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## Research Article Preparation and Dielectric Property of TiO<sub>2</sub> Doping with Silver Dispersed in Polyvinyl Alcohol and Polyurethane (TiO<sub>2</sub>@Ag/PVA-PU) Nanocomposite Materials

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### Abstract

**Background and Objective:** The hybrid inorganic-organic materials have dielectric properties such as the addition of nanosized inorganic particles into polymer matrixes is very important for energy storage application. This research aims to prepare nanocomposite material that exhibits dielectric properties that could be used for energy storage. **Material and Methods:** By applying the irradiation technique, silver nanoparticles were successfully formed onto the surface of the TiO<sub>2</sub> nanoparticles given (TiO<sub>2</sub>@Ag). The (TiO<sub>2</sub>@Ag) was formed *"in situ"* the PVA/PU matrixes showed that the homogeneous dispersion of nanosized particle "*in situ"* polymer matrixes is very easy. In the first approach, the mixture of TiO<sub>2</sub> and AgNO<sub>3</sub> with PVA/PU is followed by an *in situ* polymerization induced by gamma irradiation. The physical properties of TiO<sub>2</sub> nanoparticles are enhanced through silver dobbing. The dielectric behavior of the prepared (TiO<sub>2</sub>@Ag/PVA-PU) nanocomposite was investigated at different temperatures and frequency. **Results:** The dielectric constant values ( $\varepsilon$ ') of (TiO<sub>2</sub>@Ag/PVA-PU) are increased by increasing both temperature and frequency. **Conclusion:** The increase in  $\varepsilon$ ' values with increasing temperature indicates that the dipoles in the polymer matrix are thermally activated and have more rotational deliverance by heating.

Key words: Gamma irradiation, in situ polymerization, TiO2@Ag, polyurethane, nanocomposite, dielectric properties

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Data Availability: All relevant data are within the paper and its supporting information files.

#### INTRODUCTION

In particular, capacitors are increasingly required to store a large amount of energy then supply it instantly. These applications require ever-higher power densities along with higher capacity levels over time. This is ideal for a variety of commercial and military applications. The need for dielectric materials as energy storage systems has resulted in the production of inorganic composed of polymer materials. Inorganic materials help to increase the effective dielectric constant of the composite system without compromising the high inherent breakdown strength of polymers.

It was proposed that the properties of the interface between polymer-inorganic nanoparticles will dominate than the bulk properties of the constituents<sup>1,2</sup>. This concept goes beyond the improved dispersion of inorganic dielectric nanoparticles being enhanced by the high surface area of the inorganic nanoparticles.

Due to their unique properties and applications, nanocomposite materials have received great attention in recent years<sup>3-7</sup>. Nanocomposite materials have a high surface area to volume ratio, large length to diameter ratio, reciprocal properties and light weight have made them ideal for a variety of applications. They are widely used for filtration<sup>4</sup>, drug delivery<sup>5</sup> sensors<sup>6</sup>, tissue engineering<sup>7</sup> and protective clothing against chemical and biological hazards<sup>8</sup>, food processing and microelectronics9. There are several methods used to produce nanocomposites, such as self-assembly<sup>10</sup>, drawing<sup>11</sup>, phase separation<sup>12</sup> and electrospinning<sup>13</sup>. Among these techniques "in situ" formation is more versatile due to its simple assembly and ease of production of inorganic/organic nanocomposite, this is accomplished by swelling organic material in the desired inorganic phases to give inorganic good dispersion inside the organic phase<sup>14</sup>. A hybrid material of inorganic/organic can also be synthesized through the *in situ* incorporation of inorganic species inside a polymer matrix.

In particular, the inorganic material is ions with a size of a nanometer, with a suitable solution that can be diffused inside polymers. Here in this article gamma irradiation is used for initiation polymerization reactions of PVA/PU in hybrid with  $TiO_2@Ag$ . Gamma irradiation is a promising technique in the preparation of different advanced materials<sup>15,16</sup>.

