



Dielectric Properties of Eco-Friendly Zinc Oxide NPs Dispersed in Poly (Methyl Methacrylate) Matrix Films



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Abstract

Applying the green method in this study to synthesize the nanoparticles of zinc oxide (ZnO NPs) from reacting pomegranate peel extract with aqueous zinc nitrate in an environmentally friendly aqueous solution. The ZnO nanoparticles were characterized using various techniques: FTIR, XRD, EDX, and SEM. The results showed that the prepared particles had high purity and their sizes ranged from 24 to 44 nm, which confirmed the successful synthesis and purity of the composite produced. In order to study the electrical properties of the resulting film, the ZnO nanoparticles were embedded in polymethyl methacrylate (PMMA). Both dielectric constants, the real and the imaginary, recorded the highest values when frequencies were low and then began to gradually decrease with increasing frequency. In contrast, when the frequency is at its lowest value, the conductivity is at its lowest value but increases gradually with the frequency increase.

1. Introduction:

Due to the phenomenal properties of Zinc oxide (ZnO) nanoparticles, they are currently the most common and widely used metal oxides, leading to the development of an important research area for a wide variety of applications. ZnO nanoparticles can be synthesized using different methods. These techniques include chemical methods like solvent precipitation and sol-gel, as well as physical methods such as laser ablation, condensation, and evaporation. Additionally, it may include contemporary biological methods that utilize eco-friendly ingredients such as plant extracts; methods that reduce the production of hazardous byproducts; or methods that use water

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as a solvent instead of harmful solvents that break down into undesirable products. However, chemical techniques are less frequently favored because of the health and environmental hazards they pose from solvents, chemical reactions, as well as the associated harmful byproducts like toxic fumes and harmful compounds. Conversely, physical techniques have numerous challenges that limit their widespread use and availability, like high costs and harsh and specific operating conditions, such as high pressures and temperatures. [1].

As a result, green biosynthesis has received more attention as a straightforward and efficient economic substitute. It is ecologically safe and has a variety of antibacterial qualities that are superior to those produced by straightforward, conventional physical and chemical techniques. This kind of synthesis can generate pure nanomaterials in large quantities, without the need for complex equipment or high technical skill, as it focuses on the use of easy-to-prepare plant extracts depending on the kind of process employed [2].

One of the most commonly used extracts for producing a range of nano oxides, like zinc oxide, is pomegranate peel extract. This is because of its high concentration of various and potent plant components, like flavonoids and phenols, which serve as reducing agents, stabilize nanoparticles at specific boundaries, which are crucial in the preparation of nanocomposites, and prevent their agglomeration. These compounds convert metal ions (such as Zn^{2+}) into solid nanoparticles (ZnO) in a natural, safe, and environmentally friendly way. Additionally, they aid in preventing agglomeration and stabilizing these particles at nanoscale dimensions, thereby preserving their small size and uniform shape. This method is part of green synthesis, which is environmentally friendly and cost-effective compared to traditional chemical methods [3].

However, zinc oxide (ZnO) is a semiconductor metal oxide that has good insulating qualities, which makes it a material that shows promise for a range of photovoltaic and electronic applications. At room temperature, ZnO 's moderate dielectric constant, which varies depending on frequency, temperature, particle size, and manufacturing technique, usually ranges from 8 to 10. Both the dielectric loss and the dielectric constant decrease with increasing frequency, demonstrating frequency-dependent behavior in its dielectric characteristics. This is because slow polarizations like vacuum polarization (which leads to the appearance and disappearance of electron-positron pairs and a weak electric field) have less of an impact. High temperatures also raise the dielectric constant and improve dipole mobility. Through structural alteration or elemental additions like aluminum or gallium, ZnO nanostructures demonstrate highly adjustable dielectric characteristics, increasing their effectiveness in applications including sensors, capacitors, resonators, and piezoelectric devices [4, 5].

