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Research Article

THE SYNTHESIS, CHARACTERIZATION, AND ELECTRICAL PROPERTIES OF A ZNO NANOPARTICLE-PVC NANOCOMPOSITE

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ABSTRACT

The paper details the synthesis of ZnO nanoparticles using the Sol-Gel method. This method is commonly employed to produce nanoparticles with controlled Properties. The ZnO nanoparticles were characterized using X-ray diffraction (XRD). The XRD pattern revealed that these nanoparticles have a hexagonal crystalline structure. The grain size was determined to be 34.5 nanometers, indicating the size of individual crystalline domains in the nanoparticles. The SEM images showed that the ZnO nanoparticles had a porous and sponge-like appearance at different magnifications, including a 5µm scale bar. This porous structure can have implications for various applications, such as increased surface area and reactivity. The ZnO nanoparticles were incorporated into a PVC matrix. This implies the creation of a nanocomposite material, where the properties of the PVC are modified by the presence of the nanoparticles. Electrical measurements of the nanocomposite film were conducted. The paper reports that the dielectric constant of the film increases with the concentration of ZnO nanoparticles. This suggests that the presence of nanoparticles enhances the film's ability to store electric charge and modify its electrical properties. This paper also notes that the AC conductivity of the film increases with both the concentration of doping (the amount of ZnO nanoparticles added) and temperature. This suggests that the nanocomposite film becomes a better conductor under these conditions. The energy band gap of the film was calculated using the taut relation. It was found to be 0.375 eV for the virgin film (without ZnO nanoparticles) and 0.39 eV for the doped film (with ZnO nanoparticles). A smaller band gap for the doped film may indicate that the nanoparticles' presence has affected the composite's electronic Properties. The XRD spectra of the nanocomposite film indicated that the crystallinity of the film increased as the doping concentration of ZnO nanoparticles into the PVC matrix increased. Higher crystallinity often suggests improved structural order and stability.

INTRODUCTION

ZnO has a wide band gap of approximately 3.37 eV. This means that it requires a significant amount of energy to excite electrons from the valence band to the conduction band, making it a suitable material for various optoelectronic applications. ZnO exhibits a relatively high exciton binding energy of about 60 meV at room temperature. This characteristic is essential for applications such as light-emitting diodes (LEDs) and lasers. It helps in the efficient generation and recombination of electron-hole pairs, leading to the emission of photons. ZnO also has a high dielectric constant, which is beneficial for electronic and optical devices. It can be used in the development of capacitors and other electronic components these are Some of the notable applications of ZnO in the field of optoelectronics. ZnO-based materials are commonly used to produce ultraviolet (UV) and blue LEDs and laser diodes. The wide band gap and high exciton binding energy of ZnO make it suitable for emitting short-wavelength light, which is essential for applications like Blu-ray discs, UV curing, and medical diagnostics ZnO thin films are often used as transparent conductive coatings in electronic devices such as flat-panel displays, touchscreens, and solar cells. ZnO is sensitive to various gases, and it has been employed in gas sensors for detecting environmental pollutants, toxic gases, and even explosives. ZnO is used as an electron transport layer in some solar cell designs, helping to efficiently transport electrons generated by the absorption of sunlight. ZnO is a piezoelectric material, which means it can convert mechanical energy into electrical energy and vice versa. This property is used in sensors and energy-harvesting devices. Biomedical Applications: ZnO nanoparticles have been explored for their potential use in drug delivery, imaging, and cancer treatment due to their biocompatibility and photocatalytic properties. ZnO nanoparticles can be incorporated into coatings and textiles to provide UV

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protection, antibacterial properties, and self-cleaning capabilities.

