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Recovery of the silver contained in the radiographic plates using natural antioxidants from vegetables as reducing agents

Recuperación de la plata contenida en las placas radiográficas empleando como agentes reductores antioxidantes naturales de vegetales

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Abstract

This paper presents the results of a project whose objective is to recover the metallic silver contained in radiographic plates using plant extracts with antioxidant properties as silver-reducing agents. The use of this type of plant, whose choice is motivated by its high availability and wide geographical distribution, not only avoids the use of chemical reducing agents that are often expensive and toxic, but also results in a more economical and environmentally friendly method. This method offers vast possibilities considering the great diversity of plants with a wide variety of metabolites that include reducing and stabilizing agents in their composition. The recovery of metallic silver begins with the treatment of radiographic plates until reaching a compound where silver is complexed with ammonia, later this coordination compound is treated with plant extracts to reduce silver ionically. Ag+ to metallic silver Ag0. The silver recovered by this method is a clean and bright silver with an acceptable performance that depends on each of the extracts used.

Silver, Plant extracts, Polyphenols

Resumen

En este trabajo se presentan resultados de un proyecto cuyo objetivo es recuperar la plata metálica contenida en las placas radiográficas utilizando, como agentes reductores de la plata extractos vegetales con propiedades antioxidantes. El empleo de este tipo de plantas, cuya elección viene motivada por su alta disponibilidad y amplia distribución geográfica, no sólo evita utilizar agentes reductores químicos en muchos casos caros y tóxicos, sino que también resulta en un método más económico y respetuoso con el ambiente. Este método ofrece vastas posibilidades teniendo en cuenta la gran diversidad de plantas con amplia variedad de metabolitos que incluyen agentes reductores y estabilizantes en su composición. La recuperación de la plata metálica inicia con el tratamiento de las placas radiográficas hasta llegar al a un compuesto en donde la plata se encuentra en forma acomplejada con amoniaco, posteriormente este compuesto de coordinación es tratado con los extractos vegetales para reducir la plata de forma iónica Ag+ a plata metálica Ag0. La plata recuperada por este método es una plata limpia y brillante con un rendimiento aceptable que depende de cada uno de los extractos utilizados.

Plata, Extractos vegetales, Polifenoles

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Introduction

At present there is a growing concern regarding the environmental problems caused by human activity and how these problems can affect our health, among the polluting residues are those of the photographic industry whose waste contains silver salts, coming from the radiographic films used in all the hospitals of the world, these are disposed of in the garbage and have an impact on the contamination of the rivers.

Pure silver is a shiny, strong, ductile and malleable metal. Of all metals it has the highest reflective optical white color and the highest thermal and electrical conductivity. From the chemical point of view, it is one of the heavy and noble metals; commercially, it is a precious metal (Brown, 2001; Skoog, 2010).

It is a natural resource whose current demand is greater than its production, with the photographic industry being the largest user of silver in the world, due to its unique ability to react with light and produce images in applications such as photography and in the preparation of radiographic plates (Cabrero, 2004). Silver is the most important input in the X-ray service of any hospital or clinic in the world, unfortunately, once the radiographic plates have fulfilled their function, they end up being completely discarded and it can be affirmed that the silver contained in the x-rays goes to stop rivers and seas, silver that is rinsed from radiographic films and once it is diluted it is very difficult to recover it, so we must do everything possible to conserve and reuse such a valuable natural resource that exists in a limited amount in the planet, is a natural resource whose current demand is greater than its production, as Shayne McGuire reveals in his book entitled The Silver Bull Market Investing in the Other Gold. He mentions the importance of silver, highlighting that in a short time it will be the other gold in the financial markets (Shayne, 2013). Currently, there are methods to recover silver from the waste generated by the photographic sector, the most common reported in the literature are chemical methods that include replacement or metal substitution, electrolysis, ion exchange and precipitation (Fabregas et al., 2002). These techniques are usually related to a high initial cost in terms of equipment, operation and purification of the final products.

Due to this problem, there is interest in exploring methods that are less contaminating with the environment, such as biological methods, since these methods are widely used with success in the reduction of substances such as free radicals and metal ions, plant extracts being used with great success in the synthesis of nanoparticles of silver and other metals such as gold and copper using extracts (Mitta, 2013).

