

# Microstructure and Dispersion Parameters of Hybrid PMMA/(TiO<sub>2</sub>-ZrC) Electrospun Nanofibers for Optoelectronics and Antimicrobials Purposes

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Received: 2 March 2025, Revised: 18 March 2025, Accepted: 25 March 2025, Published: 1 August 2025

## Abstract

The method of solution electrospinning under the main conditions of 13 kV, 0.5 mL per h flow rate, and collector rotating 250 per min was used to fabricate PMMA composite nanofibers using mixed (TiO<sub>2</sub>-ZrC) nanomaterials (1:1 wt.%) as a filler in varying concentrations: 2, 4, and 6 % to utilize in futuristic photonics applications. FTIR results confirm strong interactions between the PMMA matrix and (TiO<sub>2</sub>-ZrC). Morphological analysis performed through FESEM confirmed the average diameters of the nanofibers, which showcased 82.37 nm for the polymer and increased to 139.59 nm at a weight of 6 %, while their rough surfaces are transformed into smoother ones depending on the addition of nanomaterials. EDXs provide insights into the element's composition of the PMMA encapsulating by TiO<sub>2</sub> and ZrC. A decrease in light transmittance in the UV region gives a positive indication of the protective action in solar cells. Increasing the TiO<sub>2</sub>-ZrC nanoparticle content by 6 wt.% gradually improved the indirect transition band gap ( $E_{g\text{ indir}}^{\text{opt}}$ ) for the allowed and forbidden from 3.5 and 3.2 eV for the pure polymer to 2.8 and 2.18 eV. This finding is a positive indication of its use in many optoelectronic devices. The theoretical Wemple-DiDomenico model has been implemented to evaluate the refractive index (ns), the oscillation energy (Eos), and the dispersion energy (Ed). Moreover, (TiO<sub>2</sub>-ZrC) demonstrated upper optical activity and elevated DNA decay efficiency than PMMA, validating their higher efficiency as an optical and antimicrobial agent.

**Keywords:** PMMA, TiO<sub>2</sub>, ZrC, Nanofiber, Electrospinning, Optical properties

## Introduction

Nanomaterials exhibit unique properties due to their small size and large surface area, making them useful in various fields [1]. Due to academic and industrial interests, their presence with inorganic polymeric materials has been important during the last 2 decades. Due to the significant improvements, it adds to the properties, such as mechanical, thermal, optical, and electrical properties of different composite membranes and recent developments in electrospinning technology, different types of polymers/inorganics and their nanocomposites have been produced [2,3].

Poly methyl methacrylate (PMMA), a synthetic resin derived from the polymerization of methyl methacrylate with the empirical formula (C<sub>5</sub>H<sub>8</sub>O<sub>2</sub>), exhibits a glass transition temperature of 105 °C and

demonstrates inadequate mechanical properties, including low impact resistance and susceptibility to fatigue failure. However, their optical properties, including clarity, index of refractive, haze, and light transmission, are critical features that make them uniquely advantageous for several design and engineering applications, including optics, lighting, and displays [4].

Titanium dioxide (TiO<sub>2</sub>) is a semiconducting transition material. It shows unique characteristics, such as easy control, low cost, nontoxicity, wide energy gap (3.0 to 3.2 eV), and great resistance, which can absorb ultraviolet rays and become irritated. The unique physiochemical features of metal oxide NPs, such as (TiO<sub>2</sub>), have made them widely used in biological

applications in recent decades [5]. According to several publications, TiO<sub>2</sub> NPs have been among the most investigated TiO<sub>2</sub> NPs due to their photocatalytic and antibacterial activity, which exerts good bio-related action against bacterial contamination [6]. Zirconium carbide (ZrC) is an attractive material for optical applications due to its excellent optical performance in the infrared range and high stability even at elevated temperatures. Doping ZrC nanoparticles with different elements has emerged as an effective strategy to improve their optical performance with a wide band gap of about 2.9 eV [7]. ZrC NPs have garnered attention due to their cubical shape, strong bands, stability, and applicability in biomedicine and drug delivery. Ongoing studies focus on the impact of different media on the energy gap of ZrC NPs, as the energy gap is size and shape-dependent and influenced by the surrounding medium [8]. Studies have demonstrated significant interest in nanoparticles due to their potential for various uses, such as their antibacterial properties.

Electrospinning is an economical and accurate technique for generating polymeric fibers with diameters spanning the nano-to-micron range, yielding a substantial length-to-diameter ratio [9]. Consequently, electro-spun polymer nanofibers are appealing materials for diverse applications, including tissue engineering scaffolds, filtration media, protective garments, and antimicrobial membranes [9]. It is possible to vary the carrier concentration in the matrix material by adding many elements or oxides to develop improved composite applications [10]. By electrospinning solutions with N,N-dimethyl formamide (DMF) as the solvent, Chen *et al.* [11] were able to successfully create PMMA/silica nanocomposite fibers that had up to 20 weight percent silica. Kanth *et al.* [12] successfully fabricated porous PMMA substrates with immobilized TiO<sub>2</sub> NPs in fiber shapes. These substrates were then evaluated to illustrate the influence of pollutant movement and light accessibility on the total photocatalytic performance. Sallomi *et al.* [13] succeeded in the electrospinning method to prepare PMMA/TiO<sub>2</sub> nanofibers. The influence of collector rotation speed on the diameters, morphology, and structural characteristics of the synthesized PMMA/TiO<sub>2</sub> nanofibers was examined. The primary aims of this study are to fabricate nanofibers using the electrospinning technique with PMMA and a mixture of

TiO<sub>2</sub> and ZrC, and to analyze their morphological, optical, and dispersion characteristics for prospective applications in various optoelectronic and antimicrobial fields.

