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Orthogonal Array Design for the Optimization of Silver Recovery from Waste Photographic Paper

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ABSTRACT

An orthogonal array design (OAD), OA_9, was employed as a chemometric method for the optimization of the silver recovery from waste photographic paper using external electrolysis. In this work, silver was recovered from photographic paper by dissolving the silver compounds with ammonia solution. The external electrolysis method was carried out using a brass electrode as cathode and a carbon electrode as anode. Similarly, external electrolysis was carried out without adding ammonia solution. The effects of four parameters: electrolysis time, ammonia concentration as chelating agent, solution temperature, and electrolysis voltage on the amount of recovered silver were investigated. The effects of these factors on the recovery of metallic silver were quantitatively evaluated by the analysis

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of variance (ANOVA). The results showed that silver can be effectively extracted from various sources, such as waste photographic paper and jewellery scrap, by dissolution in ammonical solutions with the aid of external electrolysis. Finally, the optimum conditions for silver recovery by electrolysis using ammonia as chelating agent were proposed. The results of ANOVA showed that 7.5-hr electrolysis time, 1.3359 mol/L ammonia concentration, and 4.5 V electrolysis voltage are optimum conditions for the silver recovery procedure.

Key Words: Orthogonal array design; Taguchi method; Silver recovery; External electrolysis; Ammonia solution; Photographic paper.

INTRODUCTION

There are several reasons to be interested in the recovery of silver from photographic processing waste. Silver is a valuable natural resource of finite supply, it has monetary value as a recovered commodity, and its release into the environment is strictly regulated. In photographic processing, silver compounds are the basic light-sensitive material in use in most of today's photographic film and paper.[1]

The photographic processing industry has four options in silver recovery: electrolytic plating, metallic replacement, ion exchange, and chemical precipitation.[2] Recirculated electrolytic desilvering has the advantage of reducing the amount of silver lost and many allow chemical conservation in some situations.[3–9] Metallic replacement is simple to use and relatively inexpensive. Refining costs and increased discharge of metal are the main disadvantages.[10–12] Ion exchange, the most frequently used method for treating large volumes of wash water, has operational difficulties, such as biological growth, resin fouling, and the stripping of silver off the resign by thiosulfate. These problems are gradually being solved by process and equipment modifications.[13–18] Chemical precipitation, the oldest silver recovery technique, is not widely used today. It has the advantage of being able to treat large volumes of concentrated solution quickly, producing very low silver concentrations. The liquid–solid separation step and possible contamination of the solution preventing reuse are the main disadvantages of precipitation techniques.[2]

The most widely used silver recovery method for large operations is electrolysis, where silver is recovered from waste photographic solution by electroplating it on a cathode. A direct electrical current is passed between two electrodes suspended in the silver-bearing solution. Silver is deposited on the cathode in the form of nearly pure silver plate. The cathodes are removed periodically and the silver is stripped off for reuse. Most silver is recovered from scraps, such as photographic films, x-ray films, and jewellery.[20] External electrolysis is a form of electrode deposition with an applied external voltage.[21] External electrolysis has been used in the recovery of silver.

Optimization is an important step in developing an extraction and recovery procedure. The theory, methodology, and application of OAD as a chemometric method for the optimization of the analytical procedure have been described in detail elsewhere.[22–30] The aim of this study was to investigate the effect of some experimental parameters on the silver recovery from waste and to find the best experimental conditions for silver recovery. Previous studies showed that electrolysis time, chelating agent concentration, electrolyte solution temperature, and electrolysis voltage have the main effects on the efficiency of silver recovery from wastes.[6–9,20] An experimental design procedure was used to investigate the effects of four parameters on silver recovery performance: electrolysis time, ammonia concentration as chelating agent, solution temperature, and electrolysis voltage.

EXPERIMENTAL

Materials

Expired unprocessed black and white photographic papers were collected from photo-processing industries in Ardakan, Iran. HPLC grade ammonia solution and nitric acid were purchased from Aldrich (Tehran, Iran).

