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ORIGINAL ARTICLE

Synergistic effect of nanostructured CdO/Ag_3PO_4 composite for excellent electrochemical and photocatalytic applications

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Abstract In this study, nanostructured cadmium oxide and silver phosphate (CdO/Ag_3PO_4) nanocomposites are synthesized and studied for their electrochemical properties as well as photocatalytic potential of these composites is also investigated. The specific capacitance of pristine CdO is found to be 416.52 F/g and the enhanced capacitance up to 1012.06 F/g is obtained for the composite material with weight ratio of 80/20 for CdO/Ag_3PO_4 nanocomposite. The Galvanic charge–discharge (GCD) study highlights the excellent charging and discharging performance and we conclude that our material is highly stable during the 5000 cycles so could be an appealing candidate for commercial devices. The photocatalysis study is used for the degradation of methyl blue dye. The sample with weight ratio 80/20 has the best photocatalytic activity and degraded the 99.9% of the dye in 120 min. Experimental results suggest that CdO/Ag_3PO_4 has an outstanding potential for use in energy storage devices as an electrode material for supercapacitors and simul-

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taneously could also be a potential candidate for waste water treatment technologies as compared to pristine counterparts.

1. Introduction

The energy storage devices such as batteries, fuel cells, and supercapacitors play a very important role in storing electrical energy for high performance applications in various electrical devices. The electrochemical studies of these devices have introduced different materials with extraordinary performances to the scientific community (Trukhanov et al., 2015, Trukhanov et al., 2018, Trukhanov et al., 2018, Almessiere et al., 2019, Zdorovets et al., 2020, Vinnik et al., 2021, Hassan et al., 2022). In the modern era, supercapacitors have gained attraction to store energy due to their long life cycle, stability, improved charge–discharge ability, high maintenance and energy density as compared to the batteries (Sawangphruk et al., 2013). Due to their long life cycle and high charge–discharge rate, the supercapacitor are more important for energy storage devices in next generation applications (Das and Mitra 2019, Castro-Gutiérrez et al., 2020, Olabi et al., 2022, Yadlapalli et al., 2022). We can store energy in various forms in several devices such as chemical, electrochemical, electromagnetic and thermal etc. using batteries, capacitors, fuel cells, flywheels, compressed air and super magnets equipped in cellular phones and electric vehicles etc. (Khurshid et al., 2018).

Different nanomaterials have been studied due to their attractive energy storage properties and the materials used in supercapacitors are mostly transition metal oxides, conducting polymers and carbon materials depending upon their applications (Chen and Dai 2013, Chen et al., 2015, Zhao et al., 2015, Demming 2016, Lu et al., 2017, Kumar et al., 2018, Chen et al., 2019, Guo et al., 2020, Ming et al., 2020, Guo et al., 2021, Qi et al., 2021, Wang et al., 2021, Li et al., 2022, Qi et al., 2022). The function of a supercapacitor is directly dependent on the electrode material (Chang et al., 2013). The surface area of electrodes controls the energy storage process and charging and discharging process of a capacitor (Ferrari and Robertson 2000). The electrical properties of any material are mainly dependent on the resistance that they offer but the resistance of any material directly depends on the dimensions of the material so by controlling the size of the material we can control the electrical properties of a material (Ueda et al., 1998). Following this constraint, a number of oxide materials such as CuO, NiO, MnO_2, Co_3O_4, etc. (Kebede 2020, Veerakumar et al., 2020, Kandasamy et al., 2021, Rezende et al., 2022) have been used in supercapacitors as electrode material due to their high capacitance and stability. Among the oxide materials, the CdO is also used as an electrode material due to its low cost and low electric resistivity (Feng et al., 2014). Cadmium oxide has unique and attractive properties among all oxides due to possessing electrical, chemical, mechanical and optical properties at the same time. The cadmium oxide has cubic structure with the direct bandgap of 2.5 eV and indirect bandgap of 1.98 eV (Mane and Han 2005).

Instead of single nanoparticle materials, nanocomposites have much better properties as compared to single counterpart for supercapacitor application (Lu et al., 2011, Yu et al., 2019, Ahmad et al., 2020, Cuña et al., 2020, Rahman et al., 2020, Varshney et al., 2020). The CdO has also been studied for supercapacitor applications with some doping agents and nanocomposite materials such as CuZn-CdO, Sr-doped CdO, CdO-$Co(OH)_2$ etc. (Tehare et al., 2017, Abbas et al., 2018, Khairy et al., 2018, Pratheepa and Lawrence 2020, Henríquez et al., 2021). Some of these composites possessed enhanced value of specific capacitance but low stability issues for higher number of cycles and vice versa. Mohamed K. et al prepared CdO/$Cd(OH)_2$ nanocomposite electrode which was highly stable but it achieved a very low specific capacitance of about 145 F/g at a discharge current of 2.0 A/g (Khairy et al., 2018). Specific capacitance value as high as 1119 F/g was obtained for $Co(OH)_2$–CdO composite but just after 1000 cycles, this value retained 54% of its original value (Tehare et al., 2017). A $CdCO_3$/CdO/Co_3O_4 composite has been prepared by hydrothermal-annealing method produced low specific capacitance value of 84 F/g but increased stability and long cycle life (92% after 6000 cycles) (Henríquez et al., 2021). So an attempt has been performed to optimize the electrochemical performance of CdO based materials which can simultaneously enhance all the associated parameters to produce better energy storage devices.

Silver orthophosphate (Ag_3PO_4) has been extensively studied as a visible-light driven photocatalyst. It has a high power of separating of photo-excited electrons and holes also possessed high quantum efficiency up to 90% which makes it an influential photocatalytic agent for the decomposition of organic compounds and O_2 evolution from water splitting under visible light irradiation (Zheng et al., 2017). S. Li et al. showed that tetrapods Ag_3PO_4 synthesized by a hydrothermal method could be employed as a supercapacitor electrode material (Li et al., 2014). However, only double-layer capacitance was observed for Ag_3PO_4 tetrapods in KNO_3 aqueous electrolyte. Following this, C. Zheng had demonstrated that Ag_3PO_4 nanospheres prepared by a sonochemical process exhibited a pseudocapacitive behavior in KOH electrolyte and they obtained specific capacitance of 832 F/g but their Ag_3PO_4 exhibited an inferior charge–discharge cycling stability. The Ag compound is also chosen due to its high electrical conductivity and it is cheaper and non-toxic compared to other metals, such as gold, platinum, and palladium (Omar et al., 2018).

In this article, we explain the synthesis of CdO/Ag_3PO_4 nanomaterial by hydrothermal method and their application as electrode material in supercapacitors and analyse the effect of concentration of different composition for electrochemical analysis. There are different methods (physical and chemical methods) that are being used to synthesized nanoparticles such as hydrothermal method, sol–gel method, vapour deposition method green synthesis, chemical method, electron beam evaporation, vacuum evaporation method and pulsed laser deposition method (Munawar et al., 2021). In all of these methods, there are some disadvantages such as high cost, cost-effective, time-consuming, toxic and not eco-friendly. But the hydrothermal method has been found the best method to synthesize the nanomaterials especially for electrochemical applications. We analyse the composite materials by different characterization techniques and calculate specific capacitance of synthesized material in supercapacitor keeping in view the advantage of silver phosphate's band gap 2.2–2.5 eV which is comparable to the CdO, the CdO/Ag_3PO_4 composite is also studied for the photocatalytic performance for the degradation of Methyl Blue (MB) dye. The as-synthesized nanocomposites possessed higher specific capacitance than the individual counter parts as compared to reported values so far (Kumar et al., 2017, Zheng et al., 2017) and are highly stable, pure and cost-effective and the results have been compared with previous studies and synthesized material is found to be an excellent composite for energy storage devices and simultaneously possessing enhanced photocatalytic activity rather than the CdO nanocomposites reported so far.

2. Experimental work

2.1. Materials

The various precursor materials such as Cadmium Chloride ($CdCl_2$), ethanol (CH_3CH_2OH), Acetone (CH_3COCH_3), PEG, $Na_4P_2O_7$ and $AgNO_3$ were purchased from Sigma Aldrich and were used without further purification.

