Comparative Study of the Efficiency of Sodium Hydroxide, Sodium Hypochlorite and Sodium Chloride as Extractors of Residual Silver From X-Ray Plates and Graphical Effluents by the Volhard Method

Carlos Alberto Paraguassu-Chaves¹, Cláudio José Pinto de Faria², Filomena Maria Minetto Brondani³, Vera Lúcia Matias Gomes Geron⁴, Nelson Pereira Silva Junior⁵, Izan Fabrício Neves Calderaro⁶, Fabrício Moraes de Almeida⁷ and Leonardo Severo da Luz Neto⁸

¹PhD in Health Sciences - University of Brasília - UnB, Brazil; PhD in Science - University of Havana (Cuba); Post-Doctor in Health Sciences - UnB and Degli Studi D'Aquila University - IT. Professor at the Federal University of Rondônia - Brazil. E-mail: carlos.paraguassu@gmail.com

²Professor at the Faculty of Education and Environment - FAEMA.

³Master in Experimental Biology from the Federal University of Rondônia - UNIR. Professor at the Faculty of Education and Environment - FAEMA.

⁴Master in Biology of Infectious and Parasitic Agents, Federal University of Pará - UFPA. Professor at the Faculty of Education and Environment - FAEMA.

⁵Master in Pharmaceutical Sciences from UNESP-Araquara. Professor at the Faculty of Education and Environment – FAEMA.

⁶Master in Regional Development and Environment (PGDRA/UFRO). TI Analyst of Federal University of Rondônia Foundation, Porto Velho, Rondônia (Brazil). Member of GEITEC/UNIR/ CNPq. Brazil. E-mail: izancalderaro@gmail.com

⁷PhD in Physics (UFC), with post-doctorate in Scientific Regional Development (DCR/CNPq). Researcher of the Doctoral and Master Program in Regional Development and Environment (PGDRA/UNIR). Leader of line 2 — Technological and Systemic Development, and Researcher of GEITEC — Federal University of Rondonia, Brazil. E-mail: dr.fabriciomoraes001@gmail.com

⁸Master in Education - Autonomous University of Barcelona, Spain. Master in Psychology - University of São Paulo. Master in Religious Studies - Faculty of Theological Education Logos of São Paulo. Bachelor of Nursing. Professional Physical Education. Bachelor in Theology. Professor at the Federal University of Rondonia, Brazil - Department of Collective Health, Researcher at the GEITEC and GEISC of the Federal University of Rondonia, Brazil. Email: lluz@unir.br

Abstract — Purpose: to compare the efficiency of sodium hydroxide, sodium hypochlorite and sodium chloride as extractors of residual silver from x-ray plates and graphical effluents used in the development of photolites by the Volhard method. Methodology: this is an applied and quantitative research using the Volhard method. Results: in relation to recovery of silver-containing metal from solid x-ray sheet residues, the results were 0.51g for NaCl, 0.205 for NaOH and 0.00g for NaClO respectively. The results obtained in the recovery of metal containing residual silver in photolite fixative in the presence of NaCl, NaOH and NaClO were 0.895, 0.835 and 1.330g respectively. Conclusions: NaClO was the best option for the extraction of silver-containing metal in fixation of photolites, being this 37.21% more efficient than NaOH and 32.70% more efficient in relation to NaCl. In relation to the recovery of this metal from x-ray film the results indicated NaCl as the best option for the extraction of silver contained in x-ray plates, which is 58.80% more efficient than NaOH.

Keywords — Silver Pullers, Residual Silver, Volhard Method.
I. INTRODUCTION

The silver (Argentum) is represented by the chemical symbol Ag, has atomic number 47, is considered a noble metal, having characteristics as white, bright, dense (density 10.5g / cm³), malleable and ductile used in many precious alloys [1]. Historical accounts show that the first metals to be manipulated and used by man were those that could be found in nature as a pure element, silver was one of them. Silver is believed to have been number three metal to be found and manipulated by man, with gold and copper preceding its discovery [2].

Research indicates that the oldest silver utensils date back to 5000 BC from India. Objects of this metal were also found 3500 BC, in the tombs of the province of Ur in India. There is also a reference to silver in the Bible, in Genesis 44: 2: "And my cup, the silver cup ...", which is a discovery about the time of Egyptian civilizations 3000 BC before Christ, [3].

The practice of natural extraction of silver is done through mining with methods of opening holes and grinding ores. Unlike gold, silver is present in many natural occurrences of minerals. The greatest abundance is represented by the silver sulfide - Ag2S (Argentite), in addition to the occurrence of deposits of native silver (not combined). It also states that because a great majority of the ores contain silver, the most important is the combination of three lead metals, copper and zinc, or each individually [4].

