ORIGINAL RESEARCH PAPER

Physics

KEY WORDS: XRD, SEM, FTIR, UV, EDAX.

# SYNTHESIS AND CHARACTERIZATION OF SILVER CHLORIDE NANOPARTICLES

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**ABSTRACT**

Silver Chloride nanoparticles were synthesized via chemical co-precipitation method from silver nitrate and sodium chloride. The formed nano particle is characterized by powder X-Ray Diffraction, Scanning Electron Microscopy, Energy Dispersive X-ray Spectroscopy, Ultra-Violet Spectroscopy and Fourier Transform Infrared Spectroscopy and Antibacterial activity. The XRD analysis confirmed the preferential growth of silver chloride nanoparticles that width is 28nm. The SEM image shows the synthesized silver chloride nanoparticles with spherical morphology. The EDAX spectra shows the elements present in the sample. The FTIR spectrum is used to study the stretching and bending frequencies of molecular functional groups in the sample. From UV spectrum, the band gap of silver chloride nanoparticles is found to be 5.3eV. From the antibacterial analysis, the silver chloride nanoparticles are found to have antibacterial activity.

## 1. INTRODUCTION

Silver nanoparticles are of interest because of the unique properties (e.g., size and shape depending optical, electrical, and magnetic properties) which can be incorporated into antimicrobial applications, biosensor materials, composite fibers, cryogenic superconducting materials, cosmetic products, and electronic components. Several physical and chemical methods have been used for synthesizing and stabilizing silver nanoparticles [1, 2]. In this article we have synthesized the Silver chloride nano particles by chemical co-precipitation method. Silver chloride is used in silver plating and to obtain pure silver. It also finds applications in photography and optics; in photo chromic glass; and in electrodes and batteries. It is used to make antiseptic silver solution. It occurs as the mineral cerargyrit.

## MATERIALS AND METHODS

Nanoparticles of silver chloride were prepared by chemical co precipitation method by adding silver nitrate and sodium chloride. Precise amounts of reagents taking into account their purity were weighed and dissolved separately in distilled water into 0.1M concentration. After obtaining a homogeneous solution, the reagents were mixed using magnetic stirring. The precipitate was separated from the reaction mixture and washed several times with distilled water and ethanol. The wet precipitate was dried and thoroughly ground using agate mortar to obtain the samples in the form of fine powder.

## 3. Tests Conducted

X-ray diffraction is an ideal technique for the determination of crystallite size of the powder samples. The basic principle for such a determination involves precise quantification of the broadening of the peaks. XRD line broadening method of particle size estimation was chosen in this investigation for determining the crystallite size of the powder sample. The morphology of the powder samples was studied by the scanning electron microscope (SEM) analysis. The elemental analysis of the sample was studied from EDAX analysis. The infra red spectroscopic (IR) studies of silver chloride nanoparticles were made by using 'SHIMADZU' FTIR 8400S model spectrometer through KBr method. The UV spectrum was taken in the absorbance mode in the wavelength range from 200 to 800 nm. The antibacterial analysis shows the antibacterial activity of the sample.

## 4. RESULTS AND DISCUSSION

### 4.1. XRD studies

### XRD – Particle Size Calculation

The XRD patterns of the prepared samples of silver chloride nanoparticles are shown in figure.1. XRD studies reveal that the samples are nano sized and crystalline. The fine particle nature of the samples is reflected in the X-ray line broadening. The size of the synthesized silver chloride nanoparticles are calculated using Scherrer equation

$$D = 0.9\lambda/\beta\cos\theta \quad (1)$$

where λ represents wavelength of X rays, β represents half width at full maximum and θ is the diffraction angle [3]. The average grain size of the particles is found to be 28nm. The XRD pattern of silver chloride nanoparticles is shown in figure 1.

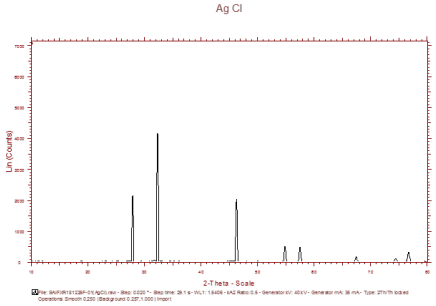


**Figure .1. XRD pattern of silver chloride nanoparticles.**

A good agreement between the Experimental diffraction angle [2θ] and Standard diffraction angle [2θ] of specimen is confirming standard of the specimen. Many peaks at 2θ values of silver chloride is observed and tabulated in table.1 and compared with the standard powder diffraction card of Joint Committee on Powder Diffraction Standards (JCPDS), silver chloride file No. 06-0480. The d-spacing values of experimental is also confirming to the standard values.

