**Synthesis and characterization of Ag nanoparticle inter Nickel/Aluminum-layer double hydroxide nano hybrids compounds**

Eussur N Alkhafaji.

College of Pharmacy, University of Ahle AL-Bait, Karbala, Iraq

Yosornoory@gmail.com

**Abstract**

This study describes the synthesis of nano hybrids compounds as silver nanoparticles AgNPs inter Ni-Al-layered double hydroxide (LDH) nano hybrid by ion exchange method. Ag as Ag NPs by reduce agent in starch solutions. The produced AgNPs are integrated on LDH surface. The material was characterized by X-ray Diffraction , Fourier Transform Infare spectroscopy , Atomic Force Microscopy and UV-Visible spectrophotomer. The expanded interlayer spacing of the Ag NPs/Ni-Al-LDH nano hybrid suggests the successful encapsulation of Ag NPs with Ni-Al-LDH. The prepared Ag NPs/Ni-Al-LDH nano hybrid compound could be suitable to use in future antimicrobial and anti bacterial applications ,the make use of, more than 99% of all types of bacteria can be killed , also the AgNPs-LDH can also efficiently inhibit the bacterial growth and prevent the bio film formation in the nutrient solutions. These recently Ag NPs-LDH may offer a talented antimicrobial solution for clinical and environmental applications.

**Key words:** AgNPs, LDH, Nano hybrid compounds

1. Introduction

Nano compounds show single properties , which cannot be observed in their bulk complement as a result of the high surface area to volume ratios and quantum confinement.\(^1\)\(^2\) Synthesis of metal nanoparticles is of attention due to their new optical, electronic, magnetic and catalytic properties which show the way to many potential applications and these properties highly depend on the size and the shape of the metal nanoparticles.\(^3\) Silver (Ag) is a several element used in photographic, textile semiconductor industries and specially it has been used to avoid and treat a diversity of diseases causing microorganisms.\(^4\)\(^5\)\(^6\) Silver nanoparticles are extremely toxic to microbes whereas there is a low toxicity towards animal cells. They have been usually used in dental resin formulations, in bone cement and in medical devices coatings.\(^7\) Silver nanoparticles react with sulfur of proteins in the microbial cell membrane which reduces the cell capability.\(^8\) Agglomeration of Ag NPs is one of the major causes which reduces the activity of Ag NPs. To defeat this problem, Ag NPs can be surface modified using a suitable modifier and further they can be integrated within inter layer chairs of a layered material which also act as a matrix to hold nanoparticles.\(^9\) In recent years much interest has been paid on the development of antimicrobial surfaces for medical devices, household products, food packaging, due to an escalating pressure exerted by the disease-causing microbes on the public health. Inorganic coatings which exhibit good mechanical strength, chemical stability, and physichemical compatibility with ceramic and metallic objects are of particular interest for antimicrobial applications. To the best of our knowledge, LDHs-coated surfaces with antimicrobial functionality have not been reported so far.\(^10\) Layered double hydroxides as well known as anion clays or hydrotalcite-like compound, consist of stacks of positively charged hydroxide layers and interlayer charge balancing anions. The structure of the LDHs can be describe by considering the structure of bruited Mg(OH)\(_2\), in which the Mg\(^{2+}\) ions are octahedral coordinated to hydroxyl groups.\(^11\) The space between the octahedral(e.g., divalent Mg\(^{2+}\), Co\(^{2+}\), Zn\(^{2+}\) and trivalent Al\(^{3+}\), Ga\(^{3+}\), Fe\(^{3+}\), Mn\(^{3+}\), Gd\(^{3+}\) etc.) layers may be busy by intercalated anions nucleotides, fluorescent molecules, radio-labeled ATP, vitamins,\(^12\) DNA, insecticide,\(^13\) and drugs, with water held in place via hydrogen bonding to the hydroxyls, using the pharmacokinetics, toxicity, and therapeutic efficacy of layer double hydroxide have been reported.\(^14\) In arrange for this LDH stage to compete with polymeric and other inorganic, it is important to show the
2. Materials and Methods

