Journal of Nano- and Electronic Physics
Vol. **18** No 3, 03028(6pp) (2026)

Журнал нано- та електронної фізики
Том **18** № 3, 03028(6cc) (2026)

OPEN ACCESS

REGULAR ARTICLE

Polymer Nanocomposite Films Doped with Piezoelectric Metal Oxide Nanoparticles

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(Received 03 March 2026; revised manuscript received 18 June 2026; published online 26 June 2026)

Polymer nanocomposite films doped with piezoelectric metal oxide nanoparticles have attracted significant attention due to their potential applications in flexible electronics, sensors, and energy harvesting devices. This research investigates the structure-property correlations in polymer nanocomposite films composed of ethylene vinyl acetate (EVA) doped with piezoelectric metal oxide nanoparticles, specifically Potassium Sodium Niobate (KNN) and Lithium Niobate ($LiNbO_3$). EVA, a copolymer of semi-crystalline polyethylene and amorphous vinyl acetate, provides tunable material properties, making it an ideal matrix for functional nanocomposites. Morphological analysis using SEM and AFM confirmed homogeneous nanoparticle dispersion across different EVA compositions. Structural characterization through XRD and FTIR revealed increased crystallinity and polymer-nanoparticle interactions in doped films. Mechanical testing showed tensile strength from 2 to 13 MPa for 15 % KNN, and 30 % $LiNbO_3$, indicating mechanical reinforcement while maintaining flexibility. These findings demonstrate the potential of KNN- and LiNbO-doped EVA films for flexible electronics, sensors, energy harvesters, and biomedical applications, providing a tunable platform where mechanical reinforcement and piezoelectric responsiveness are simultaneously achieved.

Keywords: Ethylene vinyl acetate (EVA), Polymer nanocomposites, Potassium Sodium Niobate (KNN), Lithium Niobate ($LiNbO_3$), Piezoelectric nanoparticles.

DOI: 10.21272/jnep.18(3).03028 PACS number: 81.05.Qk

1. INTRODUCTION

Nanoparticles serve as active fillers in composite systems, which can dramatically change mechanical, thermal, and electrical characteristics. It is essential in the design of advanced nanocomposites due to their high surface area and special qualities [1]. Piezoelectric nanoparticles are particularly useful since they transform into electrical signals. By doing this, their integration allows nanocomposite films to act as energy harvesters, pressure sensors, or biomedical transducers [2, 3]. For nanoparticles to be useful, they need to be uniformly dispersed and interfacial bonded to the polymer matrix. These criteria govern improvement in stability, reinforcement, and functional responsiveness of films [4]. Variation in type, shape, and surface chemistry of nanoparticles impacts overall film performance. Improvement of these aspects directly affects material adaptability and expands device-level applications in varied industries [5, 6]. Although helpful, nanoparticles bring issues that may complicate any useful application, and they may lead to agglomeration, enhance processing complexity, and impact reproducibility. Solutions to these challenges are needed to achieve scalable, reliable, and efficient nanocomposite materials [7]. Nanocomposite films are a type of engineered material that incorporates polymers with nanometer-sized fillers as hybrid mixtures that increase the mechanical strength of the system, thermal stability, and other functional properties to be applied at higher levels of usage [8]. Lightweight, flexible, and adaptable, these films can be used as part of multi-functional incorporation into new technologies. Tunable properties make it possible to modify. In this case, the key role of the structure and the properties of nanocomposite films is paramount. Morphological changes, crystallinity, and filler distribution characterize the performance outcomes of these multifunctional composite systems [9, 10]. Prior research on polymer nanocomposite films typically featured either difficulty dispersing nanoparticles evenly, weak interfacial strength, or uneven crystallinity, all of which are detrimental to mechanical reinforcement, heat stability, and piezoelectric efficiency, restricting multifunctional performance and scalability. The current research overcomes these obstacles using controlled solvent casting to achieve uniform dispersity of nanoparticles and enhance

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Cite this article as: F.T. Ayasrah et al., *J. Nano- Electron. Phys.* 18 No 3, 03028 (2026) https://doi.org/10.21272/jnep.18(3).03028

the interfacial bonding between EVA and doped oxides, as well as the improved crystallinity and matrix-filter interactions that contribute to improved mechanical strength, heat-stability, and piezoelectric performance.

1.1 Key Contribution

The research produces ethylene vinyl acetate (EVA)-based polymer nanocomposite films that were doped with KNN and $LiNbO_3$ nanoparticles with uniform dispersion and increased structural integrity.

