Influence of Strong and Weak Alkaline Medium on Microstructural and Morphological Investigation of Surfactant Based Synthesis of CdO Nanorods

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Influence of strong and weak alkaline medium on microstructural and morphological investigation of surfactant based synthesis of CdO nanorods

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Abstract

A novel microwave irradiated wet chemical method was adopted to prepare cadmium oxide nanostructures by using sodium dodecyl benzenesulphonate (SDBS) as surfactant in presence of strong and weak 0.1M alkaline medium (NaOH and NH₄OH). The X-ray diffraction measurement shows that the particle is crystallized in the cubic phase, average grain size of the samples are found to be in the range 54 nm for NH₄OH reagent and 60 nm for NaOH reagent. The XRD pattern value based lattice constant, dislocation density, X-ray density, specific surface area, microstrain–Williamson Hall and texture coefficient have been calculated. The surface functionality of the samples were analysed by using fourier transform infrared spectroscopy. The ultra visible transmittance and K-M plot analysed from the diffuse reflection spectra and photoluminescence spectra also been calculated. The morphological changes of cadmium oxide nanostructures with strong and weak 0.1M alkaline medium are clearly recorded through transmission electron microscopy. The reaction mechanism confirms that the experiment carried out in presence of 0.1M NaOH shows rod shaped nanostructures through four step mechanism such as hydration, intermediate formation, metal hydroxide formation and the formation of metal oxide.

Keywords: nanorods, nanoparticles, sodium dodecyl benzene sulphonate, texture coefficient.

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1. Introduction

Metal oxide nanoparticles are playing major role in wide range of recent technologies. Nowadays all the nanotechnologists and researchers are showing much interest to investigate the size dependent properties of metal oxide nanoparticles for their promising applications in gas sensors, supercapacitors, computer memory, device fabrications, photocatalytic and antimicrobial activities [1-4]. Smaller in size and higher in surface area is one of the important parameter to select the metal oxide nanoparticle for their suitable applications. In recent days so many metal oxide nanoparticles like ZnO, CuO, SnO₂, TiO₂, and MoO₃, etc., are synthesized and reported by so many researchers [5-7]. Cadmium oxide (CdO) nanoparticles are also a promising material for multifunctional applications such as sensors, solar cells and photodetectors with their smaller energy band gap, low resistivity and high transmission [8]. By vapour phase transport, solvothermal methods, microemulsion and template-assisted, CdO nanostructures can be synthesized [9,10]. These methods, however, require high temperatures, huge investments, multi-step development, sophisticated facilities for instruments, etc [11,12]. Hence, a simple cost effective synthesis of metal oxide nanostructures is very challenging task [13].

Among these techniques, microwave-assisted hydrothermal process by surfactants or capping agents by a one-step template-free growth develops different morphology, small particle size, high purity and short reaction time, arbitrary particle shapes [14]. Most of the literatures reports the determination of structure type, bandgap and grain size for CdO nanostructures [15]. The quantitative crystalline microstructural parameters for CdO nanoparticle variations developed by microwave-assisted hydrothermal process are analysed using X-ray intensity [16]. The quantitative determination of both texture (polar distribution) and morphology (temperature of nano-rods) of CdO nanoparticles in the case of microstrain,

dislocation density, specific surface area, X-ray density etc.can be analysed using simple X-ray powder diffraction [17,18].

This presenet study deals with the preparation of CdO nanostructures using SDBS as a surfactant in the presence of 0.1M strong (NaOH) and weak alkaline (NH₄OH) medium. The effects of surfactant and alkaline medium on structural and surface morphological behaviour of CdO nanostructures were analysed using XRD, FTIR, UV, PL, SEM and TEM. Also, determination of different microstructural parameters such as favourable orientation, particle size, RMS strain, texture coefficient etc, is reported for CdO nanostructure by X-ray line profile analysis, UV bandgap, photoluminescence properties been performed.

2. Experimental Procedure

2.1. Materials

Analytical grade of Cadmium acetate ($\text{Cd}(\text{CH}_3\text{COO})_2 \cdot 2\text{H}_2\text{O}$), SDBS, NH₄OH and NaOH were purchased from SD-fine chemicals, India. All the chemicals were used without further purification. Double distilled water was used for performing the experiments.

