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Graphene Oxide/Cadmium-Doped NiO Nanocomposites as High-Performance Electrode Materials for Supercapacitors

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Abstract

GO-based nanocomposite containing Cd doped nickel oxide (Cd-NiO) was synthesized for their possible applications as efficient electrodes in supercapacitors. It was presumed that doping of Cd into NiO and utilization of GO and Ni-MOF (BDC) can facilitate enhanced conductivity, active sites, and charge transfer. The characterizations based on XRD, FTIR, SEM, TEM, and XPS techniques revealed the presence of nanoparticles of Cd doped NiO incorporated by graphene oxide sheets to form a porous and interconnected network appropriate for ion diffusion and electron transfer. Electrochemical studies, including CV, Galvanostatic charge-discharge, EIS, and cycle stability, proved significant pseudocapacitance properties of the GO/Cd-NiO electrode. It showed excellent capacitive behavior with a specific capacitance value of 1162 F g⁻¹ and demonstrated outstanding cycling stability with a 94% capacitance maintenance of 5000 cycles, which proved the positive synergy from the Cd doping and the conductive GO matrix.

Keywords: Graphene oxide, NiO, Nanocomposites, Ni-MOF, BDC, Supercapacitors, Specific capacitance, Electrochemical performance

1. Introduction

The increasing application of portable electronic devices, electric vehicles, wearables, and energy generation systems has made it necessary for there to be increased use of electrochemical energy storage devices that can provide large amount of power density, fast charge-discharge rate, long lifetime of operation, and good cycle stability [1-3]. Super-capacitors have gained popularity among other energy storage devices due to their ability to fill the gap existing between regular capacitors and batteries. Their capability in providing high power and fast charge/discharge along with long-term cycle makes super-capacitors ideal for use in pulse power systems, regenerative braking, back-up power sources, wearables, and hybrid energy storage devices [1-4]. The limitation in the energy density of conventional super-capacitors hinders their usage in energy storage systems [4,5]. Thus, considerable research work has been conducted in order to develop electrode materials possessing high electrochemical activity,

high electrical conductivity, fast ions transportation, and high stability [5–7]. Supercapacitors' electrochemical performance depends primarily on physical and chemical properties of the electrode materials. According to the charge storage principle, there exist two types of supercapacitors that include electric double-layer capacitors (EDLCs) and pseudocapacitors. The former accumulate energy by means of a reversible electrostatic adsorption of ions of electrolytes on electrode surfaces while the latter include fast reversible Faradic reactions taking place at or close to the electrode surfaces [6–8]. There are numerous examples of carbon-based materials that can be utilized as EDLC electrodes since they are characterized by high specific surface area, appropriate electrical and chemical properties, and include activated carbon, carbon nanotubes, graphene, and graphene-based materials [9,10]. Transition metal oxides and hydroxides (RuO_2 , MnO_2 , Co_3O_4 , $\text{Ni}(\text{OH})_2$, NiO , etc.) represent pseudocapacitor electrode materials providing much higher capacitances by means of pseudocapacitive redox reactions [11–13]. Thus, creating electrode materials based on carbon-based and transition metals is a perspective way of coupling the processes of electric double-layer formation and redox reactions [12,14].

Among numerous transition-metal oxides, nickel oxide (NiO) is one of the most promising materials to be used for a pseudocapacitive electrode due to its relative inexpensiveness, abundance, stability, and favorable electrochemical behavior in alkaline electrolytes [13–16]. The NiO is a p-type transition metal oxide that exhibits reversible $\text{Ni}^{2+}/\text{Ni}^{3+}$ redox reaction and possesses a high theoretical specific capacitance value of about 2573 F g^{-1} [14,15]. In the alkaline electrochemical environment, its charge storage mechanism is usually related to the reversible transformation of NiO , $\text{Ni}(\text{OH})_2$, and NiOOH [15,16]. To take advantage of these features, NiO has been fabricated into different nanostructures, such as nanoparticles, nanorods, nanoflakes, nanosheets, hollow structures, and hierarchical porous nanostructure [16–18]. These kinds of nanostructures may increase the available electrochemical surface area and reduce ion diffusion distance. However, the electrochemical performance of pristine NiO is still much inferior to its theoretical value owing to its poor electrical conductivity, slow charge transfer kinetics, low utilization of active sites, and easy agglomeration of particles during fabrication and cycling processes [17–19]. Improving the electronic transport properties of the structure of NiO thus forms one of the major challenges to developing high-performance NiO -based supercapacitors.

Various material engineering methods such as morphology tuning, fabrication of porous structures, defect engineering, heterostructure synthesis, metal ion doping and association with electrically conductive carbon-based architectures have been developed to circumvent the natural shortcomings of NiO [18–20]. Among these techniques, metal ion doping is a feasible means to tune the crystal structure and electronic environment of NiO . The introduction of foreign ions can influence the lattice parameters, crystallite size, defects, carrier transport, and

electrochemically active sites [19,20]. More importantly, the introduction of aliovalent or mismatched ions can result in localized structural changes and affect the electrochemical performance of the oxide host. In fact, previous works have shown the possibility of improving charge transfer properties and electrochemical reactivity of doped NiO electrodes at an appropriate level of doping content and nanostructures [18–20]. In the above context, Cd^{2+} is assumed as the dopant functional for manipulation of the structural and electrochemical properties of NiO. The ionic radius and electronic nature of Cd^{2+} ions are different from those of Ni^{2+} and thus incorporation and/or interaction of Cd^{2+} ions with the NiO structure could be responsible for structural distortion, crystallite growth and defect chemistry in the oxide. These aspects may have an effect on the accessibility of the electrochemically active sites and electrolyte ion mobility and charge transfer kinetics in the system. Previous works on Cd-containing NiO systems have illustrated the significant effect of Cd incorporation on the morphology and electrochemical behavior of electrode materials based on NiO [19–21]. For instance, the Cd–Zn co-doped NiO nanorods have shown morphology dependence for enhancement of electrochemical performance, which implies the role of Cd incorporation as a way for compositional engineering of NiO-based supercapacitors [20]. Nevertheless, the use of Cd needs to be scientifically handled since cadmium is known to be a highly toxic heavy metal. Thus, in the current study, Cd is used as a controlled low level dopant for basic materials engineering instead of being the primary electrode material.

Despite the fact that Cd incorporation can change the structure and electrochemical performance of NiO, the use of Cd-doped NiO alone might be hampered by its lack of sufficient electrical conductivity and electrochemical site utilization at high current densities. Combination with the two-dimensional carbon matrix offers a possible route to tackle such issues. Graphene oxide (GO), due to its two-dimensional structure, high specific surface area, flexibility, and numerous functional groups containing oxygen atoms [21–23], has become increasingly popular as a versatile material for energy storage applications. Hydroxyl, epoxy, carbonyl, and carboxyl groups of GO sheets serve as favorable nucleation sites for metal oxides. Moreover, due to the extended two-dimensional structure, GO can provide a pathway for charge transportation and electrolyte-ion diffusion. Mechanical flexibility can be used for structural stabilization in repeated cycles of electrochemical reactions [21–24]. Thus, graphene oxide and graphene-related materials have been widely used as components for hybrid structures with transition-metal oxides. Such materials can provide increased electrical conductivity, surface availability, and cycling stability [22–25]. Specifically, NiO/GO and NiO/graphene-based structures utilize pseudocapacitive properties of NiO and electrical

double-layer and conductive networks of carbon-based materials [23-28]. Carbon-based structure can facilitate the dispersion of NiO nanoparticles, reduce the distance of electron transportation, increase electrolyte accessibility, and provide mechanical stabilization in repeated redox processes. Experimental studies have demonstrated that inclusion of GO into NiO-based electrodes can have a considerable impact on the interfacial structure and capacitive properties of such hybrid materials [24-26]. In particular, addition of graphene oxide can increase the specific capacitance of nickel oxide-based materials through the changes in the structure and interfacial charge accumulation [25]. Recently, GO-NiO nanocomposites have shown the synergetic effect of two components in electrochemical energy storage [26].

Despite these developments, most of the studies conducted so far have investigated either metal-ion doped NiO or graphene/GO-NiO hybrid systems. The combined use of Cd doping and interface engineering using GO enables one to design a novel multifunctional hybrid system wherein both materials serve their respective roles. While Cd doping could control the structural and defect environment of the NiO material, GO is expected to provide a 2-D support for dispersing nanoparticles as well as enhancing ion and electron transport properties. However, the correlation between Cd doping, structural changes, GO-based interface engineering, and their influence on charge storage processes is poorly understood. Specifically, the link between crystal structure, surface chemistry, charge transfer resistance, reversibility of reactions, and the extent of surface and diffusion controlled charge storage has not been explored extensively in Cd doped graphene/NiO hybrids.

Research gap and novelty of the present work

The main research problem that is to be considered and solved through this study involves the inadequacy of the study of the synergistic effect of the combined use of Cd-modified NiO and GO as an integral nanocomposite electrode material. Although it was demonstrated that systems based on the Cd-containing NiO and GO/NiO composite materials exhibited an improvement in electrochemical characteristics, it has not been sufficiently studied how the structural modification induced by the Cd element and the interfacial charge transport mediated by GO affect the pseudocapacitance of NiO. Hence, the novelty of this study can be attributed to the rational design of a nanocomposite GO/Cd-NiO through the systematic alteration of NiO by using the two-dimensional network of oxygen functionalized graphene oxide as the anchoring platform. Herein, unlike treating GO as merely a conductive filler, the study uses the oxygen-functionalized network of GO as an anchoring matrix for Cd altered NiO nanoparticles in order to achieve enhanced particle distribution and efficient electrochemical interfaces. The

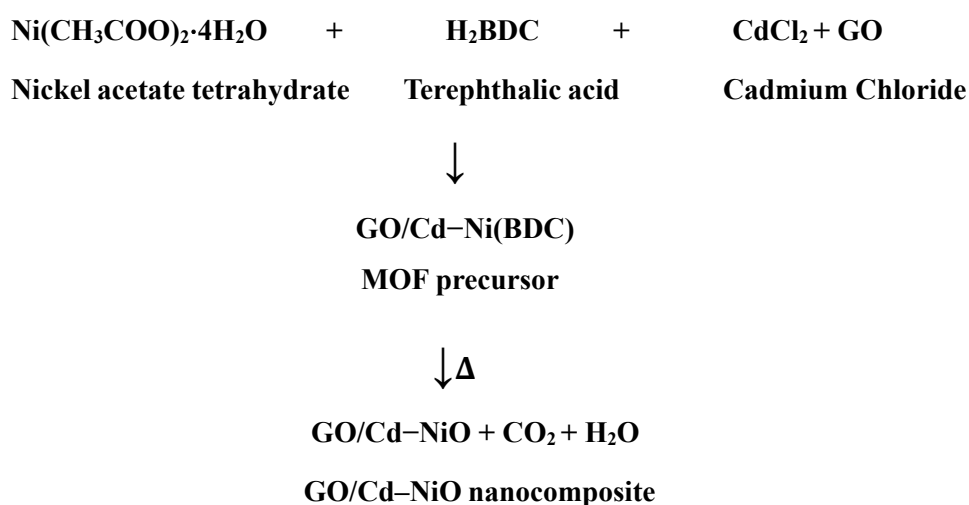
investigation is performed on the structural, spectroscopic, morphological, surface, and electrochemical properties of the formed nanocomposite in order to correlate the architecture of the material with its electrochemical storage capability.

