FABRICATION AND CHARACTERIZATION OF FLEXIBLE PIEZOELECTRIC COMPOSITE FILM KNN-Li/ZnO/PVDF FOR ENERGY HARVESTING APPLICATIONS

A Dissertation Submitted
in Partial Fulfilment of the Requirements
for the Degree of

Masters of Science
in
Physics

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Abstract

Harvesting of energy has drawn a lot of interest recently as a means of generating an electrical equipment. We must develop efficient, renewable and clean energy sources to fulfill the expanding needs and energy consumption worldwide. In this field, researchers are attempting to replace sources of energy and piezoelectricity has been proposed as a potential source. Now, the piezoelectric materials become very common to produce electricity. This article aims to develop a novel source of energy using piezoelectric materials. The main focus of this article is on piezoelectric devices as a source of energy that looks into connections between multiple devices and aims to produce the most possible power. This research explores the synthesis and characterization of a flexible piezoelectric composite film comprised of potassium sodium niobate-lithium (KNN-Li), zinc oxide (ZnO), and polyvinylidene fluoride (PVDF) for efficient energy harvesting applications. The composite film was fabricated through a multi-step process involving ball milling and drop casting methods, aiming to optimize the material properties and enhance energy conversion efficiency. The ball milling technique was employed to achieve homogeneity and fine particle size distribution of KNN-Li ceramic powder was synthesized by solid state reaction method. The fabricated composite film was fabricated by drop cast method and subjected to a comprehensive characterization using various techniques. X-ray diffraction (XRD) analysis was employed to investigate the crystal structure and phase purity, ensuring the successful formation of the desired composite. Scanning electron microscopy (SEM) was utilized to scrutinized the surface morphology and the distribution of piezoelectric phases. Fourier transform infrared spectroscopy (FTIR) was employed to analyse chemical bonding and β phase. Finally, voltage measurements were conducted to assess the piezoelectric performance of the film under mechanical stress, showcasing its potential for energy harvesting applications.

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SYMBOLS /ABBREVIATIONS

PVDF	Polyvinylidene fluoride
PEG	Piezoelectric Generator
KNN	Sodium potassium Niobate
XRD	X-Ray Diffraction
FTIR	Fourier Transform Infrared Spectroscopy
KNN-Li	Sodium potassium Niobate doped with lithium
SEM	Scanning Electron Microscopy

CHAPTER 1 AIM & INTRODUCTION

1.1 AIM: - Fabricate PEG composed of KNN-Li doped with ZnO Ceramics and Polymer PVDF.

1.2 Introduction

Our project's main objective is to yield energy using the mechanical stress or tension given to the piezoelectric sensor. Plenty of issues with the dependence on constantly replenishing energy sources could be eliminated as a result of energy harvesting. The energy produced as a result might offer relieve from

- Rising interest in using renewable energy.
- Reduce the amount of time you spend using batteries.

In current times, the extraction of electricity from natural illumination, thermal, magnetic or mechanical power is an important field of study. In this project we used mechanical stress for the generation of power. Nonetheless, it would be laborious to change the modest power source and batteries in sensor systems. Therefore, using ambient energy to power a small number of sensor systems is quite interesting.

The need for environmental energy to augment some of the electric energy used for daily living has also resulted from the shortage of accessible energy sources. For this reason, another fascinating application is using mechanical energy from trains or roadways to create power. This approach provides a modest to medium amount of electricity for running electric motors or even lightning if there are enough moving cars or trains. Using piezoelectric materials, which use the piezoelectric effect to transform mechanical vibration or strain energy into electric energy, is one of the most efficient ways to create power harvesting devices. In the last ten years, a lot of study has focused on the direct piezoelectric effect in the area of energy harvesting from the background vibrations. Because of their superior electromechanical coupling effects, piezoelectric materials are great options for mechanical energy conversion. Additionally, piezoelectric energy collection devices are far less complicated than, say, electrostatic or electromagnetic ones. Piezoelectric energy collecting systems have become extremely popular due to these features.

1.3 Piezoelectric Effect

Certain materials have the tendency to grow due to the generation of electric potential or voltage when exposed to mechanical stress. This phenomenon is known as the piezoelectric effect. In the year 1880, Pierre and Jacques made this discovery.

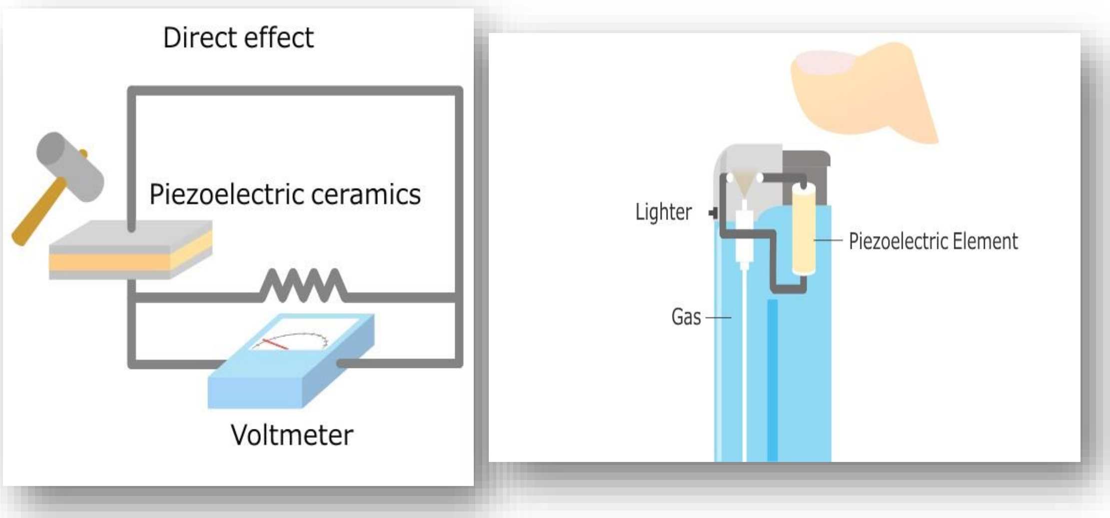

Figure 1: - This is how the piezoelectric effect operates

The basis for the operation of piezoelectric transducers is the piezoelectric effect. Certain materials produce an electric voltage when pressure or physical strain exerted along specific dimensions. Because of the piezoelectric phenomenon, the resultant voltage of these materials is inversely proportional to the force or stress imparted.

