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Original research

Stretchable electrospun PVDF/TPU nanofibers membranes: Acoustic signals detectors

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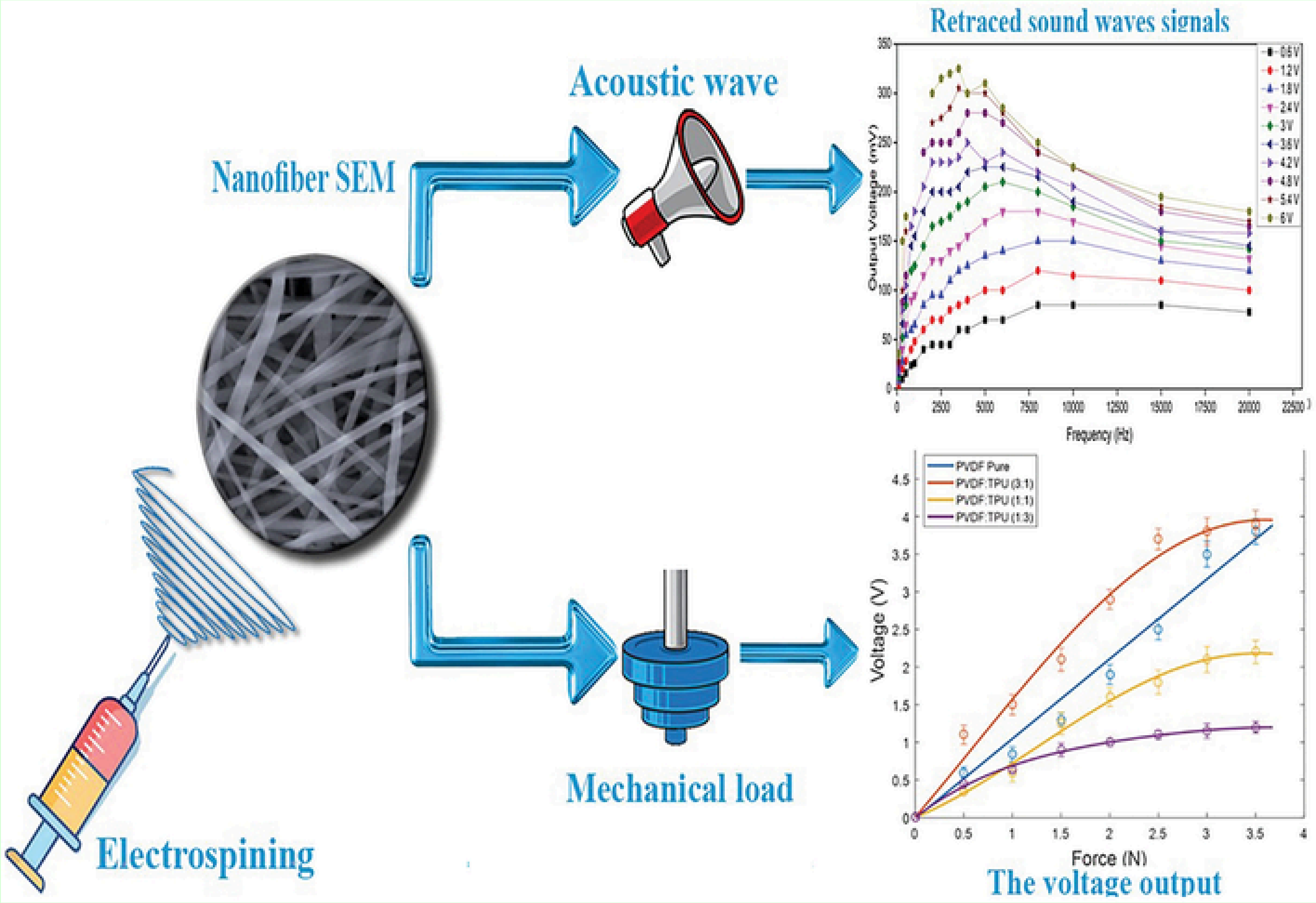
Abstract

Devices that harvest energy are crucial for reducing reliance on energy transmission and distribution systems. This helps minimize energy loss and mitigate environmental impacts. In this study, we focused on manufacturing nanocomposites using various ratios of polyvinylidene fluoride (PVDF) and thermoplastic polyurethane (TPU). PVDF, a flexible polymer, shows promise for nanogenerator applications, especially if its piezoelectric properties are enhanced. The nanofiber mat used as a transducer converts sound into electrical signals. We examined the electrical output from auditory signal excitation, analyzing the frequency of applied acoustic signals. The detected electrical signal was compared to the acoustic input signal in terms of frequency and wave distortion. PVDF, known for its piezoelectric capability, can convert mechanical or acoustic stress into electrical voltage. TPU, known for its exceptional flexibility, is widely used in the plastics sector. Our research explored the piezoresponse of nanofiber membranes with different PVDF/TPU ratios. TPU's superior mechanical stretchability enhances the piezoelectric sensitivity of PVDF/TPU nanofiber mats. This study introduces a novel application of piezoelectric electrospinning nanofiber membranes as acoustic signal detectors. Our results demonstrate that these membranes provide a promising, cost-effective, and innovative solution for capturing acoustic signals.

