Annals of Science and Technology - A, Vol 7 (2): 21-28, 2022
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ISSN: 2 544 6320

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Optimizing Silver Extraction Potential from Waste X-Ray Films Using Acid and Alkaline Leaching Agents

Gilbert U. Adie[1], Hakeem O. Shoneye[1] and Paschal O. Iniaghe[2]

[1]Department of Chemistry, University of Ibadan, Nigeria.
[2]Department of Chemistry, Federal University Otuoke, Nigeria.

Received 4th May 2022, Accepted 28th July, 2022
DOI: 10.2478/ast-2022-0006

**Corresponding author*
Paschal O. Iniaghe E-mail po.iniaghe@gmail.com Tel: +2347035191298

Abstract

In this study, the silver extraction potential from waste x-ray films using sodium hydroxide and nitric acid was studied. Sodium hydroxide and nitric acid of different concentrations were used as stripping solutions to determine the optimum leaching concentration. Optimum leaching time and leaching temperature were also investigated by varying the time and temperatures, respectively. The stripped solutions were analysed for silver concentration using Flame Atomic Absorption Spectrophotometer. Results obtained revealed that, for sodium hydroxide stripping, 26.6 mg kg^{-1} of silver ion was obtained at an optimum leaching concentration, time and temperature of 1.5 M, 900 minutes and 30 ^{o}C. For nitric acid stripping, a maximum concentration of 9830.6 mg kg^{-1} was obtained at optimum concentration, time and temperature of 4 M, 1,440 minutes and 30 ^{o}C. The result revealed that the concentration of leaching agent was a significant factor affecting silver recovery, with nitric acid possessing a much higher recovery rate compared to sodium hydroxide.

Keywords: X-ray films, silver extraction, optimisation, nitric acid

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1.0 Introduction

Silver is a precious metal that has the highest reflectivity, photosensitivity, and thermal and electrical conductivities relative to other metals (Satyanarayana and Ramesh, 2020). These properties make silver very useful in photography for making photographic images, and in electrical industries (Yacizi et al., 2010). The photographic sector alone reportedly allocates 45% of produced silver to radiographic applications (Satyanarayana and Ramesh, 2020). Silver also has great economic and industrial applications, and is often used in making jewelleries, silverware and dental fillings (Parpalliwar et al., 2015). Silver is a regulated element (US EPA, 2015), and free silver ion is reported to be extremely toxic to aquatic life (Masebinu and Muzenda, 2014). Even though silver is considered to be essentially non-toxic to humans due to its inert nature, lethal concentrations of silver salts can cause organ failure and skin pigmentation (Drake and Hazelwood, 2005). It is also listed as a secondary drinking water contaminant for potable water (Fisher et al., 2015). As a result of the aforementioned, waste photographic materials containing large amounts of silver can be regarded as being hazardous, especially when such wastes are inappropriately disposed of (White and Rycroft, 1982, Lollobrigida de Souza et al 2012; BWS, 2017). With an increasing demand for silver, approximately 2 to 2.5 % per annum (Bas et al., 2012), and a corresponding depletion in natural resource for silver (Paraguassu-Chaves et al., 2019) as well as increasing cost of silver in recent years (Aktas et al., 2010), recovery of silver from such secondary wastes is imperative (Shankar et al., 2010).

X-ray films contain approximately, 1.5 – 2 % (w/w) of metallic silver in gelatin-coated film made from polyester layer (Al-Abdalall and Al-Khalid, 2016; Lakshmi and Hemalatha, 2016). This percentage composition of silver can provide a significant resource potential for secondary silver (Satyanarayana and Ramesh, 2020). Similarly, most photographic and x-ray film wastes are reported to contain silver thiosulphate, with silver at a concentration of 5 parts per million; and the silver is usually found in the fix solution, rinse water, water baths and cleaner developer tanks (KODAK, 1999). Fully developed x-ray film is reported to contain 14 – 17 g of silver per kg of x-ray film (Abdel-Aal and Farghaly, 2007). Therefore, recovery of silver can provide significant economic and environmental benefits.