Morphological evaluation using SEM and AFM confirmed uniform nanoparticle dispersion across different EVA compositions.

The XRD and FTIR analyses indicated stronger polymer-nanoparticle interactions and improved crystalline arrangement within the matrix.

2. RELATED WORK

The research [11] improved the optical properties of polyaniline (PANI) by integrating PbS nanoparticles into PANI/PbS nanocomposite films through solution casting. Characterization techniques like transmission electron microscopy (TEM) and X-ray diffraction (XRD) revealed an increase in bandgap and optical conductivity. However, the research identified limitations in assessing long-term stability and operational performance, impacting the devices' reliability and practical application. Optical and electrical properties of polyethylene oxide and carboxymethyl cellulose films were investigated, with a focus on improvements from zinc oxide and graphene oxide nanoparticles [12]. The research concentrated on the synthesis and structural interactions of flexible composite films, although it acknowledged limits in long-term stability and mechanical performance. The investigation [13] enhanced the optical and thermal properties of polymethyl methacrylate (PMMA) thin films by incorporating metal oxide nanoparticles like zinc oxide, leading to reduced optical band-gap energy, improved optical dispersion, and greater thermal stability. However, there was insufficient assessment of long-term mechanical performance, operational stability, and overall device reliability. The research [14] of nanoparticle-reinforced sodium alginate films for antimicrobial and bioactive food packaging includes synthesizing nanocomposite films via solution casting, with conductivity tests revealing improved crystallinity and antibacterial activity. However, the constraints of long-term stability and food compatibility under storage circumstances were not extensively evaluated. The research investigated [15] the structural, optical, and dielectric characteristics of carboxymethyl films containing copper oxide nanoparticles for application in solid-state supercapacitors. Nanocomposite films were created by casting with various nanoparticle concentrations, proving their applicability for energy storage devices. However, limited research on their integration into in vivo optoelectronic systems limits their practical application and assessment. Research investigated the impact of metal oxide nanoparticles on poly (lactic acid) (PLA) films. Zinc oxide (ZnO) and zinc oxide–titanium dioxide ($ZnO–TiO_2$) nano-hybrids were developed, resulting in PLA nanocomposites. The findings revealed improved dispersion, increased tensile strength, and antibacterial properties of the nano-hybrids; however, elevated nanoparticle concentrations led to aggregation, negatively affecting uniformity and scalability. According to research, gelatin/tapioca starch bio-nanocomposite films with inorganic (zinc oxide) nanoparticles have higher thermostability and antibacterial characteristics than those with organic (chitosan) nanoparticles. However, insufficient testing in real-world food storage circumstances limits insights into the material's resilience and longevity.

2.1 Research Gap

Earlier research on polymer nanocomposite films using PbS [11], ZnO and graphene oxide [12], metal oxides such as ZnO, TiO_2, Silicon dioxide (SiO_2) [13], Nickel-doped zinc oxide (Ni/ZnO) [14] and CuO [15] had investigated the enhancement of optical, thermal, mechanical and antibacterial properties but had not given much attention to long-term stability, device performance and reliability. The present research examines KNN and $LiNbO_3$-doped EVA films, which attain monolithic nanoparticle dispersion, adjustable mechanical strengthening, and long-term piezoelectric sensitivity, in the long-term performance and the versatile application of the multifunctional device.

3. METHODOLOGY AND DESIGN

The research produced nanocomposite films of EVA that had high mechanical, thermal, and piezoelectric characteristics. The polymer matrix was EVA, which was doped with KNN and $LiNbO_3$ nanoparticles. Thin films were produced by solvent casting, and morphological, structural, mechanical, thermal, and piezoelectric characterization was made to assess the performance.

EVA copolymers were selected as the polymer matrix because of their semi-crystalline polyethylene and amorphous components of vinyl acetate, giving tunable mechanical and thermal characteristics. KNN and $LiNbO_3$ piezoelectric metal oxide nanoparticles were also chosen to impart piezoelectricity to the polymer films. EVA was dissolved in solvents of analytical grade to obtain an equal dispersion of nanoparticles.

The solvent was mixed thoroughly with EVA to produce homogeneous solutions with different polymer concentrations and content of the vinyl acetate. To prevent agglomeration and to ensure uniform dispersion of the nanoparticles, KNN and $LiNbO_3$ nanoparticles were incorporated into the polymer solutions with ultrasonic assistance. The films were dried down under ambient conditions and subsequently carefully peeled off for additional analysis.

