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A Comparative Analysis of Flexible Polymer-Based Poly(vinylidene) Fluoride (PVDF) Films for Pressure Sensing Applications

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Abstract

Recently polymers and polymer-based materials are gaining lots of interest in various fields of research and development among the research community. Similarly, polymer-based piezoelectric materials are also receiving equal attention in various applications of sensors, transducers, actuators, and portable energy harvesting devices. However, many polymers available in the market, polyvinylidene fluoride (PVDF), are marking their new milestone as a futuristic material in the applications of a self-powered electromechanical device. Therefore, in this research work, flexible PVDF thin films are fabricated via a solution casting technique for pressure-sensing applications. Further nanofillers such as Zinc oxide (ZnO) and Titanium dioxide (TiO_2) with different weight percentages (0.2,0.4,0.6 and 0.8wt.%) are doped into PVDF polymer solution to enhance the electroactive phase, which eventually increases the electro potential properties. Microstructures of nanofillers and fabricated pure PVDF and PVDF nanofilms were analyzed using scanning electron microscopy. Further, pure PVDF and PVDF nanofilms were tested for piezoelectric performance using a four-probe digital oscilloscope. Finally, the outputs were analyzed and simulated using COMSOL Multiphysics.

Keywords: PVDF, Zinc oxide, Titanium dioxide, Flexible films, Piezoelectric performance.

1. Introduction

Globally for the past two decades, the demand for energy and energy resources has increased to approximately 40%,as the usage and applications have been drastically growing with the modernization of technology. Though various conventional energy sources, such as solar, thermal and nuclear power plants, could not efficiently meet the energy crisis due to multiple restrictions. Hence it is necessary to set up an alternative energy harvesting technology without causing any destruction to humans and the environment. To solve this problem, self-powered Nanogenerators were adopted by using piezoelectric materials. Piezoelectric materials can be defined as the ability of material to convert mechanical energy into electrical energy under pressure and vice versa that are naturally accumulated within them[1–3]. And the electrical output obtained from them is called Piezoelectricity. Quartz and Rochelle's salt are well-known ancient piezoelectric crystal materials. Though the application of piezoelectricity began during the 17th century with the usage of quartz crystals as transducers and sonars, they could not stand further after the invention of ceramic-based piezoelectric materials. Though ceramic-based piezoelectric materials have high responses, they also tend to fail the race as they are restricted to economic and environmental concerns. To overcome these problems, polymer-based piezoelectric materials have stepped on a new milestone in the era of piezoelectric materials. This polymer-based piezoelectric material has been grabbing everyone's attention with its stunning piezo, pyro and ferroelectric properties [4–6].

Newly energy harvesting devices and nanogenerators are fabricated from polymer-based piezoelectric materials. These materials are portraying great attention because of their unique properties such as biocompatibility, cost-effectiveness and straightforward processing methodology[4–7]. Lijun Lu et al.2020 have reported the advance of flexible PVDF-based nanogenerators by incorporating various nanofiller[8]. Polyvinylidene fluoride (PVDF) is one of the most booming polymers-based piezoelectric materials in all research and development fields[6,9–11]. Moreover, PVDF has gained prodigious attention in significant research and development areas such as super capacitors, lithium-ion batteries, sensors, actuators, electromechanical devices, transducers, energy storage, energy harvesting devices and biomedical applications[12–14].

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PVDF – an interesting nonconductive polymer from the family of fluoropolymers, is well known for its excellent dielectric and electroactive properties. PVDF is a pure non-reactive, low-density fluoropolymer whose molecular structure is simple with repeated monomer units, as shown in Figure 1. PVDF has the lowest density of 1.75g/cm^3 and is typically 50-60% semi-crystalline with a low melting point of around 175℃[1].

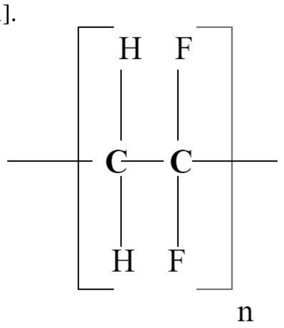

Figure 1. Molecular structure of PVDF

Due to its unique molecular structure, PVDF behaves extremely tough when exposed to extreme mechanical and thermal experiments. PVDF are more flexible in processing and can be converted into various forms, such as films and membranes[15–18]. PVDF do not restrict themselves not only to dielectric and piezoelectric properties; they are excellent in showing outstanding mechanical and thermal properties with chemical resistance under all weather conditions, and they can behave extremely resistive to all kinds of fatigue and creep. Besides all these parameters, nano-filled PVDF composites have also gained many scopes as they significantly transform the phase from nonpolar to polar[19–21]. Also, various studies have reported that the inclusion of fillers significantly improved the performance of nanofilms in multiple applications[22,23].