2.1. Synthesis of Ni/Al-LDHs by co-precipitation method

The natural NO$_3$-LDHs were prepared by a usual Co-precipitation method. A mixed solution of 0.1M of Ni(NO$_3$)$_2$·6H$_2$O and 0.05M of Al(NO$_3$)$_3$·9H$_2$O when molar ratio R=2/1. At temperature 25°C and pH was adjusted at (6.5) for the Ni/Al-LDHs by drop wise addition of 2M NaOH solution through the step by step of the mixed solution with stirring for 12 h. Separate the precipitate the washing by D.W and drying at room temperature.\textsuperscript{13,20}

2.2. Synthesis of silver nanoparticles

Prepared silver nanoparticles in 0.003M by dissolved 0.015g of AgNO$_3$ in 20 mL D.W and 10 mL ethanol, then add in solution 20mL of 2% starch solution was placed in an water bath and stirred for about 30 min using a magnetic stirrer at its temperature 60°C at pH=8. Solution was treated with starch to reduce the Ag$^+$ ions into Ag.

2.3. The nano hybrid compound Ag NPs-/Ni-Al-LDH prepare by two methods

2.3.1. First method (Indirect Ion Exchange)

Dissolved 2.9g of Ni(NO$_3$)$_2$·6H$_2$O , 1.8g Al(NO$_3$)$_3$·9H$_2$O in 100 mL ethanol when molar ratio R=2/1. Adjusted the PH at 6.5 by 2M sodium hydroxide. Gradually addition silver nanoparticles in 0.003M. Incubate the solution in water bath with shaking at 60°C for 24 h. Separate the precipitate the washing by D.W and drying at room temperature.

2.3.2. Second method (Direct ion exchange)

Dissolve 0.5g of Ni/Al- LDHs which prepare by Co-precipitation method. In 100mL ethanol. Gradually addition silver nanoparticles in 0.003M. Incubate the solution in water bath with shaking at 60°C for 24 h. The precipitate Separates, Washing by D.W and drying at room temperature.

3. Characterization techniques

The physicochemical properties of nano hybrids Ag PNs in Ni/Al –LDHs are important for their behavior, safety, bio-distribution, efficacy and evaluate the functional aspects. Characterization is performed of The Ag NPs- Ni/Al -LDHs nano hybrid materials using a various of analytical techniques, including X-ray diffractometry (XRD), Fourier transform infrared spectroscopy (FTIR) and atomic force microscopy (AFM).

3.1. UV-Visible spectrophotometer

Optical properties of the obtained yellow green color silver nanoparticle solutions were monitored on a UV-VIS spectrophotometer (Shimadzu UV-3600)

3.2. Fourier transform infrared spectroscopy (FTIR)

The chemical structure and functional group responsible for Ni/Al –LDH and Ag NPs- Ni/Al -LDHs nano hybrid was also analyzed by using FT-IR spectroscopy (JASCO FT/IR) in ratio1:50, sample: potassium bromide (KBr) and wavelength range (4000-400) cm$^{-1}$ at room temperature.

3.3. X-ray diffraction (XRD)

X-ray diffraction was conducted by XPERT-PRO using monochromatic Cu k\alpha radiation (2dsinθ=\textit{nλ}) while (λ=1.540562Å) , n=1 operated at 40 kV and 30 mA at a 2θ angle pattern. The scanning was done in the region of10°–70°. It provides information on particle phases, crystal structures, preferred texture and other structural parameters, such as degree of crystallinity, average grain size, strain, and crystal defects.
3.4. Atomic Force Microscopy (AFM)

Use the atomic force microscopy to examine the Ni/Al-LDH and Ag NPs- Ni/Al-LDHs nano hybrid compound and measurement of diameter, volumes and gatherings nanomaterials, the models were sent to the College of science/ University of Baghdad for the purpose of examination.

4. Result and Discussion

4.1. UV-Visible spectrophotometer

The UV-Vis spectrum results figure 1 established the configuration of Ag NPs which resulted a strong absorption at 340 nm. Site of the absorption bond highly depends on the particle size can be observed with the increase in size of the nanoparticle. Increase in absorption wave length explain that the size of the Ag NPs increases due to aggregation with time and bigger particles involve lesser energy, therefore longer wavelength, and color change was noted by visual watching from colorless to light yellow and, eventually, to yellow green. This color change indicates the formation of AgNP in the solution. Extract without AgNO\textsubscript{3} did not show any color changes.\textsuperscript{5}

![Figure 1. UV-Vis for silver nanoparticles solution](image)

4.2. Fourier transform infrared spectroscopy (FTIR)

Figures (2-5) explains FT-IR spectra of Silver Nanoparticles, Ni/Al – NO\textsubscript{3}-LDH and Ag NPs-Ni/Al-LDH.