Thus, this work aims at the development of GO/Cd-NiO nanocomposites as a promising candidate for pseudocapacitive electrodes in supercapacitors. This assumption is based on the ability to control the physicochemical properties of NiO through the use of cadmium modification and application of GO as a versatile two-dimensional platform capable of contributing to better dispersion of the particles, ion accessibility, conductivity, and stability of the electrode material. These two substances are supposed to work together to maximize the use of electrochemically active sites as well as charge storage kinetics of NiO-based material. At the same time, the environmental concerns associated with the use of cadmium will be taken into account.

2. Materials and Synthesis Methodology:

Synthesis of GO/Cd-NiO nanocomposite via Ni-BDC MOF precursor

A 0.5 M solution of the nickel precursor was prepared by dissolution of 0.05 mol of nickel acetate tetrahydrate (12.44 g) in 100 mL of a DMF/ethanol (1:1, v/v) solvent mixture under constant magnetic stirring. Additionally, 0.05 mol of terephthalic acid (H_2BDC , 8.31 g) was dissolved in DMF and then added drop-wise to the nickel precursor solution, forming Ni-BDC metal-organic framework precursor. Next, cadmium chloride (5 mol% relative to Ni) was dissolved in deionized water and slowly introduced into the reaction mixture under intensive stirring. Afterwards, graphene oxide (GO, 100 mg) dissolved in deionized water by ultrasonic treatment during 1 hour was added to the solution, ensuring uniform deposition of the Cadmium-containing Ni-BDC precursor onto the surface of GO. The graphical representation as shown in the Fig 1.



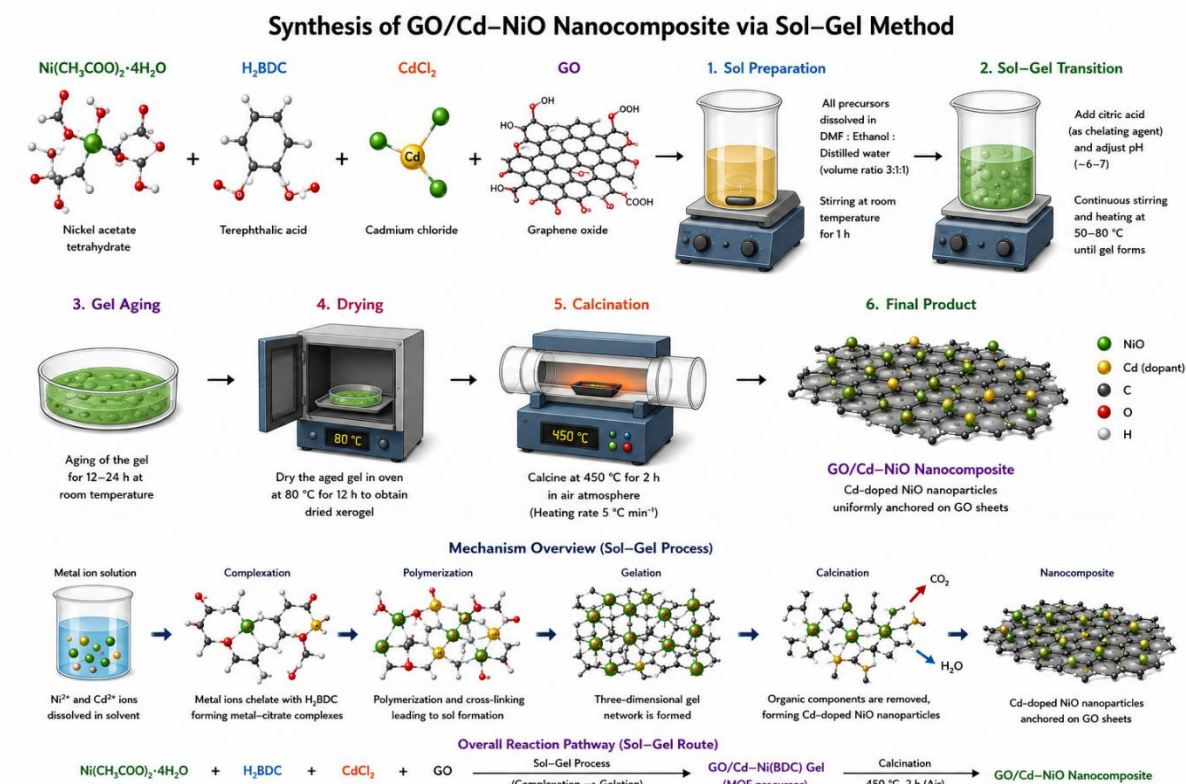
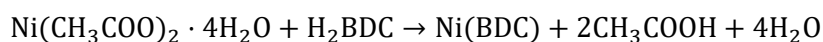


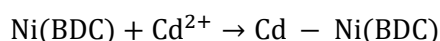
Fig 1. Graphical representation of Graphene Oxide/ Cadmium-Doped Nickel-Oxide nanoparticles

The resulting homogeneous mixture was placed in a Teflon lined stainless steel autoclave and kept at 120 °C for 12 h. The precipitate obtained was then cooled to room temperature, separated by centrifugation, rinsed extensively with ethanol and distilled water to eliminate any remaining reactants, and dried for 12 hours at 80 °C. The dried GO/Cd–Ni–BDC complex was calcined in air between 450 to 500 °C for 2 hours with a heating rate of 5 °C/min to afford the porous GO/Cd–NiO nanocomposite in which the decomposition of the Ni–BDC complex resulted in the formation of a high surface area Cd-doped NiO structure attached to the GO sheets.

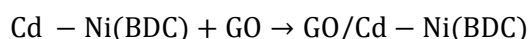
Formation of Ni–BDC MOF



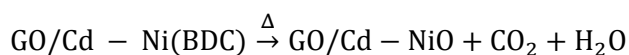
Cd incorporation into the MOF

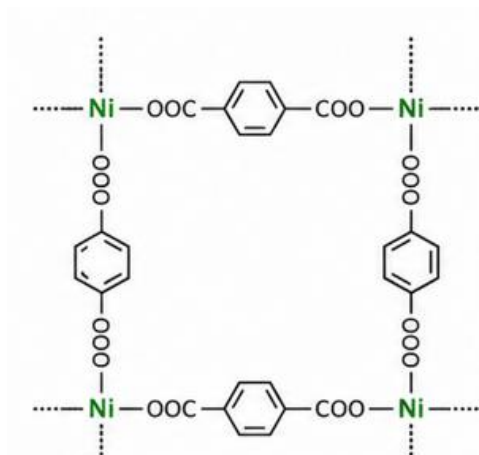


Growth on graphene oxide



Calcination





**2D Representation of Ni-BDC
Coordination Network**



**Octahedral Coordination Environment of
Ni²⁺ by BDC²⁻ Ligands**



**Ni-BDC Metal Organic Framework
(Coordination Polymer)**

2.1 Electrode Fabrication

Electrochemical investigations were carried out using a conventional three-electrode setup, employing 3 M KOH aqueous electrolyte. The working electrode consisted of copper doped nickel oxide coated nickel foam, while platinum wire and Ag/AgCl were used as the counter electrode and reference electrode, respectively. The working electrode was prepared by dispersing GO/Cd-doped NiO, carbon black, and PVDF in NMP in the weight ratio of 80:10:10. The dispersed material was then coated on nickel foam and dried at 80 °C. The loading of the active material was restricted to 2 mg cm⁻². Electrochemical investigations like CV and Galvanostatic charge/discharge were done in the potential range of -0.2 to 0.4 V vs Ag/AgCl.

3. Results and Discussion

3.1 X-ray diffraction analysis:

X-Ray diffraction (XRD) was utilized for the investigation of the crystal structure and phase purity of the graphene oxide-cadmium doped nickel oxide (GO/Cd-NiO) nano-composite. The obtained diffraction spectrum exhibits some prominent peaks which belong to the cubic phase of NiO, showing the existence of crystalline nickel oxide in the sample. These peaks are located at the angles of $2\theta = 37.2^\circ, 43.3^\circ, 62.9^\circ, 75.4^\circ, \text{ and } 79.4^\circ$, corresponding to the planes (111),

(200), (220), (311), and (222) of cubic NiO, respectively, matching with the reported XRD patterns for NiO [29]. For the GO/Cd-NiO composite, a slight shift and broadening in the reflections of NiO were seen, which can be explained by the inclusion of Cd²⁺ ions within the NiO lattice, leading to lattice strain or distortion owing to the difference in the ionic radii of Cd²⁺ and Ni²⁺ ions [30]. The existence of GO was confirmed by a weak or broad peak at about 10–12° for the (001) peak of GO or at about 24–26° for the (002) peak. The weakening of the GO peak can be explained by the exfoliation and dispersion of GO sheets with Cd-NiO nanoparticles on the GO surface. The average crystallite size (D) was calculated from the most intense diffraction peak using the Debye–Scherrer equation:

