Influence of Cr$_2$O$_3$ nanoparticles on the structural, optical, thermal and electrical properties of PEO/CMC nanocomposites

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Abstract

In the present paper, different concentrations of chromium oxide (Cr$_2$O$_3$) nanoparticles (≤ 0.6 wt%) were incorporated within PEO/CMC polymer blend to produce nanocomposite films using the casting technique. X-ray diffraction was performed on PEO/CMC-Cr$_2$O$_3$ nanocomposites. The main X-ray peaks of Cr$_2$O$_3$ were observed and defined as cubic structure and orthorhombic shape. The average particle size was calculated by Scherer’s equation in the range between 50 and 60 nm. A decrease of some IR bands after the addition of Cr$_2$O$_3$ nanoparticles was found, which was attributed to the interactions between PEO/CMC and Cr$_2$O$_3$. The effect of Cr$_2$O$_3$ nanoparticles on optical properties such as absorbance and optical energy gap ($E_g$) was characterized using UV–Vis spectroscopy. The $E_g$ was reduced after the addition of Cr$_2$O$_3$ nanoparticles. The AC conductivity ($\sigma_{ac}$), dielectric constant ($\varepsilon'$), dielectric loss ($\varepsilon''$) and the dielectric modulus ($M'$ and $M''$) were calculated at the frequency range of 0.1 Hz -7 GHz. The increase in direct conductivity ($\sigma_{dc}$) indicates the resulting free charge density or charge mobility. The calculated values of both $\varepsilon'$ and $\varepsilon''$ were decreased with increasing the frequency. The addition of Cr$_2$O$_3$ nanoparticles causes the formation of a charge-transfer complex. The Cole–Cole plot between ($M'$ and $M''$) shows a semi-circular shape which discusses according to a non-Debye method.

Keywords PEO/CMC blend · Cr$_2$O$_3$ nanoparticles · X-ray · ATR-IR · AC conductivity

1 Introduction

The blending between different polymers is an attractive method as well as providing new materials at a low cost during preparation of polymer films. Polymer blends are used extensively in the literature due to their practical importance and uses in various applications based on their properties (Patel et al. 2015; Wang et al. 2020). The physical properties of the polymeric blend depend mainly on the morphology, and preparation methods. The importance of studying the properties of the blends is due to obtain new applications.
in several fields (Wang et al. 2020; Choudhary 2017; Awad et al. 2020; Doh et al. 2013; Saputra et al. 2014; Gaabour 2020). In recent research, nanoscience has opened a new field for the uses of polymeric blending combined with some nanoparticles to be present on new nanocomposites.

Poly (ethylene oxide (PEO)) is a widely studied polymer attributed to its multiple applications, but also as a model material for understanding the basic behaviour of polyethylene with polar units in the backbone series, PEO has the range ability to form a complex with nanomaterials. PEO has moderate strength, good mechanical and electrical properties. Some optical and electrical works are reported for using the electrolyte polymer film. The chemical composition of PEO because the presence of ether end groups, and –OH makes some possibilities for the formation of the hydrogen bond which is confirmed by PEO with the other polymers during the blend method (Choudhary 2017).

Carboxymethylcellulose (CMC) polymer is a cellulose derivative, and it is used in a wide range such as a thickener, bonding linkages, and stabilizers, etc. CMC has been widely applied in medicinal products for its high-water absorption, morphogenetic, biodegradation, and biocompatibility uses. Also, CMC could have been mostly used as an additive to other polymers due to their limited processability (Awad et al. 2020; Doh et al. 2013; Saputra et al. 2014; Gaabour 2020).

