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### Abstract

The electro and electronic industry has been increasing gradually, causing an increase in the generation of waste of electrical and electronic equipment, which have several components that have added value such as - for example - gold. Aiming at environmental protection, measures are sought for the recovery of these metals, through tests with several technologies such as leaching. This consists in separating the component from the solid part, transferring it to the liquid part, using acids. It is necessary to study acids that are less harmful to the environment. Thus, microprocessors of obsolete computers were collected, their characterization was carried out and the elements present in the pins were identified. After the characterization, the leaching stage was carried out, firstly with aqua regia; then with acetic and citric acids. The leaching results presented about 10 mg / L of gold with aqua regia and about 0.5 mg / L with citric acid.

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## **Application of solid-liquid extraction with organic acids for recovery of precious metal from technological waste in disuse**

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### **Abstract**

*The electro and electronic industry has been increasing gradually, causing an increase in the generation of waste of electrical and electronic equipment, which have several components that have added value such as - for example - gold. Aiming at environmental protection, measures are sought for the recovery of these metals, through tests with several technologies such as leaching. This consists in separating the component from the solid part, transferring it to the liquid part, using acids. It is necessary to study acids that are less harmful to the environment. Thus, microprocessors of obsolete computers were collected, their characterization was carried out and the elements present in the pins were identified. After the characterization, the leaching stage was carried out, firstly with aqua regia ; then with acetic and citric*

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**Keywords:** Obsolete technological waste; Leaching; Gold; Microprocessors.

## 1. Introduction

Brazil has been facing a major problem over the years: the accumulation of waste due to population growth, urbanization and the technological revolution. All these factors are related - also - to the lifestyle and consumption of the population [GOUVEIA, 2012].

Among the several wastes, the most important are WEEE (Waste Electrical and Electronic Equipment) [WANG, 2008]. This accumulation is due to the end of the useful life of the equipment, and may also be defective, wear or often become aesthetically obsolete, still due to the technologies that have been emerging - gradually - over the years [NNOROM; OHAKWE; OSIBAJNJO, 2009]. These residues have been identified as secondary sources of several metal [WILLIAMS; OGONDO; CHERRETT, 2011]; when unrecovered, cause damage to the environment, special treatment is required for these wastes at the end of their useful life and studies for the evaluation of operations and tests that enable the recovery or recycling of these materials for new uses [NNOROM; OHAKWE; OSIBAJNJO, 2009].

According to the United Nations of Brazil (ONUBR), in 2015, the electronics industry generates - each year - around 41 million tons of electronic waste, from computers and smartphones; this number has been increasing gradually over the years [ONUBR].

According to the Ministry of Environment, the National Solid Waste Policy (PNRS), approved in 2010, provides for the prevention and reduction of waste generation. Based on this policy, the metals present in microprocessors of obsolete computers were studied by leaching with acids such as royal water and also organic acids, which are less harmful to the environment [MINISTÉRIO DO MEIO AMBIENTE].

For the identification of the elements present in the microprocessor, it is made use of the Scanning Electron Microscopy (SEM) equipment, which is able to identify and quantify the chemical elements present in the sample. In addition, it is capable of producing a high magnification image, up to 300,000 times and also high resolution.

Leaching, also called solid-liquid extraction, consists in separating a desired component from a solid phase, transferring it to a liquid phase, while the solid is contacted with the liquid phase so that leaching can occur. Both phases are in an intimate contact where the solute can diffuse from the solid to the liquid phase, which will allow a separation among the components of the solid phase [GEANKOPLIS, 1993].

Therefore, it is important to study new technologies for the leaching of this high value-added metal such as - for example - leaching using environmentally friendly acids. After the leaching, it is necessary to analyze the samples obtained in the atomic absorption equipment (AAS), where it is possible to determine the amount of analyte present in solution.

## 2. Experiments

Face to the reality described above, a partnership was established with the Information Technology sector

of University in order to collect the microprocessors of obsolete university computers that would be sent to discard. Microprocessors of a particular brand were collected; then a sample of the pins was removed with the help of pliers to analyze and characterize them in the SEM. Subsequently, the leaching was performed according to the flowchart shown in Figure 1.

