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Research Article

Study structure , optical properties of silver nanoparticles prepared chemically from Drop Casting method

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Abstract

In this research, a solution of silver nanoparticles was chemically prepared. The solutions were deposited on glass slides by pouring droplets into the air at a temperature of (333) k. The films of the nano-solution were examined by different techniques to study some of their structural optical properties. The results of the assays (XRD) showed sharp peaks indicating polycrystalline and cubic type with average crystal size of 38.9 nm for these prepared films. Atomic force microscopy (AFM) technique was used to measure the average grain size diameter and surface roughness's of the prepared films, as the obtained images give important information about the nanostructure and the results are close to (XRD) assay). It also used Fourier transform infrared spectroscopy (FT-IR) to know the molecular structure and bonds formed in the prepared films.

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Introduction

The chemical activity of nanomaterials increases and the reason is due to the large increase in their surface area with the presence of a large number of atoms on the faces of its outer surfaces, which always puts it at the top of the list of desired materials its use in various chemical applications [1]

Polyvalent silver forms different phases such as Ag_2O, AgO, Ag_3O_4, Ag_4O_3, and Ag_2O_3 through its interaction with oxygen, and the most important and stable phases that can be observed is Ag_2O [2] . It is a p-type semiconductor with an energy bandgap ranging from (1.2-3.4) eV. Ag_2O nanoparticles have gained special interest in nanomaterials research due to their unique properties. Therefore, it has been used in the manufacture of nano-devices, sensing, electrochemical conduction, catalysis, and optoelectronics It is also used in the manufacture of ointments, bio-imaging, mineral sunscreens and lotions, and drug delivery. A great potential for their use as antimicrobials has also been revealed. [3].

Experimental work

Various silver salts are used as a starting material in the synthesis of silver nanoparticles, where silver nitrate AgNO3 is the predominant raw material used in the synthesis of silver nanoparticles due to its high chemical stability and low cost. The chemical synthesis of Ag NPs is widely used [4], silver salt such as AgNO3, reducing agent such as sodium borohydride (NaBH4), and stabilizing or covering agent such as polyvinyl alcohol to control the size of nanoparticles and prevent their agglomeration [5].

The nano-solution was prepared by a simple chemical reaction, and the material used for the preparation was silver chloride) with the chemical formula (AgCl), one of its properties being that it exists in standard conditions in the form of a white crystalline solid. Equation (1) was used to find the weight of the substance to be dissolved, Wt:

$$M\left(\frac{mol}{l}\right) = \frac{W_{t(g)}}{M_{wt\left(\frac{g}{mol}\right)}} \times \frac{1000\,\frac{mol}{l}}{V(ml)} \qquad \text{---------------} \ (1) \qquad [6]$$

[6]Douglas A. Skoog, Donald M. West, F. James Holler, and Stanley R. Crouch, "Fundamentals of Analytical Chemistry, Ninth Edition", Ed. by Chris Simpson, (Mary Finch, Belmont, USA, 2014), pp 65-68.

Where M: molar concentration (mol/L), Wt: molecular weight to be dissolved (g) M_{wt}: molecular weight of the substance (g/mol), V: volume of the substance in which it was dissolved (ml), a sensitive electronic balance was used to measure the weight ratios. For the materials used for the preparation, the quickly formed, we notice a change in the color of the mixture, which indicates the formation of nanoparticles, as shown in Figure (1)

