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Recovery of silver from waste x-ray photographic films by chemical method

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Abstract

Silver is a precious metal that has the highest reflectivity, as well as the highest electrical and thermal conductivity's when compared to any other metal. X ray technique is greatly helpful for diagnosis of patient problems and hence widely used till date. Extraction of silver from the ore is expensive and harmful to the environment. In the present study, silver is recovered from waste X-ray photographic films by chemical method.

Keywords: X-ray, Photographic film, XRD, Silver recovery

Introduction

Silver is a rare precious, naturally occurring in ores like argentite, chlorargyrite, and pyrargyrite [1]. Silver has more application that is renowned. One of its most significant application is in the photographic Industry. With the highest thermal conductivity and highest optical reflectivity, it is found in abundance in the waste X-ray photographic films. The waste Xray/photographic films containing black metallic silver spread in gelatin are very good source for silver recovery compared to other types of film [3]. Naturally occurring silver is composed of two stable isotopes, 107Ag and 109Ag, of which the former is more abundant [4, 5]. Researchers claim that silver-containing wastes like used X-ray photographic film are toxic and consider them as hazardous wastes [6]. In large doses, silver and compounds containing it lead to argyria, which results in a blue-grayish pigmentation of the skin, eyes, and mucous membranes [7]. Most households dispose these wastes into land and water bodies. The recoverable silver in the x-ray films are mostly present in the "fix" and the "bleach-fix" solutions. Most photographic and X-ray wastes contain silver thiosulfate with silver at a concentration of 5 parts per million (ppm). They are found in the fixer solution, rinse water, water baths and cleaning developer tank solutions [8]. Several technologies exist to recover silver from X-ray photographic film such as burning the film, electrolysis, metal replacement, chemical precipitation and bacterial, enzymatic methods. Except chemical methods, the other methods are expensive and time consuming to recover the silver [9]. The use of chemicals like sodium cyanide, nitric acid and organic compounds cause environmental problems, while the decomposition by microorganism is slow [10]. Ion exchange processes, reduce the silver concentration in photographic effluent to levels in the range of 0.5 to 2 mg/L. Reverse osmosis (RO) and distillation recovery process are amongst the others used [11].

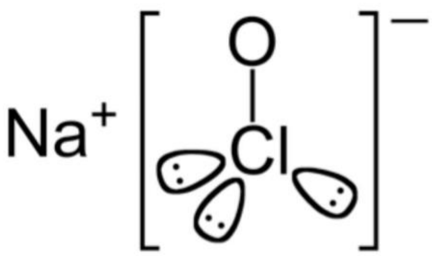
Due to the rapid depletion of natural silver resources and high silver demand, the secondary silver resources have recently become ever-important [12]. Owing to its high photosensitivity and electrical conductivity, silver is extensively used in photography and electrical industry. Approximately 40-50% of the silver production is consumed for radiography and photographic films and papers. Most of the X-ray (radiography) films (>94-98%) are used in medical applications [13]. X-ray and photographic films may contain appreciable amounts of silver in the form of silver halide e.g. AgBr. X-ray films typically contain about 4.85 g/m2 Ag [14]. During the photo processing silver halide is reduced to metallic silver on exposure to light (1) and the remaining silver halide is dissolved by thiosulphate into the waste solution (2) during the developing and fixing procedures [15]

It was claimed that during photo processing ~60% of the silver remains on the film while ~40% transfers to the solution [13]. Waste X-ray films may contain 0.7-2% silver by weight in the emulsion on the polyester film base.

Materials And Methods

1. NaOCl (Sodium hypochlorite)

Sodium hypochlorite appears as colorless or slightly yellow watery liquid with an odor of household bleach. Sodium Hypochlorite is a chlorine compound often used as a disinfectant or a bleaching agent. Sodium hypochlorite in 0.5% w/v solution is called Dakin's solution, and is used as an antiseptic to clean infected topical wounds.