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

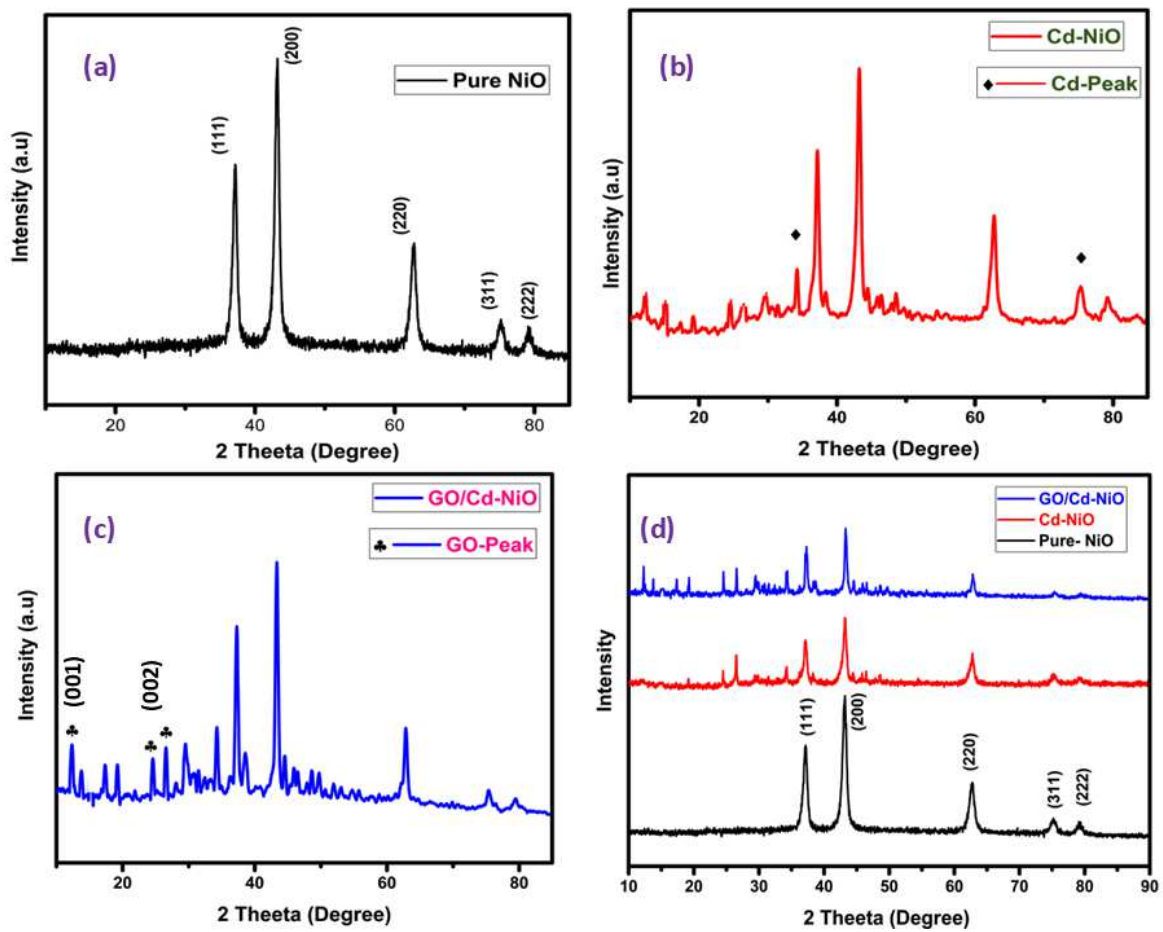


Fig 2. (a-d) represents the XRD peaks confirmation of GO/Cd-NiO nanoparticles

The crystallite size of NiO was determined to be 19.75 nm but was reduced to 11.25 nm when GO was added. It can be noted that the presence of GO has an impact on the crystal growth of NiO and contributes to the development of smaller nanoparticles as shown in the table 1 (a & b). The smaller crystal size helps in supercapacitor applications since it allows increasing the electrochemically active surface area and ion diffusion distance. Besides, the conductive properties of GO contribute to electron

transport and agglomeration inhibition of nanoparticles as shown in the Fig 2. So that the obtained GO/Cd-NiO composites gives good specific capacitance and cyclic stability.

Table 1 (a). XRD-derived structural parameters of pure NiO

S. No.	2 θ (°)	FWHM (°)	d-spacing (nm)	Crystallite size, D (nm)	Microstrain, ϵ	Dislocation density, δ (nm ⁻²)
1	37.13	0.3865	0.24275	22.65	0.0050	0.0019
2	43.16	0.54914	0.21016	16.35	0.0060	0.0037
3	51.73	0.55193	0.17717	16.76	0.0049	0.0035
4	62.71	0.37191	0.14855	26.21	0.0026	0.0014
5	75.17	0.62548	0.12673	16.79	0.0035	0.0035
Average	—	—	—	19.75	0.0044	0.0028

Table 1 (b). XRD-derived structural parameters of GO/Cd-doped NiO nanocomposites

S. No.	2 θ (°)	FWHM (°)	d-spacing (nm)	Crystallite size, D (nm)	Microstrain, ϵ	Dislocation density, δ (nm ⁻²)
1	12.30	0.58883	0.72102	14.22	0.0238	0.0049
2	24.60	0.77600	0.36281	10.98	0.0155	0.0082
3	24.60	0.68762	0.36281	0.13	0.0137	51.0459
4	37.27	0.45728	0.24189	19.21	0.0059	0.0027
5	43.33	0.49820	0.20938	18.29	0.0054	0.0029
6	62.84	0.61901	0.14827	15.76	0.0044	0.0040
7	83.11	75.73814	0.11652	0.14	0.3727	46.3274
Average	—	—	—	11.25	0.0630	13.9137

The decrease in average crystallite size from 19.75 nm for pure NiO to 11.25 nm for GO/Cd-doped NiO showed that Cd doping and GO addition limited crystal growth and resulted in formation of a smaller nanostructure. The decrease in average crystallite size enlarged the electrochemically available surface area and reduced ion-diffusion pathways, which enhanced electrolyte availability and utilization of redox centers. The average microstrain increased from 0.004451 for pure NiO to 0.063096 for GO/Cd-doped NiO, which confirmed the significant increase in lattice strain following Cd doping and GO addition. This increase in lattice strain altered the local crystalline environment and formed structural defects, which participated in the formation of new electrochemically active sites. In a similar way, the dislocation density increased and pointed at the higher presence of crystallographic defects in the structure of GO/Cd-doped NiO nanocomposite. Such defects facilitated charge transfer and electrolyte

ions' availability to the active surface. Thus, the influence of decreased crystallite size, increased microstrain, and higher dislocation density resulted in the improvement of ion/electron transport, increased pseudocapacitance and higher specific capacitance of the GO/Cd-doped NiO electrode in comparison with the pure NiO one [31].

3.2 UV-Visible Spectroscopy Analysis

Fig. 3. shows the UV-Visible spectra of the pure NiO and GO/Cd-NiO nano-composites. The wide absorption band of pure NiO is observed around 360-380 nm, which is attributed to the intrinsic charge transfer process between O^{2-} (2p) and Ni^{2+} (3d) orbitals. Beyond the absorption edge, the absorbance value decreases gradually with an increase in wavelength, implying that the energy gap of pure NiO is quite high, and hence the ability to absorb the visible light is less. On the other hand, the GO/Cd-NiO nanocomposite demonstrates an increased absorption intensity in the UV region with absorption peaks at 230-240 nm and 360-390 nm. An absorption peak of 230-240 nm relates to the $\pi \rightarrow \pi^*$ transition in the conjugated C=C double bond of the graphene oxide, while the broad absorption of 360-390 nm refers to the charge transfer process of the Cd-doped NiO [32]. Compared to the pure NiO sample, the absorption edge of the GO/Cd-NiO nanocomposite has been red-shifted to the long-wavelength range. Such absorption improvement and red-shifting edge are advantageous for enhancing light absorption capacity and increasing electron transport efficiency, which is favorable for fast charge transfer in the electrochemical energy storage system [33].

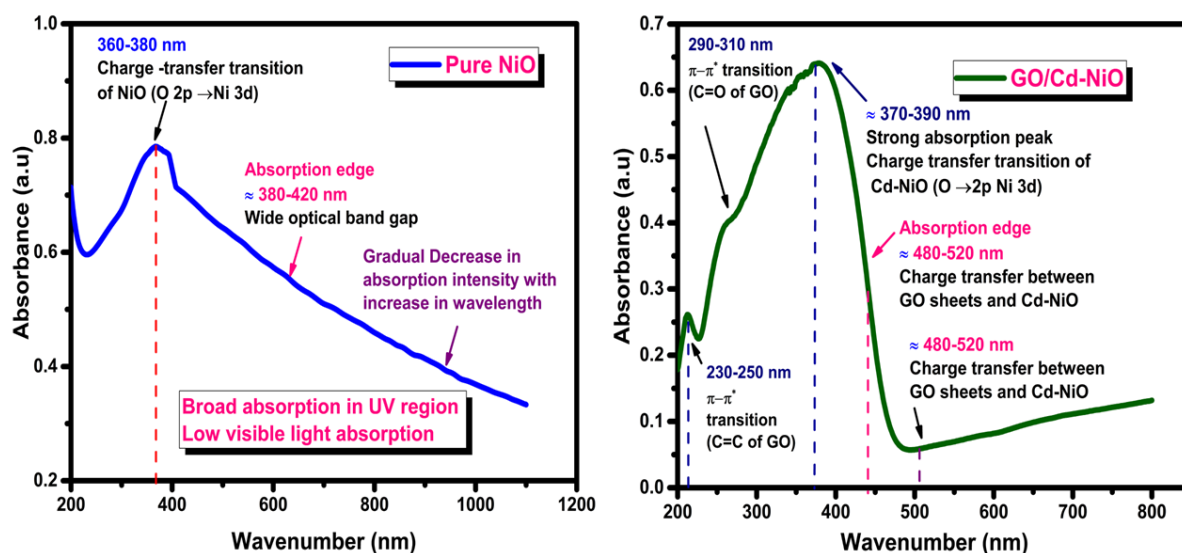


Fig 3. Shows the absorbance of pure NiO and GO/Cd-NiO nanoparticles.

