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Review article

Efficient gold-cyanide recovery from activated carbon by electrocoagulation technology

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ABSTRACT

Mexico is the 9th largest world gold producer with 1016 tonnes in reserves as of 2024. 41 % losses of gold with an average consumption of 40 g/t, is due to the escape of fine AC particles because they do not have the scope to prevent the loss of particles below 0.40 mm. The recovery of gold in Mexico is becoming more important. Given the low quantities of this metal present in the mineral ore and the difficulty of its extraction due to its encapsulation in its mineralogy, recoveries are low. Adsorption processes with activated carbon (AC) in the mining-metallurgy industry include phases that diminish the particle durability, coupled with its nature as organic matter, which tends to deteriorate and disintegrate in the adsorption systems. In this study, Electrocoagulation (EC) technology is shown as a better option to increase the efficient recovery of gold impregnated in extremely fine AC particles, without the use of chemical reagents in the metallurgical systems, by recovering the fine particles loaded with gold, EC tests were done on a batch pilot scale, on AC particles of 0.106 mm and 0.053 mm, using iron and aluminum electrodes with the best variables determined, they were interpolated at an industrial level. The trend that has been noticed is that smaller particles stick together to generate larger particles that contain aluminum hydroxide species. This means that using EC to recover incredibly fine AC particles is viable. Finally, the EC system with aluminum (Al) and iron (Fe) electrodes shows AC recoveries with gold were greater than 96 % and 88 % respectively. This study is a green technology for the effective recovery of gold, and cyanide in activated carbon thereby in increasing gold productivity in Mexico and the global market.

1. Introduction

Traditional gold extraction involves several steps: crushing, leaching, adsorption, desorption, electrodeposition, and purification. Sodium cyanide is used to leach gold into a soluble complex. Activated carbon (AC) adsorbs this complex, and the gold is then recovered through stripping and electrolysis [1,2]. AC losses occur due to attrition during processes like pulping, leaching, and reactivation. These losses result in both AC and gold losses. To optimize gold recovery, it is crucial to minimize AC losses. Current

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methods often depend on meshes or sieves, leading to 41 % AC losses. This study focuses on the recovery of the losses of fine activated carbon, which contains a significant amount of gold [3–6].

1.1. Attrition losses in adsorption-desorption reactivation (ADR) plants

Attrition losses in ADR plants vary based on factors like coal type, residence time, equipment, treatment rate, and adsorption system. While studies have identified where attrition occurs, quantifying it is challenging due to minimal weight loss per process phase. Carbon losses are distributed as follows: 41 % in final tailings, 5 % in tailing safety screen, and the remainder lost to the tailings during adsorption. The precise mechanisms of gold loss during the adsorption process are debated [3,4]. AC fines pass through sieves and move with the pulp through the adsorption cascade. Sterile pulp is sieved to recover undersized coal, while tailings are pumped to a sludge dam [7,8].

1.2. Removing coal fines in ADR plants

Fine carbon particles in fresh AC can be problematic for adsorption systems. Angular, sharp-edged particles are prone to breakage during handling, while flat or damaged particles can deteriorate during pumping. To address these issues, it is essential to remove fines before returning AC to the gold ADR circuit. A mechanical stirring process can remove fines by separating them from coarser particles precisely it requires stirring 10.0 %–20.0 % solids for 0.5–2 h in water to remove 1.0–3.0 % fines. The mixture is then sieved to further separate fines. These fines can be discarded or sold as a by-product [3].

1.3. Activated carbon particle size

Particle size distribution influences the external surface area of AC, while pore structure development has a lesser impact on specific surface area. Smaller particle sizes can lead to increased adsorption radius and faster adsorption kinetics. However, very fine particles can be challenging to separate from solutions and are prone to Fig. 1 attrition losses. Factors influencing particle size selection include difficulty in separating carbon from solutions, higher attrition losses for fine carbon from solutions, fluidization velocity considerations, and contact ion exchange phenomena. Optimizing particle size can improve AC performance in ADR plants [3,9].

