SILVER RECOVERY FROM END-OF-LIFE LED LAMPS BY THIOUREA LEACHING

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Abstract

Light Emitting Diode (LED) are gradually replacing the conventional lamps (fluorescent and incandescent) due to their saving on energy bill of up to 85%, their lifetime 5 to 8 times longer and their mercury free composition. Due to the recovery potential of precious metals and rare-earths, end-of-life LEDs are now interesting from the point of view of recycling. This paper deals with the silver recovery of printed circuit boards of end-of-life LED lamps. The characterization of the mixed batch of LED lamps was carried out to determine the initial concentration of silver. Subsequently, for recovery of silver, thiourea, an alternative leaching agent to cyanide, was used. The parameters evaluated were: time of digestion, temperature, concentration of thiourea and percentage of ferric ions. The results obtained in the characterization showed that the LED lamps had an initial concentration of 379.33mg.kg^{-1} of silver. According to the statistical analysis, the growth of the percentage of silver leachate occurred with the decrease of reaction time and temperature. The parameters thiourea concentration and the addition of ferric ions were not as relevant when correlated with the increase of silver recovery. The optimal conditions resulted in 75% extraction of the silver, presented at 25°C, reaction time of 1 hour, thiourea concentration of 30 g.L^{-1} and addition of 0.4% of ferric ions. The results obtained demonstrated the recycling potential of the end-of-life LED lamps and the technical viability for recovery of silver by thiourea.

Keywords: LED lamps, thiourea, silver, e-waste, recycling.

1. Introduction

The LED market has grown exponentially due to the increase in the variety of LED lamps applications [1]. In projections by the United States Department of Energy (US DOE), the market for LED lamps will dominate the light trade by 84% in 2030, reducing energy consumption by light sources up to 40% [2].

In addition to its rapid insertion in the market, LED lamps are valued in the recycling sector because of their potential for reuse of metals [3]. The gold and silver contained in the LED lamps, metals that arouse greater economic interest, can be recovered by means of the hydrometallurgical process, which is currently applied on an industrial scale for other electro-electronic residues [4].

Among the methods used for gold and silver recovery, cyanide leaching is widely applied because of its low cost, high recovery rate, and accelerated reaction, but due to the risk to human health and potential environmental contamination, alternative techniques can be explored [5]. Thiosulfate is a non-toxic and affordable reactant, however, to achieve a competitive recovery rate, a high amount of the leaching agent must be consumed, increasing the price [6]. The silver extraction by thiourea was considered due to the high reaction speed and lower environmental impact when compared to cyanide recovery, being optimized in this study [7]. To achieve it, the experimental planning technique was used, this method is based in an optimization process that aims to improve the performance of a system, revealing conditions in which the application of a process produces the best possible response [8].

According with the literature reviewed, the thiourea concentration, the percentage of ferric ions, the temperature and the digestion time stand out among interfering parameters in the silver recovery process of electronic scrap since they can directly influence the efficiency and indirect costs of the non-cyanide treatment [7].

The aim of this work was to study the behaviour of thiourea as an alternative lixiviant to cyanide, optimizing experimental conditions for greater efficiency in the recovery of silver from printed circuit boards (PCB) and electronic components of LED lamps.

2. *Materials and Methods*

The methodology used in this work involved two steps. The first step includes the pre-treatment of the samples and the determination of metals. The second step set the use of thiourea as an alternative leaching agent to cyanide in the extraction of silver in the PCBs and electronic components of end-of-life LED.

2.1 Characterization of LED lamps

11 kg of end-of-life LED lamps were collected, composing a mixed sample batch consisting of 16 different models. Since the samples were collected in different sources, their shapes and sizes varied according to the functionality (commercial, residential, industrial) and the manufacturer. After assessing the integrity, the LED lamps were manually disassembled. The mechanical processing of the PCBs and electronic components aimed to increase the contact surface and expose the metallic elements to the leaching agent. The samples were guillotined and after comminuted by a ring mill.