For this reason, the objective of this work was to analyze the behavior of various plant extracts with antioxidant properties in the recovery of metallic silver contained in radiographic plates, plant extracts that contain antioxidant compounds, among which the most important are polyphenolic compounds. (Marakarov, 2014) and flavonoids, compounds with high antioxidant capacity and low redox potentials. The low redox potentials of these antioxidants make the reduction of the vast majority of free radicals and some metals thermodynamically favorable. (Han, 2014).

Vegetables from the region were used: leaves of *Dysphania ambrosioides,* (epazote), *Coriandrum sativum* (Cilantro), *Petroselinum crispum* (Parsley), *Mentha spicata* (Hierbabuena), *Taraxacum officinale* (Dandelion), *Pimpinella anisum* (Anis), *Camellia sinensis.* (Black tea), *Salvia rosmarinus* (Rosemary) *and Rose leaves.*

The use of this type of plants, the choice of which is motivated by their high availability and wide geographical distribution, not only avoids using chemical reducing agents that are often expensive and toxic, but also results in a more economical and ecological method.

Composition of a radiographic plate

The radiographic films basically composed of two parts, the base and the photosensitive emulsion. The base is a rigid polyester material on which the photographic emulsion is placed, this being the element sensitive to both ionizing radiation and light and is fixed to the base by a thin adhesive sheet that prevents its detachment from it (Figure 1).

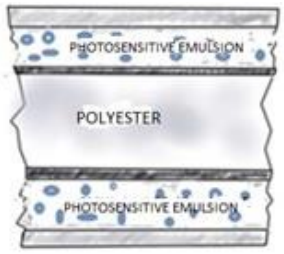

Figure 1 Image of the parts that make up a radiographic plate

The photosensitive emulsion is constituted by the gelatin in which the silver halide crystals are dispersed uniformly distributed. Halides or silver halides are chemical compounds in the form of salt that are produced by the chemical combination between some of the halogen elements (chlorine, iodine and bromine) and silver. These silver salts, under the action of radiation or visible light, undergo a series of chemical transformations that result in the production of a photographic image. The three most important halides in the manufacture of radiographic film are silver bromide (AgBr), silver chloride (AgCl) and silver iodide (AgI). In general, silver halides are sensitive to all those radiations whose wavelength reaches up to 460 nm (González et al., 1996).

The chemical reaction used to manufacture the photosensitive emulsion of the radiographic film is presented in equation (1).

$$AgNO_3 + KX \longrightarrow AgX + KNO_3 \qquad (1)$$

Silver nitrate plus potassium (bromide, chloride, iodide) will give silver halide plus potassium nitrate.

Vegetable extracts

At present, interest has arisen in the search for natural antioxidants due to their great effectiveness as reducing agents against free radicals and some metal ions, these are present in various vegetables, natural antioxidants that are generally made up of mixtures of components with high molecular diversity and biological functionality, biosynthesized by plants (in their fruits, leaves, roots, stems, seeds or other parts), components among which the most important are polyphenolic compounds and flavonoids (Marakarov, 2014; Santos et al., 2020).

Polyphenols are plant derived compounds with potent antioxidant properties found in many vegetables, fruits, seeds, and derived products (Naczk, 2006). There are several classes and subclasses of polyphenols that are defined based on the number of phenolic rings that they have and the structural elements that these rings present (Muñoz et al., 2007). Figure 2 shows the chemical structures of some of them, such as gallic acid and hydroquinone, caffeic acid, Luteolin and rosmarinic acid, present in various vegetables.

