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Synthesis and Electrochemical Investigation of Hydrothermally Synthesized CdO Electrodes for Supercapacitor Applications

Pachukant D. Holkar[1], Supriya J. Shinde[2], Vikrant P. Lalge[3], Arti A. Zaware[1], Tanaji P. Gujar[4*], Pradip B. Shelke [1*]

[1]B.P.H.E. Society's Ahmednagar College, Ahilyanagar-414001, Maharashtra, affiliated to Savitribai Phule Pune University (MS), India 413102

[2]Anekant Education Societies Tuljaram Chaturchand College, Baramati– 413102, Pune, Maharashtra.

[3]Vidya Pratishthan's Arts, Science & Commerce College, Baramati– 413133, Pune, Maharashtra.

[4]ADT's Sharadabai Pawar Mahila Arts, Commerce and Science College, Baramati –413115, Pune, Maharashtra.

*Corresponding Author: shelkepradip@gmail.com.

holkarpd28@gmail.com

Abstract
The development of advanced electrode materials is essential for high-performance supercapacitors requiring high energy and power density. In this study, cadmium oxide (CdO) thin films were synthesized on nickel (Ni) foam via a hydrothermal method followed by annealing, with growth durations varied from 2 to 12 h. Structural and electrochemical analyses revealed that hydrothermal time significantly influences material properties. FTIR confirmed Cd–O bonding, while XRD showed a cubic CdO phase with enhanced crystallinity at longer durations. Cyclic voltammetry indicated pseudocapacitive behaviour with Cd^{2+}/Cd redox activity. The 6 h sample exhibited the highest current response and capacitance retention due to optimized nanostructure and efficient ion transport. Galvanostatic charge–discharge results showed that the 8 h electrode delivered longer discharge times, indicating higher energy storage capacity. Electrochemical impedance analysis revealed low charge-transfer resistance and improved conductivity.

1.1 Introduction

In recent years, the growing demand for sustainable and efficient energy storage devices has accelerated research on advanced electrode materials for supercapacitors. Supercapacitors, also known as electrochemical capacitors, are attracting significant attention due to their high-power density, rapid charge–discharge capability, and long cycle life, making them promising candidates for next-generation energy storage systems. The performance of supercapacitors strongly depends on the structural and electrochemical properties of the electrode materials.

Among various electrode materials, transition metal oxides are particularly promising because of their multiple oxidation states and high theoretical capacitance. Cadmium Oxide (CdO) is one such material, offering good electrical conductivity, chemical stability, and excellent pseudocapacitive behaviour [1]. Hydrothermal synthesis combined with annealing has been widely used to grow metal oxide nanostructures directly on conductive substrates such as nickel (Ni) foam, which provides a three-dimensional porous network facilitating ion diffusion and electron transport [2]. Demonstrated that CdO composites grown on Ni foam exhibited enhanced capacitance due to synergistic effects among the components and efficient electron pathways [3]. Reported that CdO improved charge storage, by combining the high conductivity of CdO electrodes, showing improved cycling stability and capacitance [4]. Early studies on CdO films revealed intrinsic pseudocapacitive behaviour, further supporting its suitability as a supercapacitor electrode material [5]. Composites like CdO and CdO–

SnO_2 have shown that hybridization with other oxides or conductive scaffolds can enhance areal capacitance, rate capability, and cycling performance [6,7]. Demonstrated that CdO nanocomposites synthesized via hydrothermal methods exhibited enhanced charge storage due to the synergistic effect between the high conductivity of CdO. Comprehensive reviews on supercapacitor materials emphasize that careful selection of electrode materials, nanostructure design, and the interaction with conductive substrates such as nickel foam are crucial for achieving high energy and power densities, as well as long-term cycling stability [8].

