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Recovery of Silver from X Ray Waste From Electro Deposition

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Abstract: A **novel, simple, fast, cheap and pollution-free method was developed for recovering the silver from waste X-ray photographic films. The waste X-ray/ photographic films contain 1.5 - 2 % (w/w) black metallic silver which is recovered and reused. Around 18-20% of the world's silver needs are supplied by recycling photographic waste. Extraction of silver from the ore is expensive and harmful to the environment and low efficiency. X-ray films represent an important worldwide consumer as research on recovery of silver from exposed radiographic films must be oriented to achieve a maximum recovery and a high purity silver, with methods through the by-products will be less polluting for the environment. Global demand for silver remains steadily to about 1,000 million ounces of which around 600 million ounces are used in industrial applications. Extraction of silver from the ore is expensive and harmful to the environment and low efficiency. X-ray films represent an important worldwide consumer as research on recovery of silver from exposed radiographic films must be oriented to achieve a maximum recovery and a high purity silver, with methods through the by-products will be less polluting for the environment.**

I. INTRODUCTION

Silver is a lustrous, soft white metal bearing symbol Ag with a atomic number 47.Silver is a very important metal as it has useful properties as well as economic importance. Silver is one of the precious metals. It is often combines with other elements, including oxygen sulfur chlorine and nitrogen. Pure silver has lustrous medium grey color. Silver is a precious metal widely used in the photographic, electrical, electronics, chemical and jewelry industries. Silver is often extracted by mining or can be found at hazardous waste sites mixed with soil or water. It is also used for silver ware, electronic equipment, dental fillings besides also in mirrors, catalysis and photographic films. The photography sector alone allocates 45% of the silver to radiographic applications, which is discarded completely after its use. The electrochemical and chemical precipitation methods are majorly used to extract the silver contents from X-ray and photographic films. Silver is a rare, precious, naturally occurring metal, often found deposited as a mineral ore in association with other elements. Fifty percent of silver produced is used in photographic and imaging materials. For decades the medical sector and film industry has been using the high quality photographic films and X-ray films where both of them depend mainly on silver. Nothing has been found to equal to silver as light sensitive material where it is capable of forming photographic images because of the unique qualities of silver. The cost of silver has been increasing constantly in recent years due to increase of silver rates in international market compounded with the shortage of silver. Fortunately, much of the silver used in photographic and medical fields can be recovered and reused. The photographic waste which compromises of fixer and bleach solutions which consists a high concentrations of silver, nitrates and sulphates in the dissolved form. Besides these the photographic waste, silver can also be recovered from the hospitals such as the waste streams generated from the radiology department, scrap films... etc. Silver is unique in its ability to react with light to produce images in applications such as photography and radiography (X-rays). Major emissions of silver are from the manufacture and disposal of certain photographic and X-ray films. X-rays films used in medical applications are made of a plastic sheet (polyester film) coated with a thin coating of gelatin (protein) impregnated with silver grain. It has been reported that 25% of the world's silver needs are supplied by recycling, out of which 75% is obtained from photographic waste. The amount of silver varies between 1.5% and 2.0% by weight. With an increasing demand for silver in the world, recent attention is focused on X-ray/photographic films as one of the secondary sources of silver owing to the considerable amount of silver present in them. Pure silver has lustrous medium grey color. Silver is often extracted by mining or can be found at hazardous waste sites mixed with soil and or water. Silver has great industrial and economic applications and often used for making jewellery. It is also used for silverware, electronic equipment and dental fillings. Today, silver metal is also used in mirrors and in catalysis of chemical reactions. Its compounds are used in preparation of x-ray films and photographic films, they are called as silver halides. Dilute silver called as silver nitrate solutions are used as disinfectants and micro biocides. Silver is a rare earth metal, seen as the symbol Ag, after the Latin word Argentum, which occurs naturally in the environment only as a soft metal, coloured "silver", there are no artificial sources for it. Silver comes from a combination of mining silver, gold, copper, lead, and zinc ore. It also appears as a white powder (silver nitrate and silver chloride), or dark grey to black compounds (sulphide, silver oxide and silver). These compounds are hazardous to the environment. Photography is a sector representing an important source of silver released into the environment. The mines that produce silver and other metals are another source. Silver is a very inactive metal. It does not reacts with oxygen in air under normal conditions. However, the sulphur compounds react slowly with air. The product of this reaction is silver sulphide (Ag_2S), a black compound. Silver do not react with water, acids or many other compounds and it burns only as a silver powder. Silver has been used due to its functionality and beauty. Silver is a material used both in many industrial sectors and also as a precious metal. As an industrial metal it has thousands of uses due to its remarkable qualities. Silver has the highest electrical and thermal conductivity compared to any other element. Silver bullion stocks dropped steadily, while investments in silver are growing, which is reflected in its relatively constant price. Production will be increased to meet demand. In the last 10 years the price of silver was doubled. The ores that provide silver are limited, so silver mining cannot sustain the consumption. Moreover, the extraction of silver from scrap by products and consumer goods is important to put back into markets that rely on silver. Radiographs, still and motion industry wastes are excellent sources for silver recovery. Approximately 2 billion radiographs per year are taken around the world which include chest X-rays, mammograms and CT scan. 94-98% of the X-rays taken are in the medical fields producing photographic chemicals and scrap films as waste. Radiographic films used in the medical field are polyester sheets coated on both sides by radioactive materials which are light sensitive. Abdel-Aal and Farghaly reported that 1kg of developed X-ray film contains 14-17g of silver. Most photographic chemicals for developing X-ray films are made from silver salt. Due to the high photosensitivity of silver halide, about 8.3% of silver is used in photography. The effluent of X-ray films processing facilities can reach a silver content of 1-12g/l. The method for silver recovery in broad terms are either hydrometallurgical or pyro-metallurgical processes. The hydrometallurgical processes are through electrolysis, metallic replacement, chemical precipitation, adsorption and liquid membrane. Biosorption is also a possible technique for silver recovery which is a physico-chemical & metabolic process based on absorption, adsorption, ion exchange and precipitation mechanisms. Pyro-metallurgical process which is the traditional method for silver recovery from X-ray films include incineration, smelting, drossing, sintering and melting at high temperature. This technique has been reported to be less efficient with a recovery <95% and temperature >950°C at which X-ray film polymers are destroyed.