1.4. Electrocoagulation: A Versatile electrochemical Technique

Electrochemistry involves the interconversion of chemical and electrical energy. Electrochemical processes can generate electricity from chemical reactions or use electricity to drive chemical reactions. They have applications in various fields, including energy storage (fuel cells) and metal processing (electrolysis). Electrocoagulation is an electrochemical technique that uses electricity to treat water and wastewater. The current-assisted offers several advantages, including high efficiency, short treatment times, compact equipment, low operating costs, and hydrophobic sludge production [10–14]. In current-assisted electrocoagulation, sacrificial metal anodes (e.g. iron or aluminum) are used to produce ions that coagulate pollutants in water. The generated ions react with pollutants, facilitating coagulation and removal. Electrocoagulation is a promising technique for treating water contaminated with heavy metals, hydrocarbons, suspended solids, and other toxic substances. Its versatility and efficiency make it a valuable tool for environmental

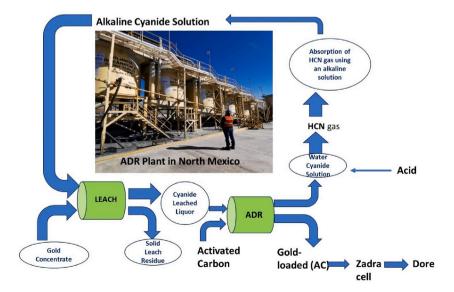


Fig. 1. Schematic diagram and picture of the plant with activated carbon ADR.

remediation [13–16]. This current-assisted electrocoagulation enhanced hydrogen gas generation with distinct and superior operational advantages [17] over the conventional methods for gold recovery from pregnant cyanide solutions, as shown in Table 1. Combining electrocoagulation with activated carbon (AC) adsorption enhanced its potential applications.

The process of EC used in the recovery of gold is very fast with a lower operation cost of less than 15 % than other technologies because it involves the use of green technology where the energy is generated from the sun, wind, and river. This makes it suitable in remote areas like the mountains where electricity is unavailable.

1.4.1. The electrodes

Electrochemical (EC) treatment uses electricity to produce hydroxide ions (OH⁻) at the cathode, which reacts with metal cations to form metal hydroxides. These hydroxides act as coagulants, removing colloidal particles. Iron electrodes produce red-brown iron (III) hydroxide, influenced by pH and ferrous ion availability. Other iron oxides, like magnetite or green rust, can also form. These oxides effectively remove contaminants through complexation and electrostatic attraction [14,19–21].

In acidic medium:

$$Fe_{(s)} \to Fe_{(aq)}^{2+} + 2e^{-}$$

$$2H_2O_{(l)} \rightarrow O_{2(aq)} + 4H_{(aq)}^+ + 4e^-$$
 (2)

$$4 \operatorname{Fe}_{(aa)}^{2+} + 10H_2O_{(1)} + O_{2(g)} \rightarrow 4 \operatorname{Fe}(OH)_{3(s)} + 8H_{(aa)}^{+}$$
(3)

In alkaline medium:

$$Fe_{(s)} \to Fe_{(aa)}^{2+} + 2e^{-}$$

$$Fe_{(aa)}^{2+} + 2OH_{(aa)}^{-} \rightarrow Fe(OH)_{2(aa)}$$
 (5)

As shown in the above equations, the iron anode is used to generate ions in the water medium. The generated iron ions are immediately hydrolyzed to form $Fe(OH)_3$, monomeric ions and polymeric hydroxyl complexes such as $Fe(H_2O)_6^{3+}$, $Fe(H_2O)_4(OH)^{2+}$, $Fe_2(H_2O)_6(OH)_4^{4+}$, $Fe_2(H_2O)_2(OH)_2^{4+}$, and $Fe(H_2O)_5(OH)^{2+}$, depending on the pH of the solution [19].

