



# The optical, electrical and mechanical performance of metal oxides incorporated PVA/rGO blend: effect of metal oxide type

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## Abstract

The effect of metal (M: Fe, Pb and Mn) oxides type on the optical, electrical and mechanical performance of PVA/reduced graphene oxide (PVA/rGO) blend has been explored. Plain PVA and 2.0 wt% of metal oxides polymeric composites (PCs) were equipped using the solution casting procedure. The structure variation due to metal oxides incorporation was examined by the FT-IR spectroscopy. The optical properties of the samples were obtained based on the UV–Vis–NIR measurements. The optical bandgap decreases from 5.41 eV (plain PVA) to 5.36 eV (plain PVA/rGO), 5.23 eV (Fe<sub>2</sub>O<sub>3</sub> PC), 5.27 eV (Pb<sub>3</sub>O<sub>4</sub> PC) and 5.22 eV (MnO<sub>2</sub> PC). The dc-electrical conductivity of the PVA/rGO blend is strongly enhanced via metal oxides incorporation. The activation energy of the host matrix decreases from 0.66 eV (plain PVA) to 0.18 eV (plain PVA/rGO), 0.05 eV (Fe<sub>2</sub>O<sub>3</sub> PC), 0.20 eV (Pb<sub>3</sub>O<sub>4</sub> PC) and 0.43 eV (MnO<sub>2</sub> PC). The dynamic mechanical analyzer (DMA) was used to investigate the effect of metal oxides incorporation on the mechanical properties of the host blend. The glass transition temperature ( $T_g$ ) value increases from 59.45 °C (plain PVA) to 61.56 °C (plain PVA/rGO) and 64.68 °C (Pb<sub>3</sub>O<sub>4</sub> PC). While it decreases to 55.04 °C (Fe<sub>2</sub>O<sub>3</sub> PC) and 58.37 °C (MnO<sub>2</sub> PC). These unique results exhibit that the optical, electrical and mechanical properties of polymeric blends could be controlled via metal oxides incorporation for applications in flexible optoelectronic devices.

**Keywords** Metal oxides incorporation · PVA/reduced graphene oxide · Optical · Electrical · Mechanical · Flexible optoelectronic devices

## 1 Introduction

In the last two decades, a researches trend has been focusing on polymer composites (PCs) due to their possible participations in many daily fields [1–4]. This scientific concern of PCs arises owing to their unique characteristics including lightweight, flexibility, availability and relatively low cost [5–9]. In addition, polymers' features qualify them to act the hosts' role for various kinds of fillers. PCs could be made to meet such an application in optoelectronics, shielding, optical devices, photocatalysts, sensors, solar cells, supercapacitors and medicine [4, 10–13]. Specifically, PCs based on polyvinyl alcohol (PVA) as a host matrix are given superior

attention due to PVA's unique physical and chemical characteristics. These characteristics include its high optical transmittance in the visible–NIR region, wide bandgap (~5.3 eV [14]) and semi crystallinity nature. The last feature arises due to the existence of the hydroxyl (OH) group in its chains which qualify it as an ideal host for a lot of fillers [5, 15, 16]. Besides, its biodegradable, water-soluble and friendly environmental features introduce it for a wide range of applications [14, 17, 18]. Moreover, the ability of blending PVA with such an amorphous polymer or a crystalline material is easy to be performed for multiple industrial applications. Carbon derivatives are promising over a lot of material to be blended with the PVA polymer to form a new kind of host materials. Reduced graphene oxide (rGO) is one of these carbon derivatives that is favorable because of its novel mechanical and electrical properties. Moreover, its excessive hardness and lightweight provide the principal host (PVA) more preferable features to the produced blend [19–24]. It is worth mentioning that rGO is optimized over other carbon derivatives to form polymeric blends since its

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high electrical conductivity, virtuous ability to materials interaction and high mobility [25, 26].

A lot of reported works in the literature concerned the PVA modification and its blends. Aslam group explored the effect of graphene oxide on the structural and opto-mechanical performance of PVA for opto-electronic devices [27]. They found that Young's modulus and tensile strength of the prepared blend were entirely enhanced. Mahendia et al. used rGO to enhance the thermal performance as well as the glass transition temperature of PVA [28]. The optical bandgap of PVA was tuned using multi-walled CNTs by Zidan researches group [29]. Furthermore, the electrical conductivity of PVA was greatly improved by blending it with GO for electronic applications [20]. In addition, the mechanical properties of PVA were reinforced by graphene for antibacterial applications [30]. Besides, a lot of previous works explored the effect of the metal oxides weight ratio on the different functional properties of PVA in the literature [31–33]. The optical and structural performance of PVA was tailored by blending with different weight ratios of iron oxide [31]. Khairy research group controlled the optical characteristics of PVA by ZrO<sub>2</sub> incorporation for UV-block applications [33]. Alrowialy et al. concluded that the optical parameter of PVA could be enhanced significantly by Y<sub>2</sub>O<sub>3</sub> embedding [32]. In our previous work, we found that PVA blended with graphene is an ideal host for FeS nanoparticles for eco-environmentally applications [13].

The current work aims to explore the effect of metal oxides' type (M: Fe, Pb and Mn oxides) on the optical, electrical and mechanical performance of PVA/rGO blend. To the best of our knowledge, such a comparative study on the functional properties of PVA/rGO PCs has not been reported before. Metal oxides are selected to play this role, since their semiconducting feature allows to tailor the optical, mechanical and electrical properties of PVA/rGO blend in a wide range for various applications. Moreover, their unique properties as availability and low cost are also welcomed. In this work, factors such as concentration, thickness, preparation conditions, etc. that may affect the functional performance of the host PVA/rGO blend are fixed, while the effect of the filler's type (metal oxides) is explored. The bonds' vibrations, optical, electrical and mechanical performance of the metal

oxides incorporated PVA/rGO blend are obtained from the Fourier transform infrared (FT-IR), UV–Vis–NIR, four-probe method and dynamic mechanical analyzer (DMA) measurements, respectively. In addition, the surface morphology of the PCs were analyzed based on scanning electron microscope examinations.

## 2 Methods and materials

Many raw materials are required to prepare the plain PVA and metal oxides incorporated PVA/rGO PCs, as listed in Table 1. No further purification was performed on the purchased materials. The solution casting procedure described by Heiba et al. was followed to prepare the plain and incorporated PCs [34]. First, 10 g of PVA granules were dissolved in 250 ml of double-distilled water at 70 °C with continuous stirring for 3 h to obtain a clear solution. Second, 50 ml of the previous solution was taken off to prepare the plain PVA sample. Third, 0.1 wt% of rGO/PVA blend was prepared by adding the required weight of rGO nanosheets into the previous PVA solution. With a magnetic stirring for 24 h at room temperature, a homogenous blend of PVA/rGO was achieved. Fourth, another 50 ml of the blend were taken off to prepare the plain PVA/rGO sample. Fifth, the remaining PVA/rGO blend was divided into three parts to obtain the metal oxide-incorporated PVA/rGO PCs by adding 2.0 wt% of the metal oxide into the previous PVA/rGO blend with stirring for 3 h to obtain the homogenous PC. Sixth, the previous five solutions were poured in glassy Petri dishes and put in an oven at 55 °C for 48 h. After that, the samples were left to reach the room temperature. Five films with three specimens of each with thicknesses of 0.20 mm ± 0.01 mm were obtained and marked (S<sub>1</sub> to S<sub>5</sub>) to perform the examinations.