1.4 Implementing Viable Options

- Growing consumer demand for renewable energy
- Offers a number of economical and effective suggestions
- Reduce dependency on the use of traditional batteries
- Intricacy of wiring
- Raise the cost of wiring
- Reduce costs for inbuilt intelligence
- Wireless networks' increasing acceptability

- Complications with the batteries

1.5 Piezoelectric Materials

1.5.1 Naturally occurring crystals: - Berlinite, Rochelle salt, Sugar Cane, Topaz, Minerals, Bone etc…

1.5.2 Ceramics created by humans: -

- LI2CO$_3$, known as Lithium Carbonate.
- Nb2O$_5$, stands for Niobium Oxide.
- KNN, abbreviated as sodium potassium niobate.
- K2CO$_3$, known as potassium carbonate.
- Na2CO$_3$, referred as sodium carbonate.
- ZNO, abbreviated for Zinc Oxide.

1.5.3 Polymer used: -

PVDF, also called Polyvinylidene Fluoride

1.6 Review of Literature

1.6.1 Pyroelectric Effect

An electric potential appears between the terminals as a result of a temperature change in the material, a process known as the pyroelectric effect.

1.6.2 Piezoelectric Films

In order to align the molecular dipoles of polar polymers, like PVDF, in a single orientation, the polarization process involves exposing films to a strong electric field. Polarized electrets attain thermodynamic stability up to roughly 90 °C. PVDF great purity and homogeneous molecular structure make it an excellent choice for producing polarized films, and its crystallization into a polarizable form further enhances this ability.

1.6.3 PVDF Piezoelectric Film Characteristics

Superior mechanical durability and stable dimensionality maintained high piezoelectric coefficients up to around 90°C.

Significant chemical inertness of PVDF.

Constant polarization for long lengths coiled around drums. Thickness or depth varying from 1mm to 9 microns.

CHAPTER 2 – WORK PERFORMED

2.1 Synthesis of KNN-Li ceramic powder

The KNN-Li ceramic powder was synthesized through a traditional solid-state reaction technique. The primary materials, namely K_2CO_3, Na_2CO_3, Li_2O_3, and Nb_2O_5, were obtained. To remove any moisture, these components were dried at 60 °C for 24 hours and then weighed according to their stoichiometric proportion. To achieve a uniform mixture, the components were ball-milled with zirconia balls in ethanol for 10 hours. The resulting mixture was placed in an alumina crucible and calcined at 950 °C for 4 hours. Calcination is the process of heating solids to a high temperature in order to extract volatiles, oxidize part of the mass, or make the material friable. For this reason, calcination is occasionally thought of as a purifying procedure. The resulting calcined powder was then reground to be used in films.