Properties of Silver

Physical properties:

Atomic number	47
Atomic mass	107.87 g.mol $^{-1}$
Electro negativity	1.9
Density	10.5 g.cm $^{-3}$ at 20°C
Melting point	962 °C
Boiling point	2212 °C
Vanderwaals radius	0.144 nm
Ionic radius	0.126 nm
Isotopes	11
Electronic shell	[Kr] $4d^{10}$ $5s^1$
Energy of first ionization	758 kJ.mol $^{-1}$
Energy of second ionization	2061 kJ.mol $^{-1}$

Chemical properties:

Silver is a very inactive metal. It does not react with oxygen in the air under normal circumstances. It does react slowly with sulfur compounds in the air, however. The product of this reaction is silver sulfide (Ag_2S), a black compound. The tarnish that develops over time on silverware and other silver-plated objects is silver sulfide. Silver does not react readily with water, acids, or many other compounds. It does not burn except as silver powder. Two naturally occurring isotopes of silver exist: silver-107 and silver-109. Isotopes are two or more forms of an element. About 16 radioactive isotopes of silver are known also.

History

Silver has been known since the ancient times. Slag dumps in Asia Minor and on islands in the Aegean Sea indicate that man had learned to separate silver from lead as early as 3000 B.C. Silver occurs native and in ores (argentite (Ag_2S) and horn silver ($AgCl_2$)); lead, lead-zinc, copper, gold and copper-nickel ores are principal sources. Silver has been known since ancient times. Silver is mentioned in the Book of Genesis, and slag heaps found in Asia Minorand on the islands of the Aegean Sea indicate that silver was being separated from lead as early as the 4th millennium BC. The silver mines at Laurium were very rich and helped provide a currency for the economy of Ancient Athens. It involved mining the ore in underground galleries, washing the ores and smelting it to produce the metal. Elaborate washing tables still exist at the site using rain water held in cisterns and collected during the winter months. Extraction of silver from lead ore was widespread in Roman Britain as a result of Roman mining very soon after the conquest of the first century AD. From the mid-15th century silver began to be extracted from copper ores in massive quantities using the liquation process creating a boost to the mining and metallurgy industries of Central Europe.

