



fenix

WP3 – PILOT PLANTS RECONFIGURATION/ IMPLEMENTATION

Task 3.4 – Pilot-scale development, testing and optimization of materials recovery processes

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ABSTRACT

One of FENIX's project main core is to realize a simple hydrometallurgical technology for valuable elements recovery from different waste streams. For this reason, two innovative technologies have been proposed and tested at both laboratory and small industrial scales. This deliverable focuses on brief description of the results achieved with both lab-scale processes and with their implementation at a larger scale. The results highlighted that is possible to achieve relatively high recovery yields and purity of degrees for the main products (Au, Ag, Cu and Sn) with both procedures.



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Abbreviations and Acronyms:

BP	Business Plan
CPU	Control Processing Unit
EU	European Union
Gold-REC1	Patent (1) in the references
Gold-REC2	Patent (2) in the references
LRE	Lo Russo Estrazioni srl
P & I	Piping and Instrumentation diagram
WPCB	Waste Printed Circuit Board



1. INTRODUCTION

In this report the main results obtained in the FENIX project at pilot plant scale are reported. In the Task 3.3 of WP3 a wide experimental campaign tests have been carried out. At this level an original chemical-biological process has been developed to recover precious and base metals from e-wastes with particular attention to Wasted Printed Circuit Boards (WPCBs), Control Processing Units (CPUs), electrical connectors plated with gold and other base and precious metals, mother boards and other kind of e-wastes.

The original process patented by UNIVAQ could be synthetically described as indicated in the follow:

1. The chemical process can be applied on the e-waste without grinding (with whole WPCB as an example or pieces of 2-3 cm as suitable dimension) avoiding considerable loss of precious metals also described in the literature;
2. The process uses a single step of metals dissolution with a chemical leaching using HCl (hydrochloric acid), H₂O₂ (hydrogen peroxide), acetic acid in water solution at room temperature with a solid/liquid ratio of 10-20%. Acetic acid could be produced by biological process;
3. Precious (Au, Ag, Pd) and base metals (Cu, Sn, Zn, Ni, Pb) are dissolved leaving the WPCB with mainly epoxy resins and fiberglass structure intact (with some residues of metals);
4. The liquid solution is easily separated from the S/L system and a selective reduction-precipitations steps are considered in the process to recover the dissolved metals. These steps are synthetically described in the follow:
 - a. Reduction and precipitation of Au by ascorbic acid (and Pd by ion in powder form and stoichiometric conditions);
 - b. Precipitation of AgCl from the leach liquor by cooling;
 - c. Reduction and precipitation of Cu by metallic Sn;
 - d. Reduction and precipitation of Sn by metallic Zn or Iron;
 - e. Elimination of Pb²⁺ by chemical precipitation adding stoichiometric H₂SO₄;
 - f. Exploitation of the residual solution adding iron in order to produce a FeCl₂-FeCl₃ solution useful for coagulation processes in the treatment of wastewaters;
5. The main products are: Au, Pd (after melting process in an inductive electrical oven adding some slug compound), AgCl, Cu and Sn in powder forms (mainly in the range of 10-90 μm), PbSO₄ and a residual chloride solution very rich in iron (extensively and usually utilized in the coagulation processes in wastewater treatments);

The process has been patented by UNIVAQ [1-2] and actually has been also implemented and updated with other suitable technical solutions with the aim to optimize the process and to propose several technical solutions that could be adapted to different e-wastes or materials containing precious and base metals. The details of the implementation activity have not been reported within this report for IPR reasons because the patenting activity is in progress.

This process has been “commercially” named Gold-REC 2 and the experimental activities carried out in Task 3.3 demonstrated that is suitable for e-waste very rich in terms of precious metals (i.e. larger than 500-800 g/t of Au) and it is not very efficient for ground materials (due mainly to the reduction of dissolved Au in presence of activated silicon or other base metals that in ground condition have a large specific area).



To cover the sector of ground materials UNIVAQ utilized the experience of the previous HydroWEEE and HydroWEEE Demo projects that are summarized in the patent “commercially” named Gold-REC 1, which has been patented by a UNIVAQ’s spin-off company.

Considering this scenario and the experience of the past hydrometallurgical projects, a design and realization of a new pilot plant has been realized in the ambit of this project.