Regarding the chemical mechanism when aluminum electrodes are used, simplified reactions occur which form a white collide, these can be described as follows:

Anode:
$$2 \text{ Al}_{(s)} \leftrightarrow 2 \text{ Al}^{3+}_{(aq)} + 6 \text{ e}$$
 (6)

$$3H_2O_{(l)} \leftrightarrow \frac{3}{2}O_{2(g)} + 6H_{(aq)}^+ + 6e^-$$
 (7)

Cathode:
$$12H_2O_{(I)} + 12 e \rightarrow 6H_{2(g)} + 12 OH_{-(aq)}$$
 (8)

$$\text{General reaction:} \quad 2 \text{ Al}_{(s)} + 9 \text{H}_2 \text{O}_{(l)} \ \leftrightarrow \ 2 \text{ Al}(\text{OH})_{3(\text{aq})} + \frac{3}{2} \text{ O}_{2(g)} + 6 \text{ H}_{2(g)} \tag{9}$$

Electrochemical treatment destabilizes suspended or dissolved particles, causing aggregation. This forms hydrophobic particles that can be easily removed. Coagulation is faster with mixed particle sizes due to better aggregation between smaller particles. EC reactor design affects efficiency and energy requirements. The choice of reactor depends on effluent characteristics, including contaminants, pH, solids, and gas formation. There are various reactor types as shown in Fig. 2 [22,23].

This study proposes an alternative method for gold recovery using a current-assisted electrocoagulation process with Al-Fe electrodes in ADR plants. SEM analysis showed a flat morphology of the solid product, suggesting reduced AC losses. The EDX analysis indicated high silicon content. XRD analysis revealed magnetite iron particles with gold, potentially useful for battery manufacturing. This method offers a safe, efficient, and economical alternative for gold recovery and cyanide regeneration.

Table 1
Comparative study of the Merril-Crowe, adsorption with activated carbon and electrocoagulation method in the recovery of gold [18].

Merrill-Crowe	Adsorption with activated carbon	Electrocoagulation
It is a well-known technology and the precipitate needs to be smelted which increases the operational cost.	The pregnant solution does not require pretreatment thereby reducing operational cost.	The cost of energy per m^3 of the rich solution is cheaper than the traditional method of treatment.
It is effective in solution with high gold content.	The concentration of the metal does not affect the suitability.	It is effective in both high and low concentrations of gold.
It has 99 % efficiency however, the cake adsorbed copper, lead, and cadmium.	It has a large surface area; however, it is applied only for adsorption of gold.	It does not require chemical reagents therefore no secondary pollution will be expected.
It is suitable also for treating superior solutions obtained from carbon elution.	No clarification is required in the pulp.	It has a low residency time for the recovery of valuable metals (in minutes).

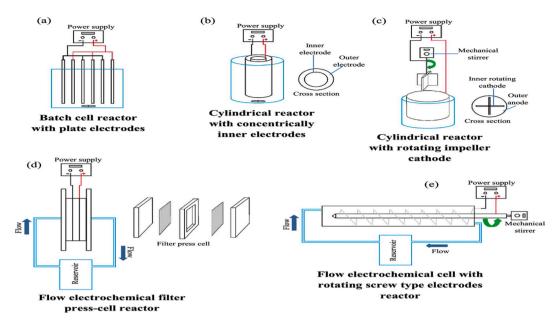


Fig. 2. The various types of electrocoagulation reactors [23]. Elsevier Copyright, 2017.

2. Experimental methodology

2.1. Adsorption experiment on activated carbon

The adsorption processes of batch-type AC were tested on a laboratory scale using an aqueous medium and a 1000 mg/L concentration of NaCN (Meyer, Lot J1121698, Catalog 2350). Finely ground AC (Calgon Carbon DG-11 6×12 Lot HB184AN-4) was made. While meshes (Wstyler Test Sieve ASTM E11 Standard) were used to screen AC and produce various particle sizes, a mortar was employed to reduce its size. The test media was adjusted to 0.5 L of synthetic solution with an Au of 10 mg/L in contact with 0.25, 0.125, 0.05, and 0.01 g of AC in varied particle sizes. The reaction was carried out in a 600-mL Pyrex beaker within 24 h.

