Fabrication of (Al₂O₃/CdO) metal and ceramic matrix composites reinforced CMC via laser ablation and studying their optical, thermal, and electrical properties for optoelectronic applications

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Abstract

Aluminum oxide and cadmium oxide nanoparticles have been reinforced carboxymethyl cellulose (CMC) nanoparticles using a laser ablation process. The effect of change the content of cadmium oxide on the optical, thermal and electrical conductivity properties have been evaluated. The synthesized films have been investigated via various techniques. The strong ion interactions and arrangement of big ion clusters that produced a significant quantity of ion clusters may have caused the $\text{Al}_2\text{O}_3$/CdO peaks to rise again. FTIR spectra of the polymer nanocomposites obtained the interactions between the $\text{Al}_2\text{O}_3$/CdO -NPs and pure CMC. The energy gap values decrease from 5.35 eV to 2.98 eV in direct transition by increasing the content of cadmium oxide nanoparticles. The values of TGA indicate that the thermal stability of the produced materials has been increased by the addition of $\text{Al}_2\text{O}_3$/CdO nanoparticles. Dielectric results found that as $\text{Al}_2\text{O}_3$/CdO NP concentrations increase, the dielectric constant and dielectric loss increase as well. Arc length in CMC/$\text{Al}_2\text{O}_3$ reduces as cadmium oxide content rises, indicating enhanced conductivity. The synthesized nanocomposite film could be suggested for optoelectronic applications.

1. Introduction

Polymer nanocomposites (PNCs) have attracted many researchers owing to their useful characteristics and high-performance by the presence of tiny quantities of metals oxides inside the polymer blend [1]. In comparison to polymer composites, PNCs with typical nanoscale nanofillers exhibit stronger property development at significantly lower loads [2]. The precise choice of the host polymer and the nanofillers is crucial for the production of PNCs since it has a significant effect on both their usage as well as the efficiency of the device [3, 4]. It was found that incorporating metal oxides to the polymeric matrix significantly improved the characteristics of the polymer [5]. The favorable electrical traits, biodegradability, and cost-effectiveness of natural polymers like carboxymethyl cellulose (CMC), starch, and chitosan make them a popular choice for various applications [6]. CMC is a basic derivative of cellulose since it has a hydrophilic carboxyl group with a hydrophobic polysaccharide backbone and a known water-soluble property [7]. Moreover, CMC is defined by a variety of properties, such as being inexpensive, environmentally friendly, semi-crystalline, and having great film-forming capabilities [8]. Because CMC can form a thin film and has potential conduction qualities, there have been numerous efforts to produce it for use in the electrolytes system [9, 10]. Nonetheless, it is important to note that CMC as a single polymer exhibits only moderate stability and miscibility in polymer electrolytes. This is shown in their efficiency when used in battery applications. Because of their strong interactions, nanoparticles (NPs) are able to aggregate effectively. Yet, it is difficult to comprehend the distribution of NPs in nanostructured materials. Modification of the nanoparticles' surfaces and ultrasonic processing using functional coupling agent groups are effective and well-known ways to increase the compatibility of nanoparticles and polymers [11].

Aluminum oxide ($\text{Al}_2\text{O}_3$) is a wide-bandgap oxide insulator with an estimated energy range of between 3.4 and 9 eV [12]. Its photocatalytic ability is restricted to the UV-light range by a wide bandgap [13]. $\text{Al}_2\text{O}_3$
has garnered significant attention due to its expansive surface area, well-organized nanopore structure, and its status as a secure and cost-efficient material with remarkable thermal stability [14]. Its remarkable photocatalyst properties and widespread use in electronic devices, biosensors, and drug delivery are a result of the nanopores' systematic arrangement [15]. According to researchers, nanostructures have noticeable optical properties and are used in optoelectronic systems [16]. Cadmium oxide (CdO) functions as an n-type semiconductor, featuring a bandgap ranging from 2.2 to 2.9 eV [17]. As a result of its shallow donors, it is a cost-effective semiconductor with good electrical conductivity [18]. The oxygen vacancies provide these shallow donors. The ionic conductivity of polymer electrolytes and the amorphous phase of materials are in harmony. To increase the ionic conductivity of solid polymer electrolyte systems, a variety of techniques have been used, including copolymerization, polymer mixing, plasticization, and the incorporation of nanofiller into polymeric composites. The objective of this research is to examine how loading Al₂O₃/CdO affects the structural, optical, electrical and dielectric characteristics of the polymer CMC for potential use in solid-state batteries.

