IJARSCT
ISSN (Online) 2581-9429
International Journal of Advanced Research in Science, Communication and Technology (IJARSCT)
International Open-Access, Double-Blind, Peer-Reviewed, Refereed, Multidisciplinary Online Journal
Impact Factor: 7.301
Volume 3, Issue 8, April 2023

Chemically Synthesized Ammonia Free CDS Thin Film for Supercapacitor Performances

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Abstract*: In the current study, we successfully created a thin film of Cadmium Sulphide (CdS) on stainless steel substrates using the quick and inexpensive Chemical Bath Deposition (CBD) method. Using various characterization techniques like XRD, FESEM, and EDAX, the prepared (CdS) thin film was examined for its structural, morphological, and compositional analysis. Subsequently, the CdS thin film has morphology similar to Nanoflakes, which is well adapted for supercapacitor application due to its naturally crack-free morphology. According to the X-Ray diffraction (XRD) study, the CdS thin film was naturally polycrystalline. Synthesized CdS thin film show super capacitance characteristic.*

Keywords: Cadmium Sulphide (CdS); Chemical Bath Deposition (CBD); Supercapacitor

I. INTRODUCTION

In recent days a major global upsurge in the depletion of fossil fuels has raised two primary concerns as a result of the rapidly expanding global economy.To the first is the depletion of existing fossil fuel supplies, and the second is correlated with an increase in greenhouse gas emissions, in particular, and environmental pollution. In general.Consequently, it is crucial to create and promote a sustainable environment[1-2].Although it has been established that faradic capacitors are favoured by large surface area, high electrical conductivity, and an abundance of channels, realising them simultaneously through an easy preparation process is still a significant challenge. Because of their potential use in biology, electronics, optics, transportation, and information technology, significant research effort has recently been devoted to creating inorganic nanocrystals[3].

Cadmium Sulphide (CdS) nanostructures for supercapacitor structural, morphological, wettability, optical, and electrical properties, as well as the impact of film thickness on these characteristics, have all been studied. Due to their exceptional optoelectronic properties, semiconductor nanostructures are sought-after materials in the research and development sector.One of the more promising materials for the creation of heterojunction solar cells and optoelectronic devices is cadmium sulphide (CdS) thin films [1].The size, shape, and crystallinity of these materials, as well as various parameters affecting their size and shape, still need to be controlled, despite numerous attempts to create these nanocrystalline thin films. Cadmium sulphide (CdS), one of the important group II-VI semiconductor materials, is one of the many different types of semiconductors and is frequently used to make thin-film. Chemical bath deposition (CBD) is the method most frequently used to deposit CdS thin films or layers.In comparison to other methods, the CBD method for the creation of thin films is straightforward, appealing, affordable, and time-efficient [4]. Its main benefit is the simplicity with which the growth rate can be controlled using its various parameters, such as time, immersions, solution concentration, pH, etc. Metal sulphide nanomaterials have attracted a lot of attention in the modern world because of their important properties and advancing uses in electronics, optoelectronics, and energy storage. The industrially important and scientifically significant metal chalcogenides (S, Se, and Te) have drawn a fair amount of attention in the past 20 years due to their unique properties. Researchers attribute these advantageous CdS properties to development of their energy storage system capacity[1].

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II. EXPERIMENTAL DETAILS

2.1 Materials

Cadmium chloride ($CdCl_2.H_2O$), sodium citrate ($C_6H_5O_7Na_3$), potassium hydroxide (KOH), pH 10-borate buffer solution, thiourea (H_2NCSNH_2), and deionized water, all the chemical are AR grade, used for the deposition. For thin film deposition, the highly polished stainless steel (SS) substrates (304 grade) were used. The 100 ml beaker was used for the deposition.

2.2 Synthesis of CdS

Cadmium chloride ($CdCl_2.H_2O$) andThiourea (H_2NCSNH_2) were used as precursors for the elements cadmium and sulphur respectively, and sodium citrate ($C_6H_5O_7Na_3$) was used as a complexing agent. The CdS films were deposited in a solution containing 20 ml of 0.05 M $CdCl_2$, 20 ml of 0.5 M $C_6H_5O_7Na_3$ (sodium citrate), 5 ml of 0.5 M KOH, 5 ml of a pH 10 borate buffer and 20 ml of 0.1 M $CS(NH_2)_2$. Deionized water was used to dilute the solution to a final volume of 100 ml. For 24 hours, the reaction solution-containing beaker was submerged in a water bath that was kept at room temperature [5].By dipping the stainless steel substrates in the precursor solution, the CdS thin films were created.After being rinsed with double-distilled water, the CdS films that had been applied were left to dry at room temperature in the open air.The slides were thermally annealed at 250 °C for 1 hour to enhance the electrical response of the semiconductor layer[6].