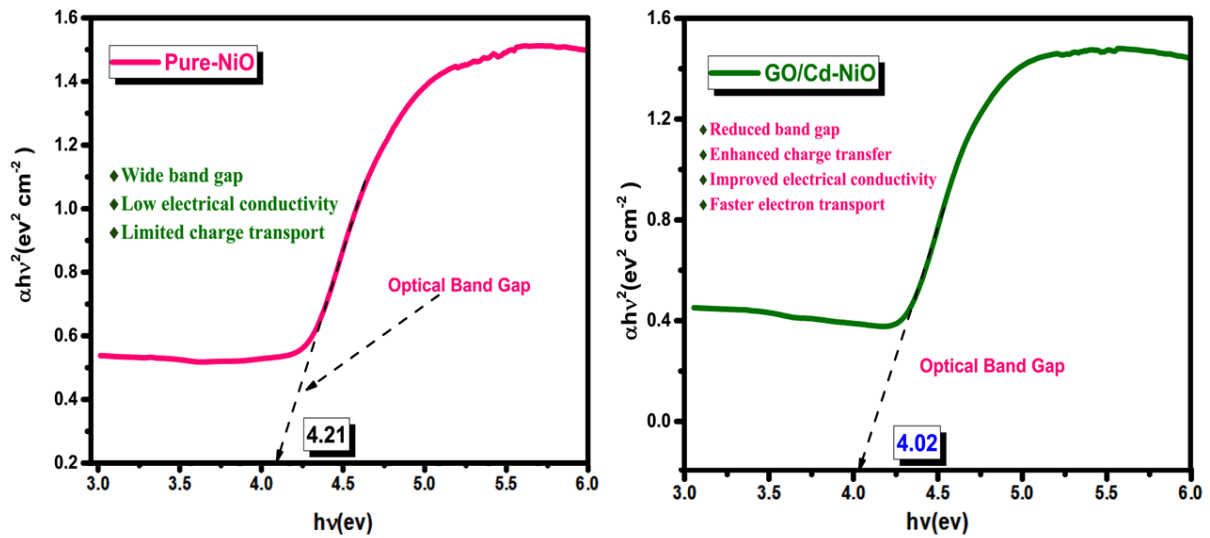


Fig 4. Shows the variation of band gap for pure NiO and GO/Cd-NiO nanoparticles.

3.3 Thermogravimetric Analysis (TGA)

Thermal stability of the GO/Cd-NiO nanocomposite was evaluated through thermogravimetric analysis (TGA), the thermogram of which is illustrated in Fig. 5. The TGA profile demonstrates multi-stage weight loss pattern, suggesting that the physical adsorption of different chemical species and the decomposition of oxygen functional groups from graphene oxide occur in an incremental fashion. The first stage of weight loss, which occurs up to about 7-10%, takes place at temperatures lower than 150 °C, and is related to evaporation of physically adsorbed water from the surface of the sample [34]. The second stage of weight loss takes place at temperatures between 150 and 300 °C, and is due to the decomposition of oxygen functional groups (hydroxyl, epoxy, carboxyl). The largest amount of weight loss takes place at temperatures of approximately 320-380 °C where there is a sharp drop in weight from around 80% to 40%. This is because of thermal reduction of graphene oxide, removal of any residual organic entities produced by the synthesis procedure, and thermal decomposition of carbonaceous entities. At temperatures above 400 °C, weight loss occurs steadily and becomes almost constant with a weight loss of about 30% at 700 °C which signifies the creation of a thermally stable inorganic framework of Cd-NiO [35]. The large residual weight shows good thermal stability of the metal oxide compound and hence successful creation of the GO/Cd-NiO nanocomposite.

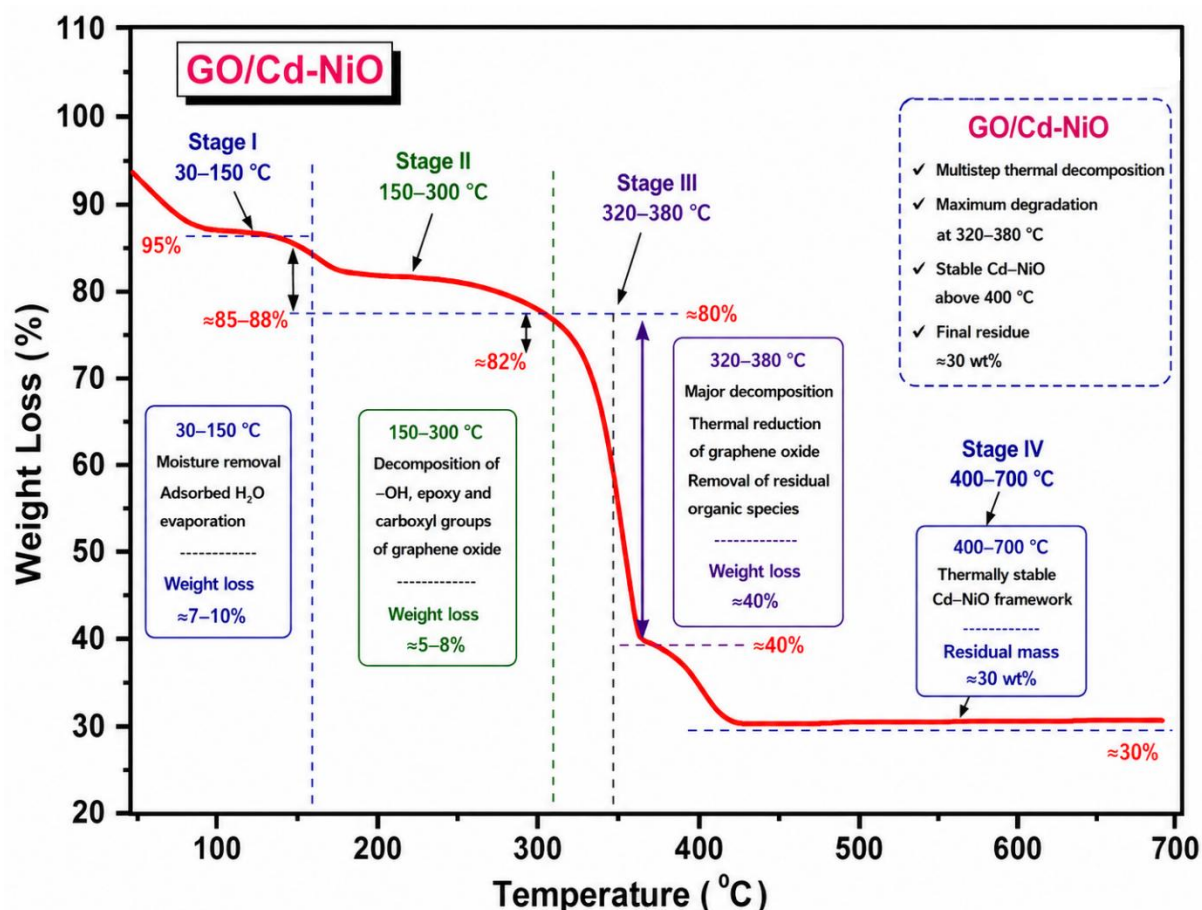


Fig 5. Shows the variation of weight loss for GO/Cd-NiO nanoparticles.

3.4 FT-IR spectroscopy analysis:

FTIR analysis was used for characterization of the distinctive functional groups existing in the synthesized GO/Cd-NiO nanocomposites and confirmation of the presence of both graphene oxide and Cd-doped NiO in the nanocomposite material. The wide band located at about 3400-3450 cm^{-1} is usually attributed to the vibration of surface OH-groups and absorbed H₂O molecules [36]. Absorption bands recorded at about 1720-1740 cm^{-1} , 1610-1630 cm^{-1} , 1380-1410 cm^{-1} , and 1040-1120 cm^{-1} correspond to oxygen-containing groups of graphene oxide such as C=O stretching of the carboxyl groups, C=C skeleton vibration / H-O-H bending, C-OH deformation and C-O-C/C-O stretching vibrations of the epoxy/alkoxy groups, respectively. The appearance of the last bands is evidence of incorporation of GO in the nanocomposite. Also, the low-frequency absorption band at about 420-470 cm^{-1} can be related to the Ni-O stretching vibration proving the formation of nickel oxide [37] as shown in the Fig. 6. As for the doped samples, minor shifting of the metal-oxygen vibration band may happen due to local environment modification induced by cadmium doping of nickel oxide. The simultaneous existence of GO-related oxygen functions and the Ni-O vibrations evidences of successful synthesis of GO/Cd-NiO nanocomposite, since graphene oxide is characterized by numerous oxygenated surface groups providing nanoparticle attachment.

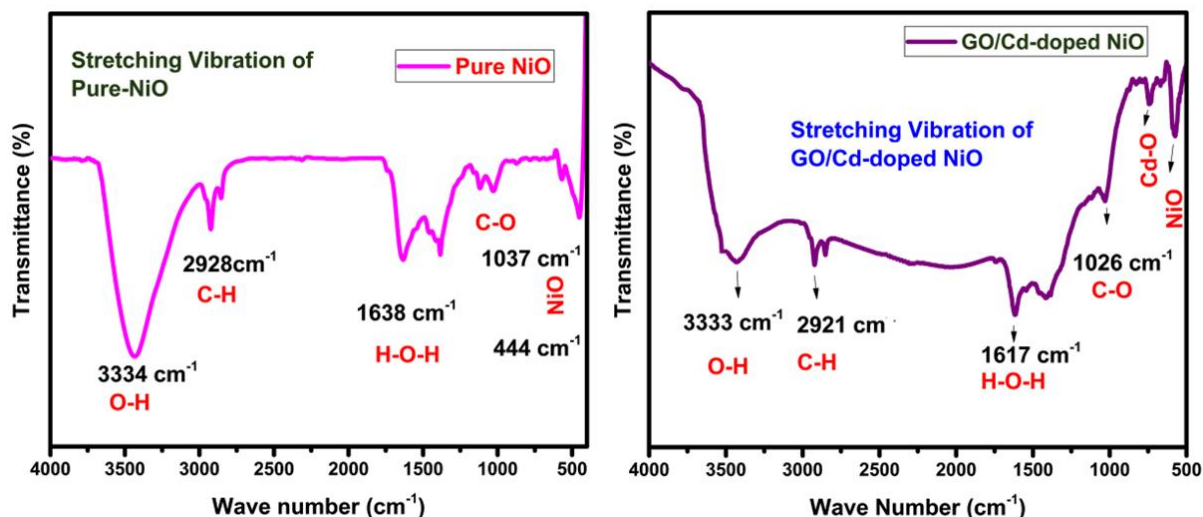


Fig 6. Shows the Stretching vibrations for pure and GO/Cd-NiO nanoparticles.