Nanoparticle materials are attracted increasing attention during the last years because of their physical properties with wide applications in different fields of technology. Several researchers have studied the dispersion of the nanoparticles in a polymer and/or polymer blend. The unique properties of the polymeric materials will enhance with the addition of some nanoparticles such as related to the composition, amount of the nanoparticles, particle size and the homogeneity between them (Zhang et al. 2005; Hassen et al. 2012). Chromium oxide (Cr$_2$O$_3$) molecules are important in different applications such as green dyes, corrosion resistance and thermal protection. Cr$_2$O$_3$ nanoparticles are attracted increasing attention in the last years because of their individual properties. Several researchers focused on the dispersion of the Cr$_2$O$_3$ nanoparticles into a polymer matrix (Singh et al. 2009; Nouh et al. 2017).

The importance of nanomaterials is due to their physical properties, which make their use in wide applications. Although there are many previous studies concerned with studying nanocomposite materials with PEO/CMC polymer blends, but there are no complete studies to analyze the effect of Cr$_2$O$_3$ on the physical properties of PEO/CMC blends. So in the present work we have prepared a new nanocomposites based on PEO/CMC blend incorporated with low weight ratios (0.0, 0.2, 0.4 and 0.6 wt.% of Cr$_2$O$_3$ nanoparticles and studied the changes of the structural, optical, AC electrical conductivity and dielectric properties, while comparing the obtained results with some of the previous reports.

2 Experimental details

2.1 Materials

Polyethylene oxide (PEO) powder with average molecular weight – 100,0000 g/mol and carboxymethyl cellulose (CMC) with average molecular weight – 90,000 g/mol, and powder of chromium (III) oxide (Cr$_2$O$_3$) nanoparticles with particles size < 100 nm with molecular weight = 151.99 g/mol were obtained by Sigma Aldrich Company, UK.
2.2 Synthesis of PEO/CMC- \( \text{Cr}_2\text{O}_3 \) nanocomposites

Double distilled water is used as the common solvent of all the components. The solution casting technique is utilized to prepare the PEO/CMC blend and PEO/CMC doped with different contents of \( \text{Cr}_2\text{O}_3 \) nanoparticles. Firstly, equal weights (2.5 g) of both PEO CMC polymers were dissolved in 200 ml of DD at 60 °C for about 4 h. Different concentrations (0, 0.2, 0.4 and 0.6 wt.%) of \( \text{Cr}_2\text{O}_3 \) nanoparticles were dispersed in DD water and added to PEO/CMC according to the formula:

\[
\text{wt\%} = \frac{w_{\text{Cr}}}{W_{\text{blend}} + w_{\text{Cr}}} \times 100
\]

where, \( w_{\text{Cr}} \) is the weight of \( \text{Cr}_2\text{O}_3 \) and \( W_{\text{blend}} \) is the weight of PEO/CMC blend. The solutions of \( \text{Cr}_2\text{O}_3 \) nanoparticles were an addition to PEO/CMC solution with stirring to prevent any agglomeration of the filler inside the blend. The final solutions of the PEO/CMC-\( \text{Cr}_2\text{O}_3 \) were cast in Petri dishes and left in an oven for about three days at 50 °C to evaporate the solvent. The thickness of the samples are prepared in the range –120 μm for X-ray, FT-IR and UV–Visible spectroscopy and in the range –300 μm for AC measurements. The obtained the PEO/CMC-\( \text{Cr}_2\text{O}_3 \) nanocomposite films were stored until characterized.

2.3 Measurements

The PEO/CMC-\( \text{Cr}_2\text{O}_3 \) nanocomposite samples were studied using the X-ray diffractometer with CuKα target and the wavelength 1.5408 Å (Model D/Max2500VB2+/PC, Rigaku Co., Japan) with Bragg’s angle 2θ = 5–70°. Fourier transform infrared (FT-IR) absorption spectra were obtained in wavenumber 4000–400 cm\(^{-1}\) using FT-IR spectrometer (Model Nicolet iS10 spectrometer, USA). The ultra and visible (UV–Vis) measurements were obtained by Jasco 570 spectrometer in the range of wavelength about 190–1000 nm. The electrical conductivity data were recorded using Novocontrol turnkey concept 40 System at a frequency of about 0.1 Hz–7 MHz.