2.2. Synthesis of cadmium oxide nanoparticles

An aqueous solution of $CdCl_2$ was prepared by mixing 4 mg of $CdCl_2$ in 25 ml distilled water. The Polyethylene glycol (PEG)-1000 was dissolved into 25 ml distilled water and resulting solutions were mixed dropwise followed by 1 hour stirring to make a homogeneous solution. This final solution was put in an autoclave and heated in the oven at 180 °C for 24 h and yellowish precipitate were collected. The precipitate solution was washed and dried at an 80 °C to obtain a powder which was calcined at 500 °C for 4 hours to obtain CdO nanoparticles. After calcination, sample was grinded in a pestle and mortar.

2.3. Synthesis of silver phosphate nanoparticles

To prepare silver phosphate nanoparticles, a 2 g of $AgNO_3$ and 2 g of $Na_4P_2O_7$ were dissolved in 40 ml water separately to get homogenous solutions which were then mixed on a magnetic stirrer. The resulting solution was stirred for 30 min at 40 °C. Then the above solution was put in an autoclave and heated at 120 °C for 15 hours in an oven. Then the obtained sample was washed and dried at an 80 °C to obtain Ag_3PO_4 nanoparticles powder.

2.4. Synthesis of CdO/Ag_3PO_4 nanoparticles composite

The composites of cadmium oxide and silver phosphate nanoparticles were made by varying the concentration of Ag_3PO_4 in cadmium oxide. Three different composite samples were prepared by adding weighted percentage of 10%, 20% and 30% of Ag_3PO_4 in cadmium oxide named as Sample-A, Sample-B and Sample-C respectively.

To prepare these samples the weighted solutions of silver phosphate were added dropwise in cadmium oxide solution and stirred for 15 min. The resulting solutions were transferred into the autoclave and then heated at 150 °C for 15 h. These solutions were then kept at room temperature for 1 hour until the precipitates were formed at the bottom. Nanoparticle of composite materials were then washed with distilled water to remove impurities and the schematic diagram of the procedure is represented in Fig. 1.

2.5. Characterization tools

The size of crystallite of the samples and the structural properties were investigated via xrays diffraction (XRD) technique. The Cu-Kα radiation with a wavelength of 1.5406167 Å was used to probe the samples on a scanning rate of 0.02°/s in the 2θ range from 20 to 70. Scanning electron microscopy (SEM) with model FEI NOV NanoSEM-450 (equipped with Energy-dispersive X-ray spectroscopy (EDX) to evaluate the elemental composition and purity of the samples) and transmission electron microscopy (TEM) with model JEOL JEM-1010 were used for the morphological study of the samples. For, electrochemical analysis, Gamry 5000-E Interface Potentiostat was used for the all Galvanic charge–discharge (GCD), Cyclic voltametry (CV) and Electrochemical impedance spectroscopy (EIS) properties. The photocatalytic properties were investigated by Jasco FP-8200 spectrophotometer.

3. Structural analysis

The XRD pattern of CdO and CdO/Ag_3PO_4 composites are shown in the Fig. 2 (a). The planes observed at the main peaks are (1 1 1), (2 0 0), (2 2 0), (3 1 1) and (2 2 2) correspond to the diffraction angles 33.02, 38.24, 54.9, 65.75 and 69.09°respectively and the cell parameters are matched with the JCPDS Card No: 05–0640 for the cubic phase. The XRD plot is between the intensity and 2θ ranging from 20 to 70°. As we increased the concentration of doped material the peaks are sharper. The sharper peaks show that the structure is highly crystalline Fig. 2. The XRD patterns were analyzed by using Analytical expert high score plus. From the XRD results, we obtained information about the crystal planes, FWHM values and d-spacing for each plane and the detailed analysis is presented in the following section. The pattern obtained was used to calculate the crystallite size by using the Debye-Scherer formula (Quan et al., 2022).

$$D = \frac{k\lambda}{\beta_{hkl} Cos\theta} \tag{1}$$

where $k = 0.94$ is shape factor for cubic crystals, $\lambda = 1.5406$°A (Cu Kα), while $\beta =$ full width at half maximum (FWHM in radians) centered at 2θ of most intense peak (1 1 1). The crystallite size was found to be 19.52 nm for pure CdO whereas for composites it was found in the range of 21.87 nm to 26.30 nm as shown in Table 1. The lattice parameter "a" was computed for each sample by using hkl plane and respective d-spacing values in the following classical formula [25].

$$a = \frac{\lambda}{2Sin\theta}\sqrt{h^2 + k^2 + l^2} \tag{2}$$

The lattice parameter of the pure CdO(4.88 Å) was greater than the CdO/Ag_3PO_4 composites.. Moreover, the unit cell volume of each sample was calculated by using the following formula:

$$V_{cell} = a^3 \tag{3}$$

The Unit cell volume of pure CdO (116.51 ($Å^3$)) is greater than CdO/Ag_3PO_4 composites following the same trend as that of lattice parameters. The strain (ε) was also calculated for each sample by using equation (4) and the strain analysis shows that the addition of Ag_3PO_4 produces a strain in the material. The overall diffraction analysis is presented in Table 1.

$$\varepsilon = \frac{1}{d^2} \tag{4}$$

The elemental composition analysis of the prepared samples was determined through Energy Dispersive X-ray (EDX) spectroscopy. The EDX plots of pure CdO and CdO/Ag_3PO_4 composite (Sample-B) are presented in Fig. 2 (b).

One can observe the peaks related to Cd and O, expressing the elemental purity of pure CdOwhile the peaks related to Ag_3PO_4 composition is highlighted by its elemental peaks of Ag, P and oxygen as shown in the Fig. 2(b). The in-set tables show the weight percentages of Cd, O, Ag and Phosphorus contents in the prepared samples which are approximately in

