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Development and Optimization of a Flexible Piezoelectric Nanogenerator for Biomechanical Energy Capture and Self-Powered Sensing

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Abstract—Harnessing the potential of readily available and environmentally friendly mechanical forces, flexible piezoelectric nanogenerators (FPNGs) made from lightweight polymers and carbon naotubes have emerged as key tools for powering modern electronics and monitoring health. In this context, we synthesized Lithium tantalate ($LiTaO_3$), a ferroelectric material, for use in an FPNG. This device effectively converts these energy sources into electrical power, utilizing a blend of polyvinylidene fluoride (PVDF), multi-walled carbon nanotubes (MWCNT), and $LiTaO_3$ nanoparticles. The process entailed incorporating $LiTaO_3$ nanoparticles into a composite film of PVDF and MWCNT, which was then sandwiched between copper electrodes to complete the FPNG assembly. Extensive experimentation was conducted to optimize the concentration of $LiTaO_3$, aiming to enhance the electrical output of the FPNG. Our optimized configuration of $LiTaO_3$ and MWCNT in the FPNG yielded consistent open-circuit voltages of about 3.4V and short-circuit currents of approximately 181nA. The FPNG proved effective in capturing various forms of biomechanical energies and demonstrated promising potential for self-powered detection of force and motion.

Keywords—Piezoelectric Nanogenerator, Lithium Tantalate ($LiTaO_3$), Multi-Walled Carbon Nanotubes (MWCNT), Polyvinylidene Fluoride (PVDF), Biomechanical Energy Conversion, Self-Powered Sensing Systems.

I. INTRODUCTION

The property that allows certain materials to convert mechanical energy into electrical energy is known as piezoelectricity [1]. Piezoelectric nanogenerators (PENGs) provide an effective way to convert mechanical energy generated by external pressure into electrical energy [2]. Operating independently without an external power source like a battery or energy storage, a piezoelectric nanogenerator utilizes environmental pressure or vibrations, making it a distinct device [3]. The nanogenerator produces electrical energy by capturing mechanical energy, such as from finger taps or bending the device's surface [3].

Pierre and Jacques Curie conducted experiments in 1880 that examined how pressure influenced the electrical properties of crystals. Their research showed that certain crystals, like quartz and tourmaline, emitted electric charges on their surfaces when subjected to mechanical stress. This crucial finding demonstrated the ability to convert mechanical strain into electrical energy [4].

Later, the Curie brothers explored the properties of piezoelectric materials more deeply and formulated mathematical principles to explain the piezoelectric phenomenon. They introduced the concept of the direct piezoelectric effect, where mechanical stress directly results in the generation of an electric charge. Additionally, they discovered the converse piezoelectric effect, in which applying an electric field to a material causes it to undergo mechanical deformation [4] [5] [6].

Later, the Curie brothers produced significant publications that outlined their discoveries and predictions in the field of piezoelectricity. Despite their influential work, their research was short-lived, causing a temporary halt in further piezoelectric studies. It wasn't

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until World War I that the practical applications of piezoelectricity emerged when Ernest Rutherford and Paul Langevin invented a device using acoustic waves in seawater to detect submarines, which led to the development of sonar [7] [8].

Later, in 1919, a researcher introduced the groundbreaking concept of using an amplifier with a crystal in a crystal oscillator control to achieve a specific frequency. This innovation was later applied during World War II to enable communication between airplanes and tanks [9]. From 1940 to 1970, researchers discovered the ferroelectric dipole properties of Rochelle salt, $BaTiO_3$, and $LiNbO_3$, which exhibited similarities to those of quartz crystal. [9]. In 2006, Dr. Z. L. Wang successfully created the first nanogenerator using zinc oxide [10] [11]. Following this, there was a significant increase in research papers focusing on the field of piezoelectricity [8].