The surfaces' morphology of the unincorporated and metal oxides incorporated PVA/rGO blend was captured using a scanning electron microscope (JOEL, Model: 3600 LA). The structure's variation of the samples was examined via FT-IR measurements using a Shimadzu (IRAffinity-1S) spectrophotometer over wavenumbers ranging from 400 to 4000 cm<sup>-1</sup>. The optical properties were obtained from the analysis of the

**Table 1** Materials used in the current study

Material	Conditions	Supplier
Ferric (III) oxide (Fe <sub>2</sub> O <sub>3</sub> )	Purity ≥ 96%	Sigma-Aldrich Co
Lead (II,IV) oxide (Pb <sub>3</sub> O <sub>4</sub> )	Purity ≥ 99%	Sigma-Aldrich Co
Manganese oxide (MnO <sub>2</sub> )	Purity ≥ 99%	Sigma-Aldrich Co
reduced graphene oxide (rGO) nano sheets	Thickness:0.55–3.74 nm, Purity ≥ 98%	Chengdu organic chemicals
PVA	(86–89% hydrolyzed, M.W.: 850,000 g.mol <sup>-1</sup> )	Alfa Aesar

UV–visible–NIR absorbance ( $A$ ) and transmittance ( $T$ ) spectra measurements measured by a JASCO (V-670) spectrophotometer with three-time rounds to confirm the reproducibility of the obtained data. The main linear optical parameters: optical absorption coefficient ( $\alpha$ ), extinction coefficient ( $K$ ), reflectance ( $\mathcal{R}$ ), energy bandgap ( $E_g$ ), Urbach energy ( $E_u$ ), refractive index ( $n$ ) and real ( $\epsilon_r$ ) and imaginary ( $\epsilon_i$ ) dielectric constants of the samples were determined using the following equations [35–44]:

$$\alpha = \frac{2.303A}{d} \tag{1}$$

$$\alpha h\nu = B(h\nu - E_g)^m \tag{2}$$

$$\alpha = \alpha_0 \exp(h\nu/E_u) \tag{3}$$

$$n = \left(\frac{1 + \mathcal{R}}{1 - \mathcal{R}}\right) + \left[\frac{4\mathcal{R}}{(1 - \mathcal{R})^2} - K^2\right]^{1/2} \tag{4}$$

$$K = \frac{\alpha\lambda}{4\pi} \tag{5}$$

$$\mathcal{R} = 1 - \sqrt{T \times e^A} \tag{6}$$

$$\epsilon_r = n^2 - K^2 \tag{7}$$

$$\epsilon_i = 2nK, \tag{8}$$

where  $d$  is the sample’s thickness,  $B$  is a constant,  $\alpha_0$  is a constant and  $h\nu$  is photons energy. While  $m$  parameter relates to the electronic transition type (direct ( $m=0.5$ ) and indirect ( $m=2$ )).

Besides, the Wemple–DiDomenico (W–D) and Sellmeier oscillator models were used to examine the refractive index at infinity ( $n_\infty$ ), energy dispersion parameters ( $E_0$  and  $E_d$ ), average inter-band oscillator wavelength ( $\lambda_0$ ), average oscillator strength ( $S_0$ ), infinite dielectric constant ( $\epsilon_\infty$ ), lattice dielectric constant ( $\epsilon_L$ ), free carrier concentration to the effective mass ratio ( $N/m^*$ ) and the plasma frequency ( $\omega_p$ ) from [9, 45]

$$n^2 = 1 + \frac{E_d E_0}{E_0^2 - (h\nu)^2} \tag{9}$$

$$\frac{n_\infty^2 - 1}{n^2 - 1} = 1 - \left(\frac{\lambda_0}{\lambda}\right)^2 \tag{10}$$

$$(n^2 - 1)^{-1} = \frac{1 - \left(\frac{\lambda_0}{\lambda}\right)^2}{S_0 \lambda_0^2} \tag{11}$$

$$S_0 = \frac{n_\infty^2 - 1}{\lambda_0^2} \tag{12}$$

$$\epsilon_\infty = n_\infty^2 \tag{13}$$

$$\epsilon_r = n^2 = \epsilon_L - \frac{e^2}{4\pi^2 C^2 \epsilon_0} \frac{N}{m^*} \lambda^2 \tag{14}$$

$$\omega_p = \left(\frac{e^2 N}{\pi C^2 m^*}\right)^{1/2}, \tag{15}$$

where  $\lambda$ ,  $C$ ,  $e$  and  $\epsilon_0$  are incident wavelength, speed of light, free-electron charge and space dielectric constant, respectively.

The dc electrical resistance ( $R$ ) performance was measured by a four-probe stage under temperature ( $T$ ) sweeping rate of 1 K/min from 300 to 400 K via an Oxford Optistat cryostat. Keithley sourcemeter (model: 6517B) was used to record the current–voltage curves. All electrical resistance measurements were performed three-time rounds. The activation energy ( $E_a$ ) of the prepared PCs was deduced using the Arrhenius relation given as [5, 46]

$$\sigma_{dc} = \sigma_0 \exp(E_a/(K_B T)), \tag{16}$$

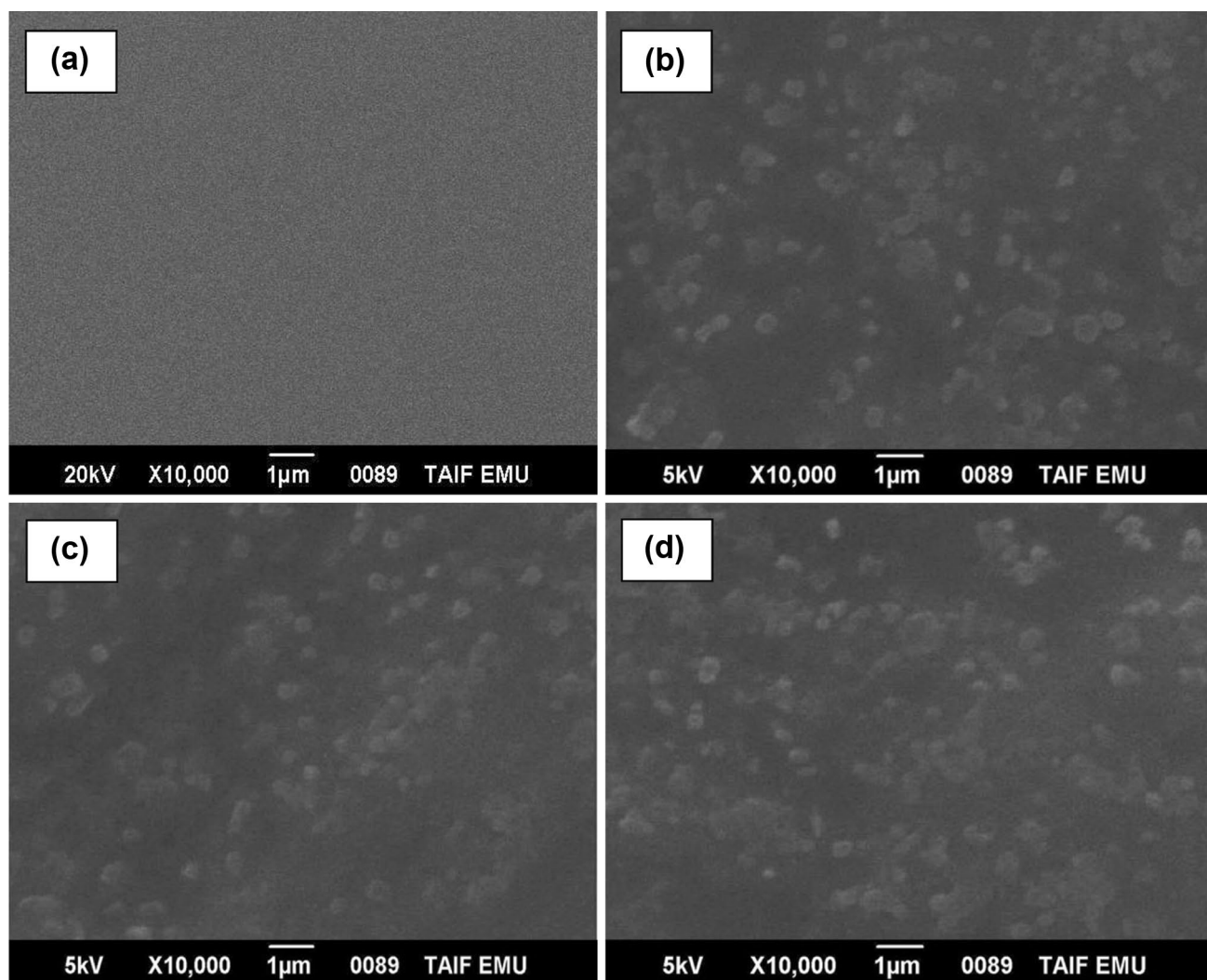
where  $K_B$  and  $\sigma_0$  are the Boltzman constant and pre-exponential factor, respectively.

The dynamic mechanical properties: storage modulus ( $E'$ ), loss modulus ( $E''$ ), stiffness and loss factor ( $\text{Tan}\delta = \frac{E''}{E'}$ ) ([47]) are measured using a dynamic mechanical analyzer (DMA, Q800). A static force of 0.01 N, strain amplitude of 15  $\mu\text{m}$  and frequency of 1 Hz was applied through temperature sweeping from 30 to 120  $^\circ\text{C}$  and a rate of 3  $^\circ\text{C}/\text{min}$ . Three-time rounds were performed to ensure the reproducibility of the obtained mechanical measurements.

