



Effect of Using Different Preparation Methods on the Properties of CdS Nanoparticles

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Abstract

This study aims to prepare cadmium sulfide nanoparticles in two methods, which are the chemical method and the biological method using two types of extracts, green tea, and starch. The prepared nanoparticles were characterized by various techniques such as X-ray diffraction (XRD), scanning electron microscope (SEM), and Energy Dispersive X-ray Spectroscopy (EDX). The synthesis of crystalline CdS nanoparticles was confirmed by various analytical techniques such as X-ray diffraction (XRD) and the pattern reveals cubic and hexagonal structures. The average crystal size for the methods respectively is 11.5, 15.9, and 5.67 nm, which have a spherical shape for nanoparticles CdS by scanning electron microscopy (SEM). The peaks of cadmium and sulfur emerged in the assay (EDX). The energy gap of the CdS has increased than the energy gap of the bulk CdS, the results were 2.63, 2.88, and 3.06 eV, respectively. This is an evidence of obtaining a nano-material for CDS by these two methods.

1. Introduction

Nanotechnology has attracted a lot of attention recently, particularly in the research and industrial communities. The capacity to design, produce, and manipulate particular nanostructured materials is key to nanotechnology's future potential. Nanomaterials are now used in practically every industry, from food and medication formulations to cosmetics and sporting gear, and from computer technology to the energy sector. The combination of semiconductor science with nanotechnology, for example, has resulted in a wide range of applications, enhancing both sciences. Nanomaterials can be grown from the bottom up or from the top down, and the growth technique should be decided according to the desired qualities of the product NPs.

The nanomaterials of cadmium-based II–VI semiconducting materials can be prepared in a variety of ways out of which vapor–liquid–solid (VLS) has been found a popular approach that can be implemented in a number of experimental routes [1]. Physical and chemical procedures are often used to synthesize NPs, but physical techniques are costly; chemical approaches, on the other hand, are harmful to the environment and living beings [2, 3, 4]. Chemical synthesis with liquid-phase precursors is a common method for making nanomaterials.

It entails a chemical reaction of the precursor species that results in nucleation, followed by the creation of nanomaterials with a wide particle size dispersion and shape. To obtain a narrow size distribution, superior crystal quality, and the appropriate shape of the output nanomaterials, adequate supersaturation, careful selection

of precursors, and species concentration, synthesis parameters and chemical reaction kinetics play a significant role [1]. Due to many restrictions and the environmental effect of physical and chemical technologies, the use of "green" and ecologically friendly processes in nanomaterial synthesis is becoming more common [5]. Green science pathways to prepare nanomaterials are less expensive and do not require the use of harmful chemicals, high pressures or extreme temperatures [6,7]. Cadmium sulfide (CdS) is a cadmium-sulfur binary chemical. It has a hexagonal wurtzite bulk structure with a melting point of 1600 °C and is insoluble in water, but soluble in dilute mineral acids. It's a semiconductor of the n-type.

Due to sulfur vacancies, it exhibits a deep donor level and conductivity. The bulk structure of CdS has a band gap energy of 2.42 eV at ambient temperature [8] and this energy value demonstrates that it is one of the most capable candidates for detecting visible light [9]. Though CdS has a variety of uses in bulk form, it possesses remarkable physical, chemical, electrical, optical, and transport capabilities when reduced to the nanoscale. XRD, transmission electron microscopy TEM or SEM, and UV-visible spectroscopy methods were used to characterize the produced material. Due to quantum confinement, these nanoparticles displayed a significant energy gap in our study.

CdS is one of the most proficient nanomaterials for solar cells [10] and there are numerous applications of this material in the field of optoelectronics, photonics, photovoltaic, and photovoltaics [11]. CdS is also used as a pigment in paints and in engineered plastic for good thermal stability [12]. In photonics, CdS is employed to make NCs [13].

A little modification in size and morphology of CdS NPs leads to its increased photocatalytic activity [14]. High-sensitivity photodetector materials have been observed from CdS NTs and NWs, proving their potential as photodetectors in technological applications with high capability at low-cost [15]. Due to high value of surface to volume ratio, CdS nanomaterial's can be used to design photocells [16], light-emitting diodes (LEDs) [17, 18], lasers [19], address decoders [20] sensors [21], optical biosensors [22], optical switches [23], hydrogen production [24], and water purification [25]. All of these striking properties points to potentials of CdS nanomaterials for ongoing and future applications.

In this research, cadmium sulfide nanoparticles prepared using two different methods: chemical method, and the biological method by using two types of extracts, the first is green tea extract, which is a reducing agent and coating for nanoparticles, while the second extract is starch as a reducing and covering agent, in order to study the effect of the preparation method on the properties of the prepared material.

