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14. ABSTRACT
Potassium sodium niobate, $K_{0.5}Na_{0.5}NbO_3$ (KNN), has emerged as a promising, lead-free, nontoxic, environment-friendly, and biocompatible material that is an alternative to high-performing, lead-based piezoelectric materials. We previously demonstrated a facile, fast, cost-effective, and scalable mechanochemical process for the synthesis of the KNN ceramic in powdered form. It is well known that polymer matrices, with relatively low thermal stability, can be enhanced by the incorporation of a secondary ceramic phase, while the functional characteristics of a brittle ceramic material can be integrated into the flexible continuous polymer matrix. Thus, rendering a polymer–ceramic composite with desirable thermal, mechanical, and functional characteristics for various applications such as actuators and sensors, where flexible and high-strength materials are required at relatively high temperatures. Here, we report our success fabricating KNN-based polymeric composite films to investigate their characteristics.

15. SUBJECT TERMS
Electromagnetic Spectrum Sciences; Mechanical Sciences; Photonics, Electronics, and Quantum Sciences; KNN; potassium sodium niobate; lead-free; piezoelectric; multifunctional; optical; nonlinear; polymer composite film; thin film; thick film

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1. Introduction

Piezoelectric materials generate internal charge in response to mechanical pressure[1] and have had applications in fields such as energy harvesting, pressure sensing, biomedical imaging, and nondestructive testing.[2–7] The best-known piezoelectric materials are lead-based ceramics, especially Pb(ZrxTi1-x)O_3 (PZT), which has demonstrated excellent dielectric and piezoelectric properties[8–11] but is also composed of more than 60% lead—a toxic heavy metal that causes irreversible damage to the human brain, kidneys, reproductive organs, etc. Furthermore, evaporation of lead oxide is inevitable during the processing of lead-based ceramics due to its low melting point (888 °C) and high vapor pressure. Lead toxicity builds up over months and years and remains in the bones, re-entering the bloodstream even after exposure ceases; the higher the exposure, the longer it lasts and the greater the damage. It cannot be easily expelled from the body.[12,13] In addition, the ecological damage caused by lead toxicity is tremendous[14] (e.g., decreased growth and reproduction in plants and animals and neurological effects in vertebrates). Hence, there has been a continuous avid search for lead-free alternatives across the world for a wide array of electronic applications. Previously, we have demonstrated and reported a facile, fast, cost-effective, and scalable mechanochemical process for synthesis of the lead-free, nontoxic, environment-friendly, multifunctional, biocompatible, and piezoelectric perovskite KNN integrated with tungsten (W-KNN) in powdered form.[15]

Composite materials have demonstrated multiple physical and mechanical properties.[16] Specifically, polymer–ceramic composites with the ceramic filler in a polymer matrix provide simultaneous strength and flexibility and have been investigated with an array of material systems. The secondary ceramic phase has been proven to enhance the polymer matrix, which has the disadvantage of a relatively low thermal stability while the flexible continuous polymer matrix could be beneficial to overcome the brittleness of the ceramic material. A polymer–ceramic composite with flexibility, high strength, and the ability to render functionalities at relatively high temperatures finds suitability in various applications such as actuators and sensors.[1] Therefore, in this work, with KNN also known to be a multifunctional material, we fabricate KNN-based composites using US Army Combat Capabilities Development Command (DEVCOM) Army Research Laboratory (ARL)-synthesized W-KNN in matrices of 2D polymers 1,3,5-tris(4-aminophenyl)benzene (TAPB) and 1,3,6,8-tetrakis(4-aminophenyl) pyrene (TAPPy) (also synthesized at ARL), and the well-known piezoelectric polymer polyvinylidene fluoride (PVDF), and characterize their properties.