Moreover, the morphology and substrate interactions play a crucial role in electrochemical performance. Studies on Ni foam-supported electrodes demonstrated that hydrothermal growth conditions, precursor concentration, and the use of additives significantly influence the nucleation, growth, and final architecture of metal oxide nanostructures, which in turn affect surface area, porosity, and ion diffusion pathways [9,10]. Optimized hydrothermal reaction duration and post-synthesis annealing are key factors in controlling crystal size, phase purity, and adhesion of CdO onto the Ni foam, directly impacting electrode stability and capacitance retention during long-term cycling [10]. Furthermore, substrate selection, surface activation, and pretreatment methods can enhance interfacial contact, electron transfer, and overall electrode conductivity, which are critical for achieving high-rate performance and energy density. Building on these findings, the present study focuses on the hydrothermal deposition of CdO on nickel foam followed by annealing. It investigates the effect of reaction time on CdO loading, surface morphology, crystallinity, and super-capacitive performance to provide insights for designing high-efficiency CdO-based electrodes with optimized electrochemical characteristics for practical energy storage applications [10].

2.1. Experimental

A nickel foam substrate was first cleaned by rinsing thoroughly with ethanol (2–3 times), followed by ultrasonication in 1 M HCl solution for 10 minutes and then in double-distilled water for another 10 minutes, then it was dried in a hot air oven at 60 °C for 4 hours. For the preparation of the precursor solution, 2 g of cadmium nitrate tetrahydrate and 500 mg of urea were dissolved in 100 mL of double-distilled water and stirred for 30 minutes using a magnetic stirrer. The prepared solution was transferred into a 150 ml hydrothermal reactor, into which the activated nickel foam substrate was placed and subjected to hydrothermal treatment for different durations (2, 4, 6, 8, and 10 hours). After the reaction, the substrates were washed with double-distilled water to remove unreacted residues and then annealed in a muffle furnace at 400 °C for 2 hours to obtain CdO films. The weight variation of the nickel foam was recorded before and after deposition to determine the CdO loading, which was found to be 11 mg (from 227 to 238 mg) for 2 hr, 26 mg (from 242 to 268 mg) for 4 hr, 21 mg (from 223 to 244 mg) for 6 hr, and 55 mg (from 223 to 278 mg) for 8hr, while the sample treated for 10 hr was similarly processed.

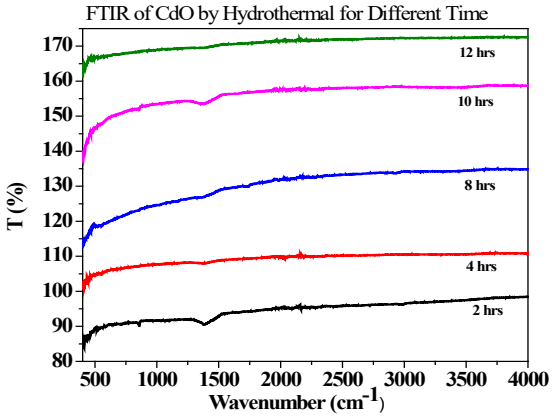

Fig.1. FTIR of CdO thin films synthesised by the hydrothermal method

The FTIR spectra of CdO thin films synthesised by the hydrothermal method for different growth times (2, 4, 8, 10, and 12 hours) on Ni foam are shown in the figure. The transmittance curves exhibit broad features without sharp peaks in the higher wavenumber region (1000–4000 cm^{-1}), indicating the absence of strong organic functional groups and confirming the purity of the CdO phase [11,12]. The main absorption bands are observed in the low wavenumber region around 400–600 cm^{-1}, which correspond to Cd–O stretching vibrations, confirming the successful formation of CdO[13]. As the hydrothermal growth time increases, the intensity and smoothness of the spectra change, reflecting modifications in the crystallinity, grain size, and bonding characteristics of the CdO. Longer growth times (10–12 hrs) show higher transmittance values, suggesting improved crystallinity and reduced defect states, whereas shorter times (2–4 hrs) result in relatively lower transmittance due to incomplete growth and higher defect concentration [14,15]. These observations indicate that hydrothermal duration significantly influences the structural and vibrational properties of CdO grown on Ni foam, with prolonged times favouring better-defined Cd–O bonding and higher material quality.