3.5 FE-SEM Studies:

SEM analysis was performed to characterize the surface morphology and microstructures of the GO/Cd-NiO nanocomposites. The SEM images mostly show a rough, porous, and interconnected surface morphology, which is highly desirable for supercapacitor electrode materials owing to increased specific surface area and easy electrolyte access. Pure NiO has the tendency to form agglomerated particles due to its high surface energy; nevertheless, upon the doping of Cd and inclusion of GO, the resulting morphology turns out to be more open and less dense. Graphene oxide flakes are generally observed in the form of wrinkled and thin-layered structures, acting as an excellent support material for the nucleation and growth of Cd-doped NiO nanoparticles [38].

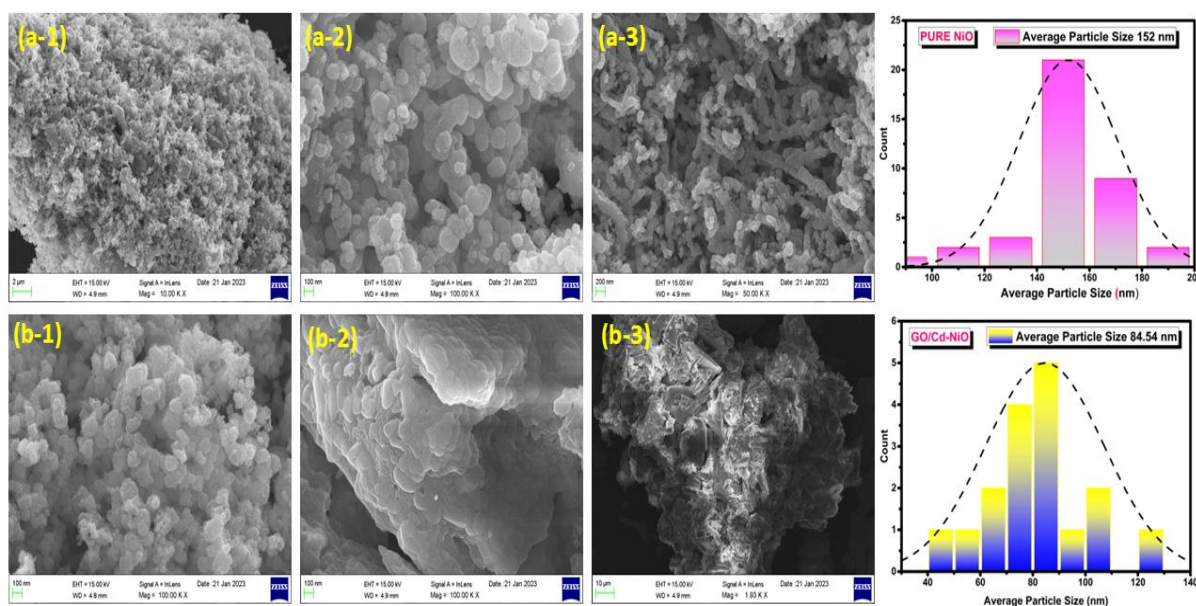


Fig 7. Shows the morphological structures for pure NiO and GO/Cd-NiO nanoparticles.

The Cd-NiO nanoparticles are dispersed on the surface of GO to create a nanoparticle network on GO sheets. Porous and interconnected morphology would prove beneficial in ion transport as well owing to reduced diffusion path and availability of many electroactive sites as shown in the Fig. 7. Moreover, close interaction between Cd-NiO nanoparticles and GO flakes should help in preventing particle agglomeration and improve electrical conductivity of the composite. Thus, the SEM images confirm the creation of a hierarchically organized morphology of nanocomposites [39].

3.6 EDX Analysis

The elemental analysis of the synthesized GO/Cd-NiO nanocomposite was carried out through the energy-dispersive X-ray (EDX) spectroscopic technique. The obtained EDX spectrum of the synthesized nanocomposite is illustrated in Fig. 8. In addition, the EDX result indicates that there exist three major elements in the nanocomposite, namely Ni, Cd, and C, representing the successful synthesis of cadmium doped NiO composite with graphene oxide. The strong characteristic peaks existing at the region of about 0.85 keV and 7.48 keV are attributed to the $L\alpha$ and $K\alpha$ emission lines of Ni atoms, respectively. The two peaks found in the region of 0.40 keV and 3.13 keV are characteristic peaks of Cd atoms' emission lines, respectively. Therefore, the existence of such characteristic peaks confirms the successful doping of the nanocomposite with cadmium. The characteristic peak existing in the region of about 0.28 keV is assigned to the presence of carbon atoms, which originated from the graphene oxide sheets in the nanocomposite. There are no other peaks existing in the obtained EDX pattern, which means that the synthesized nanocomposite does not contain any other elements other than those mentioned.

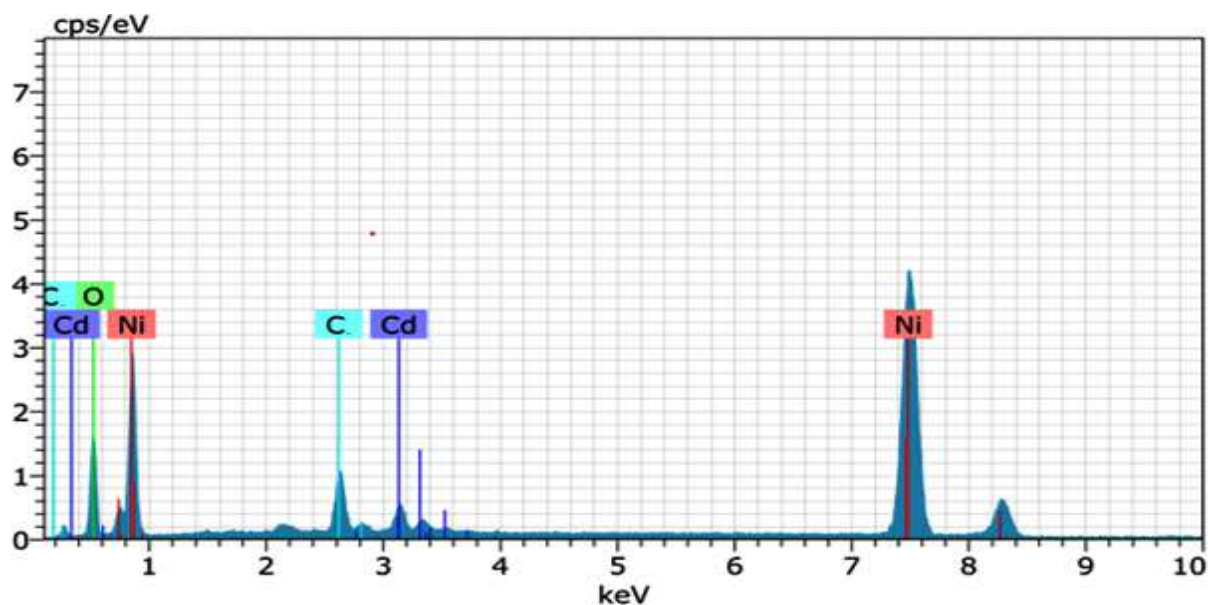


Fig 8. Shows the Elemental Composition of GO/Cd-NiO composites

Table: 1. Elemental Composition of GO/Cd-NiO composites.

El	AN	Series	unn.C [wt.%]	norm.C [wt.%]	Atom.C [at.]	Sigma) [wt.%]
Ni	28	K-series	76.57	81.95	61.85	2.08
O	8	K-series	11.16	11.94	33.07	1.60
C	6	K-series	2.92	3.13	3.91	0.13
Cd	48	L-series	2.79	2.99	1.18	0.12
Total			93.43	100.00	100.00	-

3.7 TEM-Studies:

The transmission electron microscopy technique was used to obtain more information regarding particle size, dispersion, and interfacial interaction between graphene oxide and Cd-doped NiO nanoparticles. The TEM image of the GO/Cd-NiO nanocomposite generally reveals uniformly dispersed nanosized Cd-NiO particles anchored on the transparent and wrinkled GO sheets, indicating the hybrid nature of the material. The Cd-NiO nanoparticles sizes are in the nanometer scale, ranging from 15-30 nm, which is in accordance with the crystallite size obtained from the XRD analysis. The transparent layered GO sheets act as the support matrix, while the darker contrast spots are attributed to the metal oxide nanoparticles dispersed on the carbonaceous structure [40].

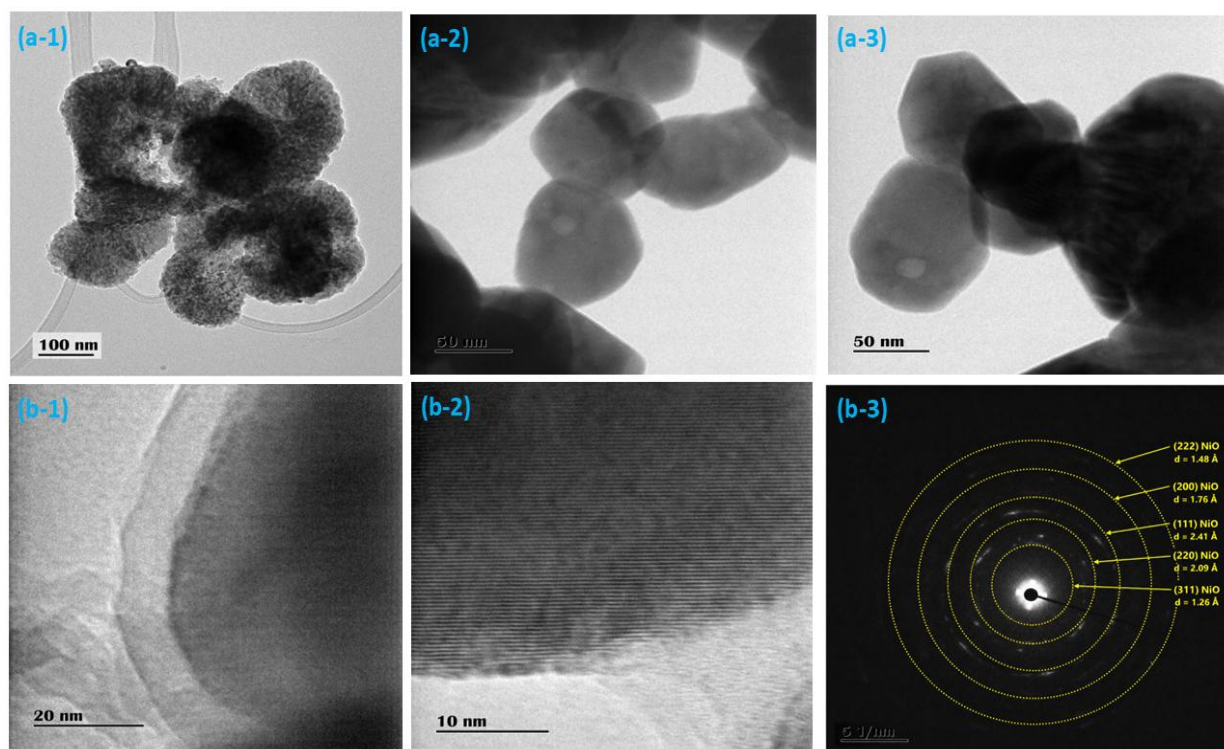


Fig 9. Shows the morphological structures for pure NiO and GO/Cd-NiO nanoparticles.