The distribution of the nanoparticles and surface morphology were assessed using Scanning Electron Microscopy (SEM), XRD and Atomic Force Microscopy (AFM). All of the analyses above demonstrated no dispersions of KNN and $LiNbO_3$ (15 and 30 %) nanoparticles in EVA that was prepared as a function of vinyl acetate concentrations (15 % and 18 %).

SEM: It is a high-resolution imaging method, which gives fine surface morphology of materials. It was used to check the homogeneous distribution of KNN and $LiNbO_3$ nanoparticles in the EVA matrix.

AFM: It is a characterization tool on a nanoscale that

is used to map the surface topography with high accuracy. It was employed to compare the roughness and nanoparticle distribution in EVA films.

XRD: It is a method of determining the existence of crystalline phases and measuring structural ordering in materials. It depends on the law of Bragg in the form of Equation (1):

$$D = \frac{J\lambda}{\beta cos\theta} \quad (1)$$

Where, D – average crystallite size, J – shape factor, λ – wavelength of the X-ray source, β – full width at half maximum (FWHM) of the diffraction peak, $cos\theta$ – Bragg diffraction angle. Tensile testing was used to measure the elastic modulus, tensile strength, and elongation at break, and showed reinforcement, but with a high degree of flexibility.

4. RESULT AND DISCUSSION

This section provides the findings of SEM, AFM, XRD, and strength analyses that were conducted to indicate correlations of nanoparticle dispersion, structural changes, mechanical reinforcement, and electromechanical behavior of EVA-KNN and EVA-$LiNbO_3$ films.

4.1 Elastic Modulus

It is the ratio between stress and strain in the linear elastic region of a material, which represents stiffness. It is involved in determining how the rigidity and mechanical performance of EVA nanocomposite films are improved by nanoparticle doping, and is given by Equation (2):

$$F = \frac{\sigma}{\epsilon} = \frac{\frac{E}{B}}{\frac{\Delta K}{K_0}} \quad (2)$$

Where F – the elastic modulus, σ – the applied stress N/m^2, ϵ – the strain, E – the applied force, B – the original cross-sectional area of the specimen (m^2), ΔK – the change in length (m), and K_0 – the initial gauge length of the specimen (m).

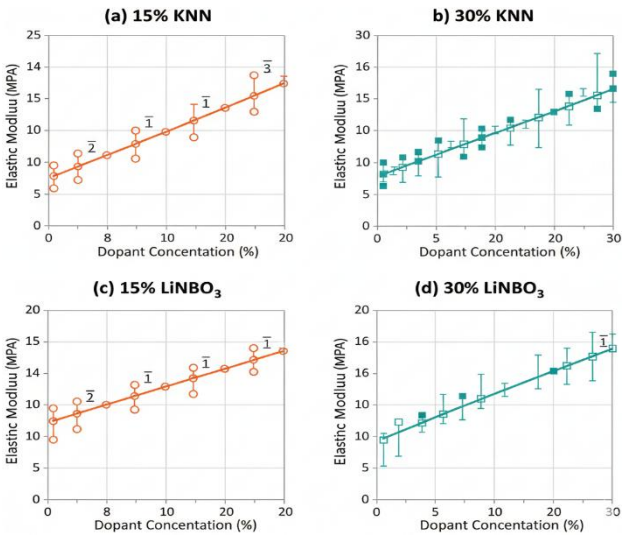

Fig. 1 – Elastic modulus variation with dopant concentration for (a) 15 % KNN, (b) 30 % KNN, (c) 15 % $LiNbO_3$, and (d) 30 % $LiNbO_3$ EVA nanocomposite films

Fig. 1 indicates that the elastic modulus of EVA nanocomposites continuously rises with dopant concentration in all systems. At 15 % KNN (a), the modulus increases between 6 and 15 Mpa, and at 30 % KNN (b), the modulus improves between 7 and 18 Mpa, which shows a good reinforcement. Likewise, 15 percent $LiNbO_3$ (c) adds a modulus between 9 and 15 MPa, and 30 percent $LiNbO_3$ (d) adds further between 10 and 17 MPa.