2.3 Characterization Techniques

XRD technique is non- destructive for characterizing crystalline materials. It provides information about crystal structure, phase, preferred crystal orientation and other structural parameters such as grain size, crystalinity, stress, and strain. XRD data is collected by using Bruker Ltd Germany, AXS D8 Advances.TEM study was done by using JEOL ASIA PTE LTD,JEM 2100 PLUS.TEM is a microscope that use a electron beam to visualize specimens and generate highly magnified image than optical microscope.SEM images are recorded using JEOL JSM-6360 instrument to study the morphology.NEX CG II, powerful second-generation energy dispersive X- ray fluorescence(EDS), spectrometer, delivers rapid qualitative and quantitative determination of majour and minor atomic elements in a wide variety of sample type-from oils and liquids to solid, metals, polymers, thin film.(EDS) microanalysis is a technique of elemental analysis associated to electron microscopy based on the generation of characteristic X- ray that reveals the presence of elements in specimens.

III. RESULTS AND DISCUSSION

3.1 XRD Analysis

The crystal structure of the electrode material was first investigated using XRD analysis [7]. The XRD pattern of the CdS thin films is displayed in Figure 1. In this pattern, the planes are (002), (101), (102) and (112) indicating that the films are polycrystalline hexagonal wurtzite and cubic structure. Peak at 2θ angle of 45 with plane 110 indicate stainless steel substrate. Cadmium sulphide thin film have low polycrystallinity, which is indicated by their broad and low-intensity peaks.
Thepolycrystallite size of the CdSnanocrystals was calculated using Scherrer Equation [1],

$$D = 0.94\, x\lambda \,/\, \beta x cos\theta$$

Where D is the crystallite size, λ is the wavelength of X-ray, β is the diffraction peak and θ is Bragg's angle of XRD peak.
As the film thickness increases, so does the relative peak intensity. The XRD pattern clearly shows that CdS thin films are polycrystalline and grain size of the particles is found to be 32nm and have a cubic crystal structure.

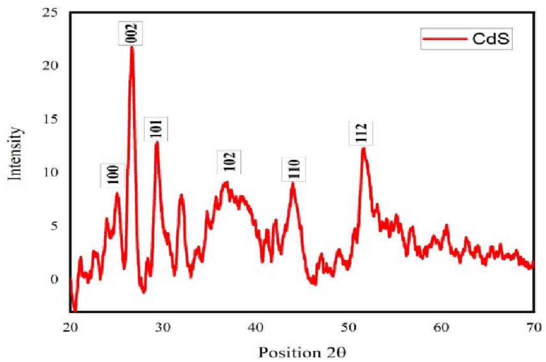

Figure 1. X-ray diffraction patterns of CdS thin films

3.2 FESEM Analysis

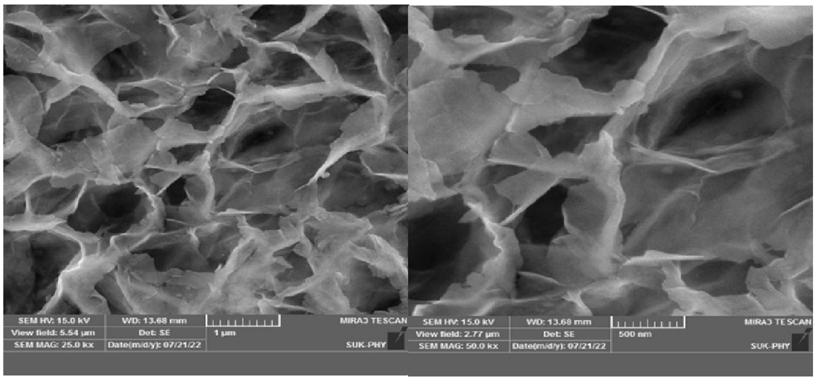

Figure 2. FESEM of CdS thin films

Field-emission scanning electron microscopy was used to examine the surface morphology and microstructural, optical behavior thin films (FESEM) [1].Above Figure 2 shows Surface also covered with agglomerates of different size and porosity space between particles . 50.0 kx, 15.0 kx and 25.0 kx magnification images of CdS thin films deposited. The aspect of the application point where each nanoflakes could contribute electrochemical reaction and intercalate using electrolyte ions was supported by the morphology of films with defined nanoflakes edges. As a result, it is the film that is best suited for supercapacitor applications [9-10].