Vacuum Zinc oxide nanoparticles can be placed in a polymer matrix to create a composite that resembles a thin film, which allows for an exact analysis of the dielectric characteristics of zinc oxide. This makes it possible to quantify the composite's AC conductivity, imaginary dielectric loss, and dielectric constant at different frequencies and temperatures. By acting as a backing matrix, the polymer helps prevent agglomeration by facilitating the even distribution of zinc oxide particles and improving the consistency of the electrical properties. By changing the polarity of the material and the interaction between the zinc oxide particles and the charge carrier mobility within the composite. The dielectric properties are also affected by the polymer. The development of high-performance composite materials for use in capacitors, sensors, and advanced electronic gadgets are produced made possible by studying these traits, which helps to elucidate the principles of electrical transport and the impact of nanoparticles on dielectric behavior. Additionally, this method makes it possible to study how the size and concentration of zinc oxide particles affect dielectric characteristics, which aids in the development of composite materials with enhanced electrical

performance [6, 7].

Hong et al. created zinc oxide (ZnO) nanocomposites using low-density polyethylene (LDPE), in which the ZnO particles were either uniformly distributed throughout the composite or in a tightly regulated heterogeneous arrangement within the composite. The dielectric constant of these composites was measured at filler concentrations of up to 40% by volume, and contrasted to that of similar composites that contained ZnO particles smaller than a micrometer in size. In order to take advantage of the large surface area of the nanoparticles, which facilitated the incorporation of highly polar molecules into the composite, the filler particles were enclosed by silane cross-linking agents and mixed with LDPE. Investigations into the impact of filler particle distribution on the dielectric constant revealed that the two types of interfaces inside the composite—the first between the contacting ZnO particles and the second between the LDPE and the ZnO particles—were responsible for the observed variations [8].

Rashmi and colleagues conducted a study to examine the impact of zinc oxide nanoparticles on the electrical and structural properties of polyvinyl alcohol (PVA) films. The resulting nanocomposites were evaluated using scanning electron microscopy (SEM), infrared spectroscopy, UV-Vis absorption, and X-ray diffraction. The UV-Vis spectra indicated that the addition of zinc oxide nanoparticles did not change the composites' absorption in the visible range. The SEM images also revealed that the nanoparticles were uniformly dispersed throughout the polymer matrix. It was found that the dielectric properties were significantly influenced by frequency and the concentration of nanofillers. The alternating conductivity (σ_{ac}) of the PVA composites grew with increasing frequency, while the dissipation coefficient ($\tan \delta$) rose with the addition of nanoparticles and decreased with increasing frequency. With moderate nanofiller levels, the composites acted like nearly lossless substances at high frequencies, making them suitable for microwave use [9].

A research team led by Hamed, S.T., successfully synthesized zinc oxide nanoparticles (ZnO NPs) with sizes ranging from 20 to 80 nm via the sol-gel method. Using the solution casting process, these particles were placed onto a polymeric blend of carboxymethyl cellulose (CMC) and polyethylene oxide (PEO) in a 70:30 ratio with varying weight proportions ranging from 0 to 6%. As the ZnO particle content increased from 0 to 6%, the resulting composite significantly increased in the refractive index from 1.79 to 1.97, and a significant decrease in the energy gap from 4.51 to 3.34 eV. Alternating conductivity (σ_{ac}) measurements at 298 K confirmed a significant improvement in the conductivity and dielectric coefficients with increasing ZnO particle concentration, reaching maximum values at 2%. The effect of temperature on the conductivity and dielectric coefficients at this optimum concentration was also studied, revealing unique properties of this material that make it suitable for use in solid-state

supercapacitors [10].

In this work, nano zinc oxide was prepared by the green method using pomegranate peel extract, then the prepared oxide was characterized using different techniques: FTIR, XRD, EDX, and SEM. Then, zinc oxide films were prepared with polymethyl methacrylate to study the real and imaginary dielectric constant and electrical conductivity.

