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Effect of Zinc Oxide Nanoparticles and Structural Properties of PVA/PMMA Composite Films

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ABSTRACT

Recent studies have been conducted on insulating polymers to convert them into semiconductor materials for use in many fields, including electronics. The polymers (PVA) and (PMMA) were chosen for their distinctive properties, availability, low production cost, and integration with each other, and nanoparticles (ZnO) were added to improve their structural and electrical properties. This research article investigates the impact of zinc oxide (ZnO) nanoparticles on the electrical and structural properties of composite films made from polyvinyl alcohol (PVA) and polymethyl methacrylate (PMMA). The researchers prepared composite films with varying concentrations of ZnO (0%, 1%, 3%, 5%, and 7% by wt.%) using a solution casting technique. The study employed several analytical methods to characterize the films. FTIR spectroscopy revealed hydrogen bonding between PVA and PMMA, indicating good compatibility between the polymers. XRD analysis showed that the films maintained a crystalline structure even at higher ZnO concentrations. SEM imaging provided insights into the structural reorganization of polymer chains and the distribution of ZnO within the composite. A key finding of the study was the significant improvement in electrical conductivity with the addition of ZnO nanoparticles. The pure PVA/PMMA blend was found to be an electrical insulator, with a conductivity of $9.9 \times 10^{-11} (\Omega . cm)^{-1}$,. However, the addition of ZnO dramatically enhanced conductivity, with the optimal concentration being 3% ZnO, which yielded a conductivity of $4.2 \times 10^{-6} (\Omega . cm)^{-1}$. This transformation effectively changed the material from an insulator to a semiconductor. The researchers concluded that the composite with 3% ZnO concentration exhibited the best overall properties. They suggest that this material could have potential applications in various fields, including digital screens, LCDs, and optical signals. The study demonstrates the potential of incorporating nanomaterials to enhance the properties of polymer composite films, opening up new possibilities for their use in various industries. This is the first study of this PVA-PMMA/ZnO composite and its transformation into a semiconductor material.

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تأثير جزيئات أكسيد الزنك النانوية على الخواص الكهربائية والتركيبية للأغشية المركبة PVA/PMMA

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الـــخُـــلاصـــة

أجريت دراسات حديثة على البوليمرات العازلة لتحويلها إلى مواد شبه موصلة لاستخدامها في العديد من المجالات ومنها الإلكترونيات، وقد تم اختيار البوليمرات (PVA) و(PMMA) لخصائصها المميزة وتوافرها وانخفاض تكلفة إنتاجها وتكاملها مع بعضها البعض، وتم إضافة الجسيمات النانوية (ZnO) لتحسين خواصها البنيوية والكهربائية. يبحث هذا البحث في تأثير الجسيمات النانوية لأكسيد الزنك (ZnO) على الخواص الكهربائية والبنيوية للأغشية المركبة المصنوعة من كحول البولي فينيل (PVA) وميثاكريلات البولي ميثيل (PMMA). قام الباحثون بإعداد أغشية مركبة بتركيزات متفاوتة من (ZnO 0٪، 1٪، 3٪، 5٪، و7٪ بالوزن) باستخدام تقنية الصب بالمحلول. استخدمت الدراسة عدة طرق تحليلية لتوصيف الأغشية، حيث كشف مطياف الأشعة تحت الحمراء عن وجود رابطة هيدروجينية بين PVA وPMMA، مما يدل على التوافق الجيد بين البوليمرات. أظهر تحليل XRD أن الأغشية حافظت على بنية بلورية حتى عند تركيزات أعلى من ZnO. وقد وفرت تقنية التصوير بالمجهر الإلكتروني الماسح رؤى ثاقبة حول إعادة التنظيم البنيوي لسلاسل البوليمر وتوزيع أكسيد الزنك داخل المركب. وكان أحد النتائج الرئيسية للدراسة هو التحسن الكبير في التوصيل الكهربائي مع إضافة جسيمات نانوية من أكسيد الزنك. وقد وجد أن مزيج PVA/PMMA النقي هو عازل كهربائي، مع توصيل كهربائي يبلغ $9.9 \times 10^{-11} (\Omega.cm)^{-1}$ ، ومع ذلك، فإن إضافة أكسيد الزنك عززت التوصيل الكهربائي بشكل كبير، حيث كان التركيز الأمثل 3% أكسيد الزنك، مما أسفر عن توصيل كهربائي يبلغ $4.2 \times 10^{-6} (\Omega.cm)^{-1}$. وقد غير هذا التحول المادة بشكل فعال من عازل إلى شبه موصل. وخلص الباحثون إلى أن المركب بتركيز 3% أكسيد الزنك أظهر أفضل الخصائص الإجمالية. ويشير الباحثون إلى أن هذه المادة قد يكون لها تطبيقات محتملة في مجالات مختلفة، بما في ذلك الشاشات الرقمية، وشاشات الكريستال السائل، والإشارات الضوئية. وتوضح الدراسة إمكانية دمج المواد النانوية لتعزيز خصائص أفلام البوليمر المركبة، مما يفتح إمكانيات جديدة لاستخدامها في مختلف الصناعات.

