



# The role of TiO<sub>2</sub> nanoparticles in enhancing the structural, optical, and electrical properties of PVA/PVP/CMC ternary polymer blend: nanocomposites for capacitive energy storage

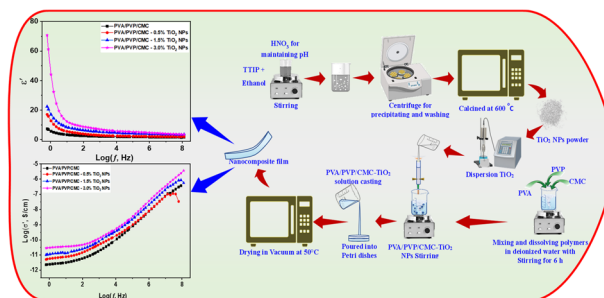
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## Abstract

Herein, titanium dioxide (TiO<sub>2</sub>) nanoparticles (NPs) were prepared via the sol–gel technique; then, they were incorporated into a ternary blend polymer matrix to design polymer nanocomposite (PNC) films through the solution casting technique. The ternary blend polymer matrix consisted of polyvinyl alcohol (PVA), polyvinyl pyrrolidone (PVP), and carboxymethyl cellulose (CMC). X-ray diffraction (XRD) analysis revealed reductions in the crystallinity structure of the polymer matrix after adding TiO<sub>2</sub> NPs. The optical study manifested increases in the refractive index and reduction in the optical bandgap values, which reduced from 4.97 eV for the pure polymer blend to 4.77 eV for the PNC film at TiO<sub>2</sub> content of 3 wt%. Additionally, the transmission edge gradually shifted towards lower energy. The PNC films exhibited considerable improvements in the dielectric constant ( $\epsilon'$ ), dielectric loss ( $\epsilon''$ ), dielectric moduli ( $M'$  and  $M''$ ), and electrical conductivity characteristics over the range of frequency range from 0.1 Hz to 10 MHz. The addition of TiO<sub>2</sub> NPs improved the electrical conductivity and dielectric constant significantly. The electrical conductivity increased by over ten times compared to the pure ternary polymer blend, and  $\epsilon'$  also rose four-fold at 100 Hz. The enhancement in the electrical and dielectric parameters of the PNC films after adding TiO<sub>2</sub> nanofiller could indicate the suitability of these samples for flexible-type energy storage applications, such as dielectric capacitors.

## Graphical Abstract



**Keywords** TiO<sub>2</sub> NPs · Sol–gel · Polymers · Nanocomposite · Energy storage

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## Highlights

- TiO<sub>2</sub>-NPs were prepared via sol–gel technique.
- TiO<sub>2</sub>-NPs were used as a nanofiller with PVA/PVP/CMC blend to prepare nanocomposites.
- The FTIR and XRD indicated the interaction between the polymer blend and TiO<sub>2</sub>-NPs.
- The band gap of PVA/PVP/CMC blend decreased with increasing TiO<sub>2</sub>-NPs content.
- The electrical and dielectric results show the use of the samples in energy storage.

## 1 Introduction

Integrating nano-sized components into polymer blends creates nanocomposites with superior properties compared to micro- and macro-composites [1, 2]. The nanocomposite blended polymer's organic and inorganic material features have positioned it for use in a wide range of technical applications, including transistors for electrical switches, solar photovoltaic cells, and the electrode of energy storage [1, 3]. Compared to pure polymers and conventional composites, the metal oxides based nanocomposites display considerable advantages in mechanical, thermal, and barrier properties, allowing them to serve as a source of high-performance new materials with several new applications [4–12].

Using the polymer blend technique can enhance the amorphous phases of a semicrystalline polymer [13]. Several polymers have been utilized to prepare solid polymer blends, such as chitosan (CH)/methylcellulose (MC) [14], polyethylene oxide (PEO)/carboxymethyl cellulose [7], PVA/sodium alginate (SA) [8], PEO/MC [13], PEO/ polyvinyl pyrrolidone (PVP) [9], and CH/PVA [15]. These prepared polymer blends exhibited enhanced electrical and dielectric properties compared to their pristine polymers.

PVA is a polymer with hydroxyl groups linked to a carbon derived from methane and has several characteristics, including excellent transparency, flexibility, and nontoxicity. PVP polymer possesses both hydrophobic and hydrophilic functional groups, making it soluble in a variety of solvents and water. CMC, which is a cellulose derivative, is used in the food, cosmetic, and pharmaceutical sectors. Furthermore, CMC has good film-forming characteristics, which results in transparent coatings [16].

Nanoparticles (NPs) incorporation into the matrix of synthetic and polymer films is one of the most successful ways to improve them. Titanium dioxide (TiO<sub>2</sub>) is a highly promising material that has been incorporated with different materials [8, 17–25] due to its many desirable qualities. It is chemically stable, transparent in the visible range, has a high dielectric constant, is nontoxic, has a low unit cost, and has a large band gap. It is extensively used in many applications, including but not limited to photo-electrochemistry [18], dye-sensitized solar cells [17], electrochromic devices [19], gas sensing [20], lithium-ion

batteries anode [23], catalysis [24, 26], waveguide applications [25], and other applications with polymers [8, 21]. Additionally, TiO<sub>2</sub> is a potential material for replacing carbon-based anodes in lithium-ion batteries [27]. TiO<sub>2</sub> NPs are also well-known for their many benefits, such as their high photocatalytic activity, hydrophilic qualities, UV blocking ability, an increase of certain physicochemical features, ability to strengthen nanocomposites, and antibacterial capacity [28].

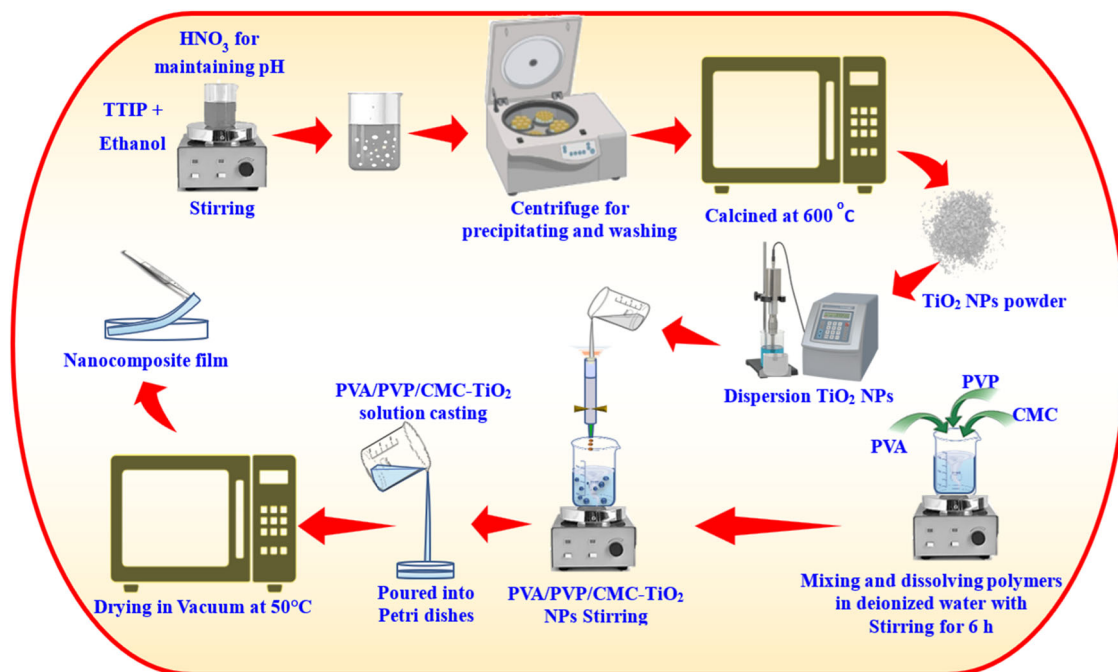
Several efforts [6, 8, 29–31] have been performed in recent years on the production of polymer nanocomposite (PNC) films by combining various polymers with TiO<sub>2</sub> NPs. By incorporating TiO<sub>2</sub>, Ren et al. [29] improved the mechanical performance of PVA/xylan composite films. According to Bisen et al. [30], PVA with minimal doping is ideal for transmitting desired properties without causing bond ruptures in the polymer host. PNCs that use a blend of PVP and CMC, along with hybrid NPs made from multi-walled carbon nanotubes and silver, were prepared and found appropriate for an assortment of applications, including optoelectronics and nanodielectrics [32].