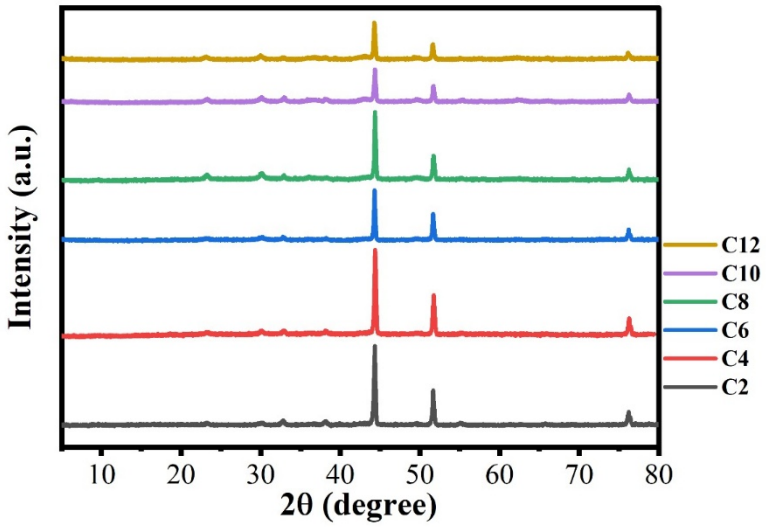

Fig.2. XRD of CdO thin film prepared by Hydrothermal method for different hrs.

The patterns (labelled C2 → C12, presumably 2,4,6,8,10,12 h hydrothermal times) show the cubic CdO phase (NaCl-type) as the dominant product: the strong CdO reflections are cantered near the expected 2θ positions for the (111), (200), (220), (311) and (222) planes and match the standard JCPDS/ICDD CdO card (05-0640). Two sharp peaks near ~44–45° and ~51–52° arise from the Ni foam substrate (metallic Ni), not from CdO. As hydrothermal time increases the CdO peaks become stronger and narrower (higher intensity and smaller FWHM), indicating improved crystallinity and growth of larger coherently scattering domains (larger crystallite size) with longer reaction time [16].

The XRD patterns confirm the formation of cubic CdO, with prominent diffraction peaks appearing near 33–34° (111), 38–39° (200), 55° (220), 65–66° (311) and 69° (222), consistent with the standard JCPDS CdO card (~05-0640,) [17]. Two additional sharp reflections at about 44.5° and 51–52° originate from the metallic Ni-foam substrate and are not related to CdO [18]. As the hydrothermal growth time increases from C2 to C12, the CdO peaks steadily intensify, indicating thicker deposits and improved crystallinity; their full width at half maximum (FWHM) narrows, reflecting an increase in crystallite size and reduced lattice strain or defects [19]. Moreover, changes in the relative intensity of specific planes particularly if the (111) reflection strengthens more than others suggest the development of preferred orientation (texture) during longer synthesis periods.

The Scherrer equation to estimate the average crystallite (coherently scattering domain) size from the FWHM of an isolated CdO peak:

$$D=K\lambda/\beta\cos\theta$$

where K ≈ 0.9 (shape factor), λ is X-ray wavelength (Cu Kα = 1.5406 Å), β is FWHM (radians, after instrument-broadening correction) and θ is Bragg angle [20]. Note this gives a lower-bound crystallite size; peak broadening can also come from micro strain, defects or instrument effects, so consider Williamson–Hall or full profile methods for size/strain separation.