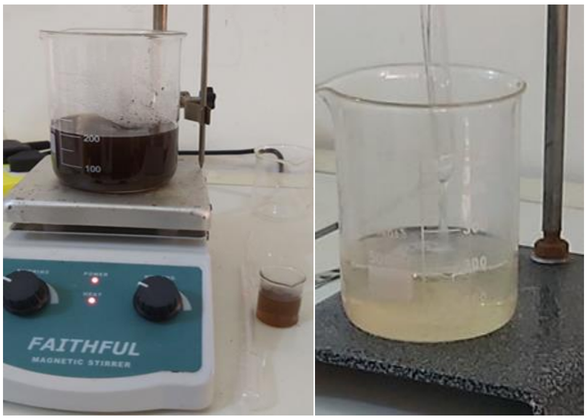


Figure 1: Stages of a chemical reaction to prepare nanoparticles

Thin Films Deposition

The nano-structured films of the prepared solution were deposited by chemical reaction method on solid bases of glass in the air by drop casting method. The Nano-solution was prepared by a chemical reaction method, and the material used for the preparation was silver chloride) with the chemical formula (AgCl), one of its properties that it exists in standard conditions in the form of a white crystalline solid. With a molecular weight of 143.321 g/mol), 14 g of silver chloride powder was taken and dissolved in (200 ml of distilled (deionized) water in a glass beaker at a molar concentration of (0.5 M) where it was not completely dissolved using the Hot Plate and magnetic heater. Magnetic Stirrer for an hour. Then he took 4g of sodium hydroxide with chemical formula (NaOH) and molecular weight (40g/mol) and dissolved it in (50ml) of high purity ethanol alcohol (99.9%), chemical formula (C2H5OH) in a glass beaker (Beaker). To obtain a solution with a concentration of 2M) by using the magnetic heater for five minutes and then quickly adding the concentration to the formed solution, we notice a change in the color of the mixture, which indicates the formation of nanoparticles. To preserve the precipitated nanoparticles, then distilled water was added until the final volume of the solution became (100ml), then the prepared solution was placed in a (Cleaner Digital Ultrasonic) device so that there would be no overlap or accumulation of the formed particles, as shown in Figure (2). This solution is to be deposited on glazes the method of pouring the droplet and studying its properties using different techniques,

Films

Figure (2) Stages of a chemical reaction to prepare nanoparticles

The thickness of the thin film is determined by two methods:

1- The weighing method

2- Maclean diffraction method

The weighted method was used in the research, as shown in the equation below: The thickness of the film is determined using an electronic balance with a sensitivity of four decimal places, by measuring the mass of the glass base prepared for deposition before the process of settling the material on it and after the deposition process. And finding the difference between the two masses, which represents the mass of the prepared membrane material, as well as measuring the area of the membrane, then the thickness of the prepared thin films (t) is calculated by applying the following relationship:

$$t = \frac{\Delta m}{A \cdot \rho_f} = \frac{(m_2 - m_1)}{A \cdot \rho_f} \qquad \text{--------------- (2)}$$

Where A: area in unit (m^2) of thin film , m_2: mass of glass substrate after deposition , m_1: mass of glass substrate before deposition, ρ_f :density of material(gm / cm3) .

It was found that the thickness of the film prepared by the chemical reaction method is 0.42 µm.

Figure (3) shows the X-ray diffraction of the film prepared using the chemical method, which was also deposited by the method of drop casting in the air on a glass substrate heated at °60C. Ultraviolet-Visible (UV-Vis) spectroscopy

A (UV-Vis spectroscopy) device was used, which was supplied by (Shimadzu) Japanese company, located in the BPC Analysis Center in Baghdad / Iraq,with a wavelength range (200-1100) UV-V is analysis can be used. To characterize nanomaterial's, and to determine many optical properties. It is used to measure the response of the sample (in terms of absorption, reflectance, and transmittance) by shining light within the ultraviolet and visible regions of the electromagnetic spectrum .

Discuss the results and conclusions

The results showed that the prepared material is silver oxide nanoparticles (Ag2ONPs). Through the examination, it was also found that the prepared membrane has a polycrystalline structure and is of a cubic type. From knowing the locations of the peaks, the presence of crystalline levels (110) (111), (111), (211) (220) was found. 311) belong to Ag2ONPs at diffraction angles (°27.83), (°31.6) (°32.19), (°46.27), (54.7°), (66.19°)(Θ2) and these values are in good agreement with the international standard card (ICDD) for X-ray diffraction (file:00-0413-1104).