In the next paragraphs the following main items are described:

- Pilot plant development
- Testing and optimization of materials recovery processes

2. PILOT SCALE DEVELOPMENT

The pilot plant was ideated and designed considering its installation in a real industrial environment to carry out experimental tests for the UNIVAQ’s researchers involved in this project. The location of this installation is close to UNIVAQ (about 40 km) at Lo Russo Estrazioni Srl (in the follow LRE) industrial site (see authorization in the following link):

<https://www.regione.abruzzo.it/system/files/ambiente/ippc/procedimenti-aia/18079/2017-dpc026-242-del-24-10-2017-lo-russo-estrazione.pdf> .

The authorization of the pilot plant has been obtained from the regional environmental authority through its technical offices (“Regione Abruzzo – ARTA”) with a standard procedure with a duration of about 8 months. To place the FENIX pilot plant in this industrial site a bilateral agreement between UNIVAQ and LRE was signed at early stages of the FENIX project.

The advantages in using the subject industrial site are:

- Working in a real environmental context;
- Availability of the e-waste necessary for the pilot plant;
- Availability of several services (compressed air supply, grinding equipment, electricity, skilled personnel to perform various required technical operations (i.e. dismantling, grinding etc.)

The following actions were carried out:

- a. Design and construction of the pilot plant;
- b. Testing activities (benchmarking of the results with pilot lab-scale tests)
- c. Production of some suitable amount of materials for their characterization and to be used by other partners of the FENIX project;

2.1. Pilot plant design

In order to extend and test the developed lab scale hydrometallurgical process at a larger scale, a small hydrometallurgical mobile plant has been designed and constructed. The Figures 1 and 2 present its P & I and the external and internal views of the mobile plant., respectively.