Zinc oxide is mostly an n-type II-IV, wide direct band gap 3.7ev semiconducting material. Hence its applications are increasing in the current decade2. It is transparent to most of the solar Spectrum, and therefore widely used as window material in solar cells, optical, waveguides, light modulators, and optical sensors3. The sol-gel method is employed to synthesize ZnO nanoparticles. This technique allows for controlled growth and manipulation of the nanoparticles, which is crucial for obtaining the desired properties and performance in subsequent applications. We use a solution-cast method to prepare PVC films. PVC is chosen as the matrix material due to its low cost, wide availability, and suitability for use in various applications. ZnO nanoparticles are incorporated into the PVC matrix in varying proportions. Doping is a common technique to modify the properties of a material, and in this case, it can enhance electrical conductivity and dielectric Constant. The nanocomposite materials are characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), and infrared spectroscopy (IR). These techniques provide insights into the structural and morphological properties of the materials. Electrical properties such as electrical conductivity (σ_{ac}) and dielectric constant (ε) are measured in the nanocomposite films. These properties are essential for understanding how the materials perform in electronic and photonic applications.

EXPERIMENTAL METHODOLOGY

Preparation of ZnO nanoparticle by Sol-Gel Method

Dissolve 1.6 grams of NaOH in 40 ccs (cubic centimeters) of distilled water. Measure the pH of the NaOH solution, which is found to be 11.57. Dissolve 9.68 grams of $ZnSO_4$ in 120 ml (Milli-liters) of water. Add the NaOH solution drop by drop at regular intervals into the $ZnSO_4$ solution with continuous stirring. Allow the mixture to stir for 30 minutes. Leave the mixture undisturbed for 48 hours to allow precipitation to occur. After 48 hours, filter the mixture using Whatman filter paper to separate the precipitated material from the solution. Wash the residue (Zinc hydroxide) several times with de-ionized water to remove any sulfate ions and impurities. Spread the washed Zinc hydroxide on a glass substrate. Place the glass substrate in an oven and subject it to alternate heating and cooling cycles. Heat the sample for one hour at 150°C and then allow it to cool for 30 minutes. Repeat this cycle for a total of five times (a total of five hours of heating). After the five cycles of heating and cooling, we collected ZnO in powder form.

Preparation of PVC film by Solution–Cast Method

Measure precisely 4 grams of PVC granules using an analytical balance (OHAUSE, U.S.A.). This ensures accurate control of the amount of PVC used in the solution. Take 20 cubic centimeters (cc) of cyclohexanone in a burette. This serves as the solvent in which the PVC will be dissolved. Place the 4 grams of PVC granules into the 20 cc of cyclohexanone. The PVC will dissolve in the cyclohexanone. Allow the PVC to dissolve completely in the cyclohexanone. This process takes 72 hours, ensuring that the PVC is fully dissolved in the solvent. After 72 hours, stir the solution for 30 minutes to ensure that it is well-mixed.

Methodology of nanoparticle-doped film

ZnO nanoparticles with a specific concentration (0.003265 gm/cc) are uniformly spread onto a solution of PVC dissolved in cyclohexanone. The mixture of ZnO nanoparticles and the PVC- cyclohexanone solution is stirred for approximately 4.30 hours. This ensures the nanoparticles are well-distributed within the solution. The nanoparticle-doped PVC-cyclohexanone solution is spread onto a glass plate floating on a mercury surface for leveling, ensuring a flat and uniform film. The glass plate with the film is placed in a vacuum oven at a controlled temperature of 50°C for approximately one week. This slow drying process allows for the evaporation of the solvent (cyclohexanone), leaving behind the PVC film with ZnO nanoparticles embedded. After the drying period, the film of PVC doped with ZnO nanoparticles is carefully removed from the glass plate

RESULT AND DISCUSSION

Figures 1(a) and 1(b) are SEM images that reveal the surface morphology of the ZnO powder. We know SEM images can show the surface structure and texture of the material. This paper is interested in observing the morphology of ZnO particles. Based on the figures, analyze the shape, size, and distribution of the ZnO particles. SEM images can provide information about the size of the particles. By measuring the size of individual particles in the images to determine the average particle size and distribution. SEM can also reveal if the ZnO particles tend to agglomerate or clump together. Understanding how the particles are distributed and whether they are well-dispersed or clustered is important for various applications. SEM images can also help assess the purity of the material, in the presence of impurities or contaminants on the surface of the ZnO particles. SEM images can sometimes provide information about the crystal structure of the particles. This result is able to identify crystal facets and structural features.

