DOI: https://dx.doi.org/10.21123/bsj.2023.7292

Synthesis and investigation of structural and optical properties of CdO: Ag nanoparticles of various concentrations

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Received 5/4/2022, Revised 4/9/2022, Accepted 5/9/2022, Published Online First 20/3/2023, Published 28/10/2023



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Abstract:

This study discussed the effects of doping with silver (Ag) on the optical and structural properties of CdO nanoparticles at different concentrations 0, 1, 2, 3, 4, 5 wt% prepared by the precipitation method. The materials were annealed at 550°C for 1 h. The structural, topographical, and optical properties were diagnosed by X-ray diffraction analysis, atomic force instrument, and visible and ultraviolet spectrometers. The results show that the average diameter of the grains depends on the percentage of added silver to the material, as the diameter decreased from 88.8 to 59.7 nm, and it was found that the roughness increased from 5.56 to 26.5. When studying the optical properties, it was noted that the energy gap was 2.53ev for the pure sample and decreased to 2.38 When 5% of silver concentration was added to cadmium oxide

Keywords: AFM, CdO Nanoparticles Doped Ag, FTIR, Method of Precipitation, UV-VIS, SEM.

Introduction:

The Interest in preparing and studying the properties of nanomaterials has increased in recent years due to their distinctive chemical and physical properties resulting from the effect of the size of the particles of this materials¹. Nanotechnology is prominent in science and research as the wide variety of methods for preparing nanoparticles². Many successful types of research in this issue are considered a challenge to think in different fields such as medicine, science, technology, etc³. Metal oxide semiconductors have attracted increasing attention because of having several intriguing properties such as large band gap, low electrical resistivity and high transmission in the visible and infrared region^{4,5}. Among them, cadmium oxide (CdO) nanoparticles are a preferred material for use as n-type carriers due to their high conductivity and optical transmission in the sunlight range⁶. In addition, CDO nanoparticles play an essential role in a wide range of applications, including optical communications⁷, gas sensors⁸, solar cells⁹.and thin film resistors ¹⁰. Also, the CdO nanoparticle was prepared with different doping elements such as aluminium 11 , tin 12 , gallium 13 , indium 14 , zinc 15 , and silver 16 for various applications. However, silver (Ag) metal has been commonly used because

of its numerous good characteristics and notable chemical reactivity in solutions¹⁷. Ag is one of the active metals that the doping process might quickly oxidize. Therefore, it is exciting to understand the role of silver in a transparent CdO matrix. Recently, scientists have worked on pure and Ag-doped CdO nanostructures through various techniques ^{16,18}. pure CdO, and many methods prepared Ag-doped CdO. nanoparticals; the precipitation technique is a simple method used in this study to prepare because of its low cost and ease of processing, on the other hand, are two of its advantages. During the synthesis process, the amount and rate of ammonia solution addition were matched correctly to obtain the necessary CdO and Cdo: Ag nanostructures. XRD, AFM, FTIR, and UV-vis spectroscopy were used to investigate the structural and optical properties of the produced nanoparticles.

This study aims to prepare nanomaterial pure CdO and doped with different from concentrations of Ag by precipitation method because of the importance of this material and its use in broad applications such as sensitizing for gases, data storage, solar cell uses, and antibacterial applications.

Materials and Procedures:

Materials and Procedu Materials:

Cd(CH₃COO)₂.2H₂O cadmium acetate dehydrates, AgNO₃ silver nitrate, NH3 ammonia as precipitator, Supplier Company CDH-Indian, were used to prepare CdO: Ag Nanoparticle.

Preparation of CdO:Ag Nanoparticle

Pure Cadmium oxide with Ag nanoparticles was prepared chemically by precipitation method as follows:

13.326g of cadmium acetate dehydrate Cd(CH₃COO)₂.2H₂O was dissolved in 100ml of distilled water using a magnetic stirrer device 1500 rpm to produce a 0.5M where the molarity is determined by relation. 1 ¹⁹.

 $M=(W/m.wt)\times(1000/V)$

where V: is The volume of distilled water, W: is the weight of cadmium acetate, and *m. wt* is Molecular weight. With continuous stirring, the ammonia solution NH₃ was added to the solution above dropwise until the pH value reached 8. The white precipitate has developed, and the mixing will continue for other 24 hours. Then, using a centrifuge, it was filtered and rinsed four times with distilled water and ethanol to remove any remaining contaminants. After that, the solution was dried for 1 hour in an oven at 100 °C, the resulting powder was calcined for 1 hour at 550 °C, and finally, we opened the yellow powder in colour.