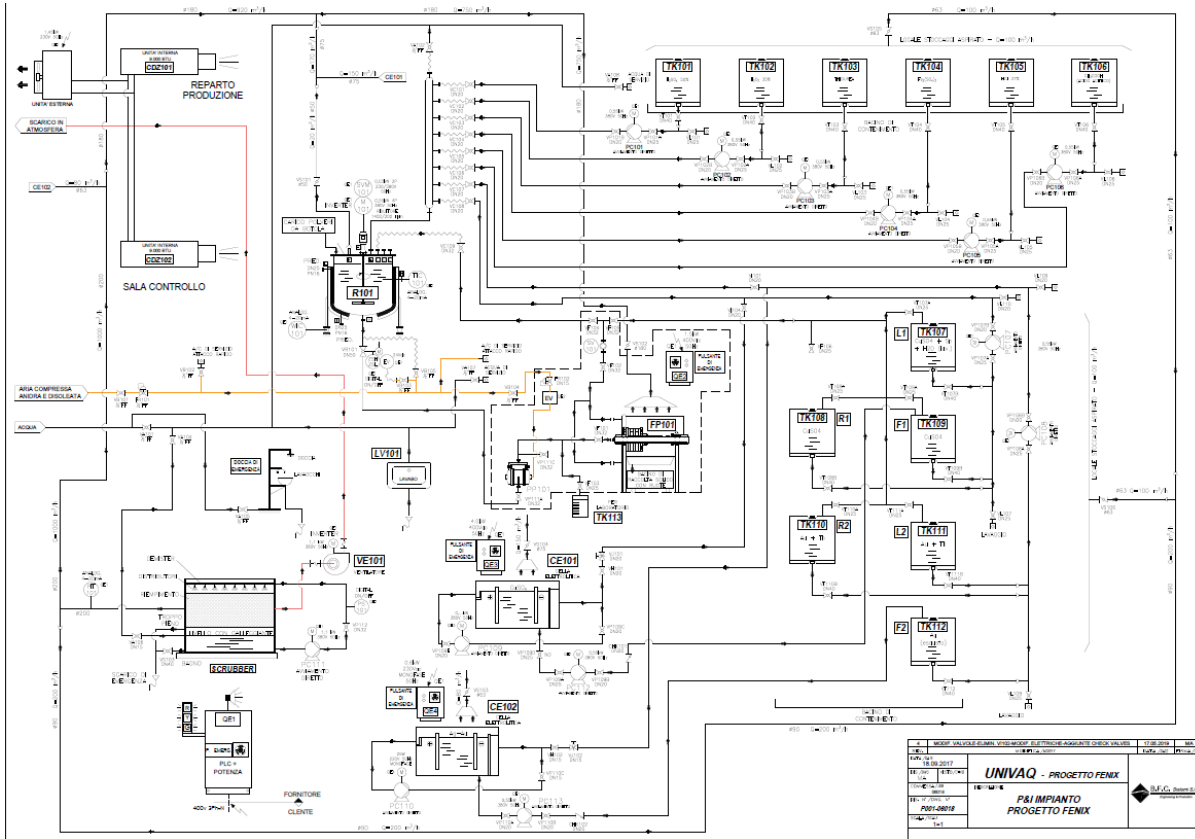


Figure 1: P&I of Fenix's hydrometallurgical plant



Figure 2: External and internal views of Fenix's hydrometallurgical plant

The container was divided in three sections, namely: one section for operator and control panel, a second section that has a chemical reactor (R101) in which leaching, precipitation and cementation will be performed, a filter press (FP 101) with its panel control, two electrochemical cells (EC1 and 2), a scrubber , and the third section with 12 storing tanks (TK 101-106 for reagents and TK 107-112 for solutions and wastewater). This plant was constructed on the site of BFC Sistemi SRL Company (the subcontractor of UNIVAQ for plant design and construction) and then moved to the actual location, the site of Lorusso Estrazione Srl Company. Till present, in order to ensure the functionality of the plant in both manual and semi-automatic mode, several tests have been performed only with water and with milled waste printed circuit boards and water.

In addition, the authorization from the Environmental Protection Agency of Abruzzo Region to perform the hydrometallurgical plant was obtained.



2.2. Public tender

The tender, having for OBJECT: "Negotiated procedure ex art. 36, paragraph 2, letter b) of Legislative Decree n. 50/2016 and s.m.i. for the supply of a hydrometallurgical pilot plant - amount based on auction euro 180,000.00 exempt value added tax (V.A.T.) ex art. 72, paragraph 1, letter c), D.P.R. 633/7 ", was carried out according to the University procedure, in compliance with the LEGISLATIVE DECREE 18 APRIL 2016, N. 50 Code of public contracts.

The main phases of the authorization procedure, (i.e. tendering, offers evaluation and winner's declaration) are shown below:

- 1) Approval of the tender for the FENIX project mobile system by the Department of Industrial Engineering, Information and Economy, to which the scientific project responsible Professor F. VEGLIO' refers (Resolution of the Council of Department of Industrial and Information Engineering and Economics n. 59/2018)
- 2) Approval of the tender for the FENIX project mobile tendering by the Board of Directors (Resolution No. 179/2018 of the Board of Directors of 5/15/2018)
- 3) Publication of the public tender notice on 06/26/2018 (deadline 06/08/2018)
- 4) Appointment of a tender commission (D.D.G. 826/2018 Prot. No. 47607 of 10/31/2018)
- 5) Contract award (Resolution No. 29/2019 of the Board of Directors of 01/30/2019)

All documentation relating to the procedure, announcement, notices, minutes and resolutions, is published in the transparency area of the University of L'Aquila. (<https://www.univaq.it/section.php?id=1878>) with CIG 73855089FF.

At the end of all these phases the Pilot Plant was fit to commence realized and installed to be ready for the required experimental campaign tests.



3. TESTING AND OPTIMIZATION OF MATERIALS RECOVERY PROCESSES

3.1. Gold-REC1 process description

This process is based on a hydrometallurgical technology which has already been patented at EU level [1]. It consists of using two different leaching systems for a selective solubilization of base metals by the precious one from milled waste printed circuit boards. The first leaching process makes use of sulfuric acid as reducing agent and hydrogen peroxide as oxidant. The second one involves thiourea as reagent and ferric sulfate as oxidant. The resulted solutions are further subjected to precipitation and electrolysis processes. The main products of this hydrometallurgical process are tin oxide, copper metal, gold and silver. Moreover, as the residual solutions have different inorganic (solution after Cu recovery) and organic substances (solution after precious metals electrolysis). The main reactions involved within the entirely hydrometallurgical flowsheet (see Figure 3) are as follows:

Cu leaching



Sn leaching and precipitation



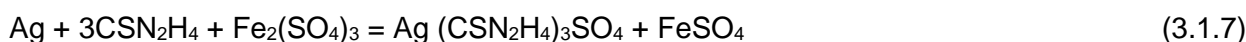
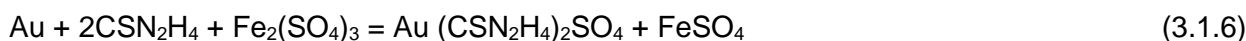
Cu electrolysis