3 Results and discussion

3.1 X-ray diffraction

The X-ray diffraction of pure \( \text{Cr}_2\text{O}_3 \) nanoparticles is shown as an insert in Fig. 1. The X-ray peaks of pure \( \text{Cr}_2\text{O}_3 \) nanoparticles found in the figure are in agreement with the literature (Zhang et al. 2005; Singh et al. 2009). The X-ray peaks at 2θ = 24.8, 32.6, 33.2, 41.4, 50.6, 85.5, 63.4 and 65.3° which are assigned to Bragg reflections (012), (104), (110), (113), (024), (116), (214) and (300), respectively. The crystalline peaks are defined cubic and orthorhombic shapes and indexed according to JCPDS 84–1616.

The X-ray diffraction of pure PEO/CMC polymer blend and the blend filled with 0.0, 0.2, 0.4 and 0.6 wt% of \( \text{Cr}_2\text{O}_3 \) nanoparticles at the Bragg’s angle 2θ from about 5–70° at room temperature is recorded in Fig. 1. From the literature, the pure PEO has two broad peaks at 20 – 19.7 and 23.8° and pure CMC is a non-crystalline structure with a broad
peak at about $2\theta - 20.4^\circ$ suggested by the amorphous nature structure for CMC pure polymer (Gaabour 2020; Abdel-Galil et al. 2014; Elashmawi et al. 2014).

The broad peak or the hump is observed in all samples. There is a decrease of the broad peak after the addition of Cr$_2$O$_3$ nanoparticles addition to other peaks at $2\theta - 32.6$, 33.9, 36.2 and 40.8.4° which is attributed to Cr$_2$O$_3$ nanoparticles because of the interactions between the OH groups inside the polymeric matrices and Cr$^{3+}$ions. The decrease in the broad peak $2\theta - 20.5^\circ$ with the disappearance of some peaks causes an increase of the amorphous region (domain amorphous phase) in the PEO/CMC-Cr$_2$O$_3$ nanocomposite samples which increase the segmental motion inside the polymeric matrices and confirming the increase of the ionic mobility and increase of the ionic conductivity of the prepared nanocomposites.

The crystallite sizes ($D$) can be calculated from Scherer’s formula as (Chandra and Kumar 2013; Rani et al. 2019):

$$D = \frac{K\lambda}{\beta\cos\theta}$$

where, $K = 9$, $\lambda = \lambda = 0.15406$ nm, and $\beta$ is the half width for the peak and half. The crystallite sizes are found to be in the range of 50–60 nm.

### 3.2 FT-IR study

The infrared spectroscopy (FT-IR) study is used to investigate and characterize the interaction between the components in the prepared nanocomposite samples. The FT-IR absorption spectra for the pure PEO/CMC polymer blend the blend doped with different concentrations of Cr$_2$O$_3$ nanoparticles is shown in Fig. 2. The position and assignments of the main peaks of PEO and CMC polymers are to relate to as in the literature.

The FT-IR spectra display the main groups of both pure polymers PEO and CMC like OH, CH$_2$, COO$^-$ groups in the CMC and C–O–C function group in the PEO polymer. The chemical interactions that occur between PEO and CMC are attributed to the formation of the hydrogen bond between the OH groups in CMC and C–O–C of PEO. occur. The absorbance bands at wavelengths about 1591 cm$^{-1}$, 1416 cm$^{-1}$ and
1347 cm$^{-1}$ are observed. The intensity of the FT-IR bands at 2871 cm$^{-1}$, 1469 cm$^{-1}$, 1347 cm$^{-1}$ and 1281 cm$^{-1}$ are nearly decreased confirming that to completely interaction and compatible between the two polymers (Patel et al. 2015; Doh et al. 2013; Basu et al. 2017; Polu and Rhee 2016; Rajeh et al. 2019).