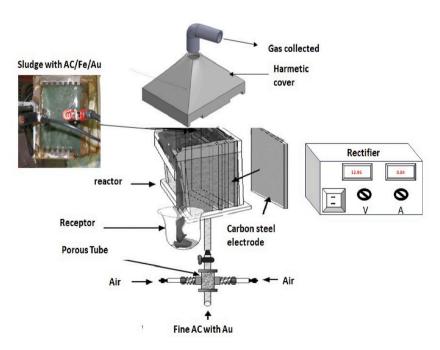


Fig. 3. Electrocoagulation prototype with vertical iron electrodes used in these tests and proposed for recovering fine AC particles loaded with gold. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

2.2. Atomic absorption spectroscopy

The Agilent Technologies Model AA 240 FS was needed to determine the amount of gold in the atomic adsorption monitoring solutions in AC, as well as to determine the amount of gold using analytical instruments and fire assay when the detection limit was the optimum choice for the process. Three repetitions per aliquot, a sample duration of 3 s, and a reading time of 5 s per duplicate in a calibration curve with four points were used in the testing [20]. The 4-point calibration curve was created using automatic micropipettes and flasks to adjust the gold matrix solution to 00.5-1.0-1.5-3.0 ppm of gold, matching the matrix to 1000 ppm of NaCN with a pH 0f 10.5-11.0.

2.3. Fire assay

Using fire assay, gold adsorbed in AC was quantitatively characterized following adsorption processes in cyanide aqueous media. With the use of an electric furnace (Cress Model C1632), nitric acid (Merck brand lot 1.00456.2510) for digestion, and a Mettler Toledo analytical microbalance (model XP–6) for gold measurement, the fire assay procedure was precisely designed for the fusing of AC particles [21].

2.4. Electrocoagulation experiment

The electrocoagulation process was carried out in a device composed of a continuous flow cell, with seven carbon iron electrodes (11.0 cm \times 11.4 cm) connected in parallel. A peristaltic pump, an air pump, and 0.222 A of current and 25 V of voltage feeder were used (Fig. 3). The reactor has an internal volume of 1200 cm³. The equipment where the electrocoagulation tests were carried out contains a sludge receiver, iron plates, an air inlet, and the flow of contaminated water through the lower part of the chamber where air is injected into a porous device with an opening of 20–80 μ m. and this allows us to generate bubbles of said diameter. This bubbling allows for more efficient removal of activated carbon with gold. Before the electrochemical process, the conductivity and pH of the solution were measured. 400 mL per minute of the solution were pushed through the EC reactor. The test was stopped, and the pH and conductivity of the sludge formed were determined after 2–4 L of sample were treated. Filtration using cellulose filter paper was used to separate the liquids and solids. In a vacuum chamber set at room temperature, the sludge that resulted from electrocoagulation was dried. In this work, atomic absorption, X-ray powder diffraction (XRD), and scanning electron microscopy (SEM) were used as chemical analytical techniques to characterize the products that came from the electrocoagulation process.

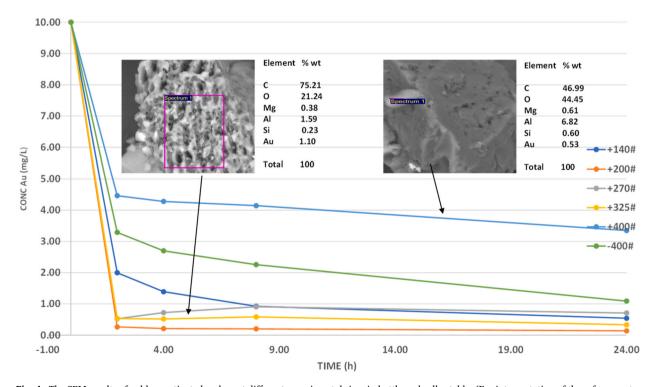


Fig. 4. The SEM results of gold on activated carbon at different experimental sizes in bottle and roller table. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

3. Analysis of the results

3.1. Gold adsorption on activated carbon in experimental sizes

After 24 h of operation on a roller table and with previously identified bottles, samples are collected corresponding to the selected monitoring times. These samples are analyzed with AA analytical instrumentation, at the wavelength for gold determination is 242.8 nm, and simultaneously they are evaluated by inductively coupled plasma optical emission spectroscopy. The results were obtained in each of the fine AC samples that were prepared with mortar, as well as the dimensions of the AC particles by contacting it with the matrix solution containing gold cyanide in ionic form. This further shows the respective gold adsorption percentage of this in the AC at different contact times (see S1 in the supplementary list).