2. Materials and Methods:

2.1 Chemicals:

The experiment involved the use of chemicals and solvents from different suppliers, such as Sigma-Aldrich, Riedel-de Haën, and SDH. The materials were used in this research as obtained without any prior purification process or modification. Peels from pomegranates were also gathered for the experiment. Based on information from a farmer northwest of Diyala Governorate, the experimental research was carried out in compliance with national and international norms, organizations, and laws pertinent to plant material research.

2.2 Devices:

The produced zinc oxide nanoparticles were characterized using FTIR, XRD, EDX, and SEM methods. A Perkin-Elmer FTIR-65 infrared spectrometer was used for FTIR testing. Utilizing an X-ray detector (Philips/PW1730, Cu, $K\alpha = 1.5406$), the composition of the sample was examined. Additionally, the material's surface characteristics were examined using a TESCAN MAIA3 scanning electron microscope (SEM).

2.3 Procedures:

2.3.1 Preparation of pomegranate peel extract:

Pomegranates, the plant used in this study, were purchased from a farm in the Diyala Governorate. Peels from pomegranates were gathered, washed with purified water, and allowed to dry at room temperature with adequate ventilation in the shade. After being partially broken into small pieces with an electric grinder, the peels were kept dry and out of the way in a paper bag until they were ready to use. Using a delicate electronic balance, 25 grams of pomegranate peels were used for the extraction procedure. To extract the bioactive components, the peels were cooked to 60–70°C for 90 minutes on a hot plate while being gently stirred, all while being contained in a conical flask with 300 ml of deionized water. After repeatedly filtering the solid waste using a cotton swab, centrifugation was used to extract it. To get a filtered solution with the extracted chemicals and no contaminants, this procedure was carried out three times. After measurement, the solution's pH was determined to be 4.6.

2.3.2 Preparation of Zinc oxide:

Zinc oxide nanoparticles (ZnO NPs) were prepared using 10 grams of zinc hexahydrate ($Zn(NO_3)_2 \cdot 6H_2O$) weighed accurately using a sensitive balance, and dissolved in 200 ml of

deionized water in a beaker. The solution was heated on a hot plate at 60°C with gentle stirring for 1 hour. Pomegranate peel extract was then slowly and gradually added using a pyrite until complete addition. Stirring was continued at the same temperature for another 1 hour. The color of the solution changed from yellow to dark green, indicating successful synthesis of ZnO nanoparticles. After the reaction was completed, the solution was allowed to cool to room temperature for 20 minutes, and the mixture was filtered to collect the resulting precipitate, which was subsequently dried in an oven at 150°C. After drying, the sample was placed in a ceramic crucible and heat-treated (such as annealing) in a furnace at 500°C for four hours, resulting in pure white powder of zinc oxide nanoparticles, confirming the success of the synthesis process.

2.3.3 Preparation of Polymer Films:

2 grams of poly (methyl methacrylate) (PMMA) are dissolved in 50 mL of tetrahydrofuran (THF) with continuous stirring until a completely homogeneous solution is formed. Then, 0.5 grams of ZnO NPs is weighed and placed in a sealed glass tube. Then, 10 mL of the previously prepared PMMA solution is added to the weighted ZnO. The mixture is stirred well and then treated in an ultrasonic bath for 15 minutes to ensure complete mixing and even distribution of the particles in the polymer solution. Finally, the homogeneous mixture is poured into a glass mold and left until the solvent evaporates completely, forming ZnO/PMMA nanocomposite film.

3. Results and Discussions:

3.1 FTIR of Zinc Oxide Nanoparticles:

The FTIR spectra of ZnO NPs within the 400–4000 cm^{-1} range following the calcination process is shown in Figure 1. The two bands in the picture, which are centered at 3455 and 1624 cm^{-1} , correspond to the stretching and bending vibrations of the O-H group, respectively, caused by the presence of moisture between zinc oxide particles and the plant's absorbed OH groups on the ZnO NPs' surface. The Zn-O group in zinc oxide is represented by the band centered at 436 cm^{-1} , which is in good accord with other previously published findings [11].

