Enhanced optical and electrical properties of CeO$_2$NPs/chitosan nanocomposites

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Abstract

Cerium oxide nanoparticles (CeO$_2$NPs) of different ratios (x = 5, 10, 15, and 20 in wt%) are successfully incorporated into chitosan (CS) to synthesize CeO$_2$NPs/CS nanocomposites by solution cast method. FTIR and XRD analysis confirmed the effective incorporation of CeO$_2$NPs into chitosan nanocomposites. TGA and DTG showed that the thermal stability of the as-prepared nanocomposites is improved. The CeO$_2$NPs/CS nanocomposites exhibited enhanced light absorption capacity in the UV-visible range as x increases, owing to the CeO$_2$NPs' large bandgap. The transmittance of UV decreased for x = 10 and 15 nanocomposites. Light scattering enhanced for x = 5 and 10 nanocomposites, increasing reflectance. Compared to CS (5.3 eV), the optical energy bandgap lowers to 4.94 eV and 5.1 eV, respectively. Impedance spectroscopy research validates the impedance spectroscopy parameters’ dependency on CeO$_2$NPs concentrations. Because of the growth of multiple polarization types, generating interfaces of numerous defects, and space charge polarization, the dielectric constant increases with increasing x (up to x = 15). The dc conductivity ($\sigma_{dc}$) and the frequency exponent (S) are estimated using the universal Josher’s power law and applied to the ac conductivity data ($\sigma_{ac}$). Obviously, (S) decreases with increasing temperature, which refers to the electrical conductivity that follows the hopping mechanism. In addition, according to the CBH model, the Coulomb barrier of charge carriers (Um) is estimated, showing decreasing values as increasing x and recording the lowest value for x = 15 nanocomposites. Nyquist plots ($Z''$ & $Z'$) indicate one semicircle arc behavior for all samples. As x rises, the radius of semicircular arcs reduces, suggesting that ($\sigma_{DC}$) increases. The enhanced characteristics of CeO$_2$NPs/CS nanocomposites make them suitable for future bio-applications.

1. Introduction

Nanotechnologies and nanomaterials are now topics of intense research activity due to the better specifications of nanoparticles in large-scale daily applications [1]. It strongly relates to the so-called quantum size effect, which improves the electrical characterizations of materials. The increase in the surface area of nanoparticles to volume ratio improves their transport and chemical interactions, resulting in distinct physical properties [2]. The incorporation of nanoparticles into biopolymers results in biopolymer nanocomposites. They are used in biosensors, solar cells, super-capacitors, drug delivery, and other disciplines [3]. Chitosan is a cationic polysaccharide obtained by alkaline N-acetylation of chitin, the second-most abundant natural polymer after cellulose [4]. Despite its specific structural qualities, CS lacks a majority of the physical properties such as optical, photovoltaic, electrical, and mechanical capabilities. The modulation of these specifications of CS could expand its employment in more applications. One of the most routes to modify the chemical structure of chitosan is mixing with inorganic fillers, such as clay [5], multi-walled carbon nanotubes [6], and TiO$_2$ [7], to produce new organic/inorganic composites. These emergent composites retain organic and inorganic components; therefore, they possess significant characteristics. The organic materials are characterized by flexibility,
insulation, ductility, and curing capacity, while the inorganic fillers are characterized by hardness, thermal stability, and toughness. Metal-organic polymers are well-known composite materials that contain an inorganic metal ion and are based on organic materials. They have a wide variety of applications, including energy storage [8, 9], sensor creation [10], catalysis [11], and gas separation. These biocompatible composite materials have been used in medicine delivery [12] and food packaging [13]. Evaluating the physical characteristics of such biopolymer nanocomposites is necessary for particular applications. CuS NPs reinforced chitosan show enhanced thermal stability, dielectric constant, ac conductivity, and mechanical characteristics [14]. Fe3O4 NPs loaded to chitosan introduced improved thermal, optical, and dynamic mechanical properties, as reported in our previous work [15]. Similarly, CeO2/ZrNPs incorporated into chitosan enhanced the thermal degradation parameters and the mechanical properties of the chitosan-based nanocomposites [16]. Depending on the dopant amount of C60, the optical constants and optical energy bandgap of C60/chitosan composites have altered substantially [17]. CeO2 NPs have been identified as a novel nano-filler that may be loaded into tissues to protect against a wide range of reactive oxygen species [18]. Furthermore, CeO2 nanoparticles promote wound healing in vitro and in vivo [19–23]. CeO2 NPs coupled with chitosan, on the other hand, boost antibacterial activity [24]. CeO2 NPs exhibited no negative impacts on liver enzyme activity, liver tissue, kidneys, or blood parameters regarding health safety. CeO2 nanoparticles, on the other hand, improved the blood redox state [25].