For the acid digestion experiments, 12 comminuted samples with a mass of 2.5 g were used, which were digested using aqua regia as a leachant. The aqua regia digestion was carried out at room temperature, the reaction time was 24 hours [9], and the solid/liquid ratio was 1:20 [10].

After the reaction, the cooled suspensions were filtered, resulting in two fractions: the leaching liquor (composed of the acid solution containing the solubilized metals) and the insoluble fraction retained in the filter paper (containing non-soluble solids, polymers and ceramics). The leaching liquors were diluted, and aliquots were analysed by the ICP OES (inductively coupled plasma optical emission spectrometry). The filter papers containing the insoluble fractions were oven dried and placed in a muffle for the loss-on-ignition process.

2.2 Silver recovery from thiourea leaching

The experimental procedure of silver leaching by thiourea was adapted according to the methodology proposed by Jing-Ying et al. [7]. In addition, to investigate the effect of thiourea concentration, percentage of ferric ions, temperature and different digestion times on solubilization of silver, the tests were performed according to the experimental design of Box-Bhenken from the levels listed in Tab. 1.

Table 1: Experimental design of Box-Bhenken - Independent parameters and their respective values for each level

Parameters	Levels		
Code	-1	0	+1
Thiourea concentration (g.L^{-1})	10	20	30
Fe^{3+} concentration (%)	0.2	0.4	0.6
Temperature (ºC)	25	30	35
Time (h)	1	2	3

Based on Box-Behnken's planning methodology, as four factors and three centre point repetitions were selected for the test, it resulted in a total number of twenty-seven experiments presented in Tab. 2.

Table 2: Box-Bhenken planning matrix.

Experiment	Temperature (°C)	Time (h)	Thiourea concentration (g.L^{-1})	Fe^{+3} (%)
7	30	2	10	0.6
1	25	1	20	0.4
6	30	2	30	0.2
2	35	1	20	0.4
4	35	3	20	0.4
8	30	2	30	0.6
3	25	3	20	0.4
9	30	2	20	0.4
5	30	2	10	0.2
15	30	3	10	0.4
10	25	2	20	0.2
11	35	2	20	0.2
14	30	1	10	0.4
16	30	1	30	0.4
13	35	2	20	0.6
18	30	2	20	0.4
12	25	2	20	0.6

17	30	3	30	0.4
20	35	2	10	0.4
22	35	2	30	0.4
26	30	3	20	0.6
19	25	2	10	0.4
21	25	2	30	0.4
27	30	2	20	0.4
24	30	3	20	0.2
25	30	1	20	0.6
23	30	1	20	0.2

For the execution of experiments, acid solutions of thiourea in concentrations of 10, 20 and 30 $g.L^{-1}$ were prepared. In an Erlenmeyer the thiourea solution was added at the solid/liquid ratio of 1.0 g sample to 80 mL leachant [7]. With manual stirring, to release the percentage of 0.2, 0.4 and 0.6% ferric ions (Fe^{3+}), respective amounts of iron (III) sulphate were added.

Then, the PCB comminuted sample was added to the Erlenmeyer and the pH was adjusted to 1.0 ± 0.3, according to Trindade [10], using a 0.5 mol/L sulfuric acid solution, because according to Birloaga and Vegliò [11], the dissolution of precious metals in thiourea is strongly influenced by the acidity.

The system was heated to the desired experimental temperatures (25, 30 and 35°C) in a shaker (Model TE-4200, Brand: Tecnal) under constant stirring at 200 ± 2 rpm. After the reaction time for each experiment, the samples were filtered, and the silver concentration was determined by Flame Atomic Absorption Spectroscopy (FAAS).

After obtaining the results generated by the sequence of DBB experiments presented in Tab. 2, response surfaces were used, which describe how the response of interest varies according to two parameters that most act upon silver recovery rate. This surface allows to evaluate and predict the parameter evolution over the experimental domain [8]. The mathematical model generated by a multivariate planning must provide high model accuracy, showing a good regression and adjustment coefficients [12]. In these terms, the analysis of variance (ANOVA) was used to validate this mathematical model, guaranteeing their suitability to the answers obtained experimentally and numerically, evaluating the quality of its adjustment and estimating the interactions of the variables on the results.