Sampling and Optimization Strategy

Photographic paper (1319 cm^2 of paper) was cut into pieces. The pieces were collected into a beaker. Then 10, 20, and 30 mL of concentrated ammonia solution was poured into the beaker that contained the waste photographic paper. These mixtures were diluted by (approximately) 250 mL of distilled water. The pieces of paper were shaken for 60 min in laboratory temperature (20°C) to dissolve the silver compound. The solution so formed was diluted to 300 mL with distilled water. After dilution, the paper pieces were removed from the electrolyte. The whole mixture was stirred very well to ensure proper mixing of the solution. After this, a brass electrode (3 × 7 cm in size; brass electrode was selected because nonferrous metal alloys, such as brass, in comparison with ferrous metal alloys, such as steel, have high conductivity, low voltage drop, better efficiency during electrolysis, and after electrolysis, desilvering process is easy[31]) connected to a carbon electrode (3.5-mm diameter by 7-cm length) was inserted into the mixture, i.e., the electrolyte was made up of silver–ammonia complex and pure ammonia dissolved in distilled water. A direct current was connected to two electrodes inserted into the electrolyte and the time was taken. To optimize the silver recovery procedure, an experimental design approach was followed. The variables (electrolysis time, ammonia concentration as chelating agent, solution temperature, and electrolysis voltage) were as shown in Table 1. All experiments replicate two times. The cathode and anode sizes, photographic paper area, extraction time, and extraction temperature were fixed during all experiments. After electrolysis, the electrodes were removed from the electrolyte. The brass electrode and precipitations were dried and weighed. The brass electrode was the cathode for the silver deposition and carbon was anode, which was sacrificed at a very slow rate. To desilver the cathode, it was removed carefully from the mounting beaker. The cathode was placed on a clean surface (a glass vial) and was carefully brushed to remove silver plating. The cathode was then recleaned with a wire brush several times before replacement in the plating solution.

External Electrolysis Without Ammonia Solution

1319 cm^2 of unprocessed photographic paper was cut into pieces and collected into a beaker, then, 300 mL of distilled water was added into beaker. After 60 min shaking beaker, the paper pieces were removed from the electrolyte. A direct current was connected to electrodes (brass cathode and carbon anode) inserted into the solution. After 24 hr, the cathode was removed, dried, and weighed before desilvering as described previously.

Determining Silver Content of Photographic Paper

To measure the total silver content of photographic paper, 1319 cm^2 of photographic paper was burned. Its ash was heated with 5 mL concentrate nitric acid to a temperature of 70–80°C with stirring. The amount of silver in solution was later determined by atomic absorption spectroscopy.

RESULTS AND DISCUSSION

Optimization is an important step in developing a recovery method. Two general systematic optimization procedures are simultaneous and sequential methods.[27,32] In sequential methods (e.g., simplex optimization), the response surface is sequentially tracked until an optimum has been located.[33] Disadvantages of sequential methods are slow convergence on complex

Table 1. Assignment of the factors and levels of the experiments by using an OA_9 (3^4) matrix and results of recovery.

Trial number	*T* (°C)	Time (hr)	*C* (mol/L)	Voltage (V)	Set 1 (g Ag)	Set 2 (g Ag)	Average (g Ag)	Percentage recovery
1	25	2.5	0.4453	1.5	0.0085	0.0107	0.0096	7.7
2	25	5.0	0.8906	3.0	0.0633	0.0550	0.05915	47.3
3	25	7.5	1.3359	4.5	0.1178	0.1105	0.11415	91.3
4	40	2.5	0.8906	4.5	0.0718	0.0637	0.06775	54.2
5	40	5.0	1.3359	1.5	0.0688	0.0763	0.07255	58.0
6	40	7.5	0.4453	3.0	0.0375	0.0436	0.04055	32.4
7	55	2.5	1.3359	3.0	0.0638	0.0693	0.06655	53.2
8	55	5.0	0.4453	4.5	0.0389	0.0315	0.0352	28.2
9	55	7.5	0.8906	1.5	0.0630	0.0587	0.06085	48.7

Note: Four parameters were optimized: electrolysis time (*t*), ammonia concentration as chelating agent (*C*), solution temperature (*T*), and electrolysis voltage (*V*).

