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CHAPTER 1

INTRODUCTION AND OBJECTIVES

1.1 Introduction

The emerging requirements for wearable electronics, self-powered sensors and bio-medical devices has led to an intensified demand for autonomous and sustainable energy technologies [1]. The primitive energy storage systems like batteries and capacitors have limitations like finite operational lifetime, environmental concerns and restricted flexibility. In order to have miniaturized electronic systems, the need of lightweight, portable and maintenance free power sources becomes important. Therefore, in order to meet these requirements, extensive research has been done on harvesting renewable forms of energy including solar, thermal, wind and mechanical energy in order to develop next generation self-sustaining systems [2].

Mechanical energy which is available from environmental vibrations, body movements, acoustic waves and structural deformations has surfaced as one of the most copious and easily available forms of energy. Nanogenerator based technologies have gained wide popularity recently, owing to their capability to convert minimal frequency along with irregular mechanical energy into electrical output. In this domain, piezoelectric nanogenerators (PENGs) have shown potential for energy harvesting because of their simple architecture, high sensitivity and flexibility [3]. These devices operate on the direct piezoelectric effect, where electrical polarization is generated when a mechanical force is applied on it externally. This effective conversion mechanism allows PENGs to act as an efficient power source for wearable electronics, biomedical devices and self-powered sensing systems [4].

Several piezoelectric materials, inorganic in nature like ²⁰lead zirconate titanate (PZT), barium titanate (BaTiO_3), zinc oxide (ZnO) and lithium niobate have shown high electromechanical conversion efficiencies [5]. However these materials suffer from brittleness, complex fabrication process, poor flexibility and toxicity due to the presence of lead-based compounds. These limitations inhibit their usage in flexible and wearable electronics where mechanical flexibility and bio-compatibility are essential parameters. Consequently, flexible piezoelectric polymers have come up as more efficient alternatives due to their low density, mechanical flexibility, lightweight structure and environmental stability.

Polyvinylidene Fluoride (PVDF) is a piezoelectric polymer which has garnered significant attention adhering to its piezoelectric, ferroelectric and pyroelectric characteristics. PVDF crystallises in several ⁷phases namely the α , β , γ and δ -phases [6]. The β -phase among all shows the highest piezoelectric activity owing to its all-trans planar zig-zag (TTTT) molecular configuration, resulting in enhanced dipole alignment and enhanced polarization behaviour. However, the pristine form of PVDF usually crystallizes in the non-polar α -phase, thereby inhibiting its piezoelectric output performance. [7]. The strategy of incorporating nanofillers within the PVDF polymer matrix has proven to be efficient for the improvement of piezoelectric performance. These nanofillers lead to β -phase nucleation through interfacial interactions, enhanced dielectric properties and improved dipole alignments within the polymer chains.

¹⁶Owing to their large surface area, excellent electrical properties and enhanced interfacial coupling 2D materials have garnered considerable attention recently. The ⁷transition metal dichalcogenides (TMDCs) are generally represented as MX_2 , where X denotes the chalcogen elements and M represents transition metals [8]. These materials have layered structures with special electronic as well as electromechanical properties required for flexible energy harvesting systems.

Tin-based TMDCs like tin disulphide (SnS_2) and tin diselenide (SnSe_2) have emerged as favourable materials because of their tunable band structures, layered morphology, enhanced surface-to-volume ratio and strong interfacial coupling [9]. Recently, heterostructure formed by combining different two-dimensional materials have also emerged as efficient strategy for improving charge separation, internal electric field generation and polarization behaviour [10]. Heterostructure engineering allows the creation of in-built electrostatic fields at interfaces which can significantly enhance stress transfer mechanisms and dipole interactions within the polymer matrices. Hence as a consequence, integration of $\text{SnS}_2/\text{SnSe}_2$ heterostructure into PVDF can potentially provide synergistic enhancement in β -phase formation and piezoelectric output [11].

Taking this forward, our present work shows a systematic investigation of TMDCs such as SnS_2 , SnSe_2 and its heterostructure ($\text{SnS}_2/\text{SnSe}_2$) used as nanofillers in integration with PVDF for piezoelectric nanogenerator applications. The choice of these materials especially SnS_2 and SnSe_2 was made due to their intrinsically layered van-der Waals structure, tunable electric properties and proven potential in electromechanical energy conversion systems [12]. This approach provides an efficient pathway towards developing highly efficient and self-powered energy harvesting systems for future wearable and portable electronic technologies.

1.2 Objectives

The prime objectives of our current study is to study the influence of transition metal dichalcogenides (TMDCs) like SnS_2 , SnSe_2 and their heterostructure $\text{SnS}_2/\text{SnSe}_2$ as nanofillers within a PVDF matrix for enhanced piezocatalytic energy harvesting applications. By incorporating 2D materials and heterostructure engineering, the parameters like interfacial interactions, β -phase crystallization, polarization behaviour and the electrical response of PVDF based PENG is anticipated to improve. The specific objectives of our present study are listed below:

- (i). Synthesis of SnS_2 and SnSe_2 powder using the hydrothermal method.
- (ii). Synthesis of $\text{SnS}_2/\text{SnSe}_2$ heterostructure powder to investigate hetero-interface formation.
- (iii). Optimization of the concentration of TMDC nanofillers within the PVDF polymer matrix.
- (iv). Fabrication of flexible PVDF/TMDC nanocomposite film with varying weight percentages of nanofillers.
- (v). Studying the effect of SnS_2 , SnSe_2 and $\text{SnS}_2/\text{SnSe}_2$ heterostructure nanofillers on the β -phase nucleation within the PVDF polymer matrix.
- (vi). Analysis and comparison of the piezoelectric response and output of PVDF based PENGs with different nanofillers systems.
- (vii). Evaluating the practical application of the fabricated PENG to power small electronic components and system.

1.3 Literature review

1.3.1: Nanotechnology and nanoparticles

Nanotechnology, being an interdisciplinary field involves the understanding and application of materials and structures at dimensions ranging from 1-100 nm [13]. Within this size regime, materials show different chemical, electrical, optical, physical and mechanical attributes which are significantly distinct from their bulk counterparts. This happens because of quantum confinement effects, enhanced surface to volume ratio and enhanced surface energy. These characteristics arising at the nanoscale level has caused the development of advanced materials with tuneable functionalities resulting in extensive applications across electronics, energy system, medicines, environmental science and material engineering.

This concept has evolved rapidly over the past few years and is currently among one of the most transformative scientific and technological fields. Due to its capability to design and engineer materials at atomic and molecular levels, nanotechnology has contributed significantly in several sectors including semiconductor industries, biomedical engineering, communication systems, sensors and renewable energy technologies. Particularly, the emergence of nanomaterials has opened new domains in the development of lightweight, flexible and strong performance electronic devices [14]. This field has also played a vital role in addressing obstacles associated with sustainable energy production, environmental and healthcare applications.

Nanoparticles are materials having dimensions in the nanoscale range and they exhibit remarkable physical and chemical properties due to their reduced dimensions. Nanoparticles exist in various forms based on their morphology including zero dimensional, one dimensional nanoparticles and nanotubes, two dimensional nanosheets and three dimensional

nanostructured architectures [15]. Their unique behaviour is because of high surface area and enhanced interfacial activity which leads to superior optical absorption, catalytic performance, charge transfer characteristics and mechanical strength.