Despite the aforementioned efforts, there is still a need for functional materials with excellent dielectric properties, low cost, and ease of preparation for energy storage applications. Herein, extensive research was conducted on the TiO<sub>2</sub> NPs prepared via the sol-gel route; then, they were used to design PVA/PVP/CMC of PNC films, analyzing their crystallinity and chemical functional groups through X-ray diffraction (XRD) and Fourier-transform infrared spectroscopy (FTIR), respectively. Additionally, their optical and morphological properties were thoroughly examined using UV–Vis and transmission electron microscope (TEM), respectively. Moreover, the conduction mechanisms of these samples were also investigated and systematically interpreted.

## 2 Experimental work

### 2.1 Materials

The polymers used in this work are PVA and CMC powder (BDH Chemicals Ltd Poole, UK) and PVP (SISCO Research Laboratory Ltd, India) powder with Mw of 14000,



**Scheme 1** A diagram illustrating the process of preparing TiO<sub>2</sub> nanoparticles and PNC films

250,000, and 72,000 g/mol, respectively. Also, titanium isopropoxide [C<sub>12</sub>H<sub>28</sub>O<sub>4</sub>Ti] with a purity of 99%, nitric acid [HNO<sub>3</sub>], and ethanol from (Sigma-Aldrich, Germany) were used during TiO<sub>2</sub> NPs preparation. Deionized water was for washing and during the preparation.

## 2.2 TiO<sub>2</sub> NPs and nanocomposites preparation

Per the previous study [26], TiO<sub>2</sub> NPs were prepared via the sol-gel technique with necessary modification and utilized as an inorganic nanofiller to prepare PVA/PVP/CMC/TiO<sub>2</sub> nanocomposites. Three hours were spent calcining the TiO<sub>2</sub> powder at 600 °C. The obtained TiO<sub>2</sub> NPs were then used to fabricate the PVA/PVP/CMC/TiO<sub>2</sub> PNC films via the solution casting process. A 120 ml of deionized water was used to dissolve (1.225 g PVA + 0.735 g PVP + 0.49 g CMC) to create the blend solution of the PVA/PVP/CMC (50/30/20 wt%). Then, the TiO<sub>2</sub> nanofiller was mixed into the blend solution at varied filling levels of 0.5 and 1.5, and 3.0 wt% while being stirred at 50 °C. Then, we produced the PNC of PVA/PVP/CMC-TiO<sub>2</sub> solutions and cast them into glass Petri plates before drying them at 50 °C. The thicknesses of the prepared PNC films range from (0.004–0.005 cm). Scheme 1 summarizes the process of preparation of TiO<sub>2</sub> NPs and PNC films.

## 2.3 Characterization

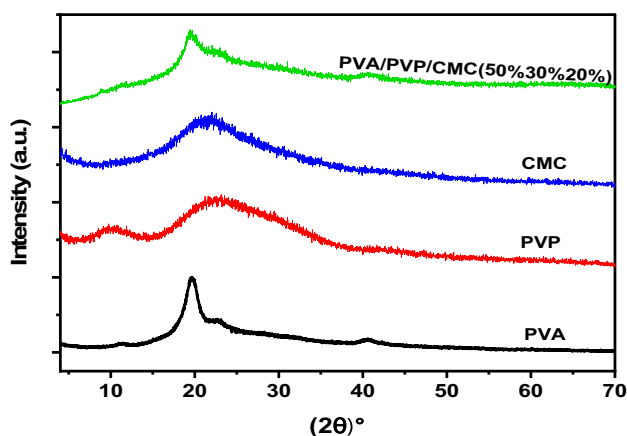
We utilized the XRD model X'Pert PRO (Malvern PANalytical, USA) to analyze the crystal structures of the

nanofiller and nanocomposite PNC films. The examination was conducted within the 3–80° range using a CuK source with a wavelength of 1.5418 Å. To obtain the FTIR spectra for the PNC films, we utilized an FTIR spectrometer, model Nicolet iS10 (Thermo Scientific, USA). Additionally, we used TEM, model JEM-2100 (JEOL, Japan), to measure the size and shape of the TiO<sub>2</sub> NPs. We utilized a spectrophotometer, model V-570-UV-VIS-NIR (JASCO, Japan), to measure transmittance spectra with a precision of 0.2 nm, between 190 and 2500 nm. The PVA/PVP/CMC-TiO<sub>2</sub> PNC films' electrical conductivity and dielectric characteristics were conducted at room temperature in a dry nitrogen atmosphere using model Concept 40 (Novocontrol Technologies, Germany). The cell that was used for electrical and dielectric measurements is a three-terminal electrode (three electrodes, i.e., top, bottom, and guard electrodes), and the applied V<sub>rms</sub> voltage is 1 V.

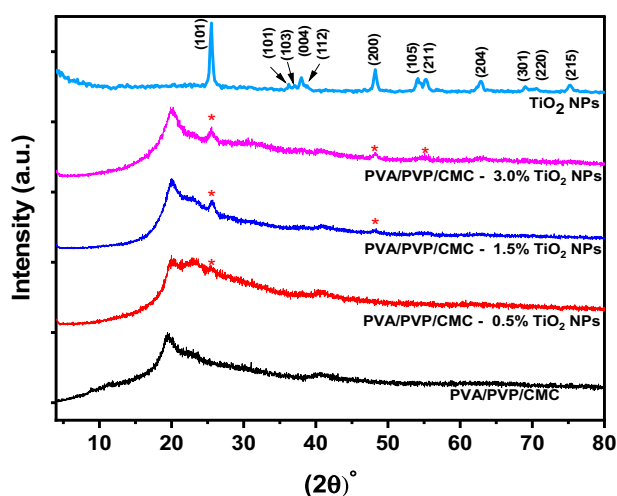
## 3 Results and discussion

### 3.1 XRD analysis

XRD investigations play a crucial role in understanding microstructural changes in polymeric materials. In this regard, we utilized XRD to determine whether the PVA/PVP/CMC-TiO<sub>2</sub> NPs PNC films had amorphous or crystalline areas and evaluate how TiO<sub>2</sub> NPs affected the PNCs' structural properties.