## Materials and methods

### Materials

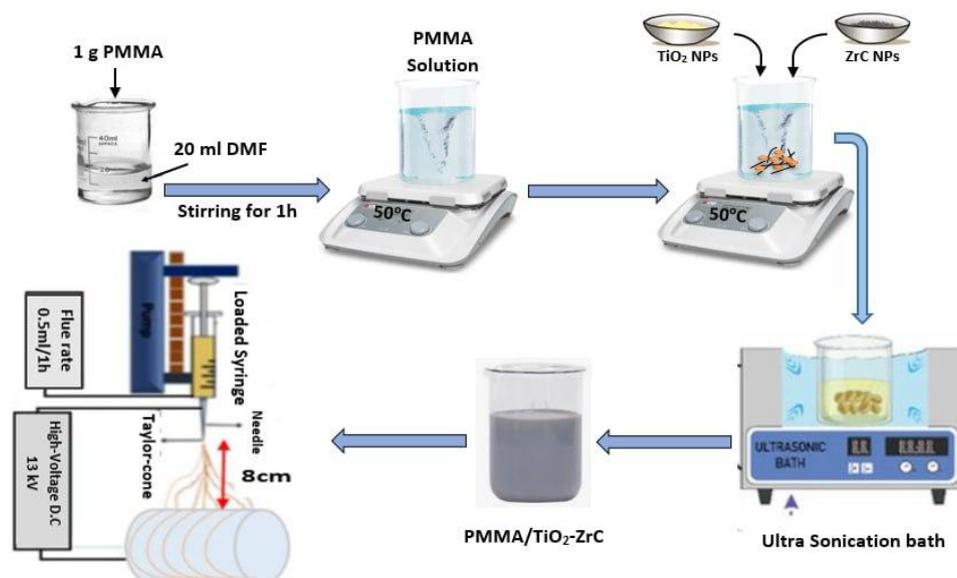
White powder of TiO<sub>2</sub>, 99 %, < 80 nm, 79.8658 g.mol<sup>-1</sup>, and black powder ZrC, 99 %, < 80 nm, 103.235 g.mol<sup>-1</sup> were bought and acquired from US Research Nanomaterial's, Inc., USA. PMMA [CH<sub>2</sub>=C (CH<sub>3</sub>) COOCH<sub>3</sub>], with a purity of minimum assay around 98 % with MW~120,000 g.mol<sup>-1</sup>, was bought from SIGMA-ALDRICH (Germany). DMF, purity > 90 %, was selected to dissolve PMMA polymer obtained from Alpha Chemical, India.

### Preparation the solution of PMMA / (TiO<sub>2</sub>-ZrC)

The preparation of a nanocomposite (NC) solution from PMMA / (TiO<sub>2</sub>—ZrC) was described by dissolving 1 g of PMMA in 20 mL of DMF and stirring for 1 h at 50 °C using a magnetic stirrer. The (TiO<sub>2</sub>—ZrC) NPs were added in a 1:1 ratio at weights equal to 2, 4, and 6 % with continuous mixing and using ultrasound many times (at least thrice) to achieve homogeneity. The solution now undergoes electrospinning to fabricate the nanofibers.

### Preparation of PMMA/ (TiO<sub>2</sub>- ZrC) nanofibers

The synthesis of PMMA/(TiO<sub>2</sub>-ZrC) composite nanofibers is delineated as follows: An aluminum foil of 10×25 cm<sup>2</sup> was encased around the drum collector, and requisite modifications were implemented to the needle tip-to-collector distance. The solution was transferred into a 5 mL glass syringe sealed with an 18-gauge blunt cannula. The electrospinning tests were performed at a flow rate of 0.5 mL per h and 13 kV within a sealed environmental room illuminated by UV light, as depicted in **Figure 1**. A metal capillary needle was employed to transfer the prepared solution into a 5-milliliter syringe, positioned 8 cm from a metal cylinder rotating at 250 revolutions per min. The film thicknesses measured approximately 20 μm.



**Figure 1** Diagram of the electrospinning to manufacturing nanofibers.

### Characterization

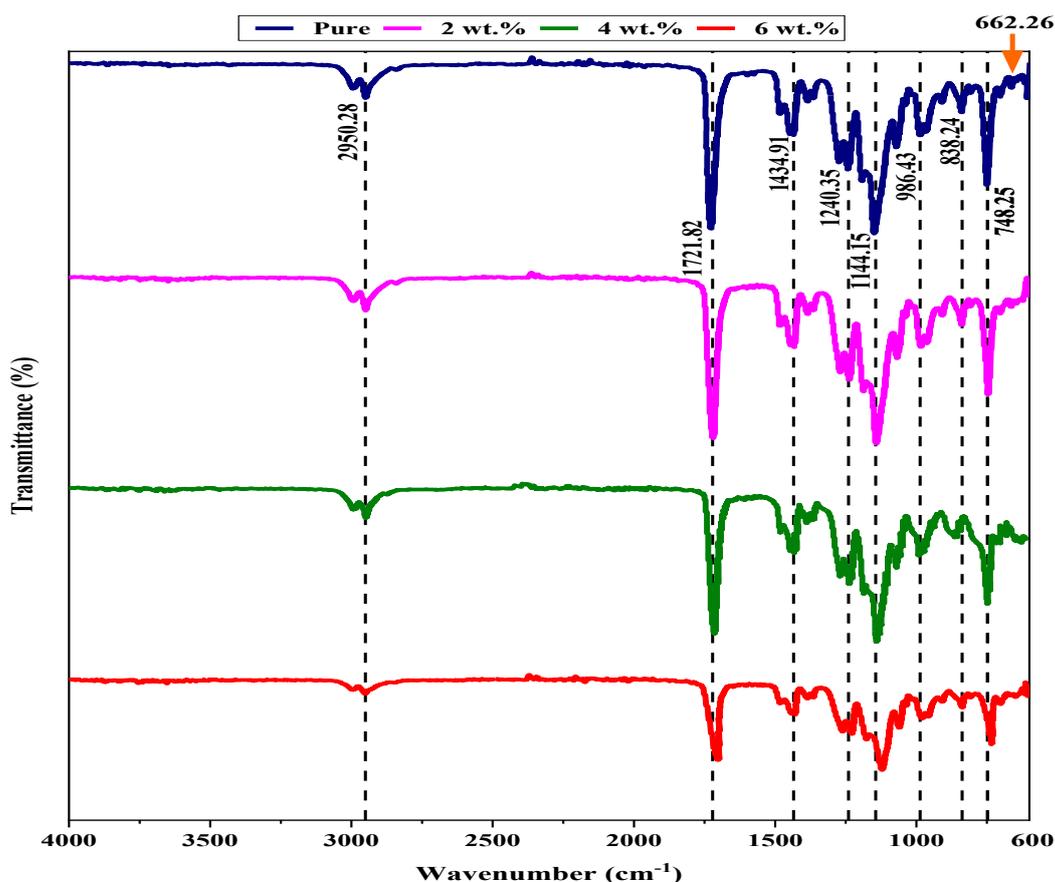
The chemical composition of the PMMA/(TiO<sub>2</sub>-ZrC) composite nanofibers was characterized using FTIR spectroscopy (Bruker company, German origin, type vertex-70) at (4000 - 400) cm<sup>-1</sup>. The surface morphology was examined using FESEM (MIRA3 TESCAN). EDXs imparted insights into the chemical composition of the PMMA encapsulating TiO<sub>2</sub> and ZrC and utilizing image processing software to calculate the sizes of nanofibers in detail. The sorption behavior was examined by (Shimadzu UV-1800) spectrophotometer over a wavelength of (200 to 1100 nm) range.