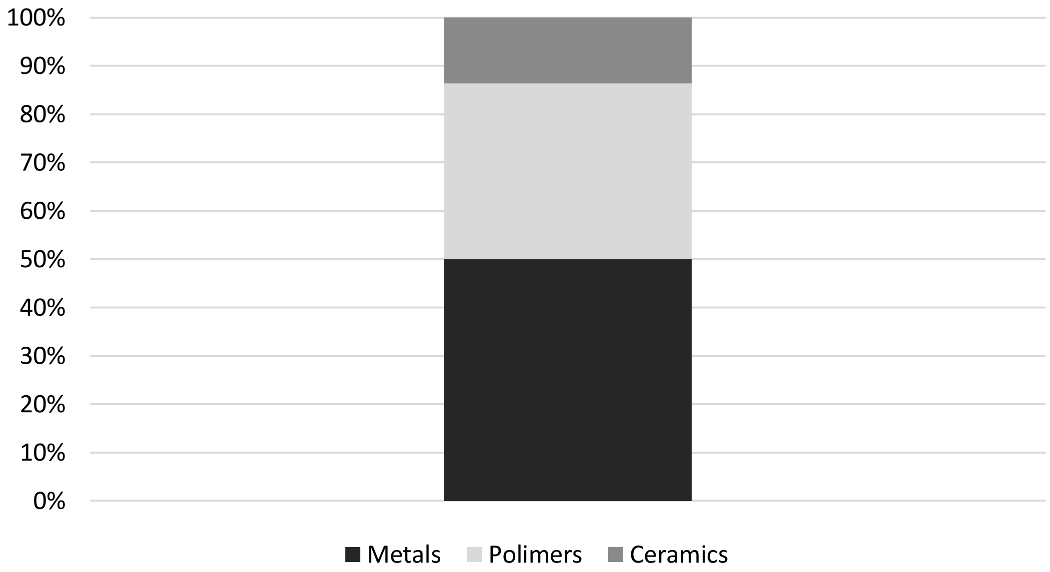
3. Results and Discussion

3.1 Characterization of LED lamps

Aqua regia leaching, loss-on-ignition and chemical analysis were performed to determine the composition of printed circuit boards in metals, polymers and ceramics fractions and to quantify the metal content.

As can be seen in Fig. 1, the metallic fraction after aqua regia digestion represented 50%, for the polymers and ceramics, the fraction presented 36% and 14% respectively. The composition of PCBs can vary depending on the type of equipment, time of use, size, manufacturer, among other factors. However, PCB containing 30% of metals are already considered economically feasible for recycling Kumar, Holuszko & Espinosa [13], which increase the potential of LED light lamps how alternative source of metal recovery from the economic point of view.

Figure 1: Composition of PCB and electronic components of end-of-life LED lamps leached by aqua regia.



With the characterization process it was possible to determine the metal content from the printed circuit boards and the electronic components of end-of-life LED lamps. While Graedel [14] affirms that the PCB composition may include more than 60 different elements, Isildar et al. [15] describes the complexity in being able to selectively leach all the elements present in this equipment. As is known, the use of aqua regia is commonly reported for characterization of metals such as gold, silver, copper and aluminium from WEEE, as can be seen in Tab. 3 [16].

Table 3: Metals concentration (mg.kg^{-1}) in PCB and electronic components of LED lamps.