Several techniques have been applied for recovering silver from x-ray films; including hydrometallurgy (leaching), pyrometallurgy (incineration), electro-deposition, metal replacement, chemical precipitation, and use of enzymes. Incineration of x-ray films normally produces yields that are less than 95 %, but also generates foul smell in the vicinity of the apparatus (Garcia, 1986; Radha and Arun, 2010; Shankar et al., 2010). Enzymatic hydrolysis, though environmentally safe, is usually time consuming (Aktas et al., 2017) and as such, may not be applicable on a commercial scale. Hydrometallurgical processes have proven highly effective in recovering silver, with recovery efficiency up to 99 % (Onlin et al., 1993; Canda et al., 2017). Leaching agents previously used for recovering silver include cyanide (Garcia, 1986, Yazici et al. 2017), NaOH (Nakiboglu et al., 2003; Erku et al., 2017), nitric acid (Abdel-Aal and Faghaly, 2007; Khunprasert, 2008; Arslan et al. 2011), oxalic acid (Marinkovic et al., 2006), thiosulphate (Yacizi et al., 2010, Ahlatci et al. 2022), etc.

The use of cyanide however causes environmental problems due to its inherent toxicity; organic acids could be time consuming as they have lesser leaching strengths compared to inorganic acids, hence lesser silver stripping capability, while the challenge of separating the leached pulp and its subsequent treatment is also very common. There have also been researches on the use of agricultural waste materials for extracting silver from waste photographic films, including plantain ash solution (Orubite-Okorosaye and Jack, 2012), red mangrove bark ash (Okorosaye-Orubite and Don-Lawson, 2016), coconut (*Cocos nucifera L*) shell aqueous extract (Gbarakoro and Akinfolarin, 2018), etc. Some of the quick wins of these latter methods are cheap raw materials, and appreciable and comparable quantities of recovered silver. However, some of the drawbacks of these methods could be longer recovery time, challenge of isolating recovered silver from the solution matrix and the release of carbon dioxide to the atmosphere during burning of agricultural wastes to obtain ash. Carbon dioxide is noted as a major contributor to global warming.

In Nigeria, recovery of silver from x-ray films is still done in an informal manner and at backyards. Interaction with those engaged in this business revealed that they had no background on the chemistry of the process and extracting solutions. They use NaOH and different types of acids not well known to them in the extraction with perceived low silver recovery and deleterious impact on both humans and the environment. It is thought that attempting to optimise this process could have economic and environmental benefits. Therefore, in this study, the ease of recovering silver from waste x-ray films by hydrometallurgy using NaOH and nitric acid that are commonly used by the informal dealers was explored, with a focus on optimisation of parameters (including concentration of leaching agents, leaching time and leaching temperature) that will improve recovery of silver and avoid wastage.

2.0 Experimental

2.1 Collection and preparation of x-ray films

The waste x-ray films, measuring 42.8 cm x 35.2 cm in size, were collected from the Radiology Department, University College Hospital, University of Ibadan, Nigeria. The films were washed with deionised water, wiped with cotton impregnated with ethanol, dried in an oven at 50 °C for 30 minutes, and shredded into 5 cm x 5 cm bits. The cut portions were segregated into pieces containing black, grey and light blue areas, respectively. However, only the portion containing 100 % black area was used for process optimisation, since this is the unexposed area of the film containing silver ions (Adani et al., 2005)