### Results and discussion

#### FTIR spectra

**Figure 2** displays the FTIR spectroscopy of PM and its nanocomposite with different weight percentages of a mixed (TiO<sub>2</sub>-ZrC) in the wavenumber range of (600 - 4000) cm<sup>-1</sup>, recorded at room temperature. The peaks seen at 2950.28, 1721.82, 1434.91, ~1240.35, and ~1144.15 cm<sup>-1</sup> are due to the asymmetric stretching of the CH<sub>2</sub> group Tommasini *et al.* [14], carbonyl group (C=O) Salloomi *et al.* [13], Torrisi *et al.* [15], C-H

deformation Ali *et al.* [16], C-O stretching and skeletal vibrations coupled to the C-H deformations Godiya *et al.* [17], respectively. The peaks at 986.43, 838.24, and 748.25 cm<sup>-1</sup> are customized to CH<sub>2</sub> rocking mode [15]. Also, it can be seen at around 662.26 cm<sup>-1</sup> new clear bands show when the concentration of (TiO<sub>2</sub>-ZrC) is high enough 4wt.%, which can be customized to vibration modes of Ti-O-Ti and Zr-O-Zr [18-20]. The results related to the FTIR technique indicate that the slight deviation in the peak positions towards greater or smaller wavelengths occurs as a result of compensation or the effect of solvents. This suggests physical, structural changes represented by the strong hydrogen bonding interaction between the polymer matrix and additive nanomaterials. Also, from such a figure, it is clear that increasing the weight percentage of additive NPs, the regularity of the nanofibers, and their diameters do not affect the functional groups of PMMA/ (TiO<sub>2</sub>-ZrC) nanofibers. This may be because the change in the physical dimensions of the nanofibers does not involve chemical reactions affecting the functional groups of the PMMA/(TiO<sub>2</sub>-ZrC) nanofibers [21].



**Figure 2** The FTIR spectra of the PMMA/ (TiO<sub>2</sub>-ZrC) composite nanofibers.

### Morphology of (TiO<sub>2</sub>-ZrC) loaded PMMA composite nanofibers

Our images shown in **Figures 3 - 6** are the FESEM (taken at magnification 15 kx and inset 60 kx), histograms of diameters with Gaussian distribution, and EDXs for pure solution PMMA and its nanocomposite with different weights 2, 4, and 6 % of a mixed (TiO<sub>2</sub>-ZrC) NPs. The nanofibers from pure solution PMMA had a porous and rough surface, as mentioned in the research Kanth *et al.* [12], while their rough surfaces were transformed into smoother ones depending on the addition of nanomaterials. The average diameter of the nanofibers was approximately 82.377 nm for a pure PMMA and increased to 96.083, 100.955, and 139.589

nm for NCs with different weights 2, 4, and 6 % (TiO<sub>2</sub>-ZrC), respectively. The consistent rotation speed of the collector correlates with the rise in fiber diameter, attributable to the additive nanoparticles that altered the charge density and viscosity of the solution, hence elucidating the variation in fiber diameter [21].

Through the typical analysis of EDXs (dominant peak represented by aluminum foil on which the film is deposited) performed on the PMMA and its composite nanofibers, shown in part c of **Figure 3**, it was found that the typical peaks are associated with carbon (C), oxygen (O), titanium (Ti) and zirconium (Zr). This suggests the possibility of obtaining nanocomposites from PMMA/ (TiO<sub>2</sub>-ZrC).

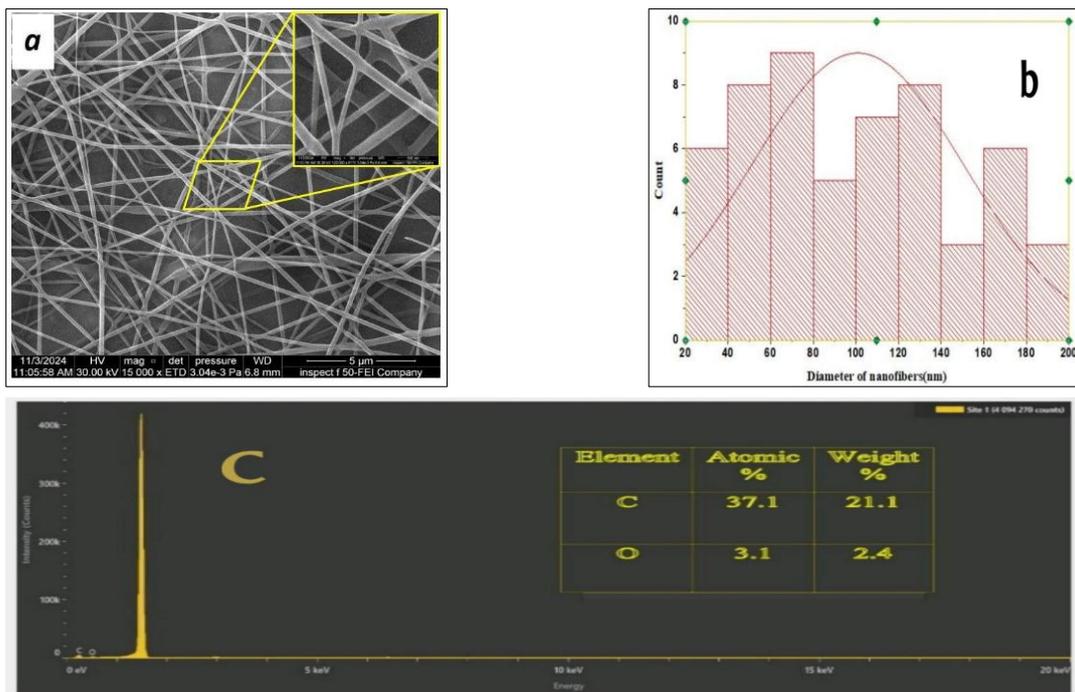


Figure 3 (a) FESEM images, (b) Gaussian distribution, and (c) EDXs of the pure PMMA.

