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# Hydrometallurgical pilot plant reconfiguration for the recycling of automotive waste for the recovery of precious and critical metals: H2020 Treasure project †

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**Abstract:** The TREASURE project aims to improve circularity in the automotive sector by developing tools and technologies to support a circular supply chain. Advocating a circular economy approach, TREASURE showcases technical and sustainable benefits for automotive companies through pilot programs focused on disassembly, recycling, and eco-design. The recycling processes for recovering precious, critical, and base metals from different types of waste from automakers, LCD manufacturers, and dismantlers are hydrometallurgical. In line with the principles of Industry 4.0, a study was carried out for the optimal reconfiguration of a mobile hydrometallurgical pilot plant to achieve higher levels of automation and circularity than current practices.

**Keywords:** hydrometallurgy; recycling; automotive waste; metals recovery; pilot plant; reconfiguration; leaching; valorization.

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## 1. Introduction

In recent years, the importance of adopting a circular economy in waste management in the automotive sector has become increasingly evident. With the increase in the production of electric and hybrid vehicles, together with the growing awareness towards environmental sustainability, the automotive industry is facing a crucial challenge: how to responsibly manage the life cycle of vehicles and minimize the environmental impact resulting from the production, use, and disposal of vehicles [1,2].

In the automotive sector, adopting a circular economy offers numerous advantages. Among the most important, we find the management of critical raw materials. The circular economy promotes the recycling and recovery of materials used to produce vehicle components. Recycling these materials reduces the pollution caused by their production and allows for secondary sources of supply. This aspect is crucial for many countries that do not have significant primary sources [3].

We find metals among the materials of most significant economic and strategic interest. Speedometers, radios, sensors, and others contain significant concentrations of precious and critical metals. The TREASURE project aims to show the benefits and feasibility for automotive companies of a circular approach to managing end-of-life devices [4].

To be able to achieve this goal, it proposes an in-depth study for the implementation of pilot processes on disassembly and recycling. Regarding the recycling phase, a study was conducted to reconfigure a hydrometallurgical pilot plant built as part of a previous European project – H2020 FENIX, grant agreement No. 760792 [5].

The pre-existing mobile pilot plant was built in a 40-foot container and was intended to treat electronic board powders with a maximum size of 2 mm. An in-depth study was carried out to identify the possible implementations of the pilot plant, starting from some criticalities highlighted during the experimental activities carried out within the FENIX project. One of the purposes of the reconfiguration was to modify and implement the plant to process the materials of interest in the TREASURE project. More generally, the goal was also to make the plant more flexible for treating different materials, not just powders. Finally, important automation work was carried out to comply with the concept of Industry 4.0 fully.

## 2. Materials, Processes, and FENIX Plant

### *2.1. Materials*

One of the purposes of the reconfiguration was to make the pilot plant more flexible to process different types of material without physical pre-treatment of size reduction and the materials of interest for the TREASURE project. The materials studied at the laboratory scale within the present project are:

- In-mold structural electronics (IMSEs);
- ITO glass;
- Printed circuit boards (PCBs).

Based on the state of the element of interest to be recycled from these samples for their hydrometallurgical treatment, no preliminary grinding step is required. IMSEs are composed of a layer of silver ink directly on the surface of the samples so that it is already exposed to the action of the acids to be dissolved. ITO glass samples are composed of a glass substrate coated with a layer of ITO on both sides; compared to the initial sample, the material is cut into squares of about 2 cm. From a chemical standpoint, no milling would be required to ensure the dissolution of indium and tin during the leaching step, only a reduction in sample size for easier handling of the solid during the hydrometallurgical process.

The two hydrometallurgical processes developed for PCBs require a disassembly phase to separate some surface mount devices (SMD) from the board [6–8].

### *2.2. Processes*

Hydrometallurgical processes treat the materials described above. These processes allow the recovery of precious and base metals with high recovery yields and a low environmental impact [8].

One of the main phases of hydrometallurgical processes is leaching, in which the reagents extract the metals of interest from the solid material. The extraction kinetics can depend on particle size, stirring, and temperature. Based on the rate-determining step, it is essential to study the optimal configuration of the leaching reactor. In this regard, the reactor in the pre-existing FENIX Plant was unsuitable for the numerous types of material treated in the TREASURE project.

Another important phase of hydrometallurgical processes is the recovery of solubilized metals during leaching. In this case, it was decided to keep the electrolysis cell previously installed in the system.

### *2.3. Pre-existing FENIX Plant*

The pilot plant has been realized inside a mobile container that could be moved to test and demonstrate the technical feasibility of the hydrometallurgical processes. The container is composed of three different areas:

- Storage area;

- Operating process area;
- Control panel area.

Figure 1 reports the layout of the hydrometallurgical pilot plant.