Metal	Average concentration (mg.kg^{-1})
Alumminium	110.923,00 ± 3.641,78
Antimony	212,50 ± 14,41
Arseny	66,00 ± 2,65
Cerium	Not detected
Cooper	94.492,00 ± 6.255,39
Galium	Not detected

Itrium	Not detected
Nickel	761,00 ± 425,54
Gold	348,50 ± 55,06
Silver	384,00 ± 22,00

In the studies of Lim et al. [17], Lim et al. [18] and Tunsu et al. [19] it is observed that the presence of noble metals is quantitatively higher in LED and MICROLED lamps when compared to incandescent and fluorescent lamps. Gold, which has been quantified in concentrations between 2.2 $mg.kg^{-1}$ and 115.9 $mg.kg^{-1}$ in LED and MICROLED lamps, is not present in incandescent and fluorescent lamps. Silver has been quantified in other types of lamps but appears in lower concentrations. In LED lamps and MICROLED was detected in the range of 159 $mg.kg^{-1}$ to 520 $mg.kg^{-1}$ and in conventional lamps from 12.20 $mg.kg^{-1}$ to 16.20 $mg.kg^{-1}$ [17, 18]. Precious metals are undoubtedly the elements that push the recycling of WEEE, and therefore, PCB can be an excellent source for recovery [20, 21].

Park & Fray [10] found that although the concentration of gold and silver make up less than 1% by mass of computer PCBs, these may correspond to approximately 80% of the financial recovery in the sale of the recovered metals. Comparing the results obtained with fluorescent and incandescent lamps it is possible to highlight that LED lamps have a superior recycling potential for gold and silver, confirming the financial viability for recovery of these metals. The concentrations of gold and silver found in PCB of LED lamps studied (Tab. 3) are similar to those found in cell phones, computers and laptops [9, 10, 22, 23].

3.2 Silver recovery by thiourea leaching

The experimental routine was performed according to the planning matrix presented in Tab. 2, so that the silver concentrations were obtained for each experiment, resulting in their respective silver recovery rates (Tab. 4).

Table 4: Silver recovery (%) obtained in each experiment.

Experiment	Silver recovery (%)
7	30.92
1	69.58
6	17.05
2	30.54
4	15.39
8	52.68
3	16.98
9	32.29
5*	11.96

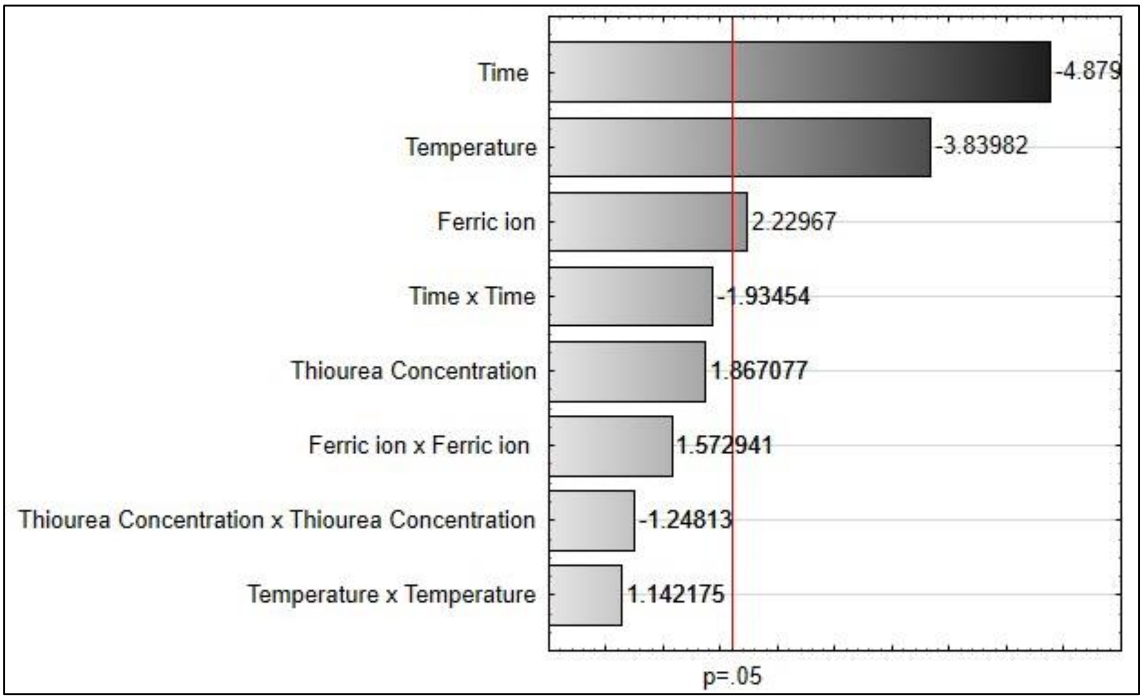
15	32.51
10	42.75
11*	11.96
14	66.85
16	75.11
13	24.56
18	38.50
12	39.56
17	49.26
20*	11.96
22	19.40
26	23.95
19	37.21
21	44.34
27	28.35
24*	11.96
25	42.44
23	39.11

