Silver recovery from photographic and X-ray films and fixer solution waste using a packed bed reactor

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Effect of nitrogen flow rate (as stirring techniques) as well as effect of temperature and pH on the cementation of silver from silver nitrate solution over copper and aluminum was investigated. Best yields of cementation was achieved at nitrogen flow rate of 2000 ml/min, 60 °C and pH = 4 or 5.5 for cementation over copper and aluminum, respectively .In other trial silver was obtained by leaching of photographic and X-ray films by different NaCN concentrations and (30% W/V) H₂O₂. The cementation over aluminium has been investigated for the removal of silver ions from solution at optimum conditions mentioned above The highest value of Ag recovery is obtained at 30 mg/l NaCN. The procedure was repeated at different volumes of H_2O_2 (30% W/V). The highest value of Ag recovery is obtained at 30 mL of H_2O_2 (30% W/V). Also silver was cemented over aluminum from fixer solution without any treatment except adjusting pH at 5.5. The cementation was determined by measuring residual Ag ion concentrations in the electrolyte by atomic absorption. Al proved to be better than Cu in this process under the present conditions. Values of the energy of activation were 4.94 and 8.94 kJ mole⁻¹ for Cu and Al, respectively, indicating that the cementation reaction is diffusion controlled at its favorite conditions. Recovery of Ag ion was almost complete. As a conclusion, cementation is a usful method for abatement of heavy metal pollution and recovery of silver from some waste material.

يتناول هذا البحث عملية الالتصاق الأسمنتي للفلزات في عملية استرجاع معدن الفضة من مخلفات الصناعة من نترات أو سيانيد الفضة. وقد أجريت التجارب المعملية في هذا البحث وتم الحصول على فلز الفضة بترسيبه من محاليل النترات أو السيانيد باستعمال أحد هذه الفلزات (الألومنيوم أو النحاس) كفلز مضحى والمتغيرات التي يتم در استها في هذا البحث هي درجة الحرارة - درجة الحموضة - درجة التقليب (معدل سريان غاز النيتروجين). وقد وجد أن أفضل النتائج لاسترجاع فلز الفضة من ترات الفضة عند تركيز ١٠٠ ملليجرام/لتر تكون باستعمال فلز الألومنيوم ومعدل سريان غاز النيتروجين ٢٠٠٠ مللي/دقيقة ودرجة حرارة محلول النترات ٦[°]م ودرجة حموضة المحلول ٥،٥ أفضل النتائج التي تم الحصول عليها من دراسة كينتيكية عملية الالتصاق الأسمنتي وكذلك تأثير العوامل السابق ذكرها عليها من المحلول المعد معملياً استخدام لاسترجاع الفضة من المخلفات الصناعية (أفلام التصوير ومعامل التحميض) بعد استخلاص الفضة من الأفلام وذلك باستخدام ٢٠٠ جرام/لتر سيانيد صوديوم و ٤٠ مللتر ماء أكسجين (تركيز ٢٠٠ ألي السابق ذكرها عليها من المحلول المعد معملياً استخدام ٢٠٠ جرام/لتر سيانيد معلية الالتصاق الأسمنتي التصوير ومعامل التحميض) بعد استخلاص الفضة من الأفلام وذلك باستخدام ٣٠ جرام/لتر سيانيد صوديوم و ٤٠ مللتر ماء أكسجين (تركيز ٣٠ %). ومن النتائج تم دراسة سرعة التفاعل وحساب ثابت التفاعل (له معاد المادة اعتمادا على أكسجين (تركيز ٣٠ %). ومن النتائج تم دراسة سرعة التفاعل وحساب ثابت التفاعل (له وحساب انتقال المادة اعتمادا على الخواص الطبيعية للمحلول المستخدم في بحث وكذلك على ثابت التفاعل (له). وتم حساب طاقة التنشيط اعتمادا على ألمواص الخواص الطبيعية للمحلول المستخدم في بحث وكذلك على ثابت التفاعل (له). وتم حساب التفايل (له ولي المادة اعتمادا على الخواص الطبيعية المحلول المستخدم في بحث وكذلك على ثابت التفاعل (له). وتم حساب طاقة التنشيط اعتمادا على ألمواص الخواص الخواص الطبيعية المحلول المستخدم في بحث وكذلك على ثابت التفاعل (له). ولماب طاقة التنشيط اعتمادا على ثابت التفاعل (له) والمستنتج طبقا للعوامل التي درست خلال هذا البحث.