The chart below shows the adsorption capacity of Au increases as the mesh size increases especially at 400 and -400 as shown in Fig. 4. This is followed by the graph which is inserted with the SEM image for a representative sample at the different mesh sizes. On the left side of this figure, we observed that the micrograph of the SEM obtained has more porosity due to the reduction of Silicon compared to the right which has more aluminum and silicon. From S1, we can observe from the SEM picture that for smaller mesh size, the surface area of the AC particle is flat hence the adsorption of Au is less because there is less porosity and from the EDX is determined more Silicon concentration that is a neutral crystal surface of adsorbing, which decreases the efficiency of the adsorption of Au while in larger mesh size, the surface area was observed to be more porous and increases the adsorption efficiency of Au and also it has fewer Silicon concentrations which agree with the investigation of Martinez-Penunuri et al. [24]. This is contrary to the report by literature that the adsorption rate of adsorption enhances higher particle size of the AC due to their increased pore length [3,25]. Mendoza et al. explained the enhancement of the recovery of gold ore because both the particular surface area and the pore volume of the activated carbon particles undergo reduction [26].

3.2. Analysis of electrocoagulation results

EC tests on AC particles of 0.106 mm size and particles of 0.053 mm were conducted to obtain the optimal working parameters. Fig. 5 shows the containers that contain the aqueous medium and AC content during the EC process in a macroscopic way. Microscopically, in the results obtained for both particle sizes using an aluminum electrode, greater coagulation was observed in the particle of 0.053 mm.

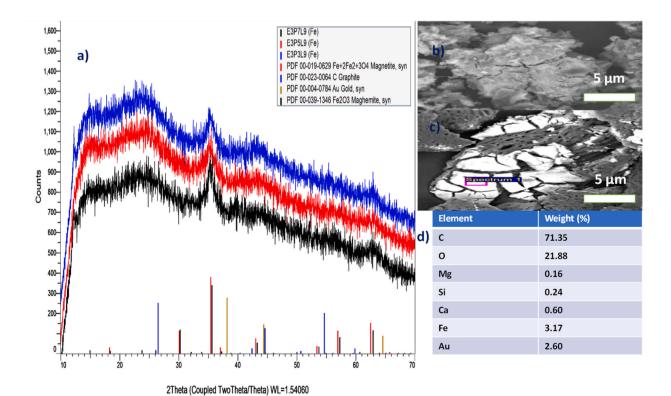


Fig. 5. a) XRD patterns b) SEM c) SEM d) EDAX from the EC process with iron electrodes.

3.2.1. X-ray diffraction with iron electrodes

The XRD technique was used on the products obtained from the EC process for the amorphous X-ray characterization of the clots from the iron electrodes to form the (Fe(OH)n(s)), generating a suspension of a gelatinous solution, allowing the elimination of fine particles of AC filled with gold from the aqueous medium either through 2 main methods 1) complex formation 2) electrostatic affinity. The next stage is coagulation, which is the formation of surface complexes, the contaminant acts as a ligand (L) to bind hydrated iron by chemical reaction [13]:

$$L - H(aq)(OH)OFe(s) \rightarrow L - OFe(s) + H_2O_{(l)}$$
(10)

The species $Fe(OH)^+_{2(aq)}$, $Fe(OH)^{2+}_{(aq)}$ + and $Fe(OH)_3$ present depends on the pH of the solution. In acidic medium, the chemical reactions are:

$$Fe_{(aq)}^{3+} + H_2O_{(l)} \rightarrow Fe(OH)_{(aq)}^{2+} + 2H_{(aq)}^+$$
 (11)

$$Fe_{(aa)}^{3+} + 2H_2O_{(l)} \rightarrow Fe(OH)_{2(aa)}^+ + 2H_{(aa)}^+$$
 (12)

$$Fe_{(aq)}^{3+} + 3H_2O_{(l)} \rightarrow Fe(OH)_{3(aq)} + 3H_{(aq)}^+$$
 (13)

In alkaline medium, $Fe(OH)^-_{6(aq)}$ and, $Fe(OH)^-_{4(aq)}$ ions may also be present.