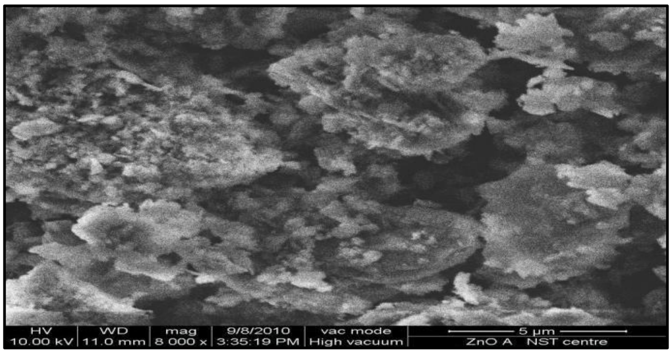

Figure 1(a) (SEM image at low magnification).

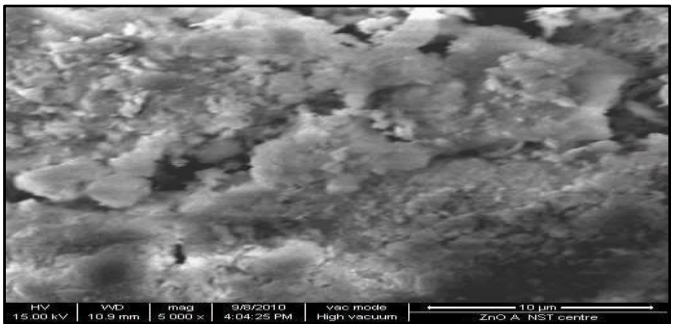

Figure 1(b) (SEM image at high magnification)

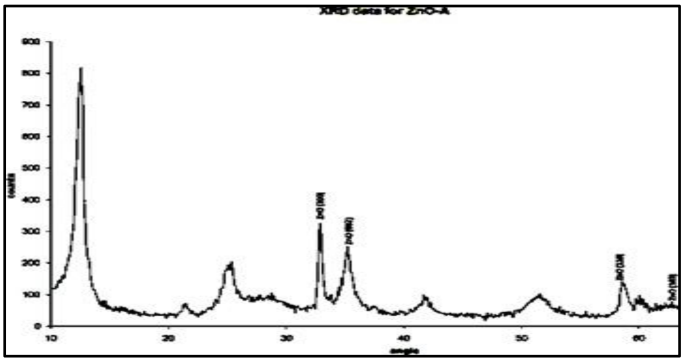

Figure 2 (XRD plot of Zn0)

Characterization through XRD

The Debye-Scherrer formula is commonly used to estimate the size of crystalline particles from X-ray diffraction (XRD) data. This formula $d = k\lambda/\beta \cos\theta$, is the correct one. Let's break down the components of this formula. d, crystallite size, which is to determine K, dimensionless shape factor (usually assumed to be 0.9) that accounts for the shape of the crystals. This factor is often approximated because the exact shape of the crystals can be challenging to determine. λ, the wavelength of the X-ray radiation used, which is 1.54 Å in this case. β, full width at half maximum (FWHM) of the diffraction peak in radians. θ, diffraction angle corresponding to the peak. The XRD analysis of ZnO powder indicates the hexagonal structure with peaks at $2\theta = 32.8^0$, 35.2^0, 58.4^0,$62.\ 48^0$ and68.8^0as shown, having the orientation [100], [002],[110],[103]and [201] as shown in fig-2. The peaks at the angle $2\theta = 12.00$ and 25.00 are due to impurity in the film. The lattice parameter value calculated for ‘a’ and ‘c’ are 3.142Å and 5.12 Å respectively and are very much close to the data (36- 1451). Table 1 shows the data observed from the diffraction pattern. The intensity counts go on decreasing with the increase in the value 2θ. The interplanar spacing d (A0) having hkl values 100, 110, 103, and 201 are very close to the Standard values of Zn0(Table-1). Some other impurities peaks are also observed. "Add references to this section, and then arrange the reference section accordingly."