Scope

Recovering silver from waste solutions such as those produced by the popular processing of medical and industrial X-ray films, photographic films and pictures, as well as graphic films, papers and plates is a concept that has been practiced for over 100 years. However the economic viability of the process has radically changed in recent years. All film i.e. X-rays, Negatives used for Newspaper or your camera spool all has Silver emulsified. When the film is processed through fixer the Silver washes off the film and the image is developed on the film. There is now silver in the fixer and some left on the film. quantity of silver in the film & fixer. X-ray film has an average of 7.5g per kilogram while negatives have 9g / kg and camera film 2g / kg. Fixer varies according to how the film is processed but average at 3.0 grams per liter.

Types of Silver

1. Fine silver which is greater than 99.9% pure
2. Coin silver with 90% silver and 10%copper
3. Silver powder that comes in several forms such as amorphous powder, dendritic crystals, atomized powder.

Market

Silver is both Industrial metal as well as a precious metal. Silver is critical in photographic printing and paper industry. Over past ten years silver bullion inventories have fallen dramatically while silver investments are steadily increasing as a result the price of silver is raising steadily over the years. Production of silver from wastes must be increased in order to keep up with the increased demand.

Silver in the environment

Silver levels in soil are not usually high except in mineral-rich areas when they can sometimes be as much as 44ppm. Plants can absorb silver and measured levels come in the range 0.03-0.5ppm.

Metallic silver occurs naturally as crystals, but more generally as a compact mass; there are small deposits in Norway, Germany and Mexico. The chief silver ores are acanthite mined in Mexico, Bolivia and Honduras, and stephanite, mined in Canada. However silver is mostly obtained as a byproduct in the refining of other metals. World production of newly mined silver is around 17.000 tonnes per year, of which only about a quarter comes from silver mines. The rest is a byproduct of refining other metals.

Health effects of silver

Soluble silver salts, specially $AgNO_3$, are lethal in concentrations of up to 2g (0.070 oz). Silver compounds can be slowly absorbed by body tissues, with the consequent bluish or blackish skin pigmentation (argiria).

Eye contact: may cause severe corneal injury if liquid comes in contact with the eyes. Skin contact: may cause skin irritation. Repeated and prolonged contact with skin may cause allergic dermatitis. Inhalation hazards: exposure to high concentrations of vapors may cause dizziness, breathing difficulty, headaches or respiratory irritation. Extremely high concentrations may cause drowsiness, staggering, confusion, unconsciousness, coma or death.

Liquid or vapor may be irritating to skin, eyes, throat, or lungs. Intentional misuse by deliberately concentrating and inhaling the contents of this product can be harmful or fatal.

Ingestion hazards: moderately toxic. May cause stomach discomfort, nausea, vomiting, diarrhea, and narcosis. Aspiration of material into lungs if swallowed or if vomiting occurs can cause chemical pneumonitis which can be fatal.

Target organ: chronic overexposure to a component or components in this material has been found to cause the following effects in laboratory animals:

- Kidney damage
- Eye damage
- Lung damage
- Liver damage
- Anemia
- Brain damage

Chronic overexposure to a component or components in this product has been suggested as a cause of the following effects in humans:

- Cardiac abnormalities
- Reports have associated repeated and prolonged overexposure to solvents with permanent brain and nervous system damage.
- Repeated breathing or skin contact of methyl ethyl ketone may increase the potency of neurotoxins such as hexane if exposures occur at the same time.

The health impact of pure solid silver on human beings is low due to its almost completely biological inert nature, hence if ingested in moderate quantities; it would pass through the human body without being absorbed into tissues. However, soluble silver salts are lethal in concentration which can cause organ failure and skin pigmentation. The Occupational Safety and Health Administration (OSHA) of the United State permissible exposure limit for silver is 0.01mg of Ag/m3 of air over an eight-hour work shift.

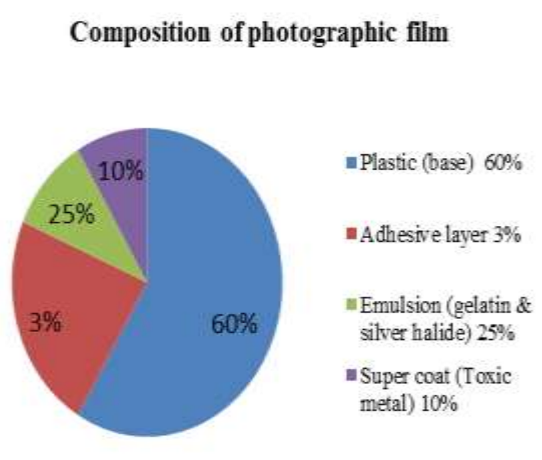
The environmental impact of silver and its salts varies depending on the concentration and chemical changes associated with the disposal body. Silver is present in all surface water but in extremely low concentrations. Silver nitrate is less toxic in seawater than in fresh water.