2.2. Synthesis of CdO Nanostructure

0.1M of cadmium solution was prepared by dissolving known volume of cadmium acetate in double distilled water. The surfactant SDBS was prepared as a solution by dissolving 4g of SDBS with 50 ml double distilled water. Both the solutions were taken in 1:1 proportion and the intial pH of the mixture is adjusted to 9.0 by the addition of 0.1M NH₄OH. The mixture is stirred well for two hours and then placed in microwave oven (Model No. IFB, 17PG1S, microwave 700W, input range 210-230 V-ac 50Hz, microwave radiation frequency 2.45GHZ, Power up to 1KW), for 3 minutes. The hot mixture is kept aside for an hour for the complete precipitation. The residual precipitate is filtered and the filtrant is used to mesure the final pH. The filtrate is washed with double distilled water until its filtrant achieves neutral pH. The neutralized filtrate or precipitate is known as pure cadmium

hydroxide, $\text{Cd}(\text{OH})_2$ and it is rinsed with ethanol for the removal of residual impurities if any. The purified $\text{Cd}(\text{OH})_2$ is weighed and annealed at $300\text{ }^\circ\text{C}$ for three hours for the development of CdO. A similar experimental procedure was repeated for the synthesis of CdO nanorods in presence of 0.1M NaOH with the initial pH of 11.0 (Scheme 1). The final product is stored in air tight container and kept it in dessicator for further investigations.

2.3 Characterization

X-ray diffraction data (XRD) were recorded using Bruker (AXS D8 Advance) diffractometer with CuK_α radiation ($\lambda = 1.5406\text{\AA}$) operating at 40kV and 30mA. Fourier transform infrared spectroscopy (FT-IR) spectra were recorded using Bio Rad Spectrometer (Mod. FTS-7) in KBr matrix in the range of $400\text{--}4000\text{ cm}^{-1}$. Diffuse reflectance spectroscopy (DRS) spectra were recorded on a Perkin Elmer UV-visible DRS spectrophotometer. Room temperature photoluminescence (PL) spectra were registered on a spectrofluorimeter Fluorolog (Mod. FL3-11). Scanning electron microscopy (SEM) and energy dispersive X-ray (EDX) analysis were recorded with JEOL Mod. JSM- 6390LV instrument. Transmission electron microscopy (TEM), High resolution transmission electron microscopy (HR-TEM) and selected area electron diffraction (SAED) images were collected by using Philips (Mod. CM 200), electron microscope with an acceleration voltage of 80kV.

Scheme 1. Schematic representation of synthesis procedure for CdO nanostructures

3. Results and discussion

3.1 Structural analysis

The structural phase composition of CdO nanostructures prepared using SDBS as a surfactant in presence of strong and weak alkaline medium (NaOH and NH_4OH) has been analysed through XRD pattern (Fig.1). The as prepared CdO nanostructure using SDBS and 0.1M NH_4OH samples (see Fig.1(a)) shows multiple (hkl) plans and low crystalline nature. The same sample was annealed at 300 °C (see Fig.1(b)) and it shows that the (hkl) miller indecies (111), (200), (220), (311) and (222) with sharp peaks. It indicates the improved crystallinity. The indexed profile are well matched with standard JCPDS(05-0640). Similar characteristic peaks were observed in presence of 0.1M NaOH samples (Fig.1(c-d)). But, the annealing have engendered identical crystallinity in presence of NH_4OH and NaOH with SDBS. Debye-scherer's equation was used to estimate the crystallite size (D) and lattice

parameter (a) of the as prepared and annealed CdO nanostructures were calculated using the formula.

$$D = \frac{K\lambda}{\beta \cos \theta} \quad (1)$$

Where, D is the crystalline size, λ is the wavelength, β is the full width at half maximum of a diffraction peak measured in radians and θ is the Bragg's angle.

$$a^2 = d^2(h^2 + k^2 + l^2) \quad (2)$$

Where, (h, k, l) are the Miller indices, while (d) is the interplanar distances which can be determined by Bragg's law [19].