Nanoparticles can be categorized into metallic, metal oxide and polymeric nanoparticles along with carbon based nanostructures and semiconductor based nanomaterials. 2D materials in the recent years have gotten considerable research attention because of their atomically thin layered structures and remarkable electromechanical properties. Several materials like graphene, TMDCs and MXenes have shown exceptional performance in applications involving sensing, energy storage and energy harvesting systems [15]. Due to their layered structure effective charge transport and strong interfacial interactions occur, making them highly suitable for incorporation within the polymer matrices.

The physiochemical properties of nanoparticles depend on their size, shape, crystallinity and synthesis method. Therefore, controlled synthesis of nanoparticles is essential for controlling their functional properties for various applications. Synthesis routes including- hydrothermal synthesis, chemical vapour deposition, exfoliation, sol-gel processing and pyrolysis method are used to obtain nanostructures with desired morphology and structural characteristics [16]. These techniques allow strict control over particle dimensions, crystal structure and surface chemistry, therefore playing a significant role in device performance.

In piezoelectric applications, nanoparticles have gained exponential importance due to their ability to enhance dielectric behaviour, dipole alignment and improved electrochemical coupling within the polymer matrix. This method of incorporation of nanofillers within piezoelectric polymers has proven to be effective for improving β -phase nucleation and piezoelectric response. In particular, layered nanomaterials which have a high surface area and strong interfacial interactions have shown considerable improvement in the performance of

piezoelectric nanogenerators. Therefore, the inclusion of nanotechnology and advanced nanoparticles has become an important strategy for developing efficient and flexible self-powered systems.

1.3.2: Nanoparticles' synthesis techniques

Nanoparticles can be synthesized by different techniques. These methods can be used to create dry particles as well as nanoparticles, which can be distributed in a liquid or gaseous media. Nanostructures can be produced by beginning with atoms or by shrinking microparticles to nanoparticle size.

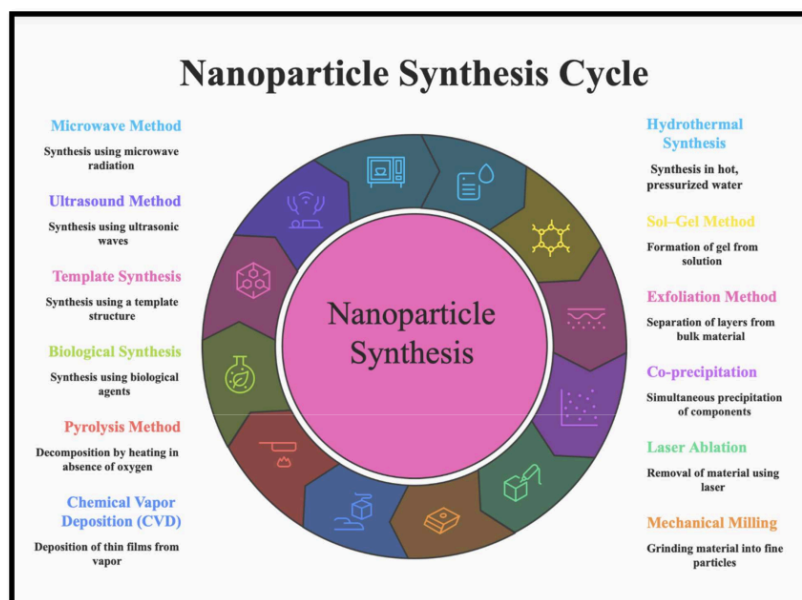


Fig.1: Nanoparticles synthesis techniques

1.3.2.1 One-Step Pyrolysis Process

One step pyrolysis is a thermal decomposition method conducted in an inert environment, commonly used for the synthesis of nanomaterials. In this technique, a precursor material is subjected to high temperatures to decompose into smaller, stable nanostructures. The process is straightforward and it is also one of the processes which is less complex and less time consuming for material synthesis. The precursor such as urea is placed in the muffle furnace at 600° C – 700° C for around 3hrs and then the sample is cooled and a powder form of the nanomaterial is obtained in the process [17].

The readily available precursors makes process economically viable, also the parameters like temperature, heating rate and precursor concentration can be tuned to achieve desired sizes and shapes of the material which is very important aspect keeping in mind the importance of particle size in the nanomaterial synthesis. It is a highly efficient and versatile technique for nanomaterial synthesis. Although it causes high energy costs and precision control but these factors can be optimized going forward.

1.3.2.2 Hydrothermal Process

The hydrothermal route is widely used to produce solids such as fluorides, ceramics, complex oxides, microporous crystals, and superionic conducting materials. Magnetic materials and luminescence phosphors are also produced with the help of it. Novel condensed materials, such as stacking sequence material, are also accessible through it. Additionally, the synthesis of thin films, helical and chiral structures, and nanoscale particles is made possible. "Hydrothermal synthesis" refers to the method of generating compounds by chemical reactions in a heated, sealed solution that is heated above ambient temperature and pressure [18].

1.3.2.3 Sol-gel process

One method used to create solid materials using tiny molecules is the sol-gel process. The sol (or solution) changes during this chemical process, becoming a biphasic system that resembles a gel. There are two phases to this system: a liquid phase and a solid phase. The liquid phase can have various morphologies, such as single particles or networks of linked polymers. Finely ground and homogenous ceramic powders can be produced via precipitation. These single- and multi-component powders at the nanoscale are produced for use in dentistry and biomedicine [19].

1.3.3 Energy harvesting

With the increase in global demand for sustainable energy and its technologies has led to heightened research towards alternate energy solutions. Conventional sources suffer from limitations like finite operational life, maintenance requirement and environmental concerns. Therefore, energy harvesting has surfaced as a potential approach to convert naturally available mechanical energy into electrical output for driving small scale electronic systems.

²⁹ The term energy harvesting refers to the process of converting energy by capturing it from surrounding sources like solar radiation, thermal gradient, wind, electromagnetic waves and mechanical vibrations into electrical energy. Among all these sources, mechanical energy is considered as readily available and accessible due to its widespread occurrence in human activities, industrial systems and environmental motion. Activities like body movements, vibrations, pressure fluctuations and acoustic waves generate mechanical energy that can be used for electrical power generation.

Several mechanisms have been developed for converting mechanical energy including electromagnetic, electrostatic, triboelectric and piezoelectric approaches. However, piezoelectric and triboelectric nanogenerators have garnered attention because of their simple

device structure, flexibility and sensitivity. These systems possess advantages like lightweight structure, low fabrication cost and compatibility with substrates that makes them relevant for wearable and self-powered electronics[20].

Due to the rapid advances in nanotechnology and material engineering, the energy harvesting efficiency has improved considerably by incorporating nanomaterials and functional composites. These developments have led to an expansion of energy harvesting systems in various domains.

1.3.4 Nanogenerators

Nanogenerators are energy harvesting devices ²⁵ which convert mechanical, thermal and vibrational energy into electrical energy at nanoscale. In recent times, these devices have gained significant popularity owing to ¹⁷ their applications in self-powered electronics and wireless sensor based systems. They are classified into ¹⁷ piezoelectric nanogenerator (PENG) and triboelectric nanogenerator (TENG) based on their operating mechanisms.

1.3.4.1 Piezoelectric nanogenerators

Piezoelectric nanogenerators are self-powered electronic devices which convert energy produced from mechanical vibrations into an electrical output by utilizing the direct piezoelectric effect [21]. They were discovered by Zhong Lin Wang in 2006, post which they have gotten significant recognition because of their capability to convert low frequency mechanical energy from sources like environmental vibrations, human activities and pressure fluctuations. The growth of high performance piezoelectric nanogenerators has become essential due to the increasing demand for self-powered electronic systems and wearable technologies.