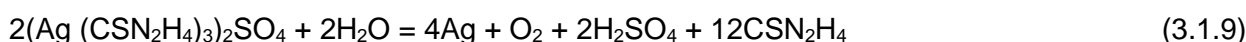
Waste water treatment



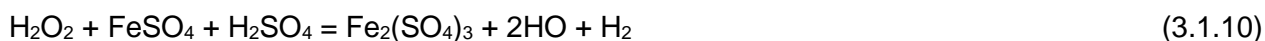
Au and Ag leaching



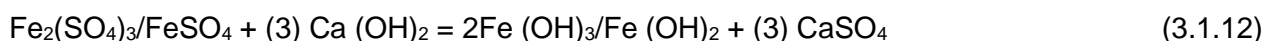
Au and Ag electrolysis



Fenton treatment



Lime treatment



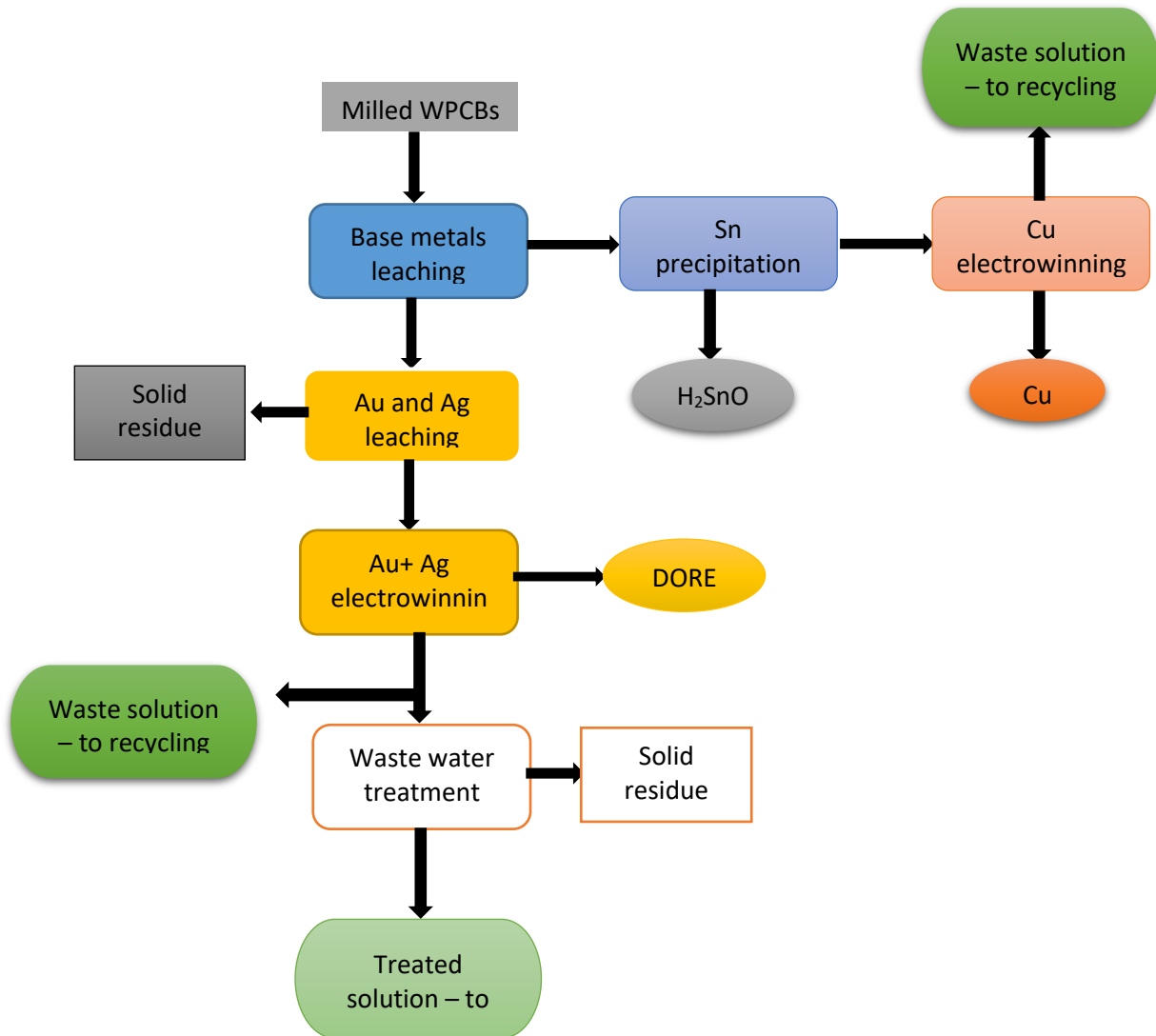


Figure 3: Gold-REC 1 process flowsheet

3.2. Gold-REC1 demonstration activities

Raw materials collection and characterization

The process was tested within the FENIX hydrometallurgical plant on waste printed circuit boards and different electronic components. These raw materials have been provided by Greentronics Company and LoRusso Estrazione. Prior to hydrometallurgical process tests performing, the chemical analysis in terms of their content of Cu, Sn, Au and Ag was performed. The determined concentrations are shown in Tables 1-4.