The estimated structural parameters of the CdO nanostructures are increased with annealing temperature. The mean crystallite sizes of the samples found to be in the range 59 nm and 60 nm. It was also observed that lattice constant value is a well matched with theoretical and experimental value of crystalline size and 2θ value. The dislocations or crystallographic defects interrupt the dislocation lines regular patterns. Dislocation densities are calculated from particle size and strain values. The data indicated that the migration density describes the concentration of crystalline deficits which is high. The crystallographic properties were highly affected by the presence of dislocation density. The dislocation density using crystalline size value (D) has been determined using the williamson and smallman's formula [20].

$$\delta = \frac{1}{D^2} (10^{14} \text{ lines} / m^2) \quad (3)$$

The volume-mass density is a powerful tool used to characterize any material (of any form bulk, thin films or powder) and its ability also to explore any changes in the crystal structure of the crystalline material. It can be calculated theoretically from X-ray diffractograms using the following equation: where n is the number of atoms per unit cell, Mw is the molecular weight, V is the volume of the unit cell and NA is Avogadro's

number. The theoretical density is also known as X-ray density because it requires information about the crystal structure of the studied compound which are deduced from X-ray diffraction analysis. Theoretical density is sensitive to the crystal structure unlike the bulk density [21].

$$\rho_x = \frac{n \cdot M_w}{N_a \cdot V} (g / cm^3) \quad (4)$$

Specific surface area (SSA) describes the properties of the material and determines its type and structure. Practically this quantity may be used for analyzing reactions on surfaces, adsorption and heterogeneity of the sample [22].

$$S_a = \frac{6}{D \times \rho_x} (10^5 cm^2 / g) \quad (5)$$

Where D is the size of the crystallite and ρ is the density of the used experimental samples. The microstrain was calculated by the Williamson-Hall method. The size of the crystallite can be measured with the help of XRD data value. The slope value of the straight line from the relation between $\beta \cos \theta$ vs $4 \sin \theta$, in Fig.2(a-b) indicated the microstrain behaviour of the CdO nanostructure in the studied samples.

$$\beta \cos \theta = \left(\frac{0.9\lambda}{D} \right) + (4\varepsilon \sin \theta) \quad (6)$$

Further, presence of imperfections in the crystal growth can bring about significant changes in the properties of nanostructures. The imperfections include: crystallite size, dislocation density, strain, W-H plot and lattice distortion leads to formation of strained crystals and have been a very important for the solid state substrate [23-25]. However, the effects of imperfections on annealed temperature (300 °C) are yet to be studied. The quantitative of all samples were correlated with their structure and morphology.

The Texture coefficient TC(hkl) value of the samples has been calculated from the XRD data using the relation [26],

$$TC = \frac{I(hkl) / I_o(hkl)}{\frac{1}{N} \sum_N I(hkl) / I_o(hkl)} \quad (7)$$

Where, $I(hkl)$ is the measured relative intensity of a plane and $I_o(hkl)$ is the standard intensity of the plane taken from the JCPDS data. The value $TC(hkl)=1$ represents films with randomly oriented crystallites, while higher values indicate the abundance of grains oriented in a given (hkl) direction. The variation of TC for the peaks of the cubic crystal structure is seen to be highest plane (111) for using 300 °C annealed temperature, which is shown in the Table 1. The Texture coefficient TC [111] value increase in preferred orientation is attributed to the increase in the grain number in this orientation.

The obtained values of the crystalline size, lattice parameters, dislocation density, X-ray density, specific surface area, texture coefficient value of CdO nanstructure using SDBS surfactant in presence of 0.1M strong and weak alkaline (NaOH and NH₄OH) medium obtained in Table 1.

Fig.1 XRD pattern of Cd(OH)₂ inpresence of 0.1M NH₄OH and NaOH (a&c) and CdO nanostructure inpresence of 0.1M NH₄OH and NaOH (b&d).

Fig.2 Williamson Hall Plot is indicating line broadening value due to the equipment of CdO nanostructure inpresence of 0.1M NH₄OH and NaOH (a&b).

Table 1: XRD parameters of CdO nanostructures developed in presence of 0.1M strong and weak alkaline medium.

XRD Parameters	NH₄OH	NaOH
Crystalline size (D) nm	54	60
Lattice Parameters Å (a)	4.69264	4.69625
	3.93 (111)	3.67 (111)
Dislocation Density	5.63 (200)	4.25 (200)
	5.71 (220)	5.93 (220)
X-ray density	8.2538	8.2348
	1.3461	1.2143
Specific surface area	0.8884 (111)	0.8596 (111)
	0.8869 (200)	0.8462 (200)
Texture coefficient	1.0934 (220)	1.0249 (220)

3.2 Functional group analysis

Fig.3 FTIR spectra of Cd(OH)₂ in presence of 0.1M NH₄OH and NaOH (a&c) and CdO nanostructure in presence of 0.1M NH₄OH and NaOH (b&d).