**Fig. 1** XRD patterns of the PVA, PVP, CMC, and PVA/PVP/CMC ternary blend



**Fig. 2** XRD patterns of the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt%

XRD patterns for the PVA, PVP, CMC, and PVA/PVP/CMC ternary blend are exhibited in Fig. 1. The PVA/PVP/CMC ternary blend is characterized by a broad peak centered at  $2\theta \approx 19.52^\circ$ , which is attributed to the semicrystalline nature of the PVA with (101) reflective plane [33, 34]. The evident broadness in this main peak confirms the semicrystalline nature of the current ternary blend.

XRD patterns of the ternary polymers blend filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt% are illustrated in Fig. 2. In the XRD patterns of the prepared nanocomposites, with the addition of TiO<sub>2</sub> NPs, it can be observed a little increase in the broadening of the broad peak (shadowed by green) seen in the pure PVA/PVP/CMC blend, suggesting decreasing in crystalline ratios in the PVA/PVP/CMC blend matrix. Besides, there is a shift, in the position of the same broad peak, towards a higher angular angle. Moreover, new sharp diffraction peaks were observed at  $2\theta = 25.52^\circ$ ,  $48.24^\circ$ , and  $55.28^\circ$ ; based on the XRD standard card

**Table 1** The calculated value of crystalline degree ( $X_{dc}$  %), indirect/direct energy gap ( $E_g$ ), and refractive index ( $n$ ) for the present films

Sample	$X_{dc}$ %	$E_g$ (eV)		$n$
		Direct	Indirect	
Pure PVA/PVP/CMC	23.03	5.43	4.97	2.00
PVA/PVP/CMC -0.5%Ti NPs	21.62	5.39	4.91	2.01
PVA/PVP/CMC -1.5%Ti NPs	17.77	5.27	4.72	2.04
PVA/PVP/CMC -3.0%Ti NPs	13.01	5.34	4.77	2.04

(JCPDS file No. 21-1272), these diffracted peaks were attributed respectively to (101), (200), and (211) atomic plane in the TiO<sub>2</sub> crystal structure, confirming the successful synthesis of the tetragonal anatase phase of TiO<sub>2</sub> NPs [35]. The intensity of these peaks also increased with the rise in nanofiller contents, indicating the effective complexation and compatibility of the TiO<sub>2</sub> NPs with the ternary blend host material. It appears that the TiO<sub>2</sub> NPs have integrated well with the blend matrix. Upon comparing the XRD of pure TiO<sub>2</sub> and PNCs filled with TiO<sub>2</sub> NPs at 0.5, 1.5, and 3 wt%, some diffraction peaks corresponding to the lattice planes of pure TiO<sub>2</sub> were not observed in the PNCs. This result could be due to two reasons: the low TiO<sub>2</sub> content (maximum 3%) or the TiO<sub>2</sub> nanofillers wrapped inside the polymer blend matrix.

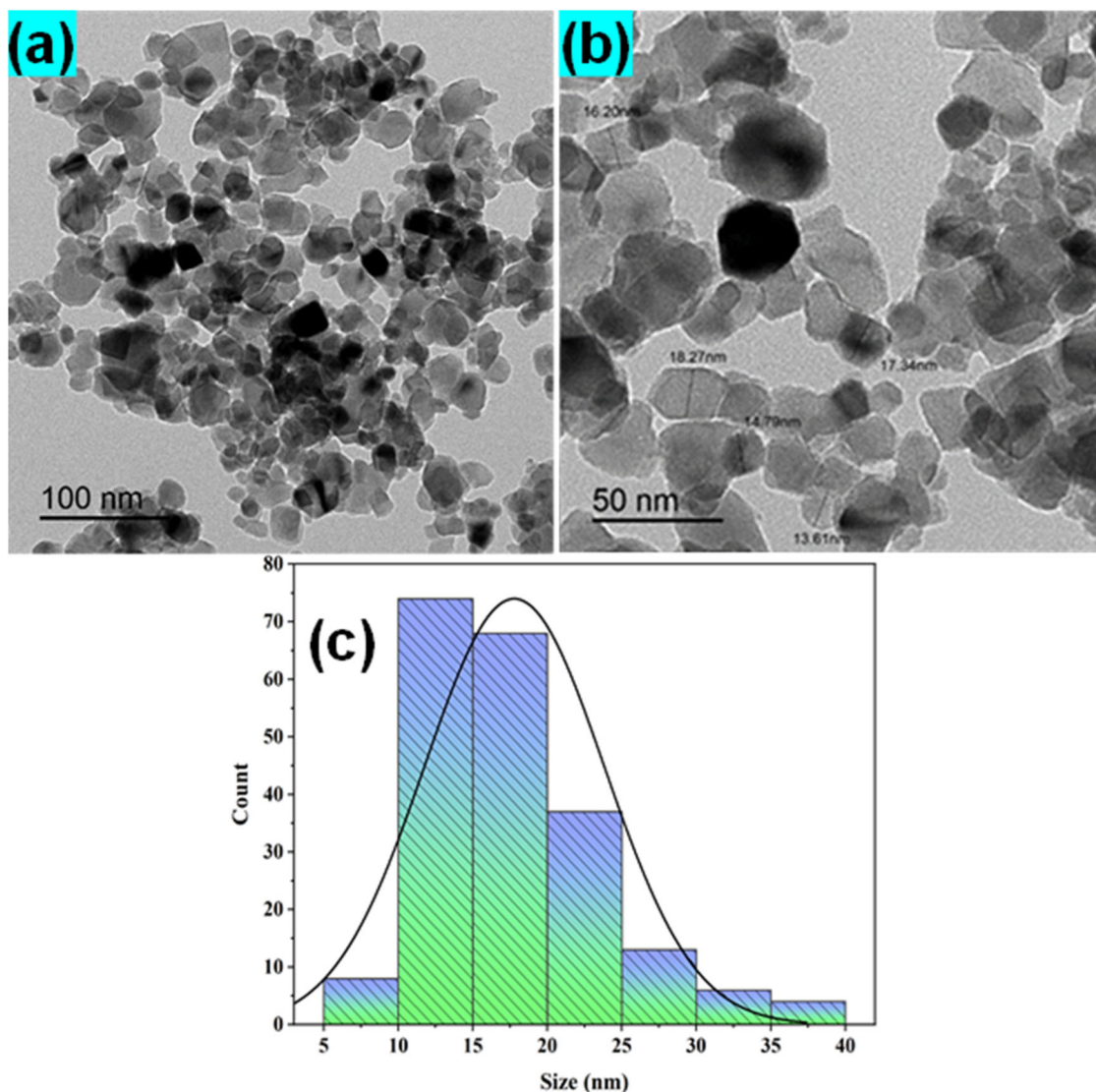
The Scherrer equation was employed to determine the average crystallite size, which involves the use of the following formula:

$$X_w = \frac{0.94 \lambda_i}{D_c \cos \theta_m} \quad (1)$$

where,  $\theta_m$  is Bragg's angle,  $X_w$  represents the full width at half maximum of the diffraction peak, and  $\lambda$  is the X-ray wavelength of the XRD machine ( $\lambda_i = 0.154056$  nm). The calculated  $D_c$  value of TiO<sub>2</sub> NPs is 12.42 nm. The diffraction peaks in the PNC films' XRD patterns were attributed to PVA, PVP, CMC, and TiO<sub>2</sub> NPs. The crystallinity degree ( $X_{dc}$  %) was determined using the Hermans-Weidinger method [36]:

$$X_{dc} = \frac{(\text{area under crystalline peaks})}{(\text{the total area under all peaks})} \times 100 \quad (2)$$

Table 1 shows the calculated  $X_{dc}$  % values of the PVA/PVP/CMC-TiO<sub>2</sub> NPs nanocomposite samples. The change in the crystalline structure could be referred to as the impact of TiO<sub>2</sub> NPs on the composition of the PVA/PVP/CMC blend matrix. Where this result suggests that adding TiO<sub>2</sub> NPs with strong crystalline properties to the PVA/PVP/CMC ternary polymer blend could physically increase the amorphous areas in the polymer blend, decreasing its semicrystalline nature. This decrease in semicrystalline nature



**Fig. 3** a, b The TEM micrographs of the TiO<sub>2</sub> NPs, (c) its congruous histograms related to the nanoparticles size distribution

could chemically indicate an increase in disorder caused by a reduction in the number of intermolecular bonds in polymer blend chains. The reduction in the crystalline structure could lead to an increase in ionic mobility and hence electrical conductivity and enhancement in its applicability in energy storage devices.