Sample	Data
Greentronics sample	
Element	(% or mg/kg) ± s.d.
Cu	33 ± 1 %
Sn	15 ± 2 %
Zn	2.0 ± 0.5 %
Au	90 ± 10 mg/kg
Ag	1400 ± 50 mg/kg
Pd	30 ± 5 mg/kg

Table 1: Example of sample treated by Gold-REC 1 process


Sample	Data
LRE sample (plates)	
Element	(% or mg/kg) ± s.d.
Cu	28 ± 1 %
Sn	< 1%
Zn	< 1%
Au	600 ± 50 mg/kg
Ag	80 ± 10 mg/kg
Pd	10 ± 5 mg/kg

Table 2: Example of sample treated by Gold-REC 1 process


Sample	Data
LRE sample (plastic chips)	
Element	(% or mg/kg) ± s.d.
Cu	10 ± 1 %
Sn	< 1%
Zn	< 1%
Au	8000 ± 500 mg/kg
Ag	1200 ± 100 mg/kg
Pd	< 10 mg/kg

Table 3: Example of sample treated by Gold-REC 1 process


Sample	Data
LRE sample (ceramics chips)	
Element	(% or mg/kg) ± s.d.
Cu	8 ± 2 %
Sn	< 1%
Zn	< 1%
Au	1000 ± 200 mg/kg
Ag	800 ± 100 mg/kg
Pd	< 10 mg/kg

Table 4: Example of sample treated by Gold-REC 1 process

As is shown in both the tables above, the variation between the elements concentration by the replication tests is large. This is due to the not non-homogeneity of the sample materials.

Tests results

Many tests of metals recovery with this technology have been applied on the milled sample of Greentronics Company. High recovery degrees for both class of metals have been obtained within all the performed tests. Bellow, a more detailed process result is reported.

Base metals recovery

The leaching process, which take place using the two-step counter current method at a solid concentration of 15 %, under continuous agitation for 2 hours for each step and a reagents concentration of 1.8 M of sulfuric acid and 20% vol/vol of hydrogen peroxide, has resulted in over 95% of Cu recovery and 60 % for Sn. Furthermore, the tin was recovered from solution in a percentage of 90% and purity of about 50% using the coagulation process with polyamine solution

in a concentration of 10%. Thereafter, the resulted solution was subjected to electrolysis process. The recovery degrees of Cu and determined current efficiency are reported within Figure 4.

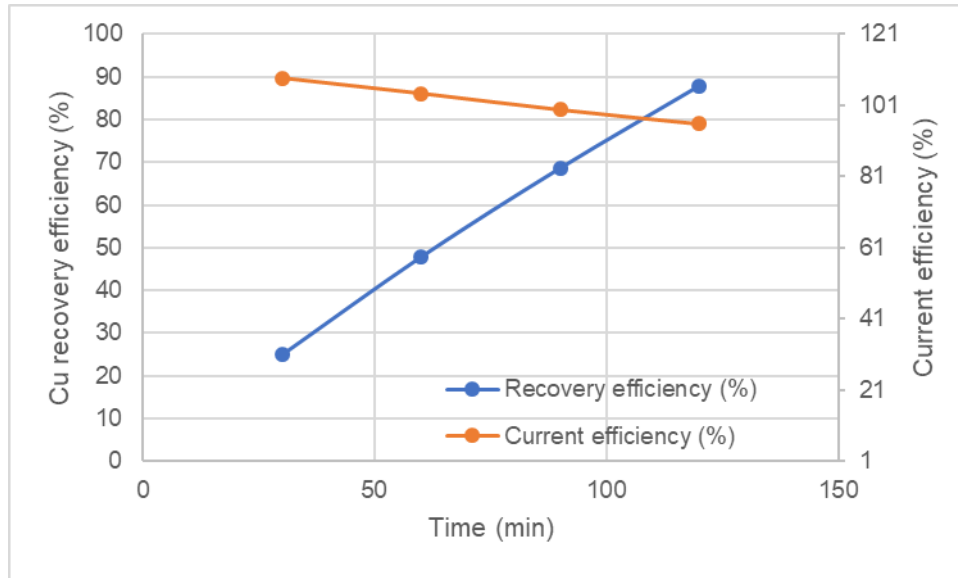


Figure 4: Cu recovery efficiency and current efficiency vs. process time

As shown, the current efficiency had the optimum value (90%) after 30 minutes of reaction and it tends to decrease with time. This is due to the parasitic reactions that takes place along the Cu reduction. At the end of the process, the purity of Cu product was of 89% and the calculated power consumption was of 2.39 kWh/kg of Cu.