2.2 Steps taken to synthesize the KNN-Li ceramic powder

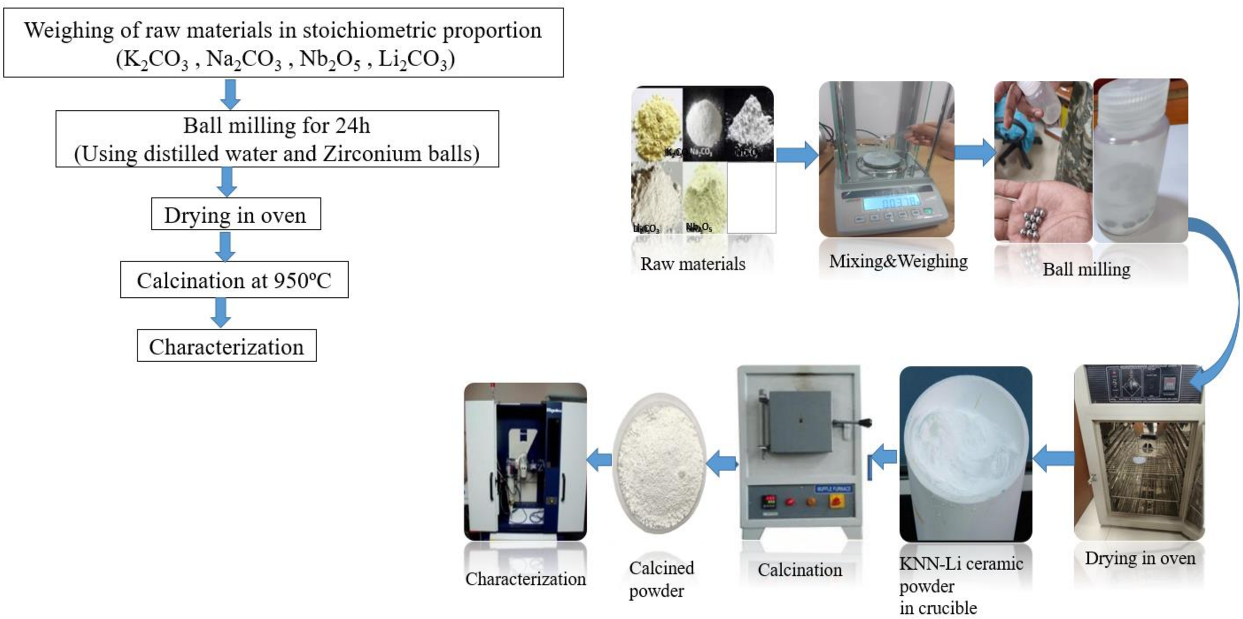

Figure 2: - Schematic presentation of synthesis of KNN-Li ceramic powder

2.3 Characterization of KNN-Li ceramic

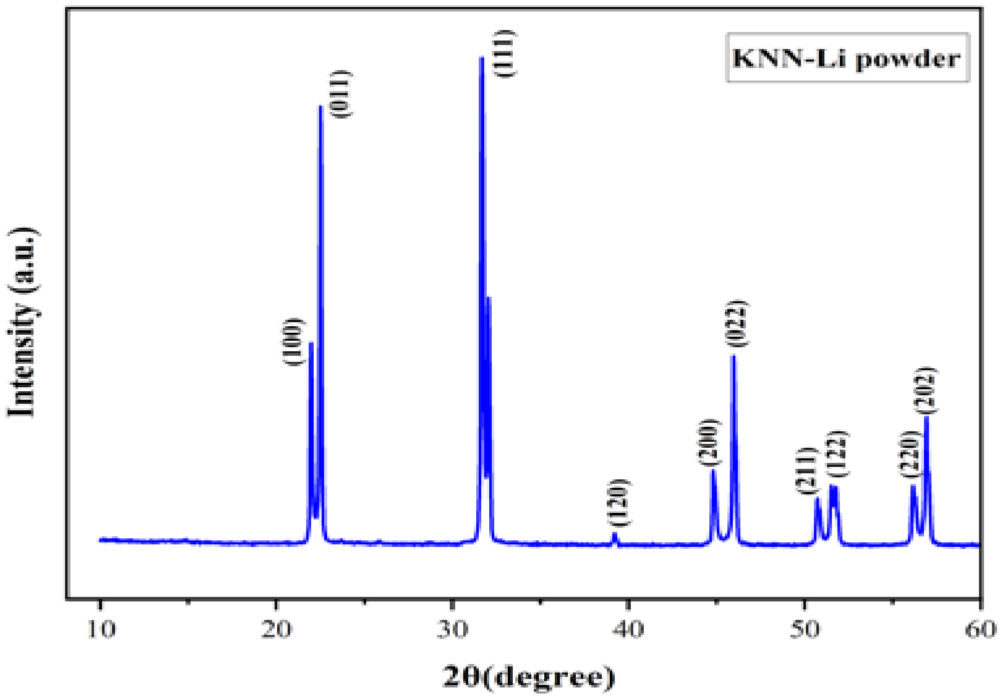

Figure 3: - XRD Plot for KNN-Li ceramic powder

XRD - Analysis

- The crystal structure of the KNN-Li powders was investigated through X-ray diffraction (XRD) analysis.
- The corresponding plot is displayed in Fig.3.
- The X-ray diffraction (XRD) pattern displays a pure perovskite structure exclusively, with no signs of impurities, suggesting the successful formation of KNN-Li powder using the solid-state reaction method.
- With reference to the standard JCPDS card no. 98-024-7572, the XRD pattern can be matched to the orthorhombic structure of perovskite KNN-Li ceramic with the *Amm2* space group.
- Furthermore, the splitting of the peaks at the [022] and [200] planes, and particularly the higher intensity of the [022] peak confirms the formation of the orthorhombic phase, which is known for its favorable piezoelectric properties.

2.4 Fabrication of KNN -Li /ZnO/PVDF Films by Drop Casting Method

2.4.1 Drop Casting Method

A popular approach for creating thin films in a variety of scientific domains, such as technology and material science, is the Drop Casting Method. In order to create a solid film, a solution or dispersion must first be deposited onto a substrate, and then the solvent must evaporate. Producing films with varying compositions and properties is made easy, affordable, and versatile with this technique.

The drop casting technique is used to produce composite films with improved ferroelectric and piezoelectric properties in the framework of KNN-Li/ZnO/PVDF. With its high dielectric constant and electromechanical coupling coefficient, KNN-Li is a lead-free piezoelectric material that is well- suited for a range of applications, including energy harvesting devices, sensors, and actuators. ZnO is doped within KNN-Li to enhance its piezoelectric properties. A good matrix material for KNN-Li composite films is PVDF, a polymer with outstanding piezoelectric responsiveness, flexibility, and durability.

Researchers can precisely regulate the film thickness and composition using the drop casting approach, allowing them to customize the final film quality. It is feasible to maximize the electrical and mechanical properties of the finished film by adjusting the concentrations of KNN-Li/ZnO nanoparticles, powders, and PVDF polymer. In order to create a homogenous layer, the deposition method entails dispersing KNN-Li/ZnO particles in a PVDF solution, casting the mixture onto an appropriate substrate, and then letting the solvent evaporate.

When the distinct qualities of KNN-Li/ZnO/PVDF are combined, the resultant film show synergistic benefits. Piezoelectric performance of the composite film is improved when KNN-Li/ZnO nanoparticles are added to the PVDF matrix. Additionally, the drop casting technique makes it simple to scale, making the production of huge area films for useful applications possible.

In conclusion, the drop casting technique offers a flexible and efficient means of creating KNN-Li/ZnO/PVDF composite films with improved piezoelectric qualities. Researchers can modify the features of the films to satisfy certain application needs by carefully adjusting the deposition conditions and composition of the film.

2.4.2 Fabrication of Pure PVDF& KNN-Li/ZnO/PVDF Film

These are the procedures had been taken in order to prepare a film utilizing the drop casting process:

2.4.2.1 Resources and Apparatus used:

- KNN-Li/ZnO powder (Lithium based Potassium Sodium Niobate doped with Zinc Oxide)
- PVDF polymer
- DMF as a solvent
- Container or Petri dish
- Magnetic Stirring
- Ultrasonicator
- Substrate
- Oven
- Spatula
- Copper wire

2.4.2.2 Preparation of KNN-Li/ZnO solution:

The required amount of KNN-Li (prepared), ZnO (purchased) and DMF as a solvent were taken and mixed together. We used ZnO because it is a conducting material. To create a uniform solution, we dissolved the KNN-Li/ZnO powder in the DMF solvent. Complete dissolving was ensured by stirring the mixture for a long enough period of time. To further improve homogeneity, we chose to sonicate the mixture for a few minutes in an ultrasonic bath.