3.2 X-ray Diffraction (XRD) of Zinc Oxide Nanoparticles:

The XRD analysis of ZnO nanoparticles (illustrated in Figure 2) reveals several sharp and well-defined peaks, indicating high purity and crystallinity. These peaks are observed in the 2 region at 31.7189°, 34.3224°, 36.2830°, 47.6227°, 54.1320°, 56.5642°, 59.9838°, 62.9247°, 66.4878°, 68.0870°, 69.1588°, 72.7933°, and 77.1045°, corresponding to the crystallographic planes (100), (002), (101), (102), (110), (103), (200), (112), and (201), respectively. Several distinct and sharp peaks are visible in the XRD examination of ZnO nanoparticles, as

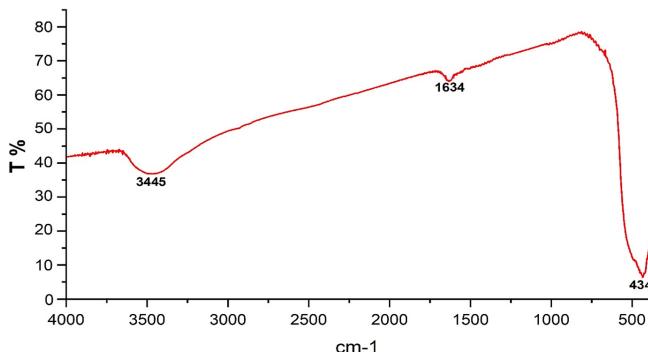


Figure 1. FTIR of ZnO NPs.

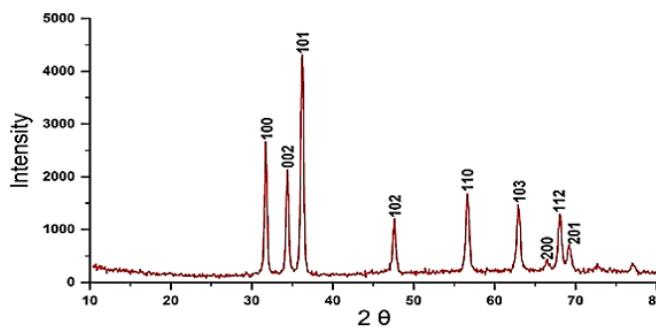


Figure 2. XRD of ZnO NPs.

shown in Figure 2, suggesting great purity and crystallinity. The crystallographic planes (100), (002), (101), (102), (110), (103), (200), (112), and (201) correspond to these peaks, which are located in the 2 region at 31.71°, 34.32°, 36.28°, 47.62°, 54.13°, 56.56°, 59.98°, 62.92°, 66.48°, 68.08°, 69.15°, 72.79°, and 77.10°, respectively. These results validate the effective production of pure ZnO nanoparticles and are in line with prior research in the literature (JCPDS Card number 36-1451). The excellent purity of the synthesized ZnO is further demonstrated by the absence of any extraneous peaks in the analyzed spectrum. The Debye-Scherer equation was used to estimate the average size of crystals[12, 13].

$$D = K\lambda / (\beta \cdot \cos\theta)$$

Where: D is the crystal size (nm), the full width at half maximum (FWHM) of the diffraction peak, the wavelength of the X-ray (1.5406), and θ is the Bragg angle. The average crystal size was found to be in the range of 13.09-47.91 nm.

3.3 Scanning Electron Microscope (SEM):

In this work, the morphology of the nanostructures was analyzed using scanning electron microscopy (SEM) to determine the microstructural differences in the samples. The round or irregularly shaped particles detected in the SEM images were zinc oxide nanoparticles. The diameters of the generated ZnO