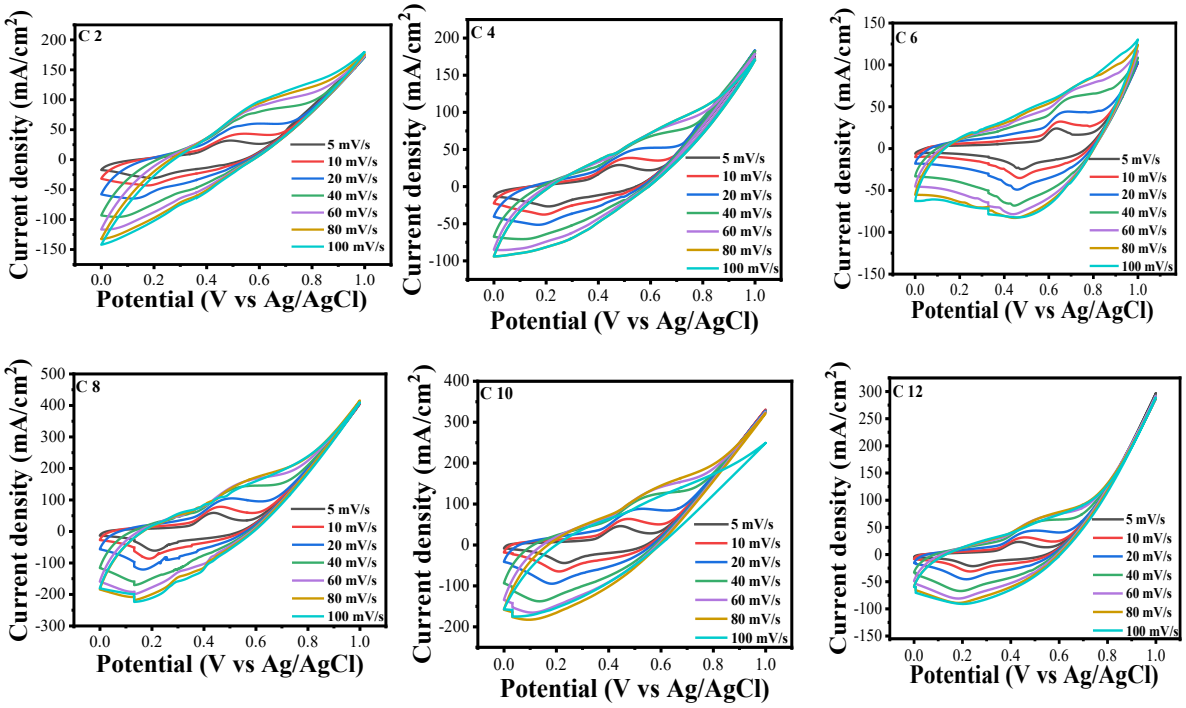

Fig.5. Cyclic Voltammetry of CdO prepared by Hydrothermal method for different hours

Based on the cyclic voltammetry data collected across multiple scan rates, the electrochemical performance of the various CdO-based electrodes (C2, C6, C8, C10, C12) demonstrates significant and systematic variation, highlighting critical differences in their charge storage capabilities and kinetic properties. All electrodes exhibit the characteristic pseudocapacitive behaviour associated with the reversible Cd^{2+}/Cd redox couple, as evidenced by the presence of broad, symmetric redox peaks [21]. However, electrode C6 emerges as the unequivocal standout performer, delivering a substantially higher current density response of approximately 150 mA/cm², which corresponds to the largest enclosed area under its voltammograms and thus indicates superior specific capacitance [22]. This exceptional performance is further reinforced by its excellent rate capability; even at the highest scan rate of 100 mV/s, C6 maintains well-defined peak shapes and minimal increase in peak potential separation, signifying highly favourable reaction kinetics, efficient charge transfer, and optimal electrolyte ion accessibility, likely owing to an ideal nanostructure or composite formulation [23].

In contrast, electrodes C8 and C10 display intermediate performance, with moderate current densities and reasonably defined redox activity, but exhibit greater peak broadening and potential separation at elevated scan rates, suggesting acceptable but comparatively slower charge storage dynamics [24-30]. The poorest performers, C2 and C12, generate the lowest current responses and show significant distortion and widening of peaks as the scan rate increases, indicating limited electroactive surface area, higher internal resistance, and inefficient ion diffusion pathways [31]. This clear performance gradient C6 >> C10 ≈, C8 > C12 ≈, C2 underscores the impactful role of material synthesis and structural design on electrochemical behaviour, positioning C6 as the most promising candidate for high-performance supercapacitor applications where both high energy and power density are crucial.