Study of Dielectric Constant and Electrical Conductivity

Dielectric constant and a.c. conductivities were carried out using the LCR Meter Bridge. A special type of Sample holder was used in which a Capacitor was formed by considering the PVC and PVC/Zn0 Composite films separately in between Copper electrodes of the same diameter. The Capacitance and Conductance were measured by the LCR Bridge at different frequencies and temperatures. The Sample holder along with the sample was then kept in a thermostat, where the temperature was controlled by a contact thermometer. The values of the dielectric constant were calculated using the relation: $\varepsilon r = cd / \varepsilon 0 A$

Where
c = Capacitance of capacitor
d = Thickness of the film
A = Area of cross-section of the film
ε0 = Permittivity of free space

Whereas the values of ac conductivities of the film were determined from the relation,

$$ac = Gd / A$$

Where ’G’ is the conductance, ’d’ is the thickness of the film, and ‘A’ is the area of cross-section.

Fig: 3 Shows the variation of dielectric constant with the frequency of virgin film of PVC at a particular temperature. The dielectric constant decreases rapidly at low frequency and slowly at high frequency. The sudden decrease in dielectric constant with frequency is due to the scattering of charge and the chaotic (disordered) thermal oscillation of the molecules. From fig., it is also observed that dielectric increases initially with temperature. The increase in dielectric constant with the temperature rise is due to the space charge polarization. At low temperatures, the dipoles are not aligned properly. As the temperature rises, the orientation of dipoles is also facilitated, and hence polarization increases, this also causes the increases in dielectric constant

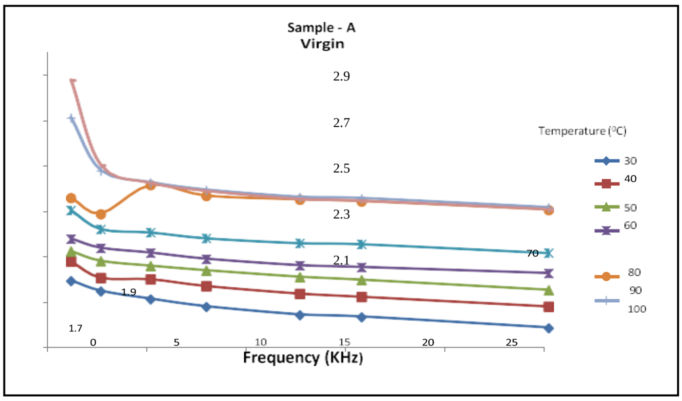

Figure 3 (Variation of dielectric constant with frequency)

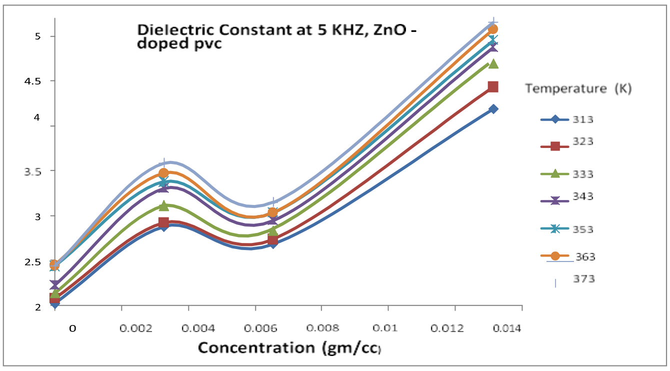

Figure 4 (Variation of dielectric constant with concentration)