There are several properties that can be imparted to inorganic material from organic material and vice versa. Generally, organic material can impart flexibility, hydrophilic character and ambient processing conditions to the coating. Inorganic materials usually exhibit high thermal stability, good electrical properties and high mechanical properties. Special interest has been given to the TiO<sub>2</sub> nanoparticles as a photocatalytic film for wastewater treatment and air purification<sup>17</sup>. Titanium dioxide (TiO<sub>2</sub>) is having semiconductor characterization like high refractive index, band gap, dielectric properties and optical transmittance. Doping silver into TiO<sub>2</sub> increased the photocatalytic activity of TiO<sub>2</sub> by enhancement the rates of interfacial electron transfer lead to extending photoresponse of TiO<sub>2</sub> into the visible range<sup>18,19</sup>. TiO<sub>2</sub>/polymer nanocomposites have a special property such as the ability to absorb intensively UV radiation and visible light transparency<sup>20</sup>. The inorganic/organic hybrid composite can be used in optical and cosmetic fields as well as food packaging material<sup>21</sup>.

Poly Vinyl Alcohol (PVA) has good chemical resistance and is can forming a transparent film with high mechanical properties and biocompatibility<sup>22</sup>. In order to further improve the properties of this polymer, the introduction of an inorganic material is useful for the creation of a composite that could be lightweight, elastic and has strong molding capability. Polyurethane (PU) is one of the most studied polymers for nano / micro composites and diblock copolymers/blends due to these characteristics. Polyurethane (PU) is an amorphous polymer with high thermal stability and high-strength. This article begins designing polymer/inorganic nanocomposite dielectrics in the hopes of finding improved properties for energy storage applications.

#### **MATERIALS AND METHODS**

**Materials:** This study carried out between May and Sep. of 2019.  $TiO_2$  nanoparticles (NPs) were purchased from the Sigma-Aldrich Corporation (Louis, MO, USA), with a dispersion concentration of 50.1 wt % and the size of particles was 50 nm. Silver nitrate AgNO<sub>3</sub> purchased from the Sigma-Aldrich Corporation (Louis, MO, USA). Polyvinyl alcohol (MW: 85,000-124,000, 87-89% hydrolyzed) was purchased from the Sigma-Aldrich Corporation (Louis, MO, USA). MDI is commercial.

**Methods:** The preparation part carried out in National Center for Radiation Research and Technology (NCRRT), Atomic Energy Authority, P.O. Box. 29, Nasr City, Cairo, Egypt and the characterization of dielectric properties was carried out in the Faculty of Science and Humanities- Jubail, Imam Abdulrahman Bin Faisal University, Jubail, Saudi Arabia. This study carried out between May and Sep. of 2019.

About 20 mL of PVA 10% by weight was dissolved in AgNO<sub>3</sub> (0.1 mM) solution of de-ionized  $H_2O$  at 70 °C and stirred for 12 h. powder of TiO<sub>2</sub> 2.5 wt % was added in the solution of

PVA/AgNO<sub>3</sub>. Nanoparticles' suspensions of TiO<sub>2</sub> and AgNO<sub>3</sub> were embedded in that PVA solution to fabricate the PVA/TiO<sub>2</sub>-AgNO<sub>3</sub>. For the fabrication of PVA/PU-TiO<sub>2</sub>-AgNO<sub>3</sub>, the 30 mL of MDI was added dropwise into the PVA/TiO<sub>2</sub>-AgNO<sub>3</sub> mixture solution by stirring for 1 h and similarly to fabricate PVA/PU-TiO<sub>2</sub>-AgNO<sub>3</sub>. This mixture was subject to 20 kGy of gamma irradiation. The color of powder turned to pink color. The resultant (TiO<sub>2</sub>@Ag/PVA-PU) nanocomposite washing several times with ethanol solvent.