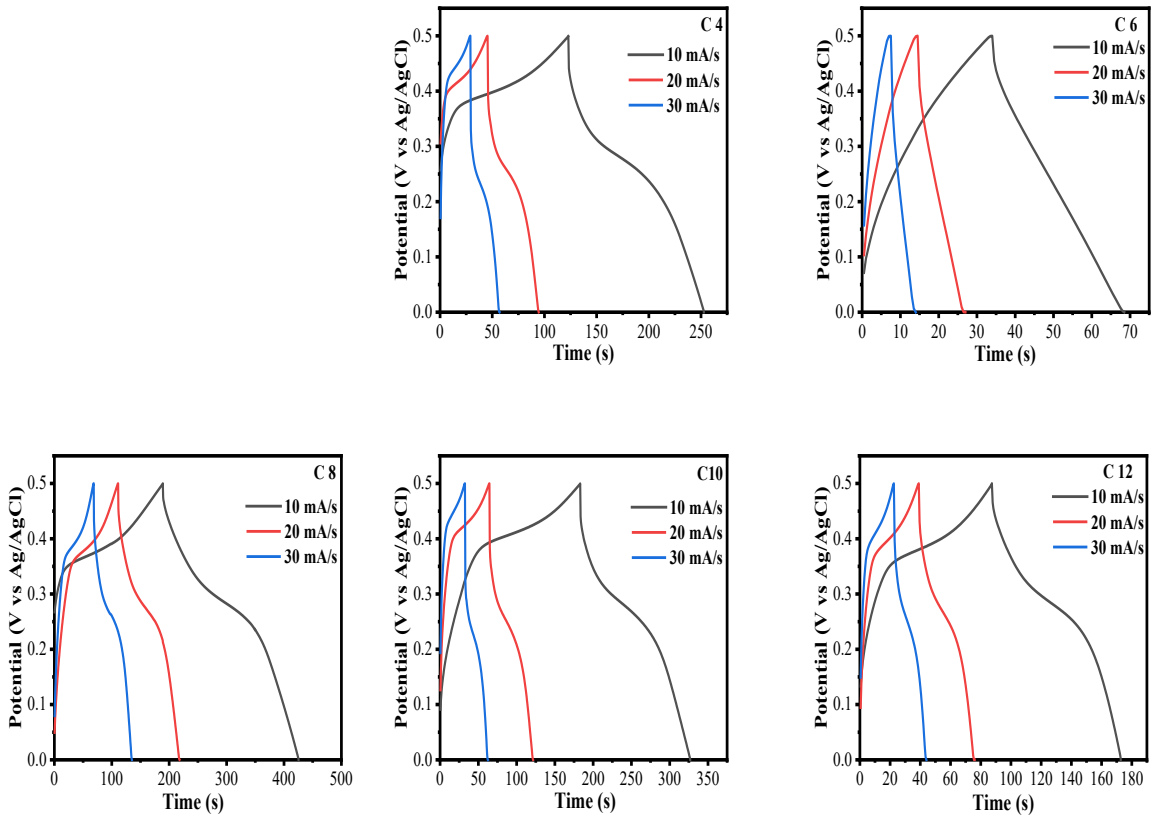

Fig.6. GCD of CdO prepared by Hydrothermal method for different hours

The galvanostatic charge–discharge (GCD) curves (Fig.6) of CdO electrodes prepared on nickel foam at different hydrothermal durations (C4, C6, C8, C10, and C12 hours) measured at current densities of 10, 20, and 30 mA/s. All samples exhibit non-linear charge–discharge profiles with distinct plateaus, confirming pseudocapacitive behaviour arising from Faradaic redox reactions [32]. At a lower current density (10 mA/s), the discharge times are longer, indicating higher specific capacitance due to sufficient ion diffusion and utilisation of the active material [33]. In contrast, at higher current densities (20 and 30 mA/s), the discharge times decrease significantly because of increased internal resistance and limited ion penetration into the electrode structure [34]. Among the samples, C8 shows the longest discharge time and the most stable GCD curve at 10 mA/s, suggesting an optimal hydrothermal growth time that yields improved electrochemical performance [35]. This indicates that the hydrothermal duration strongly influences the mass loading, morphology, and capacitive properties of CdO electrodes [36-38]