2.4.2.3 Preparation of PVDF Solution:

Desired amount of PVDF polymer was taken and mixed with DMF. Solution was then put for stirring on a magnetic stirrer with speed equal to 40 rpm and temperature around 45°C. Here DMF (Dimethylformamide) is used because it's an industrial solvent. The characteristics of PVDF, such as its high piezoelectric coefficient, appealing flexibility and outstanding stability, have advanced its usage.

2.4.2.4. Creating Film:

- After ensuring that the silicon substrate or glass is clean and clear of any impurities or dust (If required, you may use masking tape to firmly attach the substrate to a level surface), the whole surface of the substrate was poured with the PVDF solution
- To get the appropriate film thickness, the volume of the solution was modified.
- Using a dropper or pipette, the KNN-Li/ZnO solution was gradually applied to the PVDF film surface.
- The solution disseminated and self- assembled after a few minutes on the substrate surface.

2.4.2.5 Dehydration and Creation of Film:

- The substrate with the deposited solution was placed in an oven to evaporate the solvent, slowly the substrate was heated to the appropriate temperature of 75°C. (The solvent utilized may affect the precise temperature and duration).
- The substrate was continuously heated for a longer duration until the solvent gets entirely evaporated to improve film formation and adhesion.
- Prior to transferring the film, the substrate was allowed to cool down to the ambient temperature.
- The film from the surface of the substrate was carefully lifted to prevent damage.

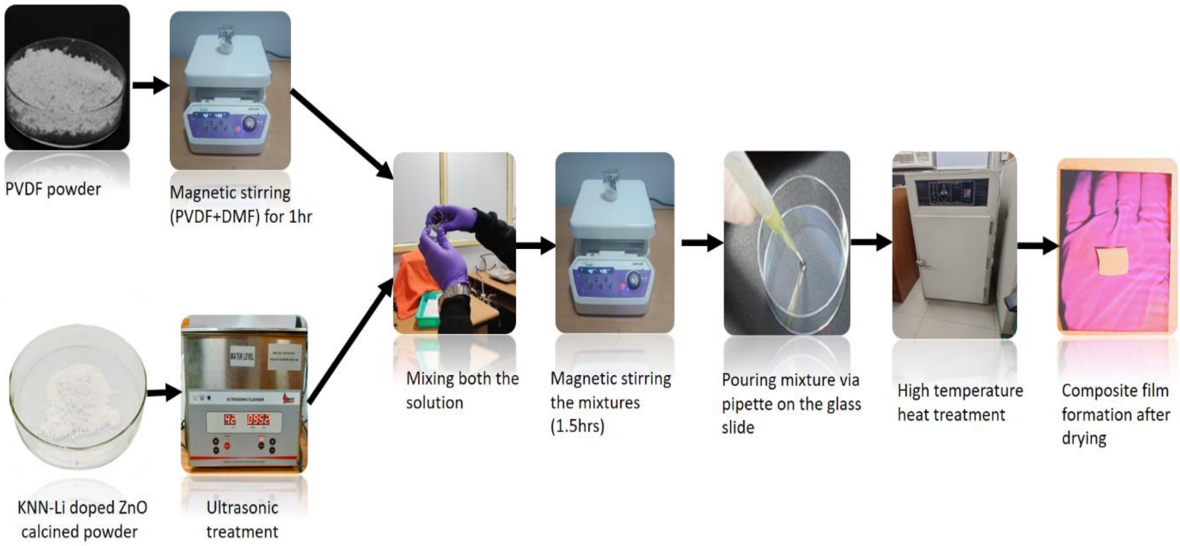

Figure 4: - Substances and resources utilized in synthesis

CHAPTER 3. CHARACTERIZATION OF FILM

The film's characteristics were assessed through the application of essential characterization techniques including X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and voltage measurements.