The uniform dispersion of nanoparticles suggests that the oxygen containing functional groups present on GO act as nucleation centers for the formation of composites. The HRTEM may also be able to show the lattice fringes of the crystallites corresponding to cubic NiO structure, having interplanar spacings of 0.24 nm for the (111) crystal plane and 0.21 nm for the (200) crystal plane as shown in the Fig 9. Overall, the TEM observations demonstrate that GO effectively disperses Cd–NiO nanoparticles and provides a conductive framework that can improve electron transport and structural stability during electrochemical cycling [41].

3.8 X-ray photoelectron spectroscopy (XPS) analysis

XPS analysis used to determine the surface chemistry and oxidation state of the elements in the GO/Cd–NiO nanocomposites. The survey spectra verify that there are indeed Ni, O, Cd, and C, and therefore prove that the hybrid composite has been successfully synthesized without impurities as shown in the Fig 10. The high-resolution Ni 2p spectrum shows two main peaks of Ni 2p_{3/2} and Ni 2p_{1/2} at 862 eV and 867 eV, respectively, together with satellite peaks [42].

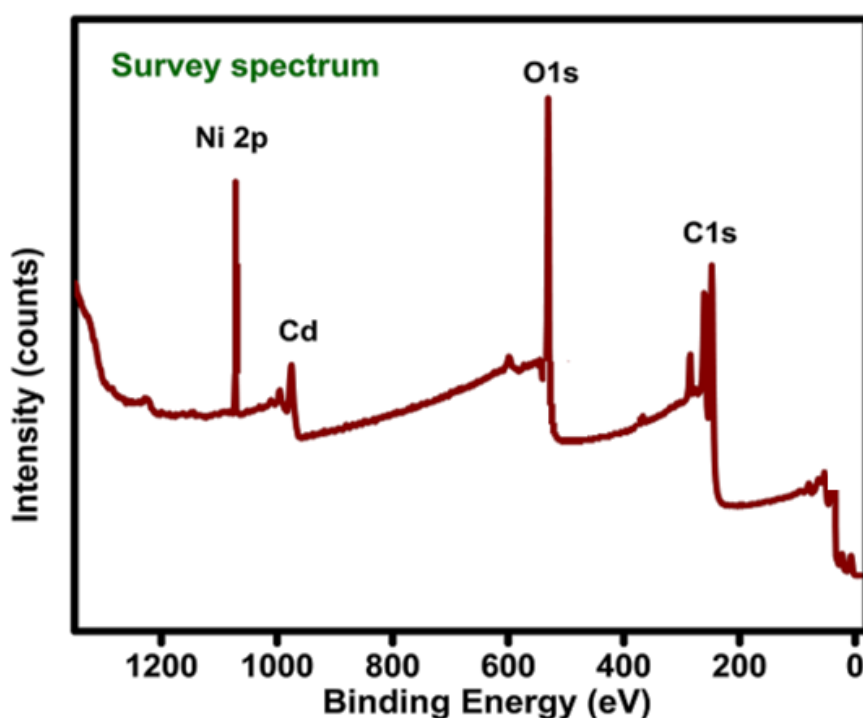


Fig 10. Shows the morphological structures for pure NiO and GO/Cd–NiO nanoparticles.

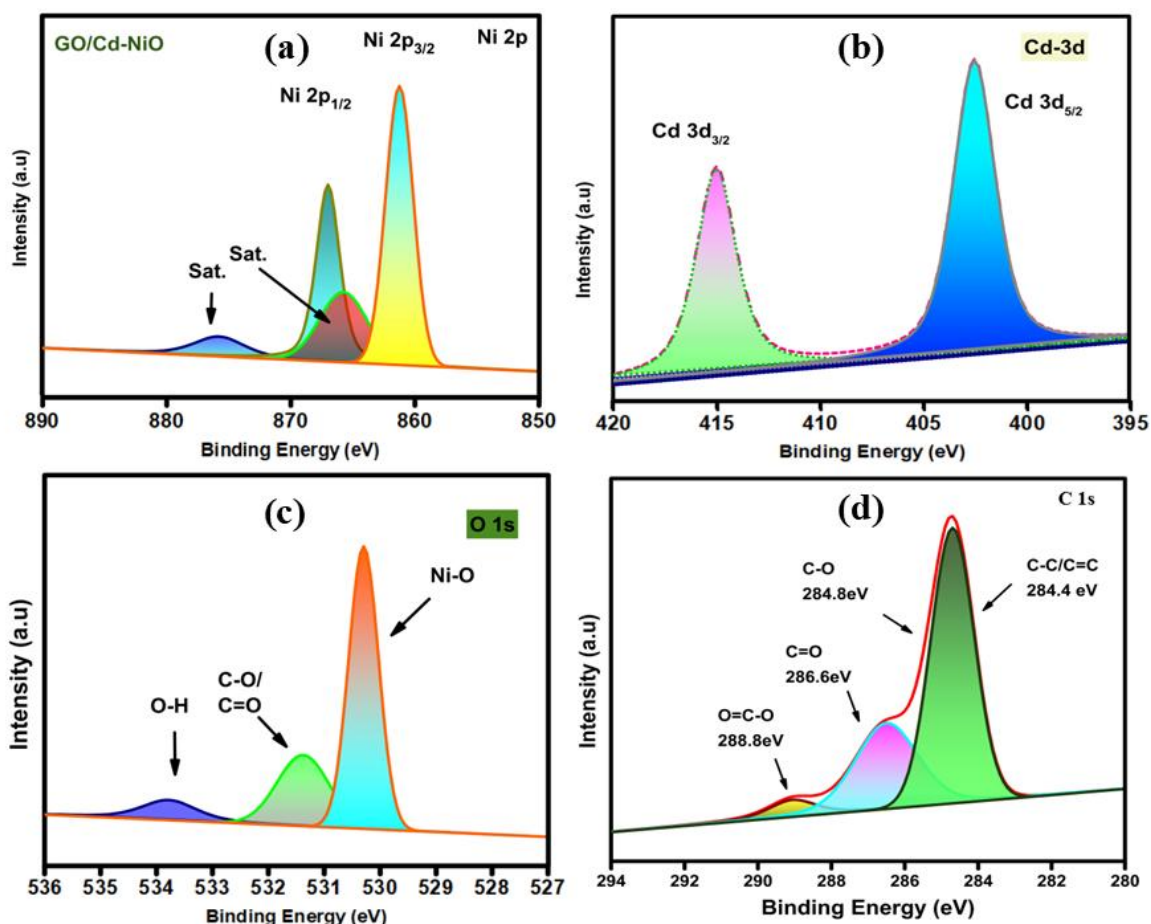


Fig .11 (a-d). Shows the morphological structures for pure NiO and GO/Cd-NiO nanoparticles.

These indicate Ni^{2+} ions in NiO, but small contributions of surface Ni^{3+} ions are possible because of surface oxidation or electrochemical activation. The O 1s spectrum can be fitted using a lattice oxygen peak at 529–530 eV, resulting from Ni–O bond, and other peaks having high binding energy at 531–533 eV, which correspond to hydroxyl groups, defect oxygen, adsorbed water, and oxygen containing functionality of graphene oxide. In the Cd 3d spectrum, there are peaks of Cd 3d_{5/2} and Cd 3d_{3/2} at 402 eV and 414 eV, respectively. The C 1s peak also reveals the binding energy corresponding to C–C/C=C, C–O, C=O, and O–C=O, which confirms the existence of graphene oxide as well as surface-oxygen functionalities as shown in the Fig 11(a-d). In summary, all XPS data clearly indicate the formation of the nanocomposites as well as their surface chemical state that leads to improved electrochemical properties [43].

3.9 Electrochemical Studies

3.9.1 Cyclic voltammetry:

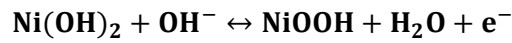
One of the major electrochemical techniques used for studying the charging behavior, redox properties, and capacitive characteristics of supercapacitor electrode materials is the technique of cyclic

voltammetry (CV). In this work, the electrochemical properties of GO/Cd-doped NiO nanocomposite electrode were studied by CV in alkaline electrolyte with the help of a three-electrode cell configuration. The obtained values shows that the resulting CVs consist of a sharp anodic and cathodic peak, which means that the charge storage mechanism takes place due to Faradic pseudocapacitance, rather than ideal double layer capacitance.

The pseudocapacitive property of the NiO electrodes comes about from the oxidation/reduction reaction of nickel species in the alkaline medium. The reaction can be expressed as:



or, in hydrated form,



For the GO/Cd-NiO electrode, the oxidation peak involves the oxidation process of Ni^{2+} to Ni^{3+} , whereas the reduction peak involves the reduction of Ni^{3+} back to Ni^{2+} during the discharge process [44,45]. The existence of redox peaks indicates that the nanocomposite is pseudocapacitive in nature. In comparison with pure NiO, the CV profile of the GO/Cd-NiO have a larger enclosed area which signifies a better charge storage ability through the synergistic effect of graphene oxide and Cd doping. Graphene oxide acts as a conducting medium while Cd doping changes the structure of the NiO lattice and generates additional active sites.