### 3 Results and discussion

#### 3.1 Surface morphology analysis

Figure 1a–d shows the captured surfaces’ morphology of the unincorporated and metal oxides incorporated PVA/rGO PCs surfaces using a scanning electron microscope (SEM). First, SEM micrographs confirm that all PCs are pinhole-free. In addition, SEM micrograph of the unincorporated PVA/rGO blend (Fig. 1a) reveals its smoothness without



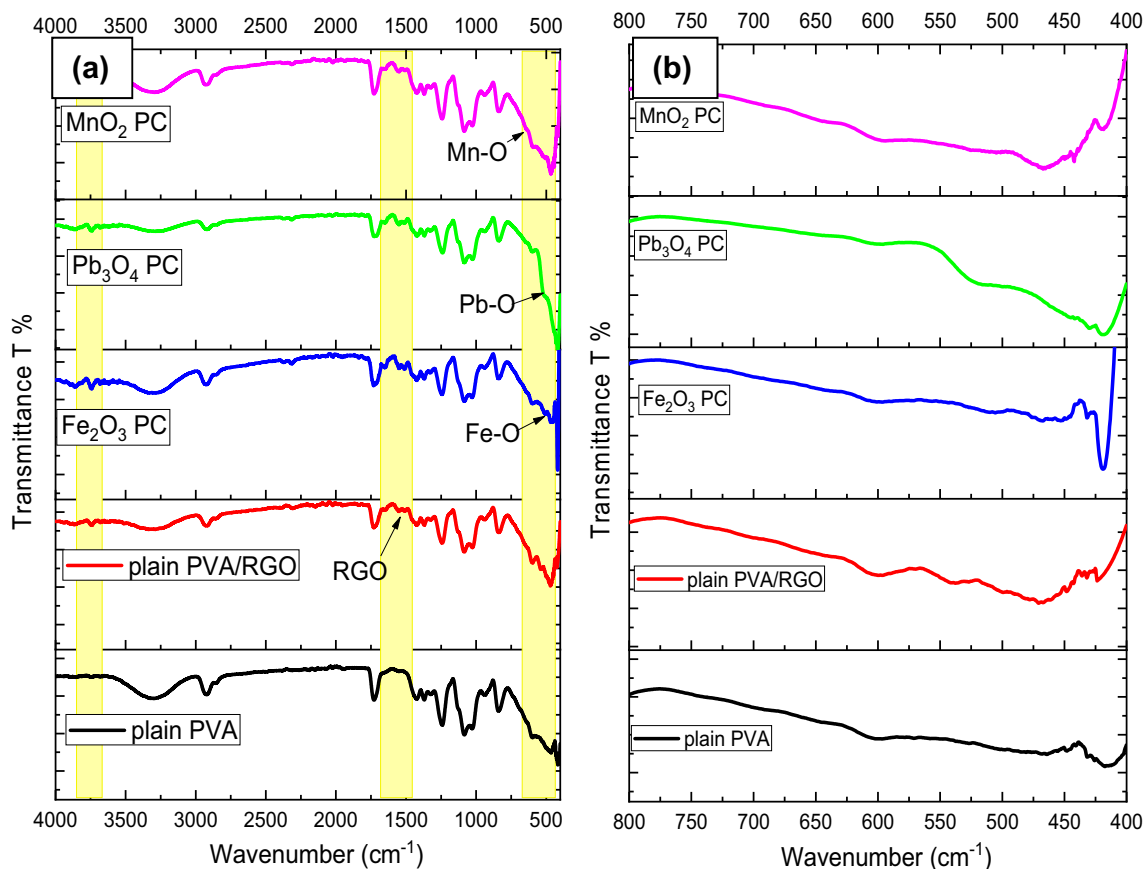
**Fig. 1** SEM micrographs of **a** unincorporated and metal (M: Fe, Pb, Mn; **b–d**) oxides incorporated PCs, respectively

any impurities. While the SEM micrographs of the metal oxides incorporated PVA/rGO PCs (Fig. 1b–d) reveal the existence of well-distributed bright spots pinned in the host matrix. In addition, the SEM micrographs of metal oxides incorporated PCs seem more roughness as compared with the unincorporated blend.

### 3.2 FT-IR analysis

The variations in the PVA and PVA/rGO blend structures due to the metal oxides incorporation have been examined using FT-IR spectroscopy. Figure 2a displays the FT-IR transmittance spectra of the plain PVA, metal (M: Fe, Pb, Mn) oxides incorporated and unincorporated PVA/rGO blend in the wavenumber range from 400 to 4000  $\text{cm}^{-1}$ . Based on the FT-IR spectrum of the plain PVA, the locations of the observed absorption bands and

their corresponding bonds' vibration are listed in Table 2. As compared with the plain PVA FT-IR spectrum, two additional small absorption peaks located at 1636  $\text{cm}^{-1}$  and 1542  $\text{cm}^{-1}$  are detected in the FT-IR spectra of the incorporated and unincorporated PCs. These absorption peaks result due to the rGO blending and correspond to the vibrations of O–H bending and C=C, respectively [8, 27]. In addition, a tiny absorption peak centered at 3740  $\text{cm}^{-1}$  relates to the stretching of the hydroxyl group (OH) on adsorbed on the rGO sheets [48]. While the metal oxides incorporation effect on the structure of PVA/rGO blend is clearly noticed in the wavenumber range from 650 to 400  $\text{cm}^{-1}$  as depicted in Fig. 2b. These additional absorption peaks as well as the variation in the absorption intensities are attributed to the M–O (M: Fe, Pb, Mn) bonds torsional and stretching vibrations [49–51]. Moreover, slight shifts in peaks' location to lower wavenumbers of the host



**Fig. 2** FT-IR transmittance spectra of plain PVA and metal (M: Fe, Pb, Mn) oxides PCs vs. wavenumbers range from **a** 4000 to 400  $\text{cm}^{-1}$  and **b** 800 to 400  $\text{cm}^{-1}$

**Table 2** Absorption band locations and their corresponding bonds' vibrations of plain PVA and metal (M: Fe, Pb, Mn) oxides incorporated and unincorporated PVA/rGO blends

Absorption band location ( $\text{cm}^{-1}$ )	Corresponding bond's vibration	References
Broad 3065—3556	Stretching of OH	[7, 55]
2925	Asymmetric stretching of CH	[29, 56]
1726	Stretching of C=O	[57, 58]
1423	Symmetric bending of $\text{CH}_2$	[29, 59]
1370	Wagging of C-H	[59]
1242	Stretching of C-C	[59]
1088	Stretching of C-O	[29]
1015	Bending of O-H	[60]
930	Rocking of $\text{CH}_2$	[29]
840	Stretching of C-C	[61]
629	Stretching of C-H	[56]

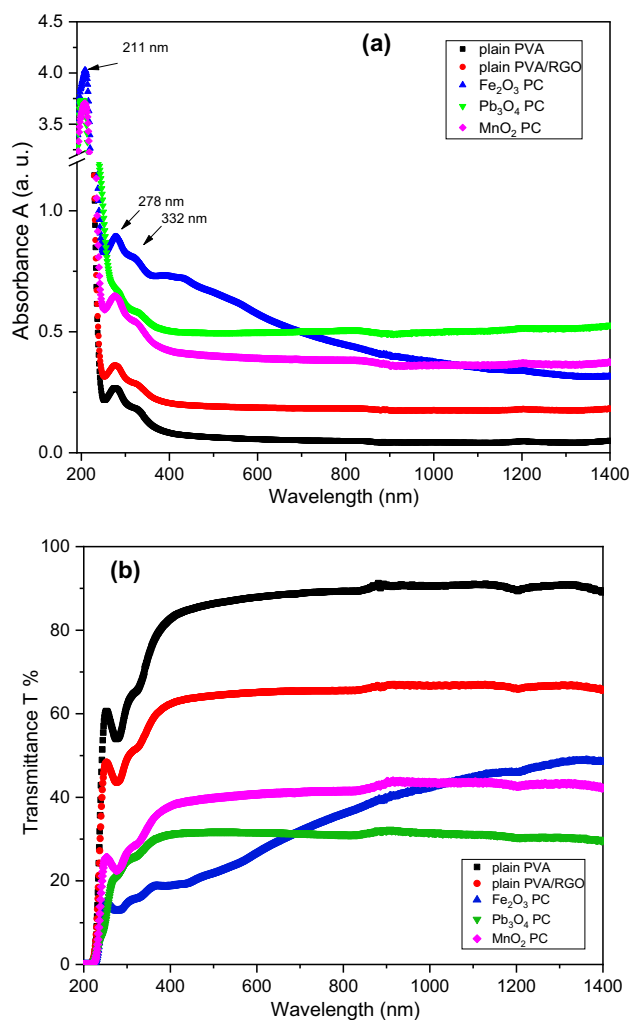
blend are detected which emphasize the positive interaction between the filler (metal oxide) and the host blend. These interactions in the blend' structure lead to clear modifications in the optical, mechanical and electrical

performance of the PVA/rGO blend as discussed below. Similar findings were reported in the literature [52–54].