Among the various applications, silver is widely used in the photographic, radiographic, electro-electronic, coin minting and jewelry industries. It is also used in welding, mainly along with copper, zinc and cadmium, in the pharmaceutical industry, in the manufacture of evaporation tanks, tubes and coils. It has wide use together with mercury in the production of mirrors [1].

Like any other residue, those caused by the inadequate disposal of the silver metal and its abandonment in the environment, can generate serious environmental problems, favoring the incorporation of contaminating agents in the trophic chain, interacting in natural physicochemical processes, giving rise to its dispersion and , therefore, to the increase of the problem. Although nature is able to renew itself in its natural course, the accumulation of waste generated by man's action, particularly chemical substances, if they exceed the limits of natural recycling of the environment there will be an imbalance in biological systems [5]. Toxic chemicals and wastes released into the soil, sea and rivers, streams, lakes and ponds cause irrecoverable damage to the life of the fauna and flora, reaching human beings sooner or later [6].

The production of solid waste is a factor of concern for the environment, the demand for the generation of this waste increases daily and among the waste produced are the solid waste generated in health and graphics, which have a significant residual silver concentration. Silver is widely employed in the photography and imaging industries as well as in general consumer electronics and its residues may pose a risk to aquatic and terrestrial organisms [7]. The inadequate disposal of silver waste generates concern regarding environmental pollution, because it has high toxic potential [1]. The high concentration of silver is harmful to humans and can be toxic to various organisms [8]. It is assimilated by the body through ingestion, inhalation, use of medical equipment and also by direct contact with the skin. It is difficult to identify the amount of silver that is absorbed or retained by the circulation of the gastrointestinal tract, lung or absorption through the skin, however in urine and feces, these quantities can be identified [9]; [10].

In large quantities in the environment, the waste of silver in its ionic form causes toxicity and for this reason, official bodies regulate the disposal of this metal [11]. Standards established by the National Environmental Council (CONAMA), by Resolution No. 357 of 2005, establish the maximum limit of silver in liquid effluent of 0.1 mg / L. [12].

Most of the silver found in soil and surface water originates from natural leaching, this metal at high concentrations is usually associated with anthropogenic activities, such as the photographic processing industry and mining [13]. Pollution of rivers, lakes, coastal areas and bays has resulted in environmental degradation due to the dumping of increasing volumes of industrial, agricultural and domestic waste, which leads to the need to adopt government policies [14].

The disposal of silver waste can generate financial loss, since it has significant added value. It should also be mentioned that silver is one of the examples of metals with risk of scarcity [15]; [16]. However, little has been done to minimize the environmental impacts caused by films, revealing solutions, radiographic fixers and residual water [17]. On the other hand, the proper treatment of the developer and fixative solutions can generate inputs, metal and the developer itself, which strengthens the economy with the possibility of reuse [18]; [10].

Liporini et al. [19] state that the recycling of radiographic waste from hospitals and clinics as well as from the photographic industry can become a major undertaking, with possibility of financial return, from the construction of a silver extraction plant of radiographs and photographic films. The generation of waste that is sent to landfills or dumps causes damage to health and the environment and the recycling of a material that is commonly destined for waste has a great significance not only to recover material, but to make it better between man and the environment.
Waste and man-made waste are unavoidable, but reducing the negative effects of litter on the environment is critical.

In the recycling of radiographic film can be done in addition to the reuse of silver, the use of plastic that can be processed into boxes and packaging can benefit micro-packaging companies that offer a quality and affordable recycled product. Thus, a simple radiological examination that could be thrown in the garbage or stored for years is an example of 100% recycled material [19].

In this perspective, in Ariquemes, a city in the Brazilian Amazon, there are several hospitals, diagnostic centers, dental offices, radiology centers, machines for the development of graphic films (photolithography) and photographic development studios, which produce ray sheets -x, in the scope of health and residual water in the process of developing graphic and photographic films, which justifies the elaboration of this proposal of recovery of residual silver, in order to minimize the effects of the disposal of these materials in sanitary landfills, taking into account as these cause contamination of soils and groundwater, among other factors.

The present study had as objective to compare the efficiency of sodium hydroxide, sodium hypochlorite and sodium chloride as extractors of residual silver from x-ray plates and graphical effluents used in the development of photolites using the Volhard Method.

II. METHODOLOGY

This is an applied and quantitative research, insofar as the objective was to compare the efficiency of sodium hydroxide, sodium hypochlorite and sodium chloride as extractors of residual silver from x-ray plates and graphical effluents used in the development of photolites using the Volhard method, quantifying the silver present in x-ray film and in fixation of photoliths used in graphic printing.

The x-ray plates were collected in a private hospital and the fixator of the process of revelation of photolithographs, both in the city of Ariquemes, in Rondônia, in the Brazilian Amazon.