response surface and difficult in dealing with response surface with high dimensionality. The methods maybe suitable as a means for fine-tuning a separation.[34] On the other hand, the simultaneous optimization methods, such as mixture designs[29,35] and factorial designs[36,37] do not suffer from these problems. The experiments carried out are previously planned. The experimental results are collected, and then, the optimum condition can be located by constructing a response surface or by retention mapping. Mixture designs are useful for experiments where the response depends on the proportions of ingredients in a mixture rather than on their amounts, while factorial designs can also deal with other variables. One obvious disadvantage of the factorial designs is that the number of experimental trials required increases geometrically with increasing number of variables. Thus, the number of experimental trials expands so rapidly that it is not feasible to implement these trials. Fortunately, this can be minimized by the use of fractional factorial experiments, such as Plackelt-Burman schemes or orthogonal array designs (OAD).[23,26,38–44] The latter methods have an advantage over the former in that three-level designs can be used, so as to extract more precise information than that obtained from the two-level designs. In OAD, orthogonal arrays (OAS) are used to assign factors to a series of experiment combinations with results that then can be analyzed by using a common mathematical procedure. The main effects of the factors and preselected interactions are independently extracted. In an OA, different combinations of numerals of any two columns have equal appearance. By arranging experiments orthogonally, different effects can be separated. For two-level design, the OA matrix is mathematically identical to the conventional factorial matrix with the same experimental trials except that the interceptions between the columns and the rows are arranged, e.g., OA_8 (2^7) matrix corresponds to the 2^3 factorial matrixes. As for three-level design, OA_9 (3^4) theoretically corresponds to the 3^2 factorials. Although three-level factorials have been described in detail, no three-level conventional factorial matrix has been constructed, whereas a three-level orthogonal array matrix is available. Experiments can be arranged orthogonally also by using conventional factorial designs. However, in OAD, by using the associated triangular table, the variables are easily assigned and quantitatively estimated.[22,45] This advantage is more significant when more complicated experiments are designed.

Electrolysis is a commonly used technology for recovering silver metal from waste photographic film and solutions.[4] The purpose of this study was to determinate how the various parameters affect electrolytic silver recovery. It has been observed that desilvering photographic film generally produces a light-colored silver plate.[4] The factors included in this study were ammonia concentration (ammonia is added to solution to accelerate the removal of ionic silver from photographic films), solution temperature, electrolysis time, and electrolysis voltage. Factors and levels tested are reported in Table 1. Data obtained by results of the experiments are given in Table 1. Data obtained by performing experiments of the factorial design were analyzed by means of the Taguchi method,[22] to calculate the main effects for each variable. (Table 2 shows the results of these computations.) The analysis of data when there is no interaction between variables, are including: (1) determine the optimum condition, (2) identify the individual influence of each factor, and (3) estimate the performance at the optimum condition. The analysis of variance (ANOVA) for the results of recovery are defined as shown in Table 3.

The mean values of the coefficients of variation for the factors at each level were calculated according to assignment of the experiment (Table 2). For example, to obtain the mean value of recovered silver for the effect of the electrolysis time (*t*) at level 1, the weight of recovered silver of the nine trials in which electrolysis time was set at level 1 (trials 1, 4, and 7) were pooled and divided by the number of values obtained (three values). The mean value of the three levels of a factor reveals how the weight of recovered silver will change when the level of the factor is changed.

In this study, the effect of electrolyte solution temperature on the recovery efficiency of silver from waste was studied. Three different temperatures (25°C, 40°C, and 55°C) were investigated. It was found that temperature of the electrolysis solution is not a significant parameter for the silver recovery procedure.

Table 2. Results of the main effects for each variable on the silver recovery efficiency.

Factor	Level	Result (g Ag)
Temperature (°C)	25	0.0610
Temperature (°C)	40	0.0603
Temperature (°C)	55	0.0542
Electrolysis time (h)	2.5	0.0480
Electrolysis time (h)	5.0	0.0556
Electrolysis time (h)	7.5	0.0718
Ammonia concentration (mol/L)	0.4453	0.0284
Ammonia concentration (mol/L)	0.8906	0.0625
Ammonia concentration (mol/L)	1.3359	0.0844
Voltage (V)	1.5	0.0476
Voltage (V)	3.0	0.0554
Voltage (V)	4.5	0.0723

Note: These results are weight of recovered silver from 1319 cm^2 photographic paper.

Table 3. ANOVA table for silver recovery procedure by using OA_9 (3^4) matrix with recovered silver weight (g) from 1319 cm^2 photographic paper as the response.

					S	*V*	*F*	*P*
Factor	Code	*f*	*S*	*V*	Pooled			
Temperature (°C)	*T*	—	1.659×10^{-4}	8.296×10^{-4}	—	—	—	—
Electrolysis time (h)	*t*	2	1.784×10^{-3}	8.918×10^{-4}	1.754×10^{-3}	8.77×10^{-4}	118.25	12.9
Ammonia concentration	*C*	2	9.550×10^{-3}	4.775×10^{-3}	9.520×10^{-3}	4.76×10^{-4}	633.12	7.0
Voltage	*V*	2	1.920×10^{-3}	9.60×10^{-4}	1.890×10^{-3}	9.45×10^{-4}	127.30	13.9
Error	—	11	0	0	1.659×10^{-4}	1.51×10^{-5}	—	66.2

Note: The critical *F* value was at 90% confidence; pooled error results from pooling insignificant effects.