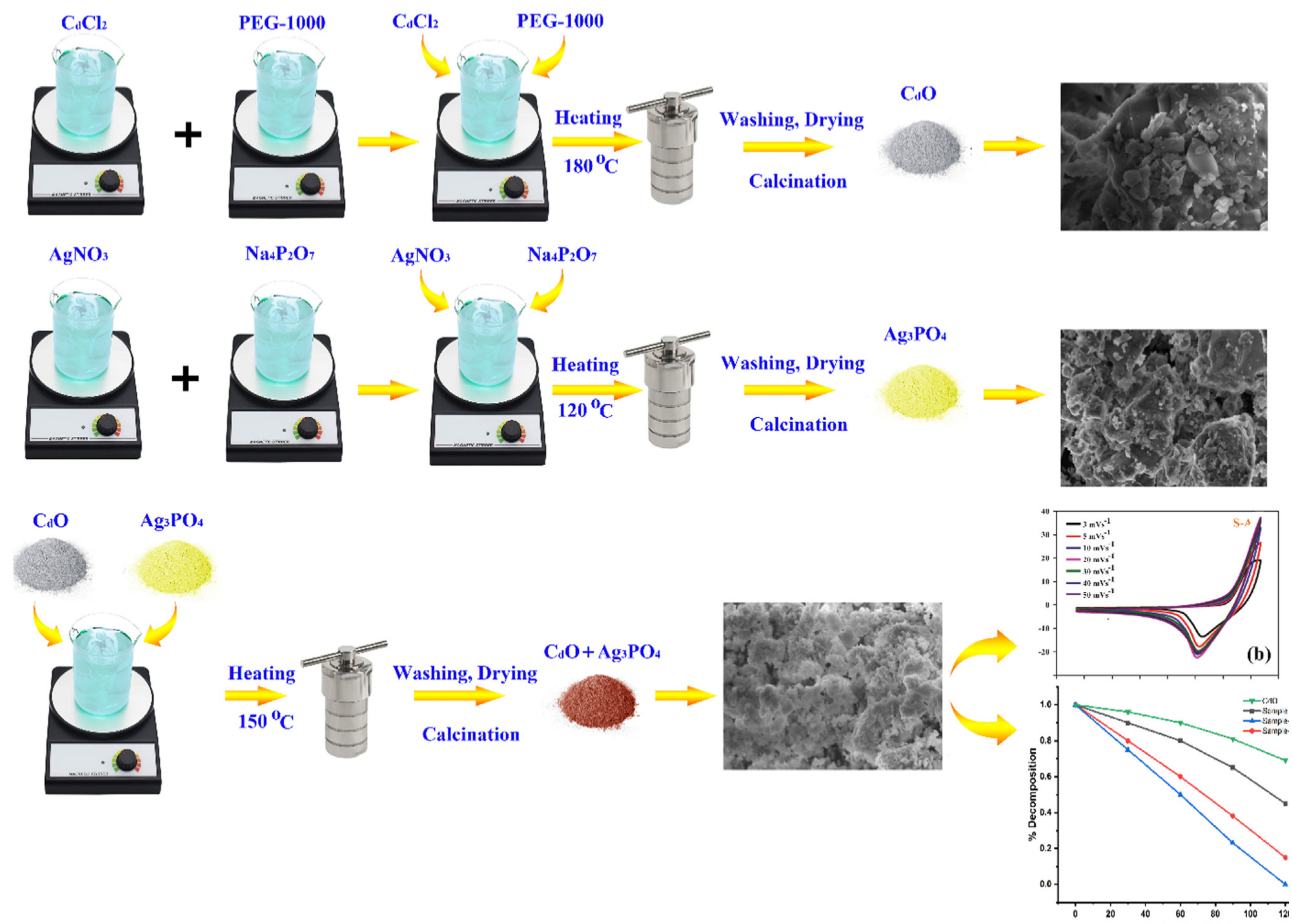

Fig. 1 Schematic diagram for the CdO/Ag_3PO_4 nanocomposite.

agreement with their used weight percentages during the synthesis process.

The surface morphology of the synthesized materials was analyzed by using Scanning Electron Microscopy (SEM) and Transmission Electron Microscope (TEM). Scanning electron microscope images of pure CdO and CdO/Ag_3PO_4 composite (Sample-B) are shown in Fig. 2 (c) & (d) respectively. The morphology of CdO is irregular and porous. The irregular shape is because the grain particles are overlapped to each other therefore the shape is not clear. It is also observed that morphology of CdO/Ag_3PO_4 (Sample-B) is also irregular and more porous than pure CdO. Due to the porous nature of surface morphology, the capacitance of our material is high because ions are easily diffused across electrodes. This higher porosity is also the signature of higher photocatalytic performance of our composite material as explained in the photocatalytic analysis section below.

The TEM was used to investigate the nanostructure morphology, shape, and size of CdO/Ag_3PO_4 composite. The TEM images of CdO and CdO/Ag_3PO_4 composite (Sample-B) are shown in Fig. 2 (e) & (f) respectively. The particles seem to be mixed spherical and irregular in shape with non-uniform distribution. The average particle size of 40 nm to 70 nm is observed for all the pure and composite materials.

4. Electrochemical analysis

The electrodes for electrochemical analysis were fabricated for pristine CdO and it,s composites with Ag_3PO_4 by combining 85% of the synthesized sample materials, 10% activated carbon (weight percentage), and 5% polyvinylidene difluoride which is used as a binder. These mixtures were completely mixed in ethanol to form a slurry which was then applied to a 1 cm^2 nickel foam surface. The resulting samples were dried at 100 °C for 12 h. A three-electrode setup was used for investigation of electrochemical properties. A KOH electrolytic solution with 1 M concentration was used for electrochemical measurements at room temperature. An Ag/AgCl electrode was used as reference electrode, platinum electrode was used as counter electrode and the working electrode was nickel active material pasted on nickel foam.

4.1. Cyclic voltammetry

After the electrode preparation, the synthesized materials (CdO, CdO/Ag_3PO_4 composites) were analyzed for their electrochemical performance to be utilized in supercapacitors. For this purpose, first of all, the cyclic voltammetry (CV) was performed by dipping the prepared electrodes in 1 M KOH electrolyte solution. For CV analysis, different scan rates (3 mV/s

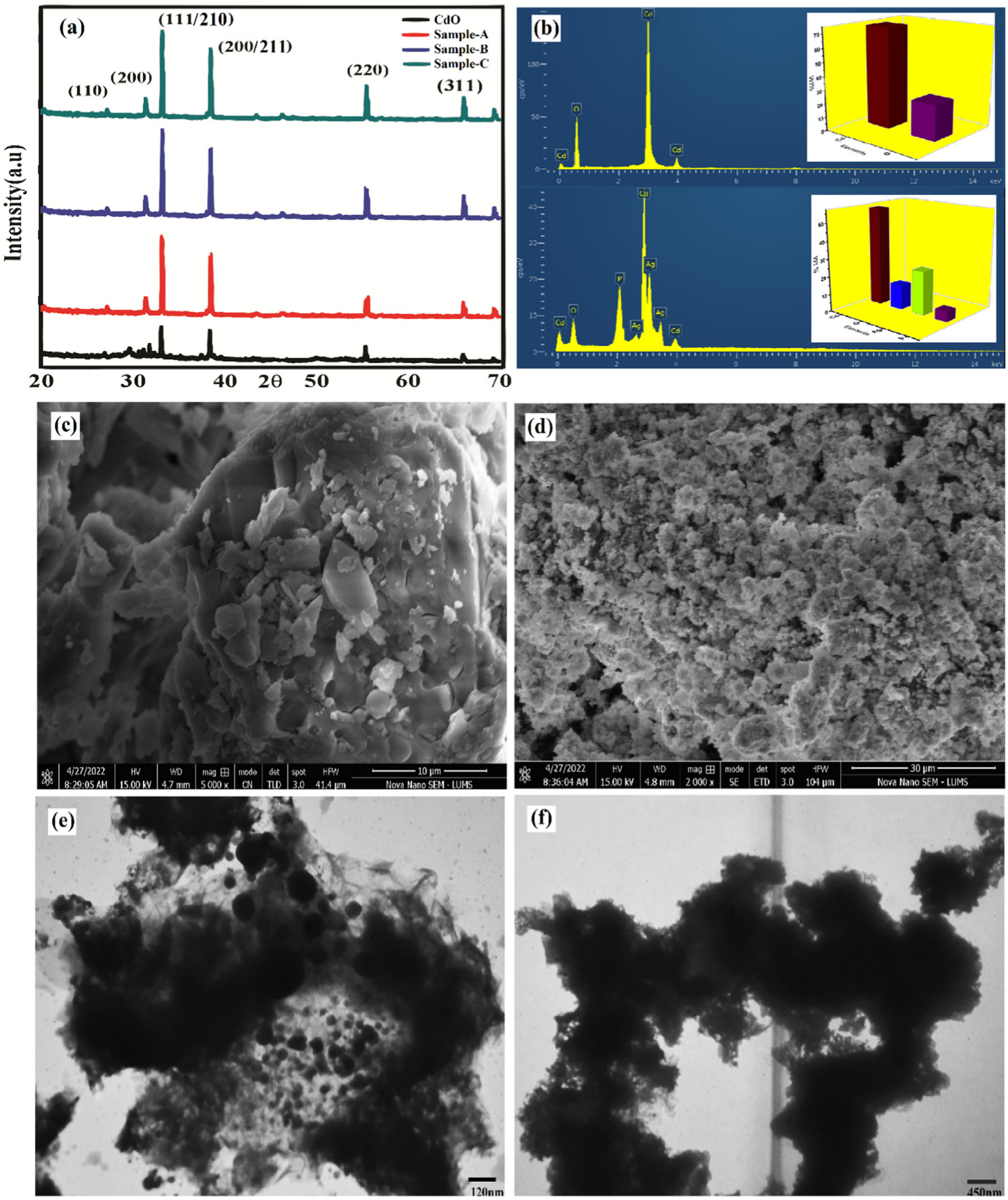

Fig. 2 (a) The XRD Pattern for pure Cadmium Oxide and CdO/Ag_3PO_4 nanocomposites. (b) EDX spectrum of (above) Pure CdO and (below) CdO/Ag_3PO_4 nanocomposite sample-B. The In-sets represent the relative concentrations of the elements. SEM micrographs of (c) pure CdO, (d) CdO/Ag_3PO_4 composite (Sample-B). TEM images of (e) pure CdO, (f) CdO/Ag_3PO_4 composite (Sample-B).

to 50 mV/s) were tested with the potential window ranging from 0.0 V to 0.6 V and the obtained curves have been depicted in Fig. 3.