2. Materials and Methods

2.1. Materials

$\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ (company QualiKems), deionized distilled water, $\text{CH}_4\text{N}_2\text{S}$ thiourea (company scharlan), NaOH, dry green tea leaves (starch Supplier local in Iraq), $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ (Thomas Baker), NH_3 (Thomas Baker). $\text{C}_6\text{H}_{10}\text{O}_5$ (starch Supplier local in Iraq), $\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$ (Supplier Company Qualikems), Na_2S (Thomas Baker).

2.2. Preparation of CdS Nanoparticles by Chemical and Biological Methods

2.2.1. Chemical Method

A solution of cadmium acetate $\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$ was prepared at a concentration of 0.1 M and put on the magnetic stirrer, then NaOH (NaOH is sluble ion compound containing the hydroxide ion OH^-), when dissolved in water or neutralized with acid it liberates substantial heat. H_2O is none electrolyte so H^+ and OH^- cannot exist in large concentration and they combine to form H_2O was added to it by droplet until the pH reached PH (8-9). Then thiourea ($\text{CH}_4\text{N}_2\text{S}$), a source of the sulfur root, was added at a concentration of 0.1 M. The materials are mixed well until the reaction is completed by placing them on the magnetic stirrer for two hours, after which the material is filtered and washed well with deionized water and then with ethanol and left to dry at room temperature. The method of work is shown in Figure (1).

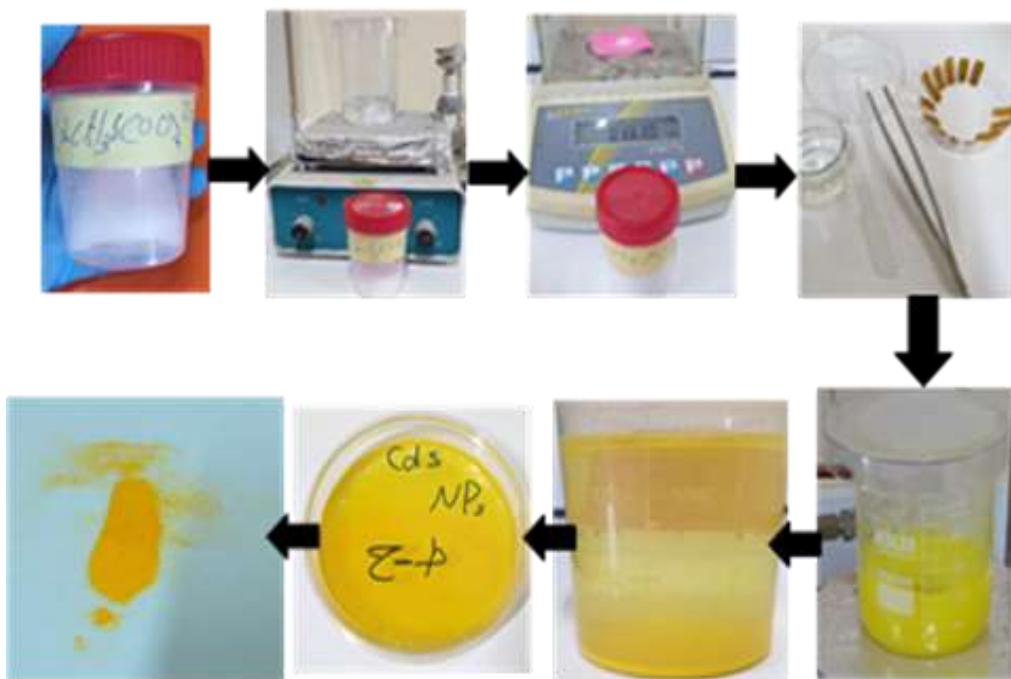


Figure (1). The method of preparing cadmium sulfide by chemical precipitation method.

2.2.2. Biological Synthesis Using Green Tea

Using tea- leaf extract as particle stabilizing agents. Tea leaves contain polyphenols, amino acids, caffeine, vitamins, minerals and antioxidants. It is anticipated that the chemical constituents of tea- leaf play a key role in the formation of CdS NPs. 20 gm of green tea leaves were taken, wash well with deionized water, and then was dry well away from heat and sunlight (at room temperature), then grind and added to 200 ml of deionized water and heat up for 30 minutes, then it is filtered and cooled completely at room temperature. In the first beaker added 3 ml of green tea extract and 0.5 M of $\text{CH}_4\text{N}_2\text{S}$ thiourea with 22 ml of deionized water and put on the magnetic stirrer for half an hour in, the molarity was calculated from Equation (1) [26]:

$$M = \frac{W}{M.Wt} \times \frac{1000}{V} \dots \dots \dots (1)$$

Where V: The volume of deionized water, W: the weight, M. wt: Molecular weight.