Legend: * Experiment presents concentration values below the quantification limit of the method (0.158 mg.L^{-1}).

It is observed in Tab. 4 that the maximum value obtained for silver recovery was 75.11% under the optimum conditions: temperature of 30°C, digestion time of 1 hour, concentration of thiourea 30 g.L^{-1} and 0.4% of ferric ions. The minimum recovery values were presented in experiments 5, 11, 20 and 24, which the detection limit of the method of 0.158 mg.L^{-1} was considered.

Based on the assumption that the analysis of the influence of the parameters on the silver recovery is essential for the optimization of the experiment, the Pareto Diagram and the ANOVA were used. The Pareto Diagram (Fig. 2) represents the standardized estimated effects that each variable exerts on the evaluated response. The dotted vertical line indicates the level of confidence, in other words, the rejection limit of the null hypothesis ($p = 0.05$), and thus, for the response evaluation, only the extreme right effects of the response should be considered.

Figure 2: Pareto Diagram of parameters effects on silver recovery.



Source: STATISTICA 10.0

The analysis of the Pareto diagram (Fig. 2) indicates that the variables time and temperature are significant and present a negative signal, so for both parameters, when their values decrease the recovery of silver increases.

To confirm the statistical significance of the variables effects in the recovery of silver from LED lamps, the analysis of variance (ANOVA) was performed as shown in Tab. 5. At Tab. 5, *p* Value indicates the probability that each variable has not be considered statistically significant for the answer, in other words, to be inside the region of acceptance of null hypothesis.

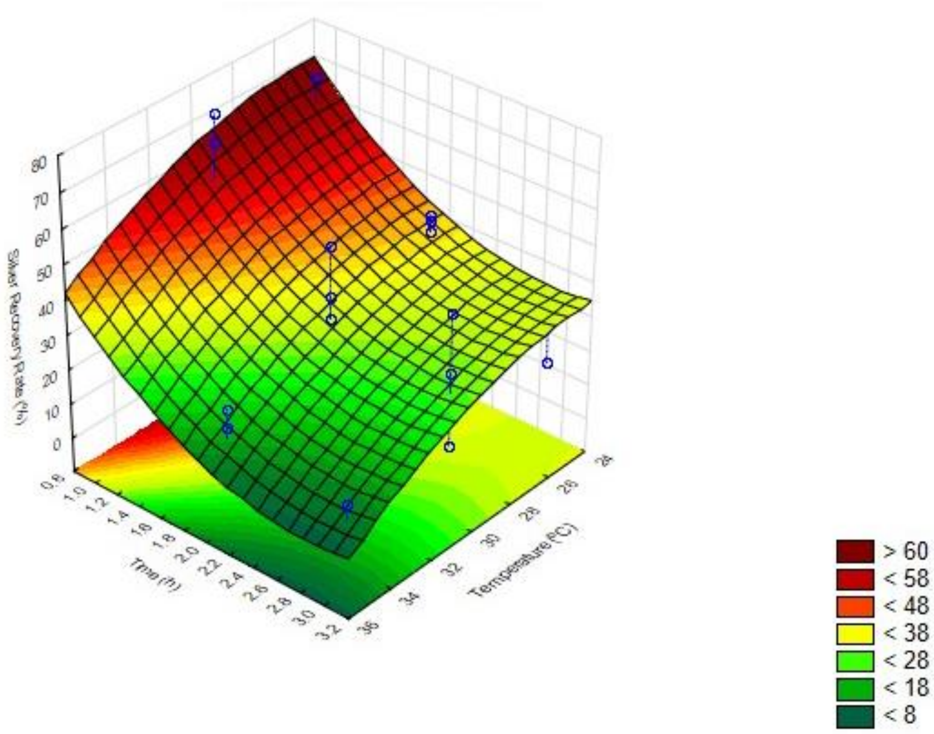
Table 5: Analysis of variance (ANOVA) for silver recovery.