**Table.1. Experimental and standard diffraction angles of silver chloride nanoparticles.**
**XRD - Expected 2θ Positions**

| Experimental | Standard – JCPDS 06-0480 | | |
|---|---|---|---|
| Diffraction angle (2θ in degrees) | D spacing (Å) | Diffraction angle (2θ in degrees) | D spacing (Å) |
| 27.86 | 3.1998 | 27.831 | 3.2030 |
| 32.266 | 2.77218 | 32.243 | 2.7740 |
| 46.253 | 1.96125 | 46.233 | 1.9620 |
| 54.835 | 1.67286 | 54.828 | 1.6730 |
| 57.486 | 1.60188 | 57.478 | 1.6020 |
| 67.435 | 1.38767 | 67.471 | 1.3870 |
| 76.71 | 1.24135 | 76.734 | 1.2410 |

The value of d (the interplanar spacing between the atoms) is calculated using Bragg's Law: **2d sinθ = n λ**

$$d = \frac{\lambda}{2 \operatorname{Sin} \theta} \quad (n = 1)$$

Wavelength λ = 1.5418 Ĺ for Cu Ka
The expected 2θ positions of all the peaks in the diffraction pattern and the interplanar Spacing d for each peak is calculated using following formula and the details are shown in table-3.

$$\frac{1}{d2} = \frac{h2+k2+l2}{a2}$$

Bragg's Law is used to determine the 2θ value: The expected 2θ and d values are close with the experimental 2θ and d values.

**Table-3. The Lattice plane and the lattice spacing from d from XRD**

| hkl | 2θ(deg) | | D((Ĺ) | |
|---|---|---|---|---|
| | Experiment | Expected | Experiment | Expected |
| 111 | 27.86 | 27.808 | 3.2042 | 3.1998 |
| 200 | 32.266 | 32.222 | 2.7745 | 2.77218 |
| 220 | 46.253 | 46.2203 | 1.96188 | 1.96125 |
| 311 | 54.835 | 54.804 | 1.67308 | 1.67286 |
| 222 | 57.486 | 57.462 | 1.60184 | 1.60188 |
| 400 | 67.435 | 67.428 | 1.38725 | 1.38767 |
| 420 | 76.71 | 76.715 | 1.24079 | 1.24135 |

**XRD – Dislocation Density**

The dislocation density is defined as the length of dislocation lines per unit volume of the crystal. In materials science, a dislocation is a crystallographic defect, or irregularity, within a crystal structure. The presence of dislocations strongly influences many of the properties of materials. The movement of a dislocation is impeded by other dislocations present in the sample. Thus, a larger dislocation density implies a larger hardness.

The X-ray line profile analysis has been used to determine the dislocation density.

The dislocation density can be calculated from equation

$$\delta = \frac{1}{D^2}$$

Where δ is dislocation density and D is the crystallite size. Results of the dislocation density calculated from the formula is given in table.2. The number of unit cell is calculated from equation

$$n = \pi\,(4/3) \times (D/2)^3 \times (1/V)$$

Where D is the crystallite size and V is the cell volume of the sample.

**Table .2. Dislocation Density and Number of Unit Cell**

| 2θ (deg) | Particle Size D (nm) | Dislocation Density ($m^2$) x1015 δ = 1 / D2 | Number of Unit Cell X$10^5$ |
|---|---|---|---|
| 27.86 | 27.84257629 | 1.28997 | 0.788133618 |
| 32.266 | 28.13128261 | 1.26363 | 0.812905757 |
| 46.253 | 28.79579452 | 1.20598 | 0.871884177 |
| 54.835 | 28.86927479 | 1.19985 | 0.878575768 |
| 57.486 | 28.13819864 | 1.26301 | 0.813505459 |
| 67.435 | 31.62290928 | 9.99992 | 1.154721002 |
| 76.71 | 27.30230031 | 1.34153 | 0.743137738 |

from XRD of silver chloride nanoparticles.