Graphical abstract



Highlights

Manufacturing nanofibers from piezoelectric materials, studying the effects of mechanical loads and sound waves on these fibers, and enhancing their functional properties by adding TPU to increase flexibility for use in sensing and energy applications.

Discussion

Our findings support the growing interest of green energy and energy harvesting new trends as it focuses on the conversion of kinetic and acoustic energies into electrical energy, thereby promoting sustainable energy sources.

The obtained date paves the way for next generation disruptive and sustainable schemes for extracting acoustic signals for potential market development in the supply chains involved usage of piezoelectric membrane in acoustic detector, energy harvesting from sounds, and wearable electronics sensors.

On focus of the assessments of PVDF/TPU nanofiber membranes as acoustic signal detectors, this research opens the need of collaborative research between academic and industrial communities to addressing carbon footprints and socio-economic implications in a globalized marketplace.

Keywords

- Acoustic waves
- Fiber
- Piezo-electric
- Sensor
- Stretchable
- Polymer

Introduction

Green technology is an innovative solution that converts energy from human movement, vibrations, and mechanical loads into usable power, addressing the global energy crisis and promoting sustainable economic growth.[1] Green technology harnessing mechanical force is crucial for sustaining wireless circuits, remote sensors, and portable battery devices.[2] They harness green electrical energy from various environmental systems.[3] Acoustic sensing applications use principles like piezo-resistance,[4, 5] piezo-capacitance,[4, 6] piezo-optics,[4, 7] and piezoelectricity.[4, 8, 9] Synthetic polymer piezoelectric acoustic sensors are highly sensitive and flexible.[10] Acoustic sensors are used in medicine, environment, industry, and scientific studies.[11]

Materials with piezoelectric capabilities have garnered researchers’ interest for energy conversion applications[12, 13, 14, 15] by converting structural deformation into electrical signals.[16, 17]

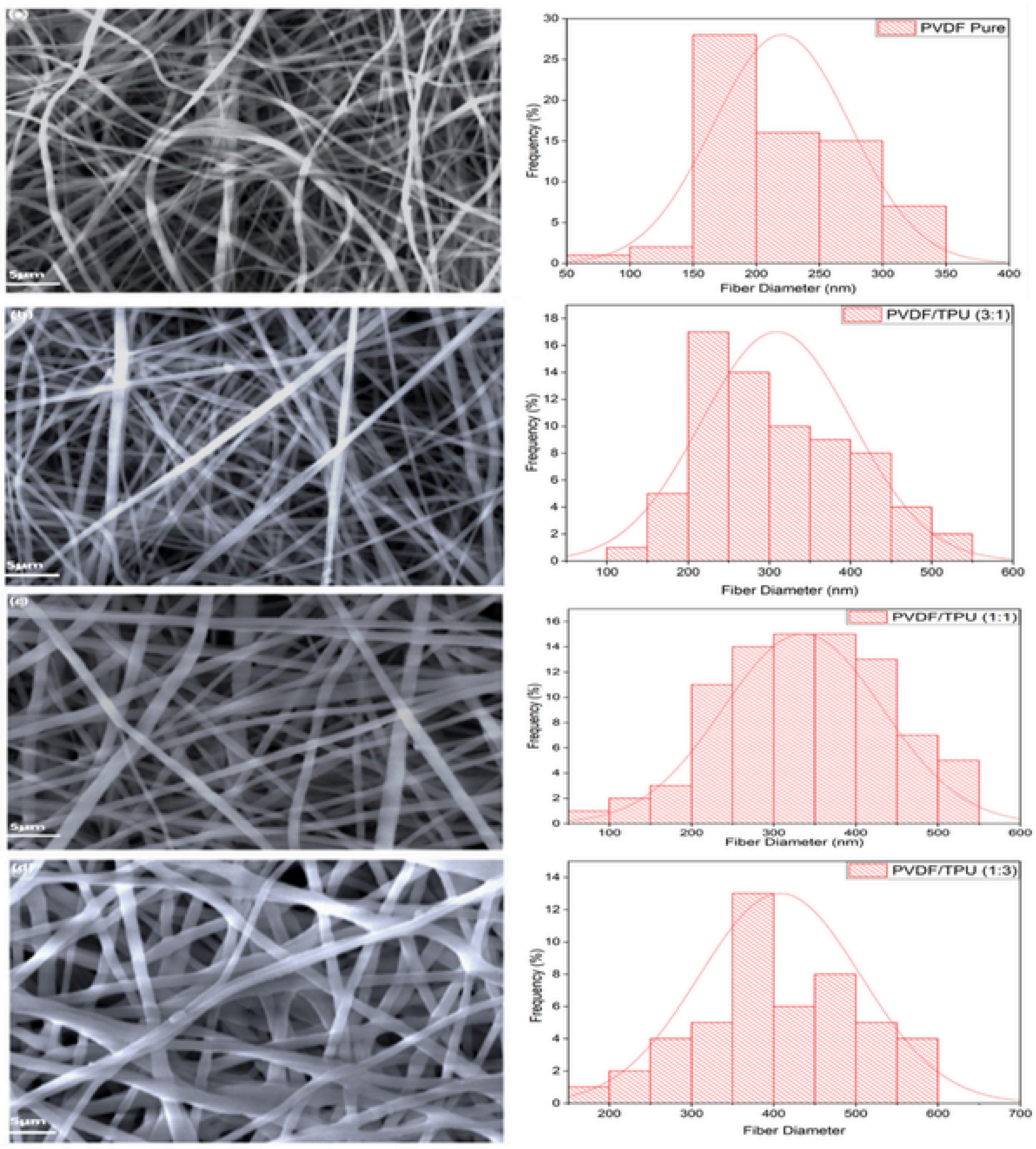
Ceramic-based materials, such as lead zirconate titanate (PZT),[18, 19, 20, 21] have been widely used in civil engineering infrastructure and structural health monitoring applications.[22] However, PZT’s intrinsic brittleness limits its tension level, posing a significant obstacle for use in adaptable electronic equipment or high-strain applications like civilian infrastructure.[23]

