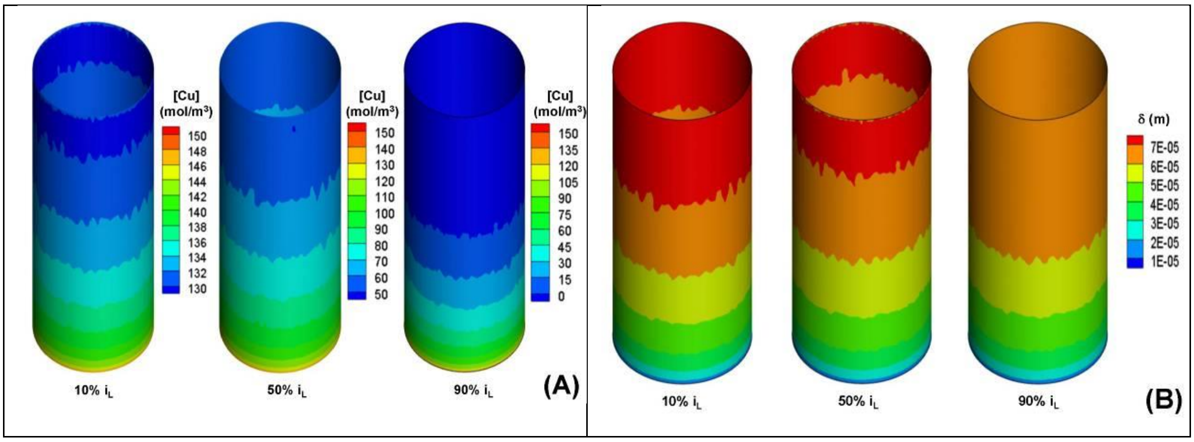


Figure 6.8. Numerical simulation results regarding the evolution of Cu ion concentration (A) and diffusion layer thickness (B) in RET at different fractions of the limiting current density (i_L), for an input concentration of 150 mol/m^3 Cu, an electrolyte flow rate of 3.83 L/min and an i_L value of 420 A/m^2.

For these reasons, our research was focused on the characterization of ceramic membranes (CM) from our laboratory equipment and also some made in the laboratory. Thus, the following were tested: Swiss-type Faience (FE), Unglazed Faience (FN), S-type Faience (FS). Also, MC tests were carried out in the laboratory from: Faience-type Slip (BF), Sandstone

Slip (BG), White Faience Slip (BFA), Raw Material-Powder (MP-P), Raw Material-EDR (MP-EDR) and Solid Ceramic Material (MCS).

The ohmic resistance measurements of the MC were performed at a current intensity of 0.15 A. Through the tests using the same solution in both compartments (electrolytic background (FE): 2 M KBr + 0.5 M. HBr and leaching solution (SL)), the data presented in Table 6.3 were obtained. where: G – MC thickness, R – MC ohmic resistance and σ is the electrical conductivity of the membrane.

Table 6.3. Parameters of ohmic resistance measurements of MC on FE and on SL.

Nr. crt.	**Name**	**G (cm)**	**R_{FE} (Ω)**	**σ_{FE} ($\Omega^{-1}cm^{-1}$)**	**R_{SL} (Ω)**	**σ_{SL} ($\Omega^{-1}cm^{-1}$)**
1.	Faience type Swiss(FE)	0,55	19,84	0,17	29,76	0,114
2.	Unglazed Faience (FN)	0,41	63,96	0,04	-	-
3.	Glazed Stoneware type S (GS)	0,37	-	-	61,52	0,037
4.	Faience type S (FS)	0,64	41,88	0,09	107,79	0,037
5.	Faience type Slip (BF)	0,36	39,17	0,06	42,54	0,051
6.	Sandstone Slip (BG)	0,32	36,34	0,05	35,92	0,058
7.	White Faience Slip (BFA)	0,37	7,20	0,32	7,75	0,302
8.	Raw Material-Powder (MP-P)	0,41	12,78	0,20	19,08	0,132
9.	Raw Material-EDR (MP-EDR)	0,35	7,75	0,28	10,45	0,212
10.	Solid Ceramic Material (MCS)	0,36	137,14	0,02	137,64	0,016

Subsequently, tests were also carried out on the solutions resulting from the leaching of exposed metals from 10 base plates [44, 45].

Based on the tests performed with the RET, the mass transport and electrode process parameters were established and then used in the numerical modeling and optimization. Finally, the RET dimensions were established, the positioning of the electrodes in it was determined in such a way as to ensure a perfectly developed flow and the piston-type flow was replaced with a uniform helical flow over the entire height of the cathode.

The creation and testing of MCs obtained from cheap raw materials demonstrated that those made from white faience slip (BFA) have maximum electrical conductivities on the electrolytic background (0.316 $\Omega^{-1}cm^{-1}$), respectively on the leaching solution (0.302 $\Omega^{-1}cm^{-1}$).

7. Research on selective and intensive electrodeposition of Cu in galvanostatic mode from real leaching solutions

As demonstrated by the results presented in Chapter 5, the use of a recycling system for MB from DPCI allows their leaching in parallel with the electrochemical regeneration of the leach solution and the electrodeposition of the leached metals. Starting from this level, we considered it absolutely necessary to investigate the extent to which, in the first stage of the recycling process, it is possible for the electrochemical regeneration of the leach solution to be carried out with the complementary process of directly obtaining a high-carrying Cu deposit, which can be exploited with maximum financial benefits.