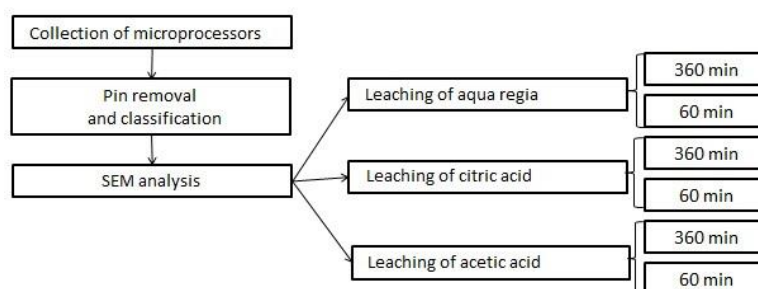


Figure 1. Flowchart of the tests performed

Based on the methodology described by Côrtes et al. (2014), leaching with aqua regia ( $\text{HCl}$  e  $\text{HNO}_3$  P.A 3:1) was started in batch,  $25^\circ\text{C}$ , in which 1 g of pins were added in contact with 50 mL of aqua regia solution, shaking the sample [CORTES et al., 2014].

During this step, other samples were leached with organic acids; the first containing 1g of pins, 0.30g of ferric sulphate and citric acid with a concentration of 20g /L and the second replacing the citric acid with glacial acetic acid, concentration 20g /L. At the end of the leaching, the solution was filtered using a vacuum pump (Primatec), the solution stored in a dark vial to avoid contact with sunlight to avoid possible degradation and the samples were analyzed for absorption of the brand GBC - Scientific Equipment.

### 3. Results and discussion

#### 3.1 Characterization of pins

When analyzing a sample of microprocessor pins in the SEM, its morphology was observed and it was scanned to analyze and identify the elements present in the sample. The analyzed areas can be observed in Figure 2, and the values in percentage corresponding to the mass of the existing components presented in Table 1.

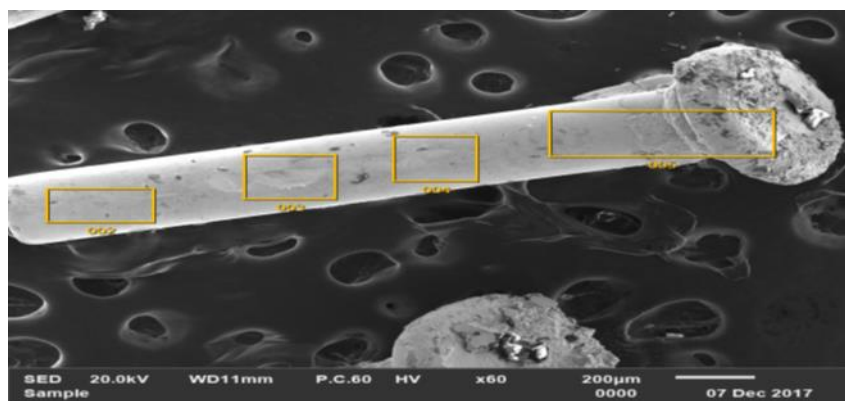


Figure 2. Points analyzed from the microprocessor pin 2.

Table 1. Percentage of components present in each analyzed area.

Points	O	Au	C	Na	Al	Sn
001	20.40	20.51	47.75	-	-	11.33
002	-	99.68	-	-	0.32	-
003	-	99.70	-	-	0.30	-
004	-	99.63	-	-	0.37	-
005	-	49.57	-	0.46	-	49.97

During the analysis, the presence of Oxygen (O), Gold (Au), Carbon (C), Sodium, Aluminum (Al) and Tin (Sn) were observed. Gold presented the highest percentage in mass; however, it is also observed that, in the upper and lower parts, the percentage was not high. In view of this, the assumption is made that the pins of these microprocessors are only gold-plated and composed of another metal in their inner part as - for example - Al and Sn.