Source	**Freedom degrees**	**Sum of squares (Adjusted.)**	**Average squares (Adjusted)**	**Value *F***	**Value *p***
Temperature (°C)	2	1692.71	846.36	8.02	0.003
Time (h)	2	2905.50	1452.75	13.77	0.000
Tiourea concentration (g.L^{-1})	2	531.98	265.99	2.52	0.108
Fe $^{3+}$ concentration (%)	2	785.30	392.65	3.72	0.044

For silver recovery, the variables time, temperature and percentage of ferric ions are outside the rejection limit of the null hypothesis. These results confirm the analysis of the Pareto Diagram (Fig. 2). The model presented R² of 77.09%, demonstrating a satisfactory adjustment for the experimental conditions implemented.

To better visualize the interactions between the significant variables and the optimum operating point to recovery silver in LED lamps, the following graph (Fig. 3) shows the response surface as a function of the analysed variables.

Figure 3: Response surface as a function of time and temperature in silver recovery rate.



Source: STATISTICA 10.0

The response surface (Fig. 3) was determined as a function of time and temperature variables, as they were significant to the silver recovery process. Analysing together all the optimized variables, it is possible to work with minimum values of thiourea concentration (10 g.L^{-1}), since the parameter does not have a significant influence on silver recovery. The value of ferric ions percentage was set up at 0.4%, since it is the medium value among the analysed ones.

It is observed in the red colour area on the surface shown in Fig. 3 that to obtain better results for silver leaching, the experiment should be conducted at temperatures lower than 25ºC and reaction times less than one hour. Silver recovery responses for digestion times greater than 2 hours were usually less than 30% because of the element precipitation when exposed to this condition.

From the bibliographic research carried out, studies using thiourea as leachate were found to recover silver from e-waste, being summarized in Tab. 6.

Table 6: Results compilation about silver extraction by thiourea in e-waste.

Reference	E-waste	Optimal conditions	Silver extraction (%)
Ficeriová *et al.* [24]	Electrical and eletronics PCB	20ºC; 2h; 10 g.L^{-1} CS$(NH_2)_2$;	83%
Lee *et al.* [1]	Computer PCB	Room temperature; 7h; 5g.L^{-1} CS$(NH_2)_2$; 0.7% Fe^{+3}	100%
Jing-Ying *et al.* [7]	Cell phone PCB	25ºC; 2h; 24 g.L^{-1} CS(NH2)2; 0.6% Fe^{+3}	50%
Lee et al. [25]	Secondary source from PCB	60ºC; 4h; 60 g.L^{-1} CS$(NH_2)_2$; 0.5 M H_2O_2	100%
Present study	LED lamp PCB	30ºC; 1h; 30 g.L^{-1} CS$(NH_2)_2$; 0.4% Fe^{+3}	75%

According to Tab. 6, Ficeriová et al. [24] recovered 83% of silver from electro-electronic waste leaching with thiourea at concentration of 10 g.L^{-1}. At concentration of 24 g.L^{-1}, Jing-Ying et al. [7] recovered only 50% of silver. Lee et al. [1] leached in concentration of 5 g.L^{-1} 100% of silver contained in computer PCB, also Lee et al. [25] recovered 100% of silver in secondary sources of WEEE, but using the concentration of 60 g.L^{-1}.