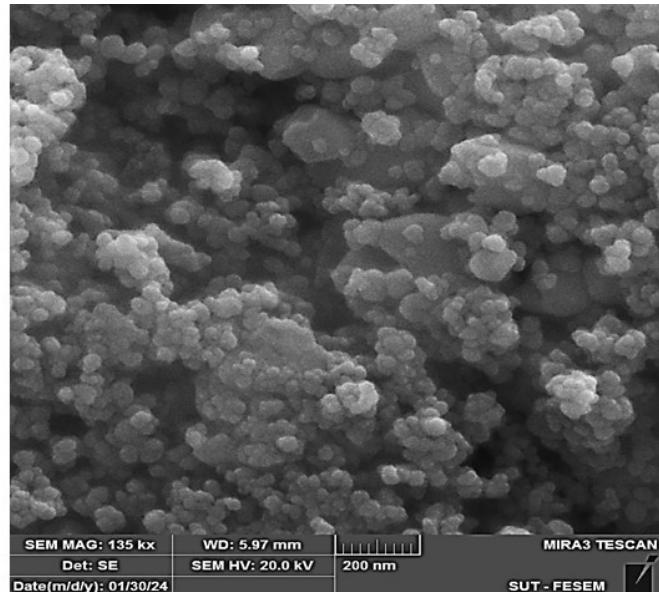


Figure 3. SEM of ZnO NPs.

nano particles were found to range from 22 to 53 nm, as shown in Figure 3.

3.4 EDX of the ZnO NPs:

The EDX analysis strongly indicates the presence of zinc in its oxide form, with high signals for both zinc and oxygen. Each element in the sample can be precisely identified using EDX, and the results show zinc and oxygen with weight percentages of 82.80% and 17.20%, respectively. The theoretical percentage values 80.339% and 19.66% zinc and oxygen with respectively, give a great convergence in the weight ratio between the theoretical and real calculations of the prepared compound. Two notable zinc peaks at 1 keV and 8.6 keV, as well as a unique oxygen peak at 0.5 keV, are visible in the EDX spectrum. The distinctive peaks of zinc and oxygen validate the elemental makeup of the ZnO nanoparticles that were produced. Furthermore, Figure 4 provides additional evidence of the purity and composition of the sample by showing the presence of trace elements, such as gold, alongside zinc and oxygen [14, 15].

3.5 Dielectric properties:

The electrical properties of the polymer nanocomposites were measured, including the real and imaginary parts of the dielectric permittivity, as well as the electrical conductivity, using an LCR meter. The measurements were conducted at various frequencies ranging from 20 kHz to 1000 kHz at room temperature [16].

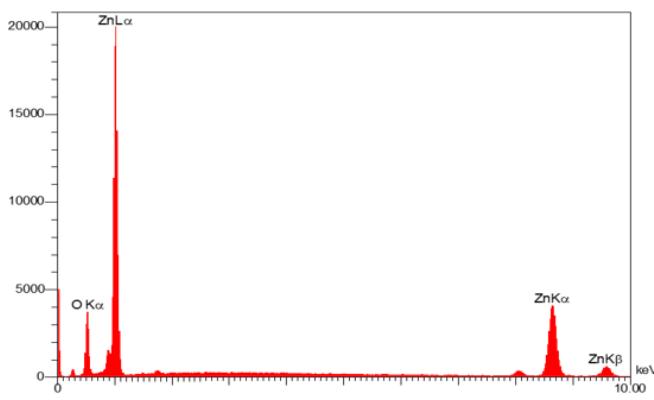


Figure 4. EDX of ZnO NPs

1. Real Permittivity (ϵ'):

The real part of the dielectric permittivity (ϵ') represents the material's ability to store electrical energy when exposed to an electric field. It is calculated using the following equation:

$$\epsilon' = \frac{C \cdot d}{\epsilon_0 \cdot A}$$

Where:

- ϵ' : Real dielectric permittivity
- C: Capacitance
- d: Thickness of the measured film
- A: Electrode area, which is equal to 1 cm²
- ϵ_0 : Vacuum permittivity (a constant with a value of $8.85 \times 10^{-12} Fm^{-1}$)

2. Imaginary Permittivity (ϵ''):

The imaginary part of the dielectric permittivity (ϵ'') represents the amount of electrical energy that is dissipated as heat over time. High ϵ'' values indicate that the material is less efficient at storing electrical energy, which is undesirable for materials used in energy storage applications.