3.1 XRD Pattern of Synthesized films

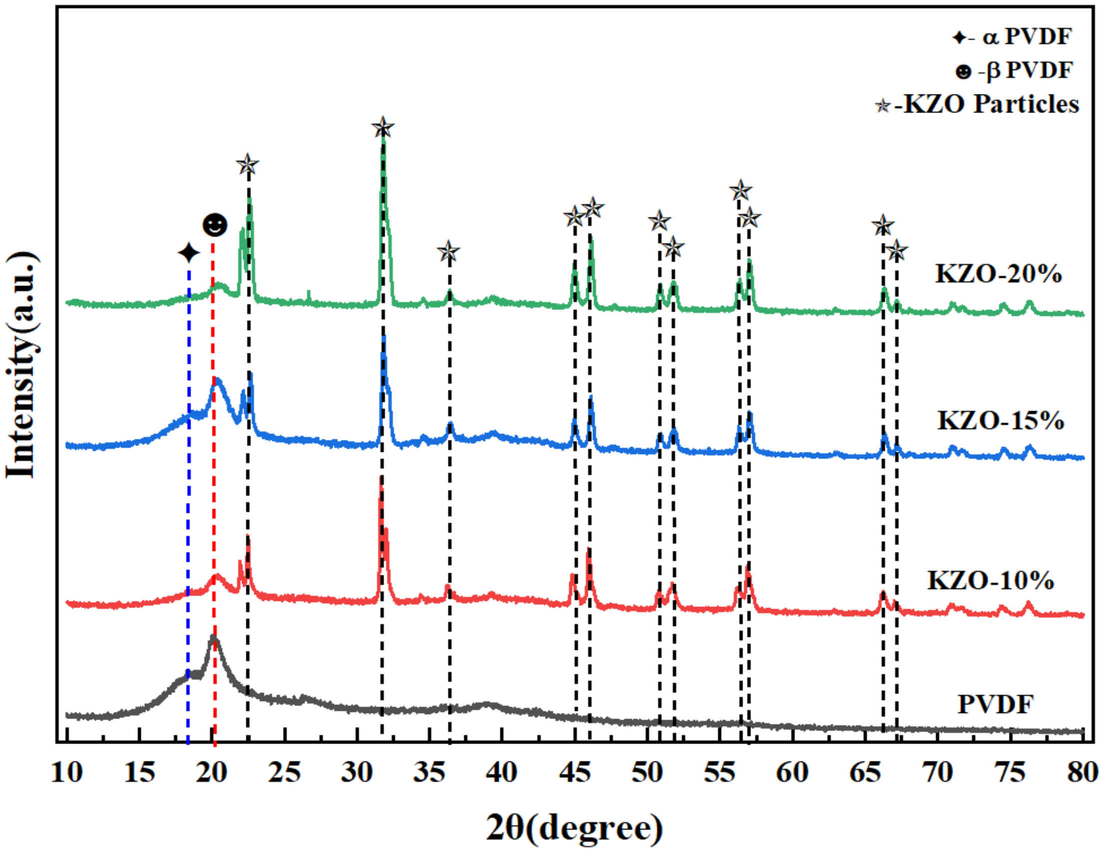

Figure 5: - XRD Pattern of synthesized films.

Analysis of the above structural data

- X-ray diffraction (XRD) analysis was carried out on a pure Polyvinylidene fluoride (PVDF) film and flexible composite films of KNN-Li/ZnO/PVDF to compile crystallographic data.
- The corresponding plots can be seen in Fig.5.
- The presence of a wide and small diffraction peak around 18.3° in all film productions is typically associated with the nonpolar α-phase of PVDF.

Meanwhile, the polar β-crystalline phase is characterized by a peak appearing at approximately 20.06°.

- Additionally, the β-phase's higher intensity in the diffraction peak when compared to the α-phase indicates that the β-phase dominates in all the films produced (see Fig. 5).
- The XRD patterns also show that as the concentration of KNN-Li/ZnO (KZO) particles increases in the PVDF matrix, the intensity of the diffraction peaks associated with the KZO particles increases, while the intensity of the PVDF-related peaks decreases.

3.2 SEM Analysis of pure PVDF and composite films

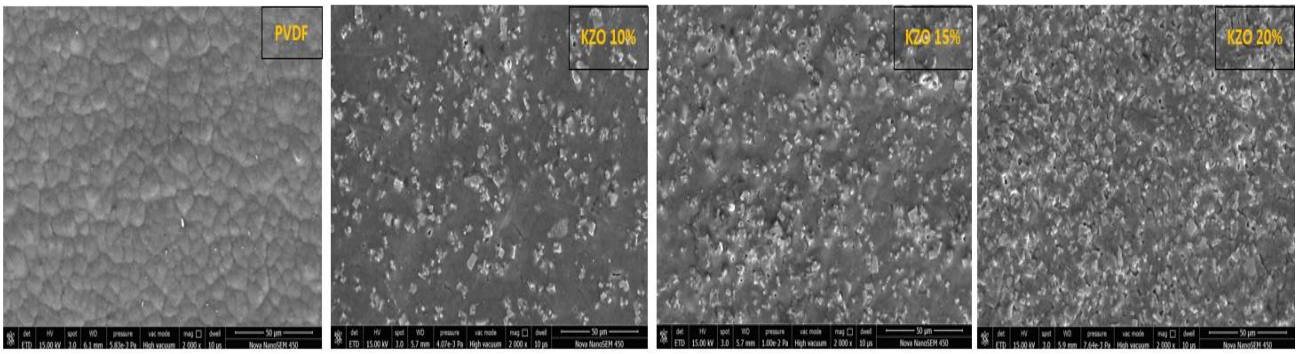

Figure 6: - SEM Analysis of pure PVDF and composite films.

- Fig.6 displays the SEM images that examine the surface characteristics of the KNN-Li/ZnO/PVDF flexible films.
- To study the morphology and surface properties of the composite film, SEM (Scanning Electron Microscopy) analysis was conducted.
- Important information on the distribution and microstructure of the integrated materials in the composite films was revealed by the SEM pictures.
- It is evident from Fig.6 that all KNN-Li/ZnO/PVDF composite films consist of homogeneous distribution of KNN-Li/ZnO ceramic particles within the PVDF matrix.

3.3 FTIR Spectra of pure PVDF and composite films.

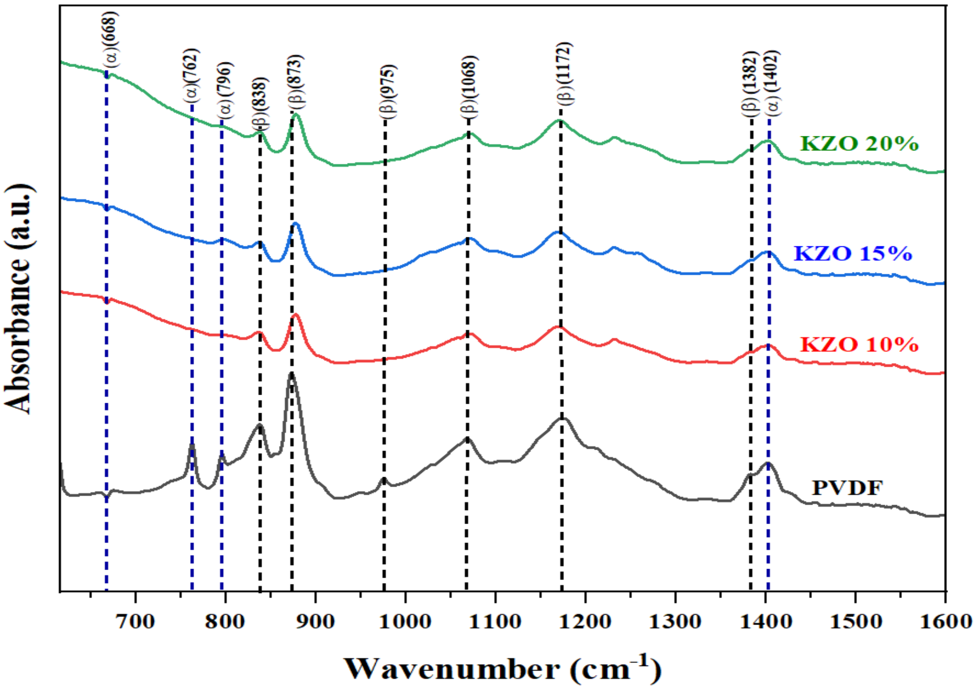

Figure 7: - FTIR Spectra of pure PVDF and composite films.