Ionic silver is extremely toxic to aquatic life and aqueous concentration of 1-5µg/liter can kill sensitive species of aquatic organisms. Sensitive aquatic plants were reported to grow poorly at 3.3 – 8.2 µg Ag/liter during exposure for 5 days and consequently died when concentration is >130 µg Ag/liter. At nominal water concentration of 0.5-4.5µg Ag/liter, accumulation in most exposed species retards their growth. The accumulation of silver by terrestrial plants from soil is low hence waste with concentrated silver from agricultural processes is low.

Ratte reported that the germination stage of plants is the most sensitive stage at which exposure to silver needs to be controlled. A concentration greater than 0.75mg Ag/liter affect lettuce adversely and 9.8mg Ag/liter would kill corn (Zea mays). A spray containing 100-1000mg Ag/liter will kill tomato (Lycopersicon esculentum) and bean plants. However, the yield of some crops has been reported to be higher on soil amended by silver laden waste.

II. COMPOSITION OF PHOTOGRAPHIC FILM

The major recording medium used in radiology is X-ray film - although the situation is changing with the introduction of new technologies in recent years. The film can be exposed by the direct action of X-rays, but more commonly the X-ray energy is converted into light by intensifying screens and this light is used to expose the film, as described above. The basic structure of the film is outlined in Figure below.



various Treatments methods to recover silver from photographic film and fixer solution. The silver to be recovered may be present in different forms as insoluble silver halide, a soluble silver thiosulphate complex, a silver ion, or elemental silver, depending upon the type of process and the stage at which it is recovered (Messerschmidt, 1988). A number of techniques are available to remove silver from silver rich photographic processing solution. Of these, three are used in virtually all practical methods of silver recovery. (KODAK, 1999).

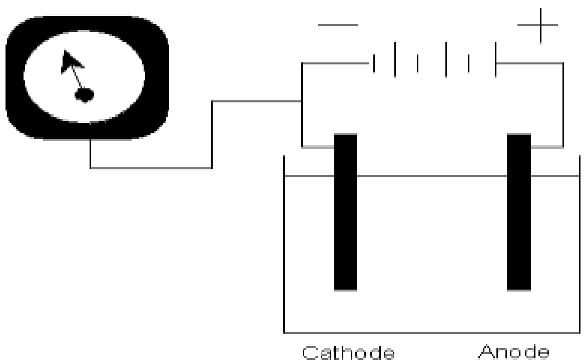
III. SILVER RECOVERY

During the development process, the film, or photographic paper, is immersed in a series of chemical solutions. The chemical solutions react with the silver and create the images on the film. During the fixing process, unprocessed silver is removed from the film, halting the developing of the image, and fixing it permanently to the film. The silver that is removed from the film bonds with the fixing chemicals and needs to be removed before the chemicals can be disposed. Places such as photo development labs, hospitals with X-ray machines, photography schools and photographers with darkrooms generate large quantities of used fixer for silver recovery. Extraction of silver from the ore is expensive and harmful to the environment and low efficiency. X-ray films represent an important worldwide consumer as research on recovery of silver from exposed radiographic films must be oriented to achieve a maximum recovery and a high purity silver, with methods through the by-products will be less polluting for the environment.

Methods for extraction of Silver from X-ray Waste

1. Chemical Precipitation
2. Electrolysis
3. Metal replacement Cartridges
4. Biological method by Enzyme production
5. Extraction of Silver by Glucose
6. Electrolysis Process:
7. When electric current is passed between two electrodes immersed in the silver-bearing x-ray solution, the silver is electronically deposited upon the cathode.
8. Electrolytic silver recovery is, in many cases, the most cost effective and efficient way to remove silver from silver-rich photographic processing solutions.
9. Recovery of metals from aqueous solution of their salts by electrolysis can be realized by two methods. The first method consists of the electrolysis of solutions obtained after leaching of the corresponding metal from ores or concentrated with the use of insoluble anodes.
10. The second method consists of the electrolytic refining of the metal In the process of electrolysis, or commonly known as electrolytic silver recovery, a direct current is passed through a silver-rich solution between a positive electrode (the anode) and a negative electrode (the cathode).
11. During this electrolytic process, an electron is transferred from the cathode to the positively charged silver, converting it to its metallic state, which adheres to the cathode.. In a simultaneous reaction at the anode, an electron is taken from some species in solution. In most silver-rich solution, this electron usually comes from sulfite