Figure 4: Shows the variation of the dielectric constant with concentration of ZnO nanoparticle doped in PVC film at a particular temperature which shows that the dielectric constant increases when the concentration of doped nanoparticle increases. This occurs when impurities, possibly ions, within the crystal capture surface electrons. This process leads to the alignment of electric dipoles at the material's surface, causing surface charge polarization. As a result, the material exhibits dielectric properties. The electron capture process, which contributes to surface charge polarization, is noted to increase with rising temperature. This leads to an increase in the material's dielectric constant. This temperature-dependent behavior is typical in many dielectric materials. At higher temperatures, a transition occurs where ionic and electronic polarization becomes significant due to a resonance process. This could involve the movement or alignment of ions and electrons within the crystal lattice. This refers to the alignment of dipoles within the material, which is influenced by temperature and other factors. This appears to be related to the

interaction between impurities and charge carriers (such as electrons and ions) within the crystal, contributing to dielectric properties. Both dipolar (orientation) and space charge polarization are strongly dependent on temperature, as noted earlier. Additionally, the concentration of ZnO particles in the crystal is mentioned as a factor affecting the dielectric constant. This suggests that the presence of impurities or defects, as well as the concentration of certain materials in the crystal, can influence the dielectric properties.

Figure 5: Shows the low-temperature range, the electrical conductivity of the virgin films remains relatively constant. This suggests that, at lower temperatures, the material does not exhibit significant changes in its electrical conductivity. As the temperature increases beyond the low-temperature range, there is a slight increase in electrical conductivity. This change indicates that the material becomes more conductive at higher temperatures. In the low- frequency regions (possibly referring to a particular frequency range), the increase in electrical conductivity is attributed to the mobility of charge carriers. This mobility is dependent on the concentration of impurities within the material. Impurities can influence the movement of charge carriers in the material, affecting its electrical properties. As the temperature continues to rise, the passage suggests that the increase in A.C.'s electrical conductivity is due to the increase in the drift velocity of thermally activated electrons. This means that at higher temperatures, more electrons gain the energy required to move through the material, leading to an increase in electrical conductivity.

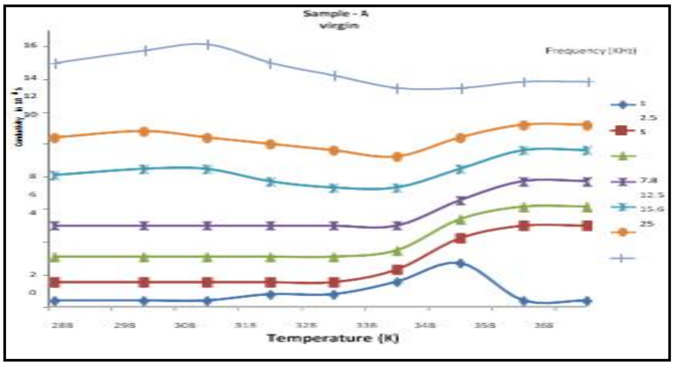

Figure 5 (Variation of conductivity with temperature).

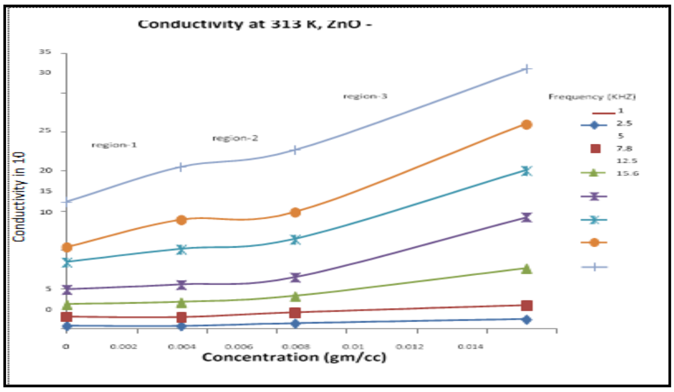

Figure 6 (Variation of conductivity with concentration)