The CV curves of pristine CdO at various scan rates ranging from 3 mV/s to 50 mV/s are shown in Fig. 3(a). The shape of the CV loop (not rectangular) indicates that the capacitor is a pseudo capacitor. The increase in current density by varying the scan rate shows that the redox reactions are due to interpolation of ions (OH^{-1}, $K^{=1}$) in an electrode as the reversible Faradaic reactions in electrolytes enhances the electrochemical performance (Forouzandeh et al., 2022). Fig. 3 (b)-(d) represent the symmetrical CV curves for the CdO/Ag_3PO_4 nanocomposites, explaining the pseudo capacitor behavior of these composites. The symmetrical shape of CV curves of as-synthesized samples shows that the material of electrodes is better for supercapacitor applications. With the help of CV curves, the

Table 1 XRD parameters Crystallite size, lattice constants (a), volume of unit cell, strain of pure CdO and CdO/Ag_3PO_4 composites.

Sample	D (nm)	a (Å)	V (Å^3)	Strain (ε)
CdO	19.52	4.88	116.51	0.0015
Sample-A	21.87	4.68	102.96	7.23
Sample-B	26.30	4.51	91.73	8.77
Sample-C	26.61	4.59	96.71	8.14

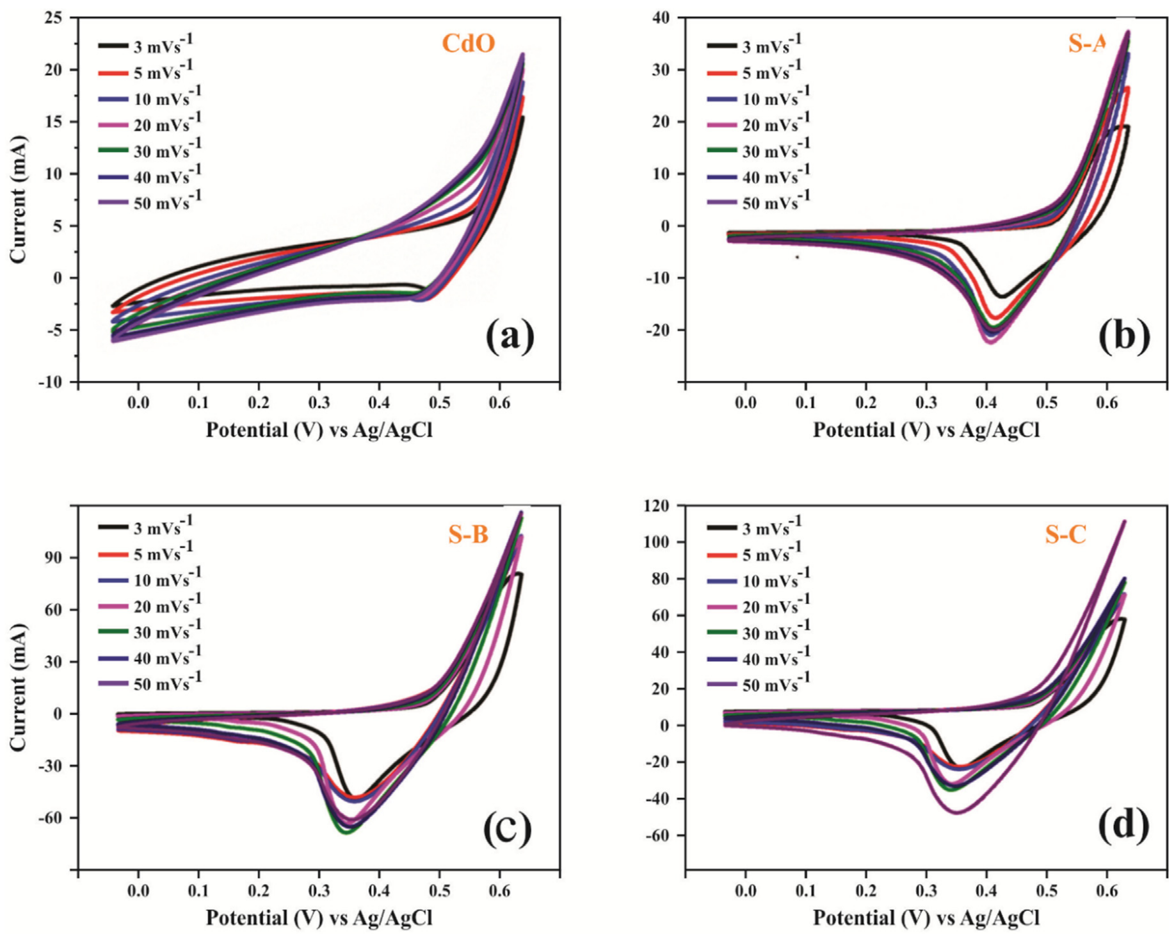

Fig. 3 The Cyclic Voltammetry Curves of CdO (a) Sample-A (b), Sample-B (c) and Sample-C (d) at different Scan rates.

specific capacitance (SC) of synthesized material was calculated by using the following equation (Zhang et al., 2022).

$$C = \int \frac{IdV}{ms\Delta v} \quad (5)$$

where I is the current of an electrode, s is the scan rate, m is the mass of electrode and ΔV is the potential window.

By using the equation (5) maximum specific capacitance values of pristine CdO at scan rate $3 mVs^{-1}$ was 416.52 Fg^{-1} which are far better than reported in the literature as mentioned below in the Table 2. The nanocomposites were also analyzed for the specific capacitance measurements and the SC values of 694.44 Fg^{-1}, 1012.06 Fg^{-1} and 785.18 Fg^{-1} were obtained for Sample-A, Sample-B and Sample-C respectively as shown in Fig. 4(a). So we conclude that the specific capacitance of CdO/Ag_3PO_4 composites is enhanced as compared to pristine CdO. The lesser capacitance of pristine CdO is due to the lower number of active sites. The enhanced electrochemically active surface area and low charge transfer resistance could result in the improved capacitive performance. As compared to the pristine samples, the composite materials have not only increased their surface area but they also increased their active sites owing to increasing the specific capacitance due to the reduction of inactive sites in composite materials. In the reduction of specific capacitance, the ion exchange process plays an important role by changing the scan rate. More ions

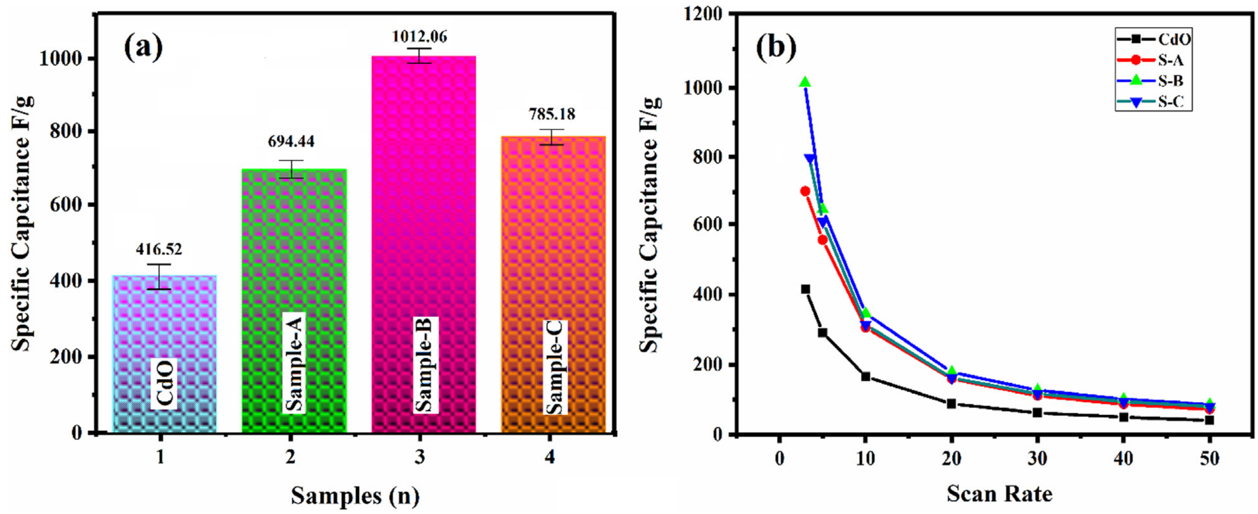

Fig. 4 The Specific capacitance of all the four samples (a) and the dependence of specific capacitance on the scan rate (b).