Next, for the actual tests on the selective and intensive electrodeposition of Cu in galvanostatic mode from real leaching solutions, a dedicated installation was designed and assembled, the scheme and photo of which are presented in Figure 7.1. The main element of the new experimental stand was represented by the REC with a volume of 1 L made of polypropylene. The REC was equipped with a plate-type EL (as a cathode) with dimensions of 2 cm x 2 cm, made of SS 316 sheet with a thickness of 0.2 mm, and the EL face not exposed to the CE was insulated. For this set of measurements, 1 L of base solution (SB) was prepared and placed over 2 kg of WPCB, decanted and adjusted to pH -0.8 with 47% HBr. Following this treatment, the concentrations of metals of interest in the solution, determined by SAAF, were: Cu – 12 g/L; Sn – 6.9 g/L; Pb – 4.4 g/L; Zn – 9 g/L; Fe – 5.6 g/L; Ni – 0.8 g/L. A volume of 400 mL was taken from this solution, which was transferred to the REC as electrolyte. The results of tests performed by CPG on the intensive and selective electrodeposition of Cu from real leaching solutions, at values of the working electrode current (I_{EL}) of -80, -160, -240, -320 and -400 mA are presented in Table 7.2, respectively the composition of the deposits in Table 7.3.

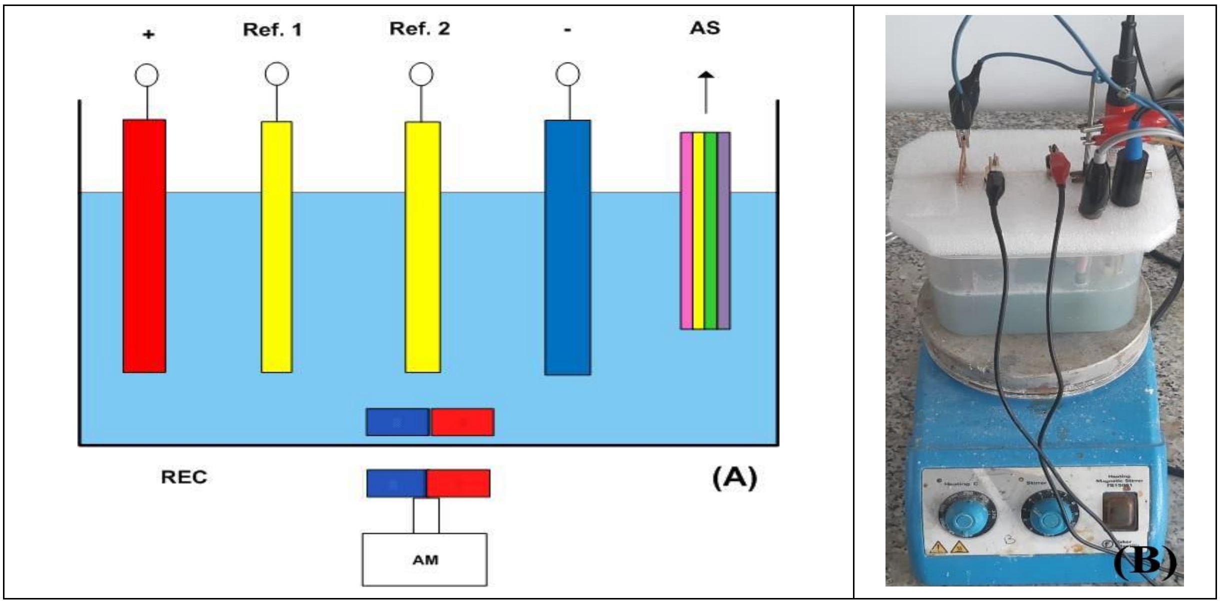


Figure 7.1. Simplified diagram of the experimental stand for studying the process of intensive selective electrodeposition of Cu from real leaching solutions (A) and image of the intensive Cu electrodeposition facility (B).

Table 7.2. Results obtained and experimental parameters used during intensive and selective electrodeposition tests of Cu from real leaching solutions [44].

Test	Deposit	I_{EL} (mA)	t (min)	i_{EL} (A/m^2)	r_F (%)	W_S (kWh/kg)	$E_{EL, M}$ (V/Ref)	ORP_M (V)
T07.01	**D07.01**	-400	127	1000	50.5	0.389	-0.363	0.047
T07.02	**D07.02**	-320	158	800	54.6	0.292	-0.346	0.031
T07.03	**D07.03**	-240	211	600	92.8	0.133	-0.343	0.020
T07.04	**D07.04**	-160	316	400	90.8	0.105	-0.311	0.018
T07.05	**D07.05**	-80	316	200	89.0	0.086	-0.287	0.032

Table 7.3. Percentage content of metals in deposits obtained by intensive and selective electrodeposition of Cu from real leaching solutions [44].

Deposit	Cu (%)	Sn (%)	Pb (%)	Fe (%)	Ni (%)	Zn (%)	Ag (%)
D07.01	98.93	0.60	0.10	0.01	0.02	0.21	0.10
D07.02	99.40	0.21	0.05	0.01	0.01	0.21	0.08
D07.03	99.67	0.09	0.03	0.00	0.00	0.15	0.05
D07.04	99.47	0.16	0.04	0.02	0.01	0.22	0.05
D07.05	99.68	-	0.04	0.01	0.00	0.22	0.04

The data obtained for the selective electroextraction of Cu in galvanostatic regime clearly show that this process can be parallel to the anodic regeneration of leaching solutions based on the $KBr/HBr/Br_2$ system.

The experimental results described in this chapter were disseminated in the form of a poster, presented at an international scientific event, specific to the field of chemistry and chemical engineering [46], as well as in the form of an ISI article [44].

8. Selective recovery of Cu, Sn, Pb in potentiostatic regime

In the natural continuation of the research, a new protocol for the selective potentiostatic electro-extraction of Cu and Sn and Pb alloys was tested. The main goal of the research was to evaluate the feasibility of the complete extraction of these metals from leaching solutions based on the KBr/HBr/Br_2 system. This original protocol consisted of a first stage of selective potentiostatic electro-extraction of Cu, followed by an intermediate stage of reducing the concentration of Cu ions by cementing them on metallic Sn, and the last stage consisted of the actual electrodeposition of Sn and Pb alloys.