Over the course of history, technology has evolved dramatically, progressing from the basic tools and techniques employed by early humans millions of years ago, such as utilizing fire for light and crafting tools and weapons from stones, to the sophisticated technologies that are commonplace today [12]. Our ancestors first used fire for illumination and rocks as tools and weapons, but throughout time they developed increasingly sophisticated technology, such as gunpowder, compasses, steamboats, and clocks [13]. The globe first experienced electricity in 1879 [13]. Instead of using lamps and candles, many individuals now have access to electricity. Steam engines, railways, cameras, telegraphs, and telephones were developed later. Significant technological advancements were made throughout the 1900s, including the development of radios, airplanes, televisions, computers, and the internet [13]. Later, these advancements paved the way for remarkable progress. Artificial intelligence, since the year 2000, has become an essential tool in the modern era and is now deeply integrated into our daily routines [13].

Over the centuries, technological advancements have led to some adverse effects that have impacted life on Earth. Notably, pollution has become a major concern, especially since the onset of the Industrial Revolution a few centuries ago [14]. As a result, contemporary society is actively searching for sustainable energy sources that do not damage the planet. In response, scientists have created technologies that utilize renewable energy sources like solar power. These advanced solar panels absorb energy from the sun's rays and transform it into electricity. Wind power is another form of renewable energy, where turbines are used to capture and convert the energy of the wind [15] [16].

Later, wind turbines harness the power of the wind to rotate their blades, converting this mechanical energy into electricity. To accommodate the increasing demand for renewable energy in future technologies, researchers are diligently exploring additional methods to generate more renewable energy [15] [16].

At the end, researchers have found various ways to extract energy from the human body through electrical, thermal, chemical, and mechanical methods. A highlighted development in a recent study is a thermoelectric generator designed to harness energy from the body's varying temperatures. This innovative device can be worn like a bracelet around the wrist and is capable of powering a wristwatch or other similar devices [17] [18] [19].

Creating a piezoelectric nanogenerator involves utilizing various piezoelectric substances, including nanocomposites, nanofillers, and the polymer polyvinylidene fluoride (PVDF). PVDF is a semicrystalline polymer composed of repeating $CH_2 – CF_2$ units. One of the advantages of PVDF film is its flexibility, allowing it to experience stress or pressure on its surface. In some cases, PVDF is combined with nanofillers like Fe_3O_4, which possesses excellent dielectric properties and magnetic behavior[20] [21] [22]. Another notable nanofiller, $BiVO_4$, is known for its favorable dielectric properties, particularly in the β-phase [22] [23]. Like the nanofillers, $LiTaO_3$ and MWCNT also enhances the dielectric properties and works well with PVDF [24] [25] [26].

Several research groups have demonstrated their piezoelectric samples using different materials. In one published study, researchers utilized $NaNbO_3$ and reduced graphene oxide (RGO) to synthesize three distinct PVDF samples. The first sample consisted of PVDF and RGO, the second sample incorporated PVDF and $NaNbO_3$, and the third sample was synthesized using PVDF, RGO, and $NaNbO_3$ [22]. The findings revealed that the β-phase was consistent across all the samples, although the voltage outputs varied among them [22]. In another research paper, a group described the synthesis of Fe_3O_4 using iron (III) acetylacetonate and oleic acid [27].

The team mixed synthesized iron oxide with PVDF to form PVDF/iron oxide. Applying a consistent force with the tip of a small metal piece, the sample produced a voltage of 35 MV/m [27]. Polyethylene glycol (PEG) is composed of numerous small particles bonded by OH groups, which enables it to cover a larger surface area [8]. Furthermore, adding PEG to PVDF enhanced the negative charge output. Research indicates that ytterbium (III) salt has self-polarization capabilities which enhance the strong ferroelectric properties of the PVDF film [28].

To improve the electrical output of a nanogenerator under stress, various nanofillers like PEG, carbon-black, and carbon nanotubes are employed. Specifically, carbon-black is adept at conducting electricity over extended distances, although its effectiveness is limited to the γ-phase [29] [30] [31]. Carbon nanotubes exhibit excellent dielectric properties, although they pose challenges in handling compared to other nanocomposites [32]. The objective of the current experiment is to develop an

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optimized nanogenerator that complements the self-polarization of PVDF, thereby enhancing the dipoles and generating a higher electrical charge. To achieve this, the nanogenerator must attain the β-phase, which is recognized for its pronounced polarization and unique zig-zag structure among the five crystalline phases (α, β, γ, δ, ε) [33] [34] [35]. The nanofiller is used to create a structured and crystalline β-phase, which stabilizes the piezoelectric properties of PVDF [22].