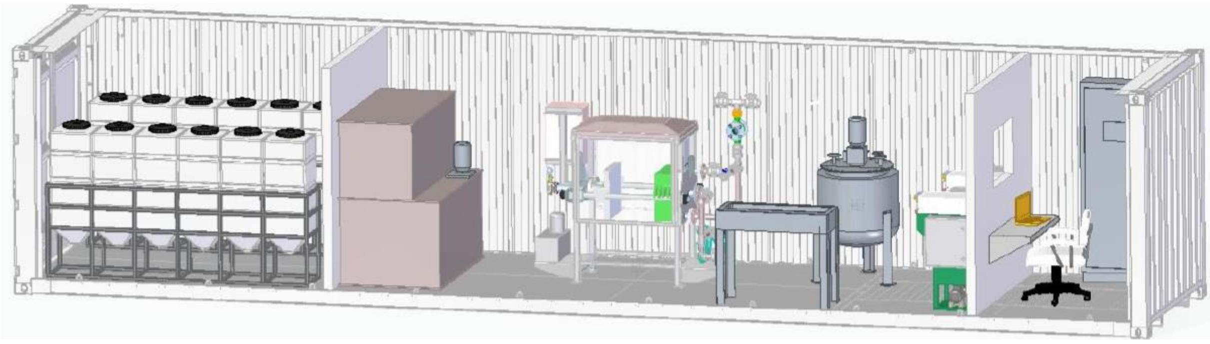

**Figure 1.** Layout of the pre-existing FENIX pilot plant.

The storage area consists of 12 tanks with a capacity of 200 L with a total of 8 centrifugal pumps for solution transfer. In this area occurs both the storage of chemicals and the temporary storage of the processing solutions. The tank of each chemical had a specific pump, while for the tanks of the processing solutions, there was a pump in service of three tanks since the leaching solutions were mainly of two types. Chemicals were fed into the tanks via a mobile pump, but without fixed piping, as the 1 $m^3$ caged IBC of the chemicals were placed outside the container.

In the operating area, the chemical operations necessary for the execution of hydrometallurgical processes take place. The operating area has a scrubber, a chemical reactor, a Plate&Frame filter, a copper electrolytic cell, and a precious metals electrolytic cell.

In the control panel area is located a PC for the management software. The plant is controlled by PLC (Programmable Logic Controller). However, it is possible to work in semi-automatic mode; in fact, only the pumps are launched via software, but almost all the valves must be manually opened/closed, except for one valve that manages the chemical reactor's discharge.

The reconfiguration of the hydrometallurgical pilot plant aims at solving the main issues that emerged during the experience of the FENIX project in the use of the pilot plant, at making the plant more flexible for the treatment of different types of materials, and more specifically, at adapting to the materials of interest for the TREASURE project.

The experimental pilot-scale activities during the FENIX project mainly concerned treating printed circuit boards' powders with a particle size below 2 mm. These tests showed the following critical points to be solved during the reconfiguration:

- Clogging of the suction pipes during the chemical reactor discharge upstream of the plate & frame filter;
- The plant's only filtration system, the plate & frame filter, was unsuitable for retaining small quantities of solid and, specifically, particles with small particle sizes, such as gold particles and generally all the metals recovered by precipitation;
- From the point of view of operations carried out by the operators, the use of an external mobile pump to transfer chemicals from the caged IBC to the corresponding tank was not a good safety practice.

In Figure 2 the clogging of the suction pipes and of the pneumatic diaphragm pump located in the line were shown.

**Figure 2.** Photographic aspect of the pipes **(left)** and pump **(right)** clogging.

## 3. TREASURE Pilot Plant

### *3.1 Reconfiguration*

To achieve the objectives explained in Chapter 2, the main implementations carried out have been listed below:

- Addition of two chemical reactors with different characteristics;
- Addition of different filtration systems, such as cartridge and bag filters;
- Movement of the chemical storage tanks to an external area directly connected to the plant by a pipeline;
- Automatization of all the valves according to Industry 4.0;
- PLC software revising with specific recipes for the materials of interest for the TREASURE project.

Figure 3 shows the layout of the reconfigured plant.