In figure 2 The Silver Nanoparticles show the bands at 3373 attributed to \(\nu\)O-H and 1624 cm\(^{-1}\) must be attributed to \(\delta\)O-H, because in the oxidation some of the hydroxyl group residue unreactive and also some moisture get absorb on the highly reactive surface of nanoparticles.\textsuperscript{22} The peak at 2359cm\(^{-1}\) attributed to uC-C band in starch,\textsuperscript{23} the bands at 1514 and 1500cm\(^{-1}\) attributed to Nitrate from AgNO\textsubscript{3}, which is preliminary metal forerunner.\textsuperscript{24} When we compared the standard FTIR spectra of AgNO\textsubscript{3}.See the table 1.

![Figure 2. FTIR spectra of Silver Nanoparticles.](image)
In figure 3 The layers double hydroxide, Ni/Al – NO$_3$-LDH show the absorption peaks to υO–H group stretches at 3443 cm$^{-1}$ and weak absorption peak of δ-OH at 1639 cm$^{-1}$. The NO$_3$ absorption peak at 1386 cm$^{-1}$ in the LDHs. Absorption peaks to υNi–O at 408 cm$^{-1}$ in layers and absorption peaks to υAl–O at 621 cm$^{-1}$ in layers. See the table 1.

![Figure 3. FT-IR spectrum of Ni/Al – NO$_3$-LDH.](image)

In figure 4 The nano hybrid compound AgNPs Ni/Al–LDH appears many anew peaks this mean successful for intercalation of Silver Nanoparticles between the layers, assimilation peaks of alkyl υC–H stretch in the 2933 cm$^{-1}$ and 2943 cm$^{-1}$ in the prepare indirect and direct respectively. The assimilation peaks of cycle υC–H stretch in the 1151 cm$^{-1}$ and 1153 cm$^{-1}$ that prepared in an indirect and direct respectively. While the weak absorption peak of δ-CH aliphatic at 705 cm$^{-1}$ and 704 cm$^{-1}$ the prepare in an indirect and direct respectively and absorption peak of cycl δ-CH at 833 and 829 cm$^{-1}$ that prepared in an indirect and direct respectively, wide absorption peak in the (3497 cm$^{-1}$ and 3425 cm$^{-1}$) is assigned to υO–H and weak absorption peak of δ-OH at 1641 cm$^{-1}$ and 1637 cm$^{-1}$ for prepared in an indirect and direct method respectively. The NO$_3$ gives an assimilation peak at 1386 cm$^{-1}$ in the LDHs, absorption two peaks of υC-O-C at 1084 cm$^{-1}$ and 1049 cm$^{-1}$ in preparer indirect but the two beak of υC-O-C at 1082 cm$^{-1}$ and 1016 cm$^{-1}$ in preparer direct. Absorption peaks to υNi–O at 432 cm$^{-1}$ and 439 cm$^{-1}$ in layers and absorption peaks to υAl–O at 563 cm$^{-1}$ and 606 cm$^{-1}$ in layers. See the table 1.

![Figure 4. FT-IR Spectra of Ag NPs Ni/Al-LDH (a) prepare by indirect method (b) prepare by direct method](image)
Table 1. Peaks of the Silver Nanoparticles, Ni/Al-NO₃-LDH and the nano hybrid compound Ag NPs Ni/Al –LDH.

| Frequency | Nanohybridcompound prepared direct ion exchange | Nanohybridcompound prepared indirect ion exchange | LDH | Silver Nanoparticles |
|-----------|-----------------------------------------------|-----------------------------------------------|-----|---------------------|
| v_{O-H}   | 3479                                          | 3425                                          | 3443| 3460                |
| δ_{O-H}   | 1641                                          | 1637                                          | 1639| 1573                |
| v_{C-H}   | 2933                                          | 2943                                          | ----| 2359                |
| v_{C-C}   | 1151                                          | 1153                                          | ----|                     |
| C-H cycle | 1543,1508                                    | 1537                                          | 1514,1500 |               |
| Ag        | 1381                                          | 1381                                          | 1386|                     |
| u_{NO3}   | 1084                                          | 1082                                          | ----|                     |
| u_{C-O}   | 1049                                          | 1016                                          | ----|                     |
| u_{C-C}   | 833                                           | 829                                           | ----|                     |
| C-H cycle | 432                                           | 439                                           | 408 |                     |
| u_{Ni-O}  | 563                                           | 606                                           | 621 |                     |
| Al-Ou     | 705                                           | 704                                           | ----|                     |