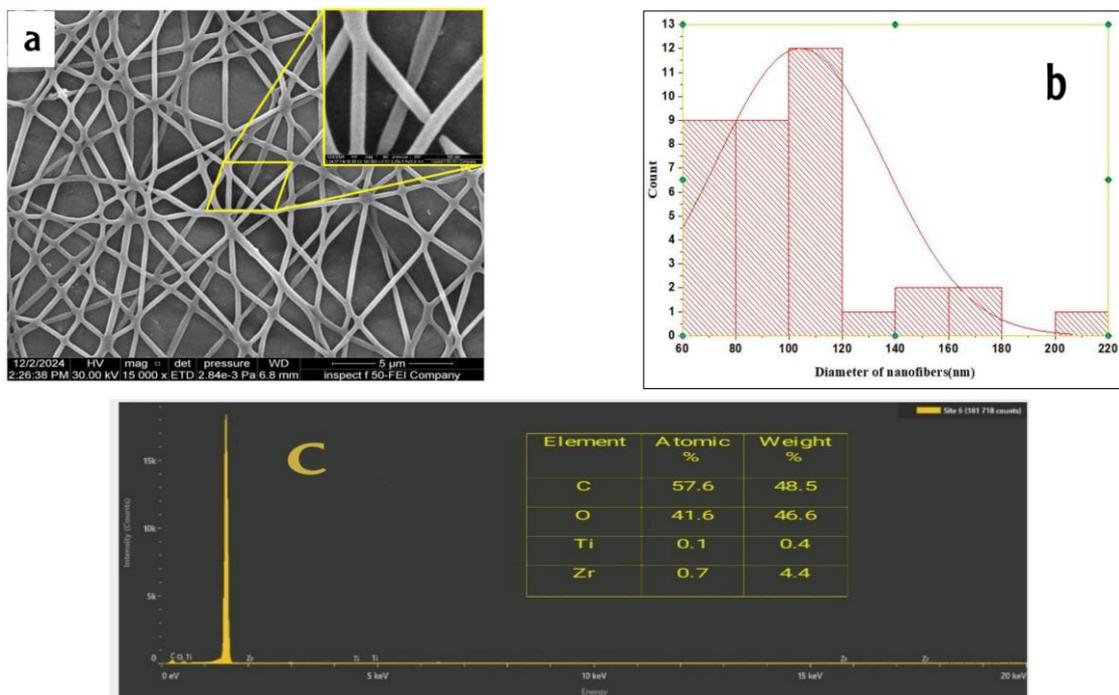


Figure 4 (a) FESEM images (b) Gaussian distribution, and (c) EDXs of PMMA/2wt.% (TiO<sub>2</sub>-ZrC).

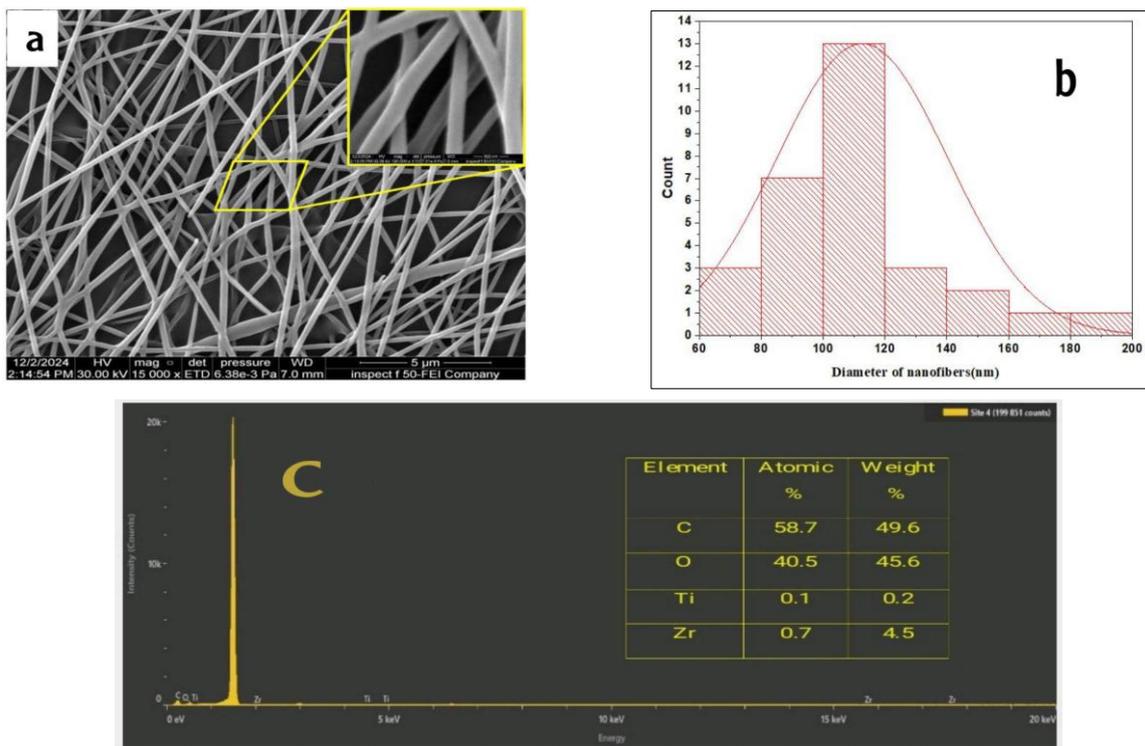


Figure 5 (a) FESEM images, (b) Gaussian distribution, and (c) EDXs of PMMA/4wt.% (TiO<sub>2</sub>-ZrC).