The material containing residual silver was sent to the Chemistry laboratory of the Faculty of Education and Environment – FAEMA in Ariquemes, Rondônia, Brazil, to carry out the analyzes.

The analyzes were performed using the Volhard Method with adaptations, according to the following subitems.

2.1 Removal of silver contained in x-ray plates

For the quantification of silver contained in x-ray plates, three different solutions were used, as shown in the sequence, namely:

- The x-ray plates were cut into squares of approximately 1x1cm, weighed into three 200g portions and placed in different beakers (1000 mL). As shown in figure 01.

- NaClO sodium hypochlorite (2.0%), sodium hydroxide NaOH (1 mol/L) and NaCl sodium chloride (1 mol/L) respectively were placed in each beaker until the 800 mL mark, placed on the shaker for 90 minutes and allowed to stand for 24 hours.

- In the case of the solution containing sodium hydroxide (NaOH) precipitates the silver hydroxide (AgOH) in the solution of the solution, and the others silver chloride (AgCl).

- After the filter paper containing precipitate was removed from the funnel and placed in porcelain crucibles, which were weighed in GEHAKA brand AG-200 model and counted with pencils for identification, as shown in Figure 03.
To finish the silver extraction process, samples were taken to the muffle, Quimis brand, model Q-318M25T at a temperature of 1000ºC for 120 minutes, according to figure 04.

Subsequently they were cooled in a desiccator containing silica. Finally, the sample was weighed and the metal content was calculated according to Equation 1:

\[
\% \frac{m}{v} = \frac{100 \times N}{P} = \frac{100 \times N}{P} \\
\text{(Equation 1)}
\]

At where:
- \(N\) = mass of ash (g) and
- \(P\) = initial sample mass (g)

**2.2 Removal of the silver contained in fixer of graphic photoliths**

For the quantification of the silver contained in fixador of graphic photoliths three different solutions were used, as it shows the sequence, to wit:

- A 200 mL sample of graphite photolyte fixative developer solution was placed in three 600 mL beakers, in beaker 01 200 mL of 2% sodium hypochlorite was added to beaker 02, 200 mL of sodium hydroxide was added to 1 mol/L and in the beaker 3, 200 ml of 1 mol/L sodium chloride. The solutions were then shaken for 1 hour and then allowed to stand for 24 hours, as shown in Figure 05.

The precipitate obtained in the decantation process was filtered by a simple method, as shown in Figure 02.

The filter paper containing precipitate was removed from the funnel and placed in porcelain crucibles, which were weighed in an AG-200 model GEHAKA analytical balance and enumerated with pencils for identification.

To finish the silver extraction process, the samples were taken to the muffle, Quimis brand, model Q-318M25T at a temperature of 1000ºC for 120 minutes, according to Figure 04.

Subsequently they were cooled in a desiccator containing silica. Finally, the sample was weighed and the metal content was calculated according to Equation 1. And the other steps were performed analogously to the first case.

The calculations to quantify the residual silver contained in the samples were made using Rule of Three, and the titration made from x-ray plate residues indicated the presence of 0.453 g for each 100 mL of sample and for the fixative sample of photoliths, 0.444 g was obtained for each 100 mL of the sample.

**2.3. Titration of samples**

For the titration of the samples, the Volhard Method was used with modifications, according to the sequences indicated below.

**2.3.1. Titration of the solution obtained from x-ray plate**
An aliquot (05 mL) of sample 01 containing silver to be analyzed was transferred to a 250 mL Erlenmeyer flask. 1mL of a saturated (~ 40m / v) solution of ammonium ferric sulfate was added.

The medium was acidified with 5mL of 6 mol / L nitric acid and titrated with standard 0.1 mol / L potassium thiocyanate solution.

The volume of potassium thiocyanate spent on the first sample was 2.1 mL.

2.3.2. Titration of the photolyte fixative solution

An aliquot (05 mL) of sample 01 containing silver to be analyzed was transferred to a 250 mL Erlenmeyer flask. 1 mL of a saturated (~ 40 m / v) solution of ammonium ferric sulfate was added.

The medium was acidified with 5mL of 6 mol / L nitric acid and titrated with standard 0.1 mol / L potassium thiocyanate solution.

The volume of potassium thiocyanate in the first sample was 2.2 mL.