الكلمات المفتاحية:

غشاء PVA/PMMA-ZnO

FTIR

XRD

SEM

والخواص الكهربائية.

1. INTRODUCTION

Recent decades have seen remarkable advances in polymer technology, resulting in the worldwide production of approximately 200 million tons of plastics annually. Polymers are extremely useful in a variety of electronic strategy applications due to their many beneficial qualities, which include high resistance, low cost, ease of manufacture, and flexibility [1]. The

subject of polymer science is now rapidly expanding in a number of interesting ways as researchers work to create high-quality polymers with a wide range of uses. PVA differs from other polymers in a number of beneficial ways, including strength, corrosion resistance, and superior thermal stability [2]. It features a backbone of carbon chains stabilized by hydroxyl functional groups present in the methane carbonate [3]. These OH groups help form effective polymer mixtures through hydrogen bonding. The amorphous thermoplastic polymer is called PMMA [4]. PMMA is a significant member of the family of methacrylic and polyacrylic esters. Due to its exceptional optical clarity, good weathering ability, high strength, excellent dimensional stability, low optical absorption due to its high transparency in the visible region, low refractive index, hardness, heat capacity, electrical performance, good mechanical properties, and ease of synthesis, PMMA has garnered a great deal of attention and remarkable features[5]. The great development in nanotechnology in recent years has led to substantial improvements in the field of materials science[6]. Nanoparticles have high resistance to harsh conditions and are stable in the presence of acids and alkalis; however, their scratch resistance is poor, which is one of the main drawbacks of this polymer [7]. Nanoparticles have attracted research and engineering interest in recent years, based on the high performance of their matrices, which allow for manipulation and structure enhancement that can improve the properties of the resulting materials, assuming that the interaction occurs between the dispersed particles and the relevant matrix. Carbonaceous materials are generally considered the most important nanofillers, allowing for a significant improvement in the properties of the materials. Nano zinc oxide, or nano ZnO, is one of the most significant nanomaterials in materials science [8,9]. Due to its special qualities, nano ZnO has been the focus of several investigations [10]. The outstanding characteristics of polyvinyl alcohol (PVA) and poly methyl methacrylate (PMMA) make them common polymers that can be utilized in the fabrication of plastic films [11]. The incorporation of nanomaterials into these films has been demonstrated in earlier research to considerably improve their characteristics [4,12]. Gaining insight into how adding nano zinc oxide would affect the structural and electrical characteristics of PVA/PMMA composite films might help providing new uses in [13]. In some studies, researchers improved the electrical properties, the DC conductivity value of PMMA-PVA/GO nanocomposites was 7.86×10^{-15} $(\Omega.cm)^{-1}$at GO ratio of 0.09%, while increasing the GO loading ratio to 0.27% caused the conductivity to increase to 4.53×10^{-9} $(\Omega.cm)^{-1}$[14]. Conducting a thorough study with cutting-edge analytical methodologies, this research attempts to assess the impact of nano ZnO addition on the structural and electrical characteristics of PVA/PMMA composite films, The electrical properties in our study were improved by a greater percentage than previous researchers.