diffused across electrodes when the scan rate was lower because of more charges transfer by oxidation and reduction reactions. Fig. 4 (b) highlights the effect of scan rate on the SC values and describes the inverse relation of specific capacitance and scan rate. As we increased the molarity of our materials the specific capacitance values also increased. The Fig. 4 also highlights that the rise in the capacitance of the composite material is more significant for the weight ratio of 80/20% (Sample-B). But as we go further to weight ratio of 70/30% (Sample-C) there is a decrease in capacitance due to redox reactions. When the impurities are further increased, the faradaic redox reaction have been effected by the impurity ions (Das and Mitra 2019). Due to which the function (diffusion) of some electrolyte ions have been affected and cannot play a role in redox reaction. So due to lesser ions diffusion and low electron transmission the electrochemical material stores charge only on outer active sites (Pratheepa and Lawrence 2020).So the overall electrochemical performance have been effected in the electrode plates and specific capacitance does not increased further for the sample-C.

4.2. Galvanostatic charge/discharge (GCD)

For practical applications, the role of cyclic stability of supercapacitor is very important (Kumar et al., 2022). The stabilities of CdO nanoparticles and CdO/Ag_3PO_4 composites were analyzed with the help of charge–discharge measurements in the potential range of 0.4 to 0.6 V which shows that the material is highly stable. We obtained Galvanostatic charge–discharge (GCD) curves of CdO and CdO/Ag_3PO_4 nanocomposites at different current densities in a 1 M KOH electrolyte solution as shown in the Fig. 5**.** It is obvious that the specific capacitance of the capacitor increases as current density decreases. We observed that the discharging time of sample B is greater as compared to the other pristine and composite samples.

From the mirror-like potential-time response behavior, these figures highlight that the charging-discharging process of the synthesized electrodes are reversible which is consistent with the CV graphs and the nonlinearity ensures the capacitive nature of the electrodes is due to their pseudo-capacitance. The concentration of alkali ions that are diffusing into and out of the electrode surface helps to control the discharging of the supercapacitor electrodes. The CdO/Ag_3PO_4 nanocomposite electrodes display a longer discharge time as compared to pristine CdO electrode. The uniform structure and higher redox reaction caused by a large number of active sites could be regarded as some main reasons for this longer discharge time of CdO/Ag_3PO_4 nanocomposite electrodes (Bai et al., 2014, Tahir et al., 2014) which is also consistent with structure and morphology of prepared samples as shown in SEM images (Fig. 3). The electrochemical adsorption–desorption phenomenon occurring at the interface of electrolyte and prepared electrode could be responsible for this charging-discharging process (Kumbhar et al., 2012).

The cyclic stabilities of the Sample-B was confirmed by carrying out 5000 CV and GCD cycles as represented in Fig. 6 (a & c). The retentions in the CV and GCD for 1st and 5000th cycles are also shown in Fig. 6(b & d). The inviolate shape of the CV curves even after 5000 cycles specifies that all the samples have an amazing degree of stability. Furthermore, after 5000 CV and GCD cycles, the prepared electrodes retained 90% and 91% of its initial charge, confirming the long-term cycling stability which is higher than that of reported for CdO or Ag_3PO_4 individually in the literature (Nallappan and Gopalan 2018).

According to above electrochemical examination, CdO/Ag_3PO_4 nanocomposite electrodes have presented the excellent cycling performance and capacitive properties even at considerably high charge and discharge currents. The reason behind this impressive performance could be categorized in three different aspects (i) the combination of CdO with Ag_3PO_4 favours the redox reactions causing by the creation of more active sites, (ii) porosity enhancement and homogeneity of composite materials (iii) Ag_3PO_4 provides more channels for electrons and ions transport to enhance the electric conductivity of the CdO/Ag_3PO_4 nanocomposite electrodes. The increased conductivity and outstanding diffusion synergistically increases the specific capacitance and cycling performance of the CdO/Ag_3PO_4 nanocomposite electrodes(Bai et al., 2014).

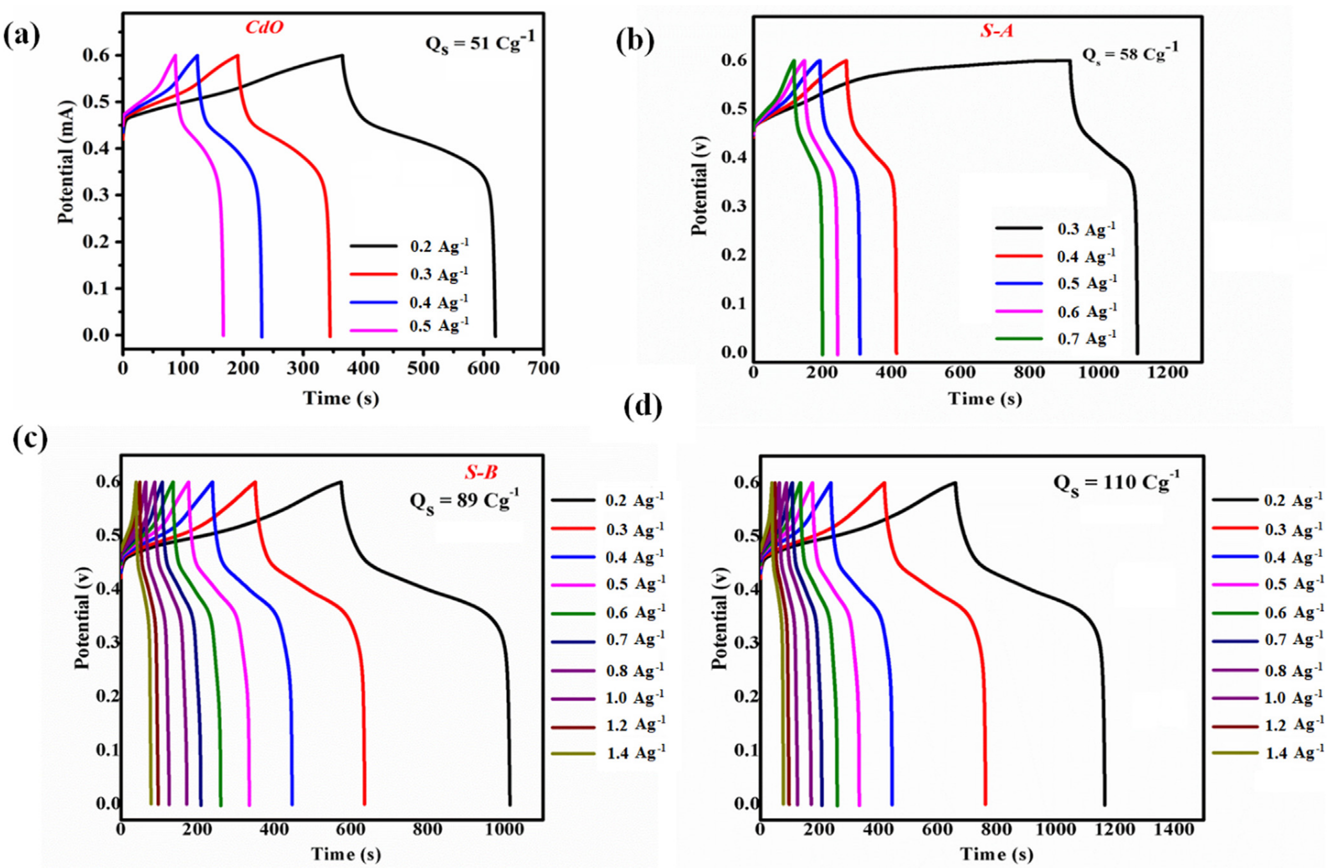

Fig. 5 GCD Curves of CdO and CdO/Ag3PO4 Composites Sample A, B and C at Different Current Densities.