In another beaker, 3 ml of green tea extract and 0.5 M of $3\text{CdSO}_4 \cdot 8\text{H}_2\text{O}$ were added to 22 ml of deionized water and placed on a magnetic stirrer for half an hour with adding drops of ammonia NH_3 and the pH was measured to be between 8-9. Then the first bowl is added to the second bowl and 50 ml of deionized water is added and placed on the magnetic stirrer for two hours, then washed, filtered, and washed well with deionized water and ethanol and left to dry at room temperature. The method of work is shown in Figure (2).

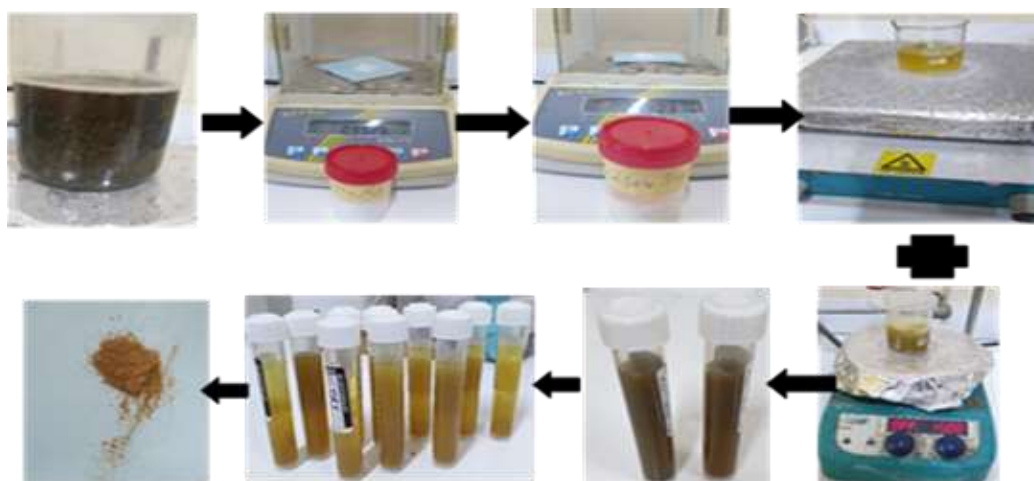


Figure (2). The method of preparing cadmium sulfide by the biological precipitation method using green tea.

2.2.3 Biological synthesis using starch

The biological method using starch (chemical formula is $C_6H_{10}O_5$) was used to prepare CdS NPs in an environmentally friendly, inexpensive way and with minimal risks of chemicals. 2 grams were weighed with a sensitive balance and added to it 400 ml of deionized water and placed on the magnetic stirrer then 0.1 M of $Cd(NO_3)_2$ concentration was prepared and the molarity was calculated from equation (1) and added to the starch solution and placed on the magnetic stirrer for 15 minutes and then added drops of NaOH to measure PH (8-9), Na_2S was added at a concentration of 0.1 M by distillation, the color of the solution change will be noticed, It turns yellow and is placed on the magnetic stirrer for two hours. After that the solution was filtration and wash by the centrifuge device for 30 min, the number of cycles is 4000 rpm to separate the unwanted material, with water and ethanol at room temperature. The method of work is shown in Figure (3).



Figure (3). The method of preparing cadmium sulfide by biological precipitation using starch.

2.3. Characterization

A Siemens D500 system was used to record the X-ray diffraction (XRD) pattern of pure CdS and doped with Sn particles using; $CuK\alpha$, $\lambda = 0.154$ nm. Scanning electron microscopy (SEM) ZEISS\SigmaVP model with the magnification of 50.00KX\Oxford Instruments UK was used to investigate particle shape and size (EDS) and mapping: oxford instruments. UK. UV diffusion was used to examine the optical characteristics. UV-VIS (Shimadzu) was used to perform reflectance spectroscopy in the wavelength range of 300 nm to 800 nm.

3. Results and Discussion

3.1. X-Ray Diffraction

To examine the structural properties of pure CdS nanoparticles prepared by chemical precipitation at room temperature. X-ray diffraction analysis of CdS NPs was performed from 20° to 80°. Figure (4) shows the compatibility between the XRD structure of the CdS NPs and that of JCDD Standard No. (96-900-8863), which has a polycrystalline structure with a hexagonal phase. The most important feature of the observed model is $2\theta = 26.7845^\circ$, assigned to (002) reflection, and other prominent features are noted in Table (1). Half Full-Width Max (FWHM) as half the height of the wave was calculated respectively, then two perpendicular lines were projected on the x-axis (2θ) from the start and end \ mid-width of the wave, then the equivalent Scherrer crystal size was calculated from Equation (2) [26]:

$$D = 0.9\lambda / \beta \cos\Theta \dots\dots\dots (2)$$

Where λ is wavelength of x-ray (Å), β is FWHM (radian) is the intrinsic full width at Half Maximum of the Peak and θ is the Bragg's diffraction angle of the respective XRD Peak. Table (1) also shows that the average crystallite size of CdS obtained using Scherrer equation (1) [1] is 11.56 nm. The results are in agreement with the results found by Narayanan et al [2] The second method (preparation of CdS NPs from green tea extract) is shown in Figure (5). The results are consistent with JCPDS card files 96-900-8840 and also (96-900-8840) for standard cubic and hexagonal data and the highest peak was (28.3193) (101) and the crystal size (15.9 nm) [27] (They are known by calculating Miller's coefficients with the lattice constant) as shown in Table (2).

While the third method (preparation of CdS NPs from starch) is shown in Figure (3). The highest peak was (23.9446) and the crystal size was (5.67 nm) and this corresponds to the (JCPDS 96-900-8863) for the standard data which had a hexagonal pattern as in Figure (6) In the presence of organic matter, these peaks widened, indicating the reduced particle size (as shown in Table 3) within the nanoscale formed by the effective covering force of the biomolecules present in the organic matter [28].

Therefore, the green synthesis mechanism using starch undergoes hydrolysis providing primary hydroxyl that con-tributes to the reduction of silver. The carboxyl and hydroxyl groups of glucose and hydrolyzed starch stabilize the silver nanoparticles by passivating their surface, thus they do not aggregate and remain uniformly distributed. This leads to the concluded that glucose plays an important role in reducing silver ions into silver nanoparticles [29].

Table (1). Crystal Size of the pure CdS nanoparticles by Chemical method.

2 θ (Deg.)	FWHM (Deg.)	d _{hkl} Exp.(Å)	G.S (nm)	d _{hkl} Std.(Å)	Phase	Hkl	card No.
25.0772	0.8924	3.5482	9.1	3.5808	Hex. CdS	(100)	96-900-8863
26.7845	0.3879	3.3258	21.1	3.3745	Hex. CdS	(002)	96-900-8863
28.3754	0.6207	3.1428	13.2	3.1632	Hex. CdS	(101)	96-900-8863
37.2612	1.2416	2.4112	6.8	2.4558	Hex. CdS	(102)	96-900-8863
44.0129	0.8148	2.0557	10.5	2.0674	Hex. CdS	(110)	96-900-8863
48.1259	1.1253	1.8892	7.7	1.9049	Hex. CdS	(103)	96-900-8863
51.6182	0.6207	1.7693	14.2	1.7629	Hex. CdS	(112)	96-900-8863
52.3554	0.8924	1.7461	9.9	1.7306	Hex. CdS	(201)	96-900-8863
			11.5nm				

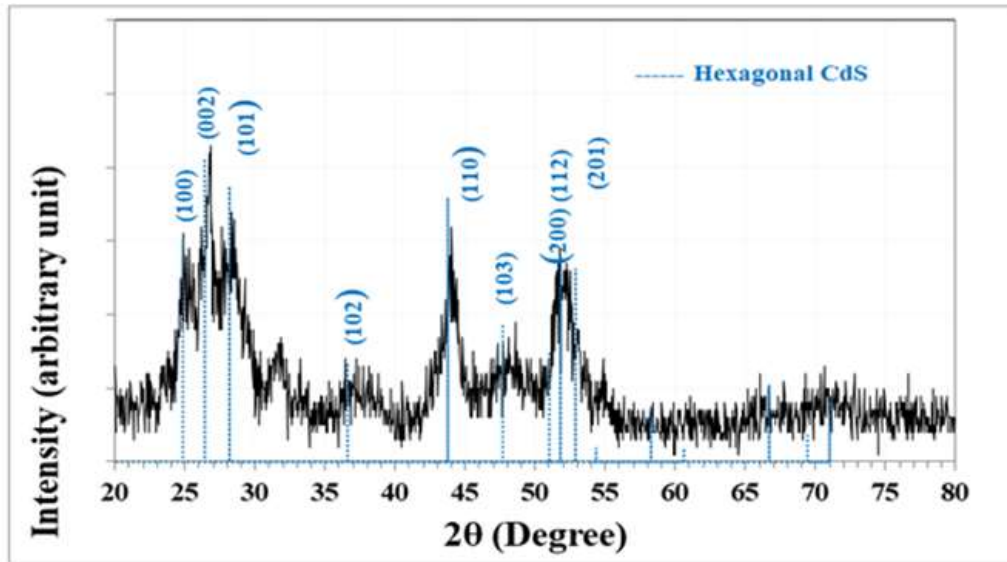


Figure (4). X-ray diffraction pattern of pure CdS nanoparticles (powder) by Chemical method.