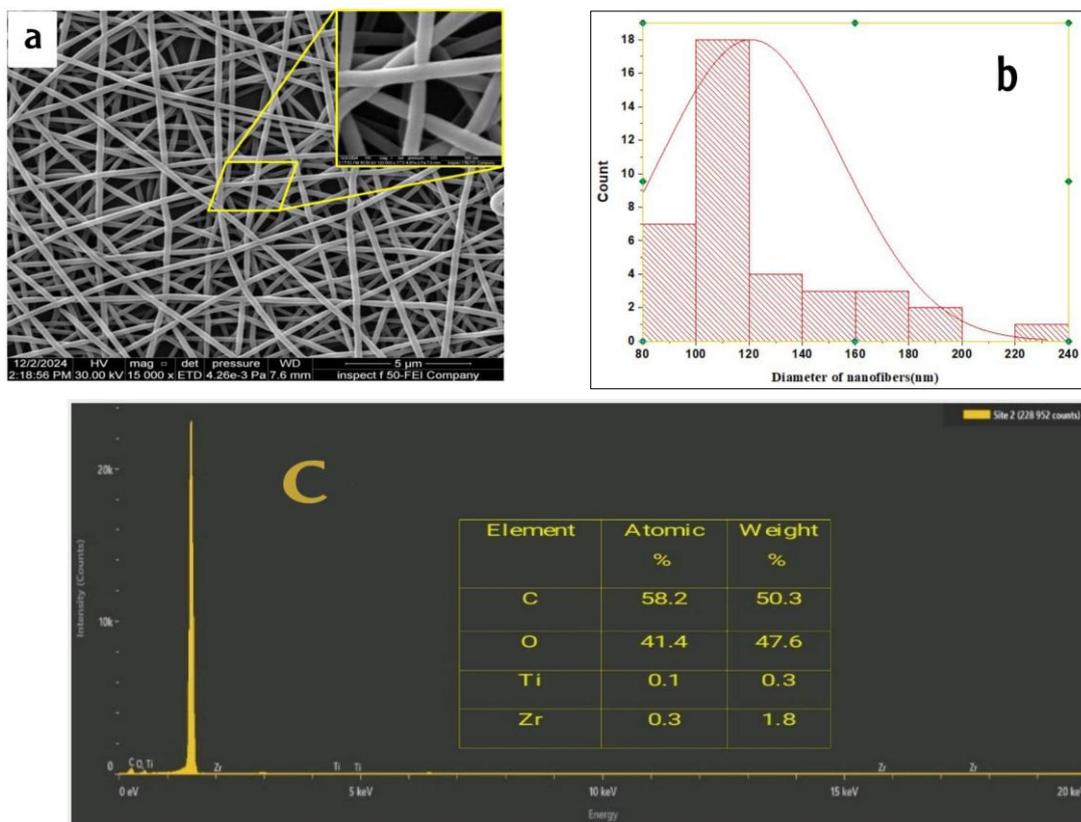


Figure 6 (a) FESEM images, (b) Gaussian distribution, and (c) EDXs of PMMA/6wt% (TiO<sub>2</sub>-ZrC).

**UV-visible spectrophotometer**

Figure 7 depicts the transmittance spectra of pure PMMA and 3 different weight 2, 4, and 6 % (TiO<sub>2</sub> - ZrC)

nanocomposites introducing into polymer matrix. The transmittance of pure PMMA film along the visible region is found to be in the range 94 - 96.5 %. At 6 wt.%

the transmittance values decrease to 87.6 % at the wavelength approaches 560 nm, since the increase of nanomaterial's could increase light absorbance and

decrease transmittance [22,23]. The reduction in light transmittance in the UV spectrum suggests a favorable protective effect on solar cells.

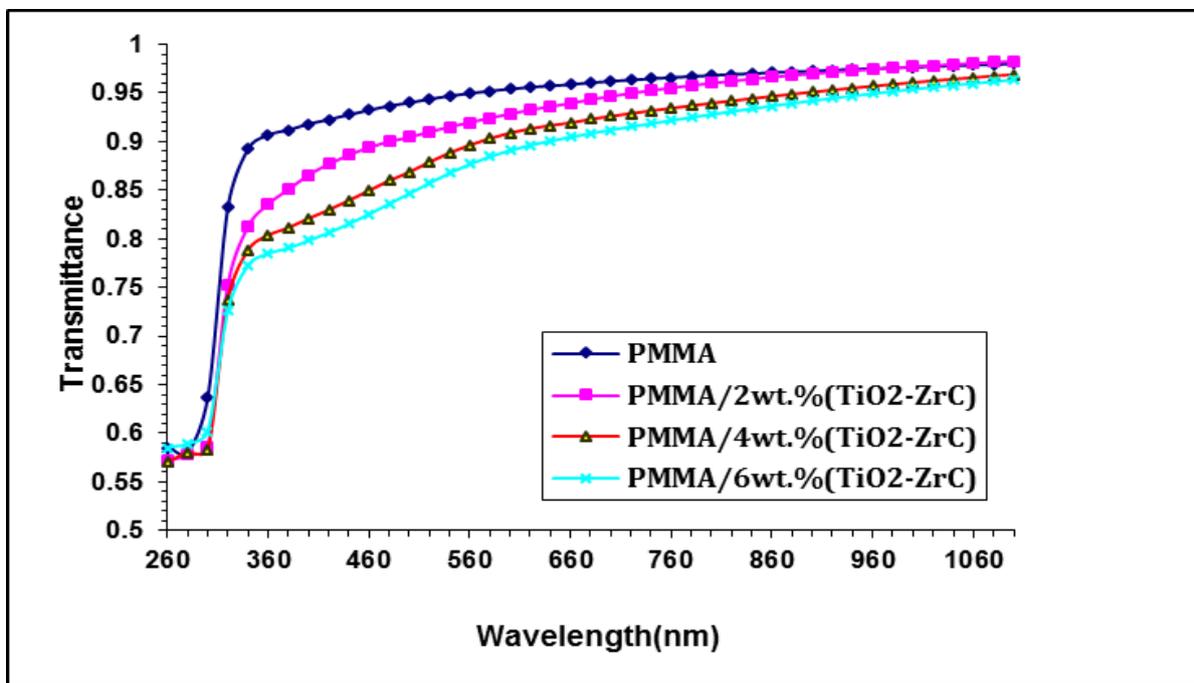


Figure 7 UV-Vis transmittance spectra of pure PMMA and 3 different (TiO<sub>2</sub>-ZrC) nanocomposites.

The type of electronic transitions between extended bands can be identified from the absorption coefficient ( $\alpha$ ) calculation [24].

$$\alpha = 2.303 \left(\frac{A}{t}\right) \text{ (A absorption and t thickness)} \quad (1)$$

From  $\alpha$  calculation, Urbach tail energy ( $E_U$ ) can be found from the relation, which shows inclusions and instabilities in a polymer matrix [25,26].