The working of PENG is based on the generation of electric polarization within piezoelectric materials under mechanical stress. The internal dipoles of the piezoelectric materials get aligned under applied pressure leading to the separation of charges which creates an electric potential across the surface of the material. When the applied force is released, reverse polarization occurs, thereby producing an alternative electrical output. This periodic cycle of deformation and relaxation generates a periodic electrical signal.

Several materials including inorganic ceramics and polymer based piezoelectric materials have been used for PENG fabrication. PVDF and its nanocomposites have gained wide importance because of their flexibility, lightweight nature and ease of fabrication. Due to these advantages, PENGs have shown potential for applications in several domains like self-powered sensing technologies and wearable electronics.

1.3.4.2 Working principle of PENGs

Piezoelectric nanogenerators operate on the direct piezoelectric effect which converts energy produced from mechanical vibrations into electrical energy via stress induced polarization [22]. On applying an external mechanical force on the piezoelectric layer, deformation occurs in the crystal structure, therefore causing displacement of charge carriers and generating a dipole moment.

Under initial equilibrium conditions and the absence of external mechanical stress, the charges are uniformly distributed and no potential difference exists across the material. When a mechanical force is applied, polarization is induced due to dipole alignment and charge accumulation happens on the opposite electrode surfaces. This creates a potential difference which carries electrons through an external circuit producing an electrical output. However, when the applied stress is removed, the material comes back to its initial state, hence leading

to depolarization and reversal of charge flow. This conversion mechanism forms the basis of PENGs operation.

1.3.4.3 Advantages of PENGs

Piezoelectric nanogenerators have gained attention because of their ability to efficiently harvest energy from mechanical vibrations and convert it into a usable electrical output. Their primary advantage is their capability to produce electrical output from low frequency mechanical stimuli like body movements, vibrations and pressure variations. PENGs also possess lightweight and flexible structures thus making them highly relevant for amalgamation into wearable and portable electronic devices [23].

PENGs also possess a simple device architecture and has a cost effective fabrication method. They can operate without any source of external power, therefore reducing maintenance requirements and environmental concerns. They are also compatible with flexible polymers and the usage of nanomaterials makes it even more durable and efficient. Therefore, owing to these characteristics, PENGs show capability for applications in self-powered sensors, biomedical devices, electronic skin as well as wireless monitoring systems.

1.3.4.4 Disadvantages of PENGs

Inspite of having substantial potential in energy harvesting applications, PENGs have limitations which can restrict their mainstream implementation. One of the major issues with PENG is that they have a relatively low electrical output especially current generation which hinders their applications in powering high energy electronic systems.

Another limitation includes the high internal impedance of PENGs devices which can cause complications in efficient energy transfer and integration with external electronic circuit. Apart

from this, its output performance depends heavily on the frequency and magnitude of the external force. Inconsistent inputs and environmental variations can impact device efficiency. Therefore, addressing these limitations ²² is essential for improving the practical applicability of PENGs in future self-powered systems [24].

1.3.5 Polyvinylidene Fluoride (PVDF)

PVDF is a high performance synthetic and flexible polymer credited due to its chemical resistance, electric and piezoelectric properties and a unique electro active behaviour. It is used in chemical processing and energy storage devices due to its durability and flexibility. This polymer has a semi-crystalline nature and is also highly fluorinated which makes it stable against acids, bases and corrosive chemicals. It is thermally stable, has high mechanical strength, is UV resistant and non-toxic. [25]

PVDF consists of 5 crystalline phases namely- alpha, beta, gamma, delta and epsilon. The alpha phase is the most stable and ground state phase which is non polar in nature hence it does not exhibit piezo or ferro electric properties. The beta phase is a polymorphic phase found at high temperatures. It consists of an all Trans planar zig zag (TTTT) chain configuration. ³² In this, all dipole moments are unidirectional, therefore creating a spontaneous polarization due to which it shows piezoelectric properties. The gamma phase is a high symmetry phase in polymers and is found under high temperature and unique processing conditions. The delta phase is less stable than others and is usually formed at a high temperature and pressure. The epsilon phase being the least common one of them all is found in extremely rare conditions, for example the iron in earth's core.

1.3.6 Two-dimensional materials

Two-dimensional (2D) layered non-centrosymmetric materials hold significant potential for electronic devices and nanoscale electromechanical systems. Recent research has

increasingly focused on atomically thin two-dimensional materials due to their extraordinary and unconventional mechanical, optical, magnetic, and electrical capabilities. In 2D transition metal dichalcogenides (TMDCs), the interlayer MX bonds are pivotal, unlike the sandwiched layers that are maintained by weak van der Waals forces, allowing for facile cleavage at the layer surface. These TMDCs are predominantly covalent compounds, commonly represented as MX_2 , where M represents a transition metal and X corresponds to a chalcogen atom. This has enabled the production of crystals with very tiny dimensions. Owing to their distinctive features such as flexibility, optical transparency, mechanical stability, and biocompatibility two dimensional materials are well suited for a wide range of advanced application. 2D materials are optimal for constructing energy harvesters such as PENGs. These materials include graphene, TMDCs, h-BN, MXenes. 2D materials have properties like high surface area, tunable bandgap, flexibility, optical transparency, high thermal conductivity. 2D materials find their applications in electronics, energy storage, optoelectronics, sensors, and in biomedical applications [26].

1.3.6.1 Tin Disulfide (SnS_2)

Tin disulphide (SnS_2) is a layered semiconductor and a transition metal dichalcogenide (TMDC) which has unique structural and electronic properties. SnS_2 has a hexagonal crystal structure, where a tin atomic layer is inserted between sulphur layers having van der Waals interactions. This layered architecture offers the possibility of the formation of ultrathin nanosheets with a high surface area and better interfacial properties, which makes SnS_2 a promising material for electronic, sensing and energy harvesting applications [27].

SnS_2 possesses many favourable properties such as appropriate band gap, high carrier mobility, environmental stability and strong electromechanical coupling behavior. In addition, the high

surface-to-volume ratio is beneficial for efficient stress transfer and enhancement of interfacial interactions with polymer matrices. Recently, SnS₂ has been reported to be an efficient nanofiller ¹³ to improve the electroactive β -phase of PVDF via dipole interaction and nucleation effects. Incorporating SnS₂ into polymer systems enhances charge polarization and dielectric properties, resulting in improved electrical output performance of piezoelectric nanogenerators.

In the present work, SnS₂ has been selected as one of the TMDC nanofillers for incorporation into the PVDF matrix owing to its layered morphology and capability to facilitate β -phase enhancement. Also, its individual role and as part of the SnS₂/SnSe₂ heterostructure system is investigated to improve piezoelectric energy harvesting performance

1.3.6.2 Tin Diselenide (SnSe₂)

Tin diselenide (SnSe₂) is a layered two-dimensional transition metal dichalcogenide (TMDC) that has gained considerable attention owing to its attractive electronic, optical and structural properties. SnSe₂ crystallizes in a hexagonal CdI₂-type layered structure, in which a tin atomic layer is sandwiched by two selenium layers and the layers adjacent to it ¹⁴ are held together by weak van der Waals forces. This kind of structure allows the production of ultrathin nanosheets with high surface area and adjustable physicochemical properties for advanced functional applications [28].