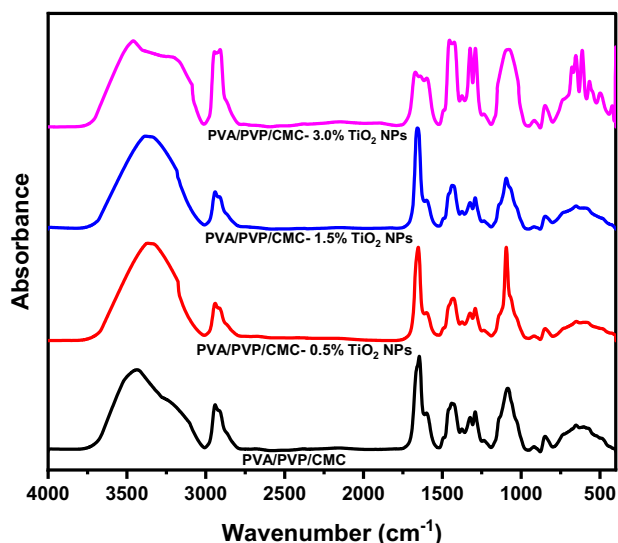
### 3.2 TEM analysis

Figure 3a, b shows the TEM micrograph of the TiO<sub>2</sub> NPs prepared by the sol-gel method, besides their congruous histograms, respectively; the histograms reveal the size and distribution sizes. The TEM micrographs show that the TiO<sub>2</sub> NPs have a uniform distribution behavior. Further, the TiO<sub>2</sub> NPs shapes vary from spherical to hexagonal and rectangular. The particle sizes were in the range between 5

and 40 nm; the particle size of the prepared TiO<sub>2</sub> NPs was nearly 17.5 nm.

### 3.3 FTIR spectroscopy

To estimate the chemical functional groups and changes in the molecular structures, FTIR spectroscopic analysis was conducted for the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt%. In the FTIR spectral charts (Fig. 4), certain bands were observed, including the stretching vibration mode for the -OH group of CMC that centers at 3438 cm<sup>-1</sup>, and the band at 2940 cm<sup>-1</sup> could belong to the asymmetrical stretching vibrational mode of the H-C-H. The band centered at 1450 cm<sup>-1</sup> is assigned to the -CH<sub>2</sub> scissoring vibrational modes [37]. The -CN stretching band of PVP located at



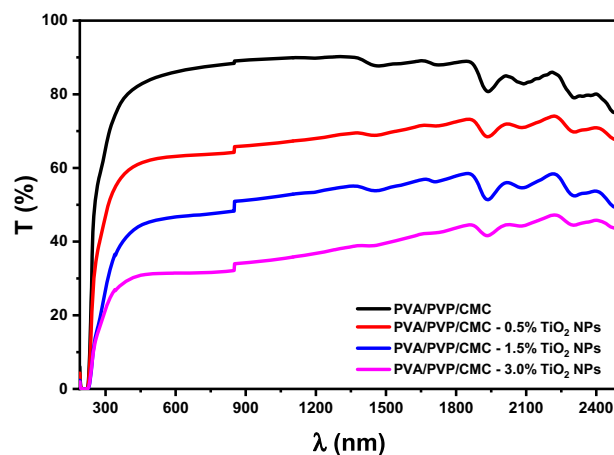
**Fig. 4** FTIR spectral charts of the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt%

**Table 2** FTIR spectra bands' assignments for the pure blend of ternary polymers of PVA/PVP/CMC and the prepared PNC samples

Wavenumber (cm <sup>-1</sup> )	Band assignment
3438	-OH str.
2940	H-C-H asymmetrical str.
1650	C=O str.
1450	-CH <sub>2</sub> scissoring <u>or</u> C=N of pyridine ring
847	-CH rocking
1292	-CN str.
923	-CH bending (out-of-plane rings)
2906	CH <sub>2</sub> symmetrical str. (shoulder)
1030	C-O str. (shoulder)
1600	-COO <sup>-</sup> str.
1374	CH <sub>2</sub> bending
1324	-CH or -OH bending
1230	-CH wagging
1086	-OH bending
734	-CH <sub>2</sub> rocking
615	-CH wagging

1292 cm<sup>-1</sup> is attributed to the pyridine ring, while the band observed at 923 cm<sup>-1</sup> could result from the bending vibration mode of the -CH group [37]. A summary of the assigned bands for the FTIR spectra of the samples can be found in Table 2.

The FTIR spectra of the PNC films prepared exhibited minor shifts in some peaks by comparing with the FTIR spectrum of the blend of ternary polymers alone. For instance, the 3438 cm<sup>-1</sup> band shifted towards a lower wavenumber to 3357 cm<sup>-1</sup> after introducing TiO<sub>2</sub> NPs. The intensity of several bands also displayed considerable



**Fig. 5** The UV/VIS spectra in the wavelength region from 190 to 2500 nm for the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt%

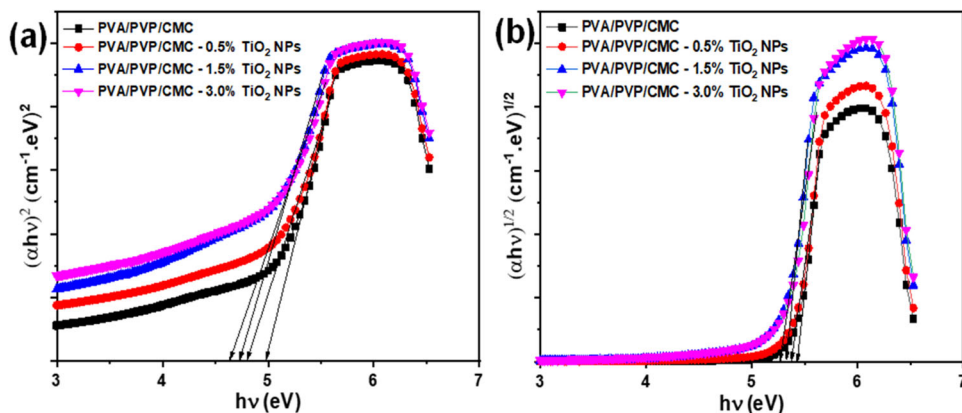
variations, which may be attributed to the physicochemical interactions between the ternary polymers' blend matrix and the TiO<sub>2</sub> NPs [38]. The variations in intensity peaks of the PNCs prepared with 0.5%, 1.5%, and 3% TiO<sub>2</sub> content could be attributed to TiO<sub>2</sub> nanofillers. These nanofillers cause an increase in the amorphous phase, as revealed by XRD analysis. The amorphous phase causes decreases in the intermolecular interactions between the chains of the PVA/PVP/CMC ternary blend, resulting in significant changes in the chemical functional groups that appear in the FTIR spectra peaks. Additionally, the two observed FTIR bands noted at nearly 1600 and 1324 cm<sup>-1</sup> were identified as the asymmetric stretching vibration mode of the carboxylate (-COO-) group and the bending vibration of the hydroxyl group (-OH), respectively. These two functional groups significantly contribute to the likelihood of intermolecular hydrogen bonding between TiO<sub>2</sub> NPs and the PVA/PVP/CMC blend [39], thus improving the structural features of their PNCs. Similar behavior was found in the CMC/polyacrylamide (PAM)/Co<sub>3</sub>O<sub>4</sub> [40] and PVA/PVP/CMC/ZnO [31] PNCs films.