Precious metals recovery

Over 85% of Au and 55% of Ag recovery degrees have been achieved using the second leaching system. Figure 5 presents the achieved recovery degrees and process current efficiencies for recovery of Au and, respectively, Ag.

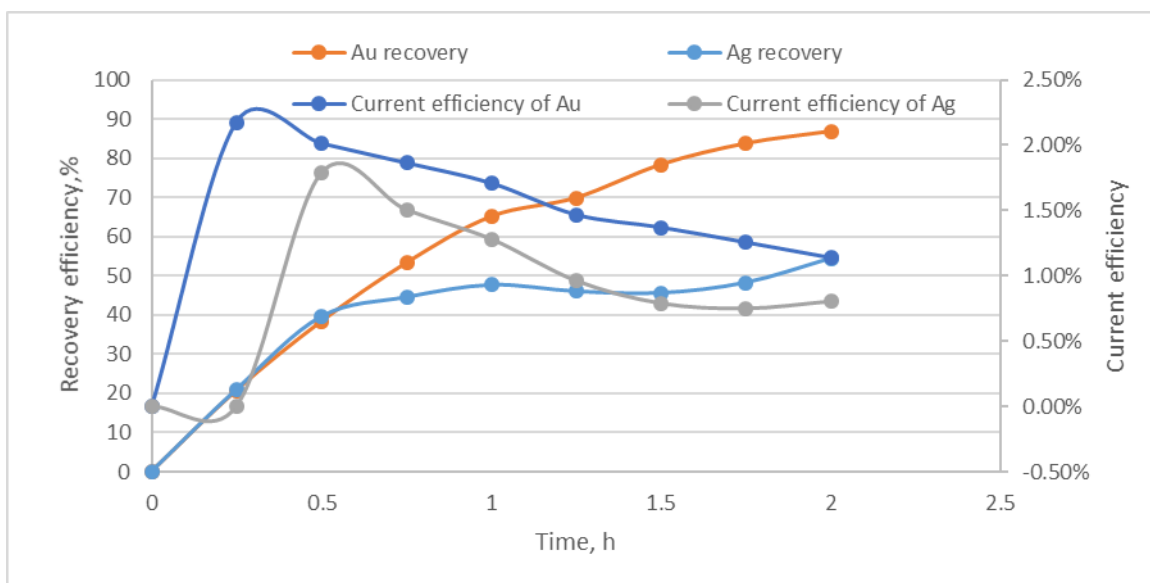


Figure 5: Au and Ag recovery efficiency and current efficiency vs. process time

These results show a very low current efficiency for both elements which is due scarce content of precious metals within solution and to the parasitic reactions. The calculated power consumption was of about 10 KWh/kg of Au. According to these achievements, a DORE product with 72% of Au was obtained at the end of the process.

To summarize the achievements of Gold-REC 1 process, Tables 5-7 summarize all the main experimental results.


Sample	Data
Copper by Electrolysis	
Element	(% or mg/kg) \pm s.d.
Cu	90 \pm 5 %
Recovery (%)	\triangleright 90%

Table 5: Example of product obtained by Gold-REC 1 process (Cu)


Sample	Data
Tin Oxyde by PA precipitation	
Element	(% or mg/kg) \pm s.d.
Sn	45 \pm 5 %
Recovery (%)	\triangleright 50%

Table 6: Example of product obtained by Gold-REC 1 process (Tin Oxyde)


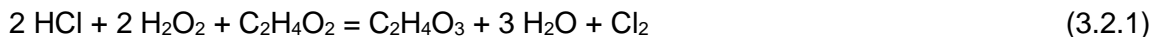
Sample	Data
Dore' (Au+Ag) by electrolysis	
Element	(% or mg/kg) ± s.d.
Au	75 ± 5 %
Ag	20 ± 5 %
Recovery (%)	➤ 80% (gold)

Table 7: Example of product obtained by Gold-REC 1 process (Au+Ag)