Analysis of the FTIR Spectra: -

- The FTIR spectroscopy of a KNN-Li/ZnO/PVDF composite film can reveal important details on its molecular interactions, bonding properties and chemical makeup.
- These are some important insights that can be drawn from the analysis of FTIR (Fourier Transform Infrared) spectra:
- Fourier Transform Infrared Spectroscopy (FTIR) is an analytical method that employs infrared radiation to examine the chemical properties of test materials. A part of the infrared (IR) light gets absorbed by the sample as it passes through it. The radiation that goes into the sample and comes out is quantified. The spectra can be employed to recognize and distinguish between molecules because different molecules exhibit unique spectra based on their respective structures.

- The crystal structure of PVDF was analyzed using FTIR to comprehend the impact of KZO ceramics on purified and prepared composite films.
- The examination of FTIR spectra revealed the presence of both α and β phases in each of the films. The peaks corresponding to these phases were illustrated in Fig.7.
- The FTIR data corresponds with the XRD data, confirming the existence of the β phase of PVDF in both the pure form and the KNN-Li/ZnO/PVDF composite films.
- The distinctive absorption peaks of the α phase were identified at 668, 762, 796, and 1402 cm−1, whereas the characteristic absorption peaks of the β phase were observed at 838, 873, 1068, and 1172 cm^{-1}.
- The β phase is crucial for the piezoelectric performance of the generator.
- Generally, FTIR spectra indicated as transmission are transformed into FTIR represented by absorbance on the y-axis to distinguish between the beta and gamma phases upon detecting absorption peaks in both.
- In Fig.7 the absorbance peak at 838 cm^{-1} corresponding to the β phase of PVDF increases as there is an increase in the concentration of KNN-Li/ZnO ceramic in the PVDF polymer up to 10% concentration, before decreasing. PVDF polymer consists of repeating units of CH_2-CF_2 monomers. Different configurations of hydrogen and fluorine atoms can lead to varied phases of the PVDF polymer.
- The positive charges on the surface of ZnO interact with CF_2 dipoles, while the negative charges on the surface of ZnO interact with CH_2 dipoles in the presence of ZnO in the composites.
- The interaction between polymer chains and ZnO may potentially result in the formation of various conformations of all Trans, such as TTTT chains that correspond to the beta phase. This suggests that the development of the beta phase could be influenced by the relationship between polymer chains and ZnO.
- The development of the β phase of PVDF in composite films containing KNN-Li/ZnO ceramic has a significant impact on the energy harvesting performance of the resulting films.

CHAPTER - 4

4.1 Construction and Mechanism of Piezoelectric Generator (PEG) with output voltage readings:

The PEG was created by prepared flexible KNN-Li/ZnO/PVDF composite films. The bottom and top electrodes of the films were then formed by attaching aluminium tape to the sides. The copper wires were subsequently attached to the aluminium tape, allowing the detection of output signals when pressure is applied upon connecting the external circuit. The constructed KNN-Li/ZnO/PVDF piezoelectric generator uses the piezoelectric effect to convert mechanical power into electric power. Using the special qualities of both KNN-Li/ZnO and PVDF materials, this technique produces an adaptable and compact generator that may be applied to a variety of uses, including sensor systems and portable energy collecting devices.

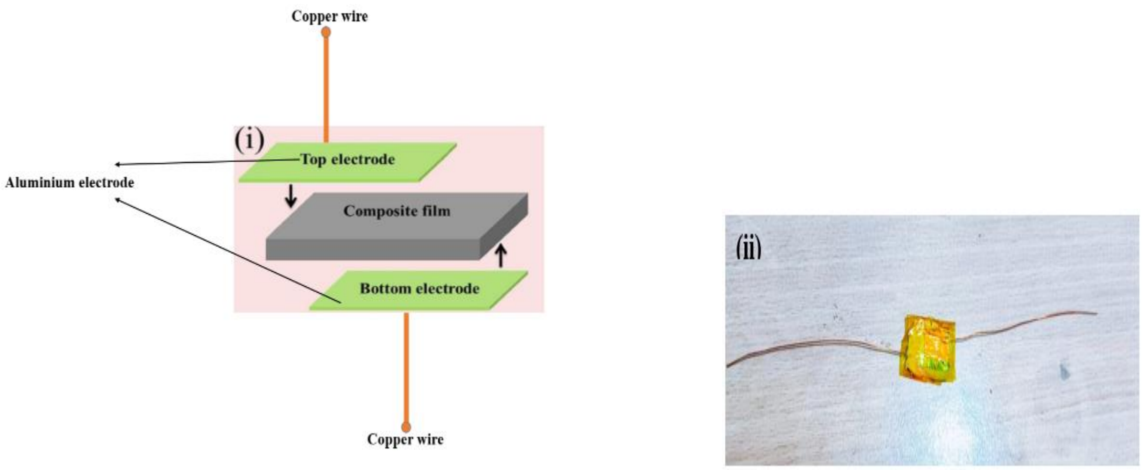

Figure 8(i-ii): - Construction of PEG

The operational mechanism of the piezoelectric generating device is such that when a perpendicular force is applied to the device, the electric dipoles align in a single direction, resulting in a potential difference across the electrodes. This leads to the flow of current through an external circuit in a top-down manner. Upon the release of the applied force, the electric dipoles reverse, causing the current to flow from the bottom electrode to the top electrode. The output signals of the PEG device were determined by performing a light finger tapping on its surface using the measurement setup depicted in Fig. 9(a-b). The PEG was connected to an oscilloscope to generate the open-circuit voltage. When pressure is applied to the device's surface, it results in generation of potential difference across the generator, which can be observed by utilizing a digital storage oscilloscope.