4.3. EIS analysis

To explore the electrochemical kinetics in more detail, the Electrochemical impedance spectroscopy (EIS) is deliberated as one of the authentic and sophisticated techniques. Apart from the stability of charging and discharging process for a large number of cycles, the material should also retain better electronic performance surety. The Fig. 7 shows the EIS graphs of the CdO and CdO/Ag_3PO_4 nanocomposite electrodes in the form of Nyquist graph. As can be seen that, in the high frequency region, the CdO/Ag_3PO_4 displays the depressed and comparatively larger semicircle and in the low frequency region its behavior is described by nearly a straight line illustrating its high charge transfer resistance. From the literature this semicircle is ascribed to the process of charge transfer at electrolyte and electrode interface that is also associated with electrical conductivity and surface area of the prepared electrodes. The charge transfer with in the electrode can be measured directly from the linear slop line also the larger semicircle in Ag_3PO_4 confirms the slow charge transfer process. The CdO/Ag_3PO_4 nanocomposites electrode possess Nyquist semicircle with smaller depressed radius as compared to the pristine CdO which ensures that the composite materials have lesser charge transfer resistance so more suitable for fast charge transfer and provides higher capacitances (Bai et al., 2014, Matheswaran et al., 2018). The more vertical line parts of EIS graphs of CdO/Ag_3PO_4 nanocomposites specifies more rapid ion diffusion of composite materials into the electrolyte as compared to pristine CdO whereas the adsorption at electrode surface is ascribed to the high surface area (Nabi et al., 2020). The superior electrochemical kinetics of the CdO/Ag_3PO_4 nanocomposites as compared to pristine CdO are obvious from these Nyquist plots. So, this synergistically improved performance could be validated to the high effective surface area, enhanced conductivity and low aggregation. The overall electrochemical study (CV, Charge-discharge and EIS) of the prepared samples showed that the CdO/Ag_3PO_4 nanocomposite have exhibited excellent cyclic and capacitive performance which could make it worth considerable candidate for supercapacitor electrodes.

For a deeper analysis, the electrochemical impedance spectroscopy (EIS) was done for the frequency range of 10^{-1} Hz to 10^5 Hz to explore the interfacial behavior and obtain more insight into the capacitive contributions to the electrochemical performance. The in-set of Fig. 7 shows the equivalent circuit diagram for composite sample-B. These findings revealed that Sample-B has the lowest values of Rct and Rs meaning that this an exceptional material with higher conductivity necessary to be used in supercapacitors. Additionally, in the lower frequencies domain the straight line represent the OH– ions diffusion resistant of the electrode and is designated as Impedance of Warburg. The EIS results also depict that the CdO/Ag_3PO_4 composites electrodes have better performance as compared to pristine CdO.

To the best of our knowledge, this interesting pseudo capacitive behaviour is rarely found in Cd-based materials

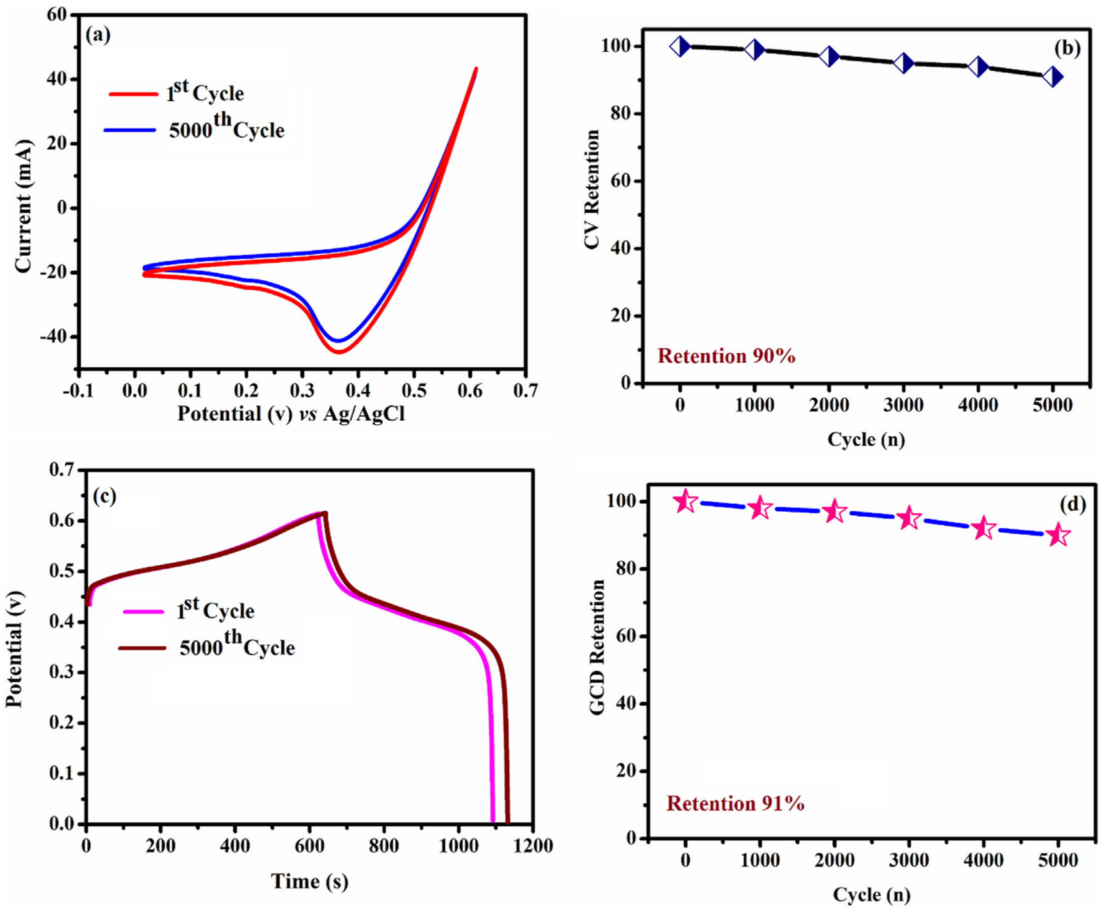

Fig. 6 The CV cyclic stability (a) CV retention (b) GCD cyclic stability (c) and GCD retention.

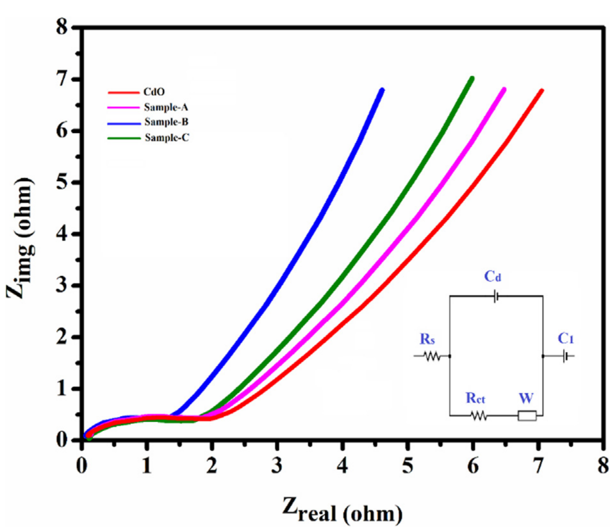

Fig. 7 The EIS graph of CdO and CdO/Ag_3PO_4 Composites samples: inset shows equivalent circuit diagram.

and this could be ascribed to the synthesis of mixed nanocomposites which provide fast electron/ ion transfer, better stress/ strain accommodation and a short diffusion path thereby enhancing the overall electrochemical performance (Khairy et al., 2018). To have a comparison, the results of this study have been related with already reported literature and is given in Table 2. It ca easily be seen that the CdO/Ag_3PO_4 have better performance and could be one of the potential candidates for supercapacitor electrode materials.