SnSe₂ is a narrow band gap material with high electrical conductivity, excellent charge transport properties and good environmental stability, which makes it an attractive material for energy storage, optoelectronic and sensing applications. The ionic radii of selenium-based compounds and their lattice polarizability are higher compared to those of sulfur-based TMDCs and can have a considerable effect on dielectric response and charge distribution. These

properties are advantageous to enhance interfacial polarization and stress transfer when incorporated into polymer matrices.

SnSe₂ has been promising as a nanofiller for piezoelectric nanogenerator application through the facilitation of dipole arrangement and the enhancement of β -phase formation in PVDF matrices. In this work, SnSe₂ has been incorporated individually and as a constituent of the SnS₂/SnSe₂ heterostructure to study its effect on polarization behavior and piezoelectric output enhancement.

1.3.6.3 SnSe₂/SnSe₂ heterostructure

A competent method to tune the electrical, optical and electromechanical characteristics of two-dimensional materials has been developed as heterostructure engineering. A heterostructure is created by integrating two or more layered materials possessing different electronic characteristics, thereby creating an interface capable of modifying charge transport and polarization behavior. In two-dimensional systems heterostructures are constrained by van der Waals interaction without requiring strict lattice matching conditions and thus allow the formation of interfaces with unique functional properties [29].

The SnS₂/SnSe₂ heterostructure offers the combined benefits of both sulfur- and selenium-based transition metal dichalcogenides (TMDCs). SnS₂ and SnSe₂ have similar crystal structures and small lattice mismatch, which can build up stable interface with strong interfacial coupling and efficient charge redistribution. The formation of a heterointerface gives rise to an internal electric field due to band offset differences and charge equilibration at the interface. This internal electric field assists in separating the charges and increasing the polarization.

In the case of piezoelectric applications, the heterostructure formation can be a significant improvement for the stress transfer mechanism and dipole interactions in the polymer matrices.

In the present work, SnS₂/SnSe₂ heterostructure was incorporated in PVDF as nanofillers to utilize the synergetic effects due to the interfacial interactions. Improved charge transfer and polarization behaviour are expected to enhance the β -phase nucleation and the electrical response of fabricated piezoelectric nanogenerator

CHAPTER 2

MATERIALS AND METHODS

2.1 Materials

Tin (IV) chloride penta-hydrate, Thio-Urea, Selenium dioxide and PVDF were bought from Sigma Aldrich. The solvents - Ethylene glycol and N,N-dimethyl formamide (DMF) were procured from Thermo Fisher and were used as received.

2.2 Synthesis of SnS₂ powder

Tin (II) Sulfide was synthesized in its powder form using hydro-thermal method. Tin (IV) chloride penta-hydrate (3.47 mmol) and Thio-Urea (4.97 mmol) were used as the precursor reagents and were mixed in 40 mL of de-ionized water. The solution was then stirred magnetically for 45 minutes at room temperature. It was then transferred into a Teflon container and sealed into a stainless-steel autoclave. The solution was then heated in the hot air oven at 200 °C for 24 hours. Post this, the sample was washed twice with de-ionized water and ethanol and centrifuged for 6 minutes at 5500 rpm. It was then left to dry in a vacuum oven overnight at 80 °C. Yellowish- brown particles of SnS₂ powder were obtained and were grinded into a fine powder using a mortar- pestle [30].

2.3 Synthesis of SnSe₂ powder

A simple hydrothermal route was used to synthesize SnSe₂ powder [31]. Tin (IV) chloride penta-hydrate (1.50 mmol) was added into 30 mL of de-ionized water and was labeled as solution 'A'. Selenium dioxide (1.50 mmol) was added into 10 mL of de-ionized water and was labeled as solution 'B'. Both 'A' and 'B' were mixed and stirred for 20 minutes. Then 3 mL of hydrazine-hydrate was added into the solution and it was magnetically stirred for 20 minutes at room temperature. It was then transferred into a teflon container and sealed into a stainless-steel autoclave. The solution was then heated in the hot air oven at 200 °C for 24 hours. Post this, the sample was washed twice with de-ionized water and ethanol and centrifuged for 6 minutes at 5500 rpm. It was then left to dry in a vacuum oven overnight at 80 °C. Black particles of SnSe₂ were obtained and were grinded into a fine powder using a mortar-pestle

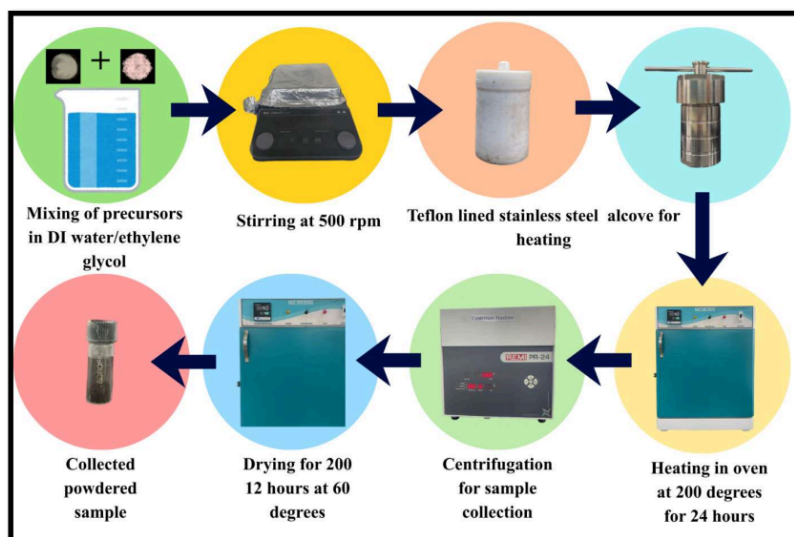


Fig. 2: Schematic illustrations for the steps of hydrothermal synthesis of Tin based TMDC powders

2.4 Synthesis of $\text{SnS}_2/\text{SnSe}_2$ heterostructure powder

The following synthesis was done using the hydrothermal method. Tin (IV) chloride pentahydrate (5.42 mmol) and Selenium dioxide (11.1 mmol) were taken and mixed into 40 mL of ethylene glycol solution. The solution was stirred magnetically for 45 minutes. After which 3 mL of Hydrazine hydrate was added into it drop-by-drop and the mixture was stirred for 30 minutes at 70 °C. The solution was then heated in the hot air oven at 180 °C for 36 hours. Post this, the sample was washed thrice with de-ionized water and ethanol and centrifuged for 10 minutes at 5500 rpm. It was then left to dry in a vacuum oven for 12 hours at 80 °C. A black powder was obtained and was grinded using a mortar- pestle for 15 minutes [32].

2.5 Synthesis Methodology of TMDCs/PVDF films

The synthesized powders were then integrated with a flexible and piezo electric polymer PVDF to synthesize thin films. For the synthesis of films 1g PVDF was dissolved in 10 mL DMF and was stirred magnetically for 3 hours. Then the respective powders were added at a weight percentage into the previously prepared solution and were stirred again for 2 hours. Post which, the solution was ultrasonicated for 30 minutes and was stirred again for 30 minutes. Then the final prepared solution was casted onto glass slides using a pipette and was left for drying in the oven at 60 °C for 12-15 hours. Then the films were retrieved from the oven and cleaned with DI water and stored in vacuum [33].