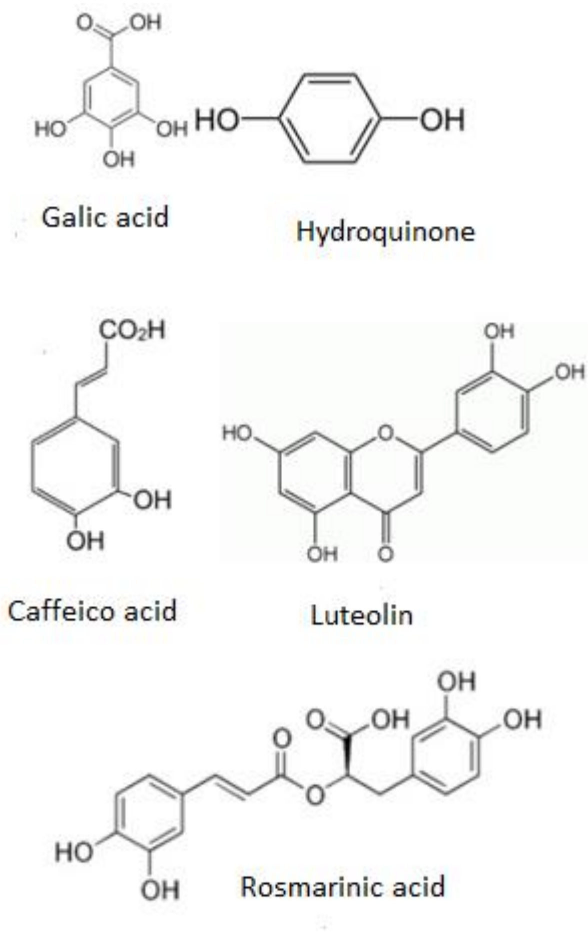

Figure 2 Chemical structure of some polyphenols present in vegetables

Polyphenols have a wide range of properties, depending on their chemical structures, including their reducing activity, which governs their antioxidant properties and their sensitivity to oxidation. The low redox potentials of these antioxidants make the reduction of the vast majority of free radicals and some metals thermodynamically favorable (Han, 2012).

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The chemical properties of polyphenols, especially their chemical reactivity and transformations, have potential implications in the field of nanotechnology, they are the most important elements in the green synthesis of nanoparticles since they play the role of reducing agents because they present chemical structures that it gives them greater reducing potential, protection and stability, since polyphenols have the ability to transfer electrons and/or hydrogen atoms during the neutralization of free radicals (Brewer, 2011).

Materials and methods

Next, the method used in the preparation of the extracts is described, as well as the steps that were followed for the recovery of the silver.

Preparation of extracts: chosen vegetables

The extraction of antioxidant compounds from plants depends to a large extent on the nature of the solvents used in this process. Polar solvents are the most used in the case of the extraction of polyphenols from plants, mainly water and alcohols, since their efficiency in the extraction of large concentrations of these compounds has been demonstrated (Sultana et al, 2009).

The preparation of extracts was carried out for each species through a solid-liquid extraction process, using water and ethanol as solvents.

The vegetables used were: *Dysphania ambrosioides leaves, popularly called Epazote, Coriandrum sativum commonly called Cilantro, Petroselinum crispum known as Parsley, Mentha spicata or Peppermint, Taraxacum officinale known as Dandelion, Pimpinella anisum or Anise, Camellia sinensis or Black Tea, Salvia rosmarinus or Rosemary and Rose* Leaves. The vegetables were previously washed, dried and subsequently crushed. At first, water was used as a solvent, which has a high polyphenol extraction capacity (Naczk, et al., 2006) and does not present any toxicity. Next, another second batch of extracts was made, replacing the water with ethanol (EtOH 96%), in order to dissolve some species with greater antioxidant capacity (Nuñez, 2008). The extracts are obtained by contacting 1.0 g of the plant with 100 ml of solvent, heating and letting it boil for 40 min at a temperature of 80 o C, after which the liquid extract is obtained by filtration.

The extracts are stored at 4ºC and protected from light.

Recovery of the silver contained in the radiographic plates

The recovery of metallic silver begins with the treatment of the radiographic plates until reaching a compound where the silver is found in a complexed form with ammonia, later this coordination compound is treated with plant extracts to reduce the silver ionic form. Ag^{+} to metallic silver Ag^{0}.

a) treatment of radiographic plates with nitric acid

200 g of radiographs crushed to an average size of 1 cm2 were placed in a 500 ml Erlermeyer flask, they were subjected to acid digestion, 600 ml of a nitric acid solution (20%, M) was added, the mixture colored blue (figure 3) was stirred for 2 hours at a temperature of 80 oC turning yellow. The yellow mixture containing the silver in the form of silver nitrate was filtered using Whatman No. 42 filter paper.