The same method used to make pure CdO nanoparticles was used to make Ag Doped CdO nanoparticles. Just before the precipitating agent (ammonia) was added, the calculated amount of dopant silver nitrate AgNO $_3$ was added to the solution in terms of 1, 2, 3, 4, 5 wt% concentration and the reaction was carried out at the same temperature. The molarity of Ag was found using the relation 2 20

%dopants=*M* (AgNO3) *molarity* /(*M* (AgNO3)+*M* (Cd(CH3COO)2.2H2O))2

where M (AgNO3) *molarity*: The molar concentration doped AgNO3 is(X), % dopants: doped concentrations 1, 2, 3, 4 and 5 %wt, M (Cd(CH3COO)2.2H2O)= 0.5 M

The colour of the solution changed to a light grey after annealing at 550°C for 1 hour in the presence of air, with the intensity of the colour varying with the concentration.

Characterization

Using $CuK\alpha$ =0.154 nm, the X-Ray diffraction (XRD) pattern of CdO: Ag nanoparticles was acquired using a Siemens model D500 system. Scanning electron microscopy (SEM) ZEISS model SigmaVP with a magnification of 50.00KX and (EDX) Oxford instruments UK were used to analyze the particle morphology and size.

Ultraviolet diffusion was used to investigate optical characteristics. Within the wavelength range of 350nm-800nm, Reflection Spectroscopy with UV-VIS (Shimadzu) was performed. The AFM revealed the morphology of the CdO: Ag NPs, and the cumulative distribution plot revealed the average grain size.

P-ISSN: 2078-8665

E-ISSN: 2411-7986

Result and Discussion: XRD analysis

Figure .1 displays the (XRD) pattern of Pure CdO nanoparticle doped with different concentrations 1, 2, 3, 4, and 5 % wt of Ag, prepared using the precipitation method.

As shown in the Figure, all produced particles had a polycrystalline reflection pattern, with the dominant reflection (111) plane at the angle 2θ = (33.2° for CdO to 32.9 for 5% Ag concentration), and the structure is FCC cubic, which is consistent well with the JCPDS card no. 05-0640 of CdO 21 as shown in Table.1. The diffraction patterns of all the samples showed strong and high-intensity diffraction peaks corresponding to the only present CdO phase, and the sharp peaks showed that the particles had an excellent crystalline structure and this agreement with the study of Salem et al. 22, and the addition of Ag causes slight off set-shifting in the diffraction angle to the less 20 with increasing in peak intensity (This shift in the peak is due to several reasons, including, 1- The link between the host and the doped particle, 2- The change in the size of the host particle, 3. The difference in binding energy and as a result of the change in mechanical properties). The angle deviation occurs due to the deformation of the lattice caused by the doping and thus caused defects in the crystal system that led to the deflection of the angle, which in turn affected the distance between the atomic levels d, where we notice an increase in d with the rise in the concentration of Ag in the material²³, based on Braques' law.

 $n\lambda=2d \sin(\Theta)$. As shown in Table. 1 ¹⁸, and this agreement with the study of Laith et al. ²⁴.

The crystallite size D was calculated from the Scherer relation 3 ²⁵.

 $D=K\lambda/(\beta \cos \theta) \dots 3$

where K is a variable that fluctuates depending on the crystallite's natural form, λ is the X-ray wavelength; λ = 1.5418 Å, β is the full width at half maximum of intensity (FWHM), and θ is Bragg's angle. It was shown that the crystallite size of produced CdO was 22.629 nm, while with add of Ag, the crystallite size decreased to 14.36 nm by increasing the doping from 0 wt% to 5 wt%; this could be due to the decrease in the concentration of

CdO with the addition of Ag this agreement with Kumar et al. ²⁶. However, no peak corresponding to Ag was observed. This may be because of the

deficient concentration of Ag or the uniform distribution of Ag into CdO. This is in agreement with Kumar et al. ²⁷.