The ratio of imaginary to real permittivity is known as the loss tangent ($\tan \delta$), which can be calculated using the following relation:

$$\tan \delta = \frac{\epsilon''}{\epsilon'}$$

Rearranging this equation, the imaginary permittivity can be calculated as:

$$\epsilon'' = \tan \delta \cdot \epsilon'$$

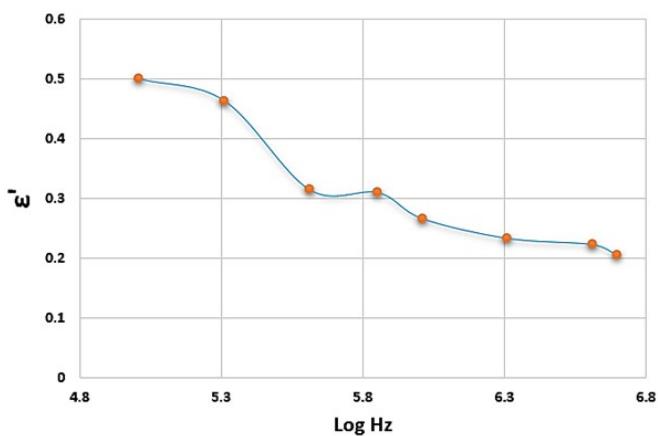


Figure 5. Relationship between the real dielectric constant and the logarithm of the frequency for ZnO/PMMA polymer nanocomposite film.

If the value of $\tan \delta$ is given as a constant symbolized by D, known as the dissipation factor or loss factor, then the equation becomes:

$$\epsilon'' = D \cdot \epsilon'$$

Where:

- ϵ'' : Imaginary dielectric permittivity
- D: Dissipation factor (loss factor)
- ϵ' : Real dielectric permittivity

Figures 5 and 6 show the relationship between the real and imaginary dielectric constants with the logarithm of frequency for ZnO/PMMA polymer film. It is clear that the real dielectric constant values for polymer composites reached their highest value at the lowest frequency, as shown in the figure, and then began to gradually decrease with increasing frequency until they reached their lowest value at high frequencies. This can be explained by the fact that at low frequencies, the field rotation is slow, providing sufficient time for the permanent and induced dipoles to align themselves in the direction of the applied field. At high frequencies, the time period is shorter than the time period needed for the molecules to align themselves in the direction of the external electric field, and therefore, the field is low [17].

The imaginary dielectric constant (ϵ'') of the ZnO/PMMA composite decreases at high frequencies due to the reduced ability of the charges to follow the changing alternating electric field. At low frequencies, there is sufficient time for ion movement and interfacial polarization (e.g., polarization at the ZnO/PMMA interface), resulting in high imaginary dielectric constant values, which are primarily related to energy losses in the material. However, as the frequency increases,

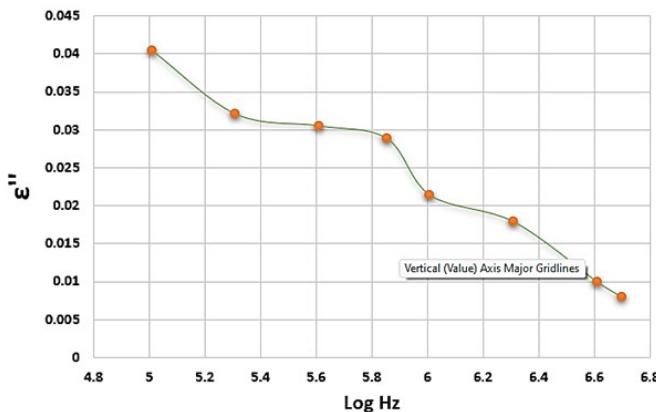


Figure 6. Relationship between imaginary dielectric constant and logarithm of frequency for ZnO/PMMA polymer nanocomposite film.

the charges or dipoles cannot reorient as rapidly as the changing field, so the polarization effect diminishes, and electrical losses decrease, resulting in a lower imaginary dielectric constant (ϵ''). This behavior reflects the reduced energy loss in the dielectric at high frequencies, making the material more efficient in high-frequency electronic applications such as thin film dielectrics and microfilters [17, 18].