As reported,[15] solid-state synthesis of W-KNN is achieved by the reaction summarized in Eq. 1, between the alkali carbonates and niobium oxide, which occurs between 400 and 700 °C as determined by thermogravimetric analysis (TGA)[17] with calcination occurring between 750 and 950 °C.

$$K_2CO_3(s) + Na_2CO_3(s) + 2\ Nb_2O_5(s) \rightarrow 4\ K_{0.5}Na_{0.5}NbO_3(s) + 2\ CO_2(g) \quad (1)$$

A coupled diffusion of alkaline and oxygen ions into niobium oxide is followed by the formation of the perovskite phase after an intermediate alkali polyniobate phase, $(K,Na)2Nb_4O_{11}$. Multiple phases other than the perovskite can be expected to form in these reaction(s).[18–22] The overall reaction rate in the ternary system is dependent on the diffusion rate and path of the slower potassium ions.[23] Also, as in any diffusion-controlled reaction, smaller particle sizes lead to shorter diffusion paths and lower input energy (heat) for the reaction.[24]

At ARL, as previously reported,[15] and using the process outlined by Lee et al.[25] as a guideline, we synthesized KNN using a Spex 8000M high-energy mixer/mill. The high-energy ball mill grinds the material in a closed vial via combined back-and-forth swings with short lateral movements, with the ends of the vial forming a figure eight for relatively uniform milling. K_2CO_3, Na_2CO_3, and Nb_2O_5 were mixed in the stoichiometric ratio and dry-milled using a tungsten-carbide vial and milling media. All powders were stored, weighed, and loaded into the vial inside the glove box, in an argon atmosphere.

TGA/differential scanning calorimetry of the 1-h milled powder determined the calcination temperature to be around 650 °C at the completion of the reaction, which is lower than the approximately 850 °C temperature found in other processes.[25,26] The resulting powder mixture from the milling process was then calcined at a temperature of 650 °C for 1 h to complete the mechanochemical reaction to achieve W-KNN. The calcined powders were then studied using X-ray diffraction, scanning electron microscopy, energy-dispersive X-ray, transmission electron microscopy, and energy dispersive X-ray spectroscopy to verify the material's structure and composition. More details on the W-KNN synthesis and material characterization are outlined in our earlier publication.[15]

2. Composite Fabrication

The synthesized KNN powder was then integrated with various polymers to obtain composite films of KNN.

PVDF is the most widely used piezoelectric polymer due to its piezoelectricity, thermoelectricity, and high dielectric properties.[27,28] Piezoelectric ceramics such as KNN intrinsically have superior piezoelectric

characteristics compared to piezoelectric polymers. Piezoelectric ceramics and PVDF composition polymers are not only efficient for obtaining β-phase PVDF, which has demonstrated better piezoelectric characteristics than other phases of PVDF, but piezoelectric ceramics have also demonstrated synergistic effects that enhance the piezoelectric performance of polymeric devices.[29,30] Piezoelectric ceramics-embedded PVDF nanocomposite thin films have demonstrated larger piezoelectric coefficients and improved response in comparison with the neat PVDF thin films.[31] We therefore fabricated a PVDF/KNN composite film using the newly synthesized W-KNN at ARL to explore its potential for US Army applications.

Composite films marrying transparent, robust material and absorbent or optically active additives allow for the design and synthesis of engineering films with tunable optical properties. Two-dimensional polymers are planar; thus, ordered molecules have emerged as a new multifunctional and composite material due to their near-infinite functionalization. A commonality among 2-D polymers is their remarkable surface area due to their inherent ordered molecular porosity, often with pore sizes in the range of 1–10 nm and surface areas well over 2000 m^2/g, making them particularly well-suited for forming composites. ARL has the unique capability of creating robust 2-D polymers with measured surface areas of 2400 m^2/g, which are transparent in the near-to-mid IR. KNN has been shown to have photonically induced charge transport and photoluminescence with tunable band gaps from nickel doping, and KNN is compatible with the 2-D polymer film synthesis process, making them a suitable active additive for optically tuned composites. Given that the band gap and absorption of KNN can be potentially tuned, lightweight composite films with tuned optical absorption could be achieved.

Specifically for this work, monomer combinations of 1) TAPPy and terephthalaldehyde (PDA), and 2) TAPB and PDA were used to form the 2-D polymers TAPPy–PDA and TAPB–PDA, respectively. Polymeric composites of KNN with the synthesized 2-D polymers TAPPy–PDA and TAPB–PDA, and the piezoelectric polymer PVDF, were synthesized and characterized as follows.