Fig.3(a, c) shows the surface functional analysis of CdO nanostructures in the range between 4000 cm^{-1} to 400 cm^{-1} . The O-H stretching mode of vibration indicates the range between 3444 cm^{-1} to 3599 cm^{-1} . The O-H vibration mode of peaks changes from broad to very sharp (Fig.3a&c), this quick change of vibration demonstrate that the weak alkaline medium (pH=9.0) does not support the complete formation of spherical nano particle, however while using strong alkaline medium in presence of 0.1M NaOH (pH=11.0) the particles are transformed to nanorod shaped particles. When pH value increases in presence of 0.1M NaOH, the vibration peak of C=O was moved up and down which is also due to the morphological changes observed from TEM images and the symmetric stretching take place 1192 cm^{-1} to 860 cm^{-1} [27,9]. This is mainly due to the step-by-step removal of hydrated water molecules from the intermediate complex by OH^- ions from sodium hydroxide. This has been confirmed in the reaction mechanism. Fig.3(b&d) shows O-H stretching mode of vibration peaks observed and it is clearly identified the O-H stretching is broad from flat peaks, when the sample is annealed at $300\text{ }^\circ\text{C}$. Here, the O-H stretching mode of vibration can plays a significant role in the morphology changes and it is clearly shows TEM images. The asymmetric stretching mode of vibration of C=O was observed 1566 cm^{-1} . Fig.3 (b&d) shown Cd-O peaks at 613 cm^{-1} , 621 cm^{-1} and 470 cm^{-1} in the finger print region [28]. The annealing has removed many weak peaks from CdO (Fig.3(b&d)). The FTIR spectrum confirms pure CdO by the presence of characteristic bands at 416 cm^{-1} and 420 cm^{-1} . For conclude from the Fig.3(b&d) the pH value increases the vibration band of CdO increases from 416 cm^{-1} to 420 cm^{-1} [10]. This wave number changes point out the morphology of cadmium oxide related to SEM and TEM interpretation.

3.3 Optical group analysis

CdO has been largely investigated for its interesting optical, optoelectronic and sensing properties. Previously, we published many papers about the synthesis of CdO

nanomaterials, investigating the effect of synthesis variables on the morphological, microstructural, electrical and optical properties of the obtained products. However, in this paper, we report for the first time, the effect of 0.1M strong and weak alkaline (NaOH and NH₄OH) medium used in the synthesis of CdO nanostructures and the evaluation of their morphological, microstructural and optical characteristics. Here, an optical characterization through diffuse reflectance spectroscopy (DRS) and PL for the CdO nanostructures synthesized has been carried out to evaluate the band gap (E_g) and photoluminescence emissions under a 349 nm excitation [29,9].

Diffuse reflectance spectroscopy has been often used for assessing the optical quality of materials, giving information on the absorption properties of bulk and thin films samples. The optical properties of 0.1M strong and weak alkaline (NaOH and NH₄OH) medium samples were measured in the wavelength range of 300 - 800 nm at room temperature. The reflectance values recorded were converted to absorbance by application of Kubelka-Munk function [30], expressed by the following relation:

$$(\alpha \cdot h\nu)^{1/2} = A(h\nu - E_g) \quad (8)$$

$$F(R) = \frac{K}{S} = \frac{(1 - R)^2}{2R} \quad (9)$$

The bandgap was estimated by means of a Tauc plot of the Kubelka–Munk function F(R), considering the indirect bandgap semiconductor nature of CdO nanostructures. For an indirect semiconductor, the Tauc plot is described by eqn (8), where α is the absorption coefficient, $h\nu$ is the photon energy, A is a constant and E is the energy gap. However, for a powder sample, absorption can be estimated as an approximation from the Kubelka–Munk function eqn (9), where k and s are the absorption and scattering coefficients respectively, and R represents the diffuse reflectance. Even though this procedure tends to underestimate the real bandgap energy of the material, giving the so-called optical bandgap, it was

demonstrated to be effective in many cases. From the plot of $(F(R) \cdot h\nu)^{1/2}$ versus the energy ($h\nu$), the bandgap energy E is estimated by extrapolating the intercept of the fitted straight line at $F(R) = 0$. As evidenced in the inset of Fig.4(a) an optical bandgap of 0.1M weak alkaline NH_4OH medium (1.8 eV) and Fig.4(b) 0.1M strong alkaline medium NaOH (2.0 eV) was estimated. As the energy band gap is known to increase as particle size of the semiconductor nanomaterials decreases, the higher value of band gap recorded for the NH_4OH samples is a confirm of the presence of nanostructures of reduced size [8].