Keywords: Cementation, Metal displacement, Water pollution, Silver recovery, Silver recycling

1. Introduction

Silver is an important industrial metal where it is used in fields such as electrical and electronic technology, photography, jewelry and silver ware...etc.

In U.S., 1999, consumption of silver, including scrap, was estimated to be about 5, 600 ton Photography is the largest end –use category, accounted for about 2,900 ton. The second largest end-use category is batteries, followed by electrical and electronic products that consumed in sterling ware, Jewelry, and silver plating [1]. The liquid waste generated in the processing of photographs and motion pictures is an excellent source of silver. In motion pictures, ferricyanide is usually employed as bleaching agent while in still photograph, ferric Ethylene Diamine Tetra-Acetic. (EDTA) Acid dominates. During processing, a large amount of dissolved silver as silver thio complex in the fixer, bleach – fix solutions), as well as ferricyanide, dichromate in the bleach solutions and the developing

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solutions are available for recovery from the toxic effluent.

In view of the escalating cost of silver and fixer chemicals and the compelling need for pollution abatement, work is currently directed towards the recovery of silver with the possibility of recycling the fixer solution. In addition, decreasing the concentration of silver in the bath improves the efficiency of the fixing operation and improves the subsequent rinsing operation; the reclained metallic silver may be recycled to the photographic process [2].

A number of different methods were employed for achieving recovery of heavy metal such as: chemical precipitation techniques, chemical recovery, electrolytic recovery, metallic replacement or cementation process and ion exchange.

Ritchie and Staunton [3] have studied rates of cementation of silver and copper in acidic nitrate and in basic cyanide media. In the cyanide medium the reaction was not as fast as in acidic solution and this was attributed to the less favorable thermodynamics of the reactions in cyanide solution.

Puvada et al. [4] studied the kinetic of cementation of silver chloro complexes from sodium chloride solutions onto copper using rotating disc electrode systems, Activation energy were found to be 10.8 and 8 kJ /mole for 3 M and 4 M. Na Cl solutions respectively. Many research activities [5-8] were carried out on cementation processes based on the standard potential theory of various metals. toxic metal classification method The proposed by Nibbler and Richardson [6] is adopted, and zinc, iron and aluminum are plated as the sacrificial metals to study the optimum operation conditions for removing soluble silver ions.

Considering, the cementation process, assume there is metal ion M_1 in wastewater, when another metal M_2 is added (M_2 is baser than M_1) the electro chemical reaction will be:

$$A M_{1^{z_{1}^{+}}} + b M_{2(s)} = a M_{1(s)} + b M_{2^{z_{2}^{+}}}.$$
 (1)

Where z is the number of equivalents per mole The aim of this work is to obtain fundamental data on the cementation of silver from aqueous solutions of silver nitrate by cementation over two metals (Cu and Al) as metal tablets. The interplay of experimental conditions and reaction conditions allows some insight into the fundamental kinetics of the reaction. The optimum conditions for silver ion recovery from synthetic silver nitrate are used for silver ion recovery, photographic and X –ray films from which silver is leached using sodium cyanide and H_2O_2 were also investigated.

2. Experimental technique

The base metals used were aluminium of purity.99.5 % and copper of purity 99.99 %. These metals were in the form of smooth tablets of 15-mm diameter and 3 mm height. One side of these tablets was isolated with an inert epoxy resin for the purpose of studying the rate of cementation accurately because this side was in contct with the bottom of the column and and the distribution of the solution is ununiform, the surface area exposed to the reaction media was equal to 3.535 cm². The surface of the metal tablets were prepared by polishing with 400 grade carborundum paper followed by 600 grade paper .They were then washed with distilled water followed by washing with acetone and drying. All chemicals (silver nitrate and nitric acid) were AR grade.

Fig. 1 shows the reaction column, which is a vertical cylindrical plexiglas column, 50 mm in diameter and 300 mm in height. The column was fitted at its bottoms with a perforated plexiglass distributor, which contains 60 holes, each hole 0.5-mm in diameter. The column was surrounded by a jacket, through which a thermostatically controlled water- was used. The temperature of water- bath can be changed from 20-100°C.