2.1 Two-Dimensional Polymer Films

Two-dimensional polymer films were cast by dissolving 150 mg of constituent monomers plus 5% and 20% monomer weight equivalent of KNN per milliliter of solution consisting of 85% trifluoroacetic acid with 5% water. Solutions were mixed and then cast using the doctor blade technique and allowed to dry in air for more than 10 min before being washed in baths of 95% ethanol and 5% triethylamine. Two 2-D polymer monomer combinations were prepared: TAPPy

and PDA to form TAPPy–PDA, and TAPB and PDA to form TAPB–PDA. Examples of the TAPPy–PDA films with 5% and 20% KNN photographed under normal and cross-polarized light can be seen in Fig. 1. The KNN did not readily dissolve in the solution, appeared to maintain its structure, and dispersed well; thus, creating uniform films that at 5% appeared homogeneous and 20% heterogeneous, with domains of varying diffraction as seen in Fig. 1d.

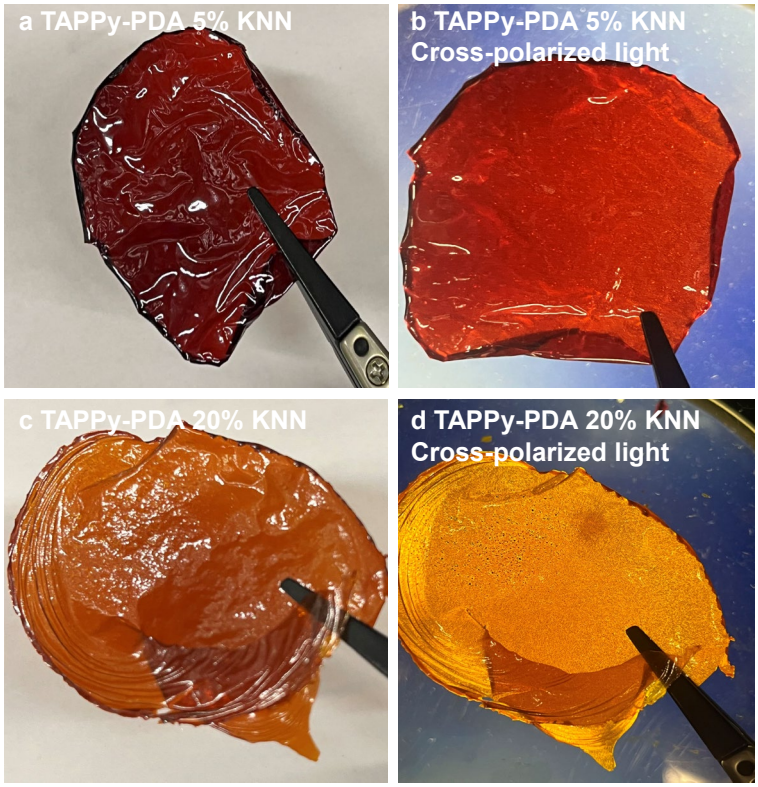

Fig. 1 Films of TAPPy–PDA with 5% KNN in a) normal light b) cross-polarized light and 20% KNN in c) normal light and d) cross-polarized light

The TAPPy–PDA/KNN composite films were further examined in a Keyence VHX7000 high-powered optical microscope with cross-polarization capability. A typical micrograph is presented in Fig. 2, with the 20% KNN TAPPy–PDA film being fairly representative of the KNN particle and 2-D polymer film morphology. Figure 2a shows the 2-D polymer/KNN composite under mixed lighting (confocal, ring, transmission); a zoomed-in view of the rectangular particulate is shown in Fig. 2b under the same lighting, displaying a faint yellow hue that is associated with birefringence in the TAPPy–PDA 2-D polymer. Increasing birefringence in semicrystalline polymers is correlated to increasing molecular orientation; as larger and larger domains of polymer become ordered, light refraction becomes increasingly dependent on the polarization of light and the angle of incidence with the polymer. Birefringence in the 2-D polymer/KNN composite can also be seen in

full ring lighting mode (Fig. 2c), again showing birefringence in the 2-D polymer adhered to the KNN particulate faces, as well as full transmission mode (Fig. 2d), which shows yellow birefringent domains in a clover-leaf pattern around some of the KNN particulates that are represented as large dark domains. The clover-leaf birefringence pattern is well documented in polymers as a result of stress concentrations that orient polymer molecules, indicating that the KNN particulates are serving as a possible source of prestrain in the 2-D polymer film, leading to regions of higher orientation.