The selective potentiostatic electrodeposition experiments of Cu and Sn and Pb alloys were performed in a facility whose simplified scheme is shown in Figure 8.1, the main elements included in the experimental stand being represented by a polypropylene RETC with a volume of 1 L divided by a porous ceramic membrane (MC), a polypropylene RA with a volume of 2 L and a Medorex TC type pump for anolyte recirculation.

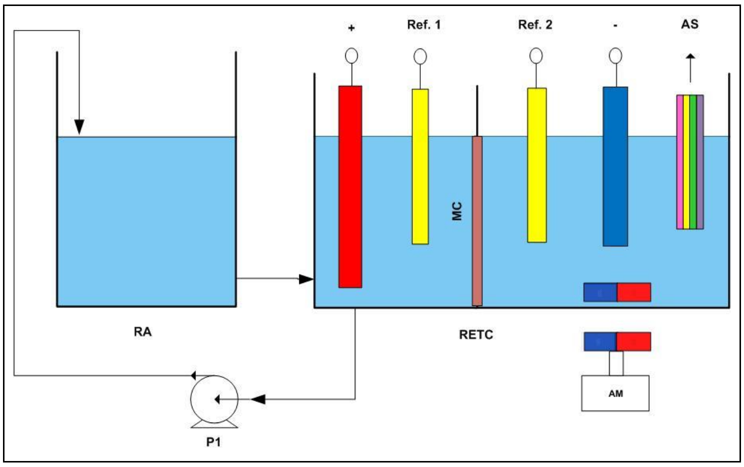


Figure 8.1. Simplified schematic of the experimental setup for potentiostatic slow selective electroextraction tests of Cu, Sn and Pb from real leaching solutions.

Following the tests performed, we concluded that the selective electrodeposition of Sn-Pb alloys, potentiostatically, from real leach solutions with extremely low Cu content, represents a feasible, efficient and cost-effective technology for advanced Sn and Pb recovery from WPCB.

Correlating the parameters evaluated during the experiments regarding the selective electrodeposition, in potentiostatic regime, of Cu and Cu and Sn-Pb alloys from the real leaching solution based on the $KBr/HBr/Br_2$ system, we came to the conclusion that the proposed and tested technologies are perfectly feasible, allowing, compared to other less ecological and efficient recycling processes cited in the literature, the obtaining of high quality and purity deposits, with a high commercial value.

The experimental results described in this chapter were disseminated in the form of an ISI article [45], as well as in the form of a poster, presented at an international scientific event, specific to the field of chemistry and chemical engineering [47].

9. Cu recovery from de-equipped WPCB

According to literature data [30 - 38] and our own results (see Chap. 5), the recovery of exposed BM from WPCB using the KBr/HBr/Br2 leaching system allows the total and unaltered removal of electronic and interconnect components from the boards, the remaining fraction being composed exclusively of 3 components: electrolytic Cu (in the form of traces), glass fiber and epoxy resin. Unfortunately, a significant mass of Cu (comprising between 5 and 10% of the total mass of WPCB) is inaccessible to the leaching process, being embedded in the WPCB structure or covered by the solder mask. To solve this problem, we proposed an original solution, consisting of three steps: (i) grinding the unequipped WPCB (WPCBD) and obtaining a ground WPCBD powder (PWPCBDM); (ii) leaching, from PWPCBDM, the Cu thus made accessible, using an extremely cheap and accessible leaching system that can be activated by simple air bubbling and (iii) recovering ultra-pure Cu by electrodeposition, in parallel with the regeneration of the pH of the leaching solution.

The simplified diagram of a proposed installation for implementing the last two stages described above is shown in Figure 9.1.

In general, the installation would include a drum chemical reactor (RCT) into which PWPCBDM and the leach solution (SL) taken with the P1 pump from the leach solution tank (RSL) are introduced. During the leaching step, the oxidative capacity of the SL is maintained by bubbling air into the RSL (or into the RCT), after which part of the dissolved Cu is extracted by electrodeposition at the CoC level of the RETC (divided by a MSC). Simultaneously with the electroextraction of excess Cu, a regeneration of the SL pH also takes place thanks to the protons coming from the CoA of the RETC via the MSC.