Fig.4 UV spectra of CdO nanostructure in presence of 0.1M NH_4OH and NaOH (a&b).

Photoluminescence (PL) characteristics of 0.1M strong and weak alkaline (NaOH and NH_4OH) medium samples have been also investigated. Room temperature PL spectra are shown in Fig.5(a-b). The weak alkaline NH_4OH medium sample exhibits a high intensity emission peak at ~ 453 nm. In the PL emission spectrum of strong alkaline NaOH medium sample is instead evidenced the strong UV emission peak ~ 458 nm. This behaviour is likely due to the different morphology of the samples. In fact, it is well known that the shape and size of the nanostructures are important factors which play a critical role in determining the intensity of the PL emissions. In case of 0.1M strong and weak alkaline (NaOH and NH_4OH) medium sample, the formed agglomerates are somewhat thicker and this hinders the radiation

to diffuse towards the surface, which results in a decrease in the intensity of the blue emission peaks [31, 9].

Fig.5 PL spectra of CdO nanostructure in presence of 0.1M NH_4OH and NaOH (a&b).

3.4 Morphology analysis

Scanning electron microscopy (SEM) analysis has been obtained great surface morphology change of using surfactant SDBS with CdO nanostructures in presence of 0.1M strong and weak alkaline (NaOH and NH_4OH) medium. Fig.6(a-b) shows the SEM image of NH_4OH annealed at 300 °C. The pictures of SDBS surfactant capped with CdO nanostructures in presence of 0.1M NH_4OH do not show any clear surface morphology, however these images says that the CdO nanoparticles smaller agglomerated with not uniform sphere look. The SEM images of SDBS capped CdO nanorods in presence of 0.1M NaOH annealed at 300 °C shown in Fig.6(c-d). In presence of 0.1M NaOH, the CdO nanoparticles appearance is agglomerated tiny nanorods. Hence, the results confirm that, when using 0.1M strong alkaline NaOH medium the smaller spherical particles altered to nanorods. The size of the CdO nanoparticle is 90 nm to 150 nm. The microwave synthesized

CdO nanostructures is obtained remarkable changes in morphology, when using different co-reagents with SDBS surfactant. Morphology results are highlighted that 0.1M NaOH change abruptly from sphere to rod like nanostructures. This kind of size and shape change of metal oxide nanostructures are welcomes to optical properties [32,8].

Fig.6 SEM images shows CdO nanostructure in presence of 0.1M NH_4OH (a&b) and NaOH (c&d).

To study the elemental composition of using SDBS surfactant with CdO nanostructures in presence of 0.1M strong and weak alkaline (NaOH and NH_4OH) medium are shown in Fig.7(a-b). Here found the EDS results are shows the entry of Cd, O and Na without any other impurities. Also expected data like mass% and atomic% is inserted in Fig.7 (a-b).

Fig.7(a-b): EDS spectrum of CdO nanostructure in presence of 0.1M NH₄OH and NaOH.

Fig.8(a-d): TEM, HRTEM micrographs and SAED pattern of CdO nanostructures in presence of 0.1M NH_4OH .

Fig.8(a-d) shows the TEM, HR-TEM and SAED images of CdO nanostructure synthesized by adding 0.1M NH_4OH . In Fig.8(a-b) exhibits number of small particles placed on a single seat. These particles are slightly aggregated together. In presence of 0.1M NH_4OH , the particles are gradually become agglomerated nanorods, as shown in Fig.8(b). HRTEM was used to investigate the crystalline structure of agglomerates. The SAED pattern shows bright spots which confirms the presence of the crystalline phase of the cubic CdO nanostructures [33,30].

Fig.9(a-d): TEM, HRTEM micrographs and SAED pattern of CdO nanostructures in presence of 0.1M NaOH .