Due to the presence of oxygen in the ether linkage, the PVP and CMC polymers could be able to engage in intermolecular hydrogen interactions with various molecules. This results in the formation of a high-intensity band at 1086 cm<sup>-1</sup>, which is indicative of -OH bending [41]. The enhancements in the intensity of this band could be a sign of the interaction between the polymers that make up the blend.

### 3.4 Optical properties

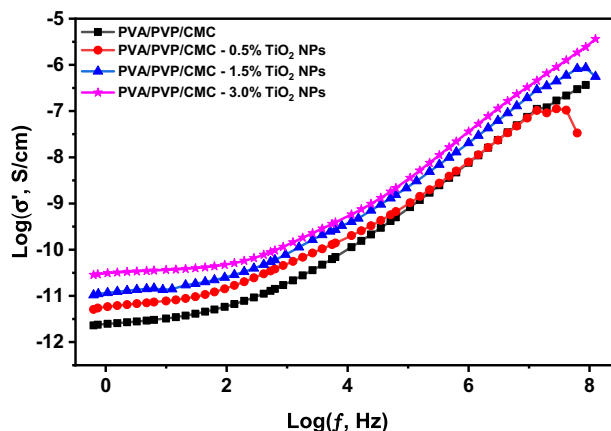
UV/VIS-NIR spectroscopy was utilized to understand how the PVA/PVP/CMC blend interacts with TiO<sub>2</sub> NPs and affects the optical behavior of the films. At room temperature, Fig. 5 showcases the PNC films' UV/VIS-NIR

**Fig. 6** Plot of (a)  $(\alpha h\nu)^2$  and (b)  $(\alpha h\nu)^{1/2}$  as functions of energy ( $h\nu$ ) for the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt%



transmittance spectra, prepared based on PVA/PVP/CMC and TiO<sub>2</sub> in the 190–2500 nm wavelength range. The UV spectrum exhibited a sharp edge at around 242 nm, with a noticeable difference in height between the pure blend and PNC samples. This variation might be attributed to the disorder caused by the presence of TiO<sub>2</sub> NPs in the polymers' host matrices. It's possible that the change observed is a result of the presence of amorphous areas within the PVA/PVP/CMC matrix alongside some crystalline regions. These findings confirm the XRD results, indicating that the prepared PNC samples are semicrystalline in nature. Additionally, the shift in the edge position from 242 nm to 255 nm clearly could suggest successful complexation between the constituents of the nanocomposites. The charge transfer absorption bands result primarily from electronic transitions, where the transitions occur from the highest occupied molecular orbital to the lowest unoccupied molecular orbital. As such, the band located around  $\approx 204$  nm is identified as the  $n \rightarrow \pi^*$  transition [42].

The optical direct ( $E_{gd}$ ) and indirect ( $E_{gi}$ ) energy gap of the pure polymers' blend and PNC samples were determined using Tauc's plots, as shown in Fig. 6a, b, respectively. From these plots, the  $E_{gd}$  and  $E_{gi}$  energy gaps were calculated. A summary of the calculated direct and indirect optical band gaps can be found in Table 1. According to the collected data, the inclusion of TiO<sub>2</sub> NPs caused a considerable reduction in the  $E_g$  values. Among all the nanocomposites, the PVA/PVP/CMC-1.5% TiO<sub>2</sub> PNC demonstrated the lowest  $E_g$  value, making it the most favorable option. These reductions in  $E_g$  values could be referred to as the presence of various polaronic contributions and imperfections that often occur in PVA/PVP/CMC-based PNCs. It has been observed that the movement direction of charge carriers is from a lower to a higher energy level during indirect transfer, as this requires only a minimal energy amount to have occurred [40]. On the other hand, direct transition necessitates a considerably larger energy amount. In contrast, the direct transition requires more energy amount. This illustration could illustrate why  $E_{gd}$ 's values are greater than  $E_{gi}$ 's.



**Fig. 7** The variation of conductivity ( $\text{Log } \sigma$ ) versus frequency ( $\text{Log } f$ ) at RT for the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt%

The refractive index ( $n$ ) values were obtained by inputting the  $E_g$  values into Eq. (3) provided in reference [43]. The findings have been compiled and presented in Table 1

$$\frac{n^2 - 1}{n^2 + 2} = 1 - \sqrt{\frac{E_g}{20}} \quad (3)$$

The increase in the prepared PNC films' refractive index by comparing it with that of the pure polymers' blend could be understood through the increase in the optical density of the PNC films. The optical density could increase because of the TiO<sub>2</sub> NPs incorporation into the polymeric matrix, which finally impacted the light penetration velocity.

### 3.5 Electric analysis

Figure 7 depicts the alternative current electrical conductivity ( $\text{Log } \sigma_{ac}$ ) variation versus frequency ( $\text{Log } f$ ) at room temperature for the virgin PVA/PVP/CMC ternary blend and the PNC films with nanofiller, i.e., TiO<sub>2</sub> NPs. It can be seen that the  $\sigma$  values of the prepared samples are enhanced with the loading of the TiO<sub>2</sub> nanofiller. The observed dispersions in the low-frequency domain are owing

**Table 3** The calculated value of DC conductivity ( $\sigma_{dc}$ ), exponentiation factor ( $s$ ), and  $\epsilon'$  (100 Hz) for the present films