2. Experimental method

2.1. Chemicals

The CMC provided by Sigma Aldrich possesses a molecular weight measuring 172,000 g.mol⁻¹. Additionally, both a high-purity aluminum and a cadmium plate were procured from Sigma Aldrich, with a purity of 99.999%.

2.2 Preparation of CMC - Al₂O₃/CdO nanoparticles

The required quantity of pure CMC was dissolved in 150 ml double-distilled water. The solution is clear and transparent after 2 hours. Firstly, the synthesized Al₂O₃ was dispersed in CMC solution by plus laser ablation in order to make the polymer nanocomposite. Secondary, the CdO was incorporated in CMC/Al₂O₃ at different concentrations (2, 4, 6 wt. %). Finally, a uniformly viscous liquid was created by thoroughly blending and agitating all the solutions together. A petri plate was then filled with the resulting solution. The nanocomposite thin films were generated through a gradual solvent evaporation process over a span of 3 days at a temperature of 50°C. The CMC- Al₂O₃/CdO nanocomposite films were removed from petri-dish and used for further investigation.

2.3. Characterizations technique

The structural characteristics of pure CMC and CMC- Al₂O₃/CdO nanocomposites films are investigated using XRD (Shimadzu XRD− 6000) with Cu Kα radiation (λ = 1.5406 nm). The chemical structures of CMC-Al₂O₃/CdO nanocomposites films were examined using Fourier transform infrared (FT−IR) in the 400−4000 cm⁻¹ range. Optical characterization was achieved by the Shimadzu UV−3600 UV−VIS−NIR spectrophotometers at room temperature (RT) in the 190−1100 nm wavelength range with an accuracy of 0.2 nm. Conducting measurements with a Novocontrol turnkey 40 System, we performed high-
resolution, broad-band dielectric spectroscopy assessments across a frequency spectrum of 0.1 Hz to 7 MHz.

3. Results and Discussion

3.1. XRD analysis

X-ray diffraction was employed to analyze how the presence of Al2O3/CdO nanoparticle nanohybrids influenced the structural characteristics of CMC. Figure 1 shows X-ray diffraction of pristine CMC, CMC/Al2O3 and CMC/Al2O3 filled with different quantities of CdO nanoparticles. The X-ray diffraction of the CMC sample displays a broad-peak appears about $\theta = 23.23^\circ$. This explains the semi-crystalline nature of pure polymer (CMC). The CMC samples were studied, and equivalent results were observed [19]. They showed that the addition of metals nanoparticles reduced the intensity of the peak at $\theta = 23.23^\circ$ for virgin CMC. Such modifications may be caused by the interactions between the pure polymer and metals oxides, which enhanced the amorphous phase of the prepared samples [20]. As the virgin CMC incorporated a sufficient quantity of dopant, free volume space and the potential for ion migration were formed. In this work, the incorporation of Al2O3/CdO was important in the increase in Al ion and Cd ion protonation, which causes the modifications of the amorphous phase. More nanofiller material was added, and this resulted in the hump's width expanding. This observation may be used to suggest the easy interaction between pure polymer CMC and Al2O3/CdO nanoparticles [21]. In Fig. 1, it is evident that the peaks attributed to Al2O3/CdO start to emerge at specific angles: $\theta = 30.87^\circ, 36.85^\circ, 44.38^\circ, 58.56^\circ,$ and $67.74^\circ$. These peaks are attributed to the Miller–Bravais indices of (220), (311), (400), (422), and (440), respectively. The detected $\theta$ values agreed with the value established by the Joint Committee on Powder Diffraction Standards (JCPDS) (file no 79-1558) [22]. The strong ion interactions and arrangement of big ion clusters that produced a significant quantity of ion clusters may have caused the Al2O3/CdO peaks to rise again. This might mean that the Al2O3/CdO concentration accelerates the routes of ion percolation.