The simple casting approach is used in this work to efficiently incorporate CeO2 NPs into chitosan at various concentrations and investigate their influence on the structural, thermal, optical properties, dielectric constant, and ACR&DC conductivity of chitosan-based nanocomposites. Furthermore, the most significant regulated factors, such as the critical concentration of CeO2 NPs for the best results, are analyzed.

2. Experimental

2.1. Materials and methods

We purchase chitosan (CS) powder of average molecular weight (100,000–300,000) and molecular formula (C6H11NO4)n from Acros Organics, and Cerium oxide of molecular formula (CeO2) and particle size of less than 50 nm from Sigma-Aldrich.

2.2. Preparation of CeO2 NPs/CS nanocomposites

We stirred a 50 ml distilled water mixture, 2% acetic acid, and 750 mg chitosan magnetically at 25°C for three hours, then x wt% of CeO2 NPs (x = 0, 5, 10, 15, 20), sonicated separately for two hours in 10 ml distilled water, was slowly added into the mixture separately and stirred for one hour. We distributed the solution into leveled hydrophobic polystyrene Petri dishes (10 cm diameter). The solution is left to dry for 24 h at 40°C.

Nanocomposite films of 50 μm thickness on average were finally peeled off from the trays and placed in sealed containers to avoid moisture exchange.

2.3. Characterizations

X-ray diffraction (XRD) patterns were performed using a 1390 Philips diffractometer with filtered Cu Kα radiation at 40 kV and 20 mA. XRD data were obtained for samples with dimensions of 2 × 2 cm², 2θ-step of 0.02°, and measurement range of 5°–70°.

The prepared nanocomposite films’ absorbance, transmittance, and reflectance were measured using a UV–visible–NIR spectrophotometer (JASCOV-670) in the range of 200–900 nm (for a 1 × 3 cm² sample area). FTIR spectra were obtained (JASCO, FTIR-300 E. Spectrophotometer) in the spectral range 400–4000 cm⁻¹, with a resolution of 4 cm⁻¹ for a 1 cm² sample area. TGA analysis was carried out (Labsys Evo (France) thermal analyzer) with a heating rate of 10°C min⁻¹ under a nitrogen atmosphere (for a 10 mg sample). To assess the dielectric constant and AC conductivity of the nanocomposites, a 3532–50 ICR HiTESTER (Hioki, Nagano, Japan) was functioned at 10⁴–106 Hz range under different temperatures (298K–333K) (for 1 cm² sample area).

The dielectric constant (ε′) and the dielectric loss (ε″) are obtained from the following relation [26]:

\[
\varepsilon^*(\omega) = \varepsilon' - i\varepsilon'' = \frac{\varepsilon(\omega)d}{\varepsilon_0 A} - i\frac{d}{\omega Z(\omega) \varepsilon_0 A}
\]

where ε' and ε'' are the real and imaginary parts of the complex permittivity, respectively. d is the thickness of the sample, A is the cross-sectional area, and the angular frequency is \(\omega = 2\pi f\). The permittivity of vacuum is \(\varepsilon_0 = 8.854 \times 10^{-12} \text{ Fm}^{-1}\).

The real \(Z'(\omega)\) and imaginary \(Z''(\omega)\) parts of the complex impedance are calculated using the following mathematical expressions [26]:
where $G$ is the measured parallel conductance.

3. Results and discussions

3.1. FTIR, microstructure, and thermal stability properties

Figure 1 represents FTIR spectra of $x$ wt% CeO$_2$ NPs/CS nanocomposites, $x = 0, 5, 10, 15$, and 20. The main characterized FTIR bands of the prepared nanocomposites are assigned and listed in table 1. Results of FTIR spectra for CS show absorbed broad bands centered at $3199$ cm$^{-1}$, $2919$ cm$^{-1}$ & $2852$ cm$^{-1}$, and $1536$ cm$^{-1}$ & $1403.8$ cm$^{-1}$, which correspond to O–H & N–H groups, CH$_2$ & CH$_3$ groups, and amide-II & CH$_2$ bending respectively. Incorporation of CeO$_2$ NPs into CS with different concentrations induced significant shifts in the characterized bands (table 1), confirming the adequate bonding of CeO$_2$ NPs into the polymer chain, which would affect the characteristic properties of the CeO$_2$ NPs/CS nanocomposites [27, 28].