### 3.3 UV-visible-NIR analysis

Exploring the optical performance of the prepared films is significant to determine their suitability in such an optoelectronic application. The absorbance ( $A$ ) and transmittance ( $T$ ) spectra of the plain PVA and metal (M: Fe, Pb, Mn) oxides PCs in the UV-Vis-NIR regions are illustrated in Fig. 3a, b, respectively. All absorption spectra of the prepared films exhibit two absorption peaks at 211 nm and 278 nm that resemble the  $\pi \rightarrow \pi^*$  inter-band electronic transitions of PVA [62, 63]. In addition, an absorption edge at 332 nm is also observed which corresponds to the  $n \rightarrow \pi^*$  electronic transition of PVA [64]. As compared with the plain PVA spectrum, no additional peaks are noted in the metal oxides PCs. In addition, the absorption amount increases and hence the transmittance decreases due to the incorporation process. Moreover, the  $\text{Fe}_2\text{O}_3$  PC film exhibits the largest absorption in the UV-Vis region, while the  $\text{MnO}_2$  one exhibits the smallest one in the indicated region. The vice versa

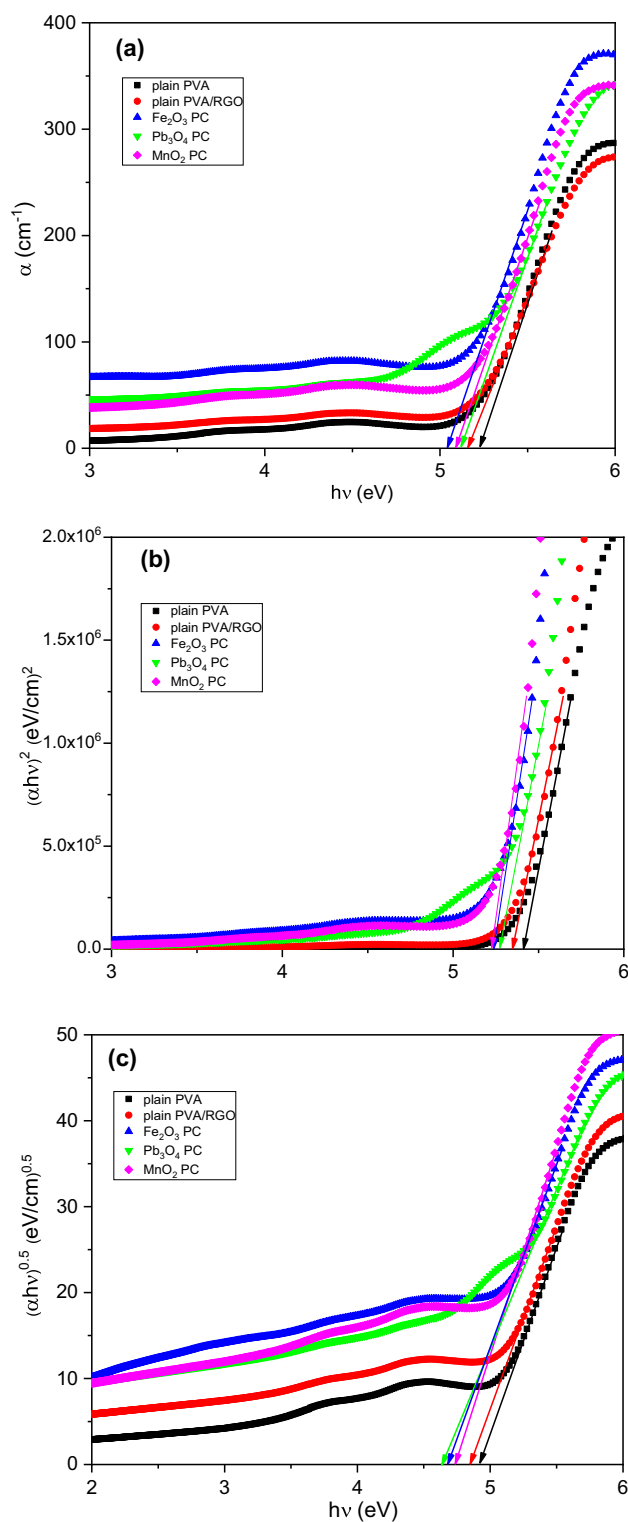




**Fig. 3** Absorbance and transmittance of plain PVA and metal (M: Fe, Pb, Mn) oxides PCs vs. wavelength

behaviour is noticed in the transmittance spectra of the samples. For example, at  $\lambda = 600$  nm,  $T$  of the plain PVA is 88% and decreases to 65% (plain PVA/rGO), 41% ( $\text{MnO}_2$  PC), 32% ( $\text{Pb}_3\text{O}_4$  PCA) and 27% ( $\text{Fe}_2\text{O}_3$  PC). This variation in the absorption and transmittance spectra is attributed to the variations in the optical bandgap of prepared films. This differentiation in the optical absorption and hence the transmittance due to varying the filler's type gives additional opportunities for new optical and optoelectronic applications.

The effect of the metal oxides incorporation on the optical absorption coefficient ( $\alpha$ ) and energy bandgap ( $E_g$ ) of the PVA/rGO blend has been explored. Figure 4a presents  $\alpha$  plots of plain PVA and metal (M: Fe, Pb, Mn) oxides PCs as a function of incident photons energy ( $h\nu$ ). The obtained energy bandgap from the absorbance measurements ( $E_{g \text{ Abs.}}$ ) of the prepared PCs are listed in Table 3. According to Fig. 4, all films absorb significantly the UV spectrum, while their absorption decrease in both the visible-NIR spectra. In



**Fig. 4** a Absorption coefficient, b direct and c indirect Tauc's plot of plain PVA and metal PCs vs.  $h\nu$

addition, red-shifts of the absorption edge of the PCs with respect to the plain one. The obtained  $E_{g \text{ Abs.}}$  value of plain PVA is 5.23 eV which is consistent with reported values [32,

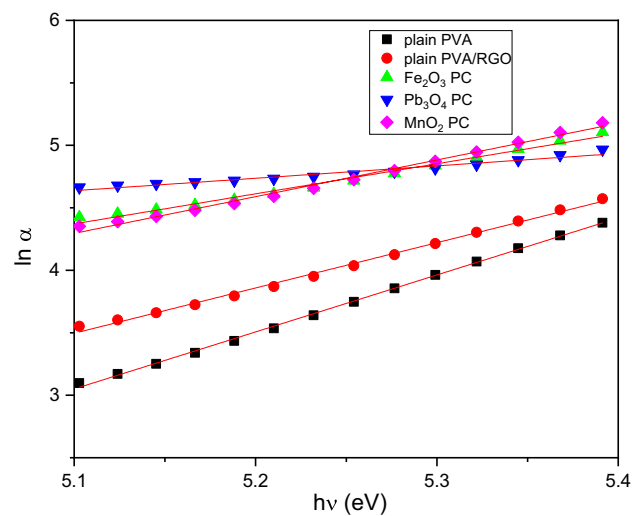
**Table 3**  $E_g$  (in eV) and  $E_u$  values of metal oxides PCs

Sample	$E_{g\text{ Abs}}$	$E_{g\text{ d}}$	$E_{g\text{ ind}}$	$E_{g\text{ ei}}$	$E_u$ (eV)
Plain PVA PVP/PVA	5.23	5.41	4.92	5.42	0.21
Plain PVA/rGO	5.16	5.36	4.85	5.35	0.28
Fe <sub>2</sub> O <sub>3</sub> PC	5.04	5.23	4.68	5.23	0.41
Pb <sub>3</sub> O <sub>4</sub> PC	5.13	5.27	4.64	5.31	0.64
MnO <sub>2</sub> PC	5.09	5.22	4.73	5.27	0.34

65]. Moreover, Fe<sub>2</sub>O<sub>3</sub> PC possesses the lowest  $E_{g\text{ Abs}}$  value (5.04 eV), while Pb<sub>3</sub>O<sub>4</sub> PC has the highest one (5.13 eV). The variation in the  $E_{g\text{ Abs}}$  values is attributed to defects' creations as a result of the incorporation process.