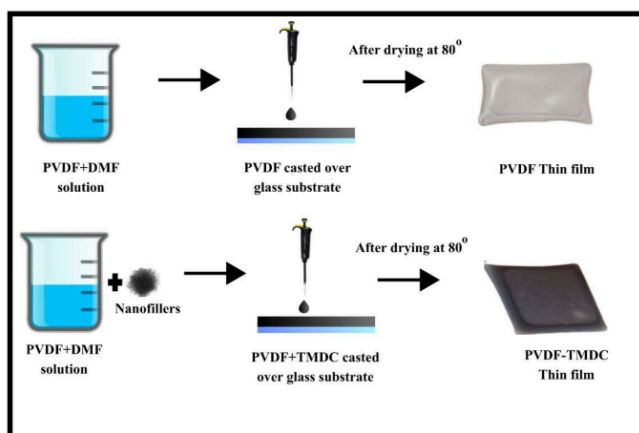


Fig. 3: Schematic illustration for the steps of film synthesis of TMDC nanofillers incorporated with PVDF using the drop casting method

2.6 Fabrication of Piezoelectric Nanogenerator

For device fabrication, the synthesized films were cut down to their requisite size of $2 \times 3 \text{ cm}^2$. This film was kept between aluminum tapes serving as electrodes for the PENG. Following this, the aluminum electrodes were connected to thin strips of copper tapes from both sides and were soldered to ensure the connection between PENG and the measurement device. The device was then sheathed with kapton tape. These fabricated devices were rectangular in shapes having dimensions of $2 \times 3 \text{ cm}^2$.

CHAPTER 3

RESULTS AND DISCUSSIONS

3.1 Characterization Techniques

XRD analysis was performed on synthesized powder samples in which the peak confirmed the correct formation of the powder using X-ray diffractometer Cu X-ray source, 3 kW (JEOL Japan model: JSM 6610LV). Fourier transform infrared (FTIR) spectra was done in order to check the content of electro active beta phase using the Perkin Elmer Spectrum II device. It confirmed the presence of the crystallization of the electro active β -phase in the synthesized thin films. Raman spectroscopy was also done using the Horiba Jobin-Yvon LABRAM-HR spectrometer in order to check the vibrational phases of the material. Field emission scanning electron microscopy (FESEM) confirmed the morphology of the surface and microstructural information of the films. A PE loop tracker was used to obtain the PE hysteresis analysis of thin films. Voltage (V_{OC}) and Current (I_{OC}) measurements were done in order to check the open loop current and voltage values of the device using the digital storage oscilloscope (Tektronix, MD0500) and a digital multimeter (Keithly DMM6500).

3.2 Characterization Results

3.2.1 XRD of powders

XRD analysis for the powders is given in Fig. 4 The JCPDS card no for SnS_2 is **00-023-0677** the JCPDS card No. for SnSe_2 is **00-023-0602**. The peaks were observed for SnS_2 at 15.20, 28.55, 32.29, 42.12, 50.24, 52.66, 55.22, 60.95, 67.43 at corresponding planes (001), (100), (002), (003), (110), (111), (200), (100), (004). For SnSe_2 the peaks were noticed at 14.59, 30.95, 40.31, 47.91, 50.28, 52.76, 57.99, 60.53, 64.17 and 67.31 at corresponding planes (001), (011), (012), (110), (111), (103), (201), (004), (202) and (113). XRD pattern confirmed the coexistence of SnS_2 and SnSe_2 phases in the heterostructure implying successful formation of the heterostructure.

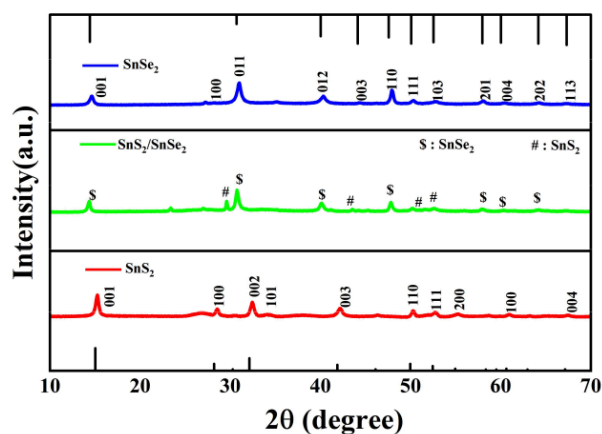


Fig. 4: XRD results of synthesised powders

3.2.2 XRD of films

For bare PVDF and PVDF/TMDCs thin films, the XRD results is shown in the Fig. 5. The peak for the non-polar α phase is detected at 18.4 degree and for the polar β phase is detected at 20.2 degree. The XRD analysis, as shown in Fig.5 * represents the diffraction peaks with respect to PVDF, the peaks for SnS_2 and SnSe_2 are represented by # and \$ respectively. This data shows the co-existence of TMDC materials and the β phase in the synthesized composite films. It shows that the electro-active β phase crystallizes in bare PVDF as well.

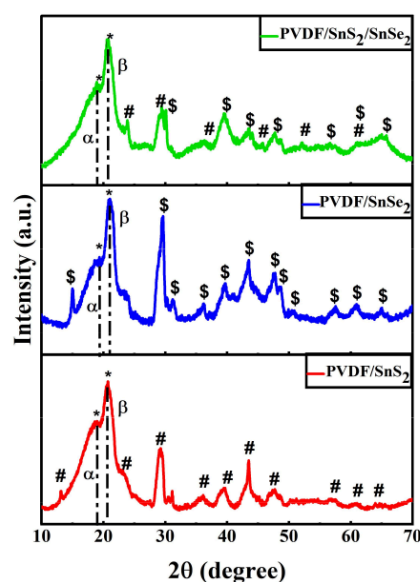


Fig. 5: XRD results of synthesized films

3.2.3 Raman spectroscopy

The Raman spectral analysis of synthesized powders is shown in the Fig. 6. Two Raman active peaks were observed namely E_{2g}^1 and A_{1g} . The E_{2g}^1 mode in the spectra is due to the in-plane vibrations between the atoms whereas the A_{1g} mode is present because of the out-of-plane vibrations. For SnS_2 , they were observed at 202.86 cm^{-1} and 311.83 cm^{-1} respectively. We know that, the E_{2g}^1 mode in SnS_2 is due to the vibration of two S atoms with respect to the Sn atom in the opposite direction [34]. The A_{1g} mode shows the out of plane vibrations of Sn-S along the c axis. For $SnSe_2$, they were detected at 179.17 cm^{-1} and 230.93 cm^{-1} respectively. The peaks for heterostructure were observed at 182.31 cm^{-1} and 291.59 cm^{-1} which matched with literature values. In our composite material, the major raman peaks do not overlap onto the individual characteristic peaks of SnS_2 and $SnSe_2$, thus confirming the synthesis of a uniformly distributed heterostructure.