Sample	$\sigma_{dc}(\text{S cm}^{-1})$	$s$	$\epsilon'_{\text{at } 100 \text{ Hz}}$
Pure PVA/PVP/CMC	$2.30 \times 10^{-12}$	0.93	2.5
PVA/PVP/CMC –0.5%Ti NPs	$5.90 \times 10^{-12}$	0.82	4
PVA/PVP/CMC –1.5%Ti NPs	$1.11 \times 10^{-11}$	0.86	6.5
PVA/PVP/CMC –3.0%Ti NPs	$2.88 \times 10^{-11}$	0.89	10

to electrode polarization or spatial charge. The  $\text{TiO}_2$  nanofiller could help to decrease potential barriers and works towards connecting two localized states, which facilitates the movement of charge carriers [44]. The relatively high conductivity of the  $\text{TiO}_2$  NPs ( $\sim 22 \times 10^{-7} \text{ S cm}^{-1}$ ) [45] also contributes to these improvements in  $\sigma_{ac}$  values of the produced PNCs. The enhanced amorphous area in PNCs samples may be responsible for this improvement in the movement of charge carriers. Jonscher's power law can represent electrical conductivity based on the following equation:

$$\sigma(\omega) = \text{DC electrical conductivity} + A(\text{angular frequency})^s = \sigma_{dc} + A\omega^s \quad (4)$$

where,  $s$  denotes the exponentiation factor. The obtained values of  $\sigma_{dc}$  and  $s$  are summarized in Table 3. The behavior of  $\sigma_{ac}$  (Fig. 7) indicates that the conduction mechanism of charge carriers in these materials utilized a correlated barrier hopping mechanism proposed by Pike [46] and Elliot [47]. It can be seen that the obtained values of  $\sigma_{dc}$  for the prepared PNCs samples reached  $2.88 \times 10^{-11} \text{ S cm}^{-1}$  for the last sample. It is known that the number of charge carriers and their mobility determine the polymeric material's conductivity. These factors can be changed by varying the amount of nanofiller present in the host ternary polymer matrix. It suggests that the adding of  $\text{TiO}_2$  NPs in the PVA/PVP/CMC ternary blend increased the favorable sites and/or presented some additional charge carriers; as a result, there may have been an improvement in the mobility and density of charge carriers, leading to an increase in  $\sigma$  values for the prepared PNCs samples. The increase in the values of both  $\sigma_{dc}$  and  $\sigma_{ac}$  indicates that these PNCs may be considered suitable applicants for electronic devices, for instance, flexible polymeric dielectric capacitors [48, 49]. Although the content of  $\text{TiO}_2$  affects the conductivity of the prepared PNC systems, it must mention here no abrupt increase in the conductivity while applying this study.

### 3.6 Dielectric properties

The dielectric constant,  $\epsilon'$ , describes the energy that has been stored in a sample, whereas the dielectric loss,  $\epsilon''$ ,

describes the energy that the sample dissipates in reaction to an outside electric field.

The values of  $\epsilon'$  and  $\epsilon''$  of the samples under examination beside the dielectric loss tangent ( $\tan \delta$ ) were determined using the following equations in terms of the capacitance ( $C$ ) and the free space permittivity ( $\epsilon_0$ ) [50–52]:

$$\epsilon' = \frac{Cd}{\epsilon_0 A} \quad (5)$$

$$\epsilon'' = \frac{\sigma}{\omega \epsilon_0} \quad (6)$$

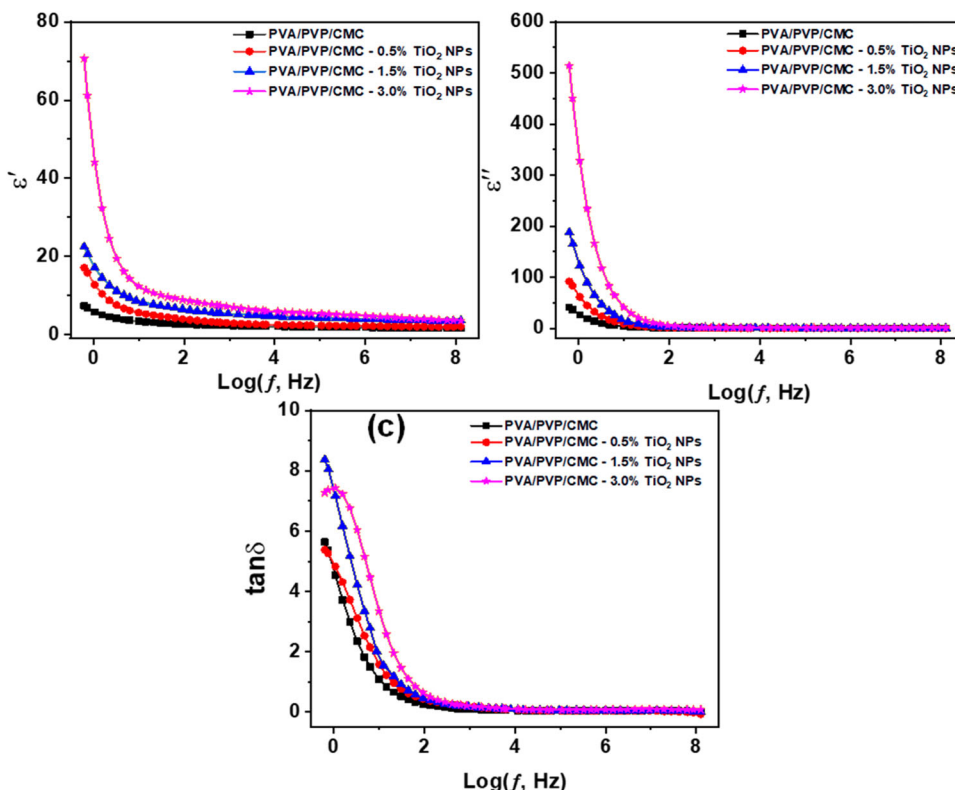
$$\tan \delta = \frac{\epsilon'}{\epsilon''} \quad (7)$$

Figure 8a, b illustrates how  $\epsilon'$  and  $\epsilon''$  values vary for all prepared PNCs films in response to various frequencies at room temperature. The obtained  $\epsilon'$  and  $\epsilon''$  spectra are observed to decrease nonlinearly when the frequencies increase. They improved when adding the  $\text{TiO}_2$  nanofillers, and similar behavior was reported in various polymers [53, 54]. At lower frequencies, interfacial polarization causes increased values of  $\epsilon'$  for the produced PNCs samples, but at higher frequencies, the value of the dielectric constant is independent of the frequency. But as the frequency of the field increases, this value gradually decreases due to the inability of all types of polarization to follow the changes in the electric field direction [40, 55]. Thus, high frequencies have a reasonably constant complex permittivity with frequency. After the addition of  $\text{TiO}_2$  nanofillers, the amount of parallel aligned dipoles in the pure ternary blend matrix increased, as evidenced by an increase in the values  $\epsilon'$ ; for instance, at 10 Hz, the value of  $\epsilon'$  for the prepared PNCs films is high in comparison to that of the pure ternary polymers' blend matrix (see Fig. 8a and Table 3). The difference between the nature of the nanofiller ( $\text{TiO}_2$  NPs) and the host polymers' blend led to the formation of micro-capacitors over the whole volume of PNCs samples [56, 57]. Additionally, it can be seen that the behavior of  $\epsilon''$  for all prepared PNC samples exhibits the same pattern of  $\epsilon'$  as observed in Fig. 8b. Also, the obtained dielectric constant value at 10 Hz in this work for PVA/PVP/CMC-5%  $\text{TiO}_2$  NPs was better than some of those mentioned in the literature (Table 4). Table 4 lists a comparison between the value of  $\sigma_{dc}$  and  $\epsilon'$  at 10 Hz obtained by this work and the published works [31, 43, 58–61].