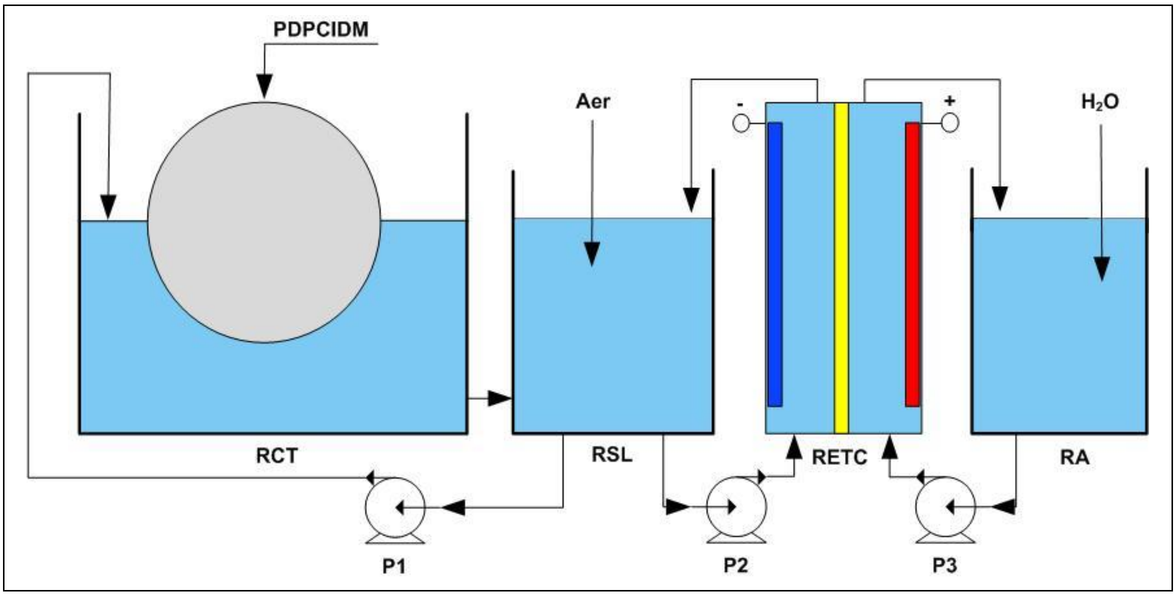


Figure 9.1. Proposed scheme for the Cu recovery plant from PWPCBDM.

For efficient and profitable extraction of Cu from PWPCBDM, a complexing leaching system based on extremely cheap and accessible raw materials (H_2SO_4, $CuSO_4$ and NaCl) can be used.

This system allowed for an efficient and complete solubilization of Cu without affecting the integrity of the composite matrix based on epoxy resin and glass fibers and without contaminating the resulting fraction with traces of MB or other compounds. Therefore, the proposed sequence of operations and processes can provide an integrated, efficient and environmentally friendly solution for the valorization of unexposed Cu from DPCI, being applicable in sustainable industrial scenarios with potential for scaling.

The results of the research described in this chapter were capitalized in the form of a patent application [48], and their corroboration with those presented in the previous chapters allowed their dissemination also in the form of two participations in scientific events in the field of invention and innovation [49, 50].

10. Preparation and electrochemical regeneration of leaching and neutralization solutions

As already presented in previous chapters, the use of the electrochemically renewable KBr/HBr/Br_2 redox system represents a feasible and advantageous alternative for the recovery of metals from WPCB. [51].

The experimental stand used for electrochemical regeneration of leach solutions is shown in Figure 10.1.

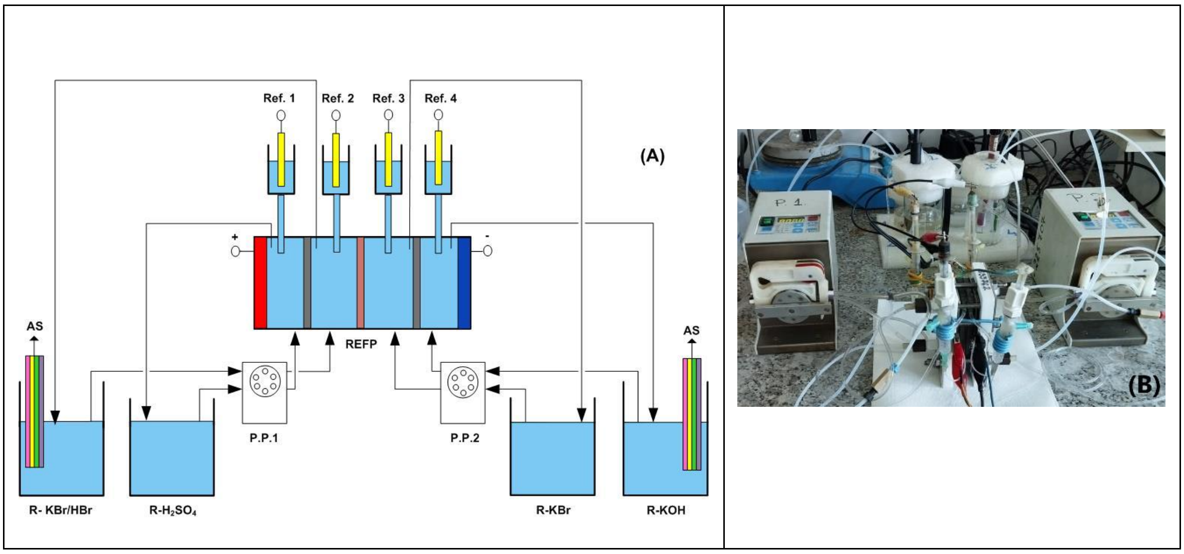


Figure 10.1. Simplified diagram of the experimental stand for electrochemical regeneration of leaching solutions, (A) and image of the experimental installation (B).

The electrochemical filter press reactor (REFP), (MicroFlowCell®), consists of 4 compartments, each with its own Ag/AgCl/KCl_{SAT} reference electrode (Ref.1-4) for potential monitoring.

Three complex electrochemical tests were performed in galvanostatic mode: Test 10.01. at 1A, Test 10.02 at 2A and Test 10.03 at 4A 10.3 (see Figure 10.2.) each structured in sequential stages with a uniform duration of 10 minutes. The tests performed at 1 A and 2 A had a total duration of 110 minutes, fully covering the 11 stages of 10 minutes each. In contrast, the test performed at 4 A was interrupted at 70 minutes, due to reaching the upper detection limit of the conductivity measurement system.

Based on the experimental data obtained, the values of the process performance parameter indicators were evaluated: current efficiency (r_F), specific energy consumption (W_S) and HBr and KOH concentration (see details in [**Error! Bookmark not defined.**]) presented for HBr in Table 10.1. and the indices "A" and "f" indicate that the corresponding terms represent the average or final values, calculated for the entire experiment, respectively at its end.

Table10.1. Average or final values of performance parameters for HBr electrosynthesis by electrodialysis using 4-compartment REFP.