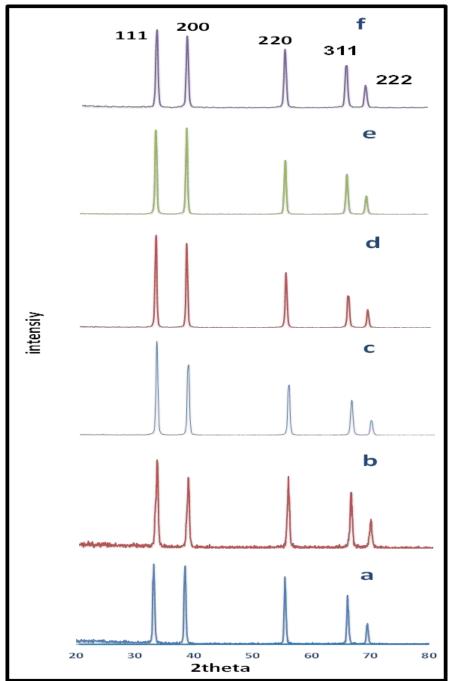


Figure 1. XRD spectrum of a: pure CdO and doped with (b: 1, c: 2, d: 3, e: 4, and f: 5) wt. % Ag nanoparticles

The Dislocation density δ is a measurement of the number of dislocations per unit volume of crystalline material. It is determined based on a relationship 4 28 .

From Table. 1, an increase in the dislocation density δ is observed, with the crystal

size decreasing due to the rise of crystal defects in the lattice with the addition of Ag; This kind of change is likely to result from recrystallization processes in nature of polycrystalline ²⁴ as well, the size of Ag ions (which have been placed into a lattice) is greater than Cd ions (ionic radii for Ag= 129 picometer, and Cd=109 picometer)

	Table 1. structural	parameters of	pure CdO and	CdO doped	1, 2, 3, 4, 5 wt% Ag
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Table 1. structural p	arame	eters of pure Co	iO and	CaO aopea	1, 2, 3, 4,	5 Wt% Ag	
Ag concentration wt%	Hkl	2theta ASTM	2theta	$d_{hkl}(A^{\circ})$	FWHM	D(nm)	$\delta = 1/D^2$
	111	33.03	33.2	2.69657	0.3994	21.6	0.00214
	200	38.31	38.52	2.3355	0.3853	21.5	0.00216
0%	220	55.308	55.48	1.65505	0.3347	24.787	0.00163
						Average of	
						D=22.629nm	
	111		33.17	2.69894	0.4982	16.65	0.0036
	200		38.45	2.33959	0.4849	17.11	0.00341
1%	220		55.42	1.6567	0.4636	17.85	0.00314
						Average of	
						D=17.2nm	
	111		33.03	2.71006	0.538	15.4	0.00422
	200		38.4	2.34252	0.564	17.87	0.00313
2%	220		55.4	1.65725	0.54	15.57	0.00413
						Average of	
						D=16.28nm	
	111		33	2.71246	0.52	15.9	0.00396
	200		38.25	2.35136	0.515	16.1	0.00386
3%	220		55.2	1.66278	0.513	16.17	0.00382
						Average of	
						D=16.05nm	
	111		33	2.71246	0.53	15.65	0.00413
	200		38.3	2.3484	0.526	15.73	0.004
4%	220		55.3	1.66001	0.538	15.42	0.0042
						Average of	
						D=15.6nm	
	111		32.9	2.72047	0.583	14.2	0.00496
	200		38.2	2.35432	0.574	14.476	0.00477
5%	220		55.2	1.66278	0.576	14.4	0.00478
						Average of	
						D=14.36nm	

Atomic Force Microscopy (AFM):

Figure. 2. a, b, c, d, e, and f shows (AFM) images of 3D surface morphology of pure CdO and CdO doped with different concentrations 1, 2, 3, 4 and 5 % wt of Ag, prepared by the chemical method. The images show that all samples have spherical and poly shapes. The decrease is clear from Fig. 2 and Table. 2 in the average diameter of the granules with an increase in the concentration of the grain at the concentration of 3%, where we notice an increase in the diameter, and this is in agreement with the results obtained from the XRD analysis, where the size decreased by increasing the concentration, but the average grain size obtained

from AFM is larger than the crystal size measured by XRD, the reason is that the AFM measures the average size of the surface grains, which are larger than the size of the inner crystals evaluated by XRD. The Table also shows that the surface roughness and the mean square root vary between increase and decrease, which could be attributable to the significant difference in particle diameters²⁸, where the results showed that the highest roughness was at the concentration of 3 and 5%. At the same time, their average grain size does not represent the highest value because the roughness is not related to the size of the grains²⁹.