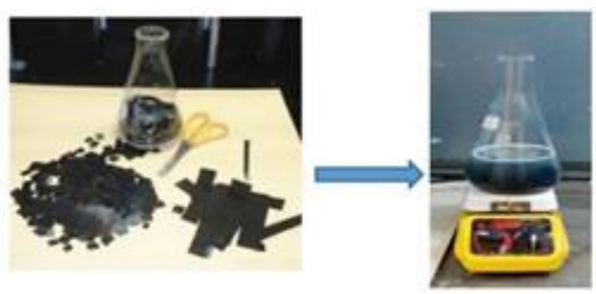

Figure 3 Acid digestion of radiographic plates

b) Synthesis of silver chloride AgCl.

The yellow filtered solution (silver nitrate) resulting from the nitric acid attack on the radiographic plates is placed in a 600 ml beaker; 2.56 g of ammonium chloride is dissolved in 40 ml of distilled water and added to the silver nitrate solution with stirring, turning milky white due to the formation of silver chloride, it is allowed to settle for one day until total precipitation of silver chloride in the form of a white precipitate. The precipitate is filtered and washed several times with distilled water; the silver chloride precipitate is then dried in a heating oven at a temperature of 25 °C for further use.

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c) Preparation of diamminsilver(I)

A 1.5 g silver chloride obtained was used in the formation of the diamminsilver(I) complex, adding 20 mL of 20% aqueous ammonia solution (Skoog, 2005), improving the reaction conditions by increasing the temperature, for the formation of the complex. diamminsilver in solution.

d) Reduction of ionic silver to metallic silver.

Once diamminsilver(I) was obtained, the recovery of metallic silver was carried out with the addition of plant extracts in aqueous solution in 100 mL alcoholic solution. Once the plant extracts were added, the silver contained in the complex was reduced to metallic silver, and finally it was filtered and weighed.

Results and discussion

The extracts obtained from the plants acquired different colors, showing that their chemical composition is not the same. Figure 4 shows the image of the plant extracts prepared using water as a solvent.

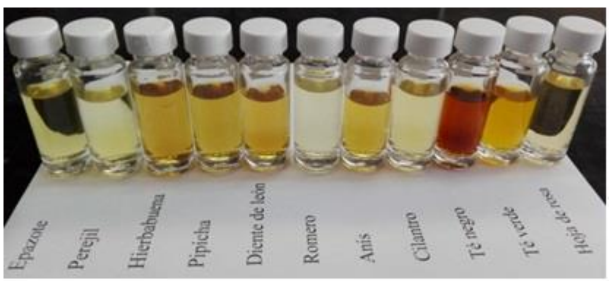

Figure 4 Plant extracts prepared in aqueous solution

Figure 5 shows the photograph of the extracts using ethanol as solvent, the shades of the colors differ totally from the extracts prepared in aqueous solution, showing a green color with different shades in most cases. It is evident that ethanol as a solvent is more selective for some of the substances present in vegetables.

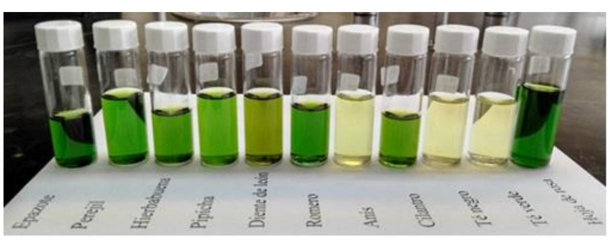

Figure 5 Plant extracts prepared in alcoholic solution

Obtaining the AgCl(s)

The dissolution reaction of the x-rays occurred on the emulsion that covers the acetate of the x-ray formed by silver halides. In the reaction with nitric acid, the x-rays discolored and formed a yellow solution, corresponding to the liquid form of the silver nitrate. Figure 6 shows the image of this chemical process and equation 2 shows the chemical reaction that occurred. (Skoog, 2010).