Table (2). Crystal Size of the pure CdS nanoparticles by Biological synthesis using green tea.

2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	G.S (nm)	d_{hkl} Std.(Å)	Phase	hkl	Card No.
25.3361	0.6723	3.5125	12.1	3.5808	Hex. CdS	(100)	96-900-8863
28.3193	0.4621	3.1489	17.7	3.1632	Hex. CdS	(101)	96-900-8863
31.3445	0.4621	2.8515	17.9	2.9090	Cub.CdS	(200)	96-900-8840
			15.9nm				

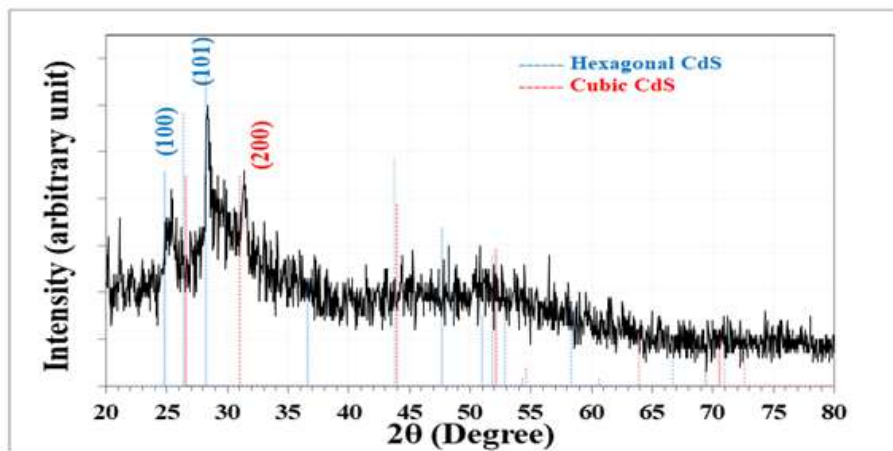


Figure (5). X-ray diffraction pattern of pure CdS nanoparticles (powder) by Biological synthesis using green tea.

Table (3). Crystal Size of the pure CdS nanoparticles by Biological synthesis using starch.

2θ (Deg.)	FWHM (Deg.)	d_{hkl} Exp.(Å)	G.S (nm)	d_{hkl} Std.(Å)	Phase	hkl	Card No.
23.9446	1.4533	3.7134	5.6	3.5808	Hex. CdS	(100)	96-900-8863
27.4740	1.4533	3.2438	5.6	3.1632	Hex. CdS	(101)	96-900-8863
44.2907	1.6609	2.0435	5.2	2.0674	Hex. CdS	(110)	96-900-8863
66.9896	1.5225	1.3958	6.3	1.4009	Hex. CdS	(203)	96-900-8863