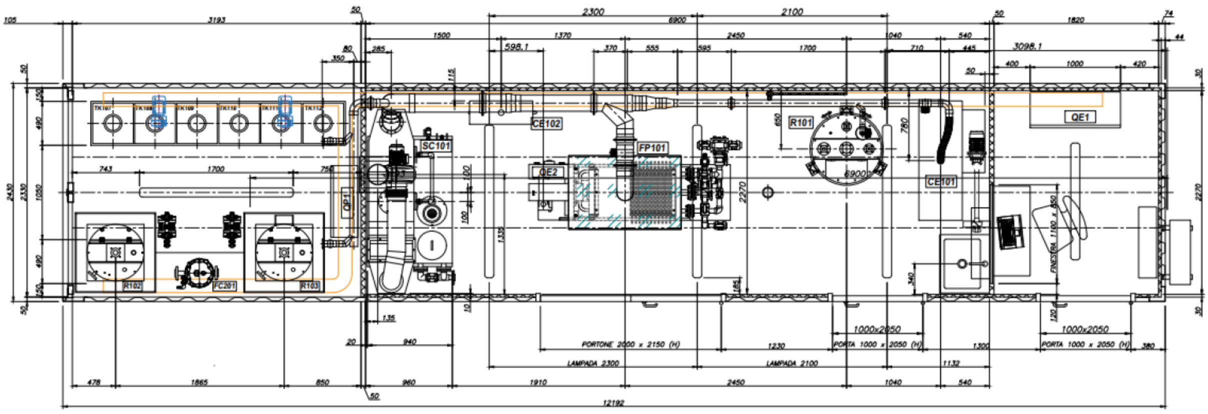


**Figure 3.** Layout of the reconfigured hydrometallurgical pilot plant, plan view.

The first two implementations are realized to the left side of the container plant by removing the chemical tanks (moved out of the container). The reactors have different geometry and functionality.

The first reactor added is like a slurry reactor intended for treating ground materials with small particle sizes; its useful volume is 100 L. The stirrer reaches almost the bottom of the reactor so that good mixing of the suspension can be guaranteed. A big filter ensures filtration to avoid the issues related to the powders clogging during discharge, and the chemical reactor position is higher than the ground floor. This type of reactor can also be used for the chemical operations like cementation and precipitation to recover metals from the leach liquor solutions or to prepare leaching solutions before starting contact with the material to be treated.

The second reactor added is intended to treat materials without any grinding or heavy powders that can create problems during the filtration; its useful volume is 100 L. It is a reactor specially designed to prevent the solid is removed from the reactor for each filtration operation. Inside the reactor, towards the bottom, is a septum with several openings of 1.2 mm that allow the passage of the solution but not of the solid, so the solid remains inside until the end of the process. During the filtration, the septum retains most of the solid so that issues related to clogging pipes are avoided. The stirring, if necessary, takes place through air/nitrogen insufflation. Nevertheless, there is a stirrer with a servo-ventilated electric motor use and gearbox, but its function is exclusively related to material discharge at the end of all leaching operations; it is not used to mix the suspension during chemical reactions occur. A rotor with 2 curved fixed blades allows the solid conveying in automatic mode for the discharge through an inspectable side door.

The pre-existing manual valves have been replaced with automatic valves to conduct the operations necessary for the process execution automatically from the computer workstation via PLC software, according to Industry 4.0.

### *3.2 Pilot Plant Test*

In order to test the new pilot plant, a leaching test on a PCBs sample received during a previous project was performed. The process is patented by Birloaga et al. [6]. In this regard, 10 kg of ground powders were introduced into the new slurry reactor with a solid/liquid concentration of 15%. Stirring was set to 200 rpm. Two leaching steps were performed with $H_2SO_4$ and $H_2O_2$ for 90 min each to remove the base metals. Subsequently, a single leaching step of the precious metals was carried out using $H_2SO_4$, $Fe_2(SO_4)_3$, and Thiourea. At the end of the 60 min, the filtration was carried out by the bag filter. The concentrations of the various reactants in the various leaching steps are the same as reported in the patent mentioned above. Table 1 shows the extraction yields of the different metals for the three leaching steps.

**Table 1.** Extraction yields of precious and base metals in the various leaching steps.

| Element | Step I | Step II | Step III | Recovery |
|---|---|---|---|---|
| Au | 0.00% | 0.00% | 85.32% | 85.32% |
| Ag | 5.26% | 1.22% | 87.81% | 87.81% |
| Cu | 84.43% | 13.41% | 0.13% | 97.84% |
| Sn | 85.22% | 5.17% | 0.38% | 90.39% |

## 4. Conclusions

The reconfiguration of the pre-existent hydrometallurgical pilot plant had different main objectives to be achieved. First, resolving the main issues arising from the trials conducted under the previous European project; second, making the pilot plant more flexible for treating different materials. Third, adapting the plant to the TREASURE materials according to their type and the processes developed at the lab scale. Moreover, finally, fully automate the plant so that PLC can ultimately govern it.

To achieve these objectives, various system solutions have been used. The Addition of some equipment, the Movement of others, and the automation of the whole process made it possible to overcome the various problems encountered and to perfectly adapt the old FENIX system to the materials of the new TREASURE project.

After the works had been completed, a start-up test was performed to check the correct working of all the equipment of the reconfigured pilot plant and to explore the operation of the PLC software based on the chemical operations to be carried out to treat the TREASURE materials.

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