2.2 Optimisation process using sodium hydroxide

Exactly 10.0 g of the shredded x-ray films was weighed and soaked in 80 mL of various concentrations of NaOH (0.1 M, 0.5 M, 1.0 M, 1.5 M and 2.0 M, respectively) in different beakers for 1,440 minutes (24 hours) at 30 °C ambient room temperature. The concentration that had the highest stripping efficiency was taken as the optimum, which was subsequently used for obtaining the optimum leaching time and temperature, respectively (Figure 1). For the optimum leaching time, exactly 10.0 g of the shredded films was weighed and

soaked in series of beakers containing 80 mL of the optimum solution of NaOH solutions at 30 °C ambient room temperature. The stripping process was monitored for 300, 600, 900 and 1,200 minutes. For evaluating the optimum leaching temperature, exactly 10.0 g of the shredded films was boiled in 80 mL of optimum NaOH solution and time, and stirred at 10 minutes interval at temperatures of 44 °C, 64 °C, 84 °C and 92 °C, respectively. Completion of the stripping process was indicated by the disappearance of the black colour of the gelatin-silver halide layer and appearance of blue colour of the polyester film.

The stripped films were removed from the stripping solution and rinsed with deionised water into the stripping solution. The sludge obtained was evaporated to dryness, followed by addition of 10 mL of 2.0 M HNO_3 to the residue with gentle heating until dissolution was complete. The resulting solution obtained was filtered into 100 mL standard flask, made up to mark with deionised water, and stored in a dark cupboard before analysis.

2.3 Optimisation process using nitric acid

For nitric acid (HNO_3) stripping, similar optimization processes for concentration, leaching time and temperature as described above for NaOH stripping, was adopted. However, the concentrations of HNO_3 solution used were: 0.1 M, 0.5 M, 1.0 M, 2.0 M and 4.0 M. Tables 1 – 3 summarizes the entire optimization process.

2.4 Instrumental analysis

The silver concentration in the extracts was analysed using the Buck scientific 211 Flame Atomic Absorption Spectrophotometer (FAAS). The appropriate lamp and resonance wavelength of the metal were used.

2.5 Quality assurance/quality control measures

All glass ware and plastics used were washed with detergent, rinsed with water, soaked with 5 % nitric acid for 24 hours and rinsed with deionised water and were dried at room temperature before use. All reagents were analytical grade reagents. Reagent blanks were analysed, and all analyses were carried out in triplicates.

3.0 Results and discussion

3.1 Effects of varying concentrations of leaching agents on silver recovery from x-ray film

Figure 2a shows the effect of varying concentrations of NaOH on the recovery of silver from x-ray films. The amount of extracted silver was observed to increase from 1.02 mg kg^{-1} when NaOH concentration was 0.1 M to 25.1 mg kg^{-1} at the optimum concentration of 1.5 M. This showed that the higher the concentration of NaOH, the more the gelatin-silver halide emulsion stripped from the polyester film into the stripping solution. This may be attributed to the fact that more hydroxyl ions (OH^-) were available to react with the silver ions (Ag^+). However, a decrease in the amount of recovered silver was observed at NaOH concentration greater than 1.5 M. This could be as a result of a threshold point in ionic balance when formation of complex silver-hydroxyl or hydrated silver-oxide complexes like $Ag_2O.xH_2O$ became more prominent (Birk, 2020). Canda et al. (2017) and Fatimah et al. (2020) also reported that silver was maximally stripped at a concentration of 1.5 M. The amount of recovered silver is also reported to be influenced by variation in pH of NaOH (Galarpe and Leopoldo, 2017). It is also thought that the H^+ ions at this point were barely enough to neutralise the OH- thereby having lesser strength to possibly dissolve completely the Ag^+ precipitates or complexes that could be formed.

Generally, the poor recovery of silver from silver-halide gelatin on x-ray polyester film using NaOH as extracting agent could be attributed to the presence of halides like Cl^- and Br^- used to make the film that have tendency of re-precipitating silver as silver halides after dissolution with acid according to these reactions (Vknn, 2012):

$$2\,Ag(aq)+ + 2\,OH\text{-}(aq) \rightleftharpoons Ag_2(OH)_2(aq) \quad \text{----------(1)}$$

$$Ag_2(OH)_2(aq)+ 2\,Cl{-}(aq) + 2\,H+(aq) \rightleftharpoons 2\,AgCl(s) + 2\,H_2O(aq) \text{ ----(2)}$$