Fig. 5a shows the characterized compounds of AC particles from iron species using XRD in the EC tests.

Fig. 5band c showed the presence of the AC particles in the aqueous medium during the EC procedure in a macroscopic and microscopic manner in the result obtained for both particle sizes, using an iron electrode while Fig. 5d showed the elemental composition using EDX Techniques. As a result, magnetite Iron particles as the end product of the EC, have a useful application in the fabrication of batteries.

Both EC processes show partial coating on the AC particles, to a greater or lesser extent, white flakes of aluminum or iron respectively are observed on the AC particles, which in principle gives us indications to seek to optimize the applied system. In the following table, we are going to show some of the principal variables used in the recovery of fine AC with Au. Some of these are the conductivity, type of electrode, connected voltage, current, residence time, and distance between the Iron and Aluminum electrodes with the percentage recovery, as seen in Table 2.

From the results, we can conclude that Al electrodes showed better recovery of AC with Au. However, Iron is the less expensive technology. The voltage variation showed that a higher induced voltage increases the current used, which directly increases clot formation and contributes to the AC fine particle recovery. The residence time showed that when the processing time is longer, it improved the AC fine particle recovery, especially using Al electrodes.

The constant distance between the electrodes showed that, with a small separation distance of 14 mm, with a higher time of residence, there is a greater current intensity (for the Al electrode) but lower current intensity (for the Fe electrode) which produces an overall better result of the EC process. While the pH is reduced at constant AC size, the variable of change in the voltage applied, and current used, increased in residence time and better EC recovery for Al of almost 100 % but lesser recovery for Fe electrodes.

It was observed that, in line with our previous report of R. Martinez-Penunuri et al. [27] according to Taguchi's experiment changing the electrodes used from Fe to Al leads to a decrease in the current used, improvement in the residence time, and better EC recovery of AC/Au.

4. Discussion

A considerable aspect of this study is the hydrophobicity of the AC particles, which according to the analysis of the research [28] reinforces that despite the higher number of pores that occur due to the larger surface area exposed to contact. This consequently leads to a decrease in the dimensions of the adsorbent mass and produces an opposite impact on the resistance to the aqueous medium of the AC. The study by Müller and Gubbins [28] showed that the selectivity of water improves with a site density of about 0.5 nm–2 site, in the presence of a carbon-free site by 104 factors. Furthermore, these carbons have oxygenated sites of lower density that lead to significant adsorption of water, but with a disadvantage of available surface for the adsorption of other contaminants.

The proposed chemistry behind the electrocoagulation process is.

1. Activated Carbon and Gold Cyanide Complexation:

Table 2Variables applied in Fe and Al for the AC fine particle recovery.

ID	pН	Conductivity	Electrode	Voltage	Current	Residence time	AC size	Electrode distance	Recovery of AC/Au
		μS/cm	Al/Fe	V	A	Min	mm	mm	%
1	7.56	645	Fe	25	0.253	5	0.053	14	85.53
2	7.40	633	Fe	15	0.128	10	0.053	14	80.57
3	8.10	640	Al	15	0.108	5	0.053	14	87.15
4	7.51	635	Al	25	0.220	10	0.053	14	97.15

Activated carbon, with its extensive network of pores, acts as an adsorbent. In gold mining, it traps gold cyanide complexes (Au $(CN)_2^-$) formed by leaching gold ore with cyanide solution (NaCN) [29]. This adsorption process is primarily physical, involving weak attractive forces between the gold complex and the carbon surface [29].