Figure 6 Liquid form of $AgNO_3$ silver nitrate.

$$4Ag_{(s)(x\text{-}rays)} + 6HNO_{3(aq)} \longrightarrow 4AgNO_{3(l)} + NO_{(g)} + NO_{2(g)} + 3H_2O_{(l)} \quad (2)$$

In the solution obtained, silver is found in an acid medium in the form of silver nitrate, this solution was reacted with $NH4Cl$ to obtain silver chloride AgCl, resulting in a white solid which was separated by decantation and dried to be treated. later (equation 3). Figure 7 shows the process of AgCl formation and precipitation. (Chang, 2002).

Figure 7 Formation of $AgCl_{(s)}$

$$AgNO_{3(ac)} + NH_4Cl_{(ac)} \longrightarrow AgCl_{(s)} + NH_4NO_3 \quad (3)$$

This process was carried out with three samples of 200 g of radiographs in order to obtain an average of the amount of AgCl obtained. Table 1 shows the amount of silver chloride obtained from each of the samples.

Radiographic plates 200 g	$AgCl_{(s)}$ obtained (g)
Sample 1	1.8209
Sample 2	1.6389
Sample 3	1.7586
Average	**1.7394**

Table 1 Amount of silver chloride obtained from 200 g of radiographic plates

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The amount of silver chloride obtained on average was 1.7394 g per 200 g of radiographs. As expected, the amount obtained in the three cases is not the same because the amount of silver depends on the conditions of the radiographic plate used.

Silver recovery

The obtained silver chloride was used in the formation of the diamminsilver(I) complex. For the study of silver recovery, 0.5 g of silver chloride was used in all cases, obtained from the treatment of x-rays, it was treated with 20% ammonium hydroxide in order to obtain diamminsilver(I). Equation 4 shows the chemical reaction of the process (Skoog, 2005).

$$AgCl_{(s)} + 4NH_{3(l)} \longrightarrow + H_2O_{(l)} \; 2[Ag(NH_3)_2]^+_{(ac)} + 2OH^-_{(ac)} \quad (4)$$

$[Ag(NH_3)^2]^+$ is a very weak oxidant with an oxidation potential (0.376 V), which reacts with the components of plant extracts prepared both in aqueous solution and in alcoholic solution (in ethanol), causing the silver ions are reduced to metallic silver by the presence of electrons originated by the oxidation of plant extracts, equation 5 shows the process (Chang, 2002).

$$\text{Plant extract}_{\text{(watery or alcoholic)}} + [Ag(NH_3)_2]^+_{(ac)} \rightarrow Ag_{(s)} + \text{Waste}_{(ac)} \quad (5)$$

The silver precipitated at the bottom of the container as was the case with the use of the mint extract, the addition of the *Epazote* extract generated the formation of a thin layer of metallic silver and with other plant extracts silver flakes were formed as in the case of using coriander extract. Figure 8 shows the images of the formation of silver with the extract of *Epazote* mint and coriander in aqueous solution.

Figure 8 Silver recovered with the aqueous extracts of *Peppermint*, *Epazote* and *Cilantro*

In both cases, with extracts in aqueous solution and extracts in alcoholic solution, it is observed that silver appears in the solution as a solid with a silvery appearance, as a result of the reduction of silver from $Ag1^+$ to Ag^0(s).

The precipitation of silver with aqueous extracts was carried out more slowly, and with greater speed when extracts with alcohol were used. Regarding the appearance of the recovered silver, it was observed that with the aqueous extracts the precipitated silver is obtained in the form of flakes and very fine powder and it was necessary to wash several times because residues of the extracts used remained, most likely by-products that formed in the course of the reaction. Figure 9 shows the appearance of silver recovered with aqueous extracts.

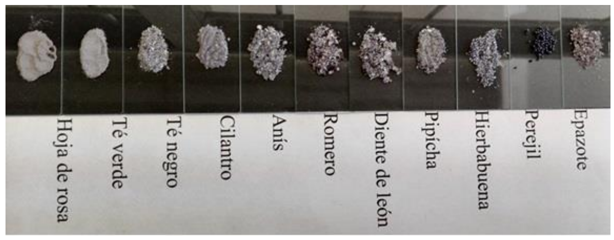

Figure 9 Silver recovered using water as solvent

On the other hand, in the case of using the extracts prepared with ethanol, the recovered silver presented a granular appearance of different sizes, from a fine powder to a powder with larger granules, silver with a cleaner appearance. These observations vary depending on the extract used and its chemical composition. Figure 10 shows the appearance of the silver recovered with ethanol extracts.