5. Photocatalytic activity

The photocatalytic activity of pure CdO and CdO/Ag_3PO_4 composite samples was examined via photo degradation of methylene blue (MB) under visible light. For this purpose a solution was prepared by adding 2 mg of MB in 500 ml of distilled water. Four beakers (A, B, C, D) were taken and 50 ml solution added in each these four beakers from previously prepared solution. Then 15 mg of each CdO, sample-A, sample-B and sample-C were added in A, B, C and D beakers. These solutions were stirred under dark environment for adsorption–desorption equilibrium. Then these solutions were kept in photocatalytic reactor and 2 ml was taken from each solution to check absorption after 0, 30, 60, 90 and 120 min. By utilizing spectrometer, the removal of methyl blue was measured.

Fig. 8(a) shows the photocatalytic performance of pure CdO and it,s composites with Ag_3PO_4 with different concentrations underneath visible light and represents the MB dye degradation rate as a function of irradiation time. From Fig. 8(a) it can be seen that pure CdO showed very low performance under visible light as compare to composite samples.

Table 2 Comparison of specific capacitance values of current work with reported literature.

Material	Synthesis Method	Electrolyte	Scan Rate/Current Density	Cs (F/g)	Ref
Sr doped CdO	Chemical	1MKOH	$1Ag^{-1}$	752F/g	(Xavier et al., 2022)
Zn doped CdO	Co-Precipitation	1 M KOH	$10mVs^{-1}$	388F/g	(Wang et al., 2020)
Ag_3PO_4	Sono Chemical	1 M KOH	$0.5mAcm^{-2}$	832F/g	(Zheng et al., 2017)
$CdO/Cd(OH)_2$	Hydrothermal	1 M NaOH	$25mVs^{-1}$	255F/g	(Khairy et al., 2018)
CdO thin film	Spray Pyrolysis	2 M KOH	$5mVs^{-1}$	344F/g	(More et al., 2019)
Nd^{3+}doped CdO	Hydrothermal	2 m KOH	$1Ag^{-1}$	593F/g	(Dhamodharan et al., 2021)
Mn doped CdO	Co-Precipitation	1 M KOH	$1Ag^{-1}$	351F/g	(Patil et al., 2015)
CdO	Hydrothermal	1 M KOH	$3mVs^{-1}$	416F/g	This Work
CdO/Ag_3PO_4	Hydrothermal	1 M KOH	$3mVs^{-1}$	1012F/g	This Work

The sample S-B showed the highest performance, which exhibited 99.9% reduction of MB after 120 min. After 120 min of irradiation time, MB degradation achieved 30.1%, 55.7%, 99.9% and 85.8% for pristine CdO, Sample-A, Sample B and Sample-C respectively. The Sample-B nanocomposite showed greater degradation rate constant hence better photocatalytic activity. It is clearly indicated that the addition of Ag_3PO_4 nanoparticles can improve the photocatalytic activity of the CdO nanoparticles. In this study, the CdO/Ag_3PO_4 nanocomposites have shown an improved photocatalytic degradation efficiency as compared to the pristine CdO or Ag_3PO_4 individually. When two or more samples with suitable band gap are combined to make composite materials there is an increase in the transfer of electrons and holes when these composites are irradiated by light and this transfer causes an increase in photoreaction. This improved photocatalytic degradation efficiency is attributed to the different factors such as rapid transfer of charge carriers between the CdO and Ag_3PO_4 interface, secondly, the surface area is enhanced and thirdly, the photogenerated electrons and holes are effectively separated which can significantly reduce the electron-hole recombination rate. The SEM study (Fig. 3) highlights that the porosity of the composite material is also enhanced which may also be attributed to positively influence the photocatalytic activity of the samples with appropriate composition. The suitable band gap energies of composite materials, their crystallite size and particle size distribution could also play a role for the improved degradation efficiencies.

Fig. 8(b) shows the emission spectra of pure CdO and the composite samples. The graph was drawn between intensity and wavelength where pure CdO showed highest peak intensity. The composite samples showed lower intensity as compare to pure CdO. While the sample S-B has shown lowest intensity as compare to pure CdO and rest of the composite samples, showing that this sample creates highest separation between electron and hole pair and accessibility of band levels for the electrons movement and it is best for photocatalytic activity of Methyl blue.

Possible mechanism: The Fig. 8(c) highlights the possible mechanism of photocatalytic degradation activity of CdO/Ag_3PO_4 nanocomposite. The performance of photocatalysis activity is usually based on the oxidation potential of photogenerated hole, the efficiency of photo-generated electrons and holes separation and the band gap of photocatalyst nanomaterials as given below (Reddy et al., 2018). As soon as the photocatalyst materials are exposed with light irradiation, electrons are transferred to Ag_3PO_4 and the holes are transferred to CdO. As a result of this transfer, the charge separation phenomenon increases and oxidation site is provided by the CdO nanoparticles. As the CdO has lower valence band edge potential as compared to Ag_3PO_4, near the interface, the electrons of the CdO transfer to the Ag_3PO_4 conduction band and, conversely, the holes from the Ag_3PO_4 could be transferred to the valence band of CdO. Electrons present in the conduction band of Ag_3PO_4 and CdO react with oxygen because the levels of conduction bands of CdO and Ag_3PO_4 are more negative than the reduction potential of oxygen. This reaction creates $O^{\cdot-}$ radicals while hydroxyl radicals $OH^{\bullet-}$ are also formed by the reaction of holes of the valence band with water. These hydroxyl radicals are the main oxidants that degrade the MB dye. The recombination of electron–hole pairs is stopped by the presence of oxygen. In this whole process the CdO act as source of photo-generated electrons and causes a shift of the Fermi level. In the CdO/Ag_3PO_4 composite, the transfers of electrons from CdO to Ag_3PO_4 occurs across interface by the process of charge equilibration and thereby enhancing the photocatalytic activity by lowering the electron–hole pair recombination rate. The MB dye molecules are absorbed on the surface of nanocomposite and then transferred into its excited state (MB*) when irradiated by sunlight. On the next stage, the electrons are transferred from the MB* to the Ag_3PO_4 conduction band causing the electrons to be trapped by the molecular oxygen. Finally, in the dye degradation process, the electrons generated by the light irradiation in the excited dyes and molecular oxygen play a crucial role. A greater specific surface area, enhanced porosity and CdO/ Ag_3PO_4 nanocomposite interface all these factors can decrease the recombination rate of electron-hole pairs which in turns improves the photocatalytic performance in the degradation of methyl blue dye under visible light than the pristine CdO and Ag_3PO_4. The overall chemical reactions that lead to the decomposition of the MB dye are mentioned below in Eqs. (6–14).

$$Ag_3PO_4/CdO + Sunlight \rightarrow e_{CB}(Ag_3PO_4)^{-} + h_{VB}(CdO)^{+} \quad (6)$$