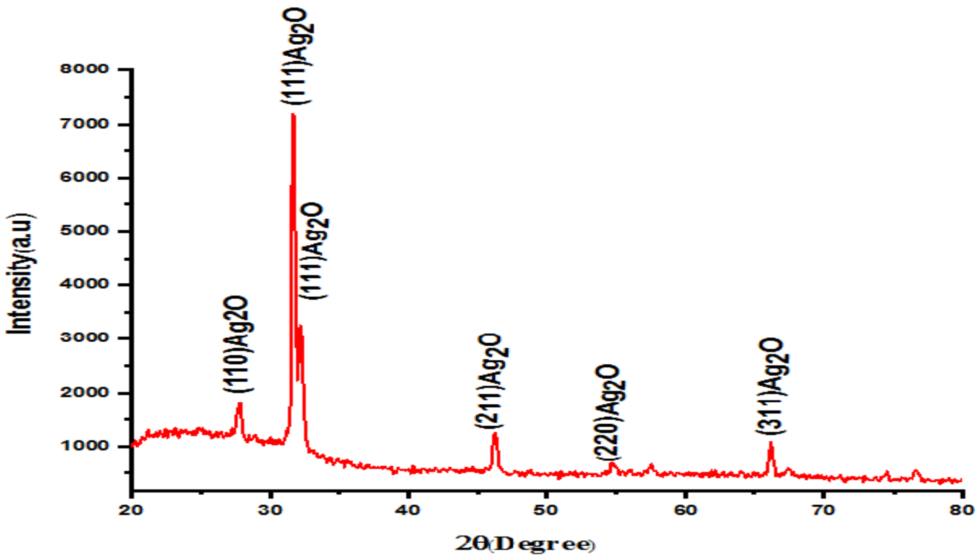
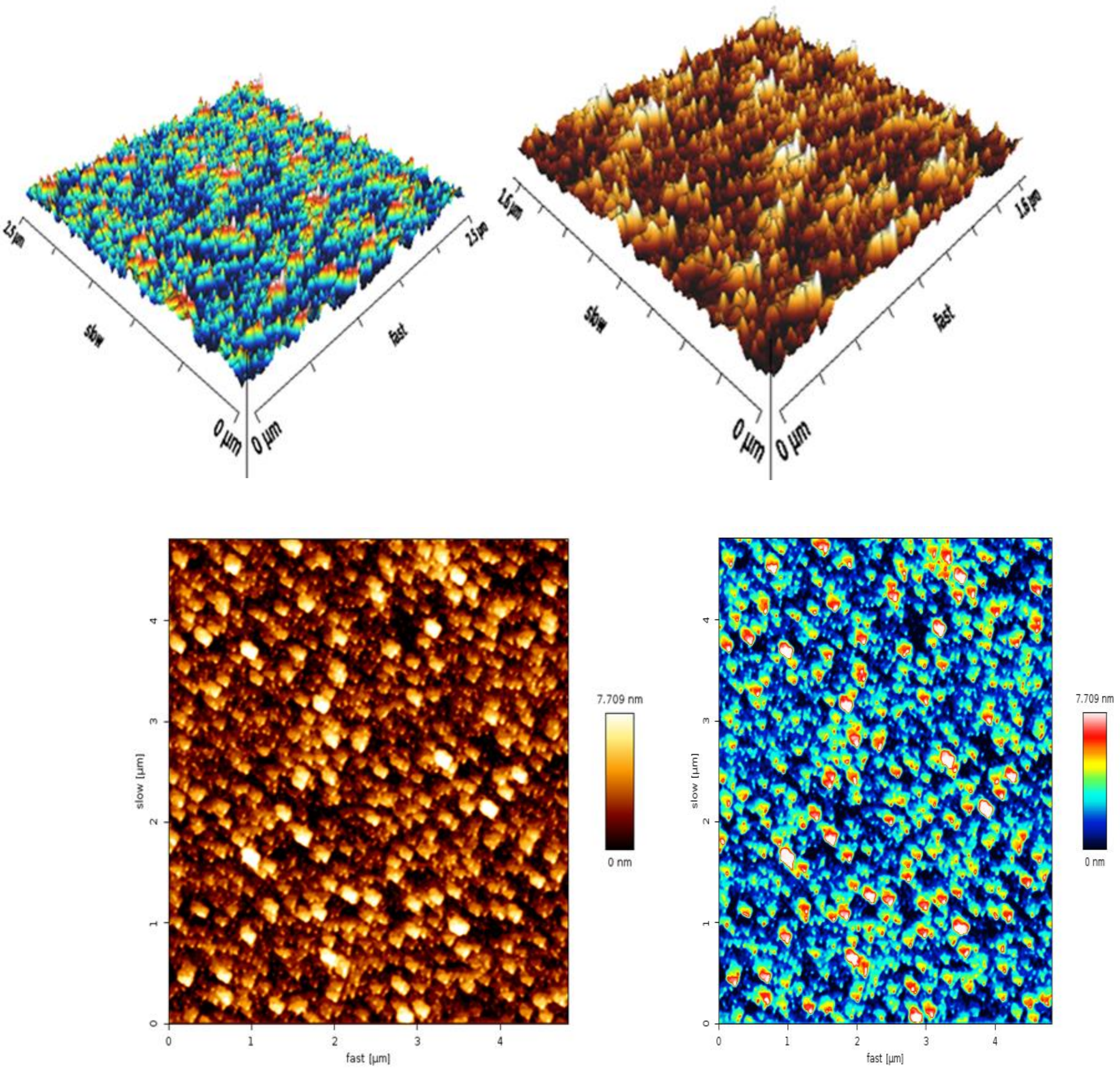