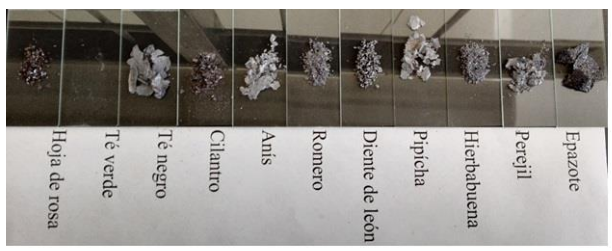

Figure 10 Silver recovered using ethanol as solvent

Figure 11 shows the photographs of silver recovered with aqueous extracts and extracts in ethanol where its appearance can be appreciated.

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Figure 11 Images of silver recovered with aqueous extracts and ethanol extracts.

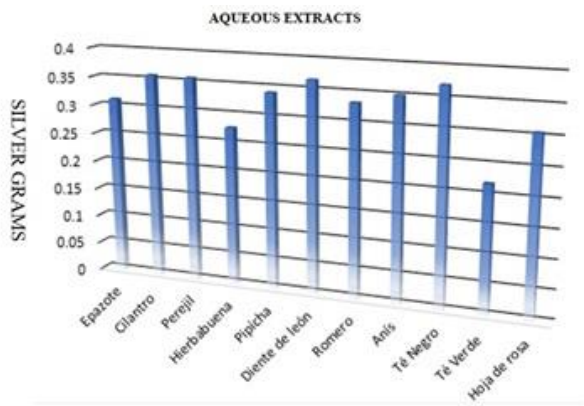
The silver obtained was filtered, washed with distilled water, then kept in the oven for 3 hours at a temperature of 100 0 C and finally weighed.

Table 2 shows the amount of silver recovered per 0.5 g of AgCl with each of the extracts in aqueous solution and ethanol.

Extract	Amount of Ag in grams (Extract in water)	Amount of Ag in grams (Extract in ethanol)
Epazote	0.3115 g	0.2447 g
Cilantro	0.3872 g	0.2952 g
Parsley	0.3766 g	0.3076 g
H. Good	0.2763 g	0.2814 g
Pipícha	0.3404 g	0.3888 g
D. de Leon	0.3659 g	0.3851 g
Rosemary	0.333 g	0.3100 g
Aniseed	0.3494	0.3100 g
Black Tea	0.3700 g	0.3000 g
Green Tea	0.2190	0
Rose Leaf	0.3055	0.3718

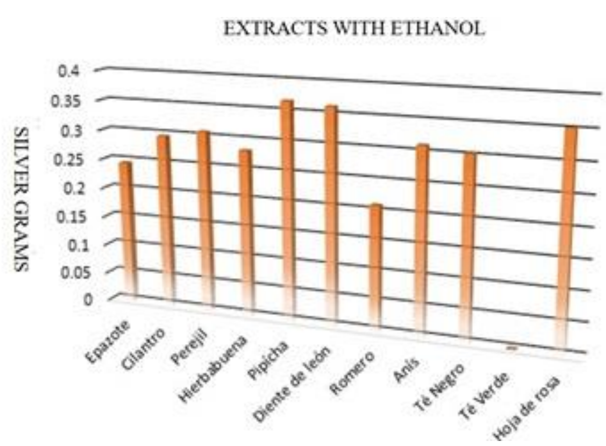
Table 2 Amount of silver recovered per 0.5 g of AgCl

Graph 1 shows the results of the amount of silver recovered with each aqueous plant extract, evidencing its behavior due to its different chemical compositions. Most likely the amount of reducing substances present in the extracts is different and consequently the amount of silver recovered. It can be seen that the best results obtained were those of cilantro, black tea, dandelion and parsley.