2.1. Recovering silver from silver nitrate solution

Before each run 500-ml of silver nitrate solution was placed in the column. The metal tablets were positioned on a perforated plexiglass plate at the end of the cylindrical portion of the column. Nitrogen gas was allowed to pass through the system at the required flow rate. For adjusting temperature

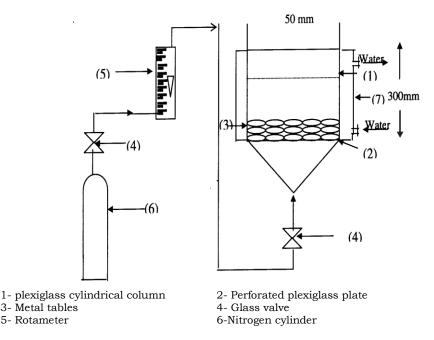


Fig. 1. Experimental apparatus.

a thermostatically controlled temperature water bath was used. Nitrogen flow- rate was measured by a rotameter. Samples of 1 ml from the solution were taken every 10 minutes to detrmine the silver ion concentration by the atomic absorption technique.

The rate of cementation of silver on the different studied metals was calculated from the measured silver ion concentration. Effects of solution temperatures, hydrogen ion concentration and nitrogen gas flow rates upon the rate of silver cementation on the studied metals were investigated.

2.2. Recovering silver from photographic, X-ray films process wastes

The used X-ray films were crushed into pieces of not more than 10 mm in length and were washed with distilled water. The film pieces having an average mass of 20 g. were treated in a glass-vessel containing certain concentration of Na CN solution and certain hydrogen peroxide of (30 % W/V) and the solution is diluted with distlled water until reached 1 liter. The solution was shaken until the black color of the film disappeared and the clear blue of the polyester or triacetate appeared with the gelatin of the film. The reaction times were recorded. The procedure was repeated at different Na CN concentrations (10, 20, 30, 40 g/l) and different volumes of hydrogen peroxide, (10, 20, 30, 40, 50 ml). After that, the cementation process was applied to remove silver ions from solution at optimum conditions stated above.

2.3. Recovering silver from fixer solution

The color bleach-fix solution obtained from photographic processes contains an average concentration of 0.8-g / 1 silver. This solution was treated with nitric acid (69 %) till reaching a pH value of about 5.5. After that the cementation process was carried out to remove silver ions from the treated solution at optimum conditions stated above.

3. Results and discussions

3.1. Cementation over copper metal

3.1.1. Effect of nitrogen gas flow rate

The effect of nitrogen gas flow-rate on the cementation of silver on copper was studied by accomplishing the cementation processes at different nitrogen gas flow-rates ranging from 0-2000 ml/min while keeping the initial silver

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nitrate concentration constant at 100 mg/l, constant pH value of 7 and the solution temperature 25°C.

The obtained results are presented in fig. 2. It is clear from this figure that the concentration of silver ion decreases with increasing nitrogen flow rates when the pH of the solution was equal to seven. This indicates that the cementation under above-mentioned conditions is a diffusion-controlled process.

3.1.2. Effect of temperature

The effect of temperature on the cementation of Ag was studied by running the cementation process at an initial silver nitrate concentration of 100 mg/l while keeping the pH of solution at 7 at different temperatures between (25-60 °C) and at nitrogen flow rate (2000 ml/min).

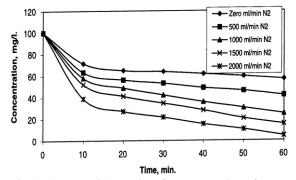
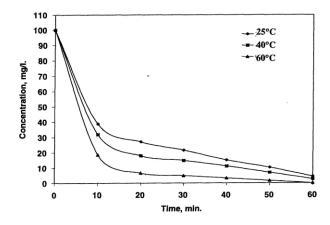
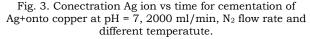


Fig. 2. Conectration vs. time for cementation of Ag+onto copper at 25 °C, pH = 7, different N₂ flow rates.





The results represented in fig. 3 show that the concentration of the residual silver ion decreases with increasing temperature.

3.1.3. Effect of pH

Fig. 4 shows the effect of pH on the cementation of Ag at a constant initial silver nitrate concentrations (100 mg/l), when the solution temperature was kept at 60° C at constant nitrogen flow rate of 2000 ml/min and at different pH values [4-7].

Table 1 and fig.4 reveal that the time needed for complete cementation of silver ions from silver nitrate solution decreases by decreasing the pH value of the solution.