Test	**i** **kA/m²**	**$r_{F,HBr,A}$** **%**	**$W_{S,HBr,A}$** **kWh/kg**	**$PR_{HBr,A}$** **kg/(m²*h)**	**$C_{M,HBr,f}$** **M**	**pH_f**
T10.01	1	63.4	27.5	1.89	0.43	-0.81
T10.02	2	62.4	36.0	3.76	0.74	-1.25
T10.03	4	71.0	40.1	8.55	0.77	-1.20

It is found from Table 10.1 that a fourfold increase in current density (i) leads to an average productivity of over 4.5 times higher (**$PR_{HBr,A}$**) and a slight improvement in the average faradaic efficiency (**$r_{F,HBr,A}$**), while the average specific energy consumption (**$W_{S,HBr,A}$**) increased by only 31.5%.

Similar data obtained for the electrosynthesis of KOH by electrodialysis are presented in Table 10.2.

Table 10.2. Average or final values of performance parameters for KOH electrosynthesis by electrodialysis.

Test	**i A/m^2**	**$r_{F,KOH,A}$ %**	**$W_{S,KOH,A}$ kWh/kg**	**$PR_{KOH,A}$ kg/(m^2*h)**	**$C_{M,KOH,f}$ M**	**λ_f mS/cm**	**T_f °C**
T10.01	1000	58,82	21,15	1,210	0,466	125,3	28,09
T10.02	2000	58,97	25,94	2,453	0,824	189,5	32,58
T10.03	4000	70,16	28,99	5,834	0,893	200,8	42,70

The evolution of the performance parameters for KOH electrosynthesis by electrodialysis is very similar to that of HBr, with the best results being obtained at the highest level tested. Current density (4 kA/m^2). Due to the better selectivity of MSC compared to MSA, the target KOH concentration (0.5 M) can be reached after only 24 min. Excellent values were also obtained for $r_{F,KOH,A}$ and $PR_{KOH,A}$, while a 4-fold increase in current density increases the value of $W_{S,KOH,A}$ by only 17.7%, but multiplies it by 5.7 times.

The results obtained regarding the simultaneous electrosynthesis of HBr and KOH by electrodialysis contribute to the optimization of the DPCI recycling process because the Br-/Br_2 leaching system is regenerated in situ and KOH will be used in the overall recycling process for pH adjustment.

Given that the proposed process can be fully automated and the necessary reagents are produced only on demand, we conclude that the savings obtained will be sufficient to cover the depreciation, operation and maintenance costs. Also, the overall carbon footprint of the proposed technology will be significantly reduced, and the process can be considered a substantial component within the circular economy concept.

11. Selective recovery of Fe, Ni, Zn

The research that will be presented in this chapter has aimed at the recovery of other base metals such as Fe, Ni and Zn (**FNZ**) present in WPCB that are solubilized but remain in the leaching solutions, after the recovery of Cu Sn and Pb, due to their low market value and high extraction costs. That is why there is little research that aims at their extraction [52, 53, 54, 55]. The relevant experiments were done on rotating disk electrodes (RDE), made of Pt, carbon paste (CP) and type 304 stainless steel (304 SS). [56]. The experimental techniques used were linear voltammetry, cyclic hydrodynamic voltammetry (CHV) and potentiostatic chronoamperometry (CAP). The experimental stand structure, the protocols used for cathode cleaning and weighing, mineralization and analysis of the obtained deposits were similar to those described in Section 8.1. and presented in [45].

For long-term CAP experiments, an electrochemical reactor was built that has a rotating disk electrode on top that is separated from the anode assembly by a horizontal ceramic membrane (Figure 11.1.).

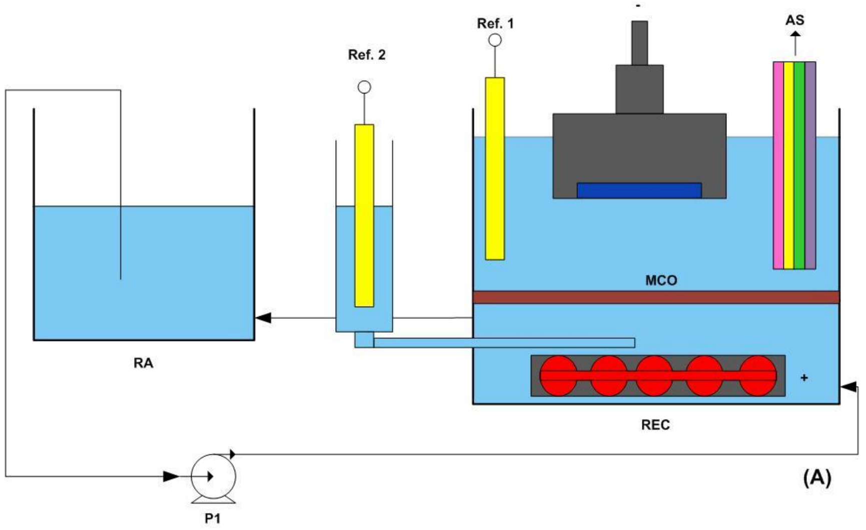


Figure 11.1. **(A)** Schematic of the experimental stand for the electroextraction of Fe, Ni and Zn from leach solutions: RA – anolyte tank; REC – electrochemical reactor; MCO – horizontal ceramic membrane; P1 – peristaltic pump; Ref1 - Ref2 – Reference electrodes; EDR – rotating disk electrode; AS – sensor assembly (pH and temperature) and **(B)** image of the experimental installation.

The electrodeposition efficiency (EED) was evaluated for all tests by relating the amount of anodic current to the amount of cathodic current. The calculated values are presented in Table 11.3.

Excluding Pt (expensive electrode material) from the data presented in Table 11.3. it is observed that for 304 SS and CP the maximum EED values of 86.8% and 95.8% respectively are obtained at pH values of 2 and 3 for $\mathbf{E_{\hat{i}nt}}$ -1.2 and -1.3 V/RE respectively.