Sodium Hypochlorite Chemical Structure

Fig. 1 Sodium hypochlorite solution

2. X Ray Photographic films (procured from diagnostic centers).
3. Nitric acid
4. Sodium hydroxide
5. Ethanol
6. Distilled water
7. Measuring cylinder
8. Glassware

   a. Beaker
   b. Conical flask
   c. Stirrer
   d. Pipette
   e. China dish
9. Filter paper
10. Burner
11. Instrument
   a. X-ray diffraction (XRD)

Methodology

1. **Recovery of silver**
2. **Characterization of precipitated silver dust**

Recovery of silver by Sodium Hypochlorite solution

2% sodium hypochlorite solution was prepared. The X-ray photographic sheets were washed with distilled water and cleaned with ethanol. These films were cut into pieces and air-dried. Washed x- rays films were dipped in the Sodium hypochlorite solution. The solution is kept without disturbing to allow particles of silver to settle at the bottom of beaker. After few hours the solution is filtered and the silver dust is collected on filter paper which is sun dried. It is further characterized using XRD.

Fig. 2 X-ray films dipped in sodium hypochlorite solution

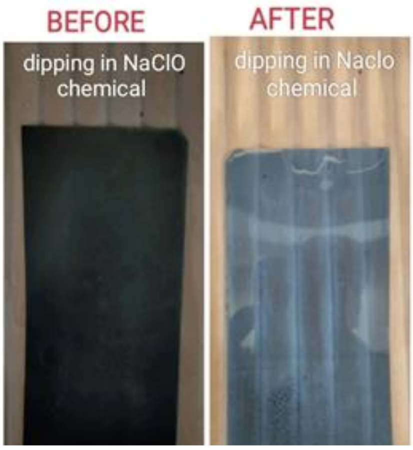


Fig. 3 X-ray films before and after dipping in solution

Fig. 4 Precipitated Silver dust

Recovery of silver by Nitric acid

The X-ray photographic sheets washed with water and cleaned with ethanol. Films air-dried and cut into pieces. Washed x- rays films dipped in 1M Nitric acid solution for approx 4 hours at 80° Celsius. Silver from photographic films is dissolved in the Nitric acid solution, which was then precipitated from sodium hydroxide solution. Precipitate of Silver hydroxide was filtered and ignited to get a piece of Silver metal.

Fig. 5 Film dipped in 1M Nitric acid solution

Fig. 6 Piece of Silver metal obtained after ignition

Characterization of silver dust by XRD:

Precipitated Silver dust was analyzed by X-ray Powder Diffraction (XRD) Instrument of Bruker's make.

X- Ray Diffraction (XRD)

X-ray diffraction analysis (XRD) is a technique used in materials science to determine the crystallographic structure of a material. XRD works by irradiating a material with incident X-rays and then measuring the intensities and scattering angles of the X-rays that leave the material.

A primary use of XRD analysis is the identification of materials based on their diffraction pattern as well as phase identification. XRD also yields information on how the actual structure deviates from the ideal one, owing to internal stresses and defects.

Fundamental Principles of X-ray Powder Diffraction (XRD)

Max von Laue, in 1912, discovered that crystalline substances act as three-dimensional diffraction gratings for X-ray wavelengths similar to the spacing of planes in a crystal lattice. X-ray diffraction is now a common technique for the study of crystal structures and atomic spacing.

X-ray diffraction is based on constructive interference of monochromatic X-rays and a crystalline sample. A cathode ray tube, filtered to produce monochromatic radiation, collimated to concentrate, and directed toward the sample, generates these X-rays. The interaction of the incident rays with the sample produces constructive interference (and a diffracted ray) when conditions satisfy Bragg's Law ($n\lambda=2d \sin \theta$). This law relates the wavelength of electromagnetic radiation to the diffraction angle and the lattice spacing in a crystalline sample. These diffracted X-rays are then detected, processed and counted. By scanning the sample through a range of 2θ angles, all possible diffraction directions of the lattice is attained due to the random orientation of the powdered material. Conversion of the diffraction peaks to d-spacings allows identification of the mineral because each mineral has a set of unique d-spacings. Typically, this is achieved by comparison of d-spacings with standard reference patterns.