4. Electrical Conductivity (σ):

Electrical conductivity can be defined as the process of transferring electric charges from one place to another in a given medium when an electric field is applied. The electrical conductivity values of the nanocomposites were studied using alternating current (AC) at different frequencies ranging between 20 kHz and 1000 kHz at room temperature. These values were calculated using the following equation:

$$\begin{aligned}\sigma_{a.a.} &= \omega \epsilon_0 \epsilon' \tan \delta \\ \omega &= 2\pi f, \epsilon'' = \tan \delta \epsilon \\ \sigma_{a.a.} &= 2\pi f \times \epsilon_0 \times \epsilon''\end{aligned}$$

Where:

σ : Electrical conductivity

ϵ_0 : Permittivity of free space (vacuum permittivity), a constant with a value of $8.85 \times 10^{-12} Fm^{-1}$

ω : Angular frequency, equal to $2\pi f$

f: Frequency used in the measurements

Conductivity is defined as the process of transfer of electrical charge from one location to another within a medium when an electric field is applied. The electrical conductivity of the polymer composites was measured, and an increase in the alternating electrical conductivity was observed with increasing frequency at room temperature, as shown in Figure 7 [18, 19]. The alternating electrical conductivity (σ) of

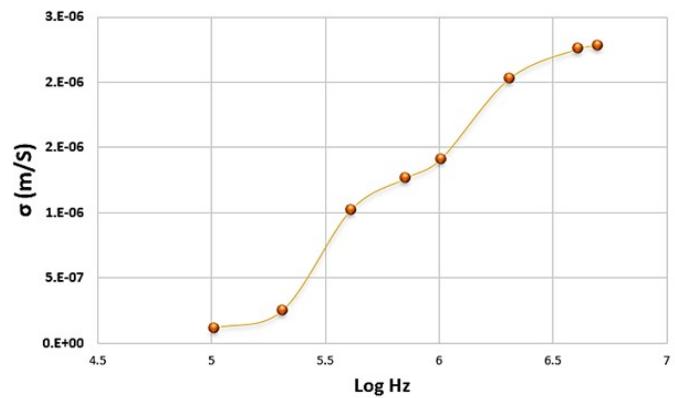


Figure 7. Relationship between electrical conductivity and logarithm of frequency for ZnO/PMMA polymer nanocomposite film.