2. EXPERIMEMTAL

The materials selected in this project are explained in some details as follows:

2.1 Matrix Materials:

PVA and PMMA resin prepared using acrylic powder with methyl methacrylate liquid monomer were used. The starting materials were selected from alkoxide-based chemicals that can be easily dissolved in solvents to produce ceramic nanoparticles. These starting materials were diluted with different solvents which are generally alcohol-based liquids such as methyl alcohol and ethyl alcohol. They were from Marlik Medical Industries Company, Iran.

2.2 Reinforcement Material:

Zinc oxide (ZnO) powder of particles with an average particle size of (315 nm) and provided by Volka and Honeywell USA was used as a reinforcing material for PMMA-PVA matrix to prepare PMMA-PVA/ZnO composite material.

2.3 PVA

Four grams of PVA with 100 ml of deionized water and magnetic stirrer mix for 6 hours at room temperature.

2.4 PMMA

PMMA liquid is mixed with the powder to produce the polymer (50%) magnetic stirrer for 6 hours at room temperature.

2.5 PVA-PMMA Sample preparation

2.5.1 Add 50% of PVA and 50% of PMMA to get the first sample.

2.5.2 At room temperature, the mixture is mixed for six hours, then zinc oxide is added to the second, third, fourth and fifth samples at a ratio of (1%, 3%, 5% and 7%) of zinc oxide (ZnO) powder, respectively, and magnetic stirrer mixed for 6 hours.

2.5.3 The samples were poured onto a glass plate for 72 hours until they dried and the sample thickness was 0.25mm. Table 1 shows the concentration and proportions of the samples. is shown in Fig.1

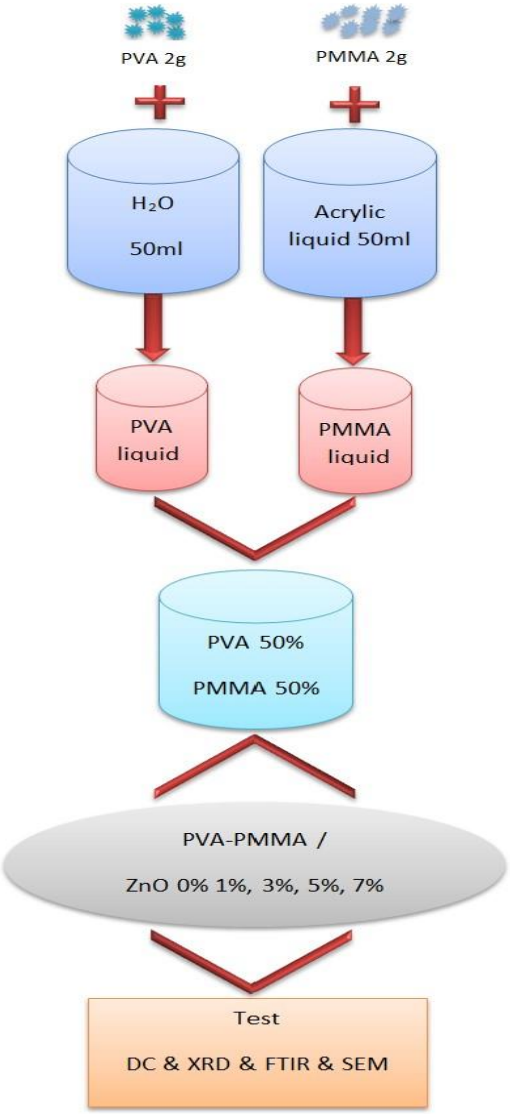


Fig.1 The method of work.