It is observed from these tabulated details, and from figure.2, figure.3 and figure.4, dislocation density is indirectly proportional to particle size and number of unit cells. Dislocation density increases while both particle size and number of unit cell decreases.

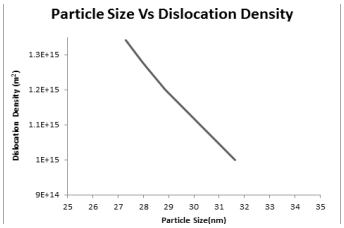


**Figure.2. Particle size Vs Dislocation density curve of silver chloride nanoparticles.**

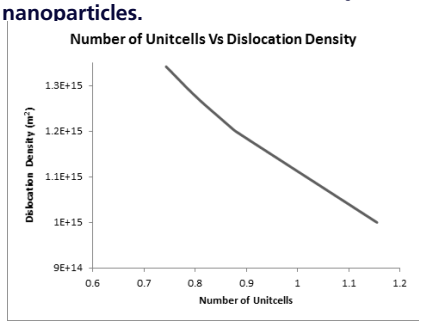


**Figure.3. Number of Unit cells Vs Dislocation density curve of silver chloride nanoparticles.**

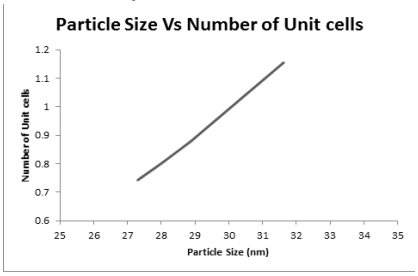


**Figure.4. Particle Size Vs Number of Unit cells curve of silver chloride nanoparticles.**

**XRD – Morphology Index**

A XRD morphology index (MI) is calculated from FWHM of XRD data using the relation

$$M.I = \frac{FWHM_h}{FWHM_h + FWHM_p}$$

Where M.I. is morphology index, FWHMh is highest FWHM value obtained from peaks and FWHMp is value of particular peak's FWHM for which M.I. is to be calculated. The relation between morphology index and particle size is shown in table .3.

**Table .3. Relation between Morphology Index and Particle size for silver chloride nanoparticles.**

| FWHM (β) radians | Particle Size(D) nm | Morphology Index (unitless) |
|---|---|---|
| 0.005129 | 27.84257629 | 0.500016248 |
| 0.005129 | 28.13128261 | 0.500016248 |
| 0.005233 | 28.79579452 | 0.494965741 |
| 0.005408 | 28.86927479 | 0.486771204 |
| 0.005617 | 28.13819864 | 0.477288942 |
| 0.005268 | 31.62290928 | 0.493304836 |
| 0.006472 | 27.30230031 | 0.442121293 |

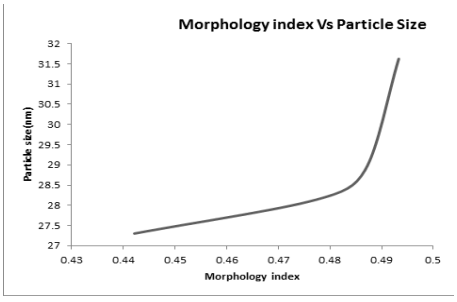


**Figure .5. Morphology Index of silver chloride nanoparticles.**

It is observed that MI has direct relationship with particle size and the results are shown in Figure .5.

**XRD – Unit Cell Parameters**

Unit cell parameters values calculated from XRD are enumerated in table .4.

**Table .4. XRD parameters of silver chloride nanoparticles.**

| Parameters | Values |
|---|---|
| Structure | Cubic |
| Space group | Fm3m [225] |
| Symmetry of lattice | FCC |
| Particle size | 28 nm |
| Lattice parameters | a=5.549 |
| Vol.unit cell(V) | 170.86 |
| Density ( ρ ) | 5.572 |
| Dislocation Density | 1.26363x1015 |
| Mass | 143.32amu |

### 4.2. SEM studies

Scanning electron microscopy was used to analyze the morphology and size of the synthesized silver chloride nanoparticles. Figure.6 shows the SEM images of the silver chloride nanoparticles at various magnifications. The SEM images of silver chloride nanoparticles show nanoparticles with spherical morphology. In this case the particles sizes are slightly increased and is also observed that the particles are distributed with agglomeration.