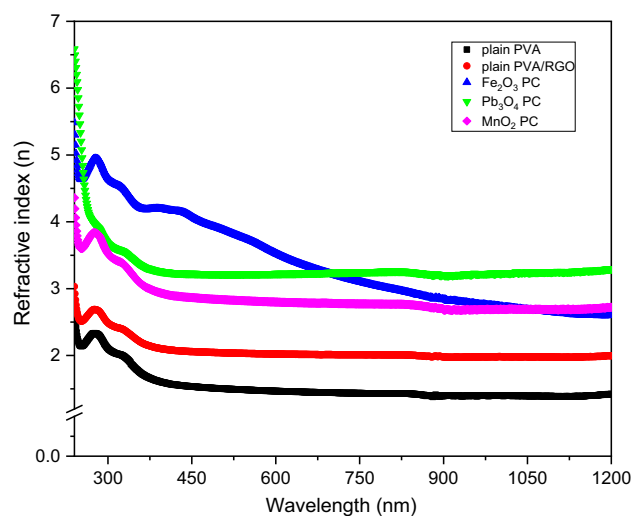
Furthermore, Tauc's relation (Eq. 2) is used to determine the direct ( $m = 1/2$ ) and indirect ( $m = 2$ ) optical bandgap of the prepared films. To perform that,  $(ah\nu)^2$  and  $(ah\nu)^{0.5}$  values of the plain PVA and incorporated and unincorporated PCs as a function of  $h\nu$  are plotted, as depicted in Fig. 4b, c, respectively. The obtained  $E_g$  values are tabulated in Table 3. It is noticed that the  $E_g$  value of the plain PVA decreases from 5.41 eV (direct) due to 0.1 wt% incorporation of rGO to 5.36 eV (direct), which also decreases as a result of metal oxides incorporation to 5.23 eV (Fe<sub>2</sub>O<sub>3</sub> PC), 5.27 eV (Pb<sub>3</sub>O<sub>4</sub> PC) and 5.22 eV (MnO<sub>2</sub> PC). In other words, controlling the optical bandgap of the host matrix could be achieved via selecting the filler's type. The decrease in  $E_g$  value is mainly referred to the formed localized energy states and defects between the occupied and unoccupied molecular orbitals of the host matrix (HOMO and LUMO) as confirmed below by Urbach energy investigations [13, 17, 32]. The obtained  $E_g$  values by Tauc's method agree with those achieved by the absorption measurements. This novel conclusion is highly appreciated in a wide spectrum of optoelectronic applications. Our optical bandgap results are in accordance with the reported data [17, 34].

Investigating the formed defects in the host matrix could be achieved by exploring the Urbach energy ( $E_u$ ) of the prepared plain and incorporated PCs [29, 45, 66]. The defects formed due to doping increase the amorphous character of the prepared PCs as compared with the plain one. The  $E_u$  value represents the tails' width of created energy states in the bandgap of the PCs [67, 68]. Equation 3 presents the exponential behaviour of the formed tails in the host blend. Figure 5 shows plots of  $\ln(\alpha)$  of the plain PVA and metal oxides PCs as a function of  $h\nu$ . The  $E_u$  values are obtained from the slopes' inverse of the fitted lines and included in Table 3.  $E_u$  value of the plain PVA is 0.21 eV, which agrees with the reported one [64, 68]. For instance, Abdullah et al. concluded that  $E_u$  of PVA increased from 0.26 to 1.28 eV due to 4 wt% of KMnO<sub>4</sub> incorporation [64]. They attributed the increment of  $E_u$  value to the created localized states in the energy bandgap of the host PVA. In addition,

**Fig. 5**  $\ln(\alpha)$  curves vs.  $h\nu$  of plain PVA and metal oxides PCs

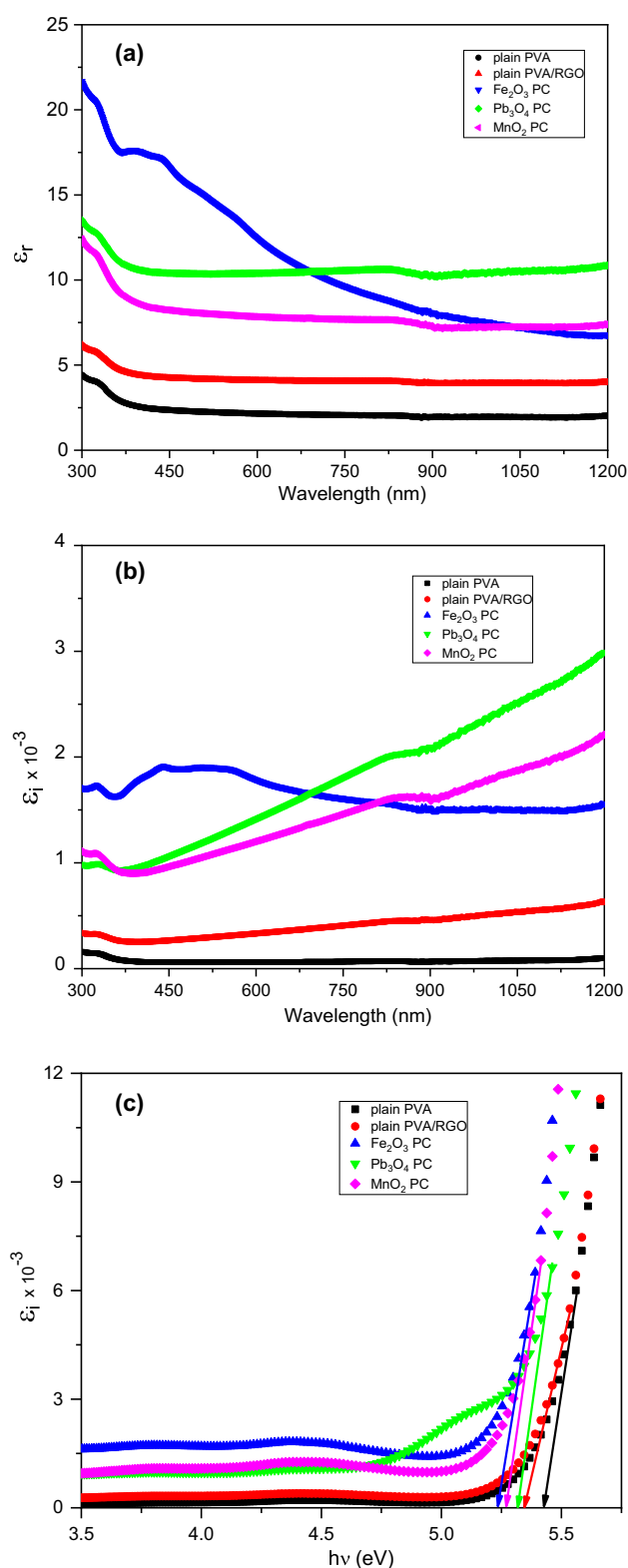
it is evident that  $E_u$  value increases to 0.28 eV (plain PVA/rGO) and more increases due to metal oxides incorporation. The  $E_u$  increment exhibits the increase in defects and voids and hence tunes the optical performance as presented above relative to the bandgap. In addition, it is clear that created defects are significantly altered as a result of the filler's type.

Moreover, the refractive index ( $n$ ) is another important optical property that must be investigated for the prepared PCs to recommend their possible applications. Equation 4 shown above is used to calculate the  $n$  values of the plain PVA and metal oxides PCs. Figure 6 illustrates  $n$  plots of the plain and incorporated PCs as a function of the scanned wavelengths. It is noticed that  $n$  of the plain PVA, plain PVA/rGO and both Pb<sub>3</sub>O<sub>4</sub> and MnO<sub>2</sub> incorporated PVA/rGO blends behaves quasi-steady in the Vis–NIR

**Fig. 6** Refractive index of plain PVA and metal oxides PCs

region, while  $n$  of  $\text{Fe}_2\text{O}_3$  incorporated PVA/rGO blend decreases as  $\lambda$  is increased in the Vis–NIR region. In addition, at wavelength  $\lambda$ ,  $n$  of the incorporated PCs is larger than that of the plain blend, which is larger than that of the plain PVA. These results can be interpreted in terms of the absorbance and reflectance responses caused by the filler's molecules in the host matrix [32, 69]. The metal oxides molecules work as reflecting centres as well as increase the whole density of the host matrix [70]. In addition, it is clear that the filler's quality plays a great role in controlling the refractive index of the host matrix. For example, at  $\lambda = 600$  nm,  $n$  value increases from 1.45 (plain PVA) to 2.02 (plain PVA/rGO) and then to 2.79 ( $\text{MnO}_2$  PC), 3.22 ( $\text{Pb}_3\text{O}_4$  PC) and 3.51 ( $\text{Fe}_2\text{O}_3$  PC). In other words,  $n$  of such a host matrix could be controlled via selecting the appropriate filler to match such applications.