Table 1. Concentration and proportions of the samples

Samples	PVA-PMMA	ZnO
1	100%	0%
2	99%	1%
3	97%	3%
4	95%	5%
5	93%	7%

3. RESULTS AND DISCUSSION

3.1 (FTIR) Fourier Transform Infrared Spectroscopy:

The chemical bonds and functional groups found in the composite films were investigated using FTIR analysis. FTIR spectroscopy may be used to study the chemical species and structure of polymers. These materials' infrared spectra change depending on their composition and can reveal complexation and interaction between different elements. The atoms or molecules in the material undergo changes in their vibrational modes due to their mutual contact, resulting in modifications of the physical and chemical characteristics of the complex's constituents[15]. Pure and PVA/PMMA-ZnO films' FTIR peaks are shown in Table 2. Because of the creation of crosslinks between the PVA/PMMA and nano ZnO, the influence of mixture on the modes of vibration can be seen in terms of an increase in band intensity and the introduction of new bands. The structural and electrical properties of pure PVA, PMMA and a 50/50 PVA/PMMA blend were studied. The sample films were prepared by solution casting method. The modification in the structural properties was described by FTIR spectroscopy. This spectroscopy reveals the formation of several new bonds and bond breaking. This suggests that as the concentration of PVA/PMMA increases, so does the functional groups of C= O [16]. The peak at 2984 cm^{-1} is usually associated with hydrogen bond stretching groups, suggesting the presence of saturated O–H stretching [17]. 1721 cm^{-1} this peak is frequently ascribed to C = O stretch bonds, which are PMMA's defining characteristics. The peak which is located at 1560 cm^{-1}, may be connected to N–O asymmetric stretch and suggest that the polymers include aromatic groups [18,19]. The peak which has a wavelength of 1360 cm^{-1}, is typically linked to C-H bond vibrations, such those seen in alkyl groups [20]. The peak which measures 1244 cm^{-1}, may be attributed to C-N bonds and indicates the existence of nitrogen-containing groups in the sample. The peak which measures 1085 cm^{-1}, may be related to C-O Stretch and suggests the existence of hydroxyl groups. The peak at 944 cm^{-1}, could be connected to the carboxylic acids found in organic molecules C–O bend [21,22] as in the Fig.2.

Table 2. shows the FTIR peak assignments for PVA/PMMA-ZnO and pure films.

Wavenumber(cm^{-1})	functional groups
2984	O–H stretch
1721	C = O stretch
1560	N–O asymmetric stretch

1360	C–H bond
1244	C–N bond
1085	C - N Stretch
944	C–O bend

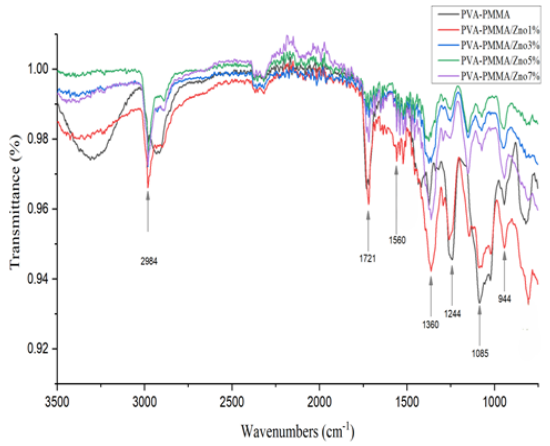


Fig.2 FTIR spectrum of PVA/PMMA-ZnO films.