### 3.2 Leaching

The leaching tests were carried out with the three acids: regia, citric and acetic water, analyzing them at different times - 60 and 360 minutes. After filtration of the solution obtained by the leaching, it was observed that the aqua regia had dissolved in solution all the pins; in tests with citric and acetic acids; however, the same can not be observed, since the pins still remained practically in their initial form, as shown in Figure 3.

In order to verify the amount of gold solution obtained from the leach, the samples were analyzed in atomic absorption spectrophotometry. From these results, it was verified that acetic acid did not present concentration of gold; the concentration in mg / L of gold presented in aqua regia and citric acid, however, can be verified in Table 2.



Figure 3. Comparative after filtration of the solution obtained by leaching.

Table 2. Concentration of gold in mg/L.

Timing (min)	Acid	Concentration
60	Aqua regia	10,045
360		10,342
60	Citric acid	0,550
360		0,493
60	Acetic acid	0
360		0

Observing the results obtained by leaching with aqua regia from Côrtes et al. (2015), the authors obtained about 42 mg / L in a time of approximately 30 minutes [CORTES et al., 2014], a superior result when compared to that presented in Table 2. These values are due to different conditions used in the accomplishment of the tests; among them, the agitation that was not the same when compared with that of the authors. The percentage of gold used to coat the pins of the microprocessors may have been reduced over the years. Concentrations in g / kg were then calculated for water and citric acid, as shown in Table 3.

Table 3. Concentration of gold in aqua regia and citric acid at different times.

Timing (min)	Acid	Concentration
60	Aqua regia	6,1745
360		6,25
60	Citric acid	0,0332
360		0,02955

It is observed that the highest concentration of gold was for the time of 360 minutes when aqua regia was used; however, from the shortest time to the greatest, there were no significant increases in the concentration extracted. Due to this fact, the leaching in a time greater than 60 minutes would not be economically feasible when applied industrially because of the high energy costs, among others, to carry out the leaching.

In the leaching with citric acid, a lower result was obtained compared to what was obtained when using aqua regia, but it is observed that from 60 min to 360 min the concentration remained practically the same, also not having a significant increase in the concentration of gold in solution.

It is observed that - although the result with citric acid has a low concentration - it can extract gold from the microprocessor pins. In this connection, it is necessary to investigate other leaching tests as well as other organic acids, which may be extracted into the liquid phase.

### 3.2.1 Scanning electron microscopy analysis after leaching

The SEM analyses were performed after leaching in order to observe if any changes occurred in the external composition of the pins. Figure 4 shows the pins leached with acetic acid in 60 minute (a) and 360 minute (b) times, and the scan was performed as analyzed in Table 4.



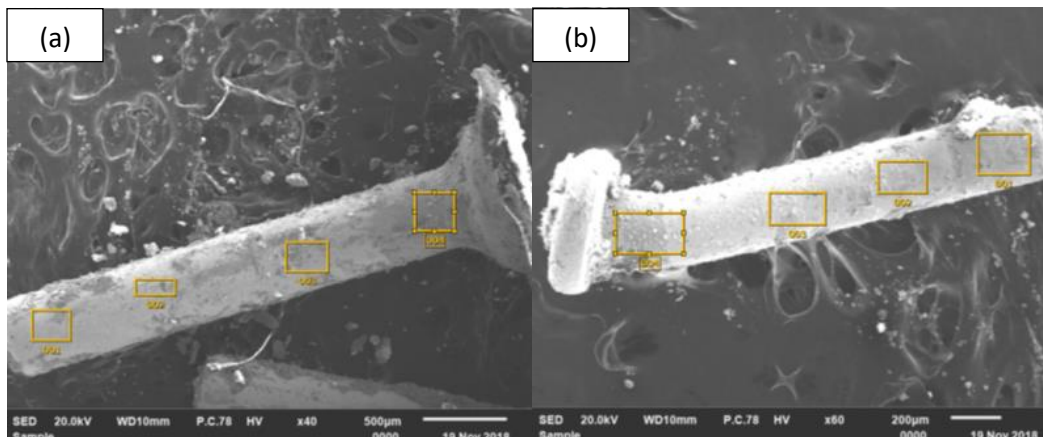


Figure 4. SEM analysis after leaching with acetic acid 60 min (a) and 360 min (b).