In this study, we produced Li_2CO_3 nanoparticles using a solid-state synthesis approach. These nanoparticles, along with MWCNT, were then integrated into a PVDF polymer matrix to develop a flexible composite film with piezoelectric features. To assemble a flexible piezoelectric nanogenerator (PENG), we attached copper tapes to both sides of the film and connected copper wires as extensions. The PENG was tested under various load frequencies, achieving a peak open circuit voltage of 4V at 50 psi and a maximum short circuit current of 180µA at 240 BPM. The device also successfully charged a 0.1µF capacitor up to 2.4V and was effectively integrated with standard electronic components. Overall, this device demonstrates significant potential for advancements in piezoelectric energy conversion and autonomous detection technologies.

II. EXPERIMENTAL PROCEDURE

A. Preparation of $LiTaO_3$ Nanoparticles

To synthesize $LiTaO_3$ nanoparticles (Figures 1a and 1b), we employed a standard solid-state synthesis technique using commercially sourced Li_2CO_3 and Ta_2O_5 as precursor materials, with no additional purification required. Specifically, we combined 0.7389g (10mM) of Li_2CO_3 and 4.41893g (10mM) of Ta_2O_5, grinding them together in an agate mortar until a uniform mixture was achieved. This mixture was then transferred to a crucible and subjected to a two-step calcination process in a muffle furnace: initially heated to 500 °C for 4 hours, followed by a rise to 1050 °C for 5 hours. Post-calcination, the resulting $LiTaO_3$ nanoparticles were purified through multiple washes with dilute HCl, ethanol, and distilled water. The purified nanoparticles were then dried in a hot-air oven at 70 °C for 3 hours, preparing them for subsequent experimentation.

B. Fabrication of PVDF-$LiTaO_3$-MWCNTs Composite Films and Flexible Piezoelectric Nanogenerators

To fabricate PVDF-$LiTaO_3$-MWCNTs thin films, we started by creating homogeneous solutions by mixing 2g of PVDF with 14 mL of DMF and 6 mL of acetone, stirring at 700 rpm for 60 minutes at room temperature. Concurrently, MWCNTs, in varying weight ratios (0.25-1.25% relative to the PVDF matrix), were dissolved in 5 mL DMF in a separate beaker and ultrasonicated for 10 minutes. After 1 hour, $LiTaO_3$ nanoparticles were incorporated into the PVDF solution, followed by the gradual addition of MWCNTs solutions to form PVDF-$LiTaO_3$-MWCNTs composite solutions (Figures 1c and 1d). These solutions were then stirred at 700 rpm for an additional 3 hours and ultrasonicated for 5 minutes every hour. To eliminate air bubbles, the solutions were placed in a vacuum chamber for 30 minutes. Once thoroughly mixed, the solutions were poured into glass petri dishes and dried at 70 °C for 5 hours (Figure 1e). The cured composite films were then peeled off, cut into 2x2 cm² pieces, and used to assemble the F-PENG. The F-PENG was constructed by placing the composite film between two copper electrodes and sealing it with Kapton tape, resulting in a flexible and durable device (Figures 1f and 1g). The optimal MWCNTs percentage was determined based on the electrical performance of the film.

