SILVER RECOVERY PROCESS FROM ELECTRONICS WASTE

Gavriş Georgeta[*], Cărăban Alina[*], Stoia Marcela[**], Hanga Mihaela[*]

**University of Oradea, Faculty of Sciences, Universitatii Street, No.1, 410084, Oradea, Romania, e-mail: georgeta_gavris@yahoo.com*
*** University "Politehnica" Timisoara,*

Abstract

Waste solid ceramic condensators coming from electronics industries are sources of important quantities of silver and require management as a hazardous waste. This paper is a study about silver possibility recovery from acid silver nitrate solutions in shape of white, crystalline, salt of silver oxalate .The optimum recovery degree performed was 98,88% like silver oxalate, because of the low value of its solubility product. The working determinate parameters were: pH 5,5, 100% excess reagent oxalic acid 0,5M as precipitation reagent and 70^0C.

Key words: silver, recovery, solide waste, silver oxalate

INTRODUCTION

In electronics factory of Curtea de Arges Romania, there are some quantities of waste ceramic condensators, because of their unavailable value of capacity. These waste solid ceramic condensators had on their surface metallic silver „Degusse"- France and a special solids resin.

Silver is considered a toxic heavy metal by the Environmental Protection Agency. The content of silver in metallic state on ceramic condensators surfaceses, may contain high silver concentration and may be dangerous.

We collect the waste for off-site treatment and reported studies on acid liaching, extraction of silver nitrate up to about 3,75g Ag/L.

The silver recovery from this waste is possible by simple mechanical operations: rub ceramic condensators down to powder: .followed by nitric acid 1:1 chemical attack, filtering and washing solids phase of solids ceramic [1]. Resulting solutions, which contains silver nitrate, are subdued to chemical recovery by chemical precipitation. The process offers an important treatment and recovery silver as silver oxalate.salt., and also, is essential both from environmental and economic point of view. At industrial level, the recovery of silver from electronics waste could be a recycling a precious metal.

In the science literature the silver recovery is made by precipitation, ion exchange, reverse osmosis, electrolysis's, chemical reduction, metallic replacement [2,3,4,5,6]. The method we are proposed are rapidly and economic advantage up to 99 % efficiency [6].

MATERIALS AND METHODS

For these studies we used Merck reagent and volumetric solutions of oxalic acid 0,5M, silver nitrate p.a. 3,75g/L, sulfuric acid 1:1 vol., chlorhidrique acid 15%. kalium permanganate 0,01N, analytical balance Tolledo, magnetic stirrer, electrical drying, derivatograph Paulic& Paulic & Erdely-MOM Hungary., Spekol 11 Jena Germany.

Silver recovery was made by chemical precipitation under magnetically stirrer, at room temperature. For the white crystalline prepared oxalate salt, is necessary 4 hours crystals growing. After this time, silver oxalate was filtered, washed and subdued to chemical analysis.

The studies are referring to the influence of different parameters of the process:

a) influence of reaction mass pH
b) influence of reagent excess
c) temperature influence
d) chemical and physical-chemical investigations on new product: silver oxalate

RESULTS AND DISSCUSSION

a) The reaction mass pH

The experimental data regarding the influnce of the pH reaction mass on the silver recovery are shown in Figure 1.