Schematic Diagram for Typical Electrolytic Silver Recovery Process:



Chemicals and materials required for electrolysis process:

1. Caustic Soda.
2. Silver nitrate.
3. Stainless steel electrode.
4. 12 volt Battery.
5. Glass container.
6. Voltage regulator.

Types of photographic films used for extraction of silver from x ray:

1. Medical X ray films
2. MRI Film
3. CT Scans
4. Mammography Film
5. Radiographs
6. Graphic Art Film
7. Non Destructive Testing Film
8. Silver Traps like Silver canisters, Silver recovery Cans
9. Fixer Solution, used and unfiltered with silver recover unit.

IV. PROCEDURE

Initially 15 liters of water is boiled up to 70^0C-80^0C 150 grams of NaOH is added to it so that it would be easier to remove the black layer present on X ray films which is the silver bearing coating this coating is then mixed thoroughly in the above solution. The X-ray films are left over in the solution for a brief time of two days so that the black layer gets completely mixed in the solution.

This solution is then taken into the 5 liter container where it is filled up to 3.5 liters. The electrodes of stainless steel are taken to carry out the electrolysis. The electrode dimensions of 14cm*11.5cm are used. The 12V battery is used along with the voltage regulator. The electrodes are washed mildly with acetone to remove any dust present on it.

The connections are made, the cathode is connected to the negative terminal of the battery is connected to the positive terminal of the battery is connected to anode these connections are again connected to the voltage regulator to give a regulated power supply. The wooden stick is used to hold the electrodes.

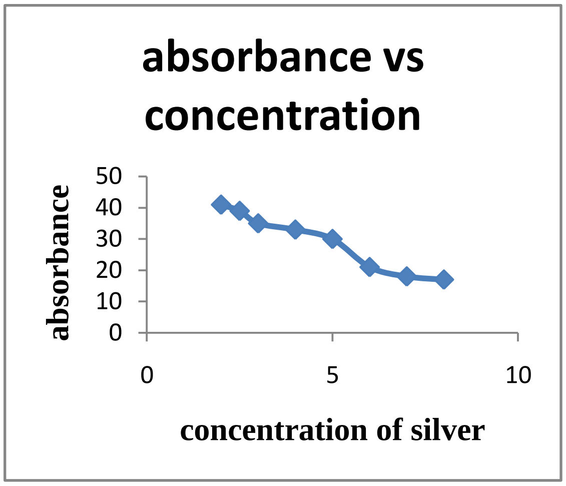
After the connections are made and the apparatus starts to run the solution is taken for every 20 minutes to find the concentration of the silver in the solution, initially the sample is taken before the experimentation. The voltage of the battery is kept at 8.5V throughout the experimentation. Standard solutions of $AgNO_3$ of different ppm were prepared.

Through the UV spectroscopy the samples were analyzed and absorbance values for solutions of different concentrations were found this is used as standard data to get the concentration of silver bearing solution. The experiment is carried out for about 160 min the samples that are taken for every 20 min are analyzed in a UV spectrometer and absorbance values are obtained the concentration values are determined from the graph absorbance vs concentration of the standard $AgNO_3$ solution.

Now, the voltage is varied in between 2V to 8V and the samples of liquid are taken for every 10 minutes. The absorbance values of these samples are also again observed in the UV-spectroscopy the concentrations of these samples are again determined from the standard data generated above.