In Figure 2b, the amount of recovered silver is shown to increase with increasing concentrations of HNO_3, with the highest recovery concentration fixed at 4 M. The maximum concentration of 4 M was selected because it was suspected that higher concentrations would be more turbulent and dangerous to work with and could cause accidental explosion. Again, higher concentrations were judged to be uneconomical as the cost of recovery cum amount of silver obtained seemed quite high. Moreover, at this acid concentration, at least 66% of the silver was already recovered (9,830 mg kg^{-1} out of the possible15,000 mg kg^{-1} reported to be present in new x-ray films). It should be noted that the concentration of silver in waste x-ray should be lower (not up to 15,000 mg kg^{-1}) than that in the new x-ray estimated to be 1.5 – 2 % (w/w), as some silver would have been stripped or converted during the process of developing the image (Harper and Siege, 2003). It is also thought that at this concentration, most of the available silver metal on the polyester film would have been stripped. The low silver recovery obtained using 0.1 M HNO_3 could be because the gelatin-silver halide layer remained unleached from the polyester base film. This could mean that the acid concentration was too low to adequately initiate the leaching process. Comparatively, leaching with nitric acid possessed a higher extraction efficiency for silver, compared to NaOH. A maximum concentration of 9,830 mg kg^{-1} was obtained when shredded photographic films were leached with nitric acid, compared to 25.1 mg kg^{-1} obtained for NaOH. Previous studies using different extracting solutions indicating comparable silver extraction have been reported (Ajiwe and Anyadiegwu, 2000 (HNO_3/cyanide solution); Bas et al., 2012 (H_2O_2 solution); Balela et al. 2020 (Oxalic acid solution). The high extraction efficiency of nitric acid certainly was due to general solubility of metals in solutions having low/ acidic pH values.

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Figure 1: Optimisation process for stripping silver from shredded X-ray films

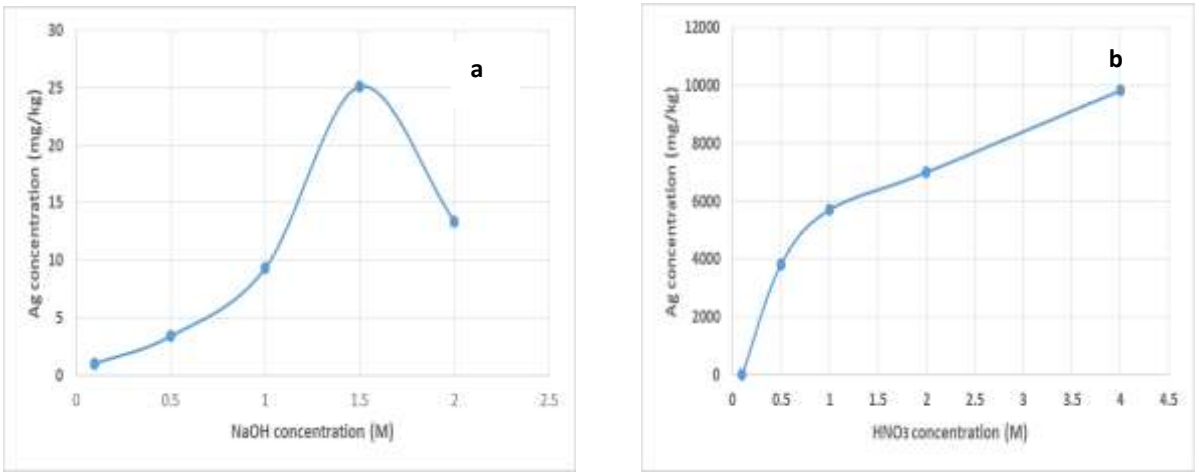


Figure 2: Effect of concentration of leaching agent on silver recovery (a) NaOH solution and (b) HNO_3 solution