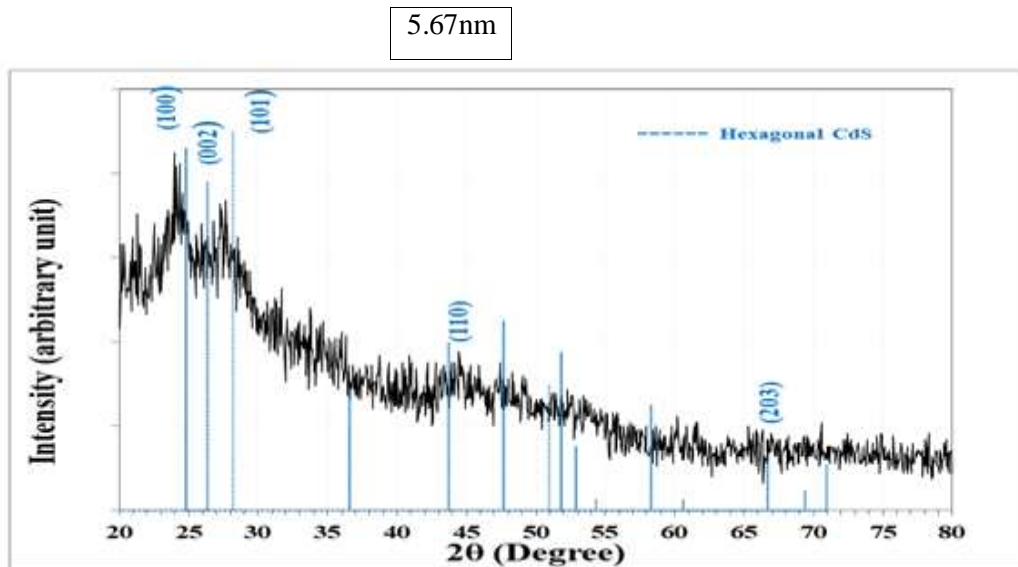
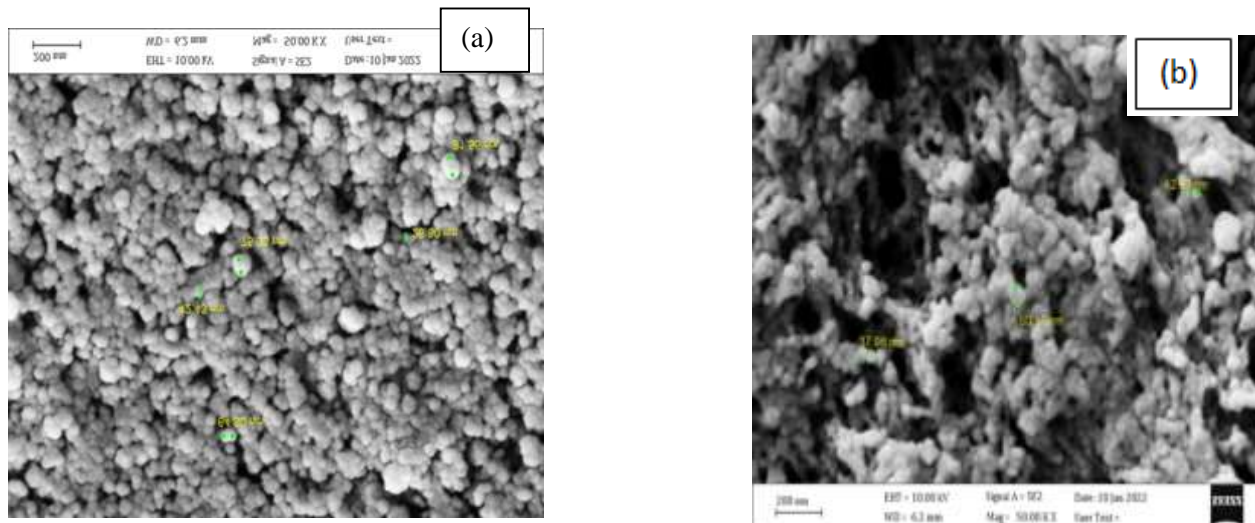


Figure. (6) X-ray diffraction pattern of pure CdS nanoparticles by by Biological synthesis using starch

3.2. FE-SEM Measurement

By examining the SEM images of CdS prepared in three different ways as shown in Figure (7), from which spherical shaped granules were found. The CdS sample prepared by the chemical method shows a size of (26-91) nm as shown in Figure (7 a), and that the particles crystallize well and are distributed with little agglomeration, in this case, an increase in the size of nanoparticles was noticed [30], while the CdS sample prepared by the biological method using green tea extract is shown with a size of (37-80) nm as shown in Figure (7 b). The cadmium sample prepared by the biological method using starch shows the size (24-76) nm as shown in Figure (7 c), with some lumps due to the added organic matter extract encapsulated on the surface of the nanoparticles [20].



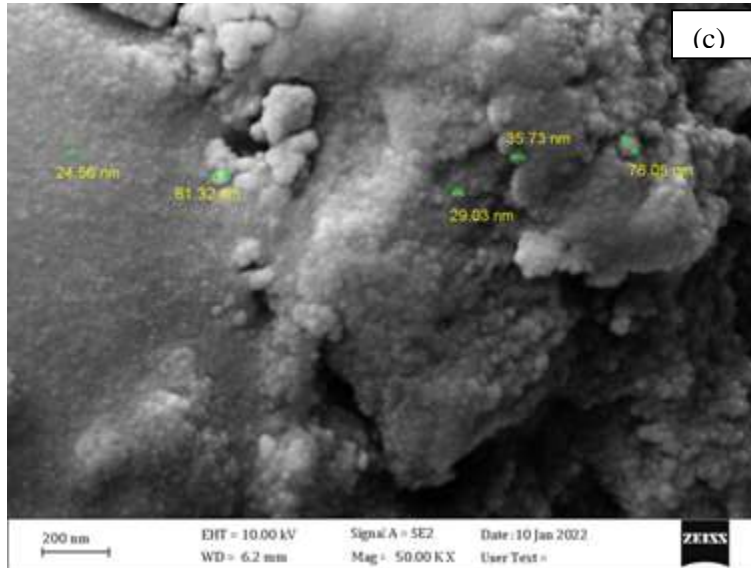


Figure (7). SEM images of (a): CdS by chemical method, (b): CdS by biological method from green tea extract, CdS by biological method from starch.