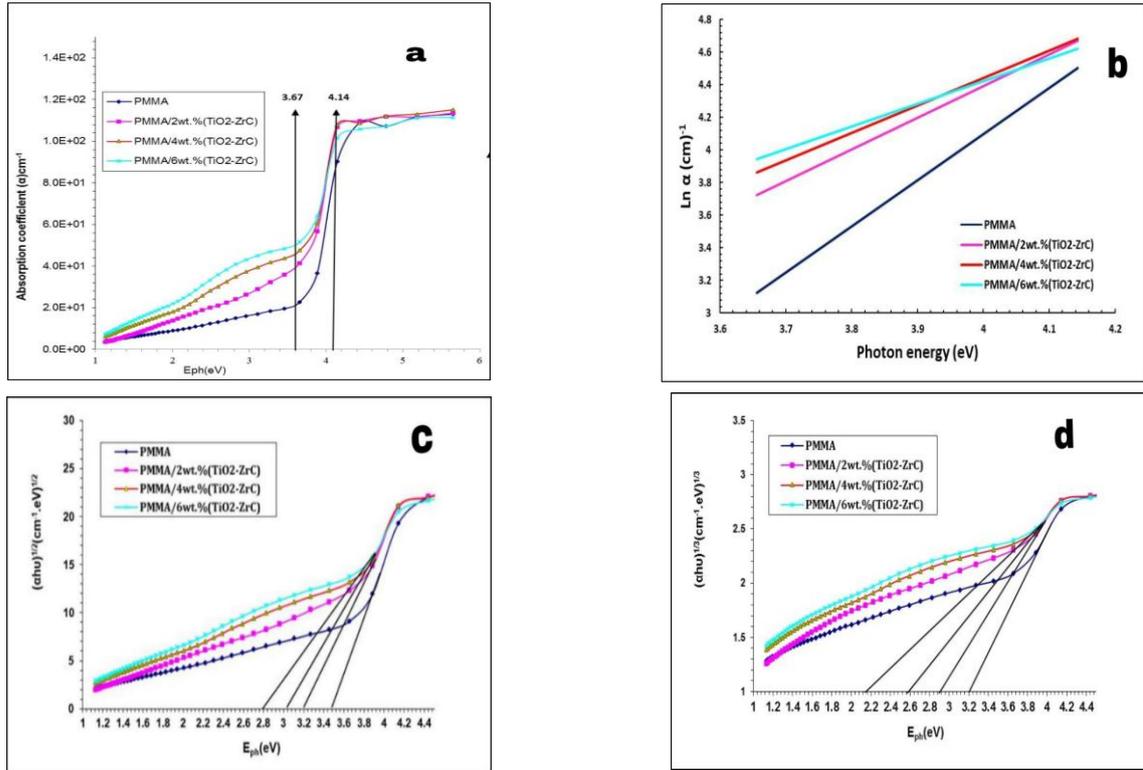
$$\ln \alpha = \ln \beta_0 + \frac{hv}{E_U} \text{ (hv signifies the energy of the photon)} \quad (2)$$

The predestined optical band gap ( $E_g^{opt}$ ) were evaluated from the following relation [27].

$$ahv = B (hv - E_g^{opt})^n \quad (3)$$

where B is defined as a constant, and the n takes a variable values of 2 and 3 for allowed and prohipted indirect transitions.

The absorption coefficient values in **Figure 6(a)** were less than  $10^4 \text{ cm}^{-1}$  indicated occur indirect transfers in all prepared samples [28]. The  $E_U$  values are obtained by fitting the graph's straight section, which is the inverse of its slope, as shown in **Figure 6(b)**, respectively, 0.35, 0.51, 0.59, and 0.72 eV for pure PMMA and 3 mixed different weight 2, 4, and 6 % (TiO<sub>2</sub>-ZrC) NCs. The allowed and forbidden transitions band gap ( $E_{g\text{indir}}^{opt}$ ) decreased from 3.5 and 3.2 eV (pure polymer) to 2.8 and 2.18 eV at a weight of 6 % nano additives, as shown in **Figure 8** and tableted in **Table 1**. The chaotic structure formed by the polymer chains upon solidification and the presence of TiO<sub>2</sub> and ZrC nanomaterials leads to the formation of disordered atoms and structure-related defects that can lead to the emergence of local states that act as donor centers close to or at the conduction band level, causes to increase in the Urbach tail. Consequently, the optical energy gap decreases. This finding provides a positive indication for use in many optoelectronics devices, like photovoltaic cell applications.



**Figure 8** (a) Spectral distributions of  $\alpha$ , (b)  $E_U$ , (c) and (d) allowed and forbidden indirect transition ( $E_g^{opt}$ ) for pure PMMA and 3 different mixed ( $TiO_2$ -ZrC) NCs.

**Table 1** The values of both allowed and forbidden indirect transitions and Urbach energy ( $E_U$ ).

( $TiO_2$ -ZrC) wt. %	Allowed $E_g^{opt}$ (eV)	Forbidden $E_g^{opt}$ (eV)	$E_U$ (eV)
0	3.50	3.2	0.35
2	3.20	2.95	0.51
4	3.00	2.6	0.59
6	2.80	2.18	0.72

The optical conductivity ( $\sigma_{op}$ ) has been specified using the following relation [29].

$$\sigma_{op} = \alpha n c / 4\pi \quad (c \text{ speed of light}) \quad (4)$$

Optical conductivity is closely linked to the optical band gap, making it an essential field of investigation.

The relationship between  $\sigma_{op}$  and  $\lambda$  for PMMA and

3 different mixed ( $TiO_2$ -ZrC) NCs is shown in **Figure 9**.  $\sigma_{op}$  depends on the  $\alpha$  and can be noticing a sharp increase in the UV region. The  $\sigma_{op}$  was determined to transmit in the Vis and NIR ranges. The augmentation of inorganic filler quantity leads to an elevation in the density of localized states within the forbidden energy gap, hence boosting  $\alpha$  and subsequently  $\sigma_{op}$  [30].