Fig.9(a-c) shows the TEM images of CdO synthesized from microwave assisted synthesis using SDBS as surfactant with 0.1M NaOH. By comparison, it is found that with the appearance of SDBS surfactant, the uniform nanorods are clearer. The reason for this phenomenon may be NaOH ions enter the interior of CdO, as well as pH, plays an important role in the growth rate of CdO nanoparticles in presence of 0.1M NaOH, modifying directly or indirectly the kinetics of the growing process, and as a result the size, structure, crystallinity and morphology change from sphere to rod structure was observed using NaOH rather than NH_4OH . The diameter of nanorods range between 20-50 nm thick and length around 300 nm. HRTEM image in Fig.9(d) shows they appear to be more compact, likely

formed by highly interconnected nanorods, with respect to one seen on previous sample. SAED pattern reported in the inset of Fig.9(d), demonstrated that they are constituted by cubic CdO nanostructures highly crystalline [34].

The final morphology and crystal structure of nanosphere and nanorods were found to affect the spacing between crystalline orders. Varying the texture coefficient, dislocation density, specific surface area, X-ray density and grain size has direct effect on the morphology. The growth mechanism and its effect of 0.1M strong and weak alkaline medium as well as surfactant have also been discussed. As of our knowledge, very few reports on CdO nanostructures prepared using SDBS as a surfactant in presence of NH_4OH and NaOH were produced by microwave irradiated wet chemical method. Here, the different morphology and microstructural value obtained from the present work is compared with literatures published elsewhere (Table 2). In this work we confirmed that the shape of nanostructure is stable as well as controllable super molecular structures and flexible physical properties due to the action of SDBS [35]. Also SDBS can proceed as exfoliation to intercalation, which means intercalation can expand the interlayer spacing of CdO material and exfoliation can break the van der waals force between the layers and external force together [36].

3.5 Growth mechanism

The growth mechanism of CdO nanostructures has been explained through four steps such as

- a) Hydration,
- b) Formation of intermediate,
- c) Formation of metal hydroxide and
- d) Formation of metal oxide.

a) Hydration:

The cadmium and SDBS solutions were prepared by using double distilled water and the same were taken in equal proportion for the experiment as doublets. Here both the components are ionized in water such as cadmium acetate will ionized in to Cd^{2+} and CH_3COO^- ions. SDBS will ionized in to Na^+ and negatively charged SDBS derivate. Meanwhile Cd^{2+} will readily form an intermediate $n[\text{Cd}(\text{H}_2\text{O})]^{2+}$.

b) Formation of intermediate:

i) In presence of strong alkaline medium:

When the initial pH of the above said a mixture is adjusted to 11.0 in presence of NaOH, the OH^- introduced in the mixture will interact with the intermediate $n[\text{Cd}(\text{H}_2\text{O})]^{2+}$.and form unstable intermediate $[\text{Cd}_n(\text{OH})_{2n}(\text{H}_2\text{O})_n]$, commonly known as olation [37]. The formation of unstable intermediate initiate the development of precipitate in the reaction mixture.

ii) In presence of weak alkaline medium:

The initial pH of the another mixture is adjusted to 9.0 in presence of 0.1M NH_4OH . Since NH_4OH is weak base due to its low dissociation constant, the intermediate formation is slow process as compared with in presence of strong alkaline medium, It was confirmed that even after the complete stirring of the reaction mixture, the development of precipitate is very poor.

c) Formation of metal hydroxide:

When the pH of the mixture is attained at 11.0 and 9.0, due the addition of 0.1M NaOH and NH_4OH , respectively in the reaction mixtures the excess $n[\text{OH}]^-$ ions eliminate the

$n\text{H}_2\text{O}$ molecules as its own ionic from the unstable intermediate $[\text{Cd}_n(\text{OH})_{2n}(\text{H}_2\text{O})_n]$ and produce $\text{Cd}(\text{OH})_2$ as precipitate. Simultaneously, the excess OH^- ions from water molecule will reacts with Na^+ ions of SDBS and enhance the alkaline (NaOH) nature in the reaction mixture and it helps to terminate the reaction with the formation of hydroxylated SDBS. It was observed that the final pH of NaOH and NH_4OH added mixture were enhanced to 13.0 and 11.0, respectively.

d) Formation of metal oxide:

The nown volume of neutral pH achieved final $\text{Cd}(\text{OH})_2$ residue is anneled at $300\text{ }^\circ\text{C}$ to get the purest form of CdO . The water molecules presrnt on $\text{Cd}(\text{OH})_2$ is completely dehydrated and the purest form of CdO obtained. From the morphological analysis it was observed that the strong alkaline medium develops nanorod shaped nano materials. It is mainly due to the step-by-step elimination of water molecule from the unstable intermediate. But in the case of weak alkaline medium the formation of perfect structure is retarded due to the presence weak electrolytic behaviour. Thus the morphological analysis simply shows the spherical shaped structure. The comparision of CdO nanoparticle development and its morphological structure changes mechanism reported in literature is shown in Table 2.

Table 2: Comparison of CdO nanoparticle development and morphological structures reported in literature.

Component			Growth mechanism	Crystallite size (nm)	Band gap (eV)	Morphology	Reference
Major	Additives	Reagent / pH					
Cadmium chloride	-	*	*	*	*	Nanowires	[38]
Cadmium acetate	PEG	NaOH / *	*	24.5	*	*	[39]
Cadmium nitrate	PEG	*	*	25	*	Nanoparticles	[40]
Cadmium chloride	-	Urea / *	*	65	3.75	Spherical, rod	[41]
Cadmium acetate	CTAB	Acetic acid / 7.3	† ¹	40 to 45	2.0 to 2.5	Spherical	[42]
Cadmium nitrate	-	NaOH / 11.0	*	*	*	Nano disk	[43]
Cadmium acetate	Glycine	NH ₃ / 12.0	*	12.6	2.35	Nanoparticles	[44]
Cadmium acetate	PEG	NaOH / 9.0	*	19.1	*	Spherical	[45]
Cadmium chloride	Triton	NaOH or HCl /	*	50	*	Sphere	[46]
	X100	2.0 to 12.0					
Cadmium acetate	-	NaOH / *	*	25	4.04 to 4.45	Plate like structure	[47]
Cadmium acetate	SDBS	NH ₄ OH / 9.0	† ²	54	1.8	Spherical	Present work
Cadmium acetate	SDBS	NaOH / 11.0	† ²	60	2.0	Nanorods	Present work

* Not reported; †¹ Micelle formation; †² Four step mechanism

4. Conclusion

Using microwave irradiation method, CdO nanostructures have been successfully synthesised using SDBS as a surfactant in presence of strong and weak alkaline medium. Uniformly synthesized CdO nanostructures clearly demonstrate that morphology and microstructures have been affected by the surfactant as well as alkaline medium. Crystalline nanorods have been developed in presence of strong alkaline medium due to step-by-step elimination of water molecule from the unstable intermediate and are found in elongated hierarchical nanostructures of a few microns in length. In order to synthesise standardized CdO nanorods for professional toxic gas sensors, this reported method can be used.

Author contributions

All authors discussed the experiment results and contributed to the writing of the paper.

Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Conflict of Interest

The authors declare no conflict of interest.

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Figures

Figure 1

XRD pattern of Cd(OH)₂ in presence of 0.1M NH₄OH and NaOH (a&c) and CdO nanostructure in presence of 0.1M NH₄OH and NaOH (b&d).

Figure 2

Williamson Hall Plot is indicating line broadening value due to the equipment of CdO nanostructure in presence of 0.1M NH₄OH and NaOH (a&b).

Figure 3

FTIR spectra of Cd(OH)₂ inpresence of 0.1M NH₄OH and NaOH (a&c) and CdO nanostructure inpresence of 0.1M NH₄OH and NaOH (b&d).

Figure 4

UV spectra of CdO nanostructure in presence of 0.1M NH₄OH and NaOH (a&b).

Figure 5

PL spectra of CdO nanostructure in presence of 0.1M NH_4OH and NaOH (a&b).

Figure 6

SEM images shows CdO nanostructure in presence of 0.1M NH₄OH (a&b) and NaOH (c&d).

Figure 7

(a-b): EDS spectrum of CdO nanostructure in presence of 0.1M NH₄OH and NaOH.

Figure 8

(a-d): TEM, HRTEM micrographs and SAED pattern of CdO nanostructures in presence of 0.1M NH_4OH .

Figure 9

(a-d): TEM, HRTEM micrographs and SAED pattern of CdO nanostructures in presence of 0.1M NaOH .

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