In addition, as we will notice in the FT-IR spectra, the cofactor is observed to interact primarily with the polymeric matrix through a carbonyl group (C = O) and a carboxylate anion (COO) group. The enhancement in the crystalline phase of pristine CMC, following the introduction of Al2O3/CdO nanoparticles, is attributed to the local structural arrangement induced by complex formation within the polymeric structure, especially at the final concentration [23].

3.2. FTIR spectra

Figure 2 displays the FT-IR spectrum of pristine CMC, CMC/Al2O3 and CMC/Al2O3 NPs doped with different quantities of CdO nanoparticles. The main vibrational frequencies of CMC were displayed in the FTIR spectrum of the virgin CMC. The spectrum included large peaks at 3303 cm$^{-1}$, which are caused by O-H stretching vibrations in CMC, and minor peaks at 2919 cm$^{-1}$, which are related to aliphatic C-H
vibration modes [24]. The characteristic CMC transmittance peaks were located at 1586, 1417, 1313, and 1027 cm\(^{-1}\) which related to asymmetric -COO- vibrations, CH2 scissoring, OH bending, and C-O-C bending, respectively. The CMC-Al\(_2\)O\(_3\)/CdO nanocomposite films showed stronger FTIR peak intensities than the virgin CMC polymer. This behavior indicated that Al\(_2\)O\(_3\)/CdO and the pure CMC exhibited some substantial intermolecular interactions. Moreover, the Al2O3/CdO nanoparticles was well dispersed in the CMC, which changed the order of the polymeric matrix and ultimately increased peak intensity. It is evident that the intensity of the –OH peak at 3303 cm\(^{-1}\) for high concentrations of cadmium oxide nanoparticles decreased after the addition of cadmium oxide in various concentrations, as well as that of the C–O and COO transmittance peaks, which expanded and decreased. This was due to the strong interactions between pure CMC, Al\(_2\)O\(_3\) NPs, and CdO NPs’ functional groups [25]. With rising CdO -NPs concentrations, the characteristics of the major functional groups, such as the (C–O–C,-OH, and C–O) groups, progressively altered and intensity decreased. As no new peaks were seen in the FT-IR spectra of the prepared samples, it was assumed that the interaction between the Al\(_2\)O\(_3\)/CdO NPs and pure CMC were physical (e.g., van der Waals forces and hydrogen bonds). Furthermore, as shown by the XRD patterns, the larger amorphous region within the doped samples is the consequence of an incorporated method that produces chain cross-linking–scission between CMC- Al2O3/CdO NPs composite.

### 3.3. Optical analysis

UV/Vis analysis is employed to measure the decrease in intensity of a light beam after it traverses through a thin film or reflects off its surface. Figure 3 displays the UV/Vis spectra for pure CMC, CMC/Al\(_2\)O\(_3\) and CMC/Al\(_2\)O\(_3\) NPs filled with various quantities of CdO nanoparticles. The appearance of a shoulder peak at 197 nm in the CMC curve can be attributed to the n → π* transition. Around 238 nm, there's another peak that belongs to π → π* [26]. The intensity of the 238 nm peak increases as cadmium oxide nanoparticle concentrations increase. The peaks' irregular changes in intensity, as shown by XRD analysis, may be caused by variations in crystallinity, which indicates complexation or homogeneity, and by variations in the optical bend gap between the CMC chain and the Al\(_2\)O\(_3\)/CdO NPs [27–30]. A new peak at 417 nm can be seen in the filled samples and may be caused by the surface plasmon resonance of Al\(_2\)O\(_3\)/CdO NPs (SPR). The intensity of surface plasmon resonance peak increases, and there is a redshift towards higher wavelengths (from 417nm to 433nm). The following Equation (Eq. 1) was used to compute the energy gap (E\(_g\)) for all obtained samples [31, 32]:

\[
(\alpha h\nu) = C(h\nu - E_g)^r
\]

where h\(\nu\) is the energy of the incident photons and C is fixed value. The values of r differs depending on whether the transitions are direct or indirect. In the k space, it has values of 2 for a direct transition and 1/2 for an indirect transition. The absorption coefficient (\(\alpha\)) can be calculated using the Beer- Lambert's formula [26].
\[ \alpha (v) = 2.303 \left( \frac{\text{absorbance}}{\text{thickness}} \right) \]