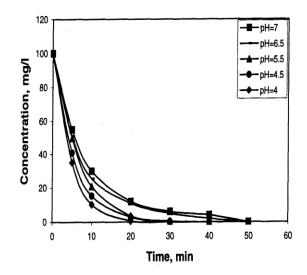


Fig. 4. Conectration oAg ion vs time for cementation of Ag+onto copper at 60 °C, 2000 ml/min, N₂ flowate and different pH.

Table 1 Time for complete cementation of silver ions on copper and aluminium as a function of pH value of the solution

pН	ching zero mg/l silver	
	on copper	on aluminium
4	20 min	15 min
4.5	30 min	15 min
5.5	40 min	15 min
6.5	40 min	20 min
7	60 min	no cementation occurs

3.2. Cementation over aluminum

3.2.1. Effect of nitrogen flow rate

The effect of nitrogen flow rate on the cementation of silver on aluminium is shown in fig. 5, under identical condition to that used for cementation over copper, the same trend was noticed , however the cementation on aluminium is less than that over copper. It is clear from fig. 5 that the concentration of silver ion decreases with increasing nitrogen flow rate . This may indicate that the cementation under the above-mentioned conditions is a diffusion controlled process. Also it is clear that the most effective nitrogen flow rate is 2000 ml/min.

3.2.2. Effect of temperature

The effect of temperature on the rate of cementation is shown in fig. 6. It is clear from that figure that the concentration of silver ion decreases with increasing temperatures. Also it is clear that the most effective temperatures is 60° C.

3.2.3. Effect of pH

Fig. 7 shows the effect of pH on rate of cementation of silver over aluminium , again the time needed for complete cementation is ecreased by decreasing pH . From results in table 1 it is seen that at pH 7 has no effect on cementation process i.e no reaction occurs. Also it is clear that pH 4, 4.5 and 5.5 has the

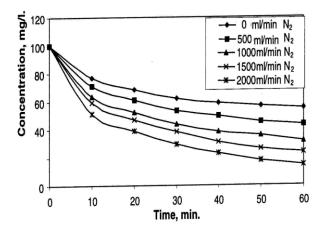


Fig. 5. Concentration v time for Aluminium at 25 °C, pH=6.5, different N₂ flow rates.

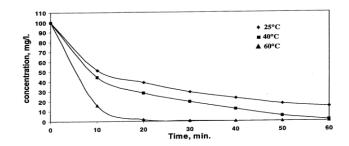


Fig. 6. Concentration of Ag ion vs. time for aluminium at pH=6.5, 2000 ml/min N₂ flow rate and different temperature .

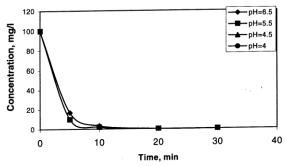


Fig.7. Concentration of Ag ion vs. time for cementation of Ag onto aluminium at 60 °C, 2000 ml/min N_2 flow rate and different pH.

same effect, since time to reach zero mg/l silver is the same (15 min) which is better than pH 6.5 (20 min).

3.3. Rate constant of cementation

Fig. 8 show the relation between $\ln C_o/C$ vs. time for cementation of silver over aluminium. The relation is a straight line which indicates that the reaction is nearly first order. The slope of this line is the rate constant. For the purpose of studying the effect of the different variables (nitrogen flow rate, initial silver ion concentration, pH value and temperature) on the value of rate constant (*k*) the following general equation was proposed:

$$k = A (variable)^{B} \quad (1)$$

Where A and B are constants. Tables 2, 3 show the relation between nitrogen flow rates and the rate constant (k) at different temperatures.

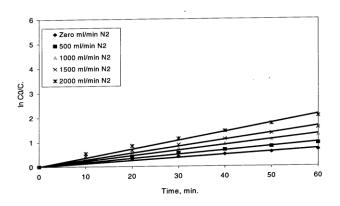


Fig. 8. Ln Co/C vs. time for cementation of Ag onto Aluminium at 25 °C, pH = 6.5, different N₂ flow rates.

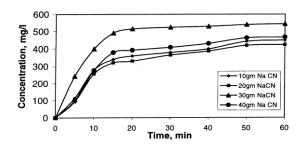


Fig. 9. Effect of sodium cyanide concentration on Ag on concentration removing from x-ray films.