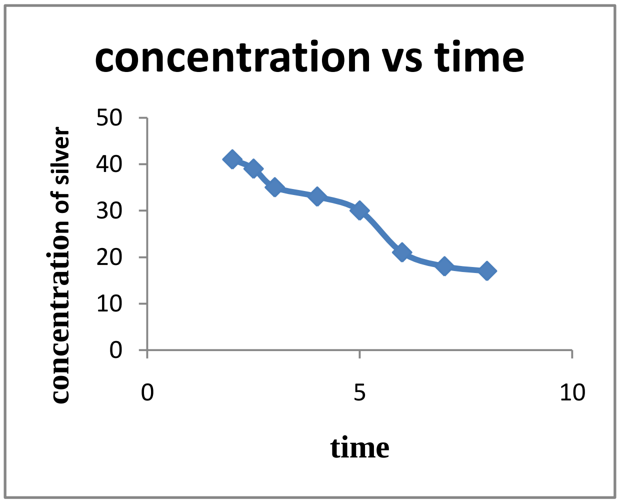
Table Absorbance vs concentration data for standard $AgNO_3$ solution

Absorbance	Concentration of silver in solution, ppm
0.0432	10
0.0707	20
0.0922	30
0.1203	40
0.1366	50
0.158	60
0.1698	70
0.2413	80
0.265	90
0.3211	100



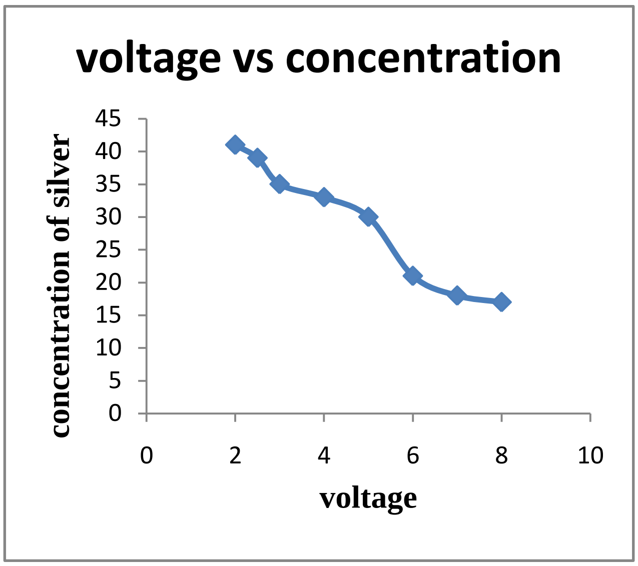
From the above, at a voltage of 8.5 volts the concentration of solution is found out for each time interval its data points are:

Absorbance	Time	Concentration of silver in solution, ppm
0.302	0	94
0.224	20	82
0.1989	40	75
0.188	60	68
0.1703	80	62
0.1698	100	58
0.1436	120	51
0.1412	140	49
0.1398	160	47

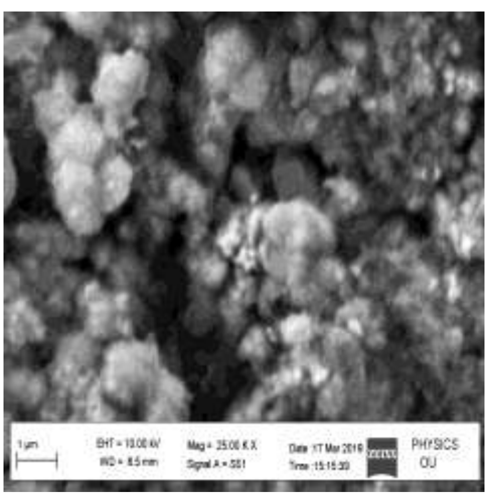


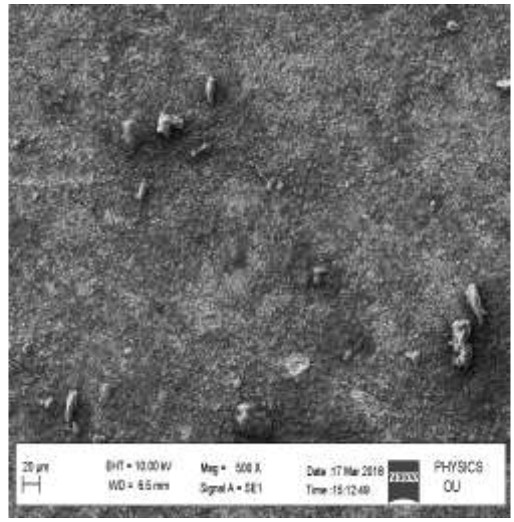
By variation of voltage the concentration of solution is found out for every 10 minutes. The data points generated are:

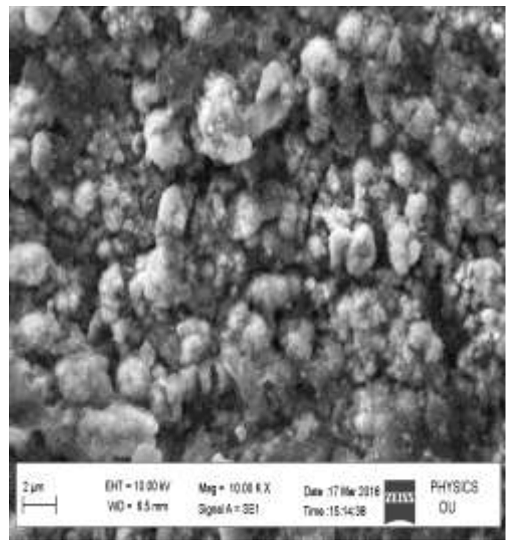
Absorbance	Voltage, volts	Concentration of Ag in solution, ppm
0.1321	2	41
0.1278	2.5	39
0.114	3	35
0.1019	4	33
0.0978	5	30
0.078	6	21
0.0732	7	18
0.0712	8	17

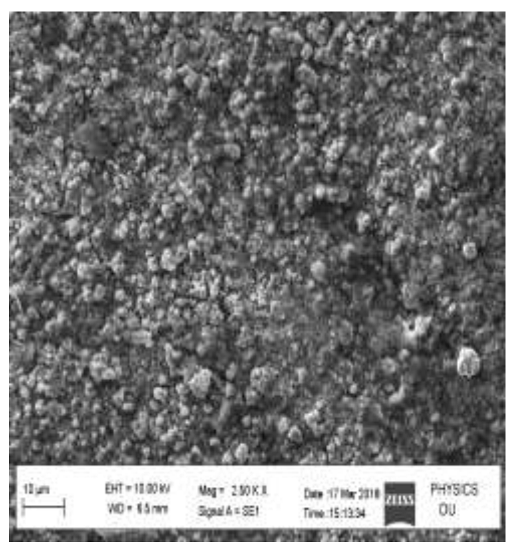


V. SEM ANALYSIS RESULTS

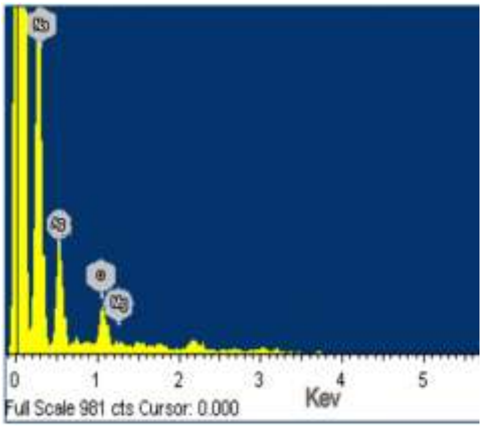








VI. EDX ANALYSIS RESULT



VII. RESULTS AND DISCUSSIONS

1. Different concentrations of $AgNO_3$ are prepared i.e. 10ppm, 20ppm, 30ppm, 40ppm, 50ppm, 60ppm, 70ppm, 80ppm, 90ppm and 100ppm.these solutions are analyzed in a UV-spectrometer, the wave length kept here is 432nm which is the wave length of silver.
2. The samples which are taken at different times those are 20, 40, 60, 80, 100, 120, 140, 160 min. During this time the voltage is kept constant at a voltage of 8.5V . These Samples are analyzed in UV to get absorbance values. From the above plot of absorbance vs concentration was drawn, the concentration of different samples are taken using the obtained absorbance values.
3. The concentration values are obtained and a graph is drawn between concentration vs time.
4. After 160 min the voltage is varied in range of 2V-8V a graph is plotted between Concentration vs Voltage.
5. In EDX analysis we found that besides Silver, Sodium, Oxygen and Magnesium.

VIII. CONCLUSIONS

1. The silver got deposited on SS electrode this was determined from EDX analysis.
2. Though the silver got deposited on plate but sodium has a higher peak since we have used 150 g of NaOH in 15 Liter solution to strip the black layer from X-ray's.
3. In graph between we found that the Concentration vs time concentration of silver drops from 100 min to 120 min and becomes almost constant at 120 min. The optimum time is found at 120 min.
4. In graph conc vs voltage the concentration of silver decreases suddenly at voltage from 5V-6V. It shows that the concentration of silver reduces effectively in this range of voltage

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