The dielectric constants (real  $\epsilon_r$  and imaginary  $\epsilon_i$  parts) of the prepared PCs are determined as a function of  $h\nu$  to endorse their probable uses. The real part of the dielectric constant presents information about the performance of the electromagnetic wave propagation through the medium and their energy-storing ability. While the imaginary part of the dielectric constant reflects the energy-loss part of the propagated electromagnetic waves [4, 18]. The calculated dielectric constants ( $\epsilon_r$  and  $\epsilon_i$ ) using Eq.'s 7 and 8 are shown in Fig. 7a, b, respectively. It is evident that  $\epsilon_r$  of plain and metal oxides PCs takes the trend of the refractive index  $n$  behaviour. This logical result is accepted in terms of the absorbance performance of the prepared samples. The real dielectric constant of plain PVA and  $\text{Pb}_3\text{O}_4$  and  $\text{MnO}_2$  incorporated PVA/rGO blends behaves quasi-steady in the Vis–NIR region, while  $\epsilon_r$  of  $\text{Fe}_2\text{O}_3$  incorporated PVA/rGO blend decreases as  $\lambda$  is increased in the Vis–NIR region. In addition, at any  $\lambda$ ,  $\epsilon_r$  values of the incorporated PCs are larger than that of the plain one. The increment in  $\epsilon_r$  value is attributed to the energy density states of the incorporated PCs as compared to the plain ones. The enhancement in  $\epsilon_r$  value of the PVA/rGO blend as well as tuning its value is highly appreciated in a lot of applications as those related to ceramics and energy storage ones. While the increase in the imaginary part of the dielectric constant as a result of incorporation is attributed to the dipole motions in the PCs [71]. The same behaviours were reported in the literature [2, 71, 72]. In instance, Soliman et al. found that the dielectric constant of PVA increased with increasing the incorporated material ( $\text{BaTiO}_3$ ) [72]. They attributed the increments in the dielectric constants to the increase in the defects' number and the disorder nature of the PCs. Furthermore, the real energy bandgap ( $E_{g\epsilon_i}$ ) of the plain PVA and metal oxides incorporated and unincorporated PVA/rGO blend could be deduced from the imaginary dielectric constant plots as illustrated in Fig. 7c. The obtained real bandgap values are also tabulated in Table 3. It is clear that the real energy ( $E_{g\epsilon_i}$ )



**Fig. 7** Dielectric constants **a** real, **b** imaginary parts vs. wavelength and **c** imaginary parts vs.  $h\nu$  of the plain PVA and metal oxides PCs



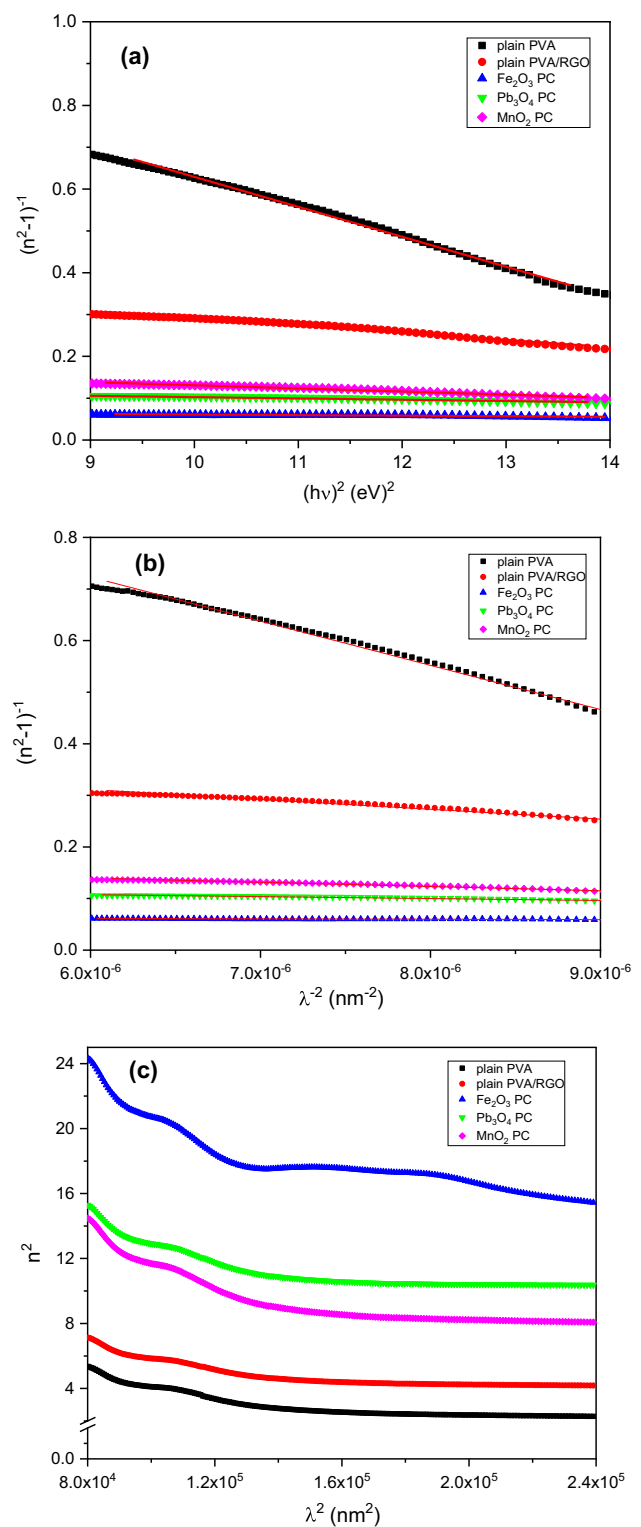
are well-matched their identical values deduced from the Tauc’s relation of the direct electronic transitions [31].

Moreover, Wemple–DiDomenico (W–D) model is used to explore the effect of the filler’s type on the dispersive parameters of the host blend [70]. Both the dispersive ( $E_d$ ) and oscillator ( $E_0$ ) energies could be investigated in the non-absorption region. The first one ( $E_d$ ) reveals the transition strength, while the second one ( $E_0$ ) indicates the average bandgap of the PCs [73]. Using W–D model (Eq. 9), curves of  $1/(n^2 - 1)$  of the samples as a function of  $(h\nu)^2$  are plotted (Fig. 8a), where both  $E_d$  and  $E_0$  are deduced and listed in Table 4. According to the obtained  $E_d$  and  $E_0$  data, a pronounced effect of the filler’s type on the dispersive energies is noticed. First, the obtained  $E_d$  and  $E_0$  of the plain PVA is well-compatible with reported [4, 7]. In addition,  $E_d$  values of the PCs are larger than that of the unincorporated blend. This result reveals that the PCs possess more inter-band transitions due to the increasing disorder character [74]. Whereas, the decrease in  $E_0$  value of the incorporated PCs relative to the plain one is accepted based on the decrease of the optical bandgap values due to the increase in the defects density [75]. Moreover, the obtained dispersive energy findings are well-agreed with those achieved by Tauc’s method as well as the reported ones [70, 75].

Besides, many other optical parameters can be determined based on the Sellmeier oscillator model and the related equations shown above (Eqs. 10–14). By plotting  $1/(n^2 - 1)$  vs.  $\lambda^{-2}$  of the prepared samples as illustrated in Fig. 8b,  $n_\infty$ ,  $\lambda_0$ ,  $S_0$  and  $\epsilon_\infty$  are estimated and also tabulated (Table 4). Furthermore, from the plots of  $n^2$  vs.  $\lambda^2$  (Fig. 8c), the values of  $\epsilon_L$  and  $N/m^*$  are obtained (Table 3). While the plasma frequency ( $\omega_p$ ) is calculated using Eq. 15. Noticeably, the effect of the metal oxides’ incorporation on the whole optical parameters as well as its type. For example, the value of the infinite dielectric constant ( $\epsilon_\infty$ ) of the metal oxides PCs is enhanced more than five times as compared with that of the plain PVA. This finding is welcomed in energy storage and supercapacitors applications. Our findings agree with the reported ones [29, 76, 77]. Our unique results introduce a valuable comparative evaluation for the optical parameters to dictate the suitable applications.