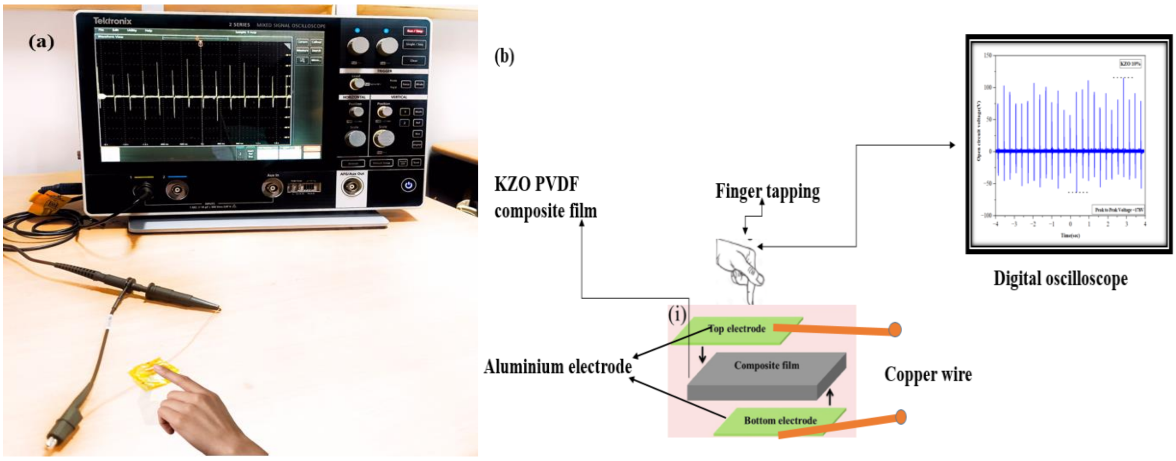

Figure 9(a-b): - Tapping the PEG device’s surface in order to measure the output signal via digital oscilloscope is shown.

4.2 Piezoelectric Generator Output Voltage

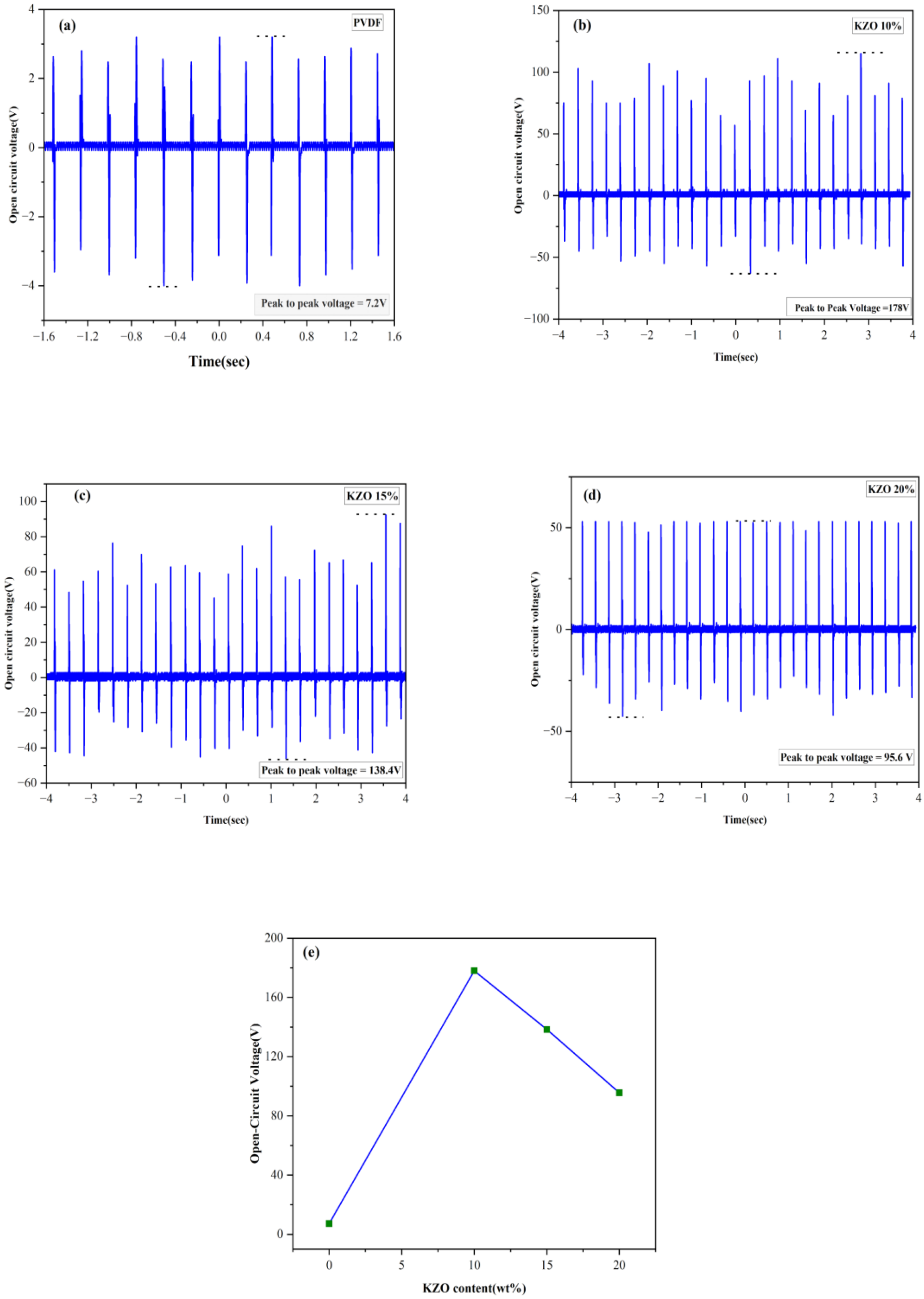

Figure 9: - Open circuit voltage generated using PEG device

Table: - Voltage generated with different wt. %

S.NO	KZO Content (wt. %)	Peak to Peak Voltage (volt)
1	0 (Pure PVDF)	7.2
2	10	178
3	15	138.4
4	20	95.6