2

Figure 4 displays the dependence of \( \alpha (v) \) on the photon energy \((h \nu)\) for pure CMC, CMC/Al\(_2\)O\(_3\) and CMC/Al\(_2\)O\(_3\) NPs filled with different concentrations of CdO nanoparticles. Table 1 displays the modified absorption edge values, which have been reduced from 4.67 eV to 2.42 eV. Since the conduction and valence bands of the electron hole changes as filler concentrations increase, the absorption edge values decrease. The plots of \((\alpha h \nu)^2\) and \((\alpha h \nu)^{1/2}\) vs \(v\) for all prepared samples are shown in Figs. 5 and 6, and Table 2 gives a summary of the \(E_g\) values that were calculated. Table 1 demonstrates that when filler concentrations increase, the energy gap values \(E_g\) decrease. For the direct transition, the energy gap values decrease from 5.35 eV to 2.98 eV, and for the indirect transition, they decrease from 4.82 eV to 1.04 eV. According to the FTIR analysis, this reduction could be brought on by coordination or interactions between pure CMC chains and Al\(_2\)O\(_3\)/CdO nanoparticles, which might result in localized states inside the band gap.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Concentration</th>
<th>Abs. edge. (eV)</th>
<th>(E_g) (eV)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Direct</td>
<td>Indirect</td>
</tr>
<tr>
<td>Pure CMC</td>
<td>0.0</td>
<td>4.67</td>
<td>5.35</td>
</tr>
<tr>
<td>CAI</td>
<td>CMC/Al(_2)O(_3)</td>
<td>2.55</td>
<td>3.51</td>
</tr>
<tr>
<td>CAICd1</td>
<td>CMC/Al(_2)O(_3)/2%CdO</td>
<td>2.47</td>
<td>3.14</td>
</tr>
<tr>
<td>CAICd2</td>
<td>CMC/Al(_2)O(_3)/4%CdO</td>
<td>2.45</td>
<td>3.06</td>
</tr>
<tr>
<td>CAICd3</td>
<td>CMC/Al(_2)O(_3)/6%CdO</td>
<td>2.42</td>
<td>2.98</td>
</tr>
</tbody>
</table>

3.4. Thermogravimetric Analysis (TGA)

Thermogravimetric analysis (TGA) was utilized to explore the thermal degradation of polymers. This technique is often applied to assess essential kinetic parameters such as degradation temperature, activation energy, and decomposition points. These parameters can clarify the thermal stability of pure CMC following the incorporation of metal nanoparticles. Figure 7 shows TGA behavior depicting weight loss as a function of temperature for CMC, CMC/Al\(_2\)O\(_3\), and CMC/Al\(_2\)O\(_3\) NPs loaded with various concentrations of CdO nanoparticles at a heating rate of 5 °C/min over the temperature range of 30 °C to 800 °C. In the first temperature range (28 °C to 247 °C), the produced films are relatively thermally stable.
In the second zone, between 247 and 479 °C, all films had a fast weight loss attributed to the chemical interaction between aluminum/cadmium and pure matrix CMC. By adding nanoparticles of metals like aluminum and cadmium, the remaining/residual weight increases significantly. This suggests that adding nanometals to CMC boosts the thermal stability of the produced samples. Table 2 lists the weight loss of films at various temperatures as determined by TGA thermograms. These values indicate that the thermal stability of the produced pure polymer has been increased by the addition of Al$_2$O$_3$/CdO nanoparticles.

### Table 2

<table>
<thead>
<tr>
<th>materials</th>
<th>T (°C)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$D_{50}$</td>
</tr>
<tr>
<td>Pure CMC</td>
<td>307</td>
</tr>
<tr>
<td>CAI</td>
<td>376</td>
</tr>
<tr>
<td>CAI CdO1 NPs</td>
<td>415</td>
</tr>
<tr>
<td>CAI CdO2 NPs</td>
<td>419</td>
</tr>
<tr>
<td>CAI CdO3 NPs</td>
<td>446</td>
</tr>
</tbody>
</table>