![Figure 1. FTIR Signals for x wt% CeO2NPs/CS nanocomposites, x = 0, 5, 10, 15, and 20.](image)

| Bands                      | $x = 0$ | $x = 5$ | $x = 10$ | $x = 15$ | $x = 20$ |
|----------------------------|---------|---------|----------|----------|----------|
| C–O stretch                | 1016.17 | 1018    | 1016     | 1016     | 1016.17  |
| C–O stretch                | 1063    | 1065    | 1065.1   | 1063     | 1061     |
| C–H stretch                | 1381    | 1381    | 1379.4   | 1381     | 1377.3   |
| C–C stretch                | 1399    | 1401    | 1401.8   | 1399     | 1399     |
| N–O asymmetric stretch     | 1526    | 1530    | 1530.4   | 1526     | 1522     |
| N–H bend                   | 1628    | —       | —        | 1628     | 1626     |
| C–H (stretch)              | 1636    | 1634    | 1632     | 1636     | 1634     |
| $\omega = \frac{G}{G^2 + \omega^2C^2}$, $\omega'' = \frac{C\omega}{G^2 + \omega^2C^2}$, (2)
size of CeO₂ NPs is determined to be in the 32 nm range using Sherrer’s equation [34]. According to the observed shift in the DTG peaks from $T = 70 \degree C$ and 163 $\degree C$ to 100 $\degree C$ and 173 $\degree C$, respectively, CS containing 5 wt% CeO₂ NPs may withstand thermal degradation better than pure CS (figure 3). This data confirms the thermal stability improvement of the 5wt% CeO₂ NPs/CS nanocomposites up to 250 $\degree C$.

3.2. Optical properties
UV–vis absorption spectra of CS and CeO₂ NPs/CS nanocomposites are represented in figure 4(a). CS films have modest absorption across the 200–800 nm wavelength range, but CeO₂ NPs/CS films demonstrate improved absorption as CeO₂ NPs concentration increases. It might be due to the CeO₂ NPs’ broad energy bandgap up to 500 nm, resulting in significant UV absorption for the CeO₂ NPs/CS nanocomposites. This data shows that the CeO₂ NPs/CS nanocomposites preserved the CeO₂ NPs’ intrinsic optical characteristics. Furthermore, as seen in the transmittance spectra in figure 4, CeO₂ NPs/CS nanocomposites virtually completely block UV light below 300 nm for $x = 5$ and extensively block UV light for $x = 10$ and 15 nanocomposites. Furthermore, the reflectance of the prepared CeO₂ NPs/CS nanocomposites films shows a substantial influence on CeO₂ NPs concentrations, as shown in figure 4(c). The nanoparticles will unavoidably
scatter light and increase reflectance, as seen for $x = 5$ and 10, before declining again as CeO$_2$ NPs rise to 15\% [35]. The UV blocking activity and low transparency of CeO$_2$ NPs/CS nanocomposites are attractive features in various applications [36]. The energy bandgap ($E_g$) of CS and CeO$_2$ NPs/CS nanocomposites is estimated by extending the straight-line in the plot of $(\alpha h\nu)^{1/2}$ against $(h\nu)$ shown in figure 5. ($E_g$) of CS is in the range of...
5.35 eV, but \( (E_g) \) of CeO\(_2\) NPs/CS nanocomposites is estimated to be 4.94 eV and 5.1 eV (for \( x = 5 \) and 10 respectively) and 6.22 eV (for \( x = 15 \)).