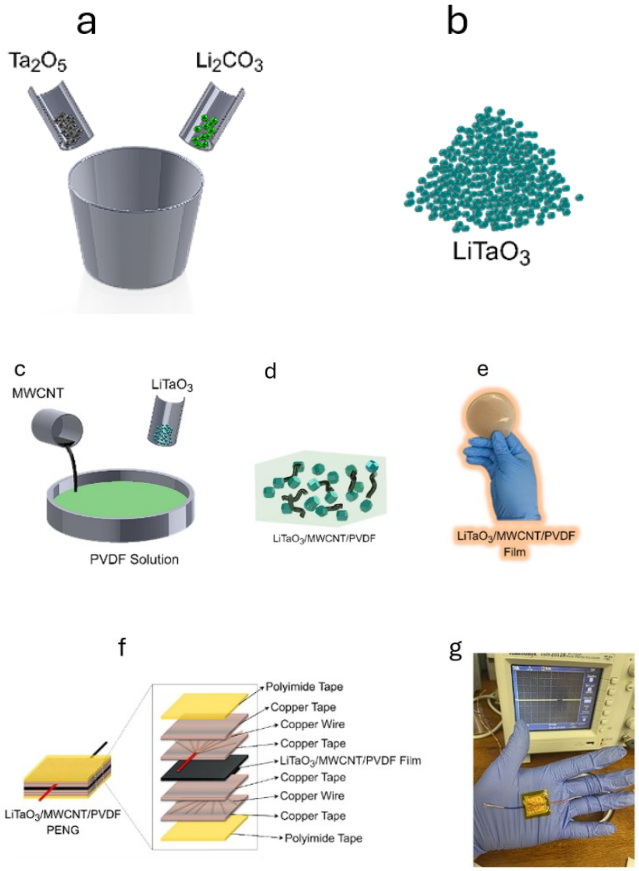

Fig. 1: (a,b) Schematic illustration of the synthesis of $LiTaO_3$ NPs via a solid-state procedure,(c-e) $LiTaO_3$/MWCNT/PVDF film synthesis, and (f,g) F-PENG fabrication.

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III. RESULTS AND DISCUSSION

In the SEM images shown in Figures 2b and 2c, the hexagonal crystal clusters of the synthesized $LiTaO_3$ material are clearly visible. Each crystal measures approximately 300-400nm in width, revealing the unique structural formation and size distribution of the $LiTaO_3$ material. These images provide crucial information about the crystal morphology and structure, enhancing our knowledge of the material's physical attributes and its applicability across different technological domains.

The electrical performance of the $LiTaO_3$/PVDF F-PENG was evaluated by applying mechanical force via an oriental motor to the surface of the PENG, maintaining an 8-inch gap between the force applicator and the PENG. A consistent force of 30psi was exerted, and the resultant open circuit voltages were recorded at a frequency of 1Hz for various nanoparticle weight percentages. The voltages measured were as follows: 0wt.% at 0.25V, 1wt.% at 0.28V, 2.5wt.% at 0.50V, 5wt.% at 0.52V, 6wt.% at 0.70V, 7.5wt.% at 0.75V, 8wt.% at 1.10V, 10wt.% at 1.20V, 11wt.% at 0.42V, 13wt.% at 0.77V, 15wt.% at 0.30V, and 20wt.% at 0.27V. The maximum voltage output was achieved with the PENG containing 10wt.% of $LiTaO_3$ nanoparticles. Following the identification of the optimal $LiTaO_3$ content, the electrical performance of the $LiTaO_3$/PVDF/MWCNT F-PENG was assessed by exerting force through an oriental motor directly onto the surface of the PENG. The gap between the force-applying surface and the PENG was consistently maintained at 8 inches, with a steady force of 30psi. The objective was to ascertain the most effective MWCNT concentration for the $LiTaO_3$/PVDF/ MWCNT F-PENG by measuring the open circuit voltage (depicted in Figure 2e).