4-3-X-ray diffraction (XRD)

The silver nanoparticles has been explained by X-Ray Diffraction. The diffracted intensities were recorded from 20° to 70° at 2θ. The show figure 5, assay the value of thin by using the Bragg low (2dsinθ = nλ)³⁰ while (λ=1.540562Å) n=1. And figure show the crystal level is (hkl). The calculated by using the observed 2θ values equivalent to each peak. The powdered silver nanoparticles show a cubic structure showing peaks at 2θ= 38 d=0.225nm at the (111) crystal level.2θ=44, d=0.2nm at the crystal level (200). 2θ=63 , d= 0.15nm (220). The high powerful bond for material is (111).²⁴,³¹

Figure 5. XRD of Silver Nanoparticles.

The LDH fig.6 has been explained by X-Ray Diffraction. The diffracted intensities were recorded from 10° to 80° at 2θ. Asssay the value of thin by using the Bragg low The figure show the crystal level is (hkl). The calculated by using the observed 2θ values equivalent to each bands .The appeared of bond at crystal level (003)spacing of 0.76 nm  2θ = 11.5°. The available data on the thickness  0.4 nm at (006),²² but the 2θ =29⁰ interlayer spacing is found to 0.3 nm at (009). Identification of the peaks
between 2θ of 37-40° shows that LDH (012) peaks on the thickness 0.25 nm. The 2θ of 60-65° shows the crystal level (110) and (113).

Figure 6. XRD of Ni/Al–NO₃–LDH

Figure 7 explains the spectra of X-Ray diffraction through the different in the value of thin layer and the crystal level after intercalation the silver nanoparticles into the layer by using the Bragg low is the show of the integration the bands of layer double hydroxide with bands of silver nanoparticles. This result show the succeed of the formation process nano hybrid compound Ag NPs-LDH. The figure show the crystal level is (hkl) from (003) until (220). The calculated by using the observed 2θ values equivalent to each bands. The appeared of bond at crystal level (003) spacing of 0.76 nm 2θ = 11.5°. The available data on the thickness 0.34 nm at (006). But the 2θ = 29° interlayer spacing is found to 0.28 nm at (009). That refers to form a cubic structure which showing in peaks at 2θ=38 d=0.25nm at the (111) crystal level. 2θ=44 , d=0.18nm at the crystal level (200). 2θ=65,d= 0.1nm (220).

Figure 7. XRD diffraction patterns of (a) nanohybrid compound prepared direct ion exchange, (b) nanohybrid compound prepared indirect ion exchange.
4.4. Atomic Force Microscopy (AFM)

Figures (8,10,12 and 14) Explain the characterized by using (AFM) for the two and three-dimensional image to silver nanoparticles, layer Ni/Al NO$_3$–LDH, and Nano hybrid compound Ag NPs-/Ni-Al-LDH . The outer surface of the silver nanoparticles was studied and the figure 9a is shown a two-dimensional image of the silver nanoparticles in where molecular clusters appear while as shown in the figure 9b a three-dimensional image that has not been cut from the surface of the silver nanoparticles is given a high molecular assemblies which has limits range (-1.43 – 31.7)nm.

![Figure 8. AFM image of silver nanoparticles](image)

Table 2 and figure 9 show the average diameters of silver nanoparticles 73.07nm. The process of preparing silver nanoparticles to obtain nanoparticles with diameters between than (40-100)nm , and the highest percentage of those nanoparticles is 11.31% particles to the diameter 95nm ,between the lowest ratio is 1.77% particles with a diameter of 40nm.