3.3. Energy Dispersive X-Ray Spectroscopy (EDX)

The EDS results demonstrated that the homogeneity of the nanoparticles was measured by the concentration ratio of the presence of two ions of cadmium and sulfur, confirming the presence of CdS nanoparticles, as shown in Figure (8):

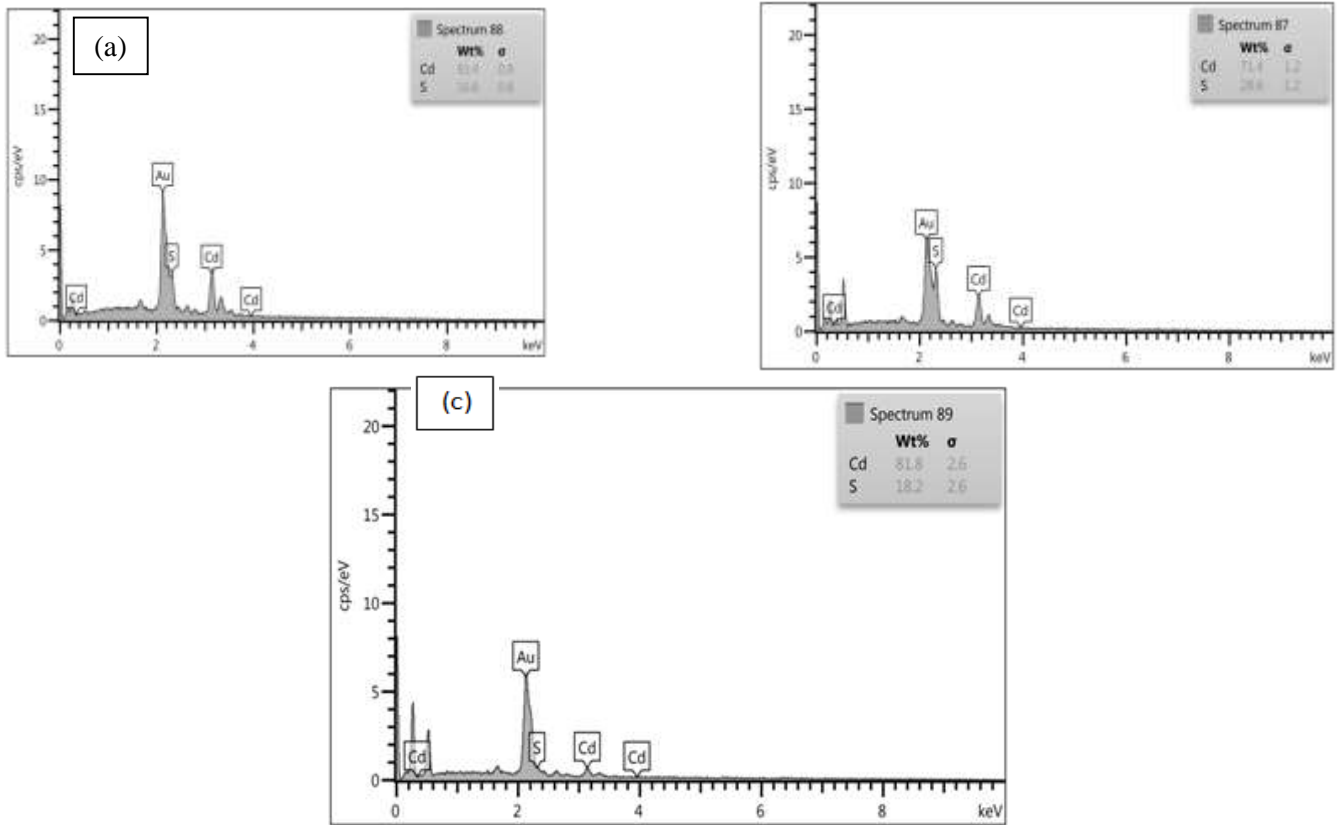


Figure (8). EDS images of (a): CdS by chemical method, (b): CdS by a biological method using green tea extract, (c): CdS by biological method using starch.

3.4. UV-Visible Spectrum

The optical properties of samples prepared by the three methods of pure CdS nanoparticles are determined from absorbance measurements in the range of (300-800) nm. Figure (6) shows the UV-visible absorption spectra of CdS at room temperature. The spectra show a UV absorption excitonic peak at 471 nm for pure CdS, while the linear part shows that the mode of transition in these powders was direct. The spectra's peak corresponds to the fundamental absorption edges in the sample and could be used to calculate the bandgap of the nanomaterial. The Optical energy band gap of CdS nanostructure was calculated by using the Planck Equation (3) [30]:

$$E = 1240/\lambda \text{ (eV)} \dots\dots\dots (3)$$

where the value of the energy gap for the sample prepared by chemical method was (2.63 eV), while the value of the optical energy gap for samples prepared by the biological method using green tea extract and starch was 2.88 eV and 3.06 eV, respectively. This may be due to the result of the volume quantization effect in the sample [31]. From the values of the energy gap, we note that the largest value was for the sample prepared in a biological way using starch, and this means that the material is subjected to more quantitative confinement than the samples prepared by other methods, and this means that the nano-material prepared in this way is the best. Quantum confinement theory says that the surface's potential barrier confines electrons in the conduction band and holes in the valence band, and so the photoelectric transmission of energy from the valence band to the conduction band will be reduced due to the confinement of both electrons and holes [32].