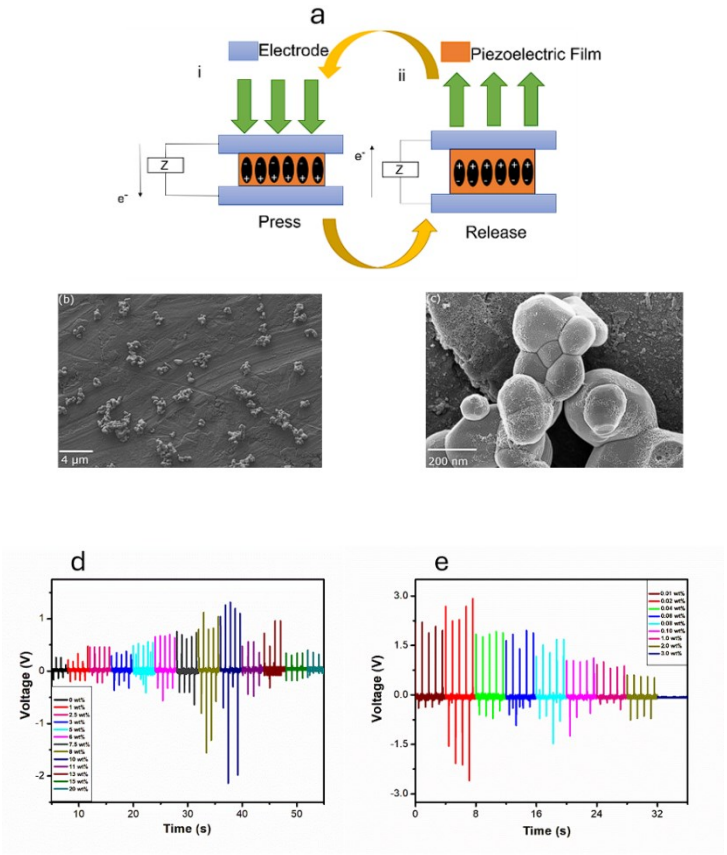

Fig. 2: (a) Schematic of the PENG mechanism during i. pressing, ii. Releasing, (b, c) $LiTaO_3$ NP SEM, (d) Voltage output of different wt.% NP incorporated $LiTaO_3$/PVDF F- PENG at 30 psi 60 BPM, and (e) Voltage output of different wt.% MWCNT incorporated $LiTaO_3$/PVDF F- PENG at 30 psi and 60 BPM.

The maximum voltage outputs at a beat frequency of 60 BPM were as follows: 0.01wt.% at 2V, 0.02wt.% at 2.8V, 0.04wt.% at 2V, 0.06wt.% at 1.9V, 0.08wt.% at 1.8V, 0.10wt.% at 1.1V, 1wt.% at 1V, 2wt.% at 0.5V, and 3wt.% at 0V. The peak output voltage was achieved with the PENG containing 0.02wt.% MWCNTs.

The analysis conducted using scanning electron microscopy (SEM) on two distinct samples (Figures 3a and 3b) —$LiTaO_3$-PVDF, and $LiTaO_3$-MWCNT-PVDF—yielded informative results. The $LiTaO_3$-PVDF sample demonstrated successful incorporation of $LiTaO_3$ particles into the polymer matrix, as seen by their presence within the film. Further, the SEM of the $LiTaO_3$-MWCNT-PVDF sample showed a dense arrangement of hexagonal $LiTaO_3$ particles along with embedded multi-walled carbon nanotubes (MWCNT), indicating effective integration of both materials into the PVDF matrix, which could enhance its piezoelectric properties. This detailed SEM examination provides valuable insights into the composite films' microstructure and component distribution, essential for exploring their potential in piezoelectric energy harvesting and other technological applications.