At slower scan rates, the electrolyte ions have enough time to penetrate into the active sites of the electrode leading to high current density and capacitance values. With the increase in the scan rate, the redox peaks experience slight shifts owing to the presence of polarization and capacitance values tend to decrease due to reduced ion diffusion ability. Nevertheless, if the CV profiles at various scan rates are almost similar, then it implies good reversibility and rate capabilities.

The specific capacitance from CV can be calculated using the equation:

$$C_s = \frac{\int I(V) dV}{m \nu \Delta V} \quad (2)$$

Where, C_s = specific capacitance (F g^{-1}), $\int I(V) dV$ = integrated area under the CV curve, m = mass of active electrode material (g), ν = scan rate (V s^{-1}), ΔV = potential window (V)

A scan rate of 5 mV s^{-1} produces ultra-capacitance values of 151.9 and 457 F g^{-1} from the calculated areal capacitance (C_{sp}) of the fabricated pure and GO/Cd-doped Nickel-oxides electrodes. In low scan rates, the K^+ ions present in the electrolyte can better intercalate and de-intercalate themselves into the bigger active sites of the electrode [46]. It was found there was a decrease in C_{sp} with an increase in the scan rate. This increased capacity performance is due to the fact that fine particles are homogeneously dispersed within the electrode which ensures better distribution of ions into the structure of the nickel oxides. This has led to increased capacity performance in the GO/Cd-doped NiO ($x = 10\%$) electrode and makes it possible to consider as an ideal material for ultracapacitors application. Fig 12. Shows the ultracapacitance of pure and metal GO/Cd-doped NiO electrodes at 5 mV/s .

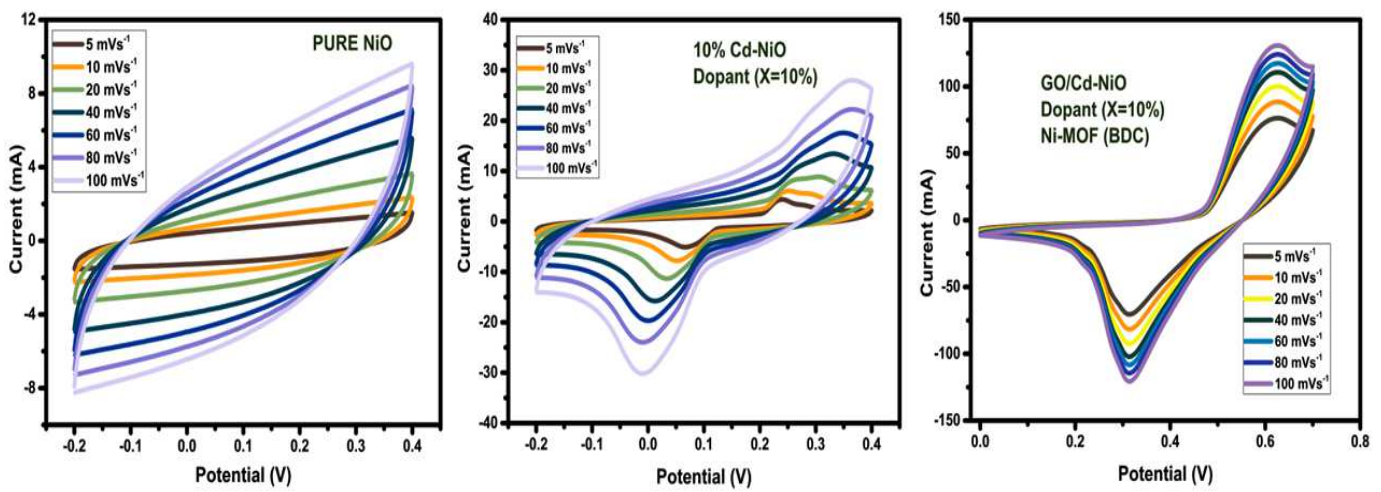


Fig 12. Shows the cyclic voltammetry curves for pure NiO and GO/Cd-NiO nanoparticles.

3.9.2 Galvanometric Charge-Discharge Studies:

The GCD method is widely used in studying such parameters as specific capacitance, reversibility of charge-discharge process, coulombic efficiency, and rate performance of the electrode of supercapacitors. The method consists of charge-discharge of the electrode at a constant current and fixed potential window, when the voltage-time dependence is measured.

As for the GO/Cd-NiO nanocomposite electrode, one should expect asymmetry of the voltage profile with plateaus rather than symmetry, as well as triangle shape curves. This electrode exhibit the behavior of the pseudocapacitor electrode, as the charge will be accumulated by means of redox reactions in the surface and subsurface areas. Longer time for discharge of GO/Cd-NiO as compared to pure NiO electrode shows higher ability of charge storage [47,48]. Enhanced GCD behavior of GO/Cd-NiO

nanocomposite can be explained by the following reasons: (a) Cd doping changes the electronic structure of NiO and increases the number of active electrochemically sites. (b) Graphene oxide is a conductive matrix for Cd-NiO nanoparticles, which facilitates electrons transfer. (c) The nanocomposite structure provides better electrolyte access and reduces the path of ions diffusion. (d) Effective anchoring of Cd-NiO nanoparticles on GO prevents agglomeration of nanoparticles and utilization of the active material.

The specific capacitance from GCD is calculated using:

$$C_s = \frac{I\Delta t}{m\Delta V} \quad (3)$$

Where, C_s = specific capacitance ($F\ g^{-1}$), I = discharge current (A), Δt = discharge time (s), m = mass of active material (g), ΔV = discharge potential window (V)

Charge-discharge galvanostatic studies of the GO/Cd:NiO thin films were performed under varying current densities (1, 1.5, 2.0, 2.5, 3.0, 3.5 and 4.0 $mA\ cm^{-2}$) in 3 M KOH electrolyte at a potential range of -0.2 to 4.0 V, as can be seen in Fig. 13. Charge-discharge profile obtained for 1 $mA\ cm^{-2}$ current density shows an extended charge-discharge time than that of others. The extended charge-discharge time attained for all current densities, reaches up to the entire potential window under constant current mode. It is clear from the non-linearity of the discharge profiles in Fig. 13 that the material follows pseudocapacitive behavior; the discharge time decreases gradually with respect to increasing current density. The ultra-capacitance values for GO/Cd-NiO and pure NiO were determined from the discharge profiles at current densities of (1, 1.5, 2.0, 2.5, 3.0, 3.5 and 4.0 $mA\ cm^{-2}$) are 1162, 1117, 1028, 833, 822, 672, and 472 $F\ g^{-1}$ and 532, 463, 410, 208, 107, 60, 46 Fg^{-1} respectively [48]. The specific capacitance obtained at lower current density of 1 $mA\ cm^{-2}$ shows higher values due to extended discharge time.

Table: 2. Comparison of capacitance values of GO/Cd-NiO composites.

Electrode material	CV capacitance	GCD capacitance	Electrolyte / notes	Reference
GO/Cd-NiO nanocomposite (Present work, theoretical)	457 F g ⁻¹ at 5 mV s ⁻¹	1162 F g ⁻¹ at 1 A g ⁻¹	Expected pseudocapacitive GO/Cd-NiO electrode	Present work
N-doped graphene/NiO nanocomposite	342 F g ⁻¹ at 5 mV s ⁻¹	320 F g ⁻¹ at 1 A g ⁻¹	NiO dispersed on N-doped graphene	[52]
NiO/graphene nanocomposite	Not explicitly reported in snippet	632 F g ⁻¹ at 2 A g ⁻¹	Hydrothermal NiO/graphene pseudo capacitor electrode / ASC device	[53]
NiO/graphene foam asymmetric supercapacitor	Not emphasized in source snippet	Not emphasized in source snippet	At 1.4 V; retained 17 Wh kg ⁻¹ at 42 kW kg ⁻¹	[54]
Layered NiO/rGO composite	Good redox-type CV behaviour reported	High specific capacitance reported for ASC cathode	Layered NiO/rGO cathode for asymmetric supercapacitor	[55]
Ni(OH)₂ nanoplates on graphene	Strong pseudocapacitive CV response	~1335 F g ⁻¹ at 2.8 A g ⁻¹ ; ~953 F g ⁻¹ at 45.7 A g ⁻¹	Direct growth on conductive graphene greatly improved capacitance	[56]
NiO quantum dots/few-layer graphene asymmetric supercapacitor	Pseudocapacitive CV behaviour reported	High-capacity retention and enhanced capacitance reported	NiO quantum dots uniformly dispersed on graphene	[57]
Pure and Al-doped NiO/GO films on grafoil	Good pseudocapacitive CV response reported	Specific capacitance, energy density and power density all reported; pure NiO film reached 181.25 F g ⁻¹	GO-coated NiO / Al-doped NiO films on grafoil sheet	[58]