### 3.3. Dielectric constant and AC&DC conductivities

Figure 6 depicts the variation of \( \varepsilon' \) with \( f (10^2-10^5 \text{ Hz}) \) for CeO\(_2\) NPs/CS nanocomposites films \( (x = 0, 5, 15, \) and 20) at various temperatures (297 K-333 K). It is obvious that \( \varepsilon' \) can be modified at different temperatures and frequencies by adding varying amounts of CeO\(_2\) NPs into the CS film. For \( x = 15 \) samples, the greatest value of \( \varepsilon' \) (270) is obtained at 100 Hz and 333 K. Because of the accumulating effect of CeO\(_2\) NPs for \( x > 15 \) nanocomposites, it seems that the \( \varepsilon' \) range grows for \( x = 5 \) and 15 nanocomposites, then decline for \( x = 20 \) nanocomposites. This improvement in \( \varepsilon' \) can be attributed to the creation of various forms of polarization due to the integration of CeO\(_2\) NPs, which produce interfaces [37, 38]. Under an external electric field, the interfaces mentioned above have a significant number of defects with uneven charge distribution and, as a result, space charge polarization [39]. \( \varepsilon' \) is substantially enhanced at lower frequencies due to dipoles’ inclination to align with the electric field. However, when \( f \) grows, the space charge polarization response cannot follow the change in the electric field, resulting in a decrease in the polarization contribution to \( \varepsilon' \). The free carriers and ionic conductivity increase as the temperature rises [40, 41]. AC spectra at various temperatures are taken to explore the dependence of the ionic conductivity of the produced materials on \( x \). In this context, electrical conduction may be defined by observing the behavior of the dispersion zones and using the well-known Josher’s universal power low (equation (3)), which can be used to determine the nature of the ionic dynamics by computing the frequency exponent \( (S) \) [42].

\[
\sigma_{AC} = \sigma_{DC} + A\omega^S
\]  

(3)

where \( (\sigma_{AC}) \) is the ac conductivity, \( (\sigma_{DC}) \) is the dc conductivity, \( (A) \) is constant, \( (\omega) \) is the angular frequency, and \( (S) \) is the frequency exponent.

Figure 7 depicts the dependency of \( \log (\sigma_{AC}) \) on \( \log (\omega) \) at various temperatures, along with the fitting curves for \( x = 0, 5, 15, \) and 20 nanocomposites. The estimated values of \( (S) \) presented in table 2 drop noticeably as temperature rises, suggesting that the electrical conductivity of these materials may follow the hopping process. For \( x = 15 \) and 20, the electrical conductivity rises. This increase in electrical conductivity by increasing CeO\(_2\) NPs content in CeO\(_2\)NPs/CS nanocomposites attributes to the function of metal oxide nanoparticles in bridging the gap between two localized states, lowering the potential barrier and allowing for a more straightforward charge carrier transfer [43]. The Coulomb barrier of charge carriers \( (U_m) \) is calculated using the Correlated Barrier Hopping (CBH) model (equation (4)), as shown in table 2 [42].
At room temperature, the $x = 15$ nanocomposites showed the lowest value of $U_m$ and continued to fall to lower values as the temperature rises. Furthermore, the $\sigma_{DC}$ of $x = 0, 5, 15,$ and 20 nanocomposites demonstrated that the $x = 15$ nanocomposites had the highest $\sigma_{DC}$ value at $T = 333 \, K$. (as shown in figure 8). These results demonstrated the role of CeO$_2$ NPs in enhancing the $dc$ electrical conductivity by reducing the Coulomb barrier at high temperatures, which suggests that thermal induction could increase the degree of overlap of potential Colombian barriers at local locations [44].

The Nyquist plots reveal one semicircle arc behavior for all samples, notably at high temperatures. A low-frequency tiny spike was detected for $x = 15$ and 20 nanocomposites at $T = 333 \, K$ due to the greatest resistivity resulting in the lowest ionic concentration and mobility (figure 9). The $x = 20$ nanocomposites have a longer spike length than the $x = 15$ nanocomposites. The electrical bulk resistance is calculated by intersecting the imperfect semicircular arc with the $Z'$ axis. Semicircular arcs with decreasing diameters confirmed the increase in $\sigma_{DC}$ for $x = 15$ and 20 nanocomposites.

$$S = 1 - \frac{6k_B T}{U_m}$$

(Figure 6. Dielectric constant ($\varepsilon'$) with frequency ($f$) under different temperatures for $x$ wt%CeO$_2$NPs/CS nanocomposites, $x = 0, 5, 15$, and 20. )
Conclusions