3.3 TEM Analysis

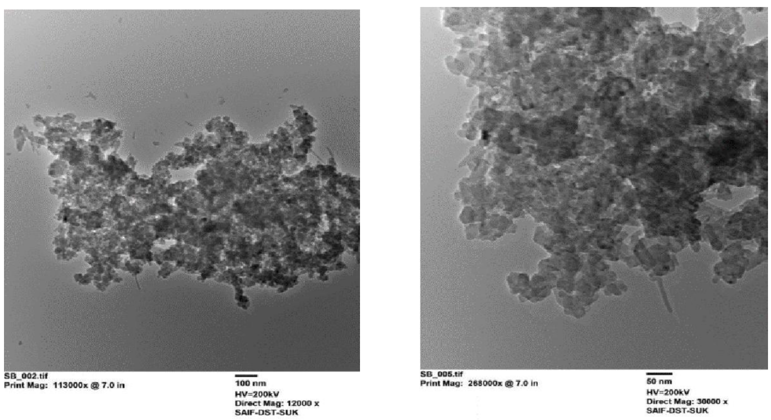

Figure 3. TEM images of CdS thin films

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Transmission electron microscopes (TEM) are microscopes that visualise specimens and produce highly magnified images using an electron particle beam. The TEM images of CdS are shown in Figure 3. According to the figure, CdS is made up of tiny, spherical nanoparticles is provided by the transmission electron microscope (TEM) and is derived from transmission spectra. Transmission electron microscopy was used to investigate the crystal structure of CdS[11]Cadmium sulfide are in form of nanorods, in variable size and their shape are visualized using TEM technique as shown in Figure 3.

3.4 EDS Analysis

Surface analysis techniques like SEM with X-ray analysis (SEM/EDS) are known to be reasonably quick, affordable, and essentially non-destructive. EDX investigates the composition and purity of goods.The technique of energy-dispersive X-ray analysis (EDS) is used for the measurement of nanoparticles. This method involves activating the nanoparticles with an EDS X-ray spectrophotometer, which is typically included in modern SEMs [1].

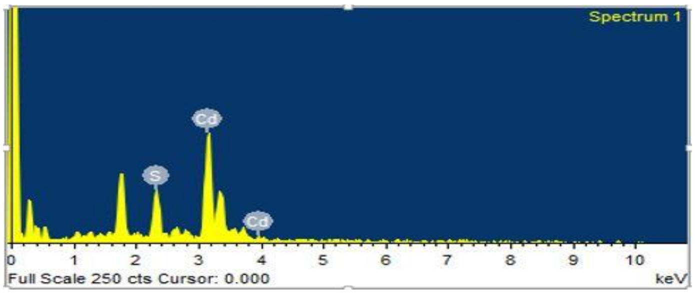

Figure 4. Energy Dispersive X-ray (EDS) of CdS thin films

The prepared samples underwent EDX analysis. The EDAX pattern exhibits Cd and S peaks, indicating that only pure Cadmium Sulphide has formed without the presence of any other elemental impurities .The CdS nanoparticle were studied briefly using scanning electron microscopy and energy dispersive X-ray (SEM/EDS)spectroscopy.

3.5 Super-Capacitive Capacitors

Cyclic Voltammetry (CV)

In a three-electrode cell made with CdS as the working electrode, platinum as the counter electrode, and SCE as the reference electrode in 1 M Na_2SO_4 aqueous solution, the super capacitive performance is evaluated using the cyclic voltammetry (CV) technique. The CVs were measured between a potential window of -1.2 and -0.2 V at various scan rates, including 100, 75, 50, 25, 10, and 5 mV/s.Figure 5 displays CdS cyclic voltammetry at various scan rates [1].

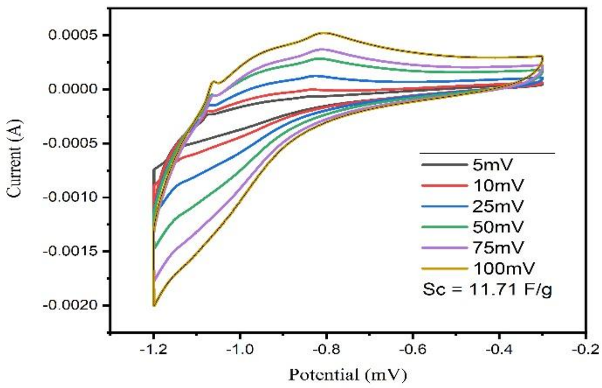

Figure 5. Cyclic Voltagramme of CdS

Table 1. Specific Capacitance at different scan rates

Scan Rate	Specific Capacitance C-1 (F/g)
5	17.75

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10	15.43
25	11.56
50	9.87
75	8.19
100	7.848

Using the following equation, the capacitance (C) of a synthesized CdS thin film is calculated. C represents capacitance (F/g),

$$C = 1/mv(Vc - Va) \int_{Va}^{Vc} I(V)dV$$

$V_c$and V_a is a potential range of -1.2 to -0.2 V, I is the applied current, and m is the weight of the deposited CdS on the electrode for Unit area (1 cm^2) dipped in electrolyte.