4.2 Tensile Strength

It is the maximum stress that a material can resist when stretched until it collapses. It is applied to determine the level of nanoparticle doping that improves the stability of EVA nanocomposite films. Tensile strength (σ_s) is calculated using Equation (3):

$$\sigma_s = \frac{E_{max}}{B} \quad (3)$$

Where σ_s – the tensile strength, E_{max} – the maximum force applied before fracture, and B – the original cross-sectional area of the specimen (m^2).

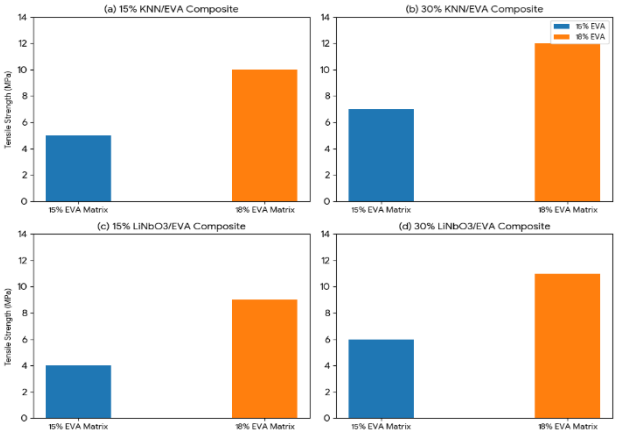

Fig. 2 – Tensile strength variation in (a) 15 % KNN/EVA, (b) 30 % KNN/EVA, (c) 15 % $LiNbO_3$/EVA, and (d) 30 % $LiNbO_3$/EVA nanocomposites

Fig. 2 depicts that the strength increases in (a) 15 % KNN, between 15 % EVA and 18 % EVA, and (b) 30 % KNN, between 6 MPa and 13 MPa, respectively. Likewise, (c) 15 % $LiNbO_3$ strengthens by a factor of about 4 MPa up to about 9 MPa, (d) 30 % $LiNbO_3$ strengthens again by a factor of about 6 MPa to about 12 MPa.

4.3 Elongation at Break

It is the force that can be held by a polymer film before breaking. The finding of high elongation proves that the doped EVA nanocomposite films have nanoparticle doping. Elongation at break (ϵ_a) is calculated using Equation (4):

$$\epsilon_a = \frac{\Delta K}{K_0} \times 100 \quad (4)$$

Where ϵ_a – the elongation at break (%), ΔK – change in length of the polymer film at the point of breaking, K_0 – original length of the polymer film before stretching.

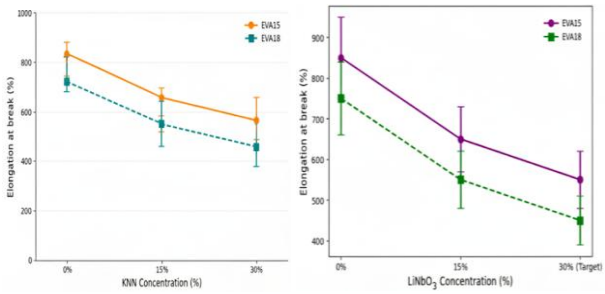

Fig. 3 – Elongation at break variation with dopant concentration for (a) EVA/KNN and (b) EVA/$LiNbO_3$ nanocomposite films

Fig. 3 indicates that both KNN and $LiNbO_3$ systems exhibit a negative dependence of elongation at break on dopant concentration. In EVA15 and EVA18 with KNN, the elongation decreases between 0 % and 30 % concentration of dopant (0 to 800 and 500 percent, respectively (a). In (b) EVA15 and EVA18 containing $LiNbO_3$, a more pronounced decrease is seen, between approximately 900 and 400-500 percent at 0 and 30 percent concentration, respectively.

4.4 Scanning Electron Microscopy (SEM)

It was used to analyze the surface morphology and nanoparticle dispersion in EVA films to ascertain a consistent incorporation of KNN and $LiNbO_3$.