Due to the differently charged fluorine and hydrogen atoms in polymeric chains, solution-processed polymers like PVDF[24] and PVDF-TrFE[25] have piqued researchers’ interest as flexible piezoelectric materials. Innovative methods like electrospinning aligned fibers[25] and crystal orientation regulation,[26] along with combining inorganic piezoelectric with conductivity-based carbon substances,[27] have enhanced these materials' piezoelectric responses.[28] PVDF is a widely commercialized piezoelectric material due to its high flexibility and piezoelectric properties.[29, 30] With a piezoelectric coefficient ten times greater than ceramics, PVDF is ideal for sensing applications.[23, 31, 32] It boasts a range of beneficial properties, including purity, lightweight, elasticity, and resistance to solvents and high electric fields.[33] Over recent decades, various PVDF fiber production technologies have emerged.[16, 34, 35] PVDF's unique properties, including biocompatibility,[1] flexibility,[36] chemical resistance,[37] high mechanical strength, UV and nuclear radiation resistance,[38] and a low dielectric constant,[39] make it suitable for actuators, sensors, and biosensors.[1]

Electrospinning is crucial for producing PVDF nanofibers, enhancing piezoelectric sensitivity by aligning them in strong electric fields.[3, 16] Research focuses on optimizing β-phase content, material composition, and structural design to boost performance.[40, 41, 42, 43, 44] This technique, shown in Fig. 1 of the supplementary file,[45] produces nano or micrometric fibers and is valued for its cost-effectiveness and wide applications,[46] aiming for properties like sub-micrometer diameters, high porosity, and improved mechanical strength.[47, 48, 49, 50]

Figure 1

SEM images of PVDF/TPU Composite nanofiber samples with fiber diameter distributions for the following samples (a) PVDF (b) PVDF/ TPU 3:1 (c) PVDF/ TPU 1:1 and (d) PVDF/ TPU 1:3 all at a scale bar of 5 µm.



Various polymers, such as polyvinyl alcohol (PVA) for food packaging[51, 52, 53] and polylactic acid (PLA) for medical uses,[54, 55] are utilized in nanofiber fabrication. Thermoplastic polyurethane (TPU) enhances PVDF with its elasticity and hydrophilicity,[12, 34, 56, 57] making it suitable for applications requiring high mechanical properties, including blood clotting,[58] purification,[59] and biosensing.[60] TPU's hydrophilic carboxyl groups and excellent mechanical qualities offer benefits like elastic strain recovery and wear resistance.[3, 61, 62, 63, 64, 65]

This paper investigates converting acoustic energy into electrical energy using polymeric nanofibers. It highlights the lack of research on electrospun piezoelastic nanocomposites for acoustics. A new prototype combining piezoactive PVDF with stretchable TPU aims to enhance acoustic energy harvesting and sensing. Electrospun PVDF/TPU mats effectively detect acoustic signals with minimal distortion.

Results and discussion

Morphological characterization

The SEM analysis examines the morphology of PVDF/TPU composite nanofibers, with Table 1 showing their average diameters. Images in Fig. 1a–d reveal the effect of TPU addition on fiber synthesis, demonstrating that optimal spinning conditions and uniform polymer mixing yield evenly dispersed fibers without beads, as illustrated in Fig. 4. The polymer solution concentration and spinning parameters significantly impact nanofiber morphology. Incorporating TPU into PVDF/TPU composites increases fiber diameter and dispersion compared to pure PVDF. For instance, pure PVDF nanofibers average 220 nm in diameter, whereas PVDF/TPU (1:1) nanofibers average 336 nm. The increase in diameter with higher TPU concentrations is attributed to TPU's higher molecular weight, which enhances viscoelastic forces in the spinning fluid, leading to greater fiber diameters due to increased chain entanglement and viscosity.[66]

Table 1

The average diameter size of nanofibers for various PVDF/TPU composite membranes.