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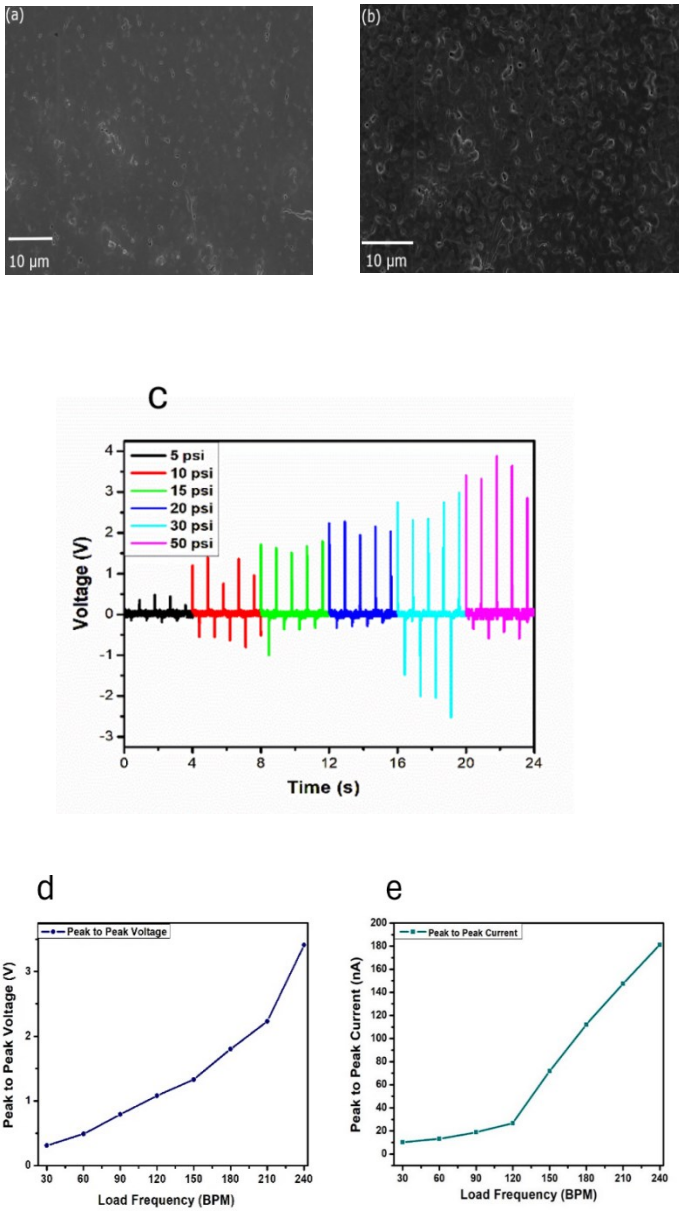

Fig. 3: (a) SEM of LiTaO3-PVDF Film, (b) SEM of LiTaO3-MWCNT-PVDF Film, (c) Pressure sensitivity of F-PENG, (d) Peak to Peak Voltage of F-PENG at Different BPM and (e) Peak to Peak Current Output of F-PENG at Different BPM.

The increased output voltage observed in piezoelectric nanogenerators when incorporating nanoparticles and nanotubes can be attributed to a combination of factors. These nanostructures enhance mechanical and electrical properties, primarily by increasing mechanical strain upon deformation, improving stress distribution, and enhancing charge separation through interfaces and heterojunctions. The higher surface area of nanoparticles and nanotubes promotes more efficient charge accumulation and separation, while their flexibility adapts the material to deformations, enabling better interaction with mechanical forces and yielding higher voltages. Additionally, enhanced electron transport within the piezoelectric material, along with synergistic effects between piezoelectric materials and nanostructures, further amplify the overall response. Incorporating nanoparticles and nanotubes thus offers a promising path to advance energy harvesting technologies, optimizing the conversion of mechanical energy into electrical energy [41]. Divided by a strain-neutral line, described PZT/PDMS layer experiences varying strain. The direct proportionality between strain rates and piezoelectric voltage/current. Higher rates yield faster charge transport, enhancing piezoelectric currents. This aligns with the fundamental piezoelectric equation. Despite current profile differences, total charges remain constant due to minimal loss during bending. The paper offers insights into strain-rate-dependent piezoelectric performance in the flexible nanogenerator [42].

The final version of the F-PENG was subjected to various levels of force applied using the oriental motor, keeping a constant tapping distance of 8 inches. During these tests, the open circuit voltage, illustrated in Figure 3c, was recorded.

International Journal of Engineering Research & Technology (IJERT)
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Vol. 13 Issue 06, June - 2024

The device displayed increasing output voltages correlating with escalating force levels, producing 0.5V at 5 psi, 1.0V at 10 psi, 1.5V at 15 psi, 2.2V at 20 psi, 2.5V at 30 psi, and peaking at 3.4V at 50 psi. This trend underscores that greater forces exerted on the nanogenerator amplify its material compression and deformation, thus enhancing the voltage generated via the piezoelectric effect [41].