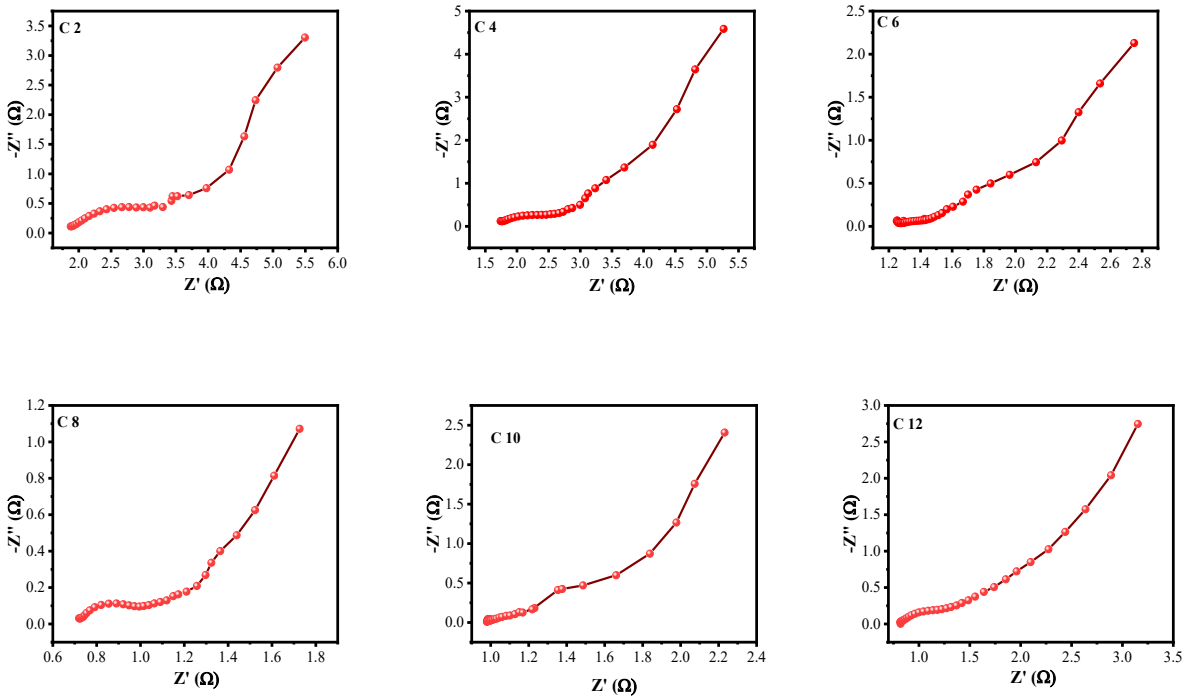

Fig.7.EIS of CdO prepared by Hydrothermal method for different hours

The plots represent the electrochemical impedance spectroscopy (EIS) Nyquist plots of CdO electrodes synthesised on nickel foam at different hydrothermal growth durations. Each spectrum shows a small semicircle in the high-frequency region, corresponding to the charge-transfer resistance (R_{ct}) at the electrode–electrolyte interface, followed by a straight line in the low-frequency region, which indicates the Warburg impedance related to ion diffusion within the porous electrode material [41]. The relatively small semicircle diameters suggest low R_{ct} values, implying good electrical conductivity and fast charge-transfer kinetics. The linear regions with slopes approaching 45° reflect efficient ion diffusion pathways and porous microstructures favourable for electrochemical storage [42]. The trend indicates that prolonged hydrothermal time influences the electrode's morphology and surface area, thereby affecting the impedance response [43]. Lower R_{ct} and more vertical low-frequency lines correspond to better capacitive behaviour, which is essential for high-performance supercapacitors. These results are consistent with previous reports on transition metal oxide–based electrodes where optimised nanostructures enhance charge transport and ion accessibility [44].

4. Conclusions

The CdO thin films successfully synthesised on Ni foam via the hydrothermal method exhibit clear dependence on growth duration in terms of structural, vibrational, and electrochemical properties. FTIR analysis confirms the formation of pure CdO, with characteristic Cd–O stretching vibrations observed in the 400–600 cm^{-1} region. The absence of significant peaks in the 1000–4000 cm^{-1} range indicates minimal organic impurities. Increasing hydrothermal time improves spectral smoothness and transmittance, suggesting enhanced crystallinity and reduced defect density. This study demonstrates that hydrothermal growth time plays a crucial role in tailoring the structural and electrochemical properties of CdO thin film electrodes on Ni foam. Short growth times (2–4 h) lead to incomplete crystallisation, higher defect density, and poor electrochemical performance. Excessively long durations (10–12 h) may cause overgrowth, agglomeration, or reduced active surface area, leading to performance degradation. An optimal growth window (6–8 h) provides a balanced morphology with improved crystallinity, better porosity, and enhanced electroactive surface area.

Declaration of competing interest

The author PDH affirms that there are no financial or personal interests that could be perceived to have influenced the research reported in this paper.

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