Figure (4) X-ray diffraction patterns of chemically prepared and precipitated silver oxide films

Table (3) the results obtained from the X-ray diffraction model of the thin films prepared by the chemical reaction method.

2θ (deg)	FWHM (deg)	D (nm)
27.83	0.147	55.160
31.60	0.246	33.389
32.19	0.246	33.439
46.27	0.344	24.968
54.77	0.393	22.634
66.19	0.147	64.014

The surfaces of the prepared films were scanned by atomic force microscopy (AFM) with dimensions of (2.5 x 2.5) cm^2,where Figure (6a) shows a two-dimensional image of the prepared thin film surfaces deposited on a glass substrate by the method of drop-casting by four drops, while Figure (6b) represents a three-dimensional image of the same thin film .



(a) (b)

Figure (5) (a) Two-dimensional (AFM) image of the prepared thin film b) Three-dimensional (AFM) image of the prepared thin film.

It is noticeable that the prepared nanoparticles were of different sizes within the nanoscale lined up horizontally, and the direction of the peaks towards the top, some of them are spherical or semi-spherical, and others have sharp peaks resembling needles, may be the reason is due to the presence of a larger number of reactants during the synthesis process but the values of the average particle size, the average surface roughness values, and the square root of the roughness are shown in Table (4) for the film prepared by the chemical reaction method.

Table (4) average grain size diameter, surface roughness average values, square root mean roughness square values resulting from (AFM) examination of films prepared by chemical reaction method

Thin films	Average Grain Size(nm)	Average Roughness (nm)	Root Mean Square(nm)
	36.8	1.113	1.114

The FT-IR spectra were determined as shown in Figure (3-5a) (3-5b) for the prepared membrane. The infrared spectrum showed a peak at -1 (550-500), which corresponds to the expansion range of silver oxide AgO [8]

Figure (7) shows the absorption spectrum as a function of the wavelength of nanoparticles prepared using the chemical method. The UV-Vis absorption spectrum showed maximum absorption at (250)nm. Surface Plasmon Resonance (SPR) is a physical process that can occur when plane-polarized light hits a thin metal film under total internal reflection conditions [9].