### 3.5. Electrical behavior

Figure 8 depicts the pristine CMC incorporated with Al203 NPs and various concentrations of CdO nanoparticles, as well as the relationship between log (f) and log ($\sigma$). The conductivity of the prepared samples improved as the nanoparticles of metal oxide increased. Low frequency dispersion was induced by interiorizations or spatial charge [33]. The electrical conductivity was improved in nanocomposites with more dopants because the molecules began to bridge the gap between the two localized states and simplify charge carrier mobility [34]. The increase of amorphous regions inside the CMC-doped polymeric material, as indicated by the XRD results, improved the electrical conductivity of the prepared samples. The Jonscher equation was used to calculate conductivity values.

$$\sigma(\omega) = \sigma_{dc} + A\omega^s$$

Where $s$ is the exponent factor, $\sigma$ is electrical conductivity, and $\omega$ is the angular frequency equal to $2\pi f$. Table 3 presents the computed values of $\sigma_{dc}$ and $s$ for all nanocomposites sample. The values of $s$, which were notably less than 1, demonstrated that these composites utilized a hopping mechanism for
charge conduction [35]. The final samples (CAICdO3 NPs) had $\sigma_{dc}$ of $2.08 \times 10^{-7}$ Scm$^{-1}$ as shown in Table 3.

<table>
<thead>
<tr>
<th>Samples</th>
<th>$\sigma_{dc}$ (Scm$^{-1}$)</th>
<th>$S$</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure CMC</td>
<td>$3.23 \times 10^{-9}$</td>
<td>0.82</td>
</tr>
<tr>
<td>CAI</td>
<td>$1.62 \times 10^{-8}$</td>
<td>0.76</td>
</tr>
<tr>
<td>CAICdO1 NPs</td>
<td>$7.37 \times 10^{-7}$</td>
<td>0.65</td>
</tr>
<tr>
<td>CAICdO2 NPs</td>
<td>$4.11 \times 10^{-7}$</td>
<td>0.51</td>
</tr>
<tr>
<td>CAICdO3 NPs</td>
<td>$2.08 \times 10^{-7}$</td>
<td>0.48</td>
</tr>
</tbody>
</table>

### 3.6. Dielectric analysis

Through a dielectric investigation, the relationship between improved charge mobility and heightened ionic conductivity is established. Evaluating the charge storage capacity of polymer composites is achieved through analysis of dielectric permittivity. The dielectric permittivity of a polymer electrolyte is determined using the following equation:

$$\varepsilon^* = \varepsilon' - j\varepsilon''$$

Here, $\varepsilon'$ represents the dielectric constant and $\varepsilon''$ represents the dielectric loss. Figrs 9 and 10 show the dependence of $\varepsilon'$ and $\varepsilon''$ on log(f) for CMC, CMC/Al$_2$O$_3$ and CMC/Al$_2$O$_3$ NPs filled with different concentrations of CdO nanoparticles. These graphs demonstrate that the non–Debye kind of behavior, also known as the refining density of the charge carriers in the region of space charge accumulation, is what leads the value of $\varepsilon'$ to increment at lower frequencies [4]. The decrease in $\varepsilon'$ and $\varepsilon''$ values in higher frequency regions is attributed to the pronounced periodic reversal of the field at the interface and the diminishing contribution of charge carriers to $\varepsilon'$ with increasing frequency. Additionally, it’s observed that elevating the concentrations of Al2O3/CdO nanoparticles results in a concurrent rise in both dielectric constant and dielectric loss.

### 3.7. Argand plot
The analysis of the Argand plot reveals that the primary factor behind the relaxation process of the polymer matrix is the ion conductivity. Figure 11 displays the Argand plot, which consists of the actual $M'$ and the fictitious $M''$, for CMC, CMC/Al2O3 and CMC/Al2O3 NPs filled with different concentrations of CdO nanoparticles. Polymeric matrix relaxation time distribution frequently deviates from a semicircular shape. It is obvious that every polymer chain has a depressed semi-circular arc. The center of this arc, which is situated along the $M'$ axis, shows the electric relaxation of nanocomposite samples. In this work, the length of the depressed semicircular arc was used to evaluate the conductivity of the prepared films [36]. Arc length in CMC/Al$_2$O$_3$ reduces as cadmium oxide content rises, indicating enhanced conductivity [37]. The Argand curves shift to the origin when cadmium oxide content increases in nanocomposite samples. The presence of diverse polarization forms, relaxation mechanisms, and intricate ion-dipole interaction collectively give rise to the non-Debye behavior of ions.