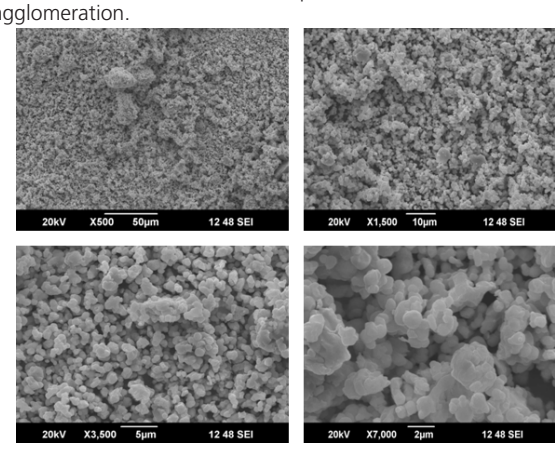


**Figure.6. SEM images of silver chloride nanoparticles at various magnifications.**

### 4.3. EDAX studies

The EDAX elemental analysis of silver chloride nanoparticles shows that the silver chloride nanoparticles was mainly composed of Ag, Cl and O elements. The O element may be due to the water molecule [4]. The EDAX spectrum of silver chloride nanoparticles is shown in Figure.7. The weight percentage of the elements are given in the table.5.

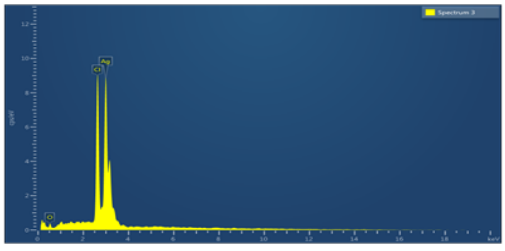

**Figure.7. EDAX spectrum of silver chloride nanoparticles**

**Table.5. The weight percentage of the elements of silver chloride nanoparticles**

| Element | Line Type | Wt% | Atomic % |
|---|---|---|---|
| O | K series | 5.12 | 19.95 |
| Cl | K series | 21.3 | 37.49 |
| Ag | L series | 73.58 | 42.56 |
| Total: | | 100 | 100 |

### 4.4. FTIR Studies

The FTIR spectrum of the silver chloride sample is shown in the figure.8.The FTIR spectrum for silver chloride nanoparticles show peak at 3429.99 $cm^{-1}$ is due to free O-H group and 1626.56 $cm^{-1}$ corresponds to the of hydroxyl group of water. The peak at 1381.57 $cm^{-1}$ represents the presence of $NO_2$ which may be due to silver nitrate solution [5]. The peaks at 759.59 and 617.74 $cm^{-1}$ are due to the presence of chloride.

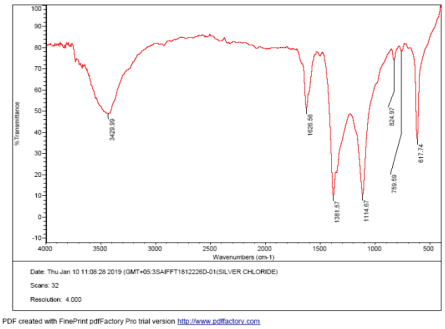


**Figure.8. FTIR spectra of silver chloride nanoparticles.**

### 4.5. UV Studies

The band gap of the prepared sample silver chloride was determined by using UV visible studies. Figure.9 shows the UV-Visible absorption spectra for silver chloride nanoparticles and the maximum absorption is at 230 nm wavelength. Figure.10 shows the graph to find the band gap of silver chloride nanoparticles. From the graph, the optical band gap of silver chloride is 5.3 eV. The optical transmittance of silver chromate nanoparticles is shown in Figure.10 and the transmittance is 96% .