Sample	PVDF PURE	PVDF/TPU (3:1)	PVDF/TPU (1:1)	PVDF/TPU (1:3)
Average fiber diameter (nm)	220 ± 30	310 ± 15	336 ± 39	408 ± 19

Nanocomposite characterization

The FT-IR spectra in Fig. 2a show PVDF bands at 838 cm^{-1} (CH2 rocking), 1175 cm^{-1} (C–F vibrations), and 1400 cm^{-1} (C–H vibrations), with additional TPU bands at 1527, 1217, and 1726 cm^{-1} corresponding to –CONH–, C=C, C–O, and C=O stretching.[67, 68, 69, 70] FTIR spectroscopy reveals PVDF's electroactive phases, including the α, β, γ, δ, and ε phases, with the β-phase enhancing piezoelectric properties. The β-phase dipoles align perpendicularly to the chain axis for spontaneous polarization. The β-phase concentration increases with higher electric fields, determined using the Beer-Lambert law.

$$F(\beta) = \frac{A_\beta}{1.26A_\alpha + A_\beta} \tag{1}$$

where F (β) is the β phase content fraction, $\boxed{A_\alpha}$ and $\boxed{A_\beta}$ correspond to absorbance intensities of 760 and 838 cm^{-1}, respectively. The β-phase content in PVDF/TPU composites was determined using absorbance intensities at 760 and 838 cm^{-1}, with pure PVDF showing 76.53% and a 25% TPU content increasing it to 81.96% before decreasing with higher TPU content. The optimal composition is PVDF: TPU at a 3:1 ratio, as TPU enhances dipole reorientation but excess TPU reduces β-phase content.[3, 42, 71] The significant PVDF peaks produced from the XRD analysis support the piezoelectric activity of combining PVDF/TPU composites as clarified in Fig. 2b. XRD and FTIR analyses confirm that this composition maintains piezoelectric properties, with the highest polymerization degree achieved at PVDF/TPU (3:1). A tensile strain test was done to investigate its mechanical characteristics of the latterly created composite, and the resulting stress–strain curve is given in Fig. 2cThe finest mechanical qualities are provided by PVDF/TPU (1:3), which has a maximum stress of 41.33 MPa and a breaking elongation of 54.3%. TPU's strong mechanical properties as a high tensile stress and flexibility elastomer polymer account for all of this. The maximum stress and elongation during the breakage of PVDF are 6.29 MPa and 11.1%, respectively, as shown in Table 2. However, the maximum stress and elongation when the break occurs of PVDF/TPU (3:1) sample are 16.21 MPa and 14.1%, respectively. This indicates that adding 25% TPU doubles the stress while increasing the elongation at break and doubling the sample's elasticity. When the TPU ratio is increased to 50%, as in PVDF/TPU (1:1), the maximal stress can increase thrice, whereas the break point elongation rises three and a half times over the pure PVDF nanofiber sample.

Figure 2

a FT-IR, **b** XRD and **c** mechanical analysis of PVDF/TPU composite nanofiber.

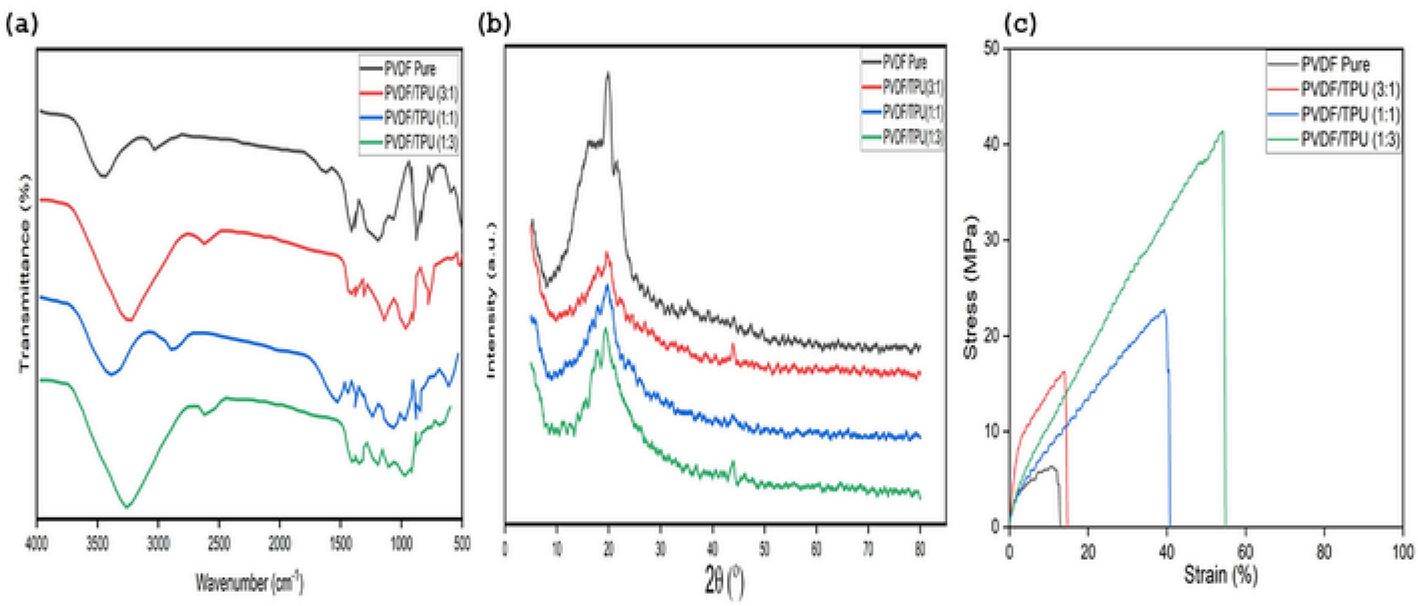


Table 2

Membrane mechanical characteristics of nanofiber composites.