It is observed in Tab. 6 that different concentrations of the leaching agent were used, corroborating with the fact that the heterogeneity of the WEEE is a determining factor in the solubilization of precious metals. In comparison to this study, the parameters of Jing-Ying et al. [7] are close to those optimized, since the extraction efficiency of 75.11% was presented for concentrations of 30 g.L^{-1}.

According to Tab. 6, Jing-Ying et al. [7] when studding the parameters that influence the leaching of silver with thiourea, concluded that the ideal temperature was about 25 ºC for leaching in 2 hours. Lee et al. [1] reached optimal parameters (100% silver recovery rate) by heating the sample at 27 ºC for a reaction time of 7 hours. Ficeriová et al. [24] determined a temperature of 20 ºC to recover 83% of the silver present in PCB, but the reaction time was established in 2 hours. Despite the recovery of 100% of silver, the reaction occurred in 4 hours and at a temperature of 60ºC, consuming more energy than the others analysed [25].

Although the temperature proposed by Jing-Ying et al. [7] is similar to the used in this work, the reaction time was higher than the optimized (one hour). In view of the results obtained and the studies presented in Tab. 6, it is noted that for reaction times of less than 2 hours greater efficiencies were obtained in silver recovery, this is explained, because in long reaction times silver tends to precipitate when in contact with the leachate. Another important factor is the temperature should not exceed 30ºC, because according to Trindade [10], thiourea is a thermally unstable compound, which loses its solubilization power at high temperatures. The study that was contrary to the above mentioned was Lee et al. [25], however this can be explained due to the use of H_2O_2 as oxidizing agent.

In contrast to many studies shown that ferric ions present as good oxidants, reducing thiourea consumption [10]. Only Lee et al. [1] and Jing-Ying et al. [7] added ferric sulfate in the silver leaching reaction from WEEE. The Fe^{3+} concentrations used were 0.7% and 0.6%, respectively, being higher than that defined in this study, 0.4%. Ficeriová et al. [24] did not add ferrous ions to the system and obtained 83% recovery of silver. With these facts, is possible to infer that further studies involving the use of this oxidizing substance are required to determine the ideal amount to be used for silver recovery.

According to Lee et al. [25] the use of H_2O_2 to the in contrast to the addition of ferric ions occurred to avoid the accumulation of impurities in their samples, but it should be noted that the oxidizing substances can be powerful consumers of the leachate, depending on the experimental conditions.

In view of the bibliography, which no study was found to recover silver from LED lamps and from the results obtained, it is worth mentioning that gold recovery may also be interesting considering the need for studies in this area, in addition to its economic viability.

For gold recovery only one study for LED lamps was found, being performed by Murakami et al. [10]. The authors obtained 100% gold recovery from the experimental cycle composed of leaching in aqua regia; commercial polyamine adsorption using thiourea as eluent; and precipitation from the addition of sodium borohydride.

An economic evaluation of the recycling of WEEE was made by Cucchiella et al. [26] which drew up a list of the ten most profitable materials (Gold, copper, palladium, plastics, silver, aluminium, tin, barium, platinum and cobalt), where gold alone represents 50.4% of potential profit of recycling, demonstrating the economic potential of recycling LED bulbs, and together with silver the total is 54%.

4. Conclusion

Due to the trend of use, application variety and replace of conventional lamps, the LED lamps recycling is both environmentally and economically necessary. For this purpose, silver thiourea recovery tests were performed and when compared to cyanidation, have advantages such as: higher reaction velocity associated with lower toxicity and cost with resulting in an efficiency above 75%.

From the parameters evaluated (thiourea concentration, percentage of ferric ions, digestion time and temperature), the sample digestion time and the solution temperature had a higher influence on silver leaching when compared to the other parameters. The solution containing 30 $g.L^{-1}$ of thiourea and 0.4% of Fe^{3+} was the most efficient for the leaching of silver. Under this condition, 75.11% of the silver was extracted from the LED bulbs of LEDs in 2h and at 30 ºC.

The results obtained in the present study confirmed the high recycling potential of end-of-life LED lamps, the technical viability for silver recovery by thiourea and the possibility of gold recovery.

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