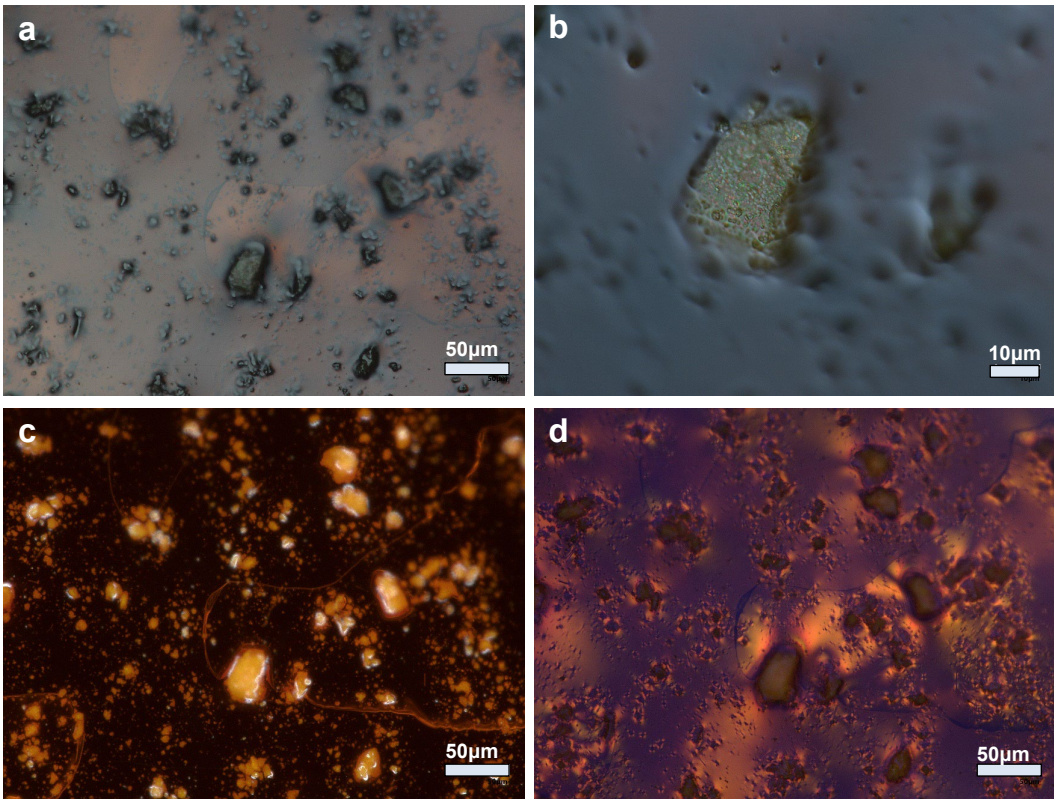

Fig. 2 Cross-polarized optical microscopy of TAPPy–PDA films with 20% wt KNN in a) all lighting modes: transmission, ring, confocal; b) zoomed-in view of KNN particles in all lighting modes; c) the same region in ring-lighting mode; and d) the same region in transmission lighting mode

2.2 X-ray Spectroscopy

Small- and wide-angle X-ray spectroscopy (SAXS, WAXS) were performed on the 2-D polymer/KNN composite films. SAXS measurements were taken at 1800-, 900-, and 370-mm camera lengths and 0.5- × 0.25-mm collimation. The WAXS measurement was taken at 55-mm camera length and 0.5- × 0.25-mm collimation. The TAPPy–PDA 5% and 20% KNN films show excellent 2-D polymer structure, in wavenumber ranges of $q \sim 0.5$ –1.6 $Å^{-1}$, and show ordering of the KNN at $q \sim$ 1.5 and 2.2 $Å^{-1}$, indicating that the KNN survived the film-casting process and the

2-D polymer was able to form in the presence of the KNN particles as seen in Fig. 3. The resulting 2-D polymer peaks in the X-ray spectra showed not only tetragonal crystalline unit cells characteristic of TAPPy–PDA, but also excellent long-range order and stacking.