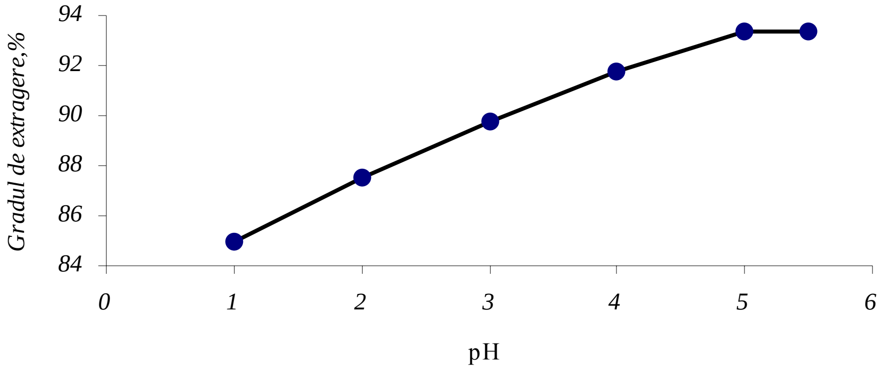

Fig 1. Silver extraction recovery dependence on the pH reaction mass

From these data, silver recovery degree is growing by increasing pH. At optimum value of pH = 5,50 silver recovery is about 93%, apart from 85% in the case of pH = 1.. The chemical equation of silver precipitation is:[8]:

$$2AgNO_3 + H_2C_2O_4 = Ag_2C_2O_4 + 2HNO3$$

$$f_{Ag2C2O4} \rightarrow Ag = 0{,}712$$

The working solution of silver nitrate, was 25 mL 3,75g$AgNO_3$/L.As corrector solution of pH was used NaOH 10% The precipitation reaction was made with 1,972mL(10%excess) oxalic acid 0,5M, 10 min. time of reaction, under magnetic stirrer, at room temperature.

b) The influence of reagent excess.

The experimental data are shown in Figure 2.

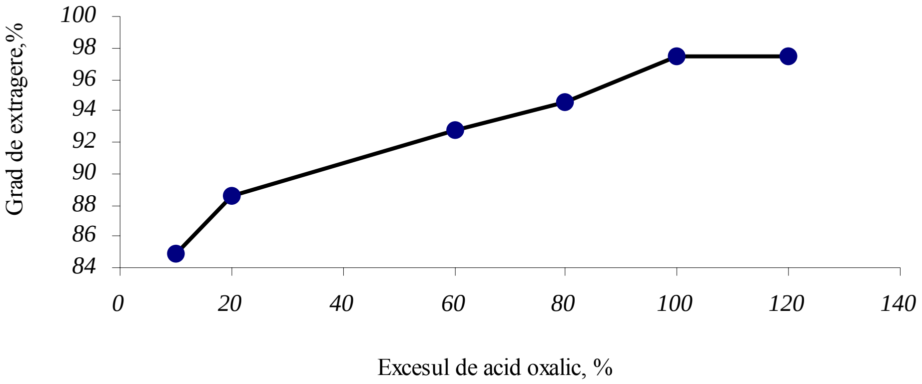

Fig. 2.Silver extraction degree dependence on the oxalic acid excess

c) The influence of the temperature reaction mass

The data regarding the influence of the temperature on the silver recovery degree are shown in Table1.

Table 1

The silver recovery degree dependence on the temperature

Nr.crt	**t^{o}C**	**α %**
1	20	97,45
2	40	97,67
3	70	98,88

The optimum recovery degree is performed for 70^{0}C.For economic conditions it could work to 20^{0}C.

d) Chemical composition

A well known quantities of silver oxalate washed and dried up to constant weight, was analyzed by dissolving it in HNO_3 1:1 warmly. The silver content was determinate by gravimetric method like halide silver, AgCl and by spctrophtometric method with 5-(4-dimthylaminobenzylidene) rhodanine, using a Spekol 11, at λ=513 nm. The results of two methods were sensible equally.

The oxalate anion was determinate with volumetric method, by kalium permanganate and sulfuric acid at 80^{0}C [9, 10, 11].

Experimental dates are shown in Table 2.

Table 2

Chemical composition of silver oxalate.

Nr.	**Ag, %**	**$C_2O_4^{2-}$, %**	**H_2O cr, %**
1	70,9992	28,9547	0,0461
2	70,9950	28,9826	0,0224
3	70,9974	28,9489	0,0537
Media	**70,9972**	**28,9620**	**0,0407**

From these data we could observed that silver oxalate is anhidrous salt.

e) Thermogravimetric and thermodipherential studies

According to [12,13], silver oxalate is blowing up at 140^{0}C and couldn't be analyzed by thermal methods

The test was made and the resulted derivatograme confirmed also chemical composition of silver oxalate (fig. 3).