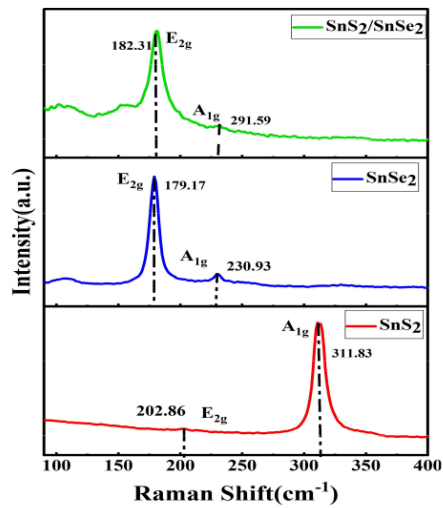
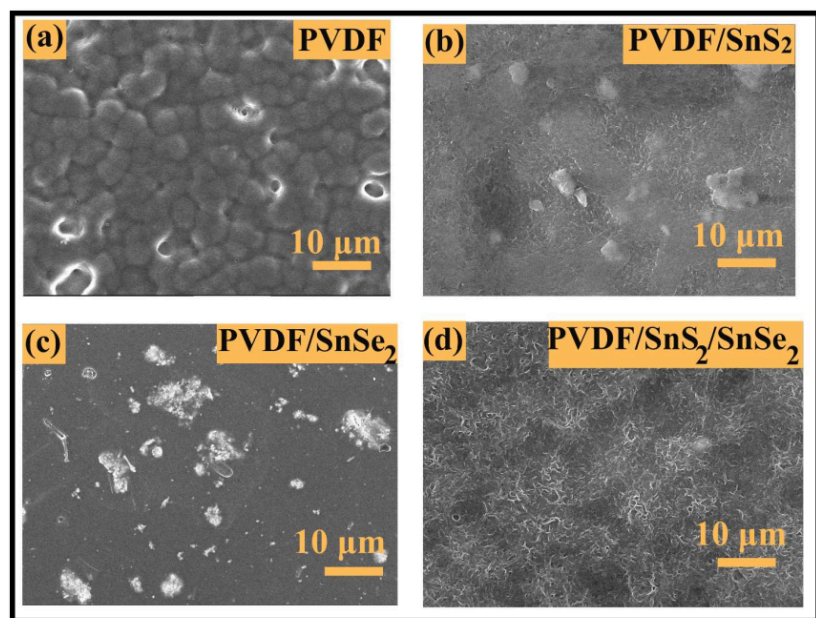


Fig. 6: Raman Spectra of synthesized TMDC powders

3.2.4 FESEM of films

The FESEM (Field emission scanning electron microscopy) morphological analysis was done out in order to study the surface morphology of the PVDF/TMDC thin films. The FESEM images of PVDF based thin films as shown in Fig. 7, depict the uniformity of the PVDF matrix. From the images shown in the Fig 7. , it can be seen that the distribution of SnS₂, SnSe₂ and SnS₂/SnSe₂ is

ubiquitous² over the PVDF polymer matrix without any signs of agglomeration which implies the effective dispersion of the nanofillers in the PVDF matrix.



²¹ Fig. 7 : FESEM micrographs for (a) PVDF, (b) PVDF/ SnS₂, (c) PVDF/SnSe₂, (d) PVDF/SnS₂/SnSe₂,

3.2.5 PE hysteresis loop of films

To compute the piezoelectric retentivity of thin films, the PE hysteresis study was done in order to elaborate on their intrinsic dipole behaviour, which is directly linked to their piezoelectric performance as shown in Fig. 8. This comparative study of hysteresis loop shows the clear presence of how polarization switches under an applied electric field, thereby confirming the presence of non centro-symmetric polarization domains in these PVDF enhanced layered dichalcogenides systems. The P_r values of PVDF, PVDF/SnS₂, PVDF/SnSe₂, PVDF/SnS₂/SnSe₂ are evaluated from the PE loop and are 6.13×10^{-2} , 7.49×10^{-2} , 7.58×10^{-2} and $8.78 \times 10^{-2} \mu\text{F cm}^{-2}$ respectively. This enhanced behavior of remanent polarization is directly linked to the generation of an enhanced piezoelectric potential under mechanical perturbation which leads to efficient charge separation and migration.

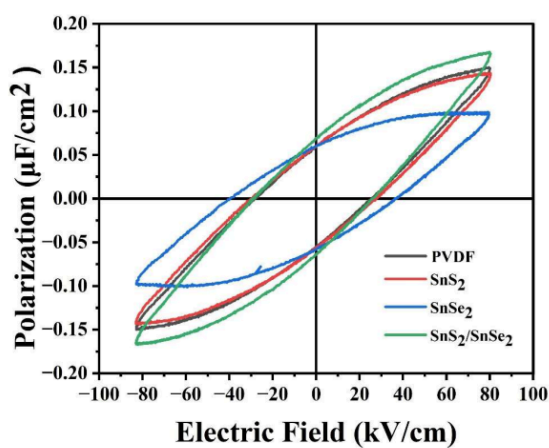


Fig. 8 : PE hysteresis loop

3.2.6 FTIR Spectroscopy of films

FTIR spectroscopy was carried out to analyze the phase transformation and to investigate the phases present in the PVDF-TMDC films synthesized at 3 wt. percentage. In the Fig.9, the beta phase is evaluated from the vibrational bands at 840 cm⁻¹ and the α -phase from the vibrational band at 762 cm⁻¹. These bands correspond to the unique bending and stretching modes related to the polar crystalline conformation of PVDF polymer matrix. The peaks in the Fig. 9 marked the presence of the beta phase, which is inherently piezoelectric and inferred that by the inclusion of TMDC materials, it gets enhanced. The beta phase was calculated by using Lambert's Beers' law using the equation [35] –

$$F(\beta) = \frac{A_{\beta}}{\left(\frac{K_{\beta}}{K_{\alpha}}\right)A_{\alpha} + A_{\beta}} \quad \text{----- (1)}$$

Where $K(\beta) = 7.7 \times 10^4 \text{ cm}^2/\text{mol}$ and $K(\alpha) = 6.1 \times 10^4 \text{ cm}^2/\text{mol}$ and these are coefficients of absorption at 840 cm⁻¹ and 762 cm⁻¹ respectively. For bare PVDF, the beta phase was evaluated to be 61 % and for 3 wt. % SnS₂, SnSe₂ and heterostructure SnS₂/SnSe₂ enhanced with PVDF, the β phase was given by 72 %, 70% and 76% respectively. Since SnS₂/SnSe₂ exhibited the maximum value of electro active beta phase, its films with PVDF at varying weight percentages (1%, 3%, 5% and 7%) were prepared and the highest value was observed at 3%. The following table shows a clear increase in the beta phase from 0 to 3 wt. %

and then a decrease on further increasing the weight due to agglomeration. We calculated β phase was as follows for different materials-

Table 01: β -Phase of synthesised films incorporated PVDF/TMDCs

Material	Electro active Beta phase
PVDF	61%
SnS ₂	72%
SnSe ₂	70%
SnS ₂ /SnSe ₂	76%

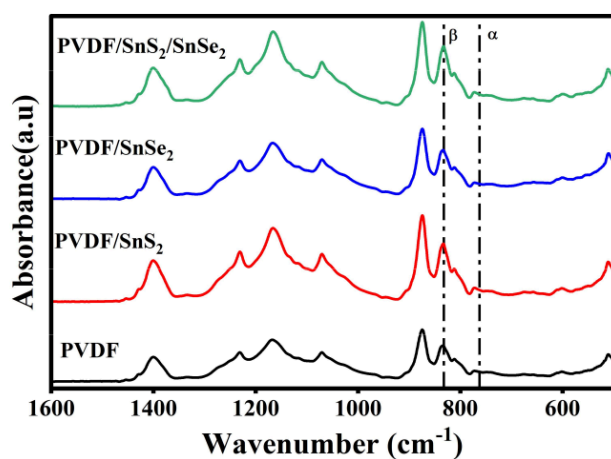


Fig. 9 : FTIR spectra of synthesized thin films

Table 02: β -Phase of synthesised $\text{SnS}_2/\text{SnSe}_2$ powder incorporated PVDF (different wt. %)