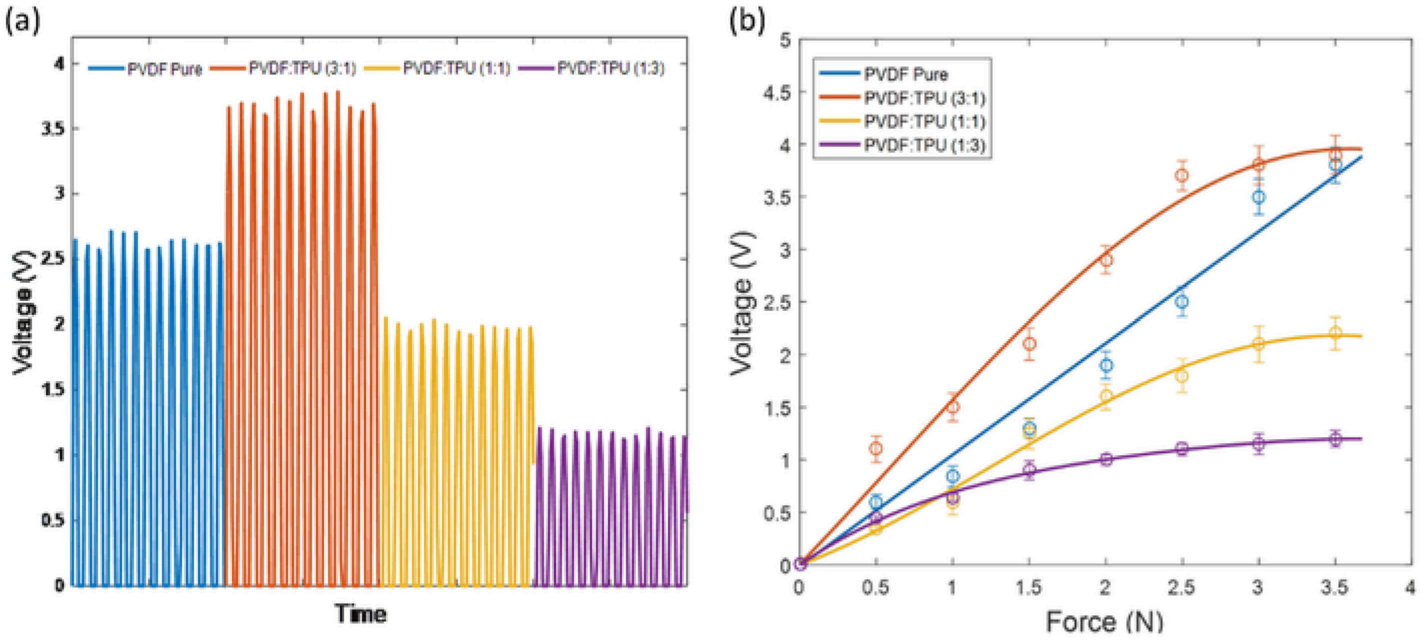
Sample	Max. stress, (MPa)	Elongation strain at break, (%)	Young's modulus (MPa)
PVDF Pure	6.29	11.1	0.73083 ± 0.06463
PVDF/TPU (3:1)	16.21	14.1	0.89825 ± 0.04948
PVDF/TPU (1:1)	22.77	39.3	0.55319 ± 0.00443
PVDF/TPU (1:3)	41.33	54.3	0.72412 ± 0.00331

Piezoelectric analysis

In this section, the output voltages of the PVDF/TPU characterizations are displayed such as the impulse response with different weights, the cyclic force with different frequencies, and the bending response as shown in Fig. 2 of the supplementary file. When analysis of peak-to-peak voltages when applied a mechanical forces at a frequency of 1 Hz, we found that the PVDF/TPU (3:1) has the best force—voltage characterization compared to other samples as shown in Fig. 3a. In terms of the impulse response with force-voltage characterization of the PVDF/TPU with various concentrations, the output voltage increases as long as the force increases as shown in Fig. 3b. However, the force-voltage curve increases linearly for the pure PVDF, while the PVDF increases with saturation behavior for different TPU blendings. In addition, the slope of the PVDF/TPU (3: 1) at range of 0: 2.5 N is linear and higher than the pure PVDF, which means that it **AQ1** has higher sensitivity than the pure PVDF within this range because of the elasticity.[72]