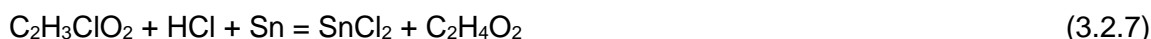
3.3. Gold-REC2 process description

This process consists of a leaching treatment to dissolve all valuable metals from the selected material without grinding. Then, after selective operations, metals can be purified by the leach liquor solution. This process is generally adopted for connectors and CPU rich in precious metals without grinding. The leaching system consists of using hydrochloric acid, hydrogen peroxide and acetic acid as chemical reagents. The leaching process of metals takes place by using chloroacetic acid as oxidant and hydrochloric acid as complexing agent. The chloroacetic acid is produced by in-situ chemical process within two steps: firstly, hydrochloric acid reacts with hydrogen peroxide and acetic acid to produce peracetic acid, water and chlorine; in the second step chloroacetic acid and hydrochloric acid are produced by the chlorination of the unreacted acetic. The main chemical reactions are reported as follows:

In situ chloroacetic production



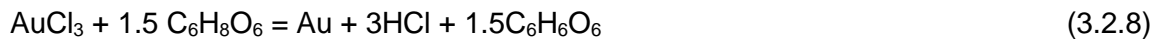
Dissolution of precious and base metals





Thereafter, in order to recover gold and silver from solution, ascorbic acid can be used as reducing agent at room temperature. Silver is recovered from solution by using the same concentration of ascorbic acid but at a lower temperature (15 °C). Accordingly, a selective recovery of these two precious metals can be achieved. The precipitation reactions are described below:

Au precipitation



Ag precipitation

By cooling the leach liquor at a temperature level lower than 15°C.

Copper is further recovered from solution in its metallic form by cementation with tin metal. There was shown that the optimal condition for Cu recovery and degree of purity for the final product is the use of tin in a concentration of 20% lower than the required stoichiometric amount.

Copper cementation



After this process step, tin is reduced from solution to its metallic form by cementation with metallic zinc powder. This is added within solution in an excess of 70 % by the required stoichiometric value.

Tin cementation



The entirely flowsheet of the hydrometallurgical process is shown in Figure 6.

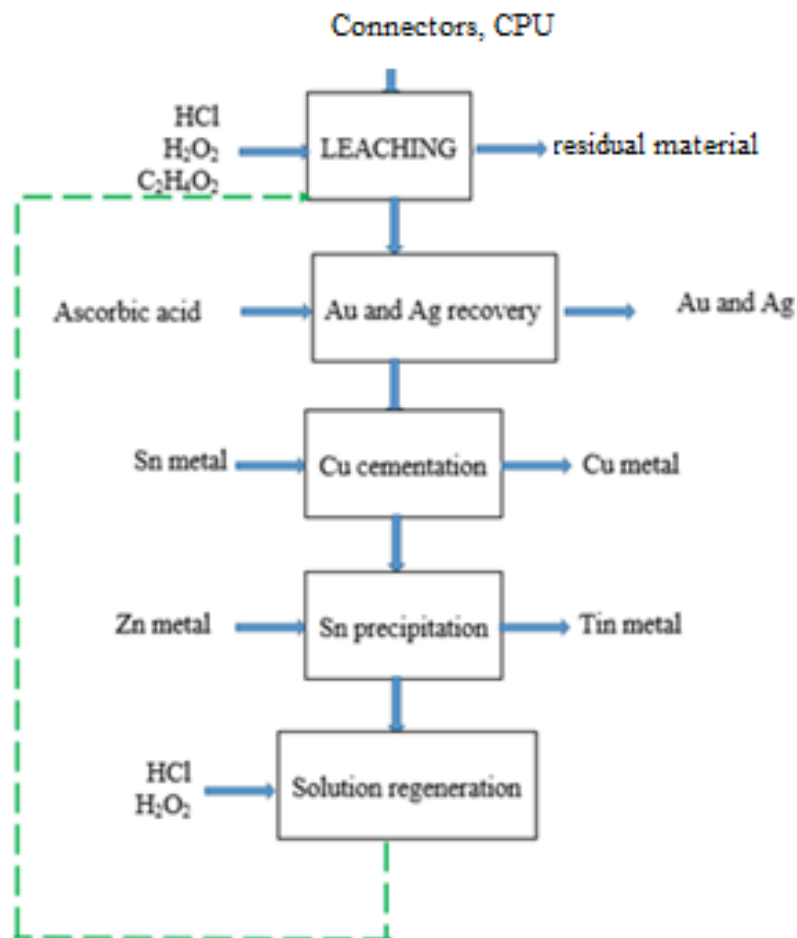


Figure 6: Block scheme of the GOLD-REC 2 process

3.4. Gold-REC2 demonstration activities

Tests have been conducted by using connectors, CPU and other very rich materials in terms of gold content (larger than 500-800 g/t Au) by application of process GOLD-REC 2. In Figure 7, CPU plated connectors and other special gold-plated materials have been utilized for this treatment, are showed as an example



Figure 7: Examples of e-wastes treated by Gold-REC 2 process

A series of tests has been performed by keeping constant the following parameters of process: solid concentration 10%; hydrogen peroxide concentration 1.96 M; acetic acid concentration 1.73 M, mechanical agitation 235 rpm. In order to evaluate recovery yield of Au, chemical attacks with aqua regia of the samples were carried out at the end of each treatment. In the next Table the most important experimental results have been shown.