**Characterizations:** The morphology of the (TiO<sub>2</sub>@Ag/PVA-PU) was investigated by transmission electron microscopy (TEM) (JSM-5300, JEOL Ltd., Tokyo, Japan) accelerated with a voltage of 12 kV. The chemical structure was studied by FT-IR (IR Prestige-21 by Shimadzu, Nagano, Japan), Wide-angle X-ray diffractions (WAXD) spectra were performed for the evaluation of the crystal structure at 25°C with (TiO<sub>2</sub>@Ag/PVA-PU) samples using a Rotaflex RT300 mA (Rigaku manufacturer, Osaka, Japan) and Nickel-filtered Cu. Ka radiation was used for measurements, along with an angular angle of  $4 \le 2\theta \le 90^\circ$ .

#### **RESULTS AND DISCUSSION**

Chemical and crystal structure of (TiO<sub>2</sub>@Ag/PVA-PU) nanocomposite: As shown in Fig. 1a, ATR-FTIR spectra is performed to investigate the chemical structure of (TiO<sub>2</sub>@Ag/ PVA-PU). The chemical interactions of functional groups between PVA and Ag and TiO<sub>2</sub> nanoparticles have been shown in Fig. 1a. The PVA show their characteristic FTIR peaks at 3276, 2917, 1410, 1117 and 869 cm<sup>-1</sup> corresponding to the vibrations of -OH, -CH<sub>2</sub>, -CH, C-C and C-30 functional groups of PVA, respectively<sup>23</sup>. The FTIR peaks of PVA show little shift compare to their individual FTIR spectrum as reported from previous literatures due to the two metal and metal oxide of the Ag and TiO<sub>2</sub> have binding with O atom of PVA. The peaks at 2278 cm<sup>-1</sup> are assigned to stretching of the isocyanate group N=C=O for polyurethane (MDI unreact). The stretching peak of Ti-O-C located at 445 cm<sup>-1</sup> and the peak at 521 cm<sup>-1</sup> corresponded to the Ti-O-Ti vibration. The peak at the 590 cm<sup>-1</sup> indicates the vibration frequency of Aq-O ionic bond groups. Therefore, these result confirmed that the composite of (TiO<sub>2</sub>@Ag/PVA-PU) are formed.

The X-ray diffraction (XRD) spectrum in Fig. 1b shows the characteristic XRD pattern of  $Ag_2O$  and  $Ag^0$  NP according to [JCPDS card No. 76-1393] and [JCPDS file No. 5-2872], respectively. According to [JCPDS card No. 76-1393] the XRD peaks observed at the diffraction angle (20) 26.5°, 32.75°, 37.9°, 54.8° and 65.3° correspond to 110, 111, 200, 220 and 311 set of lattice planes (cubic structure), respectively for





Fig. 1(a-b): (a) FTIR spectrum and (b) XRD pattern of (TiO<sub>2</sub>@Ag/PVA-PU), respectively

Ag<sub>2</sub>O. According to [JCPDS file No. 5-2872] the diffraction peaks at  $2\theta = 38.118$ , 44.304, 64.450, 77.407 and 81.550. They represented the diffraction peaks of five crystal faces of pure AgNPs at 111, 200, 220, 311 and 222, respectively.

The obtained XRD peaks show the diffraction peaks of  $Ag_2O$  are located at  $2\theta = 26.56^\circ$ ,  $32.06^\circ$  and  $54.60^\circ$  correspond to 110, 111 and 220, respectively.

In addition, the obtained XRD peaks show the diffraction peaks of Ag<sup>0</sup> NPs are located at a diffraction angle  $(2\theta) = 38.74^\circ$ , 64.27° and 77.36° correspond to (111), (220) and (311), respectively. The slight shift in the diffraction peak for both Ag<sup>0</sup> and AgO<sub>2</sub> (decrease or increase) indicates a structural change<sup>24,25</sup>. As seen the shift is more for Ag<sub>2</sub>O might due to the formation of a bond between Ag and O atoms from TiO<sub>2</sub> or PVA molecules.