Figure 3

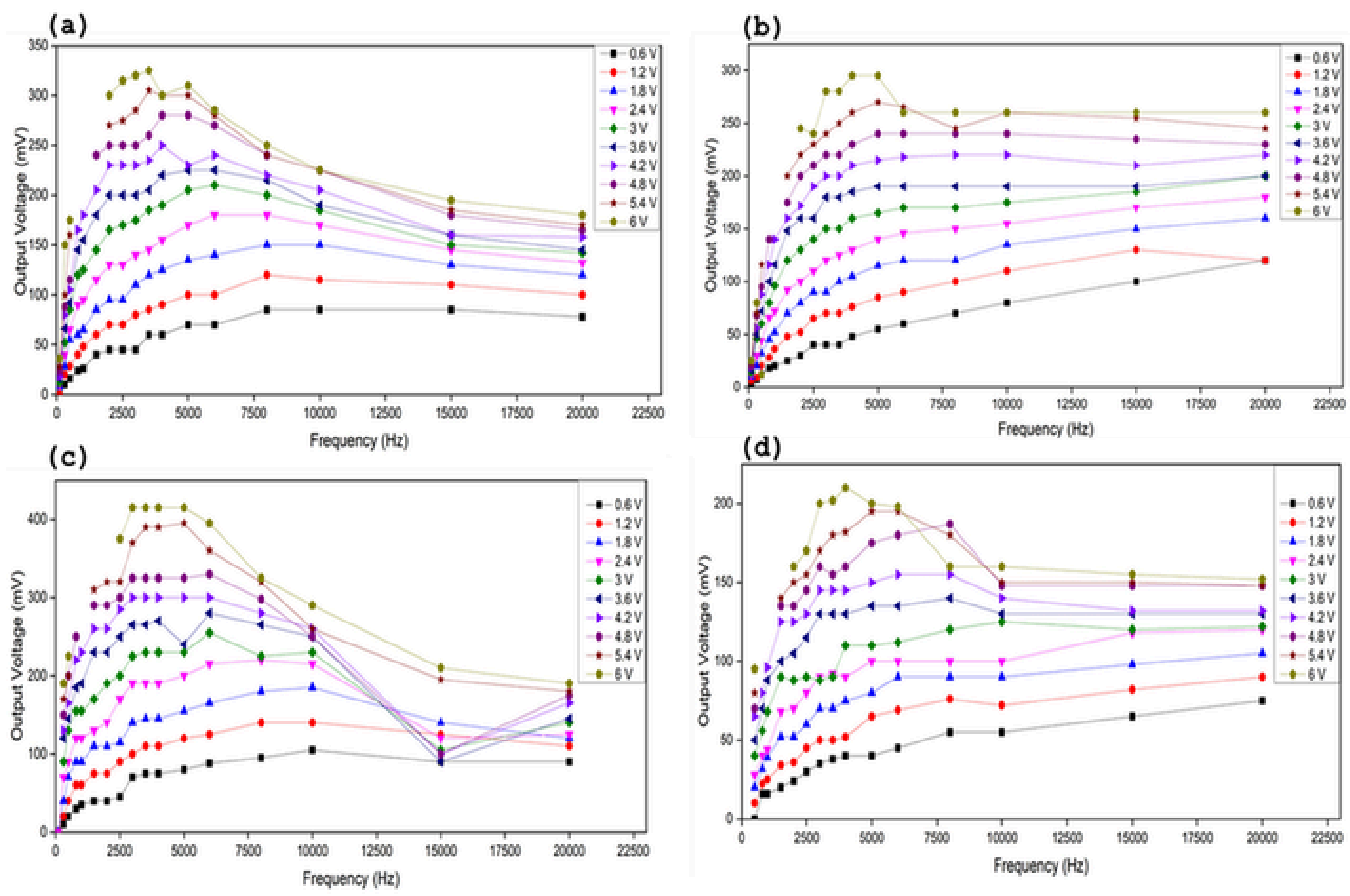
(a) analysis of peak-to-peak voltages versus applied mechanical forces at a frequency of 1 Hz and (b) The voltage output with different concentration at 2.5N.



For the frequency response, the maximum average output voltage is 5.8 V at the 1.5 Hz for the PVDF/TPU (3:1) as shown in Fig. 4a of supplementary file. The output voltages increase proportional to the frequency at applied constant force of value 3.5N as shown if Fig. 4b of supplementary file. It is worth noting that the pure PVDF saturates at 2 Hz as long as the frequency increases, while the PVDF/TPU (3:1) presents a peak at 1.5 Hz which disappears and tends to be saturated with increasing the TPU concentration.