The broad absorption band in the wavelength centred at about 3440 cm$^{-1}$ is assigned to the symmetric and asymmetric stretching of –NH$_2$, and OH groups. The band at 2871 cm$^{-1}$ is assigned to the C–H–CH$_2$ group in the pure CMC. The absorption band at 1590 cm$^{-1}$ is assigned to the asymmetric expansion of a –COO–. After the addition of Cr$_2$O$_3$ nanoparticles to the polymer blend, the intensity of the FT-IR bands at 2871 cm$^{-1}$, 1469 cm$^{-1}$, 1347 cm$^{-1}$ and 1281 cm$^{-1}$ are nearly decreased confirming that to completely miscible and compatible between the two polymers and Cr$_2$O$_3$ nanoparticles. This indicates that hydrogen gives new bonds in the catenary of the nanoparticles that these groups interact with the Cr nanoparticles as shown in Scheme 1.

![Fig. 2 The FT-IR absorption spectra for PEO/CMC blend incorporated with (0.0, 0.2, 0.4 and 0.6%) of Cr$_2$O$_3$ nanoparticles](image)

![Scheme 1 Possible interaction between PEO/CMC and Cr$_2$O$_3$ nanoparticles](image)
3.3 UV–Vis study

The ultraviolet and visible (UV–Vis) absorption spectra of pure PEO/CMC polymer blend and the blend doped with different concentrations of Cr$_2$O$_3$ are shown in Fig. 3. It is observed that the UV–Vis absorbance increased with increases of Cr$_2$O$_3$ nanoparticles inside the PEO/CMC polymeric matrices. Two absorption bands found at 251 and 373 nm in all spectra of the prepared nanocomposites are Cr$_2$O$_3$ due to the formation of charge-transfer complexes.

The optical energy gap ($E_g$) is estimated using UV–Vis spectra related to the frequency dependence of the energy. Detailed information about the band structure of nanocomposites can be obtained by studying optical absorption by exciting the electrons from a lower energy level to a higher energy level than by absorbing the amount of energy (photon) enough to transition. The optical absorption coefficient ($\alpha$) (The ability of the sample to absorb light related to the wavelength) is given from the Beer-Lambert’s relation as (Siddaiah et al. 2018; Gaur and Rana 2014):

$$\alpha = \frac{2.303A}{d}$$ (3)

where $A$ is the absorbance and $d$ is the thickness of the sample. optical absorption coefficient ($\alpha$) can be estimated using the absorbance from the equation (Sharma et al. 2016; Mohammed 2018):

$$\alpha hv = B(hv - E_g)^n$$ (4)

where $B$ is a constant and $n$ is equal 1/2, 3/2, 2 or 3 for direct allowed direct forbidden, indirect allowed and indirect forbidden transition, respectively. The graph of $(\alpha hv)^{1/2}$ against the energy of the photon ($hv$) gives us to determine the values of the optical energy gap ($E_g$) using extrapolating the linear part of $(\alpha hv)^{1/2}$ at zero as we see in Fig. 4. The value of $E_g$ of pure PEO/CMC blend agreement with previous works. After the addition of Cr$_2$O$_3$ nanoparticles, the values $E_g$ are decreased due to the increased occurrence of the interaction between Cr$_2$O$_3$ and OH groups inside the PEO/CMC polymer blend and due to incorporation of the Cr$_2$O$_3$ forms charge transfer complex inside PEO/CMC matrices.
The presence of the charge-transfer causes an increase in the AC electrical conductivity ($\sigma_{ac}$). Finally, the decrease of the optical energy band gap is due to the impurity amount in the localized state through the band gap which finally decreases the $E_g$ values. These results are consistent with the results in the X-ray study and the AC electrical conductivity data.