Figure 1b shows XRD patterns exhibited strong diffraction peaks at  $27.56^{\circ}$ , indicating TiO<sub>2</sub> in the rutile phase and



Fig. 2: TEM images of (TiO<sub>2</sub>@Ag/PVA-PU)



Fig. 3: Dielectric constant versus temperature of (TiO<sub>2</sub>@Ag/PVA-PU)

diffraction peaks at 25.03°, 37.77 and 47.96° indicating TiO<sub>2</sub> in the anatase phase. All XRD for TiO<sub>2</sub> peaks are not good agreement with the standard spectrum; JCPDS no.: 88-1175 and 84-1286<sup>26</sup>. This could due to the change of crystal structure due to binding between TiO<sub>2</sub> and PVA molecules that have XRD peak at 19.25<sup>27</sup> also is could for PU molecules<sup>28</sup>.

#### Morphology analysis of (TiO2@Ag/PVA-PU) nanocomposite:

The TEM image in Fig. 2 shown that the Ag@TiO<sub>2</sub> is nanoparticles composite. The dark spots in Fig. 2 show AgNPs on the TiO<sub>2</sub> surface. Anchoring AgNPs to the TiO<sub>2</sub> surface could produce the nanocomposites. And some of the TiO<sub>2</sub> nanoparticles have been transformed into nanorod. The morphological change in TiO<sub>2</sub> from spherical to a rod shape in the case of Ag@TiO<sub>2</sub> nanocomposites. The morphology of pure

TiO<sub>2</sub> was partially changed from spherical to nanorod during nanocomposite synthesis (Fig. 2). This was due to the effect of AgNPs influencing TiO<sub>2</sub> morphology. Similar morphological changes in TiO<sub>2</sub> have been reported<sup>29,30</sup>. The size of AgNPs on the TiO<sub>2</sub> surface is in the range of 30-45 nm, whereas the size of nanorod TiO<sub>2</sub> is (6×50) nm.

**Temperature-dependent dielectric properties for** (**TiO<sub>2</sub>@Ag/PVA-PU):** The dielectric behavior of the prepared (TiO<sub>2</sub>@Ag/PVA-PU) nanocomposite was investigated at different temperatures and frequency.

The dielectric constant ( $\epsilon'$ ) was determined using the capacitance values<sup>31</sup>:

$$\varepsilon' = \frac{C \cdot d}{\varepsilon_0 A} \tag{1}$$

where, C is the capacitance of the sample,  $\varepsilon_0$  is the permittivity of free space =  $8.85 \times 10^{-12}$  F/m, A and d are the cross-sectional area and the thickness of the sample.

The dielectric loss ( $\epsilon''$ ) was determined from the long tangent, tan ( $\delta$ ), by the relation:

$$\tan \delta = \frac{\varepsilon''}{\varepsilon'} \tag{2}$$

It was found that the phase angle gives a negative value, which indicates that  $(TiO_2@Ag/PVA-PU)$  nanocomposite is capacitive and can be represented by a connected RC network in a series connection<sup>32</sup>. The temperature dependence of dielectric constant and dielectric loss at various frequencies is presented in Fig. 3 and 4, respectively. The dielectric constant



Fig. 4: Dielectric loss versus temperature of (TiO<sub>2</sub>@Ag/PVA-PU)