Figure 4

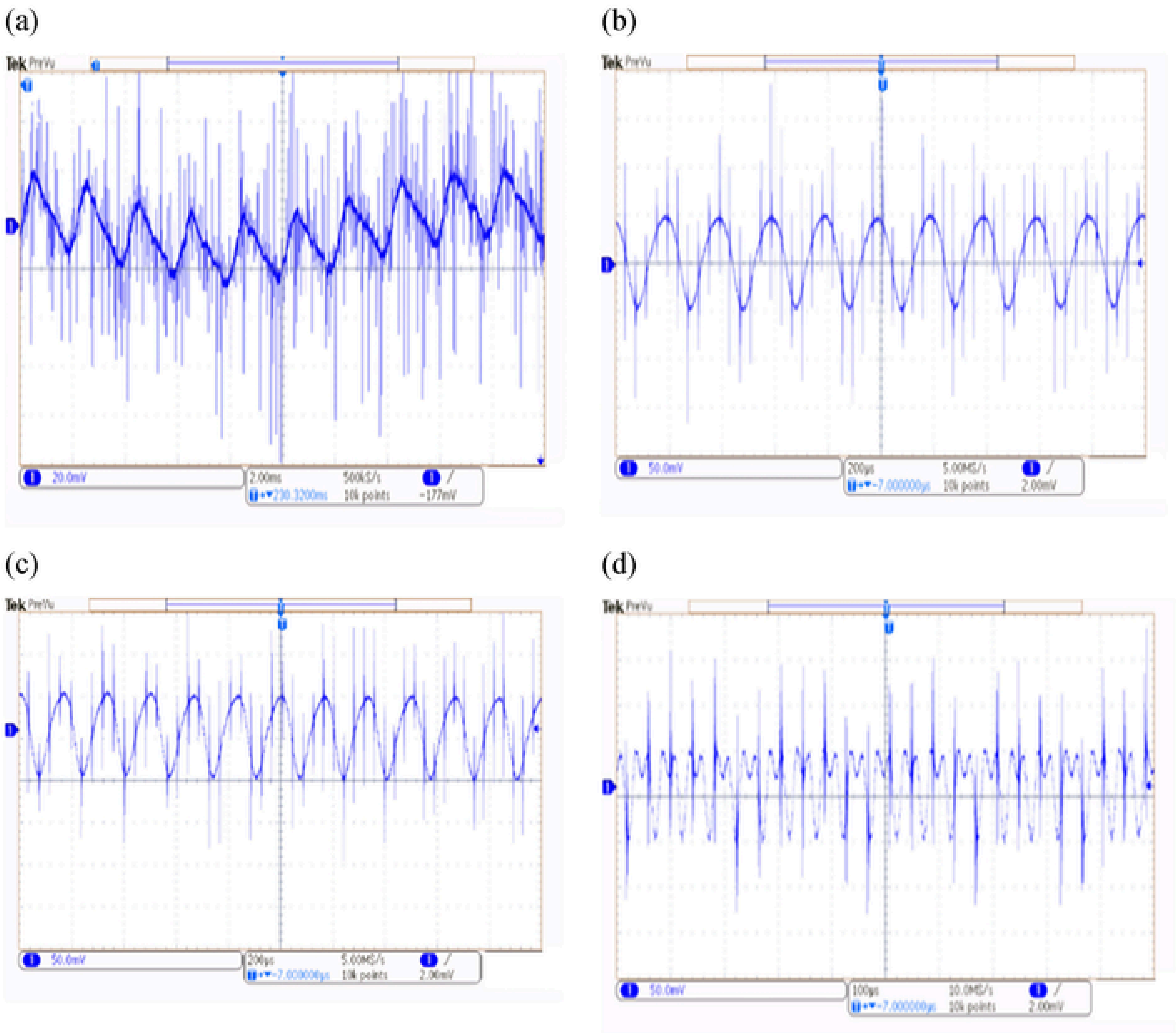
The measured voltage versus frequency at different applied voltages input and at different concentration of PVDF and TPU.



For the bending test, the maximum output voltage for the bending response is 1.8 V for the PVDF/TPU 3:1 as shown in Fig. 5a of supplementary file. When the bending angle increases, the output voltage increases linearly as shown in Fig. 5b of supplementary file. The PVDF/TPU curve's slope of (3:1) is higher than the pure PVDF, which means that the sensitivity of the PVDF/TPU (3:1) to bending response is higher than the pure PVDF. In addition, there is a similarity in the voltage output of the pure PVDF fibers as well as the formed fibers of PVDF/TPU at an added weight ratio of (1:1).

Figure 5

Examples for retraced sound waves signals at varied input frequencies for PVDF at an applied voltage of 1.2 V indicates the signal voltage output at an applied frequency (a) 500 Hz (b) 5000 Hz (c) 6000 Hz and (d) 20,000 Hz.



Acoustic sensing analysis

The acoustic sensor setup, detailed in Fig. 3 of the supplementary file, shows that pure PVDF exhibits a linear increase in voltage with frequency from 100 to 800 Hz (0.6 V to 1.2 V). This trend continues up to 4 kHz, where the output peaks at approximately 330 mV, before decreasing with higher frequencies up to 20 kHz. For low-amplitude signals, PVDF nanofibers respond consistently from 3.5 kHz to 20 kHz, with linear increases for frequencies between 100 Hz and 3 kHz. For higher amplitude signals, optimal performance occurs between 2 and 5 kHz as shown in Fig. 4a.

When TPU is added to PVDF at a 3:1 ratio, the voltage output for low amplitudes (0.6 V to 1.2 V) increases linearly up to 15 kHz, with a slight drop at 20 kHz. For higher amplitudes (1.8 V to 5.4 V), the output remains constant from 4 to 20 kHz, with a peak-to-peak voltage of 295 mV at 4 kHz, which is lower than that of pure PVDF as shown in Fig. 4b.