Figure 8c displays  $\tan \delta$  variation versus frequency for prepared PNCs. It can be observed that PNCs with 0.5%, 1.5%, and 3%  $\text{TiO}_2$  exhibit similar behavior compared to pure blend PVA/PVP/CMC. The results in Fig. 8a, c suggest that although the  $\tan \delta$  is not as small as reported in the published works [62–64], it is still enhanced compared to a pure ternary polymer blend. These results could indicate that the addition of  $\text{TiO}_2$  NPs enhances the blend's energy



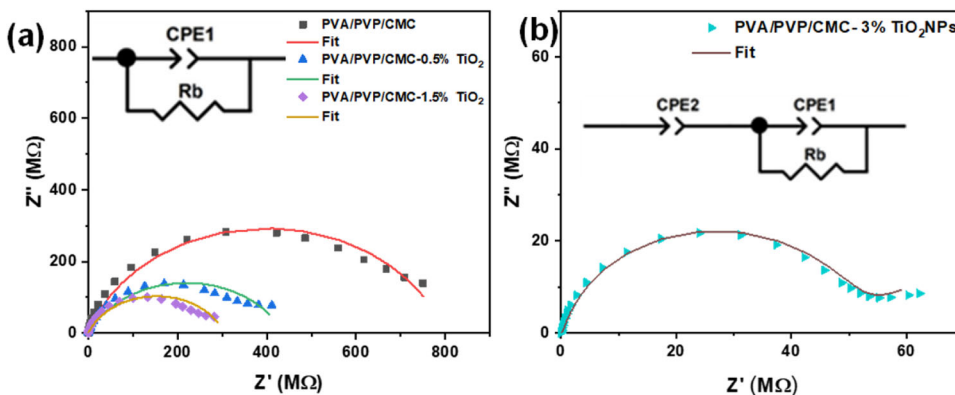
**Fig. 8** The variation of dielectric properties versus frequency for the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt.% (a), ε' (b) ε'', and (c) tan δ



**Table 4** The  $\sigma_{dc}$  and  $\epsilon'$  at 10 Hz recorded by this study and literature [31, 43, 58–61]

Work	Polymers	Filler	$\sigma_{dc}(\text{S cm}^{-1})$	$\epsilon'$
[31]	PVA/PVP/CMC	ZnO NPs (5% wt.)	$\sim 3.16 \times 10^{-11}$	$\sim 17$
[43]	PVA/CMC	SrTiO <sub>3</sub> (6%wt.)	$\sim 1.84 \times 10^{-10}$	$\sim 15$
[58]	CMC/PVA/ graphene nanoplatelets	ZnO NPs (6%)	$\sim 1.38 \times 10^{-10}$	$\sim 12.8$
[59]	PVA/PEO	ZnO NPs (5%)	$\sim 15.9 \times 10^{-12}$	$\sim 5.5$
[60]	PVP/PEO	MoO <sub>3</sub> NPs (6%wt.)	$\sim 5.60 \times 10^{-9}$	$\sim 20$
[61]	Polystyrene /polyvinyl carbazole	TiO <sub>2</sub> NPs (5%wt.)	$\sim 4.07 \times 10^{-13}$	$\sim 2.5$
This work	(PVA/PVP/CMC)	TiO <sub>2</sub> NPs (3%wt.)	$\sim 2.88 \times 10^{-11}$	$\sim 9.25$

**Fig. 9** The Nyquist plot ( $Z'$  versus  $Z''$ ) of the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt% (asterisks are the experimental data, and the solid line denotes the fitting findings)



storage ability without increasing energy dissipation. Therefore, PNCs of this ternary polymer blend with TiO<sub>2</sub> are promising nanocomposites for energy storage applications.

### 3.7 Impedance analysis

Figure 9a, b depicts the Nyquist plot ( $Z'$  versus  $Z''$ ) of the prepared PVA/PVP/CMC-TiO<sub>2</sub> samples. The conductive

properties of the bulk material are represented by semi-circles in the higher frequencies, while the tail is created at low frequencies as a result of the influence of the electrical double layer [65]. The semicircles' radii get smaller and smaller as they go closer to their origin. This suggests that the addition of TiO<sub>2</sub> nanofiller to the polymers' blend matrix could cause an increase in the disorder areas, which is supported by XRD, leading to increases in the ionic conductivity of the PNC films.

Using EIS software, the resulting  $Z''$  versus  $Z'$  data were fitted and found equal to a circuit combination. The components of this circuit are the bulk resistance (Rb) and the constant phase element (CPE) (there are two elements found in the equivalent circuit, CPE1 and CPE2). The impedances of the CPE1 and CPE2 are calculated using the following

formula [66]:

$$Z_{CPE} = 1/Q(i\omega)^n \quad (8)$$

The symbol  $Q$  represents the numerical value of  $1/Z$  at  $\omega = 1$  rad/s, while  $n$  refers to the phase of the element and indicates the deviation degree from a pure capacitor. Table 5 lists the determined fitting elements. It is worth mentioning that when the TiO<sub>2</sub> NPs were added to the prepared ternary blend, the value of (Rb) for the filled PNCs decreased compared to the value for the pure mixture, indicating that the charge transfer became more accessible inside the PNCs.

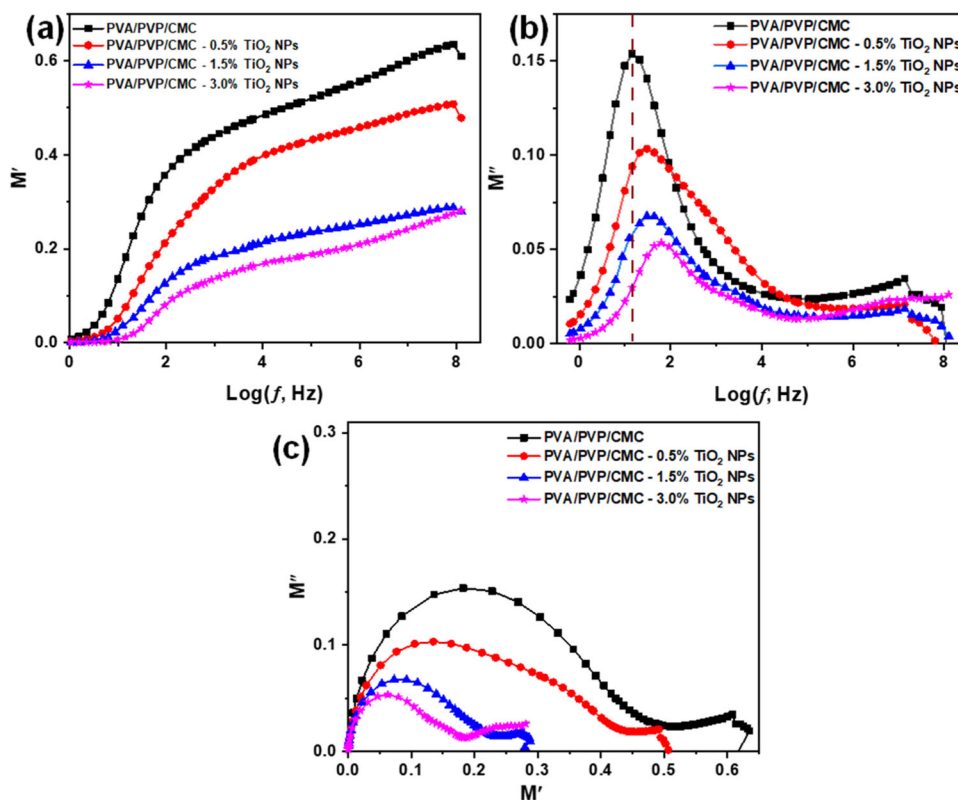
### 3.8 Dielectric Modulus

The electric modulus is the physical principle that is used to investigate the electrical relaxation mechanism of materials with ionic conductivities. Physically, electric moduli, i.e.,  $M'$  and  $M''$  are used to study the electric field relaxation in the materials when the electric displacements stay constant [67]. The prepared PNC films  $M'$  and  $M''$  measured at room temperature are illustrated in Fig. 10a, b. The dependency of  $M'$  on the frequency spectrum is depicted in Fig. 10a. The  $M'$  modulus is known as the retrograde quantity of the dielectric constant; as observed in Fig. 8a, the dielectric constant recorded higher values at lower frequencies and lower values at higher frequencies. Therefore, the  $M'$