4. Conclusion

Nanocomposite films based Al2O3/CdO and CMC were fabricated by nanosecond laser ablation. The detected $2\theta$ values agreed with the value established by the Joint Committee on Powder Diffraction Standards (JCPDS) (file no 79-1558). FT-IR spectra discussed that the dopant is found to interact with the polymeric matrix primarily through the carbonyl group (C = O) and carboxyl anion group (COO). The introduction of Al$_2$O$_3$/CdO NPs leads to an increase in the crystalline phase of the initial CMC. This is attributed to the establishment of localized ordering within the polymer matrix, facilitated by complex creation at the specified final concentration. The energy gap values decrease from 4.82 eV to 1.04 eV for indirect transition by increasing the content of cadmium oxide. The thermal analysis obtained that by adding nanoparticles of metals like aluminum and cadmium, the remaining/residual weight increases significantly. This suggests that adding nanometals to CMC boosts the thermal stability of the produced samples. Significant reduction in dielectric loss ($\varepsilon''$) is evident in the lower frequency ranges, while the mid and higher frequency ranges display only marginal changes.

Declarations

Conflicts of interest:

The authors declared that they have no conflict of interest.

Research Data Policy

The work described in this manuscript was original research that has not been published previously, and not under consideration for publication elsewhere, in whole or in part.

Author contributions statement:

M. O. Farea: Investigation, Writing - original draft, Writing - Review & Editing. M. A. El-Morsy: Investigation, Writing - original draft, Writing - Review & Editing. A. A. Menaza: Methodology, Investigation, Writing -
Data Availability Statements

Data will be available on reasonable request.

References


Figures
Figure 1

XRD patterns of pure CMC, CMC/Al$_2$O$_3$ and CMC/Al$_2$O$_3$ NPs filled with different concentrations of CdO nanoparticles.
Figure 2

FT-IR spectra of pristine CMC, CMC/Al$_2$O$_3$ and CMC/Al2O3 NPs filled with different concentrations of CdO nanoparticles.
Figure 3

UV-vis spectra of CMC, CMC/Al₂O₃ and CMC/Al₂O₃ NPs filled with different concentrations of CdO nanoparticles.
Figure 4

The relation between $\alpha(\nu)$ and $h\nu$ for CMC, CMC/Al$_2$O$_3$ and CMC/Al$_2$O$_3$ NPs filled with different concentrations of CdO nanoparticles.
Figure 5

Plots of $(\alpha h \nu)^2$ vs $h \nu$ of CMC, CMC/Al$_2$O$_3$ and CMC/Al$_2$O$_3$ NPs doped with different concentrations of CdO nanoparticles.
Figure 6

Plots of $(\alpha h \nu)^{1/2}$ vs $h \nu$ of CMC, CMC/Al$_2$O$_3$ and CMC/Al$_2$O$_3$ NPs doped with different concentrations of CdO nanoparticles.
Figure 7

TGA curve of weight loss as a function of temperature for CMC, CMC/Al$_2$O$_3$ and CMC/Al$_2$O$_3$ NPs doped with various quantities of CdO nanoparticles.
Figure 8

The dependence of log(\(\sigma\)) on log(\(f\)) for CMC, CMC/Al\(_2\)O\(_3\) and CMC/Al\(_2\)O\(_3\) NPs filled with different concentrations of CdO nanoparticles.
Figure 9

The variation of $\varepsilon'$ vs log(f) CMC, CMC/Al$_2$O$_3$ and CMC/Al$_2$O$_3$ NPs filled with different concentrations of CdO nanoparticles.
Figure 10

The variation of $\varepsilon''$ versus log(f) for CMC, CMC/$\text{Al}_2\text{O}_3$ and CMC/$\text{Al}_2\text{O}_3$ NPs filled with different concentrations of CdO nanoparticles.
Figure 11

Argond Plot for CMC, CMC/Al₂O₃ and CMC/Al₂O₃ NPs filled with different concentrations of CdO nanoparticles.