Fig. 5: The loss tangent, tan ( $\delta$ ), vs. frequency (TiO<sub>2</sub>@Ag / PVA-PU) at different temperature

values are increased by increasing both temperature and frequency. This increase in  $\varepsilon'$  values with increasing temperature indicating that the dipoles in the polymer matrix are thermally activated and have more rotational deliverance by heating. The  $\varepsilon'$  value is dramatically increased in the hightemperature region, this is due to the effect of space charge and interfacial polarization, which leads to an increase of the total polarization values. Whereas the increase in  $\varepsilon'$  values with increasing frequency indicates that the friction against the dipole's motion was constrained by the rearrangement of molecules inside the polymer matrix. This means that dipole's motion is faster than the rearrangement scale of molecules. The ε' value obtained of the (TiO<sub>2</sub>@Ag/PVA-PU) nanocomposite (from 20 to 80°C) is high compared to the dielectric constant values reported in the literature on pure PVA, PVA- InTiO, PVA-Al<sub>2</sub>O<sub>3</sub> thin films and PVP-TiO<sub>2</sub>

nanocomposites<sup>33,34</sup>. This high value of  $\varepsilon'$  supports the possibility to use it in different device applications as a dielectric layer.

The temperature dependence of  $\varepsilon$ " at various frequencies is shown in Figure 4. The  $\varepsilon$ " values are increased by increasing the temperature. This increase in  $\varepsilon$ " value with the increase of temperature is only proof that the loss mechanism is thermally activated and this is due to the increase in the number of charge carriers and the rotation of the dipole (i.e. the relaxation time decreases) with increasing the temperature<sup>35</sup>.

The electrical modulus formalism was invoked to further analyze the dielectric data<sup>36</sup>. Using the electrical modulus approach helps to gain insight into the bulk response of (TiO<sub>2</sub>@Ag/PVA-PU). This approach could, therefore, be used effectively to separate the effects of electrodes and also to help circumvent the problems caused by electrical conduction that could mask the dielectric relaxation processes. The frequency dependence of the imaginary part of complex electric modulus, M for the (TiO<sub>2</sub>@Ag/PVA-PU) nanocomposite is temperature dependence is described in Fig. 5.

The real and imaginary of modulus were calculated from the relations:

$$M' = \frac{\varepsilon^{1^2}}{\varepsilon^{1^2} + \varepsilon^{1^2}}$$
(3)

$$M'' = \frac{\varepsilon''^2}{\varepsilon'^2 + \varepsilon''^2}$$
(4)

The electrical conductivity of a (TiO<sub>2</sub>@Ag / PVA-PU) nanocomposite is improved by increasing the mobility of charge carriers and increasing their concentration. It is possible to have information for both of these two factors by studying the relationship between loss tangents, tan  $(\delta)$ , versus frequency. Figure 5 reveals the relation between tan  $(\delta)$ and the frequency at different temperatures. The area under the peak gives good information about the number of charge carriers involved in a relaxation process. As the area under the peak increase, this means that the mobility of charge carriers is increased. It is clear from Fig. 5 that the intensity of the peak increases with increasing temperature and shifted to a lower frequency. The increase of temperature leads to enhancement in the amorphosity of the polymer chains and consequently, decreases the relaxation time which results in the peak shift towards a lower frequency<sup>37</sup>. The dielectric modulus behavior is a good approach to find an explanation

Table 1: The relaxation time at different temperature

Temperature (K)	τ (µsec)
303	316
333	218
363	141
393	63

for the dielectric relaxation behavior of the electrode that is caused by the suppression of polarization. The relations M" versus frequency at different between M' and temperature ranges were presented in Fig. 6 and 7, respectively. The analysis of the modulus data and its variation with frequency gives a well-defined peak. In Figure 6, the peak shifts towards a lower frequency region and its intensity decrease with increasing the temperature. It is also noticeable the increase peak broadening by increasing the temperature. The presence of the peak in the M" vs. frequency relation (Fig. 7) and its shift towards a higher frequency indicate that the conduction mechanism of the prepared polymer electrolyte films is ionic. While the peak height and shift with an increase in temperature indicating a pluralism of the relaxation process<sup>38</sup>.