### 3.4 Electrical properties analysis

The dc-electrical properties performance of the plain and metal oxides PCs has been investigated to recommend their potential applications. The dc-electrical resistivity ( $\rho$ ) of the samples were measured vs. temperature (300–400 K) as depicted in Fig. 9a. An obvious effect of the metal oxides type incorporation is perceived. First,  $\rho$  performance of the unincorporated PVA film agrees with the literature [5, 6]. Second, rGO blending with PVA causes a decrease in the electrical resistivity and hence an increase in the

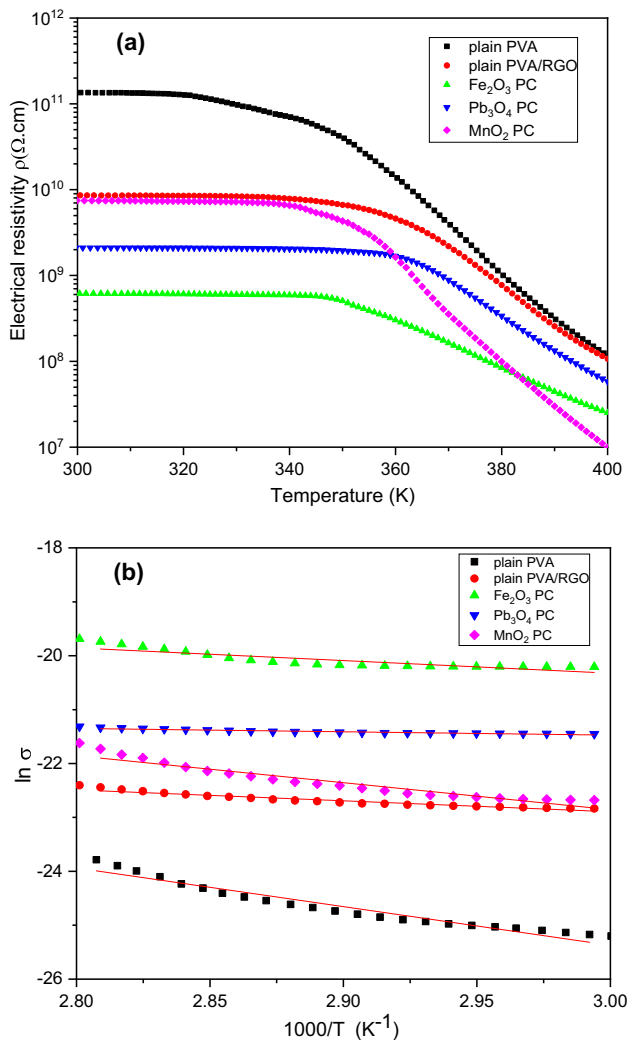


**Fig. 8** Plots of **a**  $1/(n^2 - 1)$  vs.  $(h\nu)^2$ , **b**  $(n^2 - 1)^{-1}$  vs.  $\lambda^{-2}$  and **c**  $n^2$  vs.  $\lambda^2$  of plain and metal oxides PCs

conductivity  $\sigma_{dc}$  ( $= \frac{1}{\rho}$ ) of the plain PVA. Similarly, the metal oxides incorporation causes an increase in the electrical

**Table 4** Optical parameters of plain PVA and metal oxides PCs

Sample	$E_d$ (eV)	$E_0$ (eV)	$n_\infty$	$\lambda_0$ (nm)	$S_0$ ( $\times 10^{13} \text{m}^{-2}$ )	$\varepsilon_\infty$	$\varepsilon_L$	$(\text{N/m}^2) \times 10^{57}$ ( $\text{kg}^{-1} \text{m}^{-3}$ )	$\omega_p \times 10^{15}$ (Hz)
plain PVA	5.17	5.22	1.35	262.8	1.18	1.81	2.71	0.98	1.02
plain PVA/rGO	11.31	5.20	1.78	333.3	2.01	3.22	4.48	1.75	1.06
$\text{Fe}_2\text{O}_3$ PC	51.03	4.89	2.77	186.1	19.2	7.67	19.49	1.50	1.49
$\text{Pb}_3\text{O}_4$ PC	32.27	5.16	2.95	175.4	25.1	8.69	10.56	9.29	1.60
$\text{MnO}_2$ PC	24.38	5.12	2.85	181.7	22.2	8.14	8.83	4.95	1.27

**Fig. 9** **a** DC-electrical resistivity vs. temperature and **b**  $\ln \sigma_{dc}$  vs.  $1000/T$  of the metal oxides PCs

conductivity. This result is interpreted to the increase of the charges' carriers due to the metal oxides incorporation [78, 79]. While the decrease in  $\rho$  values as the temperature is increased and hence  $\sigma_{dc}$  increase of the PCs refers to the charge carriers' transferring or/and hopping within the PCs. Correspondingly, heating the PCs causes an enhancement in

**Table 5**  $E_a$  and  $\sigma$  at 300 K of plain PVA and metal oxides incorporated and unincorporated PCs

Sample	$E_a$ (eV)	$\sigma$ at 300 K ( $\text{S.cm}^{-1}$ )
plain PVA	0.66	$7.39 \times 10^{-12}$
plain PVA/rGO	0.18	$1.16 \times 10^{-10}$
$\text{Fe}_2\text{O}_3$ PC	0.05	$1.62 \times 10^{-9}$
$\text{Pb}_3\text{O}_4$ PC	0.20	$4.74 \times 10^{-10}$
$\text{MnO}_2$ PC	0.43	$1.34 \times 10^{-10}$

the polymer's chains mobility, especially with the existence of additional created defects due to the metal oxides incorporation. Moreover, the  $\sigma_{dc}$  performance of the prepared PCs depends on the filler's type (Table 4). For example, at 300 K,  $\sigma_{dc}$  value increases from  $7.39 \times 10^{-12} \text{S cm}^{-1}$  (plain PVA) to  $1.16 \times 10^{-10} \text{S cm}^{-1}$  (plain PVA/rGO). While it is enhanced to  $1.62 \times 10^{-9} \text{S cm}^{-1}$  ( $\text{Fe}_2\text{O}_3$  PC) and  $4.74 \times 10^{-10} \text{S cm}^{-1}$  ( $\text{Pb}_3\text{O}_4$  PC) and  $1.34 \times 10^{-10} \text{S.cm}^{-1}$  ( $\text{MnO}_2$  PC). This wide variation in  $\sigma_{dc}$  values presents metal oxides PCs as competitive materials for applications in optoelectronic and elastic devices as well as electrolytes issues [71, 80].

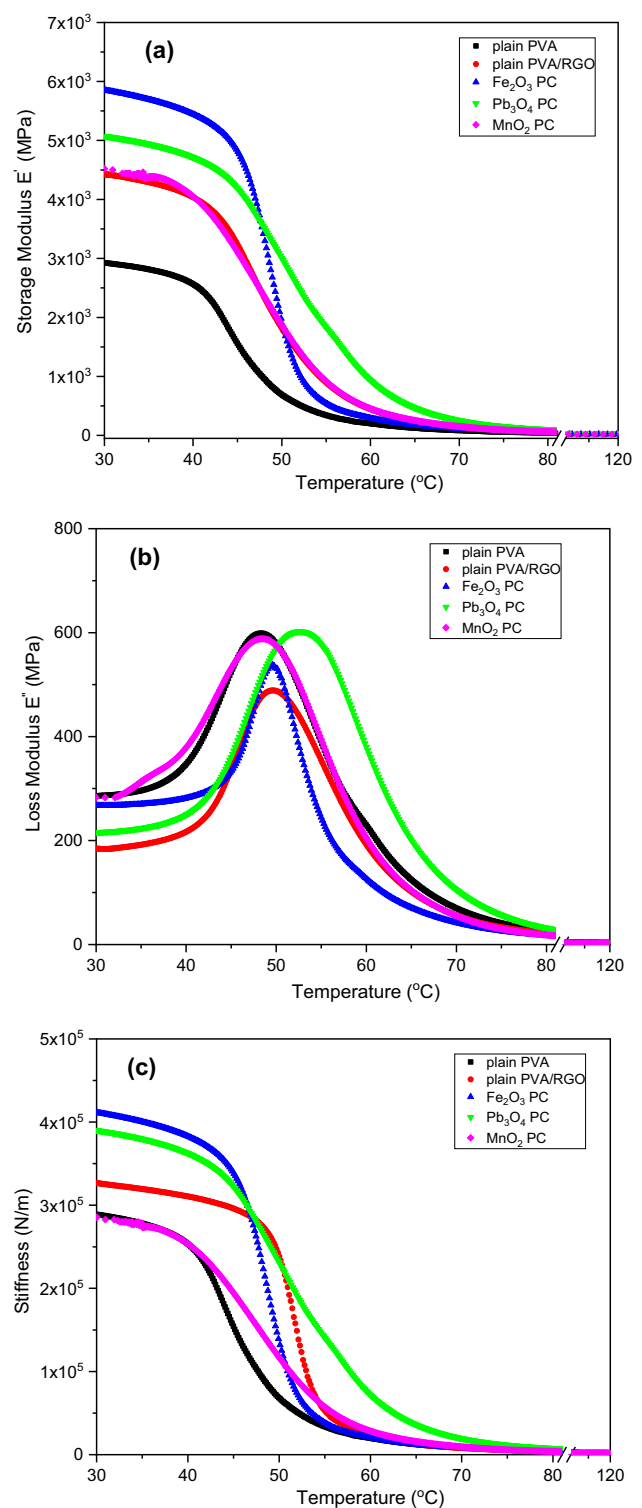
Furthermore, the activation energy ( $E_a$ ) of the plain PVA and metal oxides PCs has been investigated based on Arrhenius formula (Eq. 16). Figure 9b presents plots of  $\ln \sigma_{dc}$  vs.  $1000/T$  of the samples. The linear fits of the plotted measurements reveal their compatibility with the Arrhenius law [81, 82]. The deduced values of  $E_a$  are listed in Table 5. It is noticed that the  $E_a$  value depends on the incorporation of metal oxides' type. In addition, it is clear that the metal oxides incorporation leads to the decrease in the activation energy from 0.66 eV (plain PVA) to 0.05 eV ( $\text{Fe}_2\text{O}_3$  incorporation). This result reveals that metal oxides incorporation causes ions migration as well as production of charges' carriers [83]. Moreover, the decrease in  $E_a$  value is caused by the bandgap shrinkage as confirmed in the optical properties analysis. Our results agree with the reported ones [83, 84].