Enhancing the tapping frequency on the piezoelectric nanogenerator (PENG) reduces the time needed for electron neutralization, thereby enabling the device to generate higher currents [42]. At various frequencies, the PENG's maximum peak-to-peak voltage and current were recorded (as depicted in Figure 3d for voltage and Figure 3e for current), revealing minimal changes at lower frequencies but a linear rise at higher ones. Voltage levels increased more sharply than current levels, achieving a maximum of 3.5V for voltage and 181nA for current. This pattern is due to the PENG's material undergoing dimensional shifts and deformation under the applied loads, which modifies its resistance and alters its output characteristics as frequency increases [43]–[45].

The ability of the F-PENG to sense body movements was assessed through tests involving motions of the fist and elbow. The device recorded a voltage of 0.1 V when exposed to fist movements (illustrated in Figure 4a) at a tapping frequency of 60 BPM. For elbow movements at the same frequency (shown in Figure 4b), it registered a voltage of 0.4 V. Both actions produced similar characteristic voltage peaks during elbow bends, with higher voltages observed at increased frequencies, indicating that the PENG can detect both the type and intensity of movement.

To assess the charging efficiency of the PENG further, it was integrated into the soles of a shoe (as depicted in Figure 4c), where it was tested during jogging and running at 120 BPM and 240 BPM, respectively. The device's performance in charging 1μF and 4.7μF capacitors was recorded. During jogging, the PENG charged the 1μF capacitor to 0.8V and the 4.7μF capacitor to 0.48V within 8 seconds. In running conditions, it charged the 1μF capacitor to 1V and the 4.7μF capacitor to 0.6V in the same time frame. These results demonstrate the PENG's capability as a self-powered sensor for detecting pressure, force, and biomechanical movements.

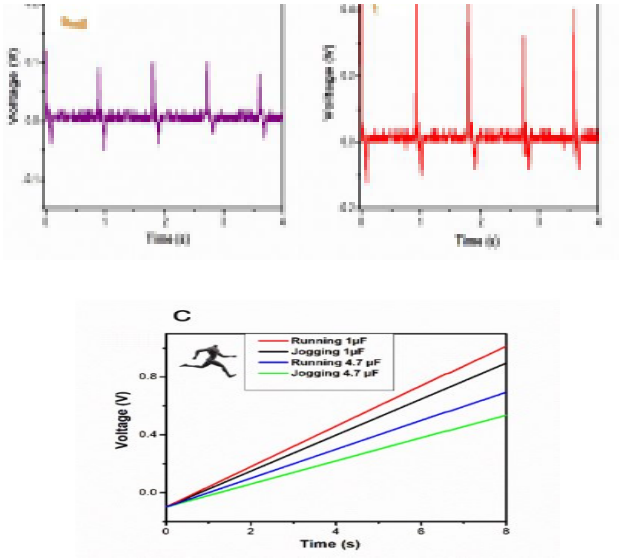

Fig. 4: (a) Body Motion Sensing: Fist Movement, (b) Body Motion Sensing: Elbow Movement, (c) Capacitor charging while jogging and running from F-PENG.

A thorough investigation was conducted to assess the long-term stability of a Piezoelectric Energy Harvesting (PENG) system operating under an actuation force of 20 pounds per square inch (psi) for 500 consecutive cycles (Figure 5). The primary focus was on monitoring the piezoelectric output voltage throughout the experiment, simulating real-world usage conditions. By subjecting the PENG device to sustained mechanical stress, this study provides crucial insights into its durability and performance over extended operational periods, contributing to the advancement and optimization of PENG technology for practical energy harvesting applications.