Wt. % of $\text{SnS}_2/\text{SnSe}_2$	β -phase (%)
0	61
1	77.8
3	81.5
5	76.2
7	75.6

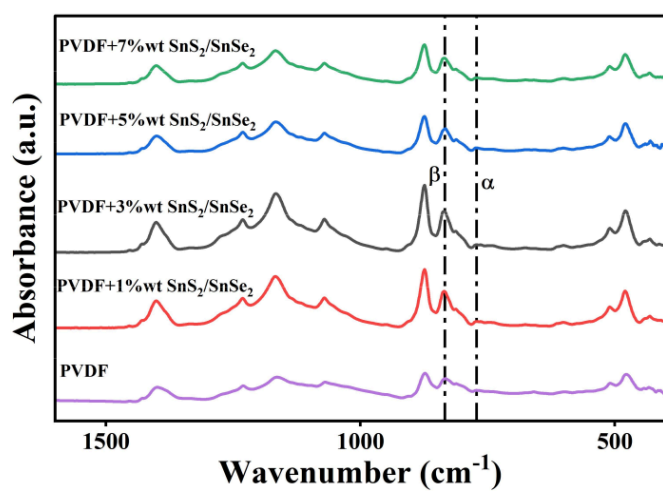


Fig. 10: FTIR spectra of synthesised $\text{SnS}_2/\text{SnSe}_2$ films incorporated with PVDF at different weight percentage

3.2.7 Electrical result Analysis

To analyze the piezoelectric response of the fabricated films incorporated with PVDF at 3 wt%, the open-circuit voltage (V_{oc}) and short-circuit current (I_{sc}) were evaluated, shown in Fig. 11 and 12. These measurements were made using the Tektronix MD034 oscilloscope and the Keysight B2985B electrometer. These values directly indicate the induced piezoelectric voltage in the sample, thereby signifying the efficient piezoelectric results of the samples. A higher value of V_{oc} and I_{sc} shows enhanced effective dipole alignment and separation of charges.

The maximum piezo response for the thin films was observed at a resonant frequency of 7 Hz. The experimental value for V_{oc} was observed to be 5.79 V for bare PVDF, however when enhanced with TMDC nanofillers, the resultant voltage was inferred to be 10.82, 7.46 and 13.20 V for SnS_2 , SnSe_2 , and $\text{SnS}_2/\text{SnSe}_2$ respectively. The values of the short-circuit current of the piezoelectric devices fabricated using PVDF and the TMDC nanofillers at 3 wt.% was reported to be 0.15, 0.57, 0.29 and 0.87 μA for bare PVDF, SnS_2 , SnSe_2 and $\text{SnS}_2/\text{SnSe}_2$ respectively.

This enhancement in the values of V_{oc} and I_{sc} corresponds to the collegial effects of improved dipole alignment arising from the broken centro and mirror symmetry in the nanofillers. As the β -phase content also complied to a similar trend, it can be inferred that by introducing asymmetry in the nanofillers, its piezoelectric response is enhanced. Therefore, the heterostructure stands out as the most promising candidate for energy harvesting applications.

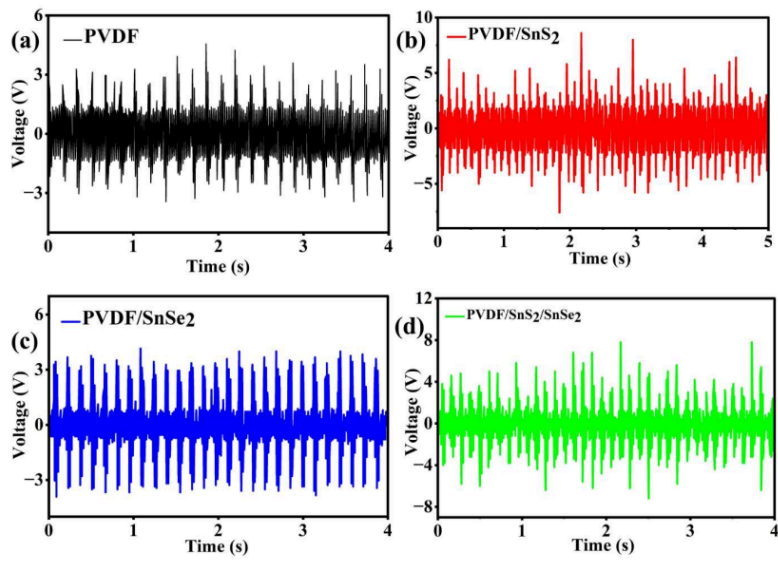


Fig. 11: Open circuit voltage for (a) PVDF, (b) PVDF/SnS₂, (c) PVDF/SnSe₂, (d) PVDF/SnS₂/SnSe₂ thin film nanogenerators at different tapping frequencies

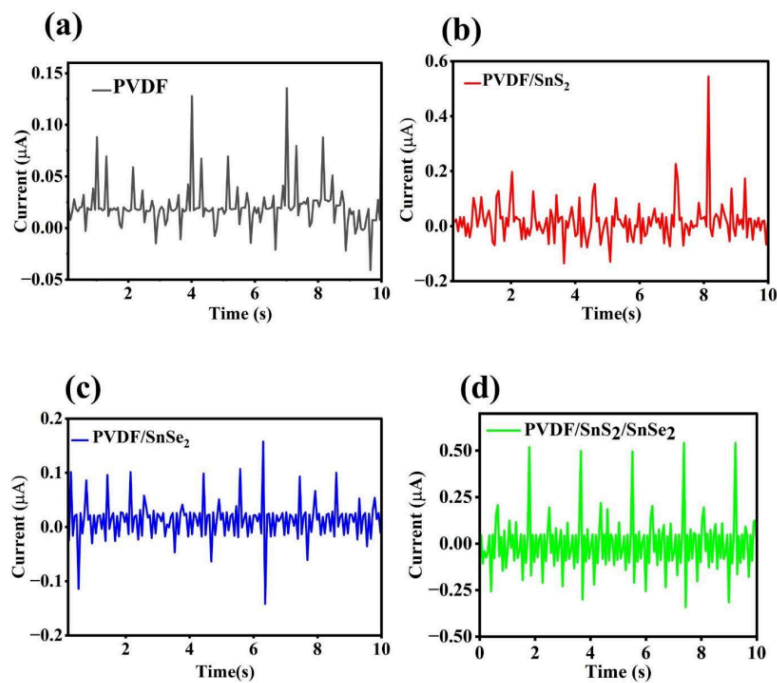


Fig. 12: Short circuit current for (a) PVDF, (b) PVDF/SnS₂, (c) PVDF/SnSe₂ and (d) PVDF/SnS₂/SnSe₂ thin film nanogenerators by continuous tapping at 7 Hz frequency.