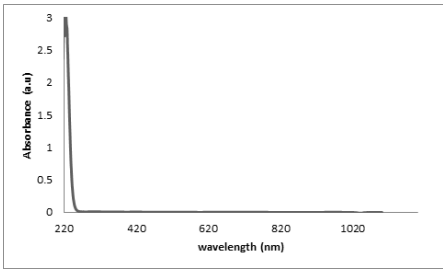


**Figure.9. UV-Visible absorption spectra of silver chloride nanoparticles.**

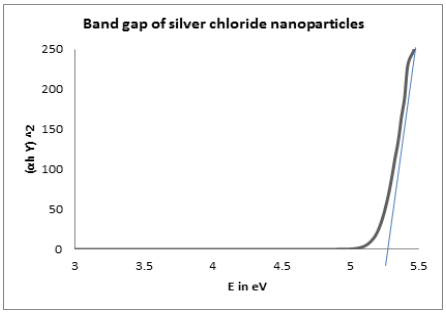


**Figure.10. Graph to find the band gap of silver chloride nanoparticles.**

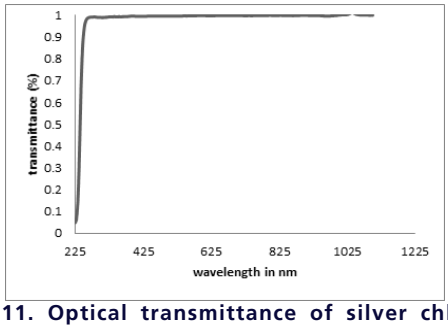


**Figure.11. Optical transmittance of silver chloride nanoparticles**

### 4.6. Antibacterial Activity

The antibacterial activity of silver chloride nanoparticles was studied in different concentrations (25, 50, 75, 100 and 150 µg/ml) against two Gram-positive (*Bacillus sp & Staphylococcus*) and two Gram-negative (*Escherichia coli and klebsiella*) as shown in Figure.12.

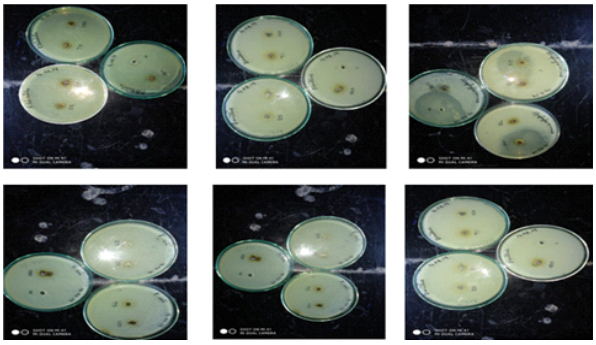

**Figure.12. Antibacterial activity of silver chloride nanoparticles**

Antibacterial potential of Silver Chloride was assessed in terms of zone of inhibition of bacterial growth. The results of the antibacterial activity are presented in the form of Table.6 and Table.7.

**Table.6.Antibacterial activity**

| Zone of inhibition (in mm) | | | | |
|---|---|---|---|---|
| **Concentration (µg/ml)** | ***E. coli*** | ***klebsiella*** | ***Bacillus*** | ***Staphylococcus*** |
| 25 | 10 | 06 | 7 | 12 |
| 50 | 14 | 10 | 10 | 18 |
| 75 | 16 | 13 | 15 | 23 |
| 100 | 18 | 15 | 19 | 26 |
| 150 | 21 | 17 | 23 | 31 |

**Table.7.Antibiotics and the culture**

| **Culture** | **Antibiotics** | | | |
|---|---|---|---|---|
| | **Gentamycin** | **Ampicillin** | **Penicillin** | **Methicilin** |
| *E. coli* | 23 | - | 13 | - |
| *klebsiella* | 22 | 16 | 18 | - |
| *Bacillus* | 23 | - | - | - |
| *Staphylococcus* | 32 | 32 | 29 | 28 |

The antibacterial activity of the sample increased linearly with increase in concentration of $AgCl_2$ ( g/ml). The results revealed that the silver chloride nanoparticles shows potential antibacterial activity.

## 5. CONCLUSIONS

The silver chloride nanoparticles have been prepared by chemical co-precipitation method. XRD analysis suggests that the average particle size is in the nano range (28nm). The SEM picture reveals the nanoparticles with spherical morphology. From the EDAX spectrum, the elements present in the sample are found. From the FTIR spectrum, the stretching and bending frequencies of the molecular functional groups in the sample are studied. From the UV spectra, the band gap was found. From the antibacterial activity of the silver chloride nanoparticles, it was found that the sample shows potential antibacterial activity.

## 6. REFERENCES

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