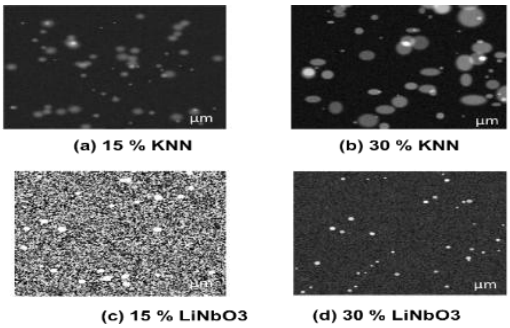

Fig. 4 – SEM of EVA 15 nanocomposites showing nanoparticle dispersion for (a) 15 % KNN, (b) 30 % KNN, (c) 15 % $LiNbO_3$, and (d) 30 % $LiNbO_3$, with corresponding property values

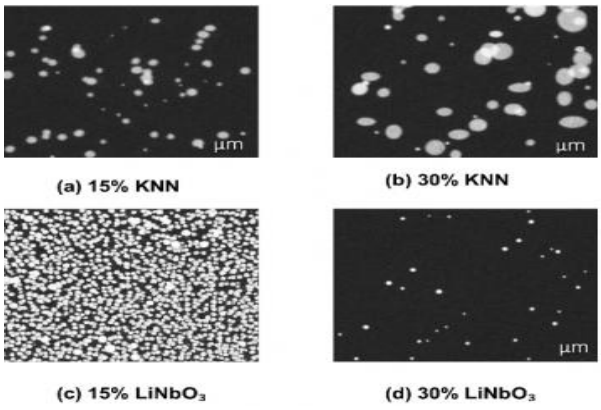

Fig. 5 – SEM of EVA 18 nanocomposites showing nanoparticle dispersion for (a) 15 % KNN, (b) 30 % KNN, (c) 15 % $LiNbO_3$, and (d) 30 % $LiNbO_3$, with corresponding property values

Fig. 4 shows the EVA 15. In (a) 15 % KNN, the unit particles are elastically supported by clusters of small size, promoting an elastic modulus of about 6-15 MPa and a tensile strength of about 4-9 MPa. In (b) 30 % KNN, the aggregates are larger, but reinforcement is stronger, with a modulus of approximately 7-18 Mpa and a tensile strength of approximately 13 MPa. In (c) 15 % $LiNbO_3$, the dense particle packing is noted, associated with a modulus of about 9-15 Mpa and a tensile strength of about 4-9 Mpa. The dispersion in (d) 30 % $LiNbO_3$ is more even with a smaller number of clusters, and it gives a modulus of 10-17 MPa and a tensile strength of 12 MPa.

Fig. 5 (a) and (b) displays the KNN particles at 15 % and 30 % doping, where particle size and aggregation increase with concentration. Fig. 5 (c) and (d) show $LiNbO_3$ particles at 15 % and 30 %, respectively, with 15 % having dense, uniform distribution, and 30 % showing sparse, scattered particles.

4.5 X-ray Diffraction (XRD)

It is employed for the crystalline structure and phase composition of EVA nanocomposite films. It shows the variation in the crystallinity of EVA, and it also confirms the presence of KNN and $LiNbO_3$ nanoparticles in the polymer structure.

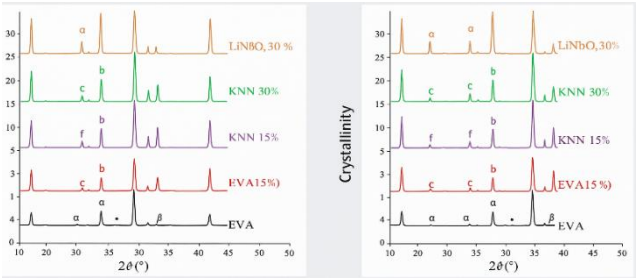

Fig. 6 – XRD patterns of (a) EVA15 and (b) EVA18 nanocomposites showing crystallinity variations with 15 % and 30 % KNN and $LiNbO_3$ doping

Fig.6 shows that the EVA nanocomposites doped with KNN and $LiNbO_3$ have a higher crystallinity. In (a) EVA15, pure EVA displays typical a and b peaks, whereas 15 % KNN and $LiNbO_3$ present sharper diffraction peaks at 22-32 °, which indicates increased ordering; 30 % doping further increases these peaks, which represent increased crystallinity. Equally, in (b) EVA18, the doped films have higher peaks around 20-35 °, and the crystallinity is the highest in 30 % $LiNbO_3$.

4.6 Atomic Force Microscopy (AFM)

It is a high-resolution imaging method applied to study the morphology and nanoscale of the surfaces of materials. The dispersion and surface interactions of piezoelectric nanoparticles in EVA films are assessed using AFM, which connects the structure and functional properties of the films.