**Table 5** The obtained parameters for fitting equivalent circuit models

Sample	Rb ( $\Omega$ )	Q <sub>1</sub> (F)	$n_1$	Q <sub>2</sub> (F)	$n_2$
PVA/PVP/CMC	$7.98 \times 10^8$	$2.73 \times 10^{-10}$	0.80		
PVA/PVP/CMC-0.5%TiO <sub>2</sub>	$4.38 \times 10^8$	$4.79 \times 10^{-10}$	0.72		
PVA/PVP/CMC-1.5%TiO <sub>2</sub>	$3.06 \times 10^8$	$5.42 \times 10^{-10}$	0.76		
PVA/PVP/CMC-3.0%TiO <sub>2</sub>	$4.79 \times 10^7$	$7.49 \times 10^{-10}$	0.90	$8.62 \times 10^{-8}$	0.35

**Fig. 10** The variation of (a)  $M'$  and (b)  $M''$  versus frequency; (c) the plot of  $M''$  versus  $M'$  behavior for the PVA/PVP/CMC ternary blend and the PNCs filled with TiO<sub>2</sub> NPs contents of 0.5, 1.5, and 3 wt%



modulus values are as expected; the  $M'$  modulus recorded the reverse behavior for the dielectric constant. It was observed that, at low frequencies,  $M'$  seems to be zero for either the pure blend polymer or PNCs with nanofiller contents (Fig. 10a). This result suggests that the contribution of the electrode/electrolyte polarization could be considered negligible [68]. Also, all PNCs reach the saturation stage at high frequencies, with  $M'$  values increasing steadily with increasing frequency (Fig. 10a). This increase in  $M'$  value is due to the electrical conductivity related to the charge/ion carriers' short-range mobility. Also, this conduct was reported [69, 70] in several PNCs systems. It can also observe that the  $M'$  values decreased while increasing the  $\text{TiO}_2$  nanofillers in the polymer matrix compared to the  $M'$  values of the pure ternary polymers; this result could suggest the effect of  $\text{TiO}_2$  NPs in the ionic conductivity of the polymer matrix. This attitude affirms that the relaxation depends upon  $\text{TiO}_2$  nanofiller.

The spectrum imaginary electric modulus,  $M''$ , of the pure ternary polymers' blend and the PNCs filled with  $\text{TiO}_2$  NPs contents of 0.5, 1.5, and 3 wt% reveals a relaxation peak (Fig. 10b). It can be noticed  $M''$  modulus manifests for the pure polymer blends, and PNCs samples a single relaxation peak that could be attributed to dc-conductivity contribution. As shown in Fig. 10b, the  $M''$  modulus relaxation peak shifted to a higher frequency when introducing the  $\text{TiO}_2$  nanofillers and continued shifting with increasing the nanofiller content. Also, it can be observed clearly, that the maximum peak of in the  $M''$  modulus decreased considerably with the increase in the  $\text{TiO}_2$  nanofillers content. As  $\text{TiO}_2$  nanofiller content increased to 3 weight percent, its intensity diminished, and its location changed. This finding suggests the possible effect of  $\text{TiO}_2$  nanofillers on the dc-conductivity contribution. Also, in the relaxation peak in the  $M''$  modulus curves, there are two parts. The first part is on the left side of the relaxation peak (at low frequency); this part represents the region of frequency in which the ions move over long distances, where the ions can easily jump between the neighbor sites. The second part is on the right side of the relaxation peak (at high frequency), where ions are trapped in their potential wells and can perform only localized motion. Asymmetry peak broadening can be noticed in the relaxation peaks, suggesting different time constants where it was found that the dielectric relaxation time ( $\tau$ ) [ $\tau \approx 1/\omega$ ] decreased considerably from 69 to 16 ms. This result could point out the non-Debye type of relaxation in the prepared PNC samples.

## 4 Conclusion

$\text{TiO}_2$  NPs were prepared via sol-gel technique; then, they were used as nanofillers with the ternary polymers' blend of

PVA/PVP/CMC to prepare PNC films. The structures, morphologies, and optical properties of the prepared pure blend polymers and the PNC films with  $\text{TiO}_2$  NPs contents of 0.5, 1.5, and 3 wt% were thoroughly examined using XRD, FTIR, TEM, and UV/VIS–NIR techniques. These techniques revealed comparatively good interactions between polymer chains and the nanofiller. The structural investigation showed that the prepared PNC samples have semicrystalline nature owing to the structure of the PVA polymer, and the addition of  $\text{TiO}_2$  NPs significantly reduced the crystallinity ratio. At the same time, the optical measurements for the ternary PNC film filled with 3% wt  $\text{TiO}_2$  exhibit decreasing in the  $E_g$  value with increasing the content of the  $\text{TiO}_2$  nanofillers. Applying these prepared PNC films, the electrical and dielectric properties were investigated to assess their suitability for dielectric capacitors. The electrical and dielectric results revealed that the  $\epsilon'$ ,  $\epsilon$ ,  $M'$ ,  $M''$ , and  $\sigma_{ac}$  values were enhanced after loading the  $\text{TiO}_2$  NPs. Also, the DC conductivity increased from  $2.30 \times 10^{-12} \text{ S cm}^{-1}$  (for pure PVA/PVP/CMC blend) to  $2.88 \times 10^{-11} \text{ S cm}^{-1}$  (for 3%  $\text{TiO}_2$  NPs). From the impedance spectra, it was found that the semicircles' radii in the Nyquist plot ( $Z''$  versus  $Z'$ ) of the prepared PNCs with  $\text{TiO}_2$  NPs samples became smaller as the  $\text{TiO}_2$  NPs content increased. The  $M'$  results indicate that the contribution of electrode/electrolyte polarization might be considered negligible. Furthermore,  $M'$  and  $M''$  values decreased as the  $\text{TiO}_2$  nanofillers content increased in the polymer matrix. The outcomes and improved electrical and dielectric features of the PNC films suggest that these samples could be suitable for flexible energy storage applications, such as capacitors and batteries.

## Data availability

The data that support the findings of this study are available from the corresponding author upon reasonable request.

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**Author contributions** MMD: Project Administration, Methodology, Review & Editing, Formal Analysis, Validation, Visualization, Funding Acquisition. AS: Supervision, Conceptualization, Methodology, Resources, Software, Data Curation, Writing—Original Draft, Review & Editing, Formal Analysis, Validation, Visualization. EMA: Writing - Original Draft, Review & Editing Formal Analysis. AMA: Writing—Original Draft, Review & Editing Formal Analysis. AYY: Conceptualization, Software, Review & Editing, Formal Analysis, Validation, Visualization. JAMA: Writing, Review & Editing. AAA-M: Supervision, Conceptualization, Methodology, Resources, Software, Data Curation, Writing—Original Draft, Review & Editing, Formal Analysis, Validation, Visualization. All authors reviewed the manuscript.

## Compliance with ethical standards

**Conflict of interest** The authors declare no competing interests.

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