CeO$_2$NPs/CS nanocomposites were successfully prepared and analyzed by XRD, FTIR, TGA, and DTG. The optical properties and ac & dc conductivities of CeO$_2$NPs/CS were investigated in order to assess the impact of included CeO$_2$ NPs on the characterizations of the chitosan nanocomposites. FTIR and XRD results confirmed the efficient incorporation of CeO$_2$ NPs into chitosan nanocomposites. TGA and DTG results show that thermal stability has improved. According to the transmittance spectra, the UV radiation shielding performance of CeO$_2$NPs/CS nanocomposites strengthened. The optical energy bandgap ($E_g$) decreases, particularly for $x = 5$ and 10 nanocomposites. The enhancement of $\sigma_{AC}$ and $\sigma_{DC}$ for CeO$_2$NPs/CS nanocomposites relates to the ability of the CeO$_2$ NPs to lower the Coulomb barrier. The lowering of ($S$) with temperature indicates that ac conductivity follows the hopping process. The semicircular arcs observed in the Nyquist plots show decreasing diameters as the CeO$_2$ NPs concentration increases. These results demonstrate the rise in $\sigma_{DC}$, particularly for $x = 15$ nanocomposites. CeO$_2$NPs/CS nanocomposites are promising for bio-applications due to their superior structural, optical, and electrical characteristics.

Table 2. The exponent of the angular frequency ($S$) and the Coulomb barrier of charge carriers ($U_m$) for $x$ wt% CeO$_2$NPs/CS nanocomposites, $x = 0, 5, 15, \text{and} 20$.

| CeO$_2$NPs | $S$ | $U_m$(eV) |
|------------|-----|-----------|
| $x$ (wt%)  | 297 K | 303 K | 313 K | 323 K | 333 K | 297 K | 303 K | 313 K | 323 K | 333 K |
| 0          | 0.971 | 0.963 | 0.933 | 0.894 | 0.854 | 5.29  | 4.24  | 2.42  | 1.58  | 1.18  |
| 5          | 0.969 | 0.967 | 0.956 | 0.927 | 0.845 | 4.96  | 4.75  | 3.68  | 2.29  | 1.11  |
| 15         | 0.962 | 0.952 | 0.922 | 0.88  | 0.813 | 4.04  | 3.27  | 2.08  | 1.39  | 0.92  |
| 20         | 0.969 | 0.962 | 0.935 | 0.902 | 0.861 | 4.96  | 4.13  | 2.49  | 1.71  | 1.24  |

Figure 7. log ($\sigma_{AC}$) versus log ($\omega$) (points) with their fitting (lines) for $x$ wt% CeO$_2$NPs/CS nanocomposites, $x = 0, 5, 15, \text{and} 20$ under different temperatures.

4. Conclusions

CeO$_2$NPs/CS nanocomposites were successfully prepared and analyzed by XRD, FTIR, TGA, and DTG. The optical properties and ac & dc conductivities of CeO$_2$NPs/CS were investigated in order to assess the impact of included CeO$_2$ NPs on the characterizations of the chitosan nanocomposites. FTIR and XRD results confirmed the efficient incorporation of CeO$_2$ NPs into chitosan nanocomposites. TGA and DTG results show that thermal stability has improved. According to the transmittance spectra, the UV radiation shielding performance of CeO$_2$NPs/CS nanocomposites strengthened. The optical energy bandgap ($E_g$) decreases, particularly for $x = 5$ and 10 nanocomposites. The enhancement of $\sigma_{AC}$ and $\sigma_{DC}$ for CeO$_2$NPs/CS nanocomposites relates to the ability of the CeO$_2$ NPs to lower the Coulomb barrier. The lowering of ($S$) with temperature indicates that ac conductivity follows the hopping process. The semicircular arcs observed in the Nyquist plots show decreasing diameters as the CeO$_2$ NPs concentration increases. These results demonstrate the rise in $\sigma_{DC}$, particularly for $x = 15$ nanocomposites. CeO$_2$NPs/CS nanocomposites are promising for bio-applications due to their superior structural, optical, and electrical characteristics.
Figure 8. $\sigma_{\text{DC}}$ versus x wt% contents for x wt% CeO$_2$ NPs/CS nanocomposites under different temperatures.

Figure 9. Nyquist plots ($Z'$ versus $Z''$) for x wt% CeO$_2$ NPs/CS nanocomposites, $x = 0, 5, 15,$ and $20$ under different temperatures.
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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Author contributions

All authors contributed to the study’s conception and design. Material preparation, data collection, and analysis were performed by EMA. The first draft of the manuscript was written by EMA and all authors commented on previous versions of the manuscript. All authors read and approved the final manuscript.

Declarations

Conflict of interest

The authors declare that they have no conflict of interest.

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