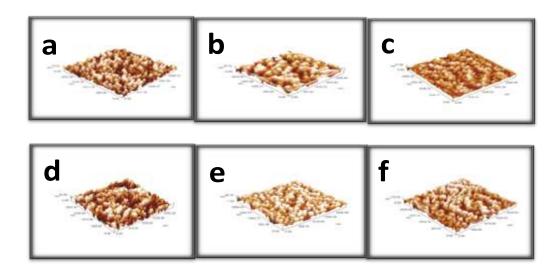


Figure 2. AFM images describing the surface morphology for nanoparticles of a: pure CdO and CdO doped with (b: 1, c: 2, d: 3, e: 4, and f: 5) wt. % Ag

Table 2. Average of diameter and roughness for Cdo: Ag Nps.

Type	Average	Average	of	rms roughness
	diameter (nm)	roughness		(nm)
		(nm)		
Pure	88.84	5.56		6.42
1%	81.21	6.56		7.79
2%	67.04	5.69		6.75
3%	69.7	11.3		13
4%	68.3	9.57		11.5
5%	59.75	26.5		31.1

Results of SEM.

Figure. 3, shows SEM images of nanoparticles pure CdO and doped with various concentrations. 1, 2, 3, 4, and 5 % wt of Ag respectively prepared by chemical precipitation method. The figure shows that all the samples have a spherical and poly-shaped structure, with a pyramid shape prevailing in the concentration of 5% and also leading to forming an agglomerate of crystals. The reason for The agglomeration of

nanoparticles results from the adhesion of the particles to each other by weak forces (Vander Waals forces) that lead to (sub-) infinitesimal entities.

P-ISSN: 2078-8665

E-ISSN: 2411-7986

In contrast, the agglomeration nanoparticles is due to the formation of covalent or metallic bonds that cannot be easily disrupted. To prevent the agglomeration of nanoparticles, some plant extracts are added. These plant extracts act as a reducing agent and a covering agent³⁰. When Ag is added to CdO, The grain size increases with increasing Ag concentration, and The intergranular pores decrease, which increases the agglomeration of the particles high; agglomeration was observed, which is related to the presence of the ionic impurity in the dopant molecules even after calcination.31. Nucleation and fusion are the product of the difference in grain size and shape that affect grain growth by changing grain boundaries³²

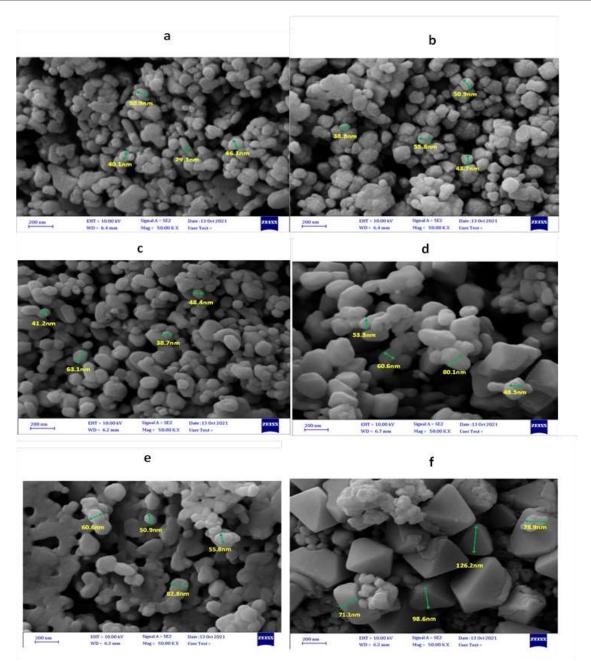


Figure 3. SEM images of (a) pure CdO (b) CdO:Ag 1% Ag (c) CdO:Ag 2%Ag(d) CdO:Ag 3% Ag \in CdO:Ag 4% Ag(f) CdO:Ag 5% Ag .

Energy-dispersive X-ray analysis (EDX)

Energy-dispersive X-ray analysis (EDX) spectra explain the composition of the components in the manufactured samples. The EDX results show that the fabricated samples are CdO and CdO: Ag nanoparticles and that the silver ions have been

introduced effectively into the lattice of CdO. The result of EDX are displayed in Fig.4. The quantity of elements growth in the sample is shown in Table. 3, which includes Cadmium Cd, C, Ag, and oxygen O. Ag acts as an impurity in cadmium oxide, affecting the sample's absorbance.