3.6 Galvanostatic Charge-Discharge

The galvanostatic charge-discharge (GCD) test was used to assess the performance of supercapacitors. The charge-discharge characteristics of a CdS thin film were investigated using the galvanostatic charge-discharge technique. The galvanostatic charge-discharge curve of CdS at current densities of 3 mA, 2 mA, 1 mA, 0.9 mA, 0.8 mA, 0.7 mA, and 0.6 mA is shown in Figure 6 with a potential window of -1.2 to -0.2 V.

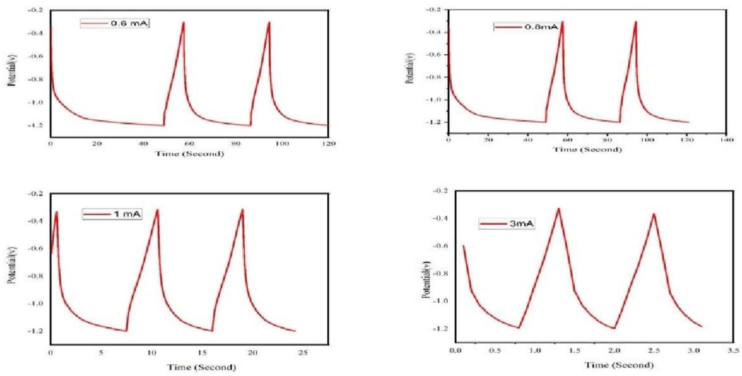

Figure 6. galvanostatic charge-discharge curve of CdS of 0.6mA, 0.8mA, 1mA and 3mA respectively.

3.7 Electrochemical impedance analysis (EIS)

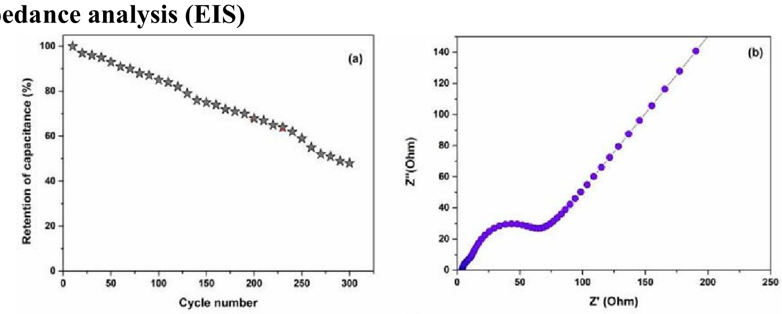

Figure 7. (a) cyclic stability test (b) the Nyquist plot for 0.5M KOH.

In order to characterise the materials, electrochemical impedance spectroscopy, a potent method for examining the capacitive behaviour of electrochemical cells, is used [12]. Measurement of the electrochemical impedance spectrum (EIS) is a useful method for examining the charge transfer at the semiconductor/electrolyte interface.As can be seen in Figure 7, the Nyquist plot for symmetric supercapacitor in 0.5M KOH is shown [13]. Utilizing impedance behaviour, the type of material response and process involved is explored.Figure 7 displays the imaginary part of impedance's frequency dependence (Z"). It shows a semicircle at high frequency and a straight line at low frequency. The electrical resistance of the electrolytes is observed to cause the initial nonzero intercept in the high frequency region at the beginning of the semicircle [12].

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IV. CONCLUSION

In conclusion, a low-cost CBD method is used to create ammonia-free CdS thin film on stainless steel substrates, and its super-capacitive properties of specific capacitance at 17.75 F. The surface morphology of the CdS thin films and nanoflakes reaches its maximum electrochemical specification. The XRD analysis showed that CdS thin films show polycrystalline hexagonal wurtzite and cubic in nature and Grain size of particles is 32nm.The SEM showed porous structure of thinfilm.TEM images revealed the spherical morphology of thin film. The elemental composition confirmed that thin film are stoichiometric

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