3.2 X-Ray Diffraction (XRD):

The crystalline structure and phase composition of the composite films were confirmed by XRD analysis. A helpful method for figuring out the crystallinity and structure of polymer matrices is X-ray diffraction [23]. To constructively establish the directions in which Bragg's rule dictates the spacing (d) between diffracting planes [24]

$$2d\sin\theta = n\lambda \dots\dots\dots (1)$$

where n is the number of orders of diffraction, θ is the Bragg angle, and λ=0.15406nm is the wavelength of the X-ray beam. Reflections are spots on the diffraction pattern that represent these particular directions. The lattice constant can be found by using equation [24,25].

$$a = \frac{n\lambda\sqrt{h^2 + k^2 + l^2}}{2\sin\theta} \qquad \dots\dots\dots (2)$$

shape factor is used in X-ray diffraction and crystallography to correlate the size of a solid's sub-micrometer particles, or crystallites, to broadening a peak in a diffraction pattern. In the Scherrer equation [24,26].

$$D = \frac{k\lambda}{\beta\cos\theta} \qquad \dots\dots\dots (3)$$

where λ is the X-ray wavelength, is the line broadening at full width at half maximum (FWHM) in radians, θ is the Bragg angle, and K is the shape factor which represents the ratio of a particle's main dimension to its minor dimension.[27]

The PVA and PMMA copolymers' XRD patterns and blend samples at ZnO are displayed in Fig.3. When PVA/PMMA is introduced to a solution, its spectrum displays no peaks. However, a peak arises at 2θ = 34°, 32°, and a slight large peak at 2θ = 36° spread in the upper amorphous area. By XRD analysis, the crystalline and amorphous phases of crystalline polymers exhibit acute reflections and diffuse scattering. Consequently, the PVA/PMMA mix in Fig.2 similarly demonstrated its crystalline nature and did not significantly alter even when a higher concentration of ZnO was added. The XRD patterns of blend samples with differing reflectance peak intensities and ZnO concentrations of 1, 3, 5, and 7 (wt%) are shown in Fig.2. Compatibility between the crystalline and amorphous copolymer components is achievable for such mixture. Therefore, the film's amorphous and crystalline structure is what causes the greater ionic diffusivity, which can

result in higher ionic conductivity in polymers with flexible backbones [28].

3.3 Scanning Electron Microscopy (SEM):

The surface morphology and microstructure of the composite films were observed at the nanoscale level using SEM imaging. By using a concentrated electron beam to scan a sample, a scanning electron microscope creates morphology of the material. The sample's electrons interact with the electron beam to produce a variety of observable signals that provide details about the composition and surface morphology of the sample. Typically, the electron beam is scanned using a raster scanning pattern, and a picture is produced by combining the beam's location with the detected signal [29].

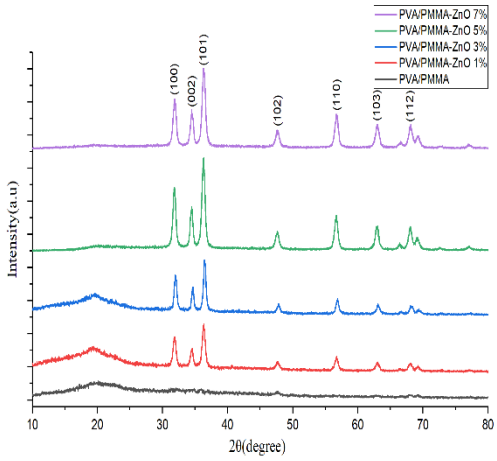


Fig.3 XRD curves of PVA/PMMA-ZnO films

The SEM is frequently used to identify phase separation and interfaces in order to investigate the compatibility of various polymer components. The structural and electrical properties of the polymer mixture are significantly influenced by the compatibility with inorganic activators [25]. The SEM pictures of the PVA/PMMA-ZnO (1,3,5,7) wt% mix and pure PVA/PMMA are displayed in Fig.4. Fig.4(a) illustrates the heterogeneous surface of the pure PVA/PMMA film, suggesting that ZnO molecules may scatter in the fine fraction phase with minimal impact on the micro-phase separation. Nevertheless, Fig.4(a) of the SEM of the pure PVA/PMMA film displays no characteristics indicative of any crystalline form; hence, Fig.4 (b, c, d, e) shows up crystallinity. The ZnO distribution fills in the gaps in the polymer with clarity, and the nanomaterial diffuses smoothly throughout the polymer. In Fig.4(c), the sample has the fewest flaws, and a branch-like form is beginning to grow. Nanoparticle become densely grouped together as a result of the more pronounced clustering of branching cluster structures. It is evident from this that the polymer chains are undergoing structural reformation.