Example	Of a table
Material used In Gold-REC 2 process	
Element	(% or mg/kg) ± s.d.
Au	950 ± 20 %
Recovery (%)	➤ 90%
Purity (%)	➤ 99%

Table 8: Example of sample and product treated by Gold-REC 2 process (S/L ratio 10%; HCl 4 M; Acetic acid 1.7 M; H₂O₂ 2 M; treatment time = 2 hours). Gold obtained by chemical reaction and treatment in oven at 1060°C with slugging compounds.

As can be observed within the Table 8, the highest recovery yields for Au are achieved when the concentration of HCl was of 4 M. Moreover, in order to achieve the complete recovery of Au an Ag but also for other elements, the cross and counter current leaching process have been furtherly tested. After leaching, gold precipitation tests have been performed by using the leach liquors. Ascorbic acid with a concentration of 5 g/L has been added in solution and test was carried out for 1 hour at 150 rpm and room temperature. In order to evaluate the recovery of gold, the solution was analysed before and after treatment. The final gold concentration in solution was less than 1 mg/L, confirming the almost complete precipitation of the metal. The grade of gold precipitate was determined to be 89.8 %. The obtained precipitate was melted in an oven at 1060 °C adding some slugging compounds reaching a purity larger than 99% in not optimized condition. Moreover, also silver concentration was measured first and after acid ascorbic treatment and was determined that any precipitation of silver occurred. The results demonstrate the gold selective recovery with this procedure. Silver recovery was obtained after cooling of the solution at 18 °C. After 1 hour of mechanical stirring at 150 rpm, a 5 % of silver recovery was obtained. The low recovery was due to the strong influence of AgCl solubility with temperature. Therefore, to increase silver recovery the treatment must be performed at a lower temperature value (< 15°C) and with an increases of process time. Figure 8 and 9 show the obtained final products.



Figure 8: Gold final product



Figure 9: Ag final product

Further tests were then carried out to recover copper and tin by cementation. Figure 10 shows the obtained products that has been sent to MBN for further characterisation studies and for the next uses considered in the FENIX project (i.e. production of some metallic pieces using a 3D printer).



Cemented Cu



Cemented Sn

Figure 10: Copper and Tin recovered by Gold-REC 2 process (Cu > 84% and Sn > 96%)

4. CONCLUSIONS

From the analysis of the results achieved in WP3 and summarized in this deliverable 3.4 it is possible to conclude that:

1. Gold-REC2 process is suitable for e-waste not grinded with good recovery of precious metals with acceptable levels of purity. As indicated in lab-scale tests this process is not suitable for grinded materials (due to reduction process of Au with the in-situ activated silica that is present within the composition of PCBs). The process seems to be more suitable to treat very rich e-waste;
2. Gold-REC 1 at the contrary is more suitable for the treatment of grinded e-waste with lower gold concentration (< 400 mg/kg). Some modification of this process has been also identified in the ambit of this project, but no details are given in this report for IPR possibilities. A new patent is under study in the ambit of the project and it will be well described in the next report as soon as IPR activities will be concluded;



3. Pilot plant tests have highlighted the necessity to implement FENIX pilot plant. These modifications will be performed coupling this activity with the training course carried out in the ambit of WP6;
4. Some products have been produced in the pilot plant such as copper (about 500 g), tin oxide, gold. Further materials will be produced during the training activities carried out in WP6 (Task in progress);
5. The obtained data will be used to implement the BP of both processes (in progress in the ambit of WP2);

REFERENCES

- [1] **WO2018215967A1** Process for the hydrometallurgical treatment of electronic boards, Inventors: I. Birloaga, F. Vegliò, I. De Michelis, F. Ferella, May 2018, Priority number - IT201700057739A-2017-05-26 (**Gold-REC1**).
- [2] **WO2019229632A1**, Hydrometallurgical method for the recovery of base metals and precious metals from a waste material, Inventors I. Birloaga, F. Vegliò, December 2019, Priority number - IT201800005826A-2018-05-29 (**Gold-REC2**)