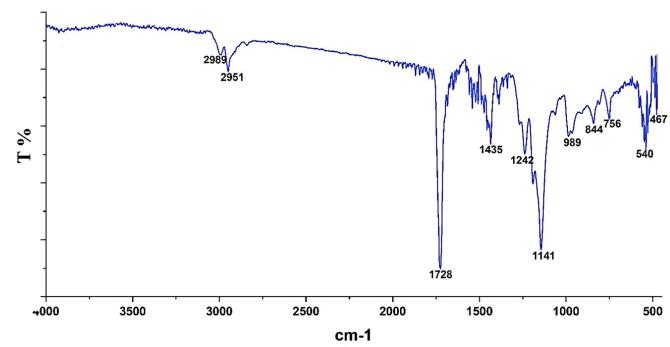


Figure 8. FTIR spectrum of the composite

ZnO/PMMA composites increases with increasing frequency due to several complex physical mechanisms, most notably the energy hopping mechanism, where zinc oxide nanoparticles allow charge transfer between non-equivalent energy sites, especially at high frequencies. The Maxwell-Wagner polarization mechanism also plays an important role, where ZnO/PMMA interfaces act as charge accumulation areas, enhancing conductivity. As frequency increases, the dielectric capacitance of the polymer material decreases, thus reducing charge transport barriers, leading to increased electrical conductivity. This behavior makes these composite nanomaterials promising for applications such as smart insulators, electronic devices, and thermoelectric sensors. This study is highly consistent with recently published articles [20, 21]. Figure 8's FTIR spectrum of the zinc oxide film distributed throughout PMMA revealed peaks at frequencies of 756, 844, and 989 cm^{-1} that were caused by the CH group's rocking motion. Additionally, a peak at 1141 cm^{-1} was noted, which was ascribed to the C–O–C bond's asymmetric stretching. At 1242 cm^{-1} , stretching vibrations of the C–C–O link in the methyl carbonyl group were seen, whereas a deformation of the C–H bond produced the peak at 1435 cm^{-1} . Furthermore,

a noticeable signal was seen at 1728 cm^{-1} , which was ascribed to the C=O carbonyl bond's stretching vibration. Also, peaks at 2951 and 2989 cm^{-1} emerged, suggesting that the C–H bond was vibrating. The presence of zinc oxide in the polymer is the cause of the bands at 467 and 540 cm^{-1} .

5. Conclusions:

The results of this study indicate that pomegranate peel extract is an important, environmentally friendly, and effective agent for preparing zinc oxide nanoparticles (ZnO NPs) with high purity and sizes ranging from 24 to 44 nm . Incorporating these particles with polymethyl methacrylate (PMMA) to form a ZnO/PMMA film showed a significant improvement in the electrical properties of the resulting film, as the real and imaginary dielectric constants reached their highest values at low frequencies, while the AC electrical conductivity gradually increased with increasing frequency, demonstrating the efficiency of the prepared materials in various electronic applications.

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Data Availability Statement: All of the data supporting the findings of the presented study are available from corresponding author on request.

Declarations:

Conflict of interest: The author declare that they have no conflict of interest.

Ethical approval: This research did not include any human subjects or animals, and as such, it was not necessary to obtain ethical approval.

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الخصائص العازلة لجسيمات أكسيد الزنك النانوية الصديقة للبيئة، والمدجحة في أغشية مصفوفة بولي (ميثيل ميثاكريلات).

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الخلاصة

في هذه الدراسة، تم تحضير جسيمات نانوية من أوكسيد الزنك ($ZnONPs$) باستخدام مستخلص قشر الرمان المحلي بالطريقة المضطربة، عن طريق تفاعل نترات الزنك المائية مع المستخلص النباتي في المحلول المائي الصديق للبيئة. تم توصيف جسيمات أوكسيد الزنك باستخدام تقنيات مختلفة $FTIR$ و XRD و $EDXSEM$. أظهرت النتائج أن الجسيمات تم تحضيرها بنقاوة عالية وبأحجام نانوية تتراوح من 24 إلى 44 نانومتر مما يؤكد نجاح التخليق ونقاء المركب الناتج. تم أيضًا درج جسيمات ZnO النانوية المحضرة ضمن بولي ميثاكريلات ($PMMA$) لدراسة الخصائص الكهربائية للفيلم الناتج. أظهرت النتائج أن الثوابت العازلة الحقيقية والتخيلية سجلت أعلى قيم لها عند الترددات المنخفضة ثم بدأت في الانخفاض تدريجياً مع زيادة التردد. في المقابل، أظهرت الموصولة الكهربائية للتيار المتردد أدنى قيمة لها عند أدنى تردد وبدأت في الزيادة تدريجياً مع زيادة التردد.

الكلمات الدالة : جسيمات أكسيد الزنك النانوية، مستخلص قشر الرمان، مركب بوليمر نانوي، طريقة التخليق الأخضر

التمويل: لا يوجد

بيان توفر البيانات: جميع البيانات الداعمة لنتائج الدراسة المقدمة يمكن طلبها من المؤلف المسؤول.

اقرارات: تضارب المصالح: يقر المؤلف انه ليس لديهم تضارب في المصالح.

الموافقة الأخلاقية: لم يتضمن هذا البحث اي تجارب على البشر او الحيوانات وبالتالي لم يكن من الضروري الحصول على الموافقة الأخلاقية.