With a 1:1 TPU composition, the output voltage is zero at 100 Hz. It increases linearly with frequency and amplitude from 0.3 kHz to 4 kHz, then declines linearly beyond 4 kHz for high amplitudes, while rising up to 10 kHz for low amplitudes before decreasing. Poor responsiveness is observed between 15 and 20 kHz and 300 to 800 Hz. The peak-to-peak voltage reaches 415 mV at 4 kHz, making this composition the most effective as shown in Fig. 4c.

When TPU concentration exceeds 50%, no output voltage is observed from 100 to 300 Hz. For low amplitudes, the voltage increases linearly from 500 Hz to 20 kHz. For medium to higher amplitudes, it rises linearly up to 10 kHz and remains constant up to 20 kHz. The signal increases linearly up to 6 kHz and then decreases up to 20 kHz. This composition has a modest response compared to others, with performance declining when TPU concentration exceeds PVDF as shown in Fig. 4d.

In summary, PVDF/TPU (3:1) is best for force-induced pressure, while PVDF/TPU (1:1) excels with acoustic wave-induced pressure. The optimal nanofiber composition is PVDF/TPU (1:1), with the best performance in the 1000 Hz to 6000 Hz range. Further research is needed to optimize nanofiber composition for maximum output voltage.

Retracing capabilities of acoustic signals

We monitored the retracement of audio signals detected by nanofiber mats, noting that signal distortion and frequency identification varied among the four samples, significantly impacting the output signal. In the pure PVDF NF mat, the signal output for low-amplitude sound waves is less distorted than at higher amplitudes. However, even at smaller amplitudes, the output signal is affected by turbulence at lower frequencies (100 to 800 Hz) and higher frequencies (10 kHz to 20 kHz). For low-amplitude sound waves in the middle frequency range of 1 to 8 kHz, there is minimal distortion compared to other frequencies. At medium frequencies, distortion affects signal output only at the highest amplitude levels, with a relatively low distortion rate. The distortion region of detected signals at 5 kHz was between 1.2 and 4.8 V applied voltage. The distortion rate rose significantly when the applied input voltage increased from 1.2 to

4.8 V, except at the middle frequency of 4 kHz, as illustrated in Fig. 5, the retracted sound wave signals at an applied voltage of 1.2 V are shown **AQ2** . Fig. 6 of the supplementary file illustrates the signals at an applied voltage of 4.8 V.

When TPU is added to PVDF at a ratio of (3:1), the signal distortion rate is lower compared to pure PVDF, with noticeable distortion mostly at frequencies over 8 kHz. Low-amplitude input signals are not considerably altered at frequencies below 1 kHz or above 8 kHz. Even at greater input amplitudes, signal distortion remains quite low, as depicted in Figs. 7 and 8 of the supplementary file.

As the ratio of PVDF to TPU approaches (1:1), the distortion rate becomes greater than that of the other two compositions. The distortion rate is higher for frequencies below 800 Hz and from 8 to 20 kHz. The signal output is found to be distorted from low to high amplitudes, with the distortion rate increasing linearly, as shown in Figs. 9 and 10 of the supplementary file.

When the TPU percentage exceeds that of PVDF at a (1:3) ratio, the distortion rate is similar to that of the PVDF/TPU (1:1) composition. Distortion is higher for frequencies below 800 Hz and between 8 and 20 kHz. The signal output shows distortion from low to high amplitudes, with the distortion rate increasing linearly, as shown in Fig. 11 and 12 of the supplementary file. Our manufactured nanofibers exhibit comparatively low retrace frequency inaccuracy across the entire auditory frequency spectrum, with the best-detected frequency having an absolute percentage inaccuracy **AQ3** of 0 to 3%.

Conclusion

In this study the performance of PVDF and TPU nanofiber mats as an acoustic signal sensor has been investigated. The nanocomposite's piezoelectric performance is connected to the PVDF, while its elasticity is related to the blended TPU. The electrospun piezoelectric PVDF/TPU NFs membranes have been employed as **AQ4** a target for varying amplitudes and frequencies of acoustic excitation waves. Based on its piezoelectric characteristics, the synthetic NFs converted auditory impulses into electric potential with harvesting up to 415 mV at detected acoustic signal of 4 kHz. Furthermore, at a specific section of the acoustic spectra with relatively low-amplitude acoustic excitations, our synthesized nanofibers mats are able to retrace the incoming auditory waves with less distortion of frequency error up to 1%. This study can be vital for the usage of piezoelectric membrane in acoustic detector, energy harvesting from sounds, and wearable electronics sensors and can pave the way for next generation disruptive and sustainable schemes for extracting acoustic signals.

Publisher's Note

Author contributions

RN, AE, NO, MG, SN, KM and GM: performed the experiments including the synthesis and the characterization of physical properties for the samples, analyzed the data; AE, RN, GM: writing original draft; AMK, MI, IK, AME, AH, IS, SG, AAD, MT and BA: review and editing and AJ, AMK, MI, AME and NS: supervision and data correction. NS: main funding's principal investigator. All authors have read and agreed to the published version of the manuscript.

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Data availability

Data are available based upon a request sent by email to the corresponding author.

Declarations

Conflict of interest

No potential conflicts of interest is reported by the authors.

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