### 3.4 Electrical conductivity study

#### 3.4.1 AC electrical conductivity

The values of AC electrical conductivity ($\sigma_{ac}$) of PEO/CMC-Cr$_2$O$_3$ nanocomposite samples is estimated from the equation (Tripathi et al. 2012):

$$\sigma_{ac} = \frac{t \cdot C}{A}$$

The variation the AC electrical conductivity (Log $\sigma$) against the frequency (Log $f$) of PEO/CMC incorporated by concentrations of Cr$_2$O$_3$ nanoparticles.
where, $t$ is thickness, $A$ the cross-sectional area and $C$ is the conductance of the used nanocomposite samples. Figure 5 shows the relation between the $\log(\sigma_{ac})$ versus frequency of PEO/CMC-Cr$_2$O$_3$ nanocomposite samples at room temperature in the range of the frequency from 1 Hz to 7 MHz. Generally, the gradual increases in the AC electrical conductivity with an increase of frequency is common behaviour for the polymer nanocomposites. There are three phases in the AC conductivity spectra as we see in the figure. The low frequency at the first region shows low values of the AC conductivity attributed to the electrode polarization related to the slow periodic reversal of the electric field. The plateau region is found at the medium range of frequency (DC conductivity could be evaluated in this region). The third region is clear at the high frequency due to space charge polarization at the blocking electrodes.

The AC conductivity values increase with an increase of the addition of Cr$_2$O$_3$ confirms to the presence of charge carriers that could be carried via hopping in defect sites along polymer chains. The raise in $\sigma_{ac}$ is discussed according to the extended chain length inside the PEO/CMC matrices that allows the hopping of the charge carriers and this supports the carriers of charge to hop between the favourable localized sites. This can be due to the tendency in polymeric samples of the dipoles to orient themselves in the direction of the field applied. However, in the high frequency range, as opposed to the lower frequency region, the growing pattern seems to be sharp. On all graphs with various concentrations of metals, this effect is found.

### 3.4.2 The dielectric permittivity

Figures 6 and 7 show the complex dielectric permittivity, real part ($\varepsilon'$) and loss part ($\varepsilon''$), spectra of PEO/CMC-Cr$_2$O$_3$ films. The values of the $\varepsilon'$ are a clear indicator of its capacity to store electrical energy, while the values of $\varepsilon''$ the energy loss in the dielectric. The values of $\varepsilon'$ are exponentially decreased as a raise of the frequency and it approaches the stable level above 300 kHz. The higher values for $\varepsilon'$ are measured at lower frequencies due to the

![Fig. 6 The plot between the dielectric constant ($\varepsilon'$) against the frequency (Log f) of PEO/CMC incorporated different contents of Cr$_2$O$_3$](image-url)
dominant contribution of the interfacial polarization effect which displays an accumulation of charges with various conductivity within the dielectric materials, obtain from the micro capacitances over the whole volume of material (Table 1).

In general, the nanocomposite samples in this article have a three phase (two for PEO/CMC blend and the other phase for Cr$_2$O$_3$ nanoparticles. Then, the values of $\varepsilon'$ are high with an increase as increases of Cr$_2$O$_3$ which is attributed to the high value of dielectric permittivity of Cr$_2$O$_3$ nanoparticles. An increase of $\varepsilon'$ is occurred due to the interactions between the Cr$_2$O$_3$ nanoparticles with the polar functional groups inside the PEO/CMC polymer blend and the changing of the dipolar ordering gives the variation of the dielectric properties. As we see in Fig. 7, the spectra $\varepsilon''$ exhibit dramatically increase up to 3 kHz which is attributed to the interfacial polarization effect.

The high value of $\varepsilon''$ confirms the raise of the energy loss per cycle inside the nanocomposite films. After the addition of Cr$_2$O$_3$, the increase of $\varepsilon''$ values. The effects of AC
conduction loss are appearing from space charges such as ions or electrons and due to the reorientation of the dipolar.