Table 3: Comparison of piezoelectric output of PVDF and TMDC nanocomposite films

Nanogenerator	V_{oc} (V)	I_{sc} (μA)	P_r ($\mu F/cm^{-2}$)
PVDF	5.8	0.15	6.13×10^{-2}
PVDF/SnS ₂	10.8	0.57	7.49×10^{-2}
PVDF/SnSe ₂	7.4	0.29	7.58×10^{-2}
PVDF/SnS ₂ /SnSe ₂	13.2	0.87	8.78×10^{-2}

3.2.8. Piezoelectric Nanogenerator (PENG)

Our present work reports the fabrication of flexible piezoelectric nanogenerators using PVDF enhanced Tin based 2D dichalcogenides namely SnS₂, SnSe₂ and their heterostructure SnS₂/SnSe₂. A PENG works on the principle of piezoelectric effect, converting any form of mechanical stress and deformities into electrical energy. In its initial state as shown in Fig. 13, that without any applied force, the dipole alignment within the PVDF based matrix is randomly oriented creating no net electrical output. However, when a periodic mechanical force is applied, the film undergoes deformation, causing an alignment within dipoles, thereby generating a piezoelectric potential across the electrodes. This causes an accumulation of charge on the electrodes producing a potential difference which causes electrons to move through the external circuitry. Upon releasing the applied force, the dipoles revert back to their original state, hence creating a reversed charge flow. This periodic process of deformation and relaxation induces alternating electrical signals corresponding to successive cycles of compression and release.

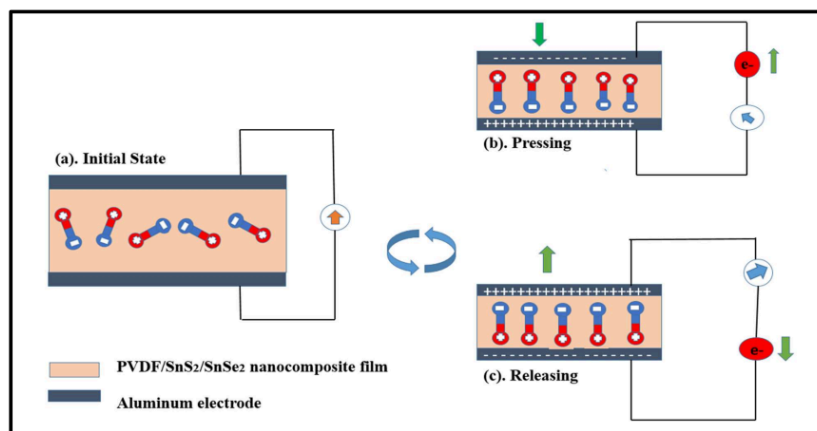


Fig.13: Schematic of piezoelectric response of PVDF/SnS₂/SnSe₂ based device with aluminium electrodes: (a) initial dipole orientation, (b) dipole alignment under pressing, and (c) reversed electron flow upon releasing

For evaluating the electrical response of the devices, the SnS₂/SnSe₂ heterostructure based PENG was exposed to periodic mechanical stress. When subjected to biomechanical stimuli, using single finger and thumb tapping, the PENG exhibited an output voltage of 0.67 V and 0.74 V respectively, as depicted in Fig. 14 (a) and (b). The resulting alternating piezoelectric response was made to pass through a bridge rectifier circuit to convert it into a unidirectional signal as depicted in Fig. 14(c). Post this, the rectified response was stored in a capacitor thus enabling effective charge accumulation and stable energy delivery to the external loads. The obtained output was thus rectified and stored in the capacitor and was successfully used to power an LED shown in Fig. 14(d).

The open circuit voltage (peak-to-peak) and short circuit current values exhibited a stable and periodic signal over multiple cycles, thereby indicating that the nanocomposite films are mechanically robust and possess good durability. Among all the devices, the heterostructure based PENG demonstrated a superior electrical output. In addition to this, the presence of SnS_2 and SnSe_2 with distinct chalcogen atoms further enhances the asymmetric charge distribution and efficient charge separation within the nanofiller induced PVDF matrix.

This improved electrical output was supported by the XRD and FTIR analysis which confirmed the enhanced dipole alignment along with the effective incorporation of nanofillers within the PVDF polymer matrix. All these results showed the potential of Tin based dichalcogenides heterostructure integrated with PVDF for its application in flexible, self-powered and wearable energy harvesting systems.

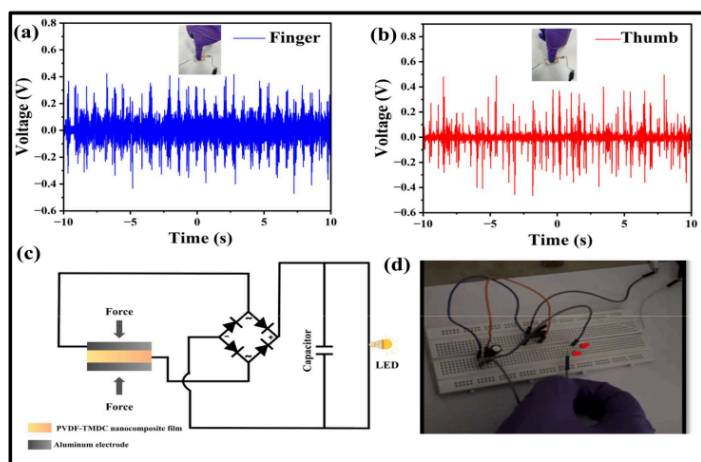


Fig. 14: Open-circuit voltage (peak-to-peak) of PENG under applied force from (a). finger (b). thumb, (c). Schematic illustration of PENG circuit for operating an LED and (d). LED powered by PENG

CHAPTER 4

CONCLUSION AND FUTURE SCOPE

4.1 Conclusion

This work successfully shows the fabrication of PVDF/TMDCs nanocomposites based films for different materials, PVDF/SnS₂, PVDF/SnSe₂ and PVDF/SnS₂/SnSe₂. The powdered samples were prepared by hydrothermal method which were then used as nanofillers within the PVDF polymer matrix as thin films using drop casting method. The SnS₂/SnSe₂ heterostructure PENG demonstrated the maximum output performance. The values of P_r values of PVDF, SnS₂/PVDF, SnSe₂/PVDF, SnS₂/SnSe₂/PVDF are evaluated from the PE loop and are 6.13×10^{-2} , 7.49×10^{-2} , 7.58×10^{-2} and $8.78 \times 10^{-2} \mu\text{C cm}^{-2}$ respectively. The output voltages were observed to be 10.82 V, 7.46 V and 13.20 V for SnS₂, SnSe₂, and SnS₂/SnSe₂ respectively. The short-circuit current was reported to be 0.15 μA , 0.57 μA , 0.29 μA and 0.87 μA for bare PVDF, SnS₂, SnSe₂ and SnS₂/SnSe₂ respectively. The fundamental reasons responsible for their enhanced response of the heterostructure based device is the improved value of electroactive beta-phase and the increased values of P_r values of the PVDF films post the incorporation TMDCs nanofillers. The device was tested under biomechanical forces via single finger and thumb tapping to show the practical application of the fabricated devices. On combining all the above obtained results, the PENG integrated with heterostructure SnS₂/SnSe₂ exhibited the most superior performance. Therefore, we propose that this device can effectively employed for mechanical energy harvesting and for powering low powered electronic devices. Our design successfully demonstrates an efficient, scalable and robust PENG with a high capability of converting mechanical energy into electrical energy.

4.2 Future Scope

- Heterostructure engineering involving other 2-D materials like Mo and W based multicomponent TMDC systems can be investigated.
- Synthesis of asymmetric Janus materials like SnSSe using chalcogen substitution for improved piezoelectric performance and output.
- Integration of PENG into wearable electronics and self-powered sensors for real-life and practical applications.
- Heterostructure powder can also be utilized for the piezocatalytic degradation of organic pollutants and azo-dyes.

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