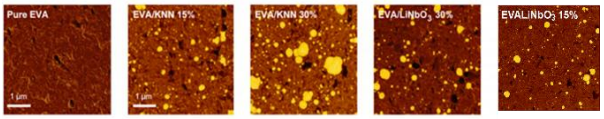

Fig. 7 – AFM images of EVA nanocomposite films showing nanoparticle dispersion

Fig.7 shows the surface morphology of EVA films with and without piezoelectric nanoparticles. Pure EVA exhibits a smooth surface, while the addition of 15 % KNN and 30 % $LiNbO_3$ nanoparticles increases surface roughness and particle aggregation, especially at higher loadings.

4.7 Discussion

The research was to establish the structure-property correlations in EVA nanocomposite films doped with piezoelectric metal oxide nanoparticles, KNN, and $LiNbO_3$. It was revealed that earlier researchers enhanced the optical and thermal characteristics of PMMA-ZnO films but did not examine long-term mechanical or device reliability [13]. Sodium alginate nanocomposites did improve crystallinity and antibacterial properties, but stability and food compatibility on storage were not sufficiently evaluated [14]. Carboxymethyl-CuO films had potential in energy storage and were poorly integrated into devices or in vivo applications [15]. PLA and gelatin/tapioca starch nanocomposites enhanced strength and antibacterial activity, yet were afflicted with aggregation of nanoparticles, scalability, and lack of real-storage validation. Uniform nanoparticle dispersion enhanced interfacial adhesion and crystallinity, as confirmed by SEM, AFM, and XRD. KNN incorporation resulted in higher stiffness and crystallinity due to strong electrostatic polymer-filler interactions, whereas $LiNbO_3$ provided better flexibility and smoother surface morphology. The tensile strength and modulus increased with nanoparticle loading, indicating efficient stress transfer, while ductility slightly declined due to restricted chain mobility. The observed trade-off between rigidity and elasticity highlights the tunable nature of EVA-based composites.

5. CONCLUSION

The structure-property correlations of EVA nanocomposite films doped with KNN and $LiNbO_3$ nanoparticles. Solvents were used to cast films, and then the films were extensively examined with the help of SEM, AFM, XRD, FTIR, and mechanical testing. The findings showed an improved crystallinity, tensile strength (2-13 MPa), 15 % KNN and 30 % $LiNbO_3$, and thermal stability; these results illustrate that EVA-based nanocomposites can balance flexibility, reinforcement, and electromechanical capabilities and become very promising in flexible electronics, sensors, energy harvesting, and biomedical applications. The investigation finds the KNN and $LiNbO_3$ doped EVA films to have configurable mechanical, structural, and piezoelectric performance relevant to applications in flexible electronic and biomedical devices. However, there is no mention of long-duration operational stability, scale-up, or in vivo evaluation. Future research should be diverted towards studies of durability, scale-up, and evaluation within a feasible biomaterial context.

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Полімерні нанокомпозитні плівки, леговані п'єзоелектричними наночастинками оксидів металів

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Полімерні нанокомпозитні плівки, леговані п'єзоелектричними наночастинками оксиду металу, привернули значну увагу завдяки їхньому потенційному застосуванню в гнучкій електроніці, сенсорах та пристроях збору енергії. Це дослідження досліджує кореляцію структури та властивостей у полімерних нанокомпозитних плівках, що складаються з етиленвінілацетату (EVA), легованого п'єзоелектричними наночастинками оксиду металу, зокрема ніобатом калію-натрію (KNN) та ніобатом літію ($LiNbO_3$). EVA, сополімер напівкристалічного поліетилену та аморфного вінілацетату, забезпечує регульовані властивості матеріалу, що робить його ідеальною матрицею для функціональних нанокомпозитів. Морфологічний аналіз за допомогою РЕМ та АФМ підтвердив однорідну дисперсію наночастинок у різних складах EVA. Структурна характеристика за допомогою XRD та FTIR виявила підвищену кристалічність та взаємодію полімер-наночастинки в легованих плівках. Механічні випробування показали міцність на розрив від 2 до 13 МПа для 15 % KNN та 30 % $LiNbO_3$, що свідчить про механічне армування при збереженні гнучкості. Ці результати демонструють потенціал плівок EVA, легованих KNN та LiNbO, для гнучкої електроніки, сенсорів, збирачів енергії та біомедичних застосувань, забезпечуючи настроювану платформу, де одночасно досягаються механічне підсилення та п'єзоелектрична чутливість.

Ключові слова: Етиленвінілацетат (EVA), Полімерні нанокомпозити, Ніобат калію-натрію (KNN), Ніобат літію ($LiNbO_3$), П'єзоелектричні наночастинки.