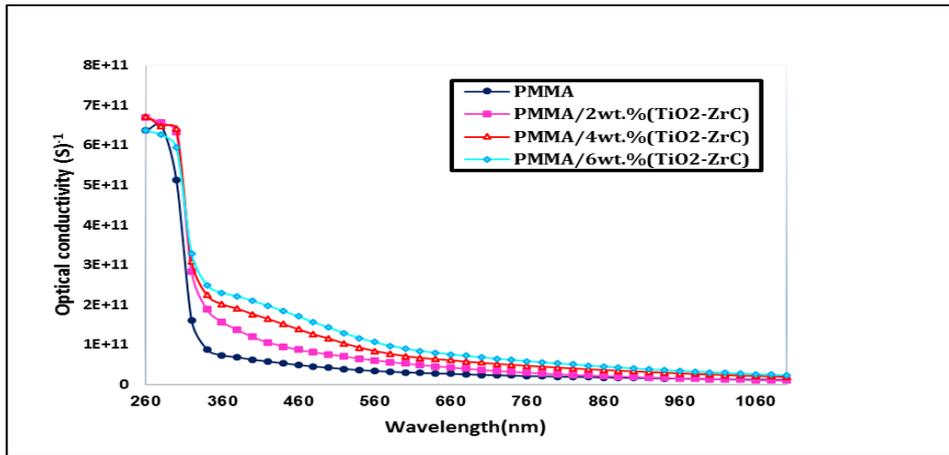


Figure 9 Optical conductivity for PMMA and 3 different mixed (TiO<sub>2</sub>-ZrC) NCs.

**Parameters of dispersion energy**

The Wemple - DiDomenico model include, the energy of single oscillator (E<sub>os</sub>), the energy of dispersion (E<sub>d</sub>), and the photon energy (hν) [31]. The Wemple DiDomenico model can be expressed mathematically as follows [31].

$$(n^2 - 1)^{-1} = \frac{E_{os}}{E_d} - \frac{1}{E_{os} E_d} (h\nu)^2 \tag{5}$$

The plotted data from  $(n^2 - 1)^{-1}$  as a function of  $(h\nu)^2$  for PMMA with various ratios of (TiO<sub>2</sub>-ZrC), forms a straight line with a y-intercept equal to  $(E_{os} / E_d)$  and a slope equal to  $(1/E_{os}E_d)$ , as in **Figure 10**. The calculated values of E<sub>os</sub> and E<sub>d</sub> displayed in **Table 2**. The energy parameter E<sub>os</sub> of PMMA reduces with the addition of (TiO<sub>2</sub>-ZrC), which corresponds to the decrease in the optical energy gap. Moreover, E<sub>d</sub> values increase, which indicates that the inclusion of (TiO<sub>2</sub>-ZrC) improved the optical transition intensity as a result of atomic diffusion to interstitial locations in the PMMA polymer [32].

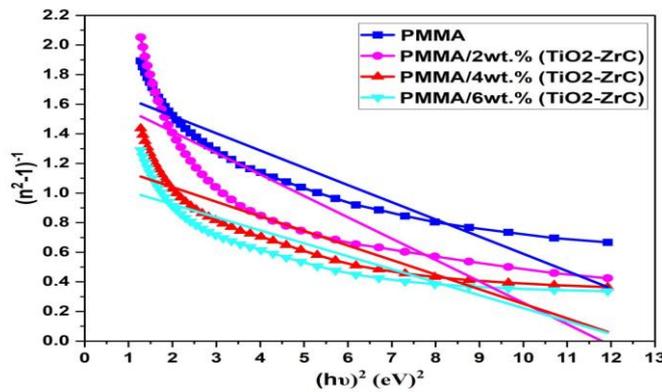


Figure 9 Plot of  $(n^2 - 1)^{-1}$  vs  $(h\nu)^2$  for PMMA and 3 different mixed (TiO<sub>2</sub>-ZrC) NCs.

The values of E<sub>os</sub> and E<sub>d</sub> can be used to calculate the dielectric constant ( $\epsilon_s$ ) and the static refractive index ( $n_s$ ) from the following equation [33]:

$$\epsilon_s = n_s^2 = 1 + \frac{E_d}{E_{so}} \tag{6}$$

These values (listed in **Table 2**) increased as the content of nanoadditives in the matrix increased. Furthermore, using (E<sub>d</sub> and E<sub>os</sub>), it is possible to compute the optical moments (M<sub>1</sub> and M<sub>3</sub>) for the current nanofiber films. These moments were determined as follows equations [34].

$$E_{os}^2 = \frac{M_{-1}}{M_{-3}} \quad \text{And} \quad E_d^2 = \frac{M_{-1}^3}{M_{-3}} \quad (7)$$

$$M_{-1} = \frac{E_d}{E_{os}} \quad \text{And} \quad M_{-3} = \frac{M_{-1}}{E_{os}^2} \quad (8)$$

By rearranged these equations, one may obtain [35]:

Increasing the (TiO<sub>2</sub>-ZrC) content resulted in a boost of the optical spectra moments, thereby strengthening the optical transitions.

**Table 2** The dispersion parameters for PMMA and 3 different mixed (TiO<sub>2</sub>-ZrC) NCs.

(TiO <sub>2</sub> -ZrC) wt. %	E <sub>os</sub> (eV)	E <sub>d</sub> (eV)	n <sub>s</sub>	ε <sub>s</sub>	M <sub>-1</sub>	M <sub>-3</sub>
0	3.88	2.21	1.25	1.5625	0.57	0.038
2	3.43	2.02	1.26	1.5876	0.59	0.049
4	3.54	2.86	1.34	1.7956	0.81	0.064
6	3.55	3.22	1.38	1.9044	0.91	0.072

### The antibacterial activity

When a material exhibits certain optical properties upon contact with bacteria, this may indicate that certain chemical or physical processes are taking place that contribute to the material's antibacterial activity and may enhance its efficiency [36]. The PMMA and 3 different mixed (TiO<sub>2</sub>-ZrC) NCs were assessed for antibacterial activities against 4 bacterial strains, gram-negative *Escherichia coli*, *klebsiella aurous*, *Pseudolysogeny*, and gram-positive *Staphylococcus aurous*, as manifested in **Figure 10**. These selected bacteria because they are among the most common causes of clinically acquired infections, along with, delayed healing of soft tissue burn wounds [37].

The maximum inhibitory power of the bacterial zone (22 mm) against *Staphylococcus aurous* was recorded when (TiO<sub>2</sub>-ZrC) nanoparticles attained 6 wt.%, as illustrated in the histogram of **Figure 11**. These results can be interpreted in the case of PMMA/(TiO<sub>2</sub>-ZrC) composite nanofibers the positively charged ion of a metallic element generated from nanoparticles cross-react with proteins at the membrane of the cell barrier, and nucleic acids (DNA), causing alterations in their structure and inhibiting microbial reproduction. Also, reactive oxygen species (ROS) are responsible for taking up nanoparticles, which causes the mechanical breakdown of the cell membrane [38,39].