The data shown in table 2 were found to fit the following equations:

cementation of silver on copper at 25 oC, pH =6.5

$$k = 0.06825 (N_2 flow rates)^{0.6319}$$
, (2)

Table 3

Table 2

Relation between the rate constant (k) and different nitrogen flow rates for cementation of Ag+ on copper at pH = 6.5

	k, \min^{-1}		
<i>N</i> ² flow rate, ml/min	25°C	40°C	60°C
0	2.4522	3.2696	3.5051
500	3.5213	4.8574	5.1400
1000	5.3604	7.1680	7.3961
1500	6.4136	8.6296	12.6183
2000	8.8491	11.9784	21.0620
Α	0.06825	0.0102	0.0037
В	0.6319	0.6175	0.7859
Variance	0.9769	0.9715	0.9343

cementation of silver on copper at 40 oC , pH =6.5

$$k = 0.1022 (N_2 \text{ flow rates})^{0.6175}$$
, (3)

cementation of silver on copper at 60 oC, pH =6.5

$$k = 0.0037 (N_2 \text{ flow rates})^{0.7859}$$
 (4)

Previous studies [9-11] on the effect of gas sparging on the rate of heat and mass transfer to the vertical wall of bubble columns have shown that the value of the velocity exponent ranges from (0.15 to 0.5), respectively. The higher exponent obtained in the present study may be ascribed to the geometry of the transfer surface where boundary layer separation and turbulent wake formation is likely to take place. Table 3 shows the relation between the rate constant (k) and different nitrogen flow rates for aluminum at pH = 6.5 at different temperatures. The data shown in table 3 were found to fit the equations:

Relation between the rate constant (k) and different nitrogen flow rates for cementation of Ag+ on aluminum at $\,$ pH = 6.5 $\,$

	k, min ⁻¹			
N ₂ flow rate, ml/min	25 °C	40 °C	60 °C	
0	1.9019	2.2949	2.9235	
500	2.6092	4.9545	10.7832	
1000	3.4740	6.0200	13.4398	
1500	4.3070	8.1733	16.4100	
2000	5.5810	9.9960	27.1644	
Α	0.0944	0.2034	0.2355	
В	0.5288	0.5054	0.5601	
Variance	0.9708	0.9445	0.8400	

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cementation of silver on aluminum at 25 $\,^{\circ}$ C, pH =6.5

$$k = 0.0944 (N_2 \text{ flow rates})^{0.5288,}$$
 (5)

cementation of silver on aluminum at 40° C, pH = 6.5

$$k = 0.2355 (N_2 flow rates)^{0.5054},$$
 (6)

cementation of silver on aluminum at 60° C, pH = 605

$$k = 0.2355 X \, 10^{-3} (N_2 \, flow \, rates)^{0.56014.}$$
 (7)

A comparison of the present data with the results of previous studies [12] at various conditions of temperature and nitrogen flow rates at pH = 6.5 shows that the best metal is aluminum followed by copper followed by lead and iron is the last one.

3.4. Determination of the activation energy of cementation:

The apparent activation energy E for a given cementation reaction can be obtained from the temperature dependence on the rate constant (k) by using Arrhenius equation.

$$K = A \exp^{-E/RT} .$$
(8)

Where (k) is the rate constant obtained from the slope of the relatioship of residual Ag+ against time at different temperatures. A is the frequency factor, E is the activation energy. Ris the gas constant and T is the absolute temperature.

$$ln k = ln A.-E/RT.$$
 (9)

When plotting ln (k) vs. 1/T a straight line is obtained. The slope of which is proportional to E. The activation energy of the process, is an important parameter for determining the rate-controlling step. If boundary layer diffusion of the aqueous species is the rate controlling step [8], E is generally 28 kj/mole. If adsorption of the species on the reaction surface and subsequent chemical reaction takes place and E values are usually 43 kj/mole. In this study E was 5, 9 kj/mole for cementation over copper and aluminum respectively which indicate that the rate controlling step is diffusion.

4. Recovering silver from photographic and X-Ray films

4.1. Effect of cyanide concentertain

Fig. (9) shows the effect of sodium cyanide concentration on Ag ion removing from X-ray films at 30 ml of H₂ 0_2 (30 % V) for one hour. The highest value of silver ion obtained by (30 g / l) Na CN. On increasing Na CN to 40 g/l the removal is decreased this is explained that when Na CN added dropwise to silver nitrate . white precipitate of silver cyanide is formed. When Na CN is added in excess, the precipitate disappears owing to the formation of cyanoargenate ions [13].