**Table 2. Diameters, sizes and aggregation of the molecular silver nanopartical in AFM .**

| Diameter (nm) | Volume (%) | Cumulation (%) | Diameter (nm) | Volume (%) | Cumulation (%) | Diameter (nm) | Volume (%) | Cumulation (%) |
|--------------|------------|----------------|--------------|------------|----------------|--------------|------------|----------------|
| 40.00        | 1.77       | 1.77           | 65.00        | 9.54       | 30.74          | 90.00        | 8.83       | 81.98          |
| 45.00        | 3.89       | 5.65           | 70.00        | 9.54       | 40.28          | 95.00        | 11.31      | 93.29          |
| 50.00        | 5.30       | 10.95          | 75.00        | 11.31      | 51.59          | 100.00       | 6.71       | 100.00         |
| 55.00        | 4.24       | 15.19          | 80.00        | 10.60      | 62.19          |              |            |                |
| 60.00        | 6.01       | 21.20          | 85.00        | 10.95      | 73.14          |              |            |                |

Avg. Diameter:73.07 nm <=10% Diameter:45.00 nm
<=50% Diameter:70.00 nm <=90% Diameter:90.00 nm
Figure 9. Percentage of diameter of silver nanoparticles

The outer surface of the layer Ni/Al NO$_3$–LDH$^{13}$ was studied and the figure figure 11a is shown a two-dimensional image of the layer Ni/Al NO$_3$–LDH in which spherical semi-shapes molecular appear while as shown in the figure 11b a three-dimensional image that has not been cut from the surface of the layer Ni/Al NO$_3$–LDH is given a high molecular assemblies which has limits range (-0.21 – 13.5)nm.

Figure 10. AFM image of(Ni/Al –LDH)(a) two- dimensional image(b) three- dimensional image.

Table 3 and figure 11 show before the intercalation process silver nanoparticles with layer Ni/Al NO$_3$–LDH the average diameters of layer Ni/Al NO$_3$–LDH 83.80nm. The process of preparing layer Ni/Al NO$_3$–LDH to obtain nanoparticles with diameters between than (45-180)nm , and the highest percentage of those nanoparticles is 8.9% particles to the diameter 75nm ,between the lowest ratio is 0.27% particles with a diameter of 140nm.
### Table 3. Diameters, sizes and aggregation of the molecular layer Ni/Al- NO₃-LDH in AFM.

| Diameter (nm) | Volume (%) | Cumulation (%) | Diameter (nm) | Volume (%) | Cumulation (%) | Diameter (nm) | Volume (%) | Cumulation (%) |
|--------------|------------|----------------|--------------|------------|----------------|--------------|------------|----------------|
| 45.00        | 4.62       | 4.62           | 90.00        | 7.07       | 63.59          | 135.00       | 1.63       | 96.74          |
| 50.00        | 3.80       | 8.42           | 95.00        | 6.25       | 69.84          | 140.00       | 0.27       | 97.01          |
| 55.00        | 5.16       | 13.59          | 100.00       | 4.62       | 74.46          | 145.00       | 0.54       | 97.55          |
| 60.00        | 4.08       | 17.66          | 105.00       | 5.71       | 80.16          | 150.00       | 0.27       | 97.83          |
| 65.00        | 7.61       | 25.27          | 110.00       | 3.53       | 83.70          | 155.00       | 1.09       | 98.91          |
| 70.00        | 8.15       | 33.42          | 115.00       | 3.80       | 87.50          | 160.00       | 0.27       | 99.18          |
| 75.00        | 8.97       | 42.39          | 120.00       | 1.09       | 88.59          | 165.00       | 0.27       | 99.46          |
| 80.00        | 8.15       | 50.54          | 125.00       | 3.80       | 92.39          | 170.00       | 0.27       | 99.73          |
| 85.00        | 5.98       | 56.52          | 130.00       | 2.72       | 95.11          | 180.00       | 0.27       | 100.00         |

Avg. Diameter: 83.80 nm  <=10% Diameter: 50.00 nm  
<=50% Diameter: 75.00 nm  <=90% Diameter: 120.00 nm

![Figure 11](image1.png)

**Figure 11.** Percentage of diameter of layer Ni/Al–LDH

The outer surface of the nanohybrid compound Ag NPsNi/Al–LDH prepared by indirect ion exchange method was studied and the figure 13a is shown a two-dimensional image of the nanohybrid compound Ag NPsNi/Al–LDH in where molecular clusters of spherical shapes appear while as shown in the figure 13b a three-dimensional image that has not been cut from the surface of the silver nanoparticles is given a high molecular assemblies which has limits range (0.09 – 44.26)nm, suggesting the manufacture of nano compound from silver nanoparticles and layer Ni/Al–LDH.