Tests performed in synthetic solutions, using EDRs (ϕ = 3 mm) made of CP or 304 SS, showed that, by appropriately choosing the pH and cathodic potential, Zn, Fe and Ni can be selectively extracted. The EED values of the metal varied between 8% and 95.17%.

Table 11.3. EED evaluated by VCH for electrodeposition of FNZ on EDR (ϕ = 3 mm) of Pt, 304 SS and CP in the ternary synthetic solution for different values of $E_{\hat{i}nt}$ and pH.

Material	**pH**	**$E_{\hat{i}nt}$ (V/ER)**			
		-1,1	**-1,2**	**-1,3**	**-1,4**
Pt	**0**	17,5	26,7	30,8	21,7
Pt	**1**	52,6	77,6	70,5	NA
Pt	**2**	77,4	86,5	87,6	78,4
Pt	**3**	90,1	93,4	87,9	75,0
304 SS	**0**	32,8	39,4	32,7	27,0
304 SS	**1**	63,5	81,2	68,6	67,5
304 SS	**2**	68,2	86,8	84,4	81,2
304 SS	**3**	73,4	80,8	85,8	72,7
CP	**0**	47,6	66,5	66,6	30,9
CP	**1**	51,9	86,5	87,6	80,1
CP	**2**	38,9	81,4	90,8	91,1
CP	**3**	72,1	89,1	95,8	95,5

Based on long-term tests (0.15 ÷ 8 h) with EDRs (ϕ = 38 mm) made of CP or 304 SS, it was demonstrated that with an appropriate polarization of the cathode and adjustment of the pH of the solution, high-purity Zn deposits and Fe-Ni-Zn alloys with purity over 98% can be obtained. Although EDRs made of CP or 304 SS are useful in laboratory studies, their industrial use is not feasible. The use of rotating cylindrical electrodes made of 304 SS would be a useful alternative, including for the circumstance that the deposits obtained would not be sufficiently adherent to the electrode.

The composition of electroextracted Fe-Ni-Zn alloys is highly dependent on experimental parameters, such as the composition and pH of the electrolyte solution, the applied cathodic potential (E_{EL}) in the case of potentiostatic operation.

12. Tests to evaluate the possibility of initiating the leaching process of MB exposed on DPCI in the absence of the initial addition of Br_2

Most of the previously described experiments targeting the selective electroextraction of MB from real leach solutions (see Chapters 5, 7, 8 and 11) were based on the exploitation of recovered electrolytes, which resulted from the chemical dissolution of BM exposed from WPCB using mixtures of KBr and HCl, in which pure Br^2 was added as an oxidizing agent, in the liquid state. Unfortunately, if the studied processes are to be implemented on an industrial scale, this approach is inadequate for at least two reasons:

- The presence of Cl^- ions in the leaching solution induces the risk of Cl_2 gas release during the electrochemical regeneration step of the oxidative power of the leaching solution because, unlike Br_2, Cl_2 is extremely poorly soluble, not forming complexes with the halide ions present.
- Under normal conditions of temperature and pressure, pure Br_2 is found in a liquid state and is extremely volatile, conditions in which its acquisition, transportation, storage and handling become extremely difficult and dangerous operations, involving major risks of work accidents and environmental pollution.

In order to eliminate the two shortcomings mentioned above, the research described in this chapter aimed at evaluating the possibilities of initiating the leaching process of MB exposed on WPCB in the absence of Cl^- ions and the initial addition of pure Br_2.

The experimental stand used to carry out these experiments is identical to the one presented in Chapter 5, with the caveat that, compared to the configuration already presented, during the last tests, the cathode formed by the 4 cylindrical graphite bars was replaced with a graphite block identical to the one used as the anode.

The results of the research described in this chapter represent a superior exploitation of the studies included in the previous chapters. Thus, taking into account the fact that HBr, at process-adapted concentrations, can be synthesized on-site by electrodialysis (see Chapter 10), it becomes evident that the leaching process of exposed MB from DPCI can be started using cheap, accessible and safe to handle reagents such as KBr, KOH, H_2SO_4, $CuSO_4$ and Na_2CO_3, eliminating the need to use pure liquid Br_2.

For the starting stage, the initial leaching solution can be represented only by SES (KBr 2 M + HBr 0.5 M), while in the cathodic flow, a SES solution can be used in which $CuBr_2$ is added so as to ensure a Cu ion concentration between 10 and 180 g/L.

The periodic inversion of the anolyte and catholyte solutions involved in the leaching process of exposed MB from the DPCI, carried out in parallel with the electrochemical regeneration of the leaching solution and the selective electrodeposition of Cu, ensures the successive and complete processing of at least 5 batches of DPCI at a solid:liquid ratio of 1:8.

Based on this protocol, the obtained Cu cathodic deposits have high purity, allowing their rapid and cost-effective utilization.

For an efficient implementation of the tested process at an industrial level, it is necessary to optimize the RETC geometry and operating parameters, an activity that can improve the extremely promising performance parameters already evaluated.

The research results described in this chapter were partially capitalized in the form of participation in scientific events in the field of chemistry and chemical engineering [57].

13. General conclusions

The literature review (see Chapter 2) allowed for a brief description of the main technologies for recycling metals from WPCB, highlighting their advantages and disadvantages. The conclusion of the study is that in order to achieve an innovative and environmentally friendly technology, it is necessary to involve electrochemical processes that allow obtaining metals (individual or alloys) of high purity at the end of WPCB recycling. In addition, electrochemical processing using aqueous solutions of WPCB minimizes the generation of volatile toxic substances, allows operation at reasonable temperatures (> 100 oC) and the creation of closed circuits in order not to unnecessarily increase the volume of solutions and the quantities of reagents used.