P-ISSN: 2078-8665

E-ISSN: 2411-7986

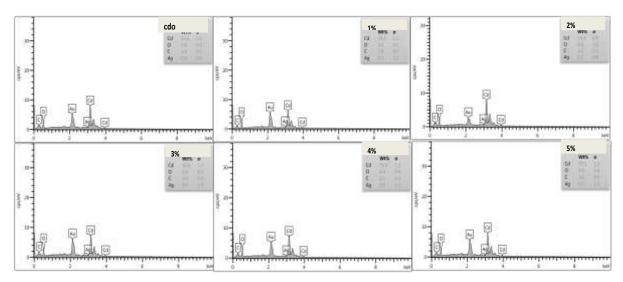


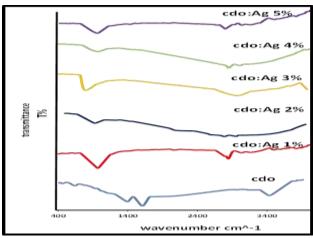
Figure 4. EDX image of pure CdO and CdO doped with 1, 2, 3, 4, 5 wt% Ag.

Table 3. Shows the proportion of the composition of the prepared materials.

g
Vt%
.2
.2
.5
.5
.7

Fourier Transform Infrared analysis (FTIR)

FTIR spectrum analysis can be used to determine the existence of functional groups. The wavelength region of 4000–400 cm⁻¹ for the synthesized Pure CdO nanoparticle and doped with Ag is presented in Fig.5. Stretching vibrations of structural OH groups have peaks that are both powerful and acute in the range of wavenumber between 3436-3448 cm⁻¹. This is in agreement with the study of Hongju et.al³³.and Ranjith Kumar et al. 34. The weak peak between 2933-2999 cm⁻¹ can be associated with C-H. The tiny band between 1625-1656 cm⁻¹ is attributed to free H₂O molecules created during the tabletting process. The absorption peaks at 1400-1600 cm⁻¹, according to the concentration of Ag, are assigned to C-O. This is in agreement with the study of Tripathi et al. 35. The peaks observed around 420-450 cm⁻¹ are connected to CdO. This is in agreement with the findings of Al-Duyyan et al. ³⁶, which emphasizes the creation of a pure catalyst. The figure also indicates the disappearance of peaks in the range about 3400 and 1600 cm⁻¹, which is due to the presence of water in the sample. This means that most of the anaesthetized samples' water has evaporated, and the presence of silver likely played a role. All peaks are shown in Table. 4.



P-ISSN: 2078-8665

E-ISSN: 2411-7986

Figure 5. FTIR of pure CdO and CdOdoped 1, 2, 3, 4, 5 % Ag.

Table 4. FTIR bonds of pure CdO and CdO doped 1, 2, 3, 4, 5 wt% Ag concentration.

Band type	Wavenumber(cm-1)
ОН	3440-3448
С-Н	2958-2999
H2O	1625-1656
C-O	1400-1600
CdO	420-450

Optical properties

The UV-VIS absorption spectrum was used to evaluate the optical characteristics of the produced CdO: Ag nanoparticle. The spectra were drawn over a wavelength range of 350 to 800 nm. Fig .6, displays Pure CdO and CdO doped with Ag nanoparticles absorption range. The absorbance increase, and the absorption peak creep to the right with increasing silver concentration (In AgNPs, the conduction band and valence band lie very close to each other in which electrons move freely. These free electrons give rise to a surface Plasmon

resonance (SPR) absorption band, occurring due to the collective oscillation of electrons of silver Nanoparticles in resonance with the light wave. The absorption of AgNPs depends on the particle size, dielectric medium, and chemical surroundings.), which leads to a decrease in the energy gap with increasing concentration of CdO, and this agreement with the study made by Kumar et al. ²⁶. These alterations can be related to the creation of donor levels within the energy gap and near the conduction band, which resulted in the absorption of low-energy photons and, as a result, a significant rise in the absorbance value. And this also led to the movement of the absorption edge. These levels are increased by increasing deflection rates, thereby reducing the width of the gap³⁷. Energy gab for Pure CdO, Ag doped CdO nanoparticle was calculated using the relation (Eg= $1240/\lambda$) Planck's law³⁸ and listed in Table.5.