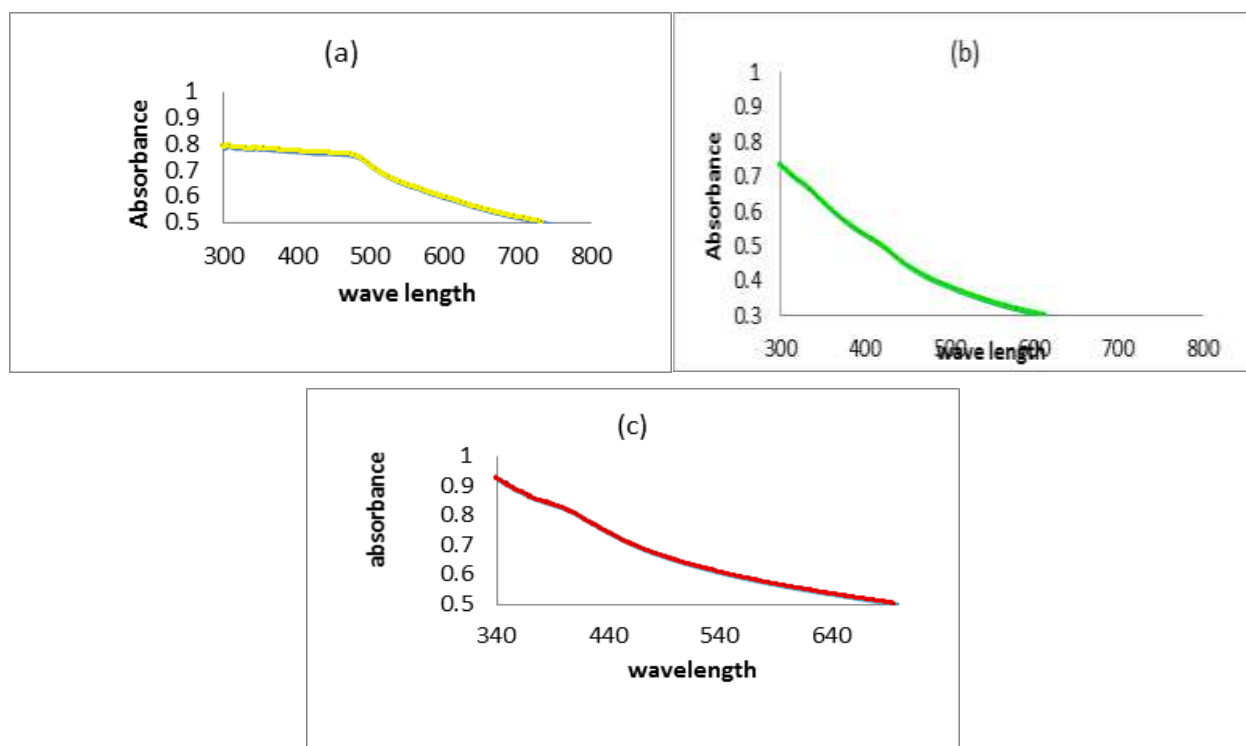


Figure (9). UV-Visible Spectrum of (a): CdS by chemical method, (b): CdS by biological method from green tea extract, (c): CdS by biological method from starch.

4. Conclusions

In this paper, CdS NPs were prepared in different ways: chemical method and biological methods, using two extracts: green tea and starch. The XRD pattern showed that the CdS nanocrystals are cuboidal and hexagonal crystals, and the particle size was within the nanoscale. As for the results of examining the SEM images, it was shown that the shape of the particles was spherical and of different sizes. As for the results of the EDS examination, it showed clear peaks of cadmium and sulfur, but the examination of the results of ultraviolet spectroscopy found that there is an increase in the energy gap than the energy gap in the bulk material of CdS. The nanomaterial prepared in a biological way using starch is the best, as from the energy gap values, we note that the largest value was for the sample prepared in this way, and this means that the material is subjected to

more quantitative confinement than the samples prepared by other methods, and therefore the nanomaterial prepared in this way is the best, and from It is clear that the prepared material has many applications such as anti-bacterial, anti-fungal, treatment of cancer cells, and it is used in solar cells and gas sensing.

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