![Figure 12](image2.png)

**Figure 12.** AFM image of Ag NPsNi/Al–LDH (a) two- dimensional image (b) three- dimensional image.
Table 4 and figure 13 show the average diameters of nanohybrid compound Ag NPsNi/Al–LDH prepared by indirect ion exchange method 80.87 nm. The process of preparing layer Ni/Al NO₃–LDH to obtain nanoparticles with diameters between than (60-150) nm, and the highest percentage of those nanoparticles is 12.47% particles to the diameter 75 nm, between the lowest ratio is 0.28% particles with a diameter of 150 nm.

Table 4. Diameters, sizes and aggregation of the molecular Ag NPsNi/Al–LDH prepared by indirect ion exchange method in AFM.

| Diameter (nm)<60   | Volume(%) | Cumulation(%) | Diameter (nm)<65 | Volume(%) | Cumulation(%) | Diameter (nm)<70 | Volume(%) | Cumulation(%) | Diameter (nm)<75 | Volume(%) | Cumulation(%) | Diameter (nm)<80 | Volume(%) | Cumulation(%) |
|-----------------|-----------|---------------|-----------------|-----------|---------------|-----------------|-----------|---------------|-----------------|-----------|---------------|-----------------|-----------|---------------|
| 60.00           | 3.32      | 3.32          | 90.00           | 11.36     | 75.35         | 120.00          | 1.66      | 98.61         |
| 65.00           | 14.13     | 17.45         | 95.00           | 6.37      | 81.72         | 125.00          | 0.55      | 99.17         |
| 70.00           | 12.19     | 29.64         | 100.00          | 4.71      | 86.43         | 135.00          | 0.55      | 99.72         |
| 75.00           | 12.47     | 42.11         | 105.00          | 5.54      | 91.97         | 150.00          | 0.28      | 100.00        |
| 80.00           | 11.08     | 53.19         | 110.00          | 4.16      | 96.12         |                |           |               |
| 85.00           | 10.80     | 63.99         | 115.00          | 0.83      | 96.95         |                |           |               |

Avg. Diameter: 80.87 nm <=10% Diameter: 60.00 nm
<=50% Diameter: 75.00 nm <=90% Diameter: 100.00 nm

Figure 13. Percentage of diameter of nanohybrid compound Ag NPsNi/Al–LDH prepared by indirect ion exchange method

The outer surface of the nanohybrid compound Ag NPsNi/Al–LDH prepared by direct ion exchange method was studied and the figure 15a is shown a two-dimensional image of the nano hybrid compound Ag NPsNi/Al–LDH in where molecular clusters of spherical shapes appear while as shown in the figure 15b a three-dimensional image that has not been cut from the surface of the silver nanoparticles is given a high molecular assemblies which has limits range (0.01 – 46.39) nm, suggesting the manufacture of nano compound from silver nanoparticles and layer Ni/Al –LDH.

Figure 14. AFM image of Ag NPsNi/Al–LDH prepared by direct ion exchange method (a) two-dimensional image (b) three-dimensional image.
Table 5  and figure 1 show the average diameters of nanohybrid compound Ag NPsNi/Al–LDH prepared by direct ion exchange method 54.68nm. The process of preparing layer Ni/Al NO3–LDH to obtain nanoparticles with diameters between than (35-75)nm , and the highest percentage of those nanoparticles is 16.53% particles to the diameter 60nm ,between the lowest ratio is 3.11% particles with a diameter of 35nm.

Table 5. Diameters , sizes and aggregation of the molecular in Ag NPsNi/Al–LDH prepared by direct ion exchange method in AFM.

| Diameter (nm)< | Volume(%) | Cumulative(%) | Diameter (nm)< | Volume(%) | Cumulative(%) | Diameter (nm)< | Volume(%) | Cumulative(%) |
|---------------|-----------|---------------|---------------|-----------|---------------|---------------|-----------|---------------|
| 35.00         | 3.11      | 3.11          | 50.00         | 10.15     | 35.19         | 65.00         | 13.91     | 78.23         |
| 40.00         | 7.53      | 10.64         | 55.00         | 12.60     | 47.79         | 70.00         | 13.91     | 92.14         |
| 45.00         | 14.40     | 25.04         | 60.00         | 16.53     | 64.32         | 75.00         | 7.86      | 100.00        |