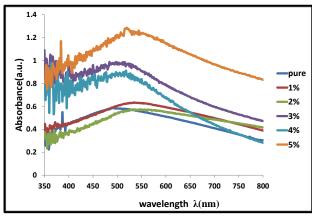


Figure 6. Absorption spectra of pure CdO and CdOdoped 1, 2, 3, 4, 5 wt% Ag concentration.

Table 5. shows the Energy band gap of pure CdO and CdOdoped 1, 2, 3, 4, 5 wt% Ag c concentration.

1.			
Wavelength	The	band	gap
(nm)	(eV)		
490	2.53		
535	2.32		
533	2.33		
497	2.49		
518	2.39		
520	2.38		
	Wavelength (nm) 490 535 533 497 518	Wavelength (nm) The (eV) 490 2.53 535 2.32 533 2.33 497 2.49 518 2.39	Wavelength (nm) The (eV) band (eV) 490 2.53 2.32 535 2.32 2.33 497 2.49 2.39

The electron capture instances (the aperture) between the valance band edge and the conduction band edge of Cadmium Oxide are generated when CdO is doped, resulting in better visible light absorption.

The transmission of charge, which may be viewed as an alternative to the excitation of an electron from orbit d of metal ions ³⁹, is responsible for increasing visible absorption. Increased

absorbance might be due to higher impurity levels inside the energy gap⁴⁰.

Conclusion:

In this study, the chemical precipitation method has successfully prepared nanoparticles of pure CdO and doped Cdo at different concentrations 1, 2, 3, 4 and 5 % wt of Ag. The UV, FTIR, XRD, AFM, and SEM analyses show that all samples' crystal structure is good, and Cadmium oxide has a cubic face-centred structure (FCC). The average particle sizes are approximately within the range of 22.629 - 14.36 nm depending on the concentration additive of Ag. It is also noted that the surface morphology of the samples is a spherical and polymorphic crystalline conglomerate. The surface roughness increases from 5.56 - 26.5 nm with increasing Ag concentration. The measurements assert that the optical energy gap decreases from 2.53 -2.32 Volts with the increase of the silver concentration. In contrast, the absorbance increases with the increase in the concentration of Ag. The results show that the manufactured samples can be used in various electrical applications, such as gas sensors, and various medical applications, such as antibacterial and anticancer therapy.

Authors' declaration:

- Conflicts of Interest: None.
- We hereby confirm that all the Figures and Tables in the manuscript are mine ours. Besides, the Figures and images, which are not mine ours, have been given the permission for republication attached with the manuscript.
- Ethical Clearance: The project was approved by the local ethical committee in University of Baghdad.

Author contributions

Nada K.Abbas and Suaad Ali contributed to the design and implementation of the research, to the analysis of the results, and to the writing of the manuscript

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تحضير ودراسة الخصائص التركيبية والبصرية لاوكسيد الكادميوم المطعم بالفضة وبتراكيز مختلفة سعاد على محمد ندى خضير عباس

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لخلاصة

تمت في هذه الدراسة مناقشة تأثير التطعيم بالفضة على الخواص التركيبية والبصرية لجسيمات اوكسيد الكادميوم النانوية ولتراكيز مختلفة (0,1,2,3,4,5)% والمحضرة بطريقة الترسيب. تم تلدين المواد عند درجة حرارة 550 درجة مئوية لمدة ساعة واحدة. تم تشخيص الخصائص التركيبية والطبوغرافية والبصرية من خلال تحليل حيود الأشعة السينية، ومجهر القوة الذرية، ومقاييس الطيف المرئية والأشعة فوق البنفسجية. أظهرت النتائج أن متوسط قطر الحبوب يعتمد على نسبة الفضة المضافة للمادة، حيث انخفض القطر من 88.8 إلى 59.7 نانومتر، ووجد أن الخشونة زادت من 55.6 إلى 65.5. عند دراسة الخواص الضوئية لوحظ أن فجوة الطاقة كانت 2.53 فولط للعينة النقية وانخفضت إلى 2.38 عند إضافة 5٪ من تركيز الفضة إلى أكسيد الكادميوم.

الكلمات المفتاحية: مجهر القوة الذرية، جسيمات اوكسيد الكادميوم النانوية المطعمة بالفضة، تحليل فورييه للأشعة تحت الحمراء، طريقة الترسيب، الاشعة فوق البنفسجية-المرئية، المجهر الالكتروني الماسح.