Previous research conducted for the development of this doctoral thesis has pursued the electrochemical recovery of MB from DPCI [30 - 38] by performing the following steps in series: (i) solubilization of MB in the chemical leaching reactor; (ii) cathodic electroextraction of MB from the leaching solution in a compartmented electrochemical reactor (REC) and (iii) regeneration of the leaching solution by passing it into the anode compartment of the REC. This approach had the disadvantage of inevitable electrochemical short circuit with negative consequences on the efficiency of the overall process and a high specific electricity consumption. In order to minimize the disadvantages of series operation, the research within this thesis began with laboratory tests in which the complete separation of the electrolyte flows from the anode and cathode circuits was performed. Thus, by imposing reasonable operating parameters (i_{EL} = 100 ÷ 300 A/m^2) they led to faradaic efficiencies and specific electricity consumption, significantly superior to the values previously obtained [40].

The initial results obtained in this way (see Chapter 5) encouraged us to carry out a more in-depth study which was presented in detail in Chapters 6 - 12.

The EDR tests presented in Chapter 6 allowed the determination of the specific mass transport parameters under the experimental conditions (limiting currents and diffusion coefficient of Cu^{2+} ions) and the establishment of the optimal current efficiency in the case of an enhanced mass transport. However, since the EDR is not suitable for large REs, a tubular electrochemical reactor (RET) with upward circulation of the electrolyte was designed, manufactured and tested. The experimental data obtained on the RET also served for numerical modeling and optimization. Based on experimental data and modeling, the RET dimensions and electrode positioning were established in such a way as to ensure a perfectly developed flux and a uniform helical flow over the entire height of the cathode. Ceramic membrane type interpolation separators were also made and tested, made of cheap raw materials (for example, white faience slip (BFA)), with a good electrical conductivity (0,316 $\Omega^{-1}cm^{-1}$ in the electrolytic background and 0,302 $\Omega^{-1}cm^{-1}$ on the leaching solution).

The successive electroextraction of base metals from the leach solution has been studied in detail: for Cu in Chapter 7, for Cu, Sn and Pb in Chapter 8 and for Fe, Ni and Zn in Chapter 11.

Research on the selective and intensive electrodeposition of Cu in galvanostatic mode from real leaching solutions (Chap. 7) on SS 316 electrodes has demonstrated that the leaching agent consisting of the $KBr/HBr/Br_2$ redox system can be regenerated anodic in parallel. If a Cu ion concentration in the leaching solution of at least 12 g/L and cathodic current densities between 200 and 600 A/m^2 is maintained, high purity Cu deposits, ~99.6%, can be obtained, which can be directly exploited due to their high market value.

Although the selective and intensive electrodeposition of Cu in galvanostatic mode from real leaching solutions was a success, the presence of Sn and Pb in the solution also required testing a selective potentiostatic electroextraction of Cu and Sn and Pb alloys. Thus, the protocol followed (see Chap. 8) consisted of a first stage of selective potentiostatic electroextraction of Cu, followed by an intermediate stage of reducing the concentration of Cu ions by cementation on metallic Sn, and the last stage consisted of the actual electrodeposition of Sn and Pb alloys. The final concentrations of Cu, Sn and Pb metals in the solution were below 0.01, below 0.1 and, respectively, below 1.5 g/L.

Since the processes for solubilizing metals from PCI, described so far, did not lead to the solubilization of Cu from the "hidden" layers (in the form of tracks) in glass fiber or epoxy resin of the DPI structure, research was needed to analyze the extraction of Cu from these as well. To solve this problem, the strategy was to grind the unequipped WPCB (WPCBD). The copper in the resulting powder became accessible for solubilization using an extremely cheap and accessible H_2SO_4, $CuSO_4$ and NaCl-based leaching system that can be activated by simple air bubbling. The final recovery of ultra-pure Cu by electrodeposition was carried out in parallel with the regeneration of the pH of the leaching solution (see Chapter 9). At the same time, the other undissolved solid components in the powder (epoxy resin and glass fibers) can follow their own recycling path. The closed-loop technological flow contributes to reducing the environmental impact and optimizes resource consumption.

To ensure an on-site regeneration pathway for the KBr/HBr/Br_2 redox system, an experimental installation was designed, built and tested to simultaneously produce HBr and KOH, in-site, by electrodialysis (see Chapter 10). Tests performed with a 4-compartment filter press type RE showed that at a current density of 4 kA/m^2, anodic current efficiencies of 73% (for HBr) and cathodic current efficiencies of 82% (for KOH) are obtained, respectively a specific energy consumption of 40 kWh/kg HBr and 27 kWh/kg KOH. This allows for efficient use of the essential raw materials (KBr and KOH) through recirculation in the process and eventual utilization of the hydrogen in fuel cells. After the electroextraction of Cu, Sn and Pb, the resulting solutions mainly contain Fe, Ni and Zn (**FNZ**). These metals must also be recovered by cathodic electroextraction, even if their value is not high, because this way the resulting solutions can be more easily recycled in the process.

Experiments carried out for this purpose (see Chapter 11), using different electrode materials (Pt, glassy carbon, carbon paste and 304 SS stainless steel) have demonstrated that by controlling the pH= and cathodic potential, it is possible to selectively extract FNZ with extraction yields between 8% and 95% and purity of zinc or FNZ alloys of over 98%.

The actual leach solutions from which the base metals (BM) were extracted (see Chapters 5, 7, 8 and 11) were derived from the solubilization of these metals in WPCB. Initial solutions containing mixtures of KBr and HCl were used for solubilization, to which pure liquid Br_2 was added as an oxidizing agent.