### 3.4.3 The complex dielectric modulus

The complex dielectric modulus ($M^*$) is used to study the relaxation phenomena in AC electrical behaviour. The modulus ($M^*$) is calculated as (Baskaran et al. 2004):

$$M^* = \frac{1}{\varepsilon_s} = M' + M''$$  \hspace{1cm} (6)

$$M' = \frac{\varepsilon'}{\varepsilon_r^2 + \varepsilon''^2} \quad \text{and} \quad M'' = \frac{\varepsilon''}{\varepsilon_r^2 + \varepsilon''^2}$$  \hspace{1cm} (7)

where $M'$ is the real part of the electric modulus and $M''$ is the imaginary part of the electric modulus. Figures 8 and 9 obtain the behaviour of both $M'$ and $M''$ against the log of...
the frequency. From the figures, at low frequencies, the values of both $M'$ and $M''$ attend almost zero, assigned to the negligible contribution of electrode polarization.

The low values of the two complex modules suggest that the electrode effect has a marginal electrode polarisation contribution and can thus be overlooked. The step-like transformation approaches an asymptotic value relative to the frequency and represents the extremely capacitive nature of the samples. At medium of frequency, the values of $M'$ are linear increase with increases of frequency and exponentially increase at high frequency.

In the $M''$ graph, it found a relaxation peak that offers an indicator of the existence of the phase transition that occurs attributed to the transition regions from long-range ionic (translation) to short-range (dipolar) mobility. The magnitude of $M''$ peak increases with increases of the Cr$_2$O$_3$ nanoparticles. The shift for the position of the peak to low frequency occurs as an increase of Cr$_2$O$_3$ nanoparticles increases the conductivity relaxation time.

Figure 10 shows the diagram between the real part ($M'$) and the imaginary ($M''$). The diagram shows the half semicircle which discusses according to the Debye style relaxation mechanism and the absence of contact effects. This specific function is attributed to the grain site or due to the mobility of free charges caused by interactions between both the components of the blend. Then, relaxation is due to the complex of conductivity relaxation. With the rise in Cr$_2$O$_3$ nanoparticles, the collected semicircle increases, indicating that this sample is more conductive.

Two relaxation regions are appeared below and above the $M''$ peak. The DC conductivity arising from the continuous hopping phase in which the charge carriers are mobile over long distances can be connected to the area of low frequencies. Although the area of the high-frequency side is due to the mechanism of relaxation polarisation and indicates the spectrum of frequencies at which the ions are confined spatially to their potential wells and the ions can only pass inside the wells in the short range. The AC conductivity-related right side of the peak benefits from the reversible motion of ions over a small space. Therefore, the peak frequency reflects the change from long to short of the mobility as an increase in frequencies.
4 Conclusions

In the present paper, different concentrations of chromium oxide (Cr$_2$O$_3$) nanoparticles (≤ 0.6 wt%) were incorporated within PEO/CMC polymer blend to produce nanocomposite films using the casting technique. X-ray diffraction was performed on PEO/CMC-Cr$_2$O$_3$ nanocomposites. The main X-ray peaks of Cr$_2$O$_3$ were observed and defined as cubic structure and orthorhombic shape. The average particle size was calculated by Scherer’s equation in the range between 50 to 60 nm. A decrease of some IR bands after the addition of Cr$_2$O$_3$ nanoparticles was found, which was attributed to the interactions between PEO/CMC and Cr$_2$O$_3$. The effect of Cr$_2$O$_3$ nanoparticles on optical properties such as absorbance and optical energy gap (E$_g$) was characterized using UV–Vis spectroscopy. The E$_g$ was reduced after the addition of Cr$_2$O$_3$ nanoparticles. The AC conductivity ($\sigma_{ac}$), dielectric constant ($\varepsilon'$), dielectric loss ($\varepsilon''$) and the dielectric modulus ($M'$ and $M''$) were calculated at the frequency range of 0.1 Hz–7 GHz. The increase in direct conductivity ($\sigma_{dc}$) indicates the resulting free charge density or charge mobility. The calculated values of both $\varepsilon'$ and $\varepsilon''$ were decreased with increasing the frequency. The addition of Cr$_2$O$_3$ nanoparticles causes the formation of a charge-transfer complex. The Cole–Cole plot between ($M'$ and $M''$) shows a semi-circular shape which discusses according to a non-Debye method.

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Declarations

Conflict of interest There is no conflict of interest.

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