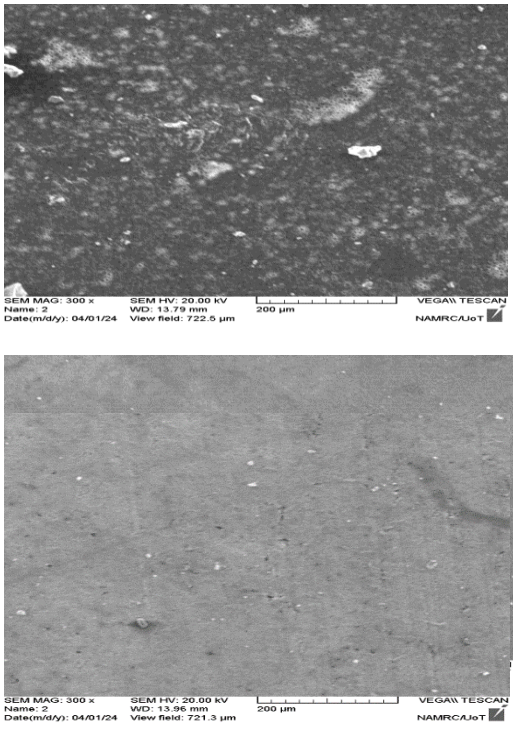
3.4 Electrical conductivity:

A DC current was used to examine the electrical properties of the composite films. The electrical conductivity of the samples was measured, and the electrical conductivity was shown as in Table 3, where the pure PVA/PMMA polymer is considered an electrical insulator and its value is equal to $9.9 \times 10^{-11}(\Omega.cm)^{-1}$. When adding ZnO nanoparticles, the electrical conductivity improved significantly, and at a percentage of ZnO 3% , the nanoparticles that reached $4.2 \times 10^{-6}(\Omega.cm)^{-1}$were the ideal ratio of conductivity due to the lack of defects, as shown in examining sem and converting samples from insulator to semiconductor as shown in Fig.5. These

results were better than previous studies, including: the DC conductivity value of PMMA-PVA/GO nanocomposites was 7.86 × 10^{-15} $(\Omega.cm)^{-1}$ at GO ratio of 0.09%, while increasing the GO loading ratio to 0.27% caused the conductivity to increase to 4.53 × 10^{-9} $(\Omega.cm)^{-1}$.[14]

Table 3. Shows electrical conductivity values of PVA/PMMA-ZnO and pure films.

Simple	Conductivity $(\Omega.cm)^{-1}$
PVA/PMMA	9.9×10^{-11}
PVA/PMMA-ZnO 1%	6.6×10^{-8}
PVA/PMMA-ZnO 3%	4.2×10^{-6}
PVA/PMMA-ZnO 5%	7.6×10^{-7}
PVA/PMMA-ZnO 7%	4.9×10^{-9}