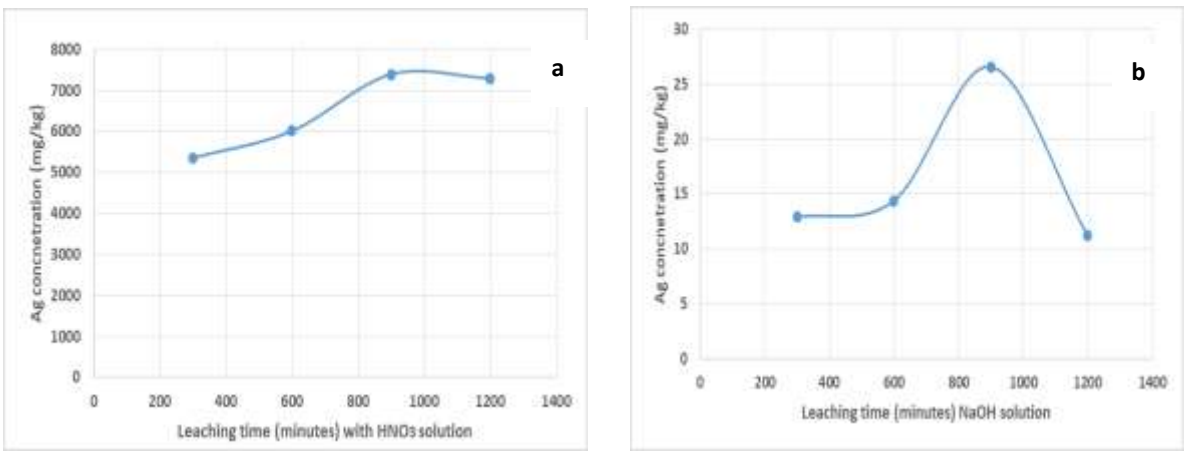


Figure 3: Effect of leaching time on silver recovery using (a) HNO_3 solution and (b) NaOH solution

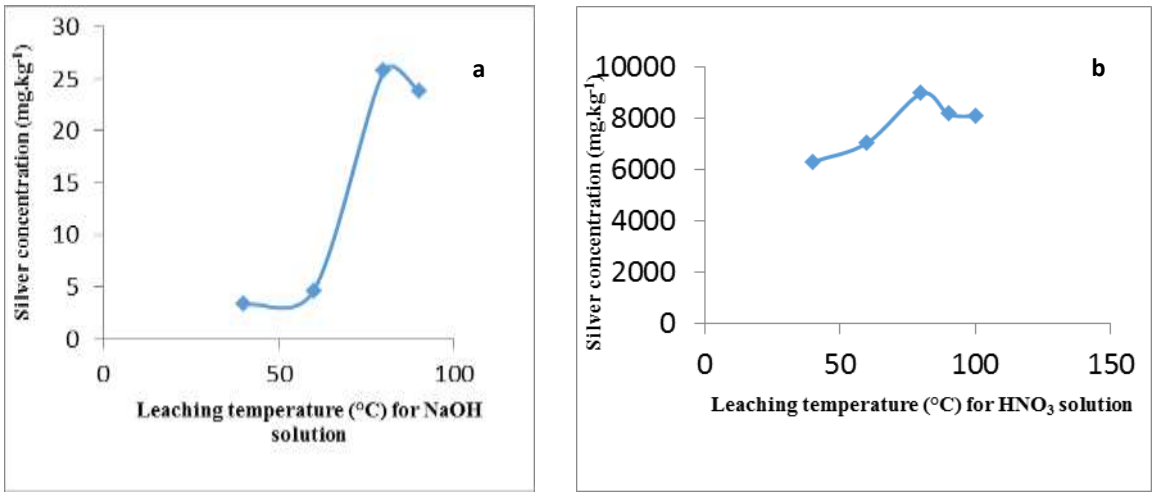


Figure 4: Effects of leaching temperature on silver recovery in (a) NaOH solution and (b) HNO_3 solution