4.3. Observations and Results:

- Fig.9 (a-e) illustrates the open-circuit voltage charts of the piezoelectric generators made with different concentrations of KNN-Li/ZnO i.e. (KZO) ceramic powder in the PVDF matrix.
- Fig.9 (e) demonstrates how the concentration of KZO ceramic particles affects the variation in open-circuit voltage.
- The plot reveals that when the KZO content in the PVDF matrix is increased to 10wt%, the open-circuit voltage initially increases and then decreases as the KZO concentration reaches 20 wt. % in the PVDF matrix.
- Among the different samples, the one with 10wt% KZO particles exhibits the highest open-circuit voltage, measuring 178V.
- The open-circuit voltages for pure PVDF, 10% KZO, 15% KZO, and 20% KZO based piezoelectric generators are 7.2V, 178V, 138.4V, and 95.6V, respectively.
- The observed decrease in the output voltage could be attributed to the presence of over 10 wt. % of KNN-Li/ZnO additives in the PVDF matrix, which hinders the nucleation of the PVDF phase and enhances the likelihood of defects emerging in the polymer matrix.
- The KNN-Li/ZnO/PVDF piezoelectric generator's adaptability makes it possible to include it into a variety of compatible and portable gadgets permitting the extraction of energy from the movement of the body thrills from the environment.
- In summary, the created piezoelectric generator presents a viable approach to power generation and energy collecting in modular and portable devices. Because of its

adaptability, film offers opportunities for autonomous vehicles, self- powered gadgets, and other applications needing effective energy conversion.

CHAPTER – 5

5.1 Evaluation of electric charge output for the purpose of piezoelectric power generation

One way to generate power is by harnessing piezoelectric materials, which provide actuators capable of converting physical stress into current. Ambient motion can be converted into electric charge that can be preserved and utilized to power other devices.

Conventional power sources that are impractical in the scenarios of portable systems can be produced by using the power harvesting devices.

5.2 Applications based on Piezoelectric Generator

Piezoelectric generators utilize the piezoelectric effect to generate electric charge due to the mechanical stress. In today's world, this technology has a wide range of application ranging from energy harvesting to sensors and actuators that are discussed below: -

1. Energy Harvesting:

   Piezoelectric generator transforms the mechanical energy from the environment to electrical energy. The application include:

   - **Wearable electronics**: Piezoelectric materials can be used in shoes or clothing to harvest energy from human body movements to power electronic devices such as smartwatches, medical sensors etc…
   - **Automobiles Systems**: Piezoelectric materials that are embedded in the roads can produce energy via road vibration which can easily power the sensors, streetlights, traffic signals etc…on the roadways.

2. Medical Applications:

   Ultrasound Imaging: The ultrasound waves are generated and received via piezoelectric crystals which is crucial for imaging tissues inside the body.

3. Electronics:

- Piezoelectric materials are used as touch sensors such as in touch screens and buttons through which signals are generated when pressed or touched which provides the sensory feedback and haptic feedback for improving interaction between user and device.
- Piezoelectric materials are used in speakers and microphones to convert sound waves into electric signals and vice versa.

4. Industrial Applications:

   Piezoelectric materials can be employed as actuators in manufacturing semiconductor devices, precision machining, as dampers in machinery and buildings and as sensors to monitor vibrations as well as to improvise the stability and performance.

5. Defense:
   - **Aerospace**: Piezoelectric sensors are used to monitor the quality of structures of aircrafts by detecting cracks and other deformations if present.
   - **Sonar navigation**: Transducers made up of piezoelectric material are utilized in sonar system for navigation, detection and communication in the underwater region.
   - Armed Forces: For troops' boots, the military contemplated using piezoelectric materials to power radios and other transportable electronic gadgets

5.3 Advantages

- **Elevated frequency response**: Piezoelectric devices/materials offer a fairly high frequency action, which enables the sensing of rapidly fluctuating parameters.
- **Elevated transient response**: Piezoelectric transducers may produce linear output and can detect events that happen in microseconds.

5.4 Drawbacks

A few of the disadvantages of piezoelectric sensors are as follows:

- **Minimal output**: Due to the limited output of the piezoelectric sensors, an additional electronic circuit needs to be connected.
- **Raised impedance**: Measurement error may occur because piezoelectric crystals have a high impedance and must be linked to both the amplifier and the additional circuit.

To avoid these issues, lengthy cables and high input impedance amplifiers should be used.

- **Establishing**: Giving the crystals the correct shape and strength is really difficult.

5.5 Conclusion

An electric field is produced when pressure is applied to the device's face because the charge carriers within the crystals distort. Furthermore, we may charge the battery of our mobile phones or other devices using this electric potential. It is amazing to think that piezoelectric technology may convert human movement into electrical power.

In the next ten years, energy harvesting is predicted to rule the technological world. As the number of electronic devices in use grows exponentially, there is an increasing focus on harvesting green energy from the surrounding environment. The piezoelectric effect allows for the generation of electricity from such biological energy, enabling smaller devices. With advancements in electronics, improved synthesis of piezoelectric crystals, and more thoughtful installation design, more electricity might be produced and could be viewed as the next viable method of power generation.

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