All diffraction methods are based on generation of X-rays in an X-ray tube. These X-rays are directed at the sample, and the diffracted rays are collected. A key component of all diffraction is the angle between the incident and diffracted rays. Powder and single crystal diffraction vary in instrumentation beyond.

X-ray diffractometers consist of three basic elements: an X-ray tube, a sample holder, and an X-ray detector.

Fig. 7 Bruker's X-ray Diffraction

Sample Preparation

Grind the sample to a fine powder of less than ~10 μm (or 200-mesh) in size is preferred. Place into a sample holder. Care must be taken to create a flat upper surface and to achieve a random distribution of lattice orientations unless creating an oriented smear.

Fig. 8 Sample Preparation for powder XRD

The geometry of an X-ray diffractometer is such that the sample rotates in the path of the collimated X-ray beam at an angle θ while the X-ray detector is mounted on an arm to collect the diffracted X-rays and rotates at an angle of 2θ. For typical powder patterns, data is collected at 2θ from ~5° to 70°, angles that are pre-set in the X-ray scan.

Characterization of Silver piece

The Silver piece obtained by ignition of the Silver hydroxide precipitate was sent to National computerized gold testing laboratory, which shows the silver obtained is 98.47% pure.

Results and Discussions:

Weight of X-ray films used= 445 g

Weight of silver dust = 0.768 g

XRD of obtained Silver dust was carried out on Brukers's instrument. The peaks obtained is similar to XRD of pure silver.

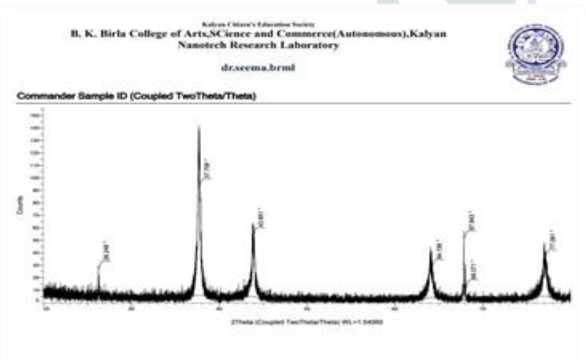


Fig. 9 XRD of precipitated Silver dust

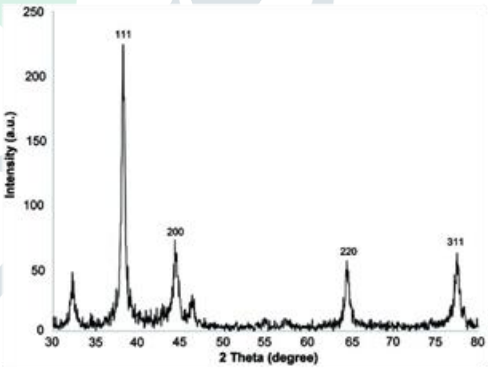
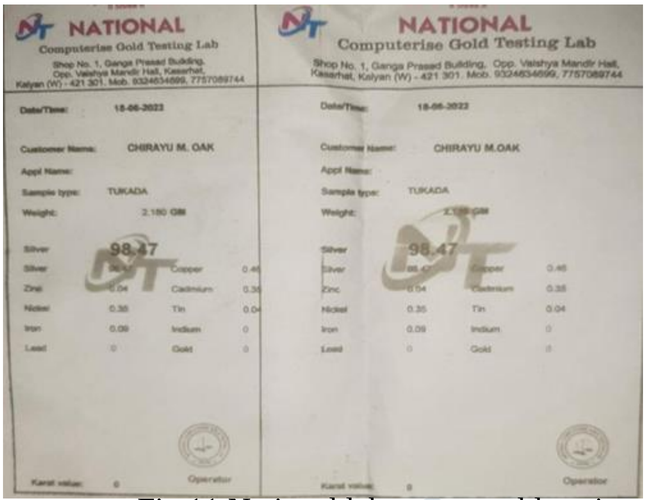


Fig. 10 XRD of pure silver

INDEX	ANGLE	D VALUE	REL. INTENSITY	FWHM
1	26.246°	3.39271 A°	26.9%	0.100
2	37.706°	2.38380 A°	100.0%	0.100
3	43.851°	2.06294 A°	56.0%	0.100
4	64.156°	1.45045 A°	27.5%	0.100
5	67.843°	1.38033 A°	56.0%	0.100
6	68.071°	1.37626 A°	10.7%	0.100
7	77.061°	1.23657 A°	35.0%	0.100

Above table of peak values which shows 100% relative intensity at 37.706° confirms the presence of silver.