2. Electrocoagulation Setup:

The EC system consists of electrodes (anode and cathode) submerged in a tank containing the gold-loaded activated carbon particles suspended in the cyanide solution. An electric current is applied, triggering various chemical reactions at the electrode surfaces.

3. Anode Reactions:

At the anode (typically made of inert materials like stainless steel), water decomposes:

$$2H_2O_{(l)} \rightarrow O_{2(aq)} + 4H_{(aq)}^+ + 4e^-$$
 (14)

The generated hydroxyl radicals (OH•) play a crucial role in the process.

4. Metal Ion Desorption and Reduction:

The hydroxyl radicals attack the gold cyanide complexes adsorbed onto the activated carbon, weakening the bonds and facilitating their desorption from the carbon surface. Desorbed gold ions (Au⁺) then migrate toward the cathode.

5. Cathode Reactions:

At the cathode (made of iron or aluminum), reduction reactions occur. Ideally, the reduction reaction should involve water:

$$2H^{+}_{(an)} + 2e^{-} \rightarrow H_{2(g)}$$
 (15)

However, depending on the cathode material and applied voltage, reduction of gold ions can also take place:

$$Au_{(aa)}^+ + e^- \rightarrow Au_{(s)} \tag{16}$$

6. Recovering Gold:

The reduced gold precipitates as elemental gold particles (Au(s)) on the cathode surface or in the solution. These gold particles can be subsequently collected and refined.

7. Cyanide Management:

The Cyanide-metal complex has an impact on the sensitivity when the pH drops to 8. The efficiency of HCN gas release is enhanced by the bubbling of air at the bottom of the EC reactor. As illustrated in Fig. 1, the adsorption of HCN gas takes place when it bubbles in an alkaline (NaOH) solution to produce NaCN which is recycled back.

$$HCN_{(g)} + NaOH_{(g)} \rightarrow NaCN_{(aq)} + H_2O_{(l)}$$
 (17)

The present invention provides an Electrocoagulation (EC) process of fine activated carbon particles with gold content, thus allowing their recovery and subsequent benefit. Electrocoagulation is a technique that consists of inducing an electric current in a solution using an iron/aluminum electrode, which causes chemical reactions that allow the formation of aggregates that can subsequently be removed by some secondary separation method. The process allows fine particles smaller than 0.106 mm to coagulate and recover between 90.0 and 99.0 % of the activated carbon particles.

5. Conclusion

"This study demonstrates that activated carbon (AC) effectively adsorbs gold, particularly in the absence of heavy metals such as copper, cadmium, silver, and iron. The leaching of gold from AC is enhanced when the AC contains lower concentrations of ions like silicon, aluminum, and magnesium. SEM analysis revealed that smaller particles (<20 µm) exhibit reduced gold adsorption due to their flat surface, possibly influenced by the presence of silicon ions. Electrocoagulation (EC) emerges as a highly efficient technique for recovering gold particles from AC. The iron species in magnetite and maghemite contribute to gold adsorption, leading to electroflotation and agglomeration. Using iron and aluminum electrodes, EC achieves excellent gold recovery rates exceeding 90 %. Aluminum electrodes offer advantages such as higher residence time (10 min), lower energy consumption (5.5 W), and lower pH (7.51) of the leach solution. This novel technology presents a promising approach to gold recovery in Mexico. EC also produces magnetite-gold cyanide complexes, XRD, and SEM analysis confirmed the presence of electrostatic forces between gold, and magnetite/maghemite, likely attributed to the magnetic properties of these materials. The EC process stands out as an innovative

technology in gold technology in extractive metallurgy, offering the potential for optimizing operating costs in ADR plants by recovering fine gold particles from AC. Industrial applications include regenerating sodium cyanide and magnetite-gold particles for use in battery manufacturing".

CRediT authorship contribution statement

Jose Refugio Parga Torres: Visualization, Supervision, Project administration, Methodology, Investigation, Funding acquisition, Formal analysis, Data curation, Conceptualization. Nelson Oshogwue Etafo: Writing – review & editing, Writing – original draft. Gregorio Gonzalez Zamarripa: Visualization, Validation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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