Figure 6: Shows the variation of Conductivity with Concentration, which increases with the increase of the concentration of the nanoparticle. At low concentration (region1), with the increase of nanoparticles, the conductor sites increase in the matrix for the electron hopping mechanism. In Region 2, conductivity is impeded by the increase of nanoparticles. In this region, Vander Waals interaction causes agglomeration of nanoparticle and PVC matrix.The plateau region in the material's electrical conductivity represents a compromise between two factors: electron hopping effects and the agglomeration of nanoparticles. As the number of nanoparticles increases, this compromise is influenced by both the frequency and temperature of the system. The real conductivity of the material increases with increasing frequency, and this increase is directly proportional to the frequency. This suggests that the material's conductivity is influenced by the frequency of the applied electrical signal.The increase in conductivity at higher frequencies and temperatures suggests the presence of a polaron hopping mechanism in the sample. Polaron hopping involves the movement of charge carriers within the material. The total electrical conductivity of the sample is described as the sum of two components: σo and σac. σo is frequency-independent and temperature- dependent, representing the direct current (dc) electrical conductivity, likely associated with the drift mobility of charge carriers. σac, on the other hand, is frequency and temperature- dependent. It increases as both the frequency and temperature of the system increase. The sharp increase in conductivity in Region 3 is attributed to contact with thin polymer-coated or uncoated nanoparticles, which enhances the connectivity between opposite sites and forms a network of nanoparticles. The increase in conductivity with temperature is attributed to a hopping mechanism occurring between coordination sites, local structural relaxations, and the segmental motion of the polymers. As the amorphous region of the material increases, the polymer chain undergoes faster internal modes in bond rotation, resulting in increased segmental motion. This, in turn, promotes the hopping of ion movements both within and between polymer chains, leading to higher electrical conductivity in the material.

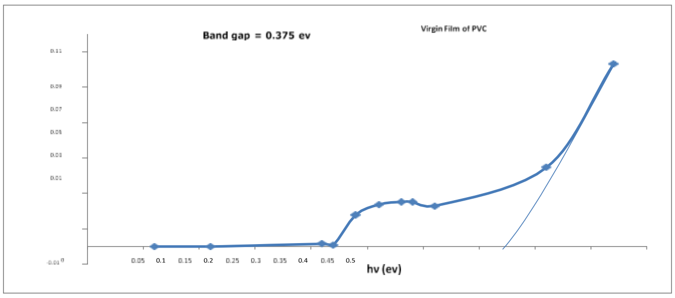

Figure 7 Absorption spectrum of ZnO to determinethe band gap of virgin film of PVC.

Optical measurement: The optical band gap of materials is associated with the photon energy and absorbance by the given relation13.

$$\alpha hv = A(hv- E)n \text{ Putting } n = 1/2$$

Fig. (**7**) and **Fig.** (**8**) shows the FTIR absorbance spectrum of virgin and doped Zn0 nanoparticle in the PVC matrix. α (alpha) is the absorbance, ν (mu) is the frequency of light, h is Planck's constant, and n represents different types of electronic transitions (e.g., n = 1/2, 2, 3/2, 3) for direct allowed, indirect allowed, direct forbidden, and indirect forbidden transitions, respectively. The formula $(\alpha h\nu)^2 = A(h\nu - E)$ relates the square of the absorbance to the energy gap (E) of the film. In this context, the energy gap of the film is determined by extrapolating linear plots of $(\alpha h\nu)^2$ vs. hν. The energy gap of the virgin film is calculated to be 0.375 eV through the extrapolation of the mentioned linear plots. Meanwhile, the energy gap of the ZnO nanoparticle-doped film is calculated to be 0.39 eV. This suggests that doping nanoparticles have an impact on the energy band gap of the polymer film. The text mentions that the interaction between the ZnO nanoparticles and the polymer matrix leads to a shift in the absorbance peaks and the formation of new peaks. This indicates changes in the optical properties of the film due to doping. The energy gap of the ZnO-doped film is found to be higher than that of the virgin PVC film, suggesting that the introduction of ZnO nanoparticles affects the electronic structure of the film. The text also briefly mentions the diffraction pattern of pure PVC film, indicating some impurity peaks at specific angles (2θ = 18.50 and 27.00) and noting the amorphous nature of pure PVC.