NATIONAL
Computerise Gold Testing Lab
Shop No. 1, Ganga Prasad Building, Opp. Vaishya Mandir Hall, Kasarhat, Kalyan (W) - 421 301.

Date/Time:	18-06-2022
Customer Name:	CHIRAYU M. OAK
Appl Name:	
Sample type:	TUKADA
Weight:	2.180 GM
Silver	98.47

Silver	98.47	Copper	0.46
Zinc	0.04	Cadmium	0.35
Nickel	0.35	Tin	0.04
Iron	0.09	Indium	0
Lead	0	Gold	0

Karat value: 0 Operator

Fig.11 National laboratory gold testing report

The above test report confirms the presence of 98.4% pure Silver.

Conclusion

The above work shows that Sodium hypochlorite solution can be readily used for the leaching of silver from waste X-ray films with almost complete recovery of silver.

The silver recovered by using waste x-ray sheet and Sodium hypochlorite yields silver dust. The analysis of silver dust was done using XRD. The peak value obtained from the graph confirm the presence of silver in the sample.

Silver was also recovered from photographic films using nitric acid treatment. Piece of silver obtained was almost 99% pure.

Since Silver being a precious metal it can be converted into Nano silver for various applications of Nano silver.

References:

1. ATSDR., "Toxicological profile for silver", Agency for Toxic Substances and Disease Registry U.S. Public Health Service, December 1990.
2. Riedel, S., and Kaupp, M., "The highest oxidation states of the transition metal elements", Coordination Chemistry Reviews 253 (5–6), 2009, 606-624.
3. Masser, S.H., "Method of recovering silver from waste photographic film and paper", American patent, 4759914, 1988,CI 423-439.
4. IUPAC., "Atomic weights of the elements", International Union for Pure and Applied Chemistry, Technical report, 2007.
5. Bjelkhagen, H.I., "Silver-halide recording materials: for holography and their processing". Springer, 1995, 156-166.
6. White, I.R., and Rycroft, R.J.G., "Contact dermatitis from silver fulminate-fulminate itch". Contact Dermatitis, 8, 1982, 159- 163.
7. Samson, O.M., and Edison, M., "Review of Silver Recovery Techniques from Radiographic Effluent and X-ray Film Waste", Proceedings of the World Congress on Engineering and Computer Science, 2, 2014, San Francisco, USA.
8. Kodak., "Recovering silver from photographic processing solutions", Publication no. J-215 Eastman Kodak Company, 1999.
9. Rawat, J.P., and Iqbal, S., Kamoonpuri, M., "Recovery of silver from laboratory wastes", Journal of Chemical Education, 1986, 63 (6), 537.

10. He, J., and Kappler, A., "Recovery of precious metals from waste streams", Microbial Biotechnology, 10, 1194-1198, 2017.
11. Goshima, T., Hori, K., Yamamoto, A., "Recovery of silver from radiographic fixer", Oral Surgery, Oral Medicine, Oral Pathology, 77(6),1994, 684-688
12. Zhouxiang, H., Jianying, W., Ma, Z., Jifan, 2008. A method to recover silver from waste x-ray films with spent fixing bath, Hydrometallurgy, February, Vol. 92, No. 1, pp 141-151.
13. Khunprasert, P., Grisdanurak, N., Thaveesri, J., Danutra, V., Puttitavorn, W., 2008. Radiographic film waste management in Thailand and cleaner technology for silver leaching, Journal of Cleaner Production, January, Vol 16, No 1, pp. 28-36.
14. Kodak, 1998. Sources of silver in photographic processing facilities, Eastman Kodak Company, Publication No: J-210, 8 pp.
15. Kodak, 1999. Recovering silver from photographic processing solutions, Eastman Kodak Company, Publication No: J-215, 11 pp.