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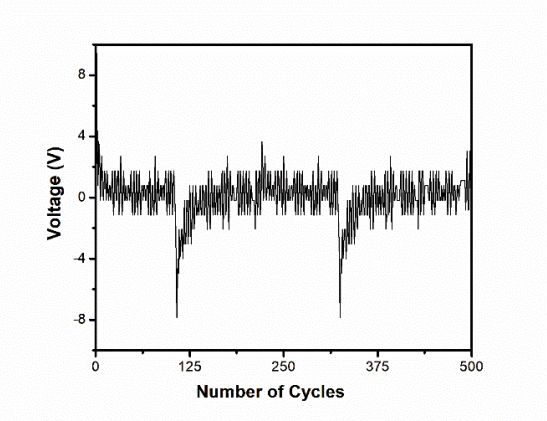
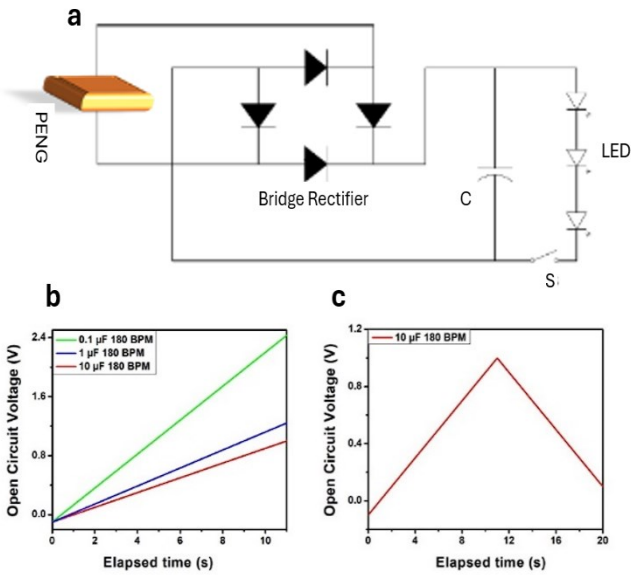


Fig. 5: The long-term stability test of PENG with an actuation force of 20 psi, piezoelectric output voltage for 500 cycles.



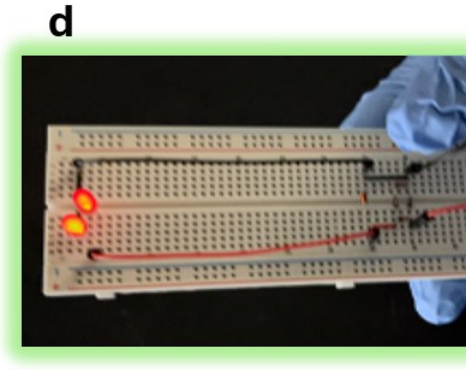


Fig. 6: (a) Circuit Diagram When PENG is Connected to LEDs with Rectifier and Capacitor (b) Charging Different Capacitors for 12s at 180 BPM, (c) Charging and Discharging of 10µF Capacitor, (d) Lighting of LEDs with PENG.

To test the energy harvesting and storage performance of the PENG, a circuit was designed (Figure 6) featuring a full wave bridge rectifier connected to capacitors, resistors, and LEDs. The PENG's voltage output was evaluated while charging capacitors of 0.1µF, 1µF, and 10µF at a tapping frequency of 180 BPM over a 12-second interval. The findings indicated that the 0.1µF capacitor achieved a voltage of 2.4V, while the 1µF and 10µF capacitors recorded voltages of 1.3V and 0.9V, respectively. Notably, the smallest capacitor displayed the highest voltage, aligning with the expectation that higher capacitances lead to greater charge losses [44]. Additionally, the charge and discharge behaviors of the 10µF capacitor were observed, showing charging tied to the external forces exerted on the PENG and a steady discharge rate dictated by the capacitor's characteristics [44], [46]. Furthermore, the PENG managed to light up two LEDs following 30 seconds of tapping at a frequency of 180 BPM, highlighting its effectiveness in energy harvesting and transformation.

In this research, we effectively created $LiTaO_3$ nanoparticles and embedded them within a PVDF polymer framework with MWCNTs to develop an advanced piezoelectric energy harvester. This device achieved a peak open circuit voltage of 3.4V and a maximum short circuit current of 181nA under the influence of hand tapping at 240 BPM and 50 psi. Additionally, it efficiently powered two LED lights and demonstrated strong potential in self-powered force and motion sensing, suggesting opportunities for developing cost-effective, easily manufactured sensors. The energy-conserving method used to fabricate the nanorod to composite film structure underlines the capability of $LiTaO_3$ nanoparticle-enhanced nanogenerators for energy harvesting and autonomous sensing applications. This opens up possibilities for developing cost-effective, easily manufactured sensors for a wide range of both experimental and real-world biological environments.

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