Avg. Diameter:54.68 nm <=10% Diameter:35.00 nm <=50% Diameter:55.00 nm <=90% Diameter:65.00 nm

5. Conclusion
This work can be prepare nano hybrid compound by intercalation of silver nanoparticles in layer double hydroxide as nano hybrid compound Ag NPs Ni/Al-LDH was prepared by ion exchange method. Ag NPs were synthesized by reduction of silver ions using starch solution. The synthesized surface modified Ag NPs were encapsulated with LDH by the inset synthesis of Ni/Al-LDH which works as a matrix to hold the silver nanoparticles. The prepared nano hydride compound capacity Ag NPs/Ni-Al-LDH nanohybrid material could be appropriate to use in antimicrobial applications like wound and burn dressing.

References
[1] Madusanka N, Sandaruwan, C, Kottegoda, N and Karunaratne, V 2014 Journal of Applied Science and Technology Synthesis of Ag Nanoparticle/Mg-Al-Layered Double Hydroxide Nanohybrids. European International I(1), I-7.
[2] Tricoli A and Pratsinis SE 2010 Nat Nanotechnol Dispersed nanoelectrode devices. 5(1),54–60.
[3] Abdul W W, Abdul K , Nursiah LA-N, Nurafni and Wayan S 2018 ORIENTAL JOURNAL OF CHEMISTRY Synthesis of Silver Nanoparticles using Muntingia calabura L. Extract as Bioreductor and Applied as Glucose Nanosensor. 34(6), 3088-3094.
[4] Devi LS, Joshi SR 2012 Mycobiology Antimicrobial and synergistic effects of silver nanoparticles synthesized using soil fungi of high altitudes of eastern Himalaya. 40(1), 27–34.
[5] Yan H, Xi Z, Zhiyun D and Kun Z 2013 International Journal of Nanomedicine Green synthesis of silver nanoparticles by Chrysanthemum morifolium Ramat. extract and their application in clinical ultrasound gel. 8 , 1809–1815.
Crystalline Silver Nanoparticles

One Pot Synthesis of Ag/AgCl nanoparticle decorated layered dou

Hydroxides

Agasit N and Kaushik N K 2014 American Journal of Nanomaterials One Pot Synthesis of Crystalline Silver Nanoparticles. 2(1),4-7

Cai Y, Tan F, Qiao X, Wang W, Chen J and Qiu X 2016 RSC Adv. Room-temperature synthesis of silica supported silver nanoparticles in basic ethanol solution and their antibacterial activity. 6(22), 18407–18412.

Tzounis I and Logothetidis S. 2017 Mater. Today-Proc Fe3O4 and SiO2 core shell particles as platforms for the decoration of Agnanoparticles. 4(7), 7076–7082.

Nocchetti M, Donnadio A, Ambrogi V, Andreani P, Bastianini M, Pietrella D, and Latterini L 2013 Journal of Materials Chemistry B Ag/AgCl nanoparticle decorated layered double hydroxides: synthesis, characterization and antimicrobial properties. J(18), 2383-2393.

Chen C, Gunawan P, Lou X W and Xu R 2012 Advanced Functional Materials Silver nanoparticles deposited layered double hydroxide nanoporous coatings with excellent antimicrobial activities. 22(4), 780-787.

Yuser N A, Abbas M B and Salih M H 2011 Karbala journal university Synthesis of Zn/Al-layered double hydroxide nano hybrids with 2,4- dichloro and 4- chlorophenoxy acetate.9(1), 147-164

Noor A A and Eussur N A 2019 Karbala journal university Synthesis and Characterization of nano hybrids ascorbic acid in Ni/Al –LDHs by direct ion exchange and Its Potency for Inhibition to Pseudomonas aeruginosa. 139, 4691

Eussur N A, Maryam M M and Alaa A. A 2019 Journal of Engineering and Applied Sciences Preparation and Characterization of Mg/Fe-LDH Nano Hybrids with(O, O-Diethy1 O-3, 5, 6Trichloro-2-Pyridyl Phosphorothioate(Chlorpyrifos) by Direct and Indirect Ion Exchange Method. 14(9) , 10723-10732

Noh W, Sun X, Ramachandran A, Rearick C, and Dey S K 2012 Int. J. Mater. Sci. Synthesis and characterization of silver (core)/layered double hydroxide (shell) nanoparticles. 2, 83-87.