4.2. Effect of H_2O_2 concentration

Fig. (10) show the effect of different volumes of hydrogen peroxide (30 % W/V) at a constant concentration of sodium cyanide (30 g/l).

The silver ion concentration increases with the increase of the volume of hydrogen peroxide (10-40 ml) and on increasing the volume of H_2O_2 more than (40 ml) the silver ion concentrations does not increase.

Fig. 11 shows the concentration of silver ion from leaching solutions (X-ray films) at optimum conditions (pH = 5.5, 2000 ml/min nitrogen flow rate, 60° C after cementation on aluminum.

4.3. Recovering silver from fixer solution

Recovering of silver from a fixer solution by cementation could be achievely by only adjusting the pH to 5.5.

Fig. 12 show the concentration of silver ion from fixer solution at optimum conditions (*pH* = 5.5, 2000 ml /min nitrogen's flow rate), after cementation on aluminum.

From fig. 11 and 12 it is seen that full cementation of silver from fixer solution takes a very short time (nearly 15-min) less than

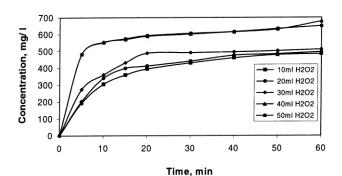


Fig. 10. Effect of different H₂ O₂ volume on (Ag) ion concentration removing from X-ray films.

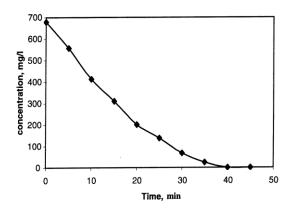


Fig. 11. Cementation of silver ion over Al from leaching solutions of X-ray films at optimum condition (60 °C, pH = 5.5 and 2000 ml/min N₂ flow rate).

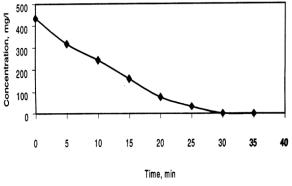


Fig. 12. Cementation of silver ion over Al from ficer solution at optimum condition 60 °C, pH = 5.5 and 2000 N_2 ml/min.

that from leaching solutions). Fig. 13 and 14 show a quantitative flow diagram for silver recovery from the photographic X-ray films and fixer solution respectively by cementation process every half an hour.

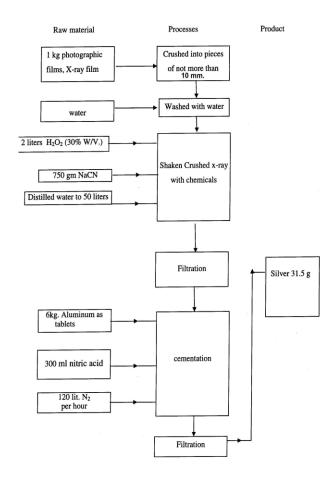


Fig. 13. Quantitative flow sheet for silver recovery from photographic and X-ray films by cementation process every one galf hour.

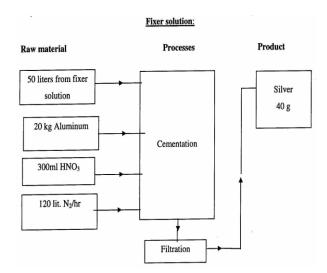


Fig. 14. Quantitative flow sheet for silver recovery from fixer solution by cementation process over Al every one half hour.

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5. Conclusions

This study of the rate of cementation of silver from Ag NO₃ solution on Cu and Al at different conditions of temperature, pH and nitrogen flow rate revealed that best yields of cementation were obtained at 60° C and nitrogen flow rate of 2000 ml/min and pH 4 - 5.5 on using Cu and Al, respectively. Al proved to be better than Cu for cementation of silver.

Values of the energy of activation were 4.94 and 8.94 kj mole⁻¹ for Cu and Al, respectively, indicating that the cementation reaction is diffusion controlled.

Silver was leached from photographic and X-ray films and the resulting solution was cemented over Al at the optimum conditions (3.39 g of Ag ion was obtained from 100 g X-ray films and photographic films on using 30 g/1 NaCN and 40 ml H₂O₂ (30 % W/V).

Also silver from fixer solution was cemented from fixer solution on aluminum with adjustment of pH at 5.5 and a yield of 0.8 g Ag ion per liter of fixer solution.

In conclusion the present method has proved to be effective in complete Ag recovery from photographic waste solutions .

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