Table 1: Optimized parameters for obtaining optimum leaching concentration

Leaching agents	Varying leaching times (minutes)	Optimum concentration (Molar)
NaOH	300, 600, 900 and1,200	1.5
HNO_3	300, 600, 900 and 1,200	4.0

Table 2: Optimized parameters for obtaining optimum leaching time from the optimum concentration

Leaching agents	Varying concentrations of leaching agents (Molar)	Leaching time (minutes)
NaOH	0.1, 0.5, 1.0, 1.5 and 2.0	1,440
HNO_3	0.1, 0.5, 1.0, 2.0 and 4.0	1,440

Table 3: Optimized parameters for obtaining optimum leaching temperature from the optimum concentration and time

Leaching agents	Varying leaching temperatures (°C)	Optimum concentration (Molar)	Optimum time (minutes)
NaOH	44, 64, 84 and 92	1.5	900
HNO_3	44, 64, 84 and 92	4.0	900

3.2 Effects of varying leaching time on silver recovery from x- ray film

Figure 3a presents an assessment of leaching time to deduce the effect of time on the stripping of silver ions with HNO_3 solution. The concentration of silver in the extract was observed to increase gradually to over 7296 mg kg^{-1} at 900 minutes, after which a decline was observed. The decline in silver concentration could be due to completion of silver stripping from the film. In the same vain, as indicated in Figure 3b, silver concentration increased steadily to about 26.6 mg kg^{-1} at around 900 minutes also, after which a sharp decline was observed. This could be adduced to the same reason as acid stripping explained above. The low silver recovery using NaOH as earlier mentioned could be because of re-precipitation of silver halides after acid dissolution, thereby making some of the silver ions already precipitated unenviable for detection.

3.4 Effects of varying temperatures of leaching agents on silver recovery from x-ray film

As shown in Figure 4a and b, 80 ^{0}C appeared to be the highest leaching temperature for silver stripping using both NaOH and HNO_3 solutions. Temperature is reported to be one of the most important factor that increases silver stripping from the x-ray films (Anuradha and Valli, 2011). Higher temperatures are noted to increase exposure of surface area to the stripping solution. However, very high temperatures are not recommended for silver stripping from x-ray films because at elevated temperatures greater than 100 ^{o}C, there is tendency of disintegration of the stacked gluten employed in the in the manufacture of x-ray films. Nakiboğlu et al. (2003) reported that, the most appropriate stripping temperature range for gelatin-silver layer from used photographic film is 70 – 80 ^{o}C., while Aktas et al. (2010) reported 70 ^{o}C as optimal temperature for dissolution of silver from radiographic films. These corroborates our findings, which is in line with the results obtained in this study.

4.0 Conclusion

This study assessed the possibility of optimising the commonly used leaching solutions (NaOH and HNO_3) by backyard recyclers in Nigeria to strip metallic silver from waste x-ray films. Three conditions were optimised, namely: leaching concentration, leaching time and leaching temperature. It was revealed that optimum leaching concentrations of 1.5 M and 4 M for NaOH and HNO_3, respectively, showed good promise to strip a good percentage of silver from the film. Furthermore leaching time reaching 900 minutes for both solutions indicated optimum leaching of silver, and the best leaching temperature was about 80 ^{o}C. The quantity of silver metal stripped from our findings was comparable to previous studies using different leaching solutions from our study. In a whole, optimising hydrometallurgical methods involving stripping of silver metal from x-ray films using low cost and readily available NaOH and HNO_3 was achieved by noting the optimum concentration, leaching time and leaching temperatures with concomitant benefits on the environment, health and economy. Further studies should consider sequential stripping to evaluate the strengths of leaching solutions after more than one stripping, longer leaching time and more acid volume.

Conflict of Interest

The authors declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

Individual Author's contribution:

Conception: G.U. Adie
Design: G.U. Adie, H.O. Shoneye
Execution: H.O. Shoneye
Interpretation: G.U. Adie, P.O. Iniaghe
Writing the paper: G.U. Adie, P.O. Iniaghe

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