Yuser N A, Abbas M B and Salih M H 2012 Karbala journal university Thermal Analysis for Nano hybrid Compounds and degradation degree Thermal found. 10(3) , 36-43.

Zubair M, Daud M, McKay G, Shehzad F and Al-Harthi M A 2017 Appl. Clay Sci. Recent progress in layered double hydroxides (LDH)-containing hybrids as adsorbents for water remediation. 143, 279–292.

Ruiz-Hitzky E, Aranda P, Darder M and Rytwo G 2010 J. Mater. Chem. Hybrid materials based on clays for environmental and biomedical applications. 20(42), 9306–9321.

Mishra G, Dash B, Pandey S and Mohanty P P 2013 J. Environ. Chem. Eng. Antibacterial actions of silver nanoparticles incorporated Zn-Al layered double hydroxide and its spinel. 1(4), 1124–1130.

Gohi, Bi Foua C A, Hong-Yan Z, Sheng X, Kai-Min Z, Binyao L, Xiu Li Hand Xiao-Ju C 2019 International Journal of Molecular Sciences Optimization of ZnAl/Chitosan Supra Nano Hybrid Preparation as E cient Antimicrobial Material. 20(22), 5705

Goh K H, Lim T T and Dong, Z 2008 Water research Application of layered double hydroxides for removal of oxyanions: a review. 42(6-7), 1343-1368.

Loo YY, Chieng BW, Nishibuchi M, Radu S 2012 Int J Nanomedicine. Synthesis of silver nanoparticles by using tea leaf extract from Camellia sinensis. 7, 4263–4267.

Janardhanan R, Karuppuiah M, Hebalkar N and Rao T N 2009 Polyhedron Synthesis and surface chemistry of nano silver particles. 28(12), 2522-2530.

Theivasanthi T and Alagar M 2011 Nano Biomedicine and Engineering Electrolytic synthesis and characterizations of silver nanopowder. arXiv preprint arXiv:1111.0260.

Muzamil M, Khalid N, Aziz M D and Abbas S A 2014 In IOP Conference Series: Materials Science and Engineering Synthesis of silver nanoparticles by silver salt reduction and its characterization. Vol. 60, No. 1, 012034

Ballarin B, Migmani A, Mogavero F, Gabbanini S and Morigi M 2015 Appl. Clay Sci. Hybrid material based on ZnAl hydrotalcite and silver nanoparticles for deodorant formulation. 114, 303–308.

Hussein M Z, Bahar F A and YAHYA H 2010 J.Iran.ChemSoc. Synthesis and characterization of hippurate-layered double hydroxide nanohybrid and investigation of its release property. Vol 7, 42-51
[27] Chen Y.F, Zhou S H., Li F and Chen Y W 2010 J. Mater. Sci. Synthesis and photoluminescence of Eu-doped Zn/Al layered double hydroxides. 45, 6417-6423.

[28] Silverstein R M and Bassler G C 1962 Journal of Chemical Education Spectrometric identification of organic compounds. 39(11), 546.

[29] Silverstein R M, Robert M and Bassler G C 1963 Wiley, New York Spectrometric Identification of Organic Compounds. N. Y, 84.

[30] Bragg W L 1913 Containing papers of a mathematical and physical character The structure of some crystals as indicated by their diffraction of X-rays. Proceedings of the Royal Society of London. Series A, 89(610), 248-277.

[31] AL-Rufaiie M, and AL-Zubaidi H Y 2018 Middle East Research Journal Biosynthesis of silver nanoparticles using (Carissa macrocarpa) leaves extract and study its antibacterial activity.3(46),1-21

[32] Tezuka S, Chitrakar R, Sonoda A, Ooi K. and Tomida T 2004 Green Chemistry Studies on selective adsorbents for oxo-anions. Nitrate ion-exchange properties of layered double hydroxides with different metal atoms. 6(2), 104-109.

[33] Xue X Y, Zhang S H and Zhang H G 2015 Am. J. Anal. Chem. Structures of LDHs Intercalated with Ammonia and the Thermal Stability for Poly (vinyl chloride). 6, 334–341.

[34] Hashim S. S, Abdul Kadhim A. A, & Matrood B. A 2014 Karbala Journal of Pharmaceutical Sciences Preparation of nanohybrid compound from the food preservative octyl gallate and studying some of its biological activities., 5(7), 277-289.