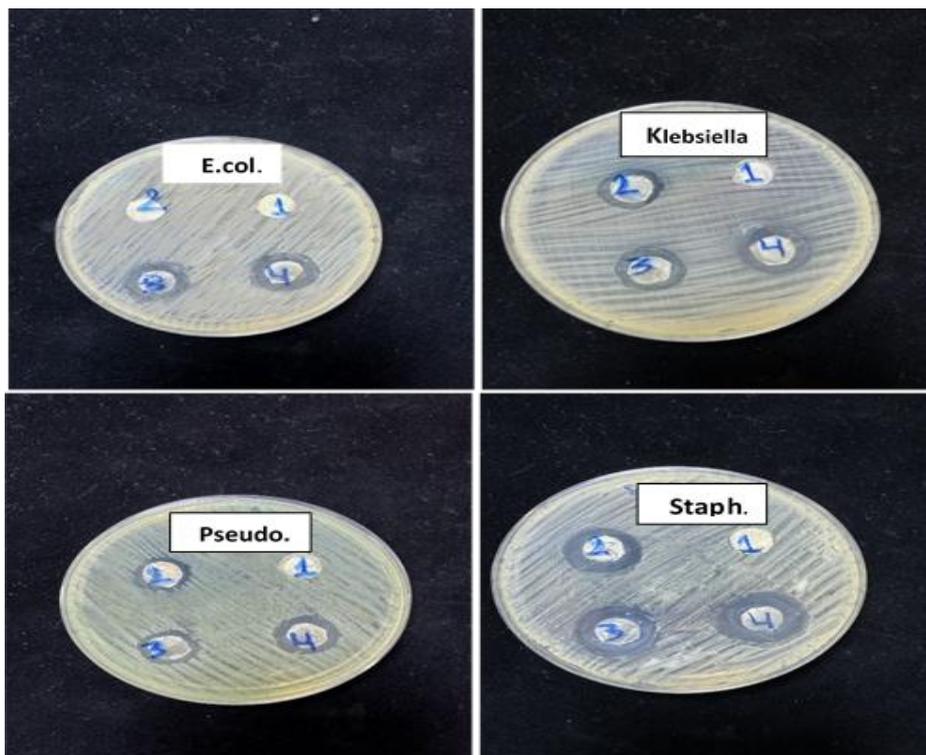


Figure 10 Antibacterial activity for PMMA and 3 different mixed (TiO<sub>2</sub>-ZrC) NCs.

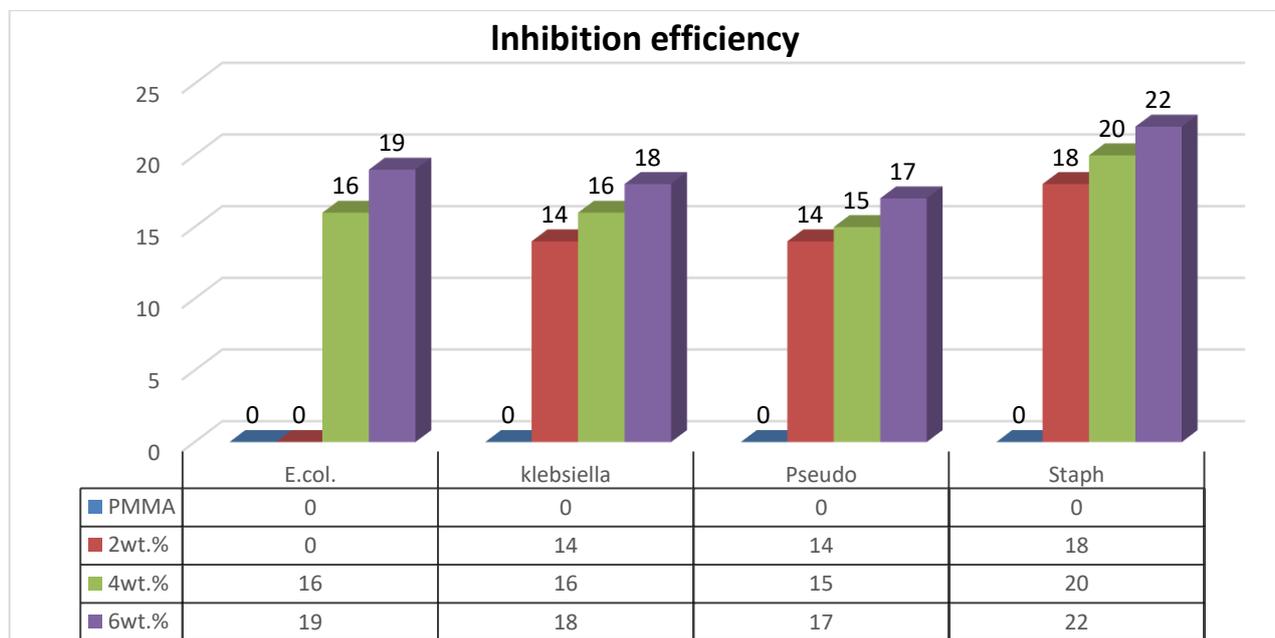


Figure 11 The effect of contain (TiO<sub>2</sub>-ZrC) NCs on inhibiting the growth of 4 bacterial strains.

### Conclusions

A successful attempt was made to fabricate PMMA/(TiO<sub>2</sub>-ZrC) nanofiber composites through electrospinning. The physical interaction between PMMA and (TiO<sub>2</sub>-ZrC) was approved through FTIR analysis. FESEM images verified that the electrospun

PMMA and its composite nanofibers measured between 82.37 and 139.59 nm. The reduction in light transmittance in the UV spectrum is a favorable indicator of the protective function in solar cells. The allowed and forbidden transitions decreased from 3.5 and 3.2 eV (pure polymer) to 2.8 and 2.18 eV at the

mixture of TiO<sub>2</sub> and ZrC nano additives reached a weight of 6 %, which makes an encouraging sign for use in many optoelectronic devices. A study of the broad optical spectra indicated that the E<sub>d</sub>, n<sub>s</sub>, ε<sub>s</sub>, M<sub>-1</sub>, and M<sub>-3</sub> increased with increasing (TiO<sub>2</sub>-ZrC) content. The greatest inhibition zone of gram-positive (*Staphylococcus aureus*) bacteria was about 22 mm at a weight of 6 %, making it usable as an alternative to conventional antibiotics.

### Acknowledgements

Thanks to University of Babylon, Iraq for support.

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