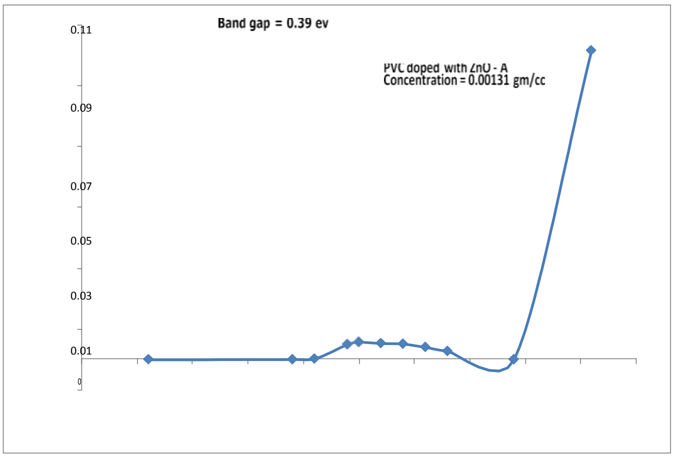

Figure 8 Absorption spectrum of ZnO to determine the band gap of PVC film-doped nanoparticle.

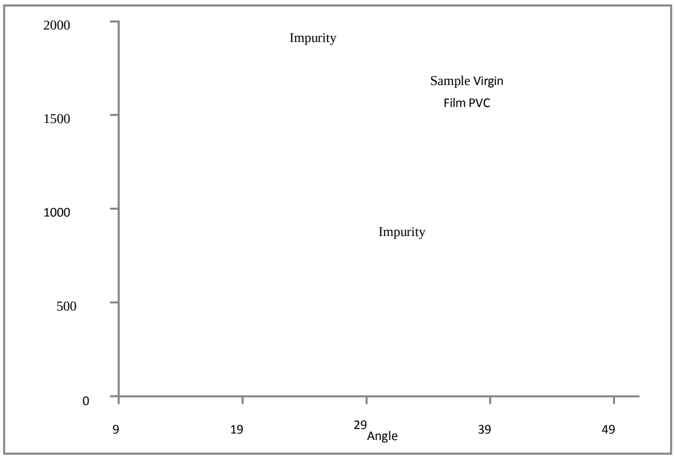

Figure 9 (Intensity Count versus angle of the virgin film)

Figures 10 and 11 are XRD patterns, which are used to analyze the crystallographic structure and order of a material. Two different doping concentrations of ZnO nanoparticles are considered: 0.003265 g/cc and 0.00653 g/cc. The observation that the intensity of peaks in XRD (X- ray diffraction) patterns is higher when the doping concentration is higher is a common phenomenon in materials science and can be related to the increase of the dopant or the crystallinity of the material. This could be indicative of an increase in the crystalline order or the presence of more well- defined crystallographic features when a higher concentration of ZnO nanoparticles is introduced into the PVC matrix. The text concludes that after doping, the film shows a higher degree of structural order. This implies that the presence of ZnO nanoparticles has influenced the structural properties of the PVC matrix, potentially leading to improved crystalline order or more pronounced crystallographic features.