### 3.5 Mechanical properties analysis

The effect of the metal oxides incorporation on the dynamic mechanical properties performance of the PVA/rGO blend has

been examined using a dynamic mechanical analyzer (DMA) in temperature swept mode. The importance of mechanical properties investigation arises due to its great role in many applications as those needed the elasticity as well as hardness natures. For example, the storage modulus ( $E'$ ) reflects the energy-storage ability of the sample [85]. While the loss modulus ( $E''$ ) symbolizes the energy dissipated in it [86]. Figure 10a–c illustrates the storage modulus ( $E'$ ), loss modulus ( $E''$ ) and stiffness of plain PVA and metal oxides incorporated and incorporated PVA/rGO blend vs. temperature. A great effect on the whole mechanical properties due to the rGO blending as well as metal oxides' incorporation and their types. According to Fig. 10, each curve can be divided into three regions. The first one is called the glassy region lies from 30 °C to about 45 °C, where the quasi-stability feature is noticed. The second region lies in the temperature range from 45 °C to about 65 °C that called the glassy-leathery transition region. In the transition region, the mechanical properties are greatly altered. For example, the storage modulus and stiffness behavior decrease steeply, while the loss modulus increases sharply. However, the third region lies in the temperature region more than 70 °C, the mechanical properties return to stability character. The third region is mostly called the leathery region, where the samples are in their viscous status. Furthermore, the storage modulus, as well as the stiffness of the incorporated PCs, are enhanced as compared with the plain ones. The obtained storage modulus values at 30 °C are listed in Table 6. It is noticed that  $E'$  is duplicated due to  $\text{Fe}_2\text{O}_3$  incorporation in PVA/rGO blend. This enhancement of the mechanical properties is attributed to the restriction feature of the host polymer chains as a result of the incorporation as compared to the plain one. As a novel result, the mechanical properties of the PVA could be enhanced via metal oxides incorporation to meet different applications in elastic stiffening devices. Comparable conclusions were accomplished in [86–88].

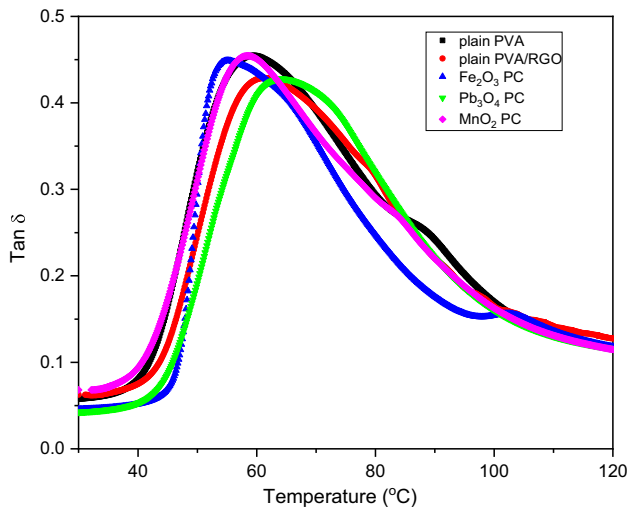
Moreover, the glass transition temperature ( $T_g$ ) is an important thermal property that must be investigated to direct the applications of the prepared PCs in such fields.  $T_g$  is the temperature that matches the midpoint between the glassy and rubbery region of the PCs [14]. To perform that, curves of the damping factor ( $\text{Tan}\delta (= \frac{E''}{E'})$ ) of the samples must be plotted as a function of temperature, as shown in Fig. 11.  $T_g$  values match the maxima of the  $\text{Tan}\delta$  values [89, 90]. The achieved  $T_g$  values are included in Table 6. This novel result of controlling the  $T_g$  value of the PVA polymer via incorporation with metal oxides is appreciated in many applications.



**Fig. 10** **a** Storage modulus, **b** loss modulus and **c** stiffness of metal oxides PCs

**Table 6** Storage modulus ( $E'$ ) at 30 °C and glass transition temperature ( $T_g$ ) of plain PVA and metal oxides incorporated and unincorporated PCs

Sample	$E'$ ( $\times 10^3$ MPa) at 30 °C	$T_g$ (°C)
plain PVA	2.93	59.45
plain PVA/rGO	4.46	61.56
Fe <sub>2</sub> O <sub>3</sub> PC	5.94	55.04
Pb <sub>3</sub> O <sub>4</sub> PC	5.08	64.68
MnO <sub>2</sub> PC	4.52	58.37

**Fig. 11** Plots of Tan  $\delta$  vs. temperature of metal oxides PCs

## 4 Conclusions

Plain PVA sample and 2.0 wt% of metal (M: Fe, Pb and Mn) oxides incorporated PVA/reduced graphene oxide polymeric composites (PVA/rGO PCs) were equipped using the solution casting procedure. Clear variations in the bands' absorption spectra have been noticed in the FT-IR spectra of the prepared samples. The effect of the metal oxide's type on the optical properties of the host PVA/rGO blend has been investigated. The energy band-gap decreases from 5.41 eV (plain PVA) to 5.36 eV (plain PVA/rGO), 5.23 eV (Fe<sub>2</sub>O<sub>3</sub> PC), 5.27 eV (Pb<sub>3</sub>O<sub>4</sub> PC) and 5.22 eV (MnO<sub>2</sub> PC). While the Urbach energy increases from 0.21 eV (plain PVA) to 0.28 eV (plain PVA/rGO), 0.41 eV (Fe<sub>2</sub>O<sub>3</sub> PC), 0.64 eV (Pb<sub>3</sub>O<sub>4</sub> PC) and 0.34 eV (MnO<sub>2</sub> PC). The refractive index ( $n$ ) at 600 nm wavelength increases from 1.45 (plain PVA) to 2.02 (plain PVA/rGO) and then to 2.79 (MnO<sub>2</sub> PC), 3.22 (Pb<sub>3</sub>O<sub>4</sub> PC) and 3.51 (Fe<sub>2</sub>O<sub>3</sub> PC). The infinite dielectric constant ( $\epsilon_\infty$ ) of the metal oxides PCs is enhanced more than five times as compared with that of the plain PVA. These results

reveal the increase in the defects and vacancies due to the incorporation process. The dc-electrical conductivity of the PVA/rGO blend is strongly enhanced via metal oxides incorporation. The activation energy of the host matrix decreases from 0.66 eV (plain PVA) to 0.18 eV (plain PVA/rGO), 0.05 eV (Fe<sub>2</sub>O<sub>3</sub> PC), 0.20 eV (Pb<sub>3</sub>O<sub>4</sub> PC) and 0.43 eV (MnO<sub>2</sub> PC). The effect of metal oxide's type on the PVA/rGO blend mechanical properties (storage modulus, loss modulus and stiffness) and glass transition temperature has been examined. The storage modulus ( $E'$ ) of the plain PVA is improved from  $2.93 \times 10^3$  MPa (plain PVA) to  $4.46 \times 10^3$  MPa via blending with rGO. While  $E'$  of the PVA/rGO blend is enhanced to  $5.94 \times 10^3$  MPa,  $5.08 \times 10^3$  MPa and  $4.52 \times 10^3$  MPa due to Fe<sub>2</sub>O<sub>3</sub>, Pb<sub>3</sub>O<sub>4</sub> and MnO<sub>2</sub> incorporation, respectively. It is noticed that  $E'$  is duplicated due to Fe<sub>2</sub>O<sub>3</sub> incorporation in PVA/rGO blend. The glass transition temperature ( $T_g$ ) value increases from 59.45 °C (plain PVA) to 61.56 °C (plain PVA/rGO) and 64.68 °C (Pb<sub>3</sub>O<sub>4</sub> PC). While it decreases to 55.04 °C (Fe<sub>2</sub>O<sub>3</sub> PC) and 58.37 °C (MnO<sub>2</sub> PC). Our unique results introduce a new procedure for controlling the optical, electrical and mechanical properties of polymeric blends via metal oxides incorporation for applications in flexible optoelectronic devices.

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## Declarations

**Conflict of interest** The authors declare no conflict of interest.

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