The presence of Cl^- ions in the leaching solution induces the risk of Cl_2 gas release during the electrochemical regeneration step of the oxidative power of the leaching solution because, unlike Br_2, Cl_2 is extremely poorly soluble, not forming complexes with the halide ions present. In addition, the handling of pure Br_2 induces major risks of occupational accidents and environmental pollution. For these reasons, the possibility of initiating the leaching process of exposed BM from WPCB in the absence of Cl^- ions and the initial addition of pure Br_2 was tested (see Chap. 12). Specifically, the leaching of exposed MB from WPCB was performed with an initial SES leaching solution (KBr 2 M + HBr 0.5 M), and as the initial catholyte, SES solutions in which $CuBr_2$ was added to maintain a copper ion concentration between 10 and 180 g/L. The influence of current density and electrolysis time on the structure, quality and purity of Cu deposits deposited from the catholyte solution were studied. The r_F and W_S associated with the cathodic and anodic processes were calculated.

In summary, the research conducted within this doctoral thesis led to the following original contributions:

- Design, construction and testing of 10 experimental stands;
- Design, construction and testing of drum-type chemical reactors and compartmentalized or non-compartmentalized electrochemical reactors of various configurations (tank, filter press, tubular, etc.);
- Identification of an optimal system for the solubilization of base metals from WPCB based mainly on the Br_2 / Br^- redox couple;
- Establishing optimal conditions for regeneration of solubilization solutions including through on-side anodic generation of Br_2;
- Determination of conditions for Cu recovery by galvanostatic or potentiostatic cathodic electrodeposition;
- Selective successive recovery of Cu, Sn and Pb in potentiostatic regime;
- Selective recovery of Fe, Ni and Zn in potentiostatic regime.

The results of the original research from the doctoral thesis were disseminated through the publication of 4 ISI articles, the presentation of 7 papers (posters) at international scientific events specific to the field of chemistry and chemical engineering, and the filing of a patent application.

Referințe bibliografice

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Scientific articles

1. **Marian-Iosif Frîncu**, Eniko Covaci, Sorin-Aurel Dorneanu, Petru Ilea, *Selective electroextraction of base metals from leaching solutions obtained during the recycling of waste printed circuit boards. I. Intensive galvanostatic electrodeposition of copper,* Studia Universitatis Babes-Bolyai, Seria Chemia, **2020**, LXV, 3, p. 33-44. **FI 0.447**
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Conference participation

1. **Marian Iosif Frîncu**, Florica Imre-Lucaci, Sorin Aurel Dorneanu, Petru Ilea, *Electrochemical regeneration of the leaching solution for metals recovery from WPCBs*, RICCCE 21- 2019, 4-7 Septembrie **2019**, Mamaia-România, Poster.
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4. **Marian-Iosif Frîncu**, Sorin-Aurel Dorneanu, Eniko Covaci, Petru Ilea, *Selective electroextraction of Sn-Pb from Cu free leaching solution obtained during the recycling of waste printed circuit boards using the KBr-HBr system,* Symposium SICHEM – 2020, 17-18 Septembrie **2020**, București-România, Poster.
5. **Marian-Iosif Frîncu**, Sorin-Aurel Dorneanu, Petru Ilea, *Innovative circular process for the electrochemical recovery of the base metals from waste printed circuit boards*, EUROINVENT 13-2021, 20-21 Mai **2021**, Iași-România, Poster și PRO INVENT 19-2021, 20-22 Octombrie **2021**, Cluj Napoca-România, Poster.

6. **Marian-Iosif Frîncu**, Sorin-Aurel Dorneanu, Petru Ilea, *Process optimization for base metals recovery from waste printed circuit boards using the electrochemical regenerable $KBr/HBr/Br_2$ system*, Symposium SICHEM – 2024, 11-12 Aprilie **2024**, București-România, Poster.

7. Gabriele-Mario Bogdan, **Marian-Iosif Frîncu**, Sorin-Aurel Dorneanu, *Electrochemical preparation and regeneration of raw materials used in metals recovery from waste printed circuit boards*, RICCCE 23 - 2019, 4-7 Septembrie **2024**, Mamaia-România, Poster.

Patent application

1. Petru Ilea, Sorin-Aurel Dorneanu, **Marian-Iosif Frîncu**, Florica Imre-Lucaci, Arpad Imre-Lucaci, *Process and plant for recovering materials from waste printed circuit boards (without electronic components),* Patent application, nr. A **2021** 00133 RO 135981 A2.

Awards

1. ” Silver Medal”, ” Diploma of Excellence - INCD Urban INCERC” - **Marian-Iosif Frîncu**, Sorin-Aurel Dorneanu, Petru Ilea, *Innovative circular process for the electrochemical recovery of the base metals from waste printed circuit boards*, EUROINVENT 13-2021, 20-21 Mai **2021**, Iași-România.

Participation in research projects

1. *Innovative technologies for advanced recovery of materials from IT and telecommunications waste* (PN-III-P1-1.2-PCCDI-2017-0652, Financing contract nr. 84PCCDI / 2018), coordinated by the Technical University of Cluj-Napoca (**UTCN**) having partners: Babeș Bolyai University (**UBB**), INCDO-INOE 2000 Branch Research Institute for Analytical Instrumentation (**IC-INOE**), "December 1, 1918" University of Alba-Iulia (**UAB**), Gheorghe-Asachi Technical University of Iași (**UTIASI**) and the National Research Institute for Chemistry and Petrochemistry (**ICECHIM**). Component Project UBB: *Efficient and environmentally friendly electrochemical and chemical technologies for advanced recovery of materials from printed circuit board (PCB) waste from IT and telecommunications equipment*, **2018-2021**.

2. Project POCU/380/6/13/123886, ” *Entrepreneurship for innovation through doctoral and postdoctoral research*”, Project co-financed by the European Social Fund through the Human Capital Operational Program, **2020-2021**.