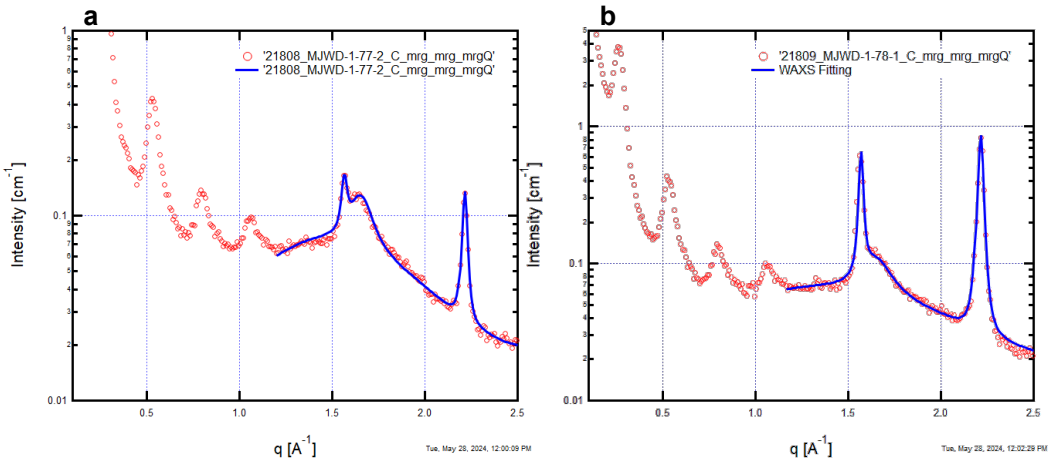

Fig. 3 SAXS and WAXS of films: a) TAPPy–PDA with 5% wt KNN and b) TAPPy–PDA with 20% wt KNN

2.3 UV–Vis Spectroscopy

UV–vis spectroscopy was performed on the TAPPy–PDA/KNN composite films sweeping wavelengths of 300–2400 nm, as seen in Fig. 4. The 2-D polymer/KNN composite films show a progressively increasing shift in reflection, increasing from 5% to 20% wt KNN, without adding any obvious absorption peaks into the spectral response (Fig. 4a). There is a distinct reflection peak response at around 450 nm in reflection (Fig. 4b) that is shifted from the neat films. In addition to no distinct absorption peaks appearing, the broad absorption of the 2-D polymer/KNN composite films does not change significantly with added KNN content, beginning at about 600 nm.

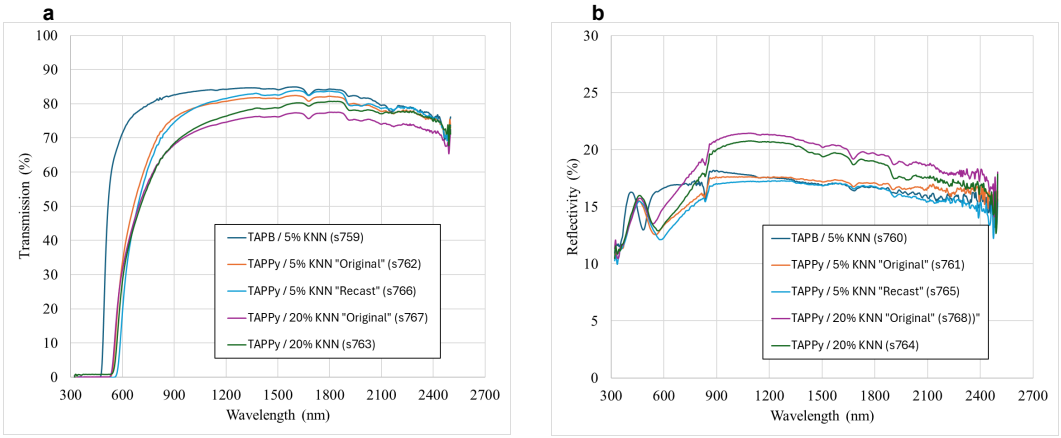

Fig. 4 UV–vis spectroscopy of TAPPy–PDA films with 5% and 20% wt KNN in a) transmission and b) reflection