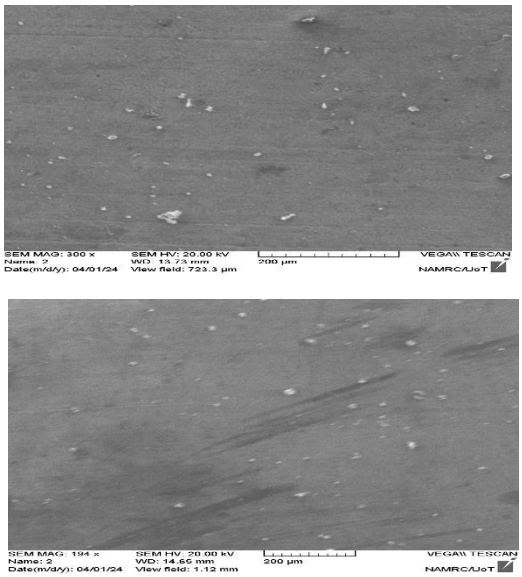


Fig.4 SEM images of
(a)PVA/PMMA
(b)PVA/PMMA-ZnO 1%
(c)PVA/PMMA-ZnO 3%
(d) PVA/PMMA-ZnO 5%
(e)PVA/PMMA-ZnO 7%

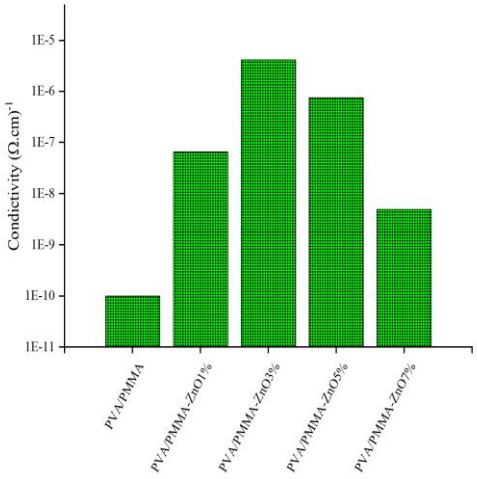


Fig.5 Shows electrical conductivity of films

4. CONCLUSION

It can be seen that when the percentage of ZnO increases, the absorption edge in the membranes increases. It can be concluded that the hydrogen bond formation between PVA/PMMA can be visualized in the analyzed composition ranges based on the observed data from XRD, FTIR, DC, and SEM. Using FTIR and by examining the shifts in XRD peaks. ZnO showed significant improvement in optical properties which increased with increasing loading ratio of ZnO nanoparticles in the composites. The fabrication of the new nanocomposites was successfully achieved such that homogeneous and fine dispersion of nanoparticles was observed in the samples, and both the contribution of ZnO and the increases in loading ratio contributed to the significant improvement of the electrical properties based on DC electrical conductivity test. These nanocomposites were successfully prepared using the casting method in this study. All FTIR spectra of the blend samples display variations in peak strength when the amount of ZnO in the polymer matrix decreases, indicating a strong interaction between PVA/PMMA and ZnO. Studies using X-ray diffraction (XRD) on samples blended with 1%, 3%, 5%, and 7% weight content of ZnO showed that the crystalline structure of the films is generally preserved but exhibits an amorphous character of PVA/PMMA. The SEM image of the polymer blend displays the shape of the dendrites. At 3% ZnO, the electrical conductivity appears and is equal to $4.2 \times 10^{-6}(\Omega.cm)^{-1}$, which is a higher value than the rest of the samples. Electrical conductivity increased as a result of the good distribution of nanoparticles in the matrix, the other percentages decreasing as a result of the agglomeration of nanoparticles in the matrix, and the best distribution of nanoparticles at the percentage of 3% ZnO. The new nanocomposites were successfully fabricated, resulting in homogeneous and finely dispersed ZnO nanoparticle in the samples. The DC electrical conductivity test showed a significant improvement in the electrical properties, which was attributed to both the increased loading ratio and the contribution of ZnO. The polymer is transformed from an insulator to a semi-conductor. The test findings indicate that these films have potential applications in the automobile and smartphone sectors, as well as in related fields such as UV filters, solar cells, light diffusers, and gas or humidity sensors, including digital screens, LCDs, optical signals, and others[14]. In subsequent studies, it is possible to improve the mechanical and optical properties of the composite. We have had problems with thick film if the weather is wet, so we recommend making films in suitable weather conditions such as dry weather.

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