Graphic 1 amount of silver recovered from 0.5 g of AgCl with the aqueous extracts

Graph 2 shows the amount of silver in grams with the plant extracts in ethanol. It is important to point out that the color of the ethanol extracts is different from the color of the water extracts. From this observation it can be inferred that water does not dissolve all the substances present in the plant, ethane being more selective with respect to the substances it dissolves. In the same way that occurs with the aqueous extracts when the extract in alcoholic solution is added to the complexed silver in the form of diaminesilver(I), the silver was reduced from ionic silver Ag+ to metallic silver Ag0 and, as previously mentioned, it does so in less time. The appearance of the silver is granular and apparently cleaner.



Graphic 2 Recured silver of 0.5 g of AgCl with the extracts in alcoholic solution

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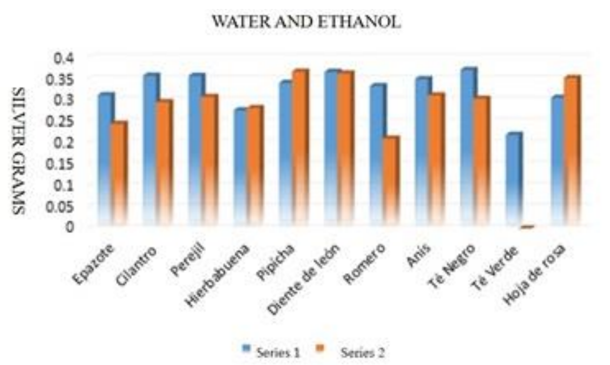
The amount of silver recovered is different, depending on the extract used, and it is evident that its composition with respect to the oxidizing agents it contains is different. It can be seen in the graph that among the best results in terms of the amount of silver recovered are the extracts Pipicha, Dandelion and Rose Leaf.

Graph 3 shows the amount in grams of silver recovered with aqueous extracts and ethanol extracts, it can be seen that in most cases the amount of silver recovered is greater with aqueous extracts, and in some, as in the cases of the extracts in ethanol, of the Pipicha and Hoja de rosa, the amount of silver recovered is greater.



Graphic 3 Amount of silver recovered with the aqueous extracts (series 1) and alcoholic extracts (series 2)

Regarding the yield obtained with each of the extracts, we can see from Table 3, that it also depends on the extract used.

extracts	Percentage of silver recovered aqueous extract.	Percentage of silver recovered alcoholic extracts
Epazote	82.77%	65.02 %
Cilantro	94.92 %	78.44%
Perejil	94.76 %	81.74%
H. Buena	73.42%	74.78 %
Pipícha	90.45 %	97.42 %
D. de León	97.23%	96.19 %
Romero	88.49%	55.80 %
Anís	92.85%	82.38 %
Té Negro	98.45 %	80.52 %
Té Verde	58.19%	0%
Hoja de Rosa	81.18%	93.48%

Table 3 Percentage of silver recovered with the aqueous extracts and ethanol extracts

Better yields were obtained with the aqueous extracts than with the ethanol extracts, most probably this is due to the fact that water is the liquid that dissolves more substances, this property is due to its ability to form hydrogen bonds with other substances, since these dissolve when they interact with polar water molecules.

The best yield of the silver recovered with the aqueous extracts was that of black tea with a yield of 98.45%. In the case of the ethanol extracts, it was that of Pipicha with a yield of 97.42%. In the case of green tea, 59.19% of the silver was recovered with the aqueous extract, with the ethanol extract, recovery was not possible, forming a brown solid. In general, it is observed that good yields are obtained using plant extracts in both water and ethanol.

Conclusions

The use of aqueous plant extracts in the recovery of the silver contained in the radiographic plates is an efficient, low-cost and environmentally friendly option that is part of the so-called green chemistry. This method offers vast possibilities taking into account the great diversity of plants with a wide variety of metabolites that include reducing and stabilizing agents in their composition.

It was possible to obtain clean shiny silver in the form of powder and flakes with an acceptable yield, which depends on each one of the plant extracts and the solvents used.

Finally, I consider that the reduction of silver with plant extracts is a viable alternative to recover it in metallic form, reducing contamination, for which reason it is proposed to continue the studies with other plants, as well as to carry out analytical studies of the recovered silver to determine its purity.

On the other hand, it is also proposed to carry out studies of plant extracts to better understand their chemical composition.

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