Table 4. Percentage of components present in each analyzed area.

60 minutes							360 minutes		
Points	O	Au	Al	Cl	Sc	Sn	O	Au	Sn
001	-	99,66	0,34	-	-	-	10,17	89,83	-
002	14,61	85,39	-	-	-	-	8,42	91,58	-
003	12,33	87,67	-	-	-	-	13,33	86,67	-
004	8,08	-	-	2,33	2,08	87,52	18,07	42,94	38,98

When analyzing the results obtained compared to the previous analysis of the leach, it is observed that the percentage of gold remained practically in the same range. This fact was already expected, due to the low extraction performed by acetic acid. It is also observed that there is a greater number of metals and other elements in the analysis of 60 minutes, a fact that does not occur for the analysis of 360 min: this only presents Au and Sn of metals.

SEM analyses were performed after leaching - also for the leached pins with citric acid analyzed in 60 minutes (c) and citric acid in 360 minutes (d), as can be verified in Figure 5. Then, the scanning was performed as shown in Table 5.

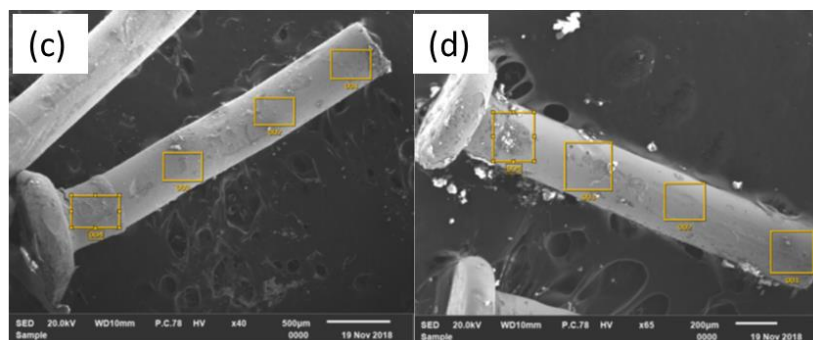


Figure 5. Percentage of components present after leaching with citric acid 60 min (c) and 360 min (d).

Table 5. Percentage of components present after leaching with citric acid.

Points	60 minutes						360 minutes		
	O	Au	Al	Si	Sc	Sn	O	Au	Sn
001	14,16	85,64	-	-	-	-	-	99,70	0,30
002	-	99,63	0,37	-	-	-	-	100,00	-
003	-	99,53	0,47	-	-	-	-	99,68	0,32
004	4,15	15,04	-	1,22	2,24	77,34	27,61	72,39	-

Analyzing the results from the leaching with citric acid, it is also discussed that the Au values remained in the same percentage range when compared with those obtained with the non-leached strip. It is also seen the presence of other metals in the 60 min analysis that are not present in the 360 min sample. It is believed that for both acetic and citric acid the presence of more metals in the first time than in the second one is due to the leaching time, in which more metals can be leached in 360 minutes, which were not analyzed in atomic absorption to verify the present concentration.

## 5. Conclusion

By performing the analysis of the obsolete microprocessors through the SEM, it is verified that there is a high percentage of Au at the analyzed points, which justifies the interest in recovery of this high value-added metal. In addition to Au, Al and Sn were also present. After the leaching at 60 and 360 min, the concentration of Au present in the solution of aqua regia, citric acid and acetic acid could be analyzed in atomic absorption spectrophotometry. The concentrations of Au present in water solution ranged from 10,045 mg / L and 10,342 mg / L for the 60 and 360 minute times, respectively. For citric acid, the concentrations presented were 0.550 mg / L for 60 minutes and 0.493 mg / L for 360 minutes; however, for acetic acid, it did not present Au in solution, thus concluding that acetic acid was not able to extract Au from the microprocessors. From the above, it can be considered that - with the use of organic acids - it is possible to perform Au extraction from obsolete microprocessors; however, it is necessary to carry out more in-depth studies to improve the extraction conditions, allowing a more efficient extraction.

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