### III. RESULTS AND DISCUSSION

3.1. Metal recovery data containing silver from x-ray sheets with different extractors

The results contained in Table 01 were obtained from 200g of sample x-ray plates with different extractors. NaCl, NaOH and NaClO, respectively.

| Sample Used         | Extraction Solution | Sample mass | Metal obtained containing Ag g/100g |
|---------------------|---------------------|-------------|-----------------------------------|
| X-Ray Plates        | NaCl                | 200g        | 1.02g                             | 0.51 |
| X-Ray Plates        | NaOH                | 200g        | 0.41g                             | 0.205|
| X-Ray Plates        | NaClO               | 200g        | 0.00g                             | 0.00 |

In relation to the recovery of silver-containing metal from solid x-ray sheet residues, the results were 0.51g for NaCl, 0.205 for NaOH and 0.00g for NaClO respectively, with NaCl being the best option for the extraction of Ag contained in the plates of x-ray, being that 58.80% more efficient than NaOH and in relation to the NaClO it was not possible to establish comparison for not having extracted metal of the sample.

3.2. Silver metal recovery data obtained from different phototractor fixators

The results contained in Table 02 were obtained from 200 mL of photolite fixative sample with different extractors. NaCl, NaOH and NaClO, respectively.

| Sample Used         | Extraction Solution | Sample mass | Metal obtained containing Ag g/100g |
|---------------------|---------------------|-------------|-----------------------------------|
| Photolithographic Fixer | NaCl                | 200mL       | 1.79g                             | 0.895 |
| Photolithographic Fixer | NaOH                | 200mL       | 1.67g                             | 0.835|
| Photolithographic Fixer | NaClO               | 200mL       | 2.66g                             | 1.330|

The results obtained in the recovery of metal containing residual silver in photolite fixer in the presence of NaCl, NaOH and NaClO were 0.895, 0.835 and 1.330g respectively, which evidenced NaClO as the best option for the extraction of metal containing silver in residues of photolyte fixative. In quantitative terms, it is worth noting that NaClO was 37.21% more efficient than NaOH and 32.70% more efficient than NaCl.

When comparing the results obtained from the titration, which was 0.474 g of silver per 100 mL of solution, with NaCl extractors of 0.895 g per 100 mL, NaOH of 0.835 g NaClO of 1.330 g per 100 mL of sample, it was observed that the titration showed smaller values than those recovered with the extractors from photopolymer fixative residues. This difference could be related to sample storage time, formation of silver precipitate and titration errors.

With respect to the data quantified by titration of the x-ray plate sample, 0.453 g of silver per 100 mL of the sample were obtained, and the NaCl extractor recovered 56.29% of silver in relation to titration data. NaOH 22.73% and 0% NaClO. The results are the same as those found by Faria [20].

The results obtained by Bortoletto et al., [21] for silver recovery, using NaOH, NaCl and Activated Carbon as precipitating agents, demonstrated that NaOH as precipitating agent provided low silver removal, there was the appearance of undesired substance besides increase of the pH of the solution in the course of the process. The NaOH solution presented precipitation results when its concentration was changed to 1.5 mol/L, removing only...
10.3% of the silver present in the fixative, when its concentration was increased to 1.8 mol/L promoted a significant increase of 40.1% in silver removal, however, the pH of the medium increased to 13.0. In the same study, it was observed that babassu coconut coal showed higher affinity with silver thiosulphate, adsorbing around 0.42 mg Ag g⁻¹ of coal. Coals of the coconut shell and 119 (charcoal type) showed almost equal adsorption capacity [21]; [22].

Silva et al., [23] obtained a yield of 13.95% using NaClO and 12.39% using NaCl as precipitating agents, but considered as a non-viable process, as their yield is low compared to the recovery of silver from electrolytic bath, for example. Several authors have stood out with studies on treatment of silver residue, recovery of silver and in broader terms of health waste in general. Among these researchers are, Maciel; Liu; Cardoso [24]; Melo [25]; Bampi; Sechi; Gonçalves [26]. Carvalheiro; Mion; Liporini; Libardi [27]; Grigoletto; Santos; Albertini; Takayanagui [28]; Hoceva; Rodriguez [29]; Camargo; Motta; Lunelli; Severo [30]; Gunther [31]; Navarro, et al., [32]; Garcia; Zanetti-Ramos [33]; ANVISA [34]; Tramontini et al., [35]; Antunes [36]; Lippel; Baasch [37].

IV. CONCLUSIONS

With the results obtained it was possible to conclude that in the silver recovery process from x-ray plates and graphite fixator with the NaCl, NaOH and NaClO extractors, NaClO was the best option for the extraction of silver containing metal in fixator, being this 37.21% more efficient than NaOH and 32.70% more efficient in relation to NaCl. In relation to the recovery of this metal from x-ray film the results indicated NaCl as the best option for the silver extraction contained in the x-ray plates, being this 58.80% more efficient than NaOH and in relation to NaClO it was not possible to establish a comparison because it did not extract metal from the sample, which suggests the performance of new tests with this extractor. It is worth noting that the tests carried out indicated that it is feasible to recover residual silver from x-ray plates and fixator of photolithography, which may contribute to minimize the environmental contamination generated by inappropriate disposal of these materials.

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