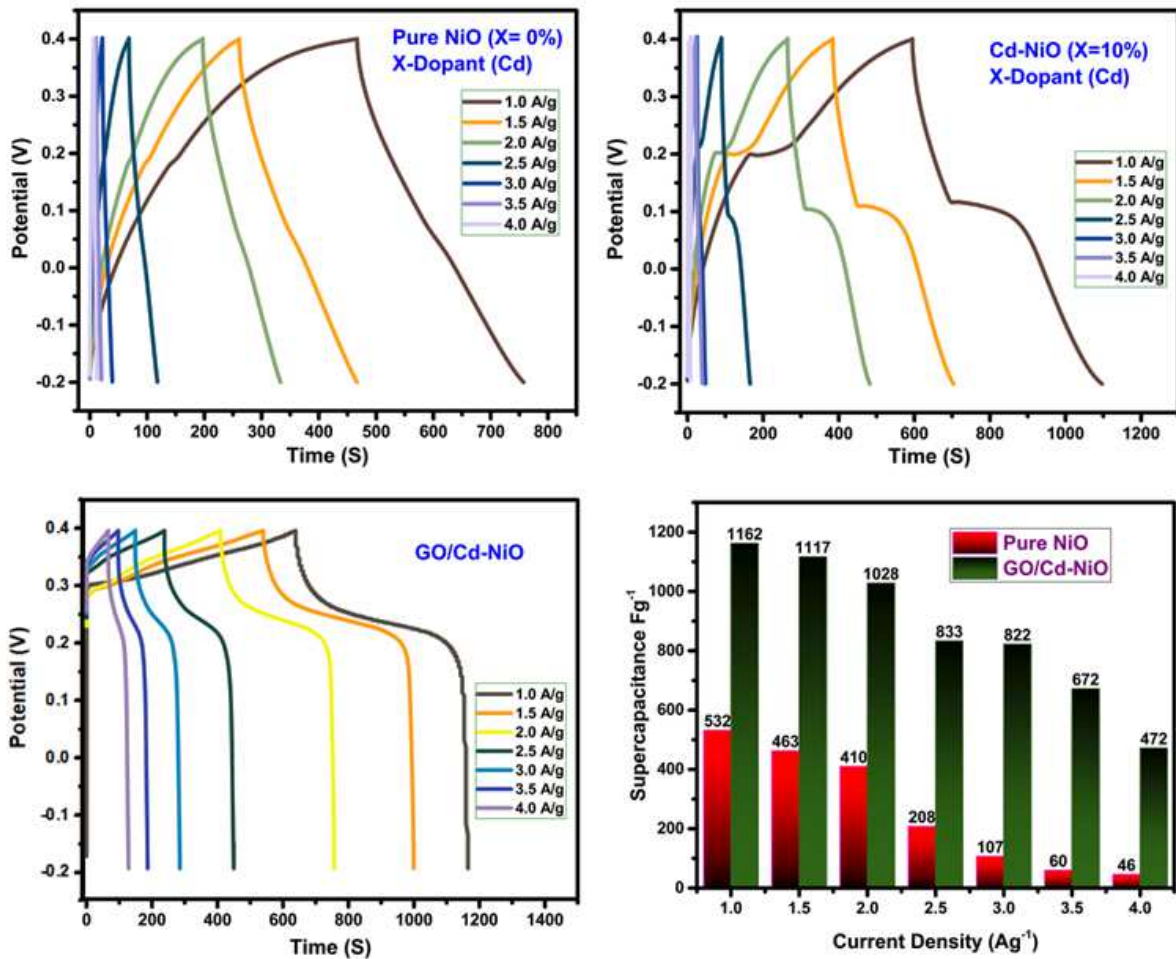


Fig 13. Shows the charge and discharge characteristics for pure NiO and GO/Cd-NiO nanoparticles.

3.9.3 Electrochemical Impedance Spectroscopy:

EIS was done to analyze the characteristics of charge transfer, interfacial resistance, and ion diffusion in the GO/Cd-NiO electrode. A Nyquist plot usually comprises a small semi-circle in the high-frequency regime and an inclined or almost vertical straight line in the low-frequency regime. The intersection of the graph on the real axis in the high-frequency regime denotes the solution resistance (R_s) comprising resistance due to electrolyte, current collector, and electrode itself. The diameter of the semi-circle is the charge transfer resistance (R_{ct}) related to the Faradaic reactions occurring at the electrode/electrolyte interface. In the case of NiO electrodes containing GO, the existence of graphene-derived sheets reduces charge-transfer resistance due to creation of conducting pathways for electrons [48]. Hence, the GO/Cd-NiO electrode would have a smaller diameter of semi-circle compared to the bare NiO electrode. The greater slope of the impedance graph at the low-frequency range implies better capacitive nature and faster diffusion rate of the ions across the porous electrode structure. This decreased impedance value is due to the porous nanocomposite structure, the conductive nature of GO, and the change in electronic configuration of NiO after addition of Cd as shown in the Fig. 14. All

together, the EIS study confirms the better electrochemical property of GO/Cd–NiO, as indicated by the decreased impedance and increased transport properties of the electrode/electrolyte interface [49].

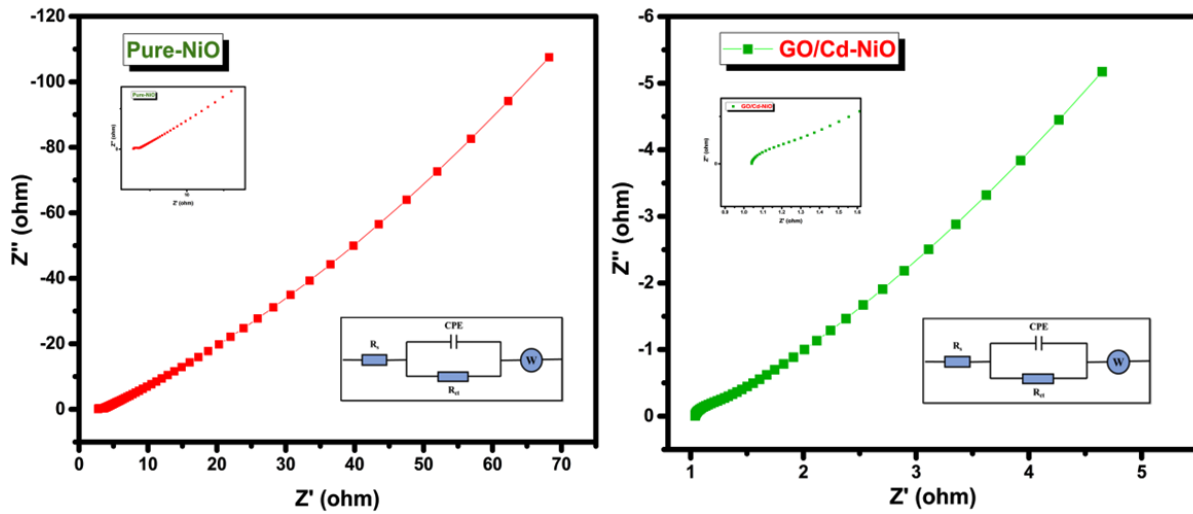


Fig 14. Shows Nyquist plots of (a) pure NiO and (b) GO/Cd–NiO nanocomposite electrodes.

3.9.4 Cyclic stability analysis

Cycling stability is one of the most important characteristics determining the real-world potential of supercapacitor electrodes materials. The cycling stability of the GO/Cd–NiO electrode is usually determined by cyclic galvanostatic charge-discharge tests conducted within thousands of cycles with a constant current density [50].

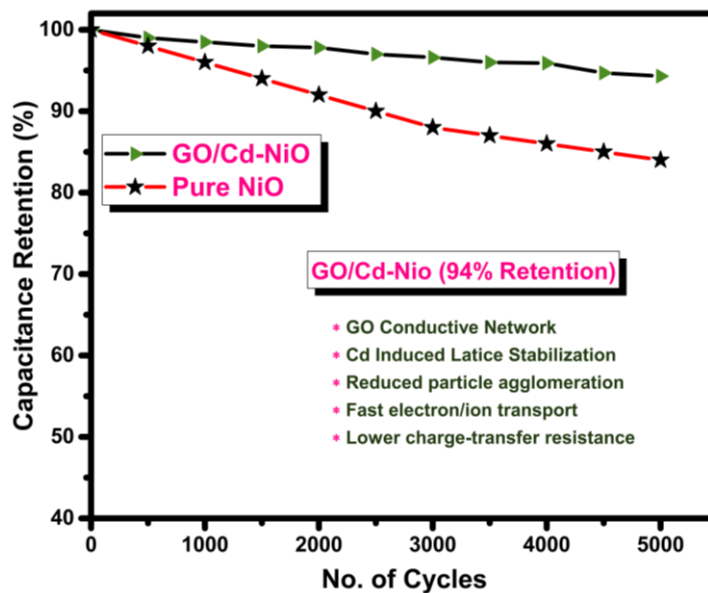


Fig 15. Shows the cyclic stability of pure NiO and (b) GO/Cd–NiO nanocomposite electrodes.

The GO/Cd–NiO nanocomposite possess a high degree of capacitance retention after many cycles, which is an evidence of good reversibility of electrochemical processes and stable structure. The improved properties of cycling can be explained by the combined effect of graphene oxide and Cd-doped NiO. The role of GO layers is the creation of a flexible conductive matrix accommodating the volumetric changes of the oxide during redox processes, thus providing the structural integrity and better adhesion of the material. Simultaneously, the small size of Cd–NiO particles decreases the ion-diffusion limitations and increases the efficiency of charge transfer. Good interactions between the oxide nanoparticles and graphene oxide can prevent their aggregation and detachment from the current collector during numerous cycles and attains the cyclic stability of GO/Cd–NiO is 94% at 5000 Cycles as shown in the Fig 15. Thus, the nanocomposite GO/Cd–NiO nanocomposites preserve a high part of the original capacitance even after long-term cycling [51,61].

Conclusion

To conclude, graphene oxide and cadmium-doped NiO-based composites are promising electrode materials for the fabrication of supercapacitors owing to their redox ability of NiO and high conductivity and large surface area of GO. The results of characterization have demonstrated that the synthesis of Cd-doped NiO combined with GO was successful and the porous microstructure of the hybrid is favorable to providing a large number of electroactive centers and effective paths for ions/electrons transfer. The electrochemical tests reveal that the GO/Cd–NiO nanocomposite is characterized by higher pseudocapacitance 1162 F/g because of reversible Faradaic reactions on Ni-containing materials. The use of GO provides increased electrical conductivity and structural stability, and Cd doping contributes to the tuning of the electron configuration of NiO and active site accessibility. Thus, the nanocomposite under consideration should demonstrate higher specific capacitance, lower charge transfer resistance, enhanced rate capability, and cycling stability 94% of 5000 cycles in comparison with pure NiO. As a result, such nanoparticles exhibit enhanced electrochemical properties and show strong potential as efficient electrode materials in nanoparticle-based energy storage applications.

Author contribution

CK & AP: conceptualization, methodology, review & editing, supervision. GSS & DR: conceptualization, methodology, writing—original draft. PJ: review & editing, visualization, formal analysis.

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Data availability

Data sharing is not applicable to this review article as no datasets were generated or analyzed during the current study.

Declarations

Conflict of interest

The authors declare that they have no known conflicts of interest associated with this publication.

Research involving human and animal participants

The research did not involve any human participants or animals

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