The values of the extraction efficiency for three levels of ammonia concentration were calculated according to assignment of the extraction experiments (Table 4).

The result of electrolysis without ammonia (as chelating agent) showed that, in the absence of ammonia, the silver was not recovered from unprocessed photographic papers, because silver halide (on the photographic papers) has low solubility in pure water and could not extract to the solution.

The ANOVA results of these experiments indicate that (at 90% confidence level) except temperature, all variables (electrolysis time, ammonia concentration, and voltage) have significant effects on silver recovery procedure. In this work, the interactions between the variables were not considered. The results of ANOVA showed that enhancing electrolysis time from 2.5 to 7.5 hr increased the silver recovery. Also, by increasing ammonia concentration from 0.4453 to 1.3350 mol/L, the recovery of silver was increased; on the other hand, by increasing electrolysis voltage from 1.5 to 4.5 V, the efficiency of silver recovery was enhanced.

Recall that for a bigger and better quality characteristic, the study of the main shows that the optimum conditions proposed according to the results of ANOVA are: 7.5-hr electrolysis time, 1.3359 mol/L ammonia concentration, and electrolysis voltage at 4.5 V.

As a general rule, the optimum performance will be calculated using the following expression:

$$Y_{\mathrm{opt}} = \frac{T}{N} + \left(t_x - \frac{T}{N}\right) + \left(C_y - \frac{T}{N}\right) + \left(V_z - \frac{T}{N}\right)$$

Table 4. The results of experiments for determination efficiency of extraction process.

Ammonia concentration (mol/L)	Extracted silver (g Ag)	Extraction efficiency (%)
0.4453	0.04925	39.4
0.8906	0.0851	68.1
1.3359	0.1195	95.6

Note: The amount of silver in the extracted solutions, was determined by atomic absorption spectroscopy. The extraction time was 60 min and extraction temperature was 20°C. These results are weight of extracted silver from 1319 cm^2 photographic paper.

where Y_{opt} is average of performance + contribution of t_x, C_y and V_z above average performance and where, T is the grand total of all results, N is total number of results, Y_{opt} is performance at optimum condition, and t_x, C_y, and V_z are optimum electrolysis time, ammonia concentration, and electrolysis voltage, respectively.

The procedure for calculation of the confidence interval of the optimum performance is discussed as follows. The CI is given by:

$$\mathrm{CI} = \pm\sqrt{\frac{F_\alpha(f_1, f_2)V_e}{n_e}}$$

where $F_\alpha(f_1, f_2)$ is variance ratio for degree of freedom (DOF) f_1 and f_2 at the level of significance α (in this work, $\alpha = 90\%$) and the confidence level is $(1 - \alpha)$. $f_1 =$ DOF of mean (which always equals 1), $f_2 =$ DOF of the error term, $n_e =$ number of equivalent replications, and given by $n_e =$ number of trials/DOF of mean (always 1) + DOF of all factors resulted used in the estimated.

The results of determining the optimum value and CI for the estimated value of the maximum silver recovery efficiency showed that in optimum conditions, the amount of recovered silver will be 0.112 ± 0.004 g Ag/1319 cm^2 or 0.849 ± 0.001 g Ag/m^2 of photographic paper (between 86.4% and 92.8% of total silver content of photographic paper). On the other hand, the result of atomic absorption spectroscopy showed that the silver content of photographic paper is 0.125 g Ag/1319 cm^2 or 0.948 g Ag/m^2 of photographic paper.

CONCLUSION

The purpose of this study was to provide an understanding of the effect of different parameters, such as solution temperature, electrolysis time, ammonia concentration as chelating agent, and electrolysis voltage on the efficiency of silver recovery from waste.

The use of an OAD for the optimization of silver recovery by electrolysis procedure was demonstrated. The factorial design provided a systematic procedure that could be used to obtain the optimum conditions within the range of experimental conditions investigated. The experiments proved that by using this method, silver can be effectively recovered or recycled from expired unprocessed black and white photographic paper and can be used to prepare such compounds as metallic silver.

By considering the results of present study, this method has compared with previous methods (metallic replacement, ion exchange, and chemical precipitation methods[3–18]) in terms of efficiency, facility, and cost.

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