2.4 PVDF Composites

2.4.1 Thin Films

PVDF powder was magnetically stirred in dimethylformamide (DMF) at a 15 wt% concentration until all PVDF was dissolved. The PVDF/DMF solution was cast onto a glass slide and enclosed using a silicone border. The cast solution was then submerged into a bath of deionized (DI) water to remove the DMF. The thin films were submerged for 1 h. The resulting thin films were then removed and dried in air for 24 h. Composite thin films containing KNN at varying concentrations (10 wt%, 20 wt%, 30 wt%, 40 wt%, 50 wt%) were fabricated using the same casting approach after KNN was mixed into a solution of PVDF/DMF.

2.4.2 Wet-spun Fibers

After obtaining solutions for PVDF/DMF or PVDF/KNN/DMF using the same approach as with the fabrication of thin films, the solution was transferred to a syringe with a needle gauge of 26 (inner diameter of 0.30 mm). A syringe pump positioned vertically to a DI water bath was used to extrude the solution through the needle, as shown in Fig. 5. The syringe needle was submerged in a rotating DI water bath. The solution was then spun into the DI water at a rate of 3 mL/h. Continuous neat PVDF or PVDF/KNN fibers were collected. The fibers were then washed with DI water and dried in air.

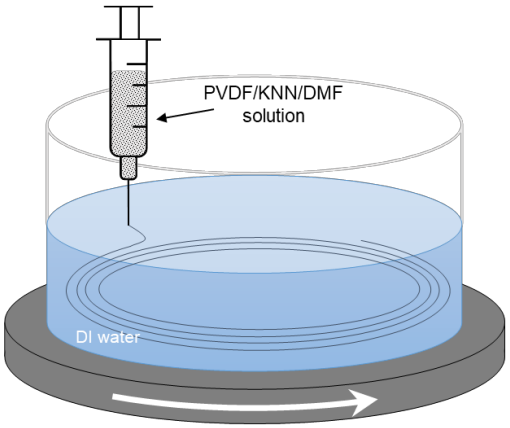

Fig. 5 Schematic of the wet spinning apparatus

The PVDF/KNN films did not demonstrate any optical transmission characteristics during the UV–vis spectroscopy—unlike the 2-D polymeric films. Further characterization of these composites is in progress for various applications.

3. Conclusions

W-KNN–based polymeric composite films and fibers were successfully fabricated. The KNN appeared to maintain its structure and rendered well-dispersed uniform films that at 5% appeared homogeneous and 20% heterogeneous, with domains of varying diffraction. Birefringence was observed in the 2-D polymer adhered to the KNN particulate faces, as well as full transmission mode. Birefringent domains in a clover-leaf pattern were observed around some of the KNN particulates as a result of stress concentrations that orient polymer molecules, indicating that the KNN particulates are serving as a possible source of prestrain in the 2-D polymer film, leading to regions of higher orientation. UV–vis spectroscopy indicated broad absorption of the 2-D polymer/KNN composite films does not change significantly with added KNN content. Further characterization of all synthesized composites is underway to explore the path forward for a variety of US Army applications.

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List of Symbols, Abbreviations, and Acronyms

2-D	two-dimensional
ARL	Army Research Laboratory
DEVCOM	US Army Combat Capabilities Development Command
DI	deionized
DMF	dimethylformamide
IR	infrared
KNN	potassium sodium niobate, $K_{0.5}Na_{0.5}NbO_3$
PDA	terephthalaldehyde
PVDF	polyvinylidene fluoride
PZT	Pb(ZrxTi1-x)O_3
SAXS	small-angle X-ray spectroscopy
TAPB	1,3,5-tris(4-aminophenyl)benzene
TAPPy	1,3,6,8-tetrakis(4-aminophenyl)pyrene
TGA	thermogravimetric analysis
UV	ultraviolet
WAXS	wide-angle X-ray spectroscopy
W-KNN	piezoelectric perovskite KNN, integrated with tungsten

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