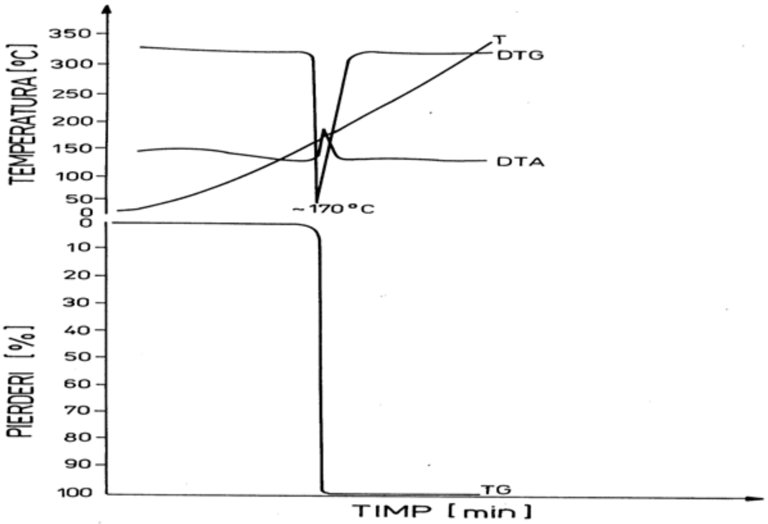

Fig.3. TG, DTG, DTA and T curves for silver oxalate

CONCLUSIONS

The optimum parameter silver recovery by chemical precipitation method are following: pH optimum =5,50, optimum excess precipitation reagent is 100%, versus stoichimometric chemical equation at room temperature. In these conditions we obtained a good value recovery degree of silver: 98,88%.All the investigations confirmed chemical composition of silver oxalate and his purity.

The influence of temperature upon recovery degree is slightly and we prepared silver oxalate at room temperature.

The purging process of solids waste ceramic condensators as oxalate, from electronics industry as oxalate, has the following advantages:

- speed of cleaning, filtering, washing- superior to the amorphic forms of silver halides or sulfur.
- low and compact volume of the precipitate
- the transformation silver oxalates, initially obtained, into other salts
- a precipitation reagent that is economically accessible (synthesis, price, transport, chemical stability)
- The optimum pH values are easy to obtain using suitable substances, also dose of oxalic acid and the temperature if it necessary.
- The crystalline white silver oxalate can be is stable against day light versus silver halides

The oxalic acid excess and can be eliminated by treating the filtered product and scouring waters with 10% milk of lime. Thus, the oxalate anion is varied as calcium oxalate and the pH of the solution is corrected, The effluent can be disposed according to the environment and waters quality legislation. The recovery silver as silver oxalate resolves the environmental problems as well as the recovery of transitional metals.

REFERENCES

1. Buzilă Lenuţa, Georgeta Gavriş, 1983, Procedeu pentru separarea argintului şi paladiului din deşeuri de pastă Pd-Ag, Certificat de Inventator Nr. 83472
2. Gavriş Georgeta, Almasi Diana, 2008, Metodă de epurare recuperativă a ionului de argint din soluții reziduale, Brevet Nr. 122093 B1
3. http://www.freepatentsonline.com/4755453.html (US Pat)
4. http://www.med.cornell.edu/ehs/updates/silver.html
5. http://www.patentstorm.us/patents/5409582/description.html
6. C.Liteanu,1961, Chimia analitică cantitativă, Ed.Tehnică, Bucureşti
7. C.Macarovoci, 1979, Analiza chimică cantitativă anorganică, Bucureşti
8. Manualul inginerului chimist, 1974, Vol II, Ed Enciclopedica, Bucuresti
9. E. Merk, The testing of water, Darmastadt
10. Polacious-Mendoza,. 1984, US Pat.No.4437889, Method for recovering silver from solid and liquid photogrphic waste