The relaxation time was calculated from  $f_{max}$  position and the results were shown in Table 1. It is clear from Table 1 that *t* decreases with increasing temperature; this means the ionic mobility increase with increasing temperature. The temperature reduces the relaxation time and this leads to a rapid response of the polar dipoles to the applied external field, thus increasing the electrical conductivity value by increasing the temperature.

lonic conductivity of (TiO<sub>2</sub>@Ag / PVA-PU): Figure 8 illustrates the relation between electrical conductivity (s<sub>ac</sub>) and the frequency at different temperatures. From Fig. 8 it is noticed that the  $s_{ac}$  value of the prepared composite is low compared to its value at high frequency. This is due to the space charge polarization or interfacial impedance resulting from the accumulation of charges at the electrode<sup>39</sup>. At high frequency, the mobility of the ions increases and therefore increases the value of the electrical conductivity. It is also clear from the figure that the  $s_{ac}$  value is highly dependent on temperature. Figure 9 shows the relation between s<sub>ac</sub> value and the temperature in the temperature range from 303 K up to 393 K at a different frequency. It is clear that s<sub>ac</sub> increases linearly on increasing temperature due to the increase of the ion motilities and charge carriers density as well as the dipolar relaxation.



Fig. 6: The real part of modulus (M') vs. frequency (TiO<sub>2</sub>@Ag / PVA-PU) at different temperatures



Fig. 7: The imaginary part of modulus (M") vs. frequency (TiO<sub>2</sub>@Ag / PVA-PU) at different temperatures



Fig. 8: Frequency-dependent conductivity at various temperatures for (TiO<sub>2</sub>@Ag / PVA-PU)



Fig. 9: Temperature-dependent conductivity at various Frequency for (TiO<sub>2</sub>@Ag / PVA-PU)

The thermal activation energy (Ea) was calculated at a different frequency according to the relation:

$$\sigma = \sigma_{o} \exp(-E_{a} / kT)$$
 (5)

where, k is the Boltzmann's constant,  $\sigma_o$  is pre-exponential factor, Ea is activation energy and T is absolute temperature The calculated values of Ea which determined from the slope of the curves in Fig. 9.

The activation energy (Ea) for (TiO<sub>2</sub>@Ag / PVA-PU) is low due to the amorphous nature of the PVA polymer that facilitates the motions of the fast ions in the polymer matrix. The amorphous nature also provides a bigger free volume in the polymer electrolyte system with an increase in temperature<sup>40</sup>.

#### CONCLUSION

Herein, (TiO<sub>2</sub>@Ag-PVA/PU) was successfully synthesized by *in situ* polymerization induced by gamma irradiation. The binding formation between TiO<sub>2</sub> and Ag with PVA was confirmed by FTIR and XRD instrument. Based on the characterization TEM results, it was concluded that Ag NPs have good dispersion onto the surface of TiO<sub>2</sub> particles. The morphology of pure TiO<sub>2</sub> was partially changed from spherical to nanorod during nanocomposite synthesis. AC conductivity increases and the activation energy for (TiO<sub>2</sub>@Ag / PVA-PU) is increased with increasing frequency. The dielectric constant values are increased by increasing both temperature and frequency. This increase in  $\varepsilon'$  values with increasing temperature indicating that the dipoles in the polymer matrix are thermally activated and have more rotational deliverance by heating. The  $\varepsilon'$  value is dramatically increased in the hightemperature region, this is due to the effect of space charge and interfacial polarization, which leads to an increase of the total polarization values.

#### SIGNIFICANCE STATEMENT

This study discovers the giant dielectric (TiO<sub>2</sub>@Ag / PVA-PU) nanocomposite materials that can be beneficial for energy storage. This study will help the researcher to prepare polymers serving as the matrix and inorganic material used to increase the effective dielectric constant. The interface polymer inorganic plays a critical role that many researchers were not able to explore in defining the properties of nanocomposites. As a result of nanoparticles of Ag@TiO<sub>2</sub> addition to a polymer of PVA-PU, a reduction in the activation energy due to the decrease in particle size is observed. This is due to the effect of space charge and interfacial polarization, which leads to an increase in the total polarization values.

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