$$e^{-}_{CB} + O_2 \rightarrow O^{\cdot-} \quad (7)$$

$$h^{+}_{VB} + H_2O \rightarrow OH^{-} + H^{+} \quad (8)$$

$$MB + hv \rightarrow MB^{*} \quad (9)$$

$$MB^{*} \rightarrow MB^{\cdot+} + O_2^{\cdot-} \quad (10)$$

$$O_2^{\cdot-} + H^{+} \rightarrow HOO^{\cdot} \quad (11)$$

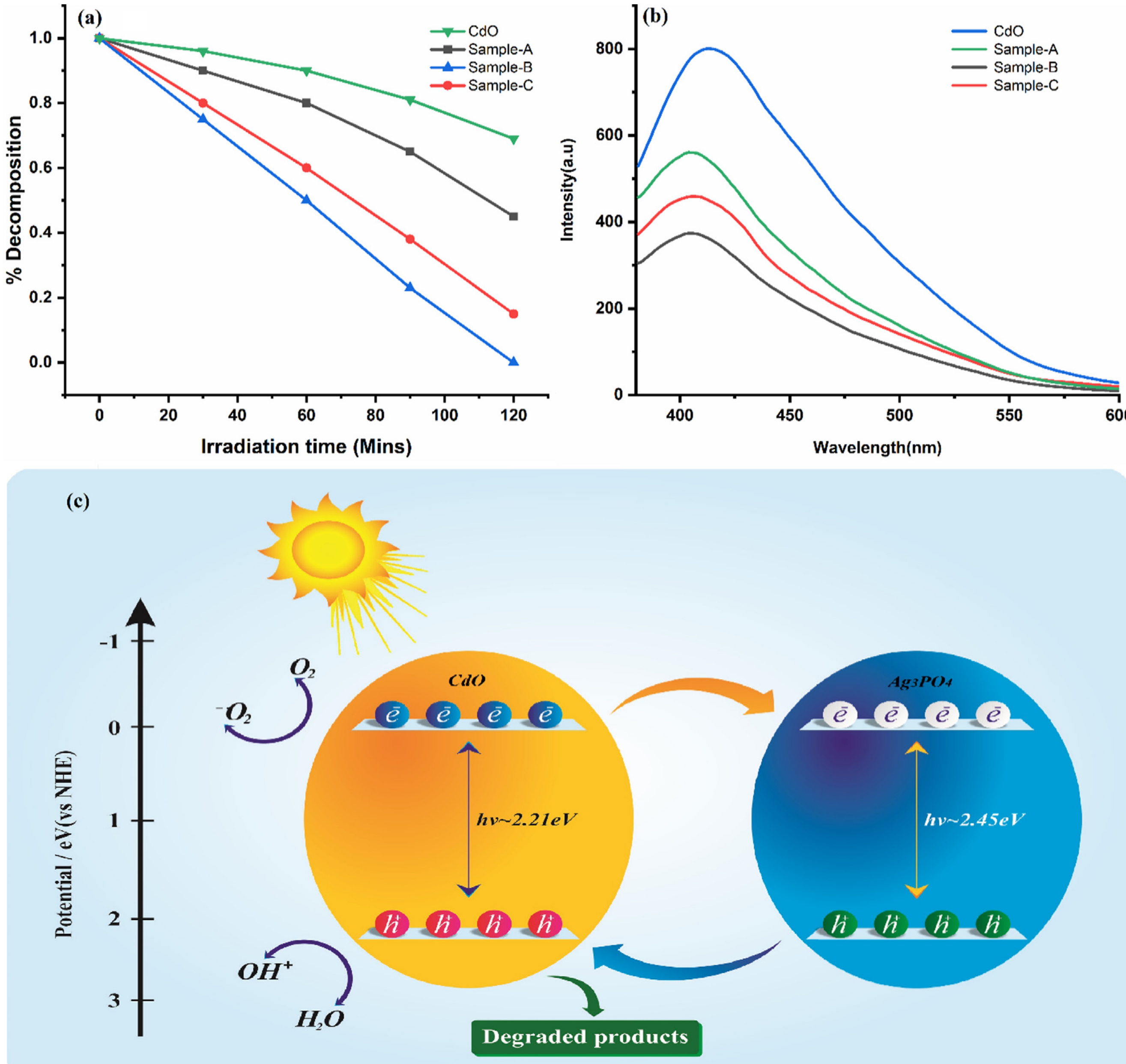

Fig. 8 (a) Photodegrading activity of pure CdO and it's composite with Ag_3PO_4. (b) PL spectra of pure CdO and it's composite with Ag_3PO_4. (c) Proposed photocatalytic mechanism of CdO/Ag_3PO_4 nanocomposite under visible light irradiation.

$$2HOO^{\cdot} \rightarrow H_2O_2 + O_2 \tag{12}$$

$$H_2O_2 \rightarrow 2HO^{\cdot} \tag{13}$$

$$MB + MB^{+} + HO^{\cdot}/O_2^{-} \rightarrow Degraded\,products \tag{14}$$

The efficiency of CdO/Ag_3PO_4 nanocomposites was found to be one of the best efficiencies reported in the literature so far for methyl blue degradation in CdO composite in such a short time. A. Taufik et al. worked out CdO nanoparticles degradation efficiency for methyl blue and they obtained 77% degradation in 120 mins (Taufik et al., 2018) and it's CdO composite with reduced graphene oxide totally degraded the MB dye in 110 mins (Kumar et al., 2016). It's composite with Fe_3O_4 ferrite showed 92.85% efficiency in 150 min (Nallendran et al., 2019). The Ag_3PO_4 on the other hand was also the topic of interest for the researcher for its photocatalytic properties. It's composite with graphene oxide degraded the MO dye with the efficiency of 91% in 120 min (Yan et al., 2017) and it's component with Nb_2O_5 showed 96% efficiency for methyl orange dye (Osman et al., 2021). Besides such efforts some composites of interest related to this study and their corresponding used dyes and efficiencies are listed in the Table 3 for the readers to have also a review on the related work for future concern.

Table 3 Comparison of photocatalytic performance of current work with reported literature.

Material/Composite	Dye used	Time for degradation	Efficiency obtained	Ref
CdO	MB	120 min	77 %	(Taufik et al., 2018)
rGO-CdO	MB	110 min	100%	(Kumar et al., 2016)
CdO-Fe_3O_4	MB	150 min	92.85 %	(Nallendran et al., 2019)
CdO–NiO	MO	180 min	89.44 %	(Nallendran et al., 2018)
CdO-TiO_2	Reactive Green-19	180 min	94.53 %	(Dhanalekshmi et al., 2021)
CdO/ZnO	MB	4.5 h	100%	(Weldegebrieal et al., 2021)
CdO/ZnO	MB	4 h	96.7%	(Yousef et al., 2012)
Graphene-CdO/SnO_2	MG	120 min	94 %	(Sirohi et al., 2019)
	RhB		82 %	
Polyaniline/CdO	MB	4 h	71% (sunlight) 59% (UV light)	(Gülce et al., 2013)
SnS_2-CdO	RhB	210 min	86.11 %	(Srivind et al., 2020)
	CR		92.86 %	
ZnO/Ag/CdO	Textile effluent	210 min	> 90 %	(Saravanan et al., 2015)
ZnO/CdO	MB	6 h	100%	(Saravanan et al., 2011)
CuO/CdO	MB	180 min	98.78%	(Sajid M. Mansoori 2021)
Ag_3PO_4@Ni_3S_2	RhB	40 min	80 %	(Yan et al., 2019)
Ag_3PO_4/Nb_2O_5	MO	60 min	96 %	(Osman et al., 2021)
Ag_3PO_4/Ag	RhB	90 min	Nearly 98 %	(Kai Huang 2015)
	MO		40 %	
	MB		78 %	
GO/Ag_3PO_4	MO	120 min	91 %	(Yan et al., 2017)
Ag@Ag_3PO_4	2-naphthol	3 h	80 %,	(Gong et al., 2012)
	MO	45 min	100%	
CdO/Ag_3PO_4	MB	120 min	99.9%	This work

6. Conclusion

In the current work, the nanoparticles of Cadmium Oxide/Silver Phosphate composites were synthesized by hydrothermal method to investigate the effects of composite material by changing their concentration on crystal structure, morphology, electrochemical and photocatalytic properties. With the help of CV, we calculated the specific capacitance of composite materials and concluded that the capacitance of a capacitor increases by decreasing its scan rate. The capacitance of sample-B (with 80/20% CdO/Ag_3PO_4 composition) is the highest as compared to pure CdO and rest of the composite materials. The GCD confirms the excellent charging and discharging rate and we conclude that our material is highly stable during the 5000 cycles. The synthesized composite electrodes showed 91% capacitive retention after 5000 charge–discharge cycles so could be the best choice for supercapacitor electrodes. Developing low cost nanocomposite electrodes is certainly a research direction that should be extensively investigated in the future and the use of conducting CdO based composite electrode would open a new potential for energy storage device. The photocatalytic studies revealed that the above mentioned sample degraded 99.9% of the Methyl blue dye in 120 min. The comparison of the synthesized composites with reported literature also concluded that the photocatalytic activity of CdO/Ag_3PO_4 is far better for dye degradation. These results confirm the dual behavior of CdO/Ag_3PO_4 for their prospective electrochemical performance and simultaneously excellent application for decontamination of wastewater from toxic organic pollutants.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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