Table (5) the bonds formed by the NPsAg prepared by the chemical reaction method

Wave number Cm^{-1}	Type of Bonds	Species
3741	O-H stretching	phenols،Alcohols
3251	O-H	،H-bonded alcohols phenols
	N-H stretching	primary، secondary amines and amides
-2360 2381	C≡N stretching	Nitriles
2110	C≡O	Alkyne
1635	C=O	Amide
1426-1451-1516	N-H bend	primary، secondary amines and amides
509	C-Br stretching	alkyl halides

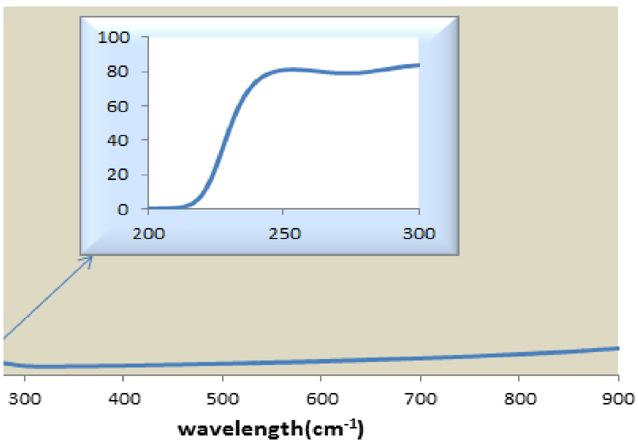


Figure (7) Absorbance spectrum of Ag2O NPs prepared by chemical reaction method

Estimation of the Optical Band Gap (Eg)
Figure 8 show the energy gap was determined by plotting the graphical relationship between photon energy (hν) versus $(\alpha h\nu)^{1/r}$ and by choosing the best linear tangent that cuts the energy axis when (0=α) where it represents a point
The intersection is an optical energy gap value, the energy gap for the permissible direct electronic transitions was found when (r = 1/2) for the prepared nanoparticles, while the photon energy was found and to find the absorption coefficient.

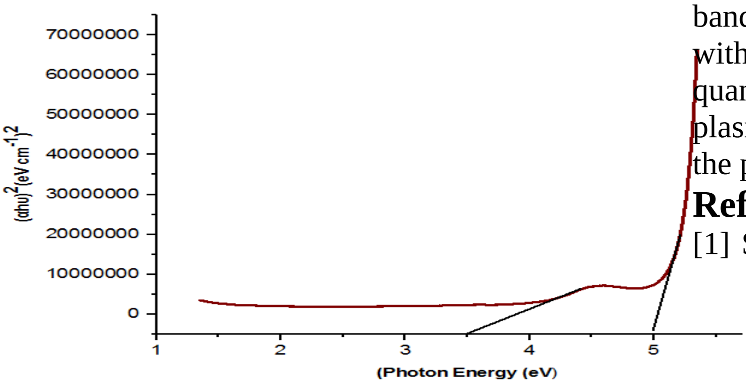


FIgure 8 Determination of the optical energy gap of the direct transmission of nanoparticles

As for the reason for the existence of two energy gaps, it may be attributed to the oscillation of the absorption edge, which is due to the structure of the energy band and the change of state density with the energy level [10]. May be The high value of the energy gap indicates that silver nanoparticles have dielectric behavior to induce polarization when strong radiation falls on the material. The absence of absorption bands in the visible region and the large energy gap confirm that the silver oxide nanoparticles are suitable for optical applications, and also indicate that the defect concentration in the oxide particles, Silver nanoparticles are very low [11].

The chemical reaction method is considered a convenient and fast way to produce nanoparticles, but it requires more reactants, and it also needs to undergo a particle cracking process so that a process of gathering together does not occur of developing particles and the occurrence of the so-called superposition phenomenon.

X-ray diffraction (XRD) technique showed that the prepared films possess a polycrystalline structure of cubic type and the average crystal size of the prepared particles is (38.9nm).

The results of atomic force microscopy (AFM) showed that the prepared film is more rough and relatively high, and the reason may be due to the use of many reactants during the chemical preparation process.

The absorbance spectrum (UV-vib) shows an increase in the amount of the optical bandgap compared to the macroscopic size with two energy gaps due to the effect of quantum confinement with the presence of plasmon resonance in the ultraviolet region of the prepared particles interaction.

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