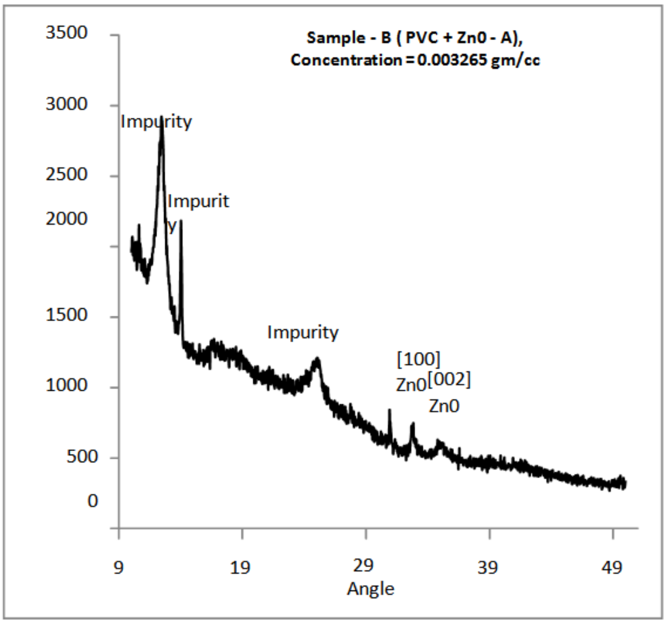

Figure 10 (Intensity Count versus angle of the doped film).

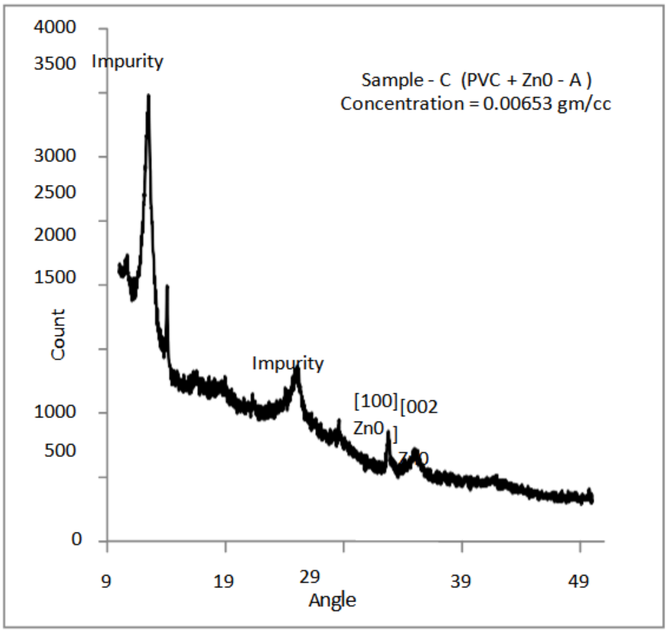

Figure 11 (Intensity Count versus angle of the doped film)

CONCLUSION

1. The ZnO nanoparticles are found to have a size of 34.5 nm and a hexagonal structure. Their lattice parameters closely match that indicating their crystalline nature and the specific crystal structure.
2. The dielectric constant (permittivity) of the material is mentioned to decrease rapidly at lower frequencies, up to 7 KHz, and then decreases more gradually at higher frequencies, up to 25 KHz. This behavior suggests a frequency-dependent dielectric response,

which can be characteristic of materials with different polarization mechanisms.

3. The dielectric constant is reported to increase at both low and high concentrations of ZnO nanoparticles. However, it remains at a low value in the concentration range of
4. 0.004 to 0.007 gm/cc of doped nanoparticles. This indicates that the dielectric constant is influenced by the concentration of nanoparticles and exhibits specific behavior in this concentration range.
5. The alternating current (a.c.) conductivity is said to increase with the increase in the concentration of ZnO nanoparticles within the PVC matrix. However, there is an impediment in the conductivity between the concentration range of 0.003 to 0.006 gm/cc. This is attributed to Vanderwaal interactions leading to the agglomeration of nanoparticles, which can impede the conductivity.
6. The energy gap of the virgin PVC film is reported to be 0.375 eV, while the energy gap of the doped film is calculated to be 0.39 eV. This difference in energy gaps suggests that there is an interaction between the ZnO nanoparticles and the polymer matrix, which likely affects the electronic properties of the material.
7. The XRD (X-ray diffraction) plot of the virgin PVC film shows an amorphous nature, while the nanocomposite film exhibits a crystalline nature. Additionally, the crystallinity is stated to increase with the higher concentration of ZnO nanoparticles. This indicates that the introduction of nanoparticles leads to a transition from an amorphous to a crystalline structure, and the degree of crystallinity is concentration- dependent.

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