Zinc oxide (ZnO) is a crystalline wide-gap semiconductor from the II-VI group with a wide band gap of 3.3eV at room temperature. Also, a single crystal of ZnO is considered to possess piezoelectric and pyroelectric properties. A study has reported that ceramics and polycrystalline thin films containing ZnO may show microscopic piezoelectricity[24]. Titanium dioxide is a ceramic oxide with twelve polymorphs and high thermal conductivity. Studies have reported that when titanium dioxide is deposited in thin films, its refractive index makes it more reflective for dielectric mirrors and UV-resistant materials coatings. Various literature studies said that titanium dioxide and zinc oxide are considered to be less harmful when compared to other chemical proportions[11]. Sabry et al. 2019 have investigated nanogenerators based on nanocomposites PVDF/ZnO with different concentrations by fabricating nanofibers using the electrospinning technique. It was observed that the nanocomposite had an increase in potential with the addition of ZnO nanofillers[25]. Fakri et al. 2019 have developed a flexible hybrid piezoelectric nanogenerator based on ZnO nanorod with PVDF nanofibers to enhance output. The output power was improved compared to pristine ZnO NRs and PVDF NFs nanogenerators[26]. Nikhil DilipKulkarni et al. 2022 have investigated nanogenerators based on nanocomposites PVDF/ TiO2 using a solvent casting approach. PVDF-TiO_2 films showed a significant increment in remnant polarisation (Pr) values, indicating enhanced piezoelectric performance[27].RahulMitra et al. 2022 have developed flexible piezoelectric nanogenerators based on TiO_2 with PVDF films by solution casting technique. It resulted in those highest piezoelectric voltage responses in all the different mechanical modes[28].

The COMSOL Multiphysics software suite is an advanced tool for various computer simulations in various research, development and learning fields[29,30]. It is versatile simulation software adopted for modelling designs, devices and processes. Additionally,COMSOL Multiphysics can convert the resulting models into simulation applications. Precisely, COMSOL Multiphysics coversevery step of the modelling workflow,from defining the geometry, material properties, and physics that describe a particular phenomenon for solving and post-processing the model with accurate and reliable results[13,31].

After an extensive literature survey, it was noticed that minimal work was done on PVDF nanocomposites using a combination of ZnO and TiO_2nanofillers. In this research article, the fabrication and characterisation of flexible PVDFthin filmswith ZnO and TiO_2 were fabricated using the solution casting technique. The piezoelectric performance of neat and nanofilms was tested,andfinally, the samples were simulated using COMSOL Multiphysics software.

2. Experimental Procedure

The entire methodology carried out in this experimental study is represented in Figure 2. Preliminarily, neat PVDF and PVDF nanofilms were fabricated using the solution casting technique. Then the fabricated films were tested for piezoelectric performance using a four-probe digital storage oscilloscope. Further, the experimental results were analysed using COMSOL Multiphysics. The morphological characterisation of nanofilms was studied using Scanning Electron Microscopy (SEM), X-ray diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy were studied.

2.1. Materials

PVDF with a molecular weight of (M_w≈ 534,000) and density of 1.73 g/cm^3 was procured from Sigma-Aldrich Malaysia. Solvent N, N-dimethyl formamide with 98% purity were procured from Kavins scientific Pvt Ltd, India. Zinc oxide (ZnO) and Titanium dioxide (TiO_2) nanofillers were also purchased from Sigma-Aldrich, and all these materials were used directly without further processing.

2.2. Fabrication ofNEAT PVDF films

Initially, (10wt. %) 1 gram of PVDF is dissolved in 9 grams of DMF to fabricate neat PVDF films. The entire solution is ultrasonicated for less than 40-50 minutes at a temperature of 50ºC to obtain a homogeneous mixture of PVDF polymer matrix. Because DMF is considered one of the best (strongest) solvents for PVDF, stirring continually with PVDF yielded the best results. Then the polymer matrix is poured into a Petri dish and castedin a hot air oven at 100ºC for 50 minutes to get transparent and flexible, Figures 3 (a) and 3(b) shows the images of neat PVDF and its flexibility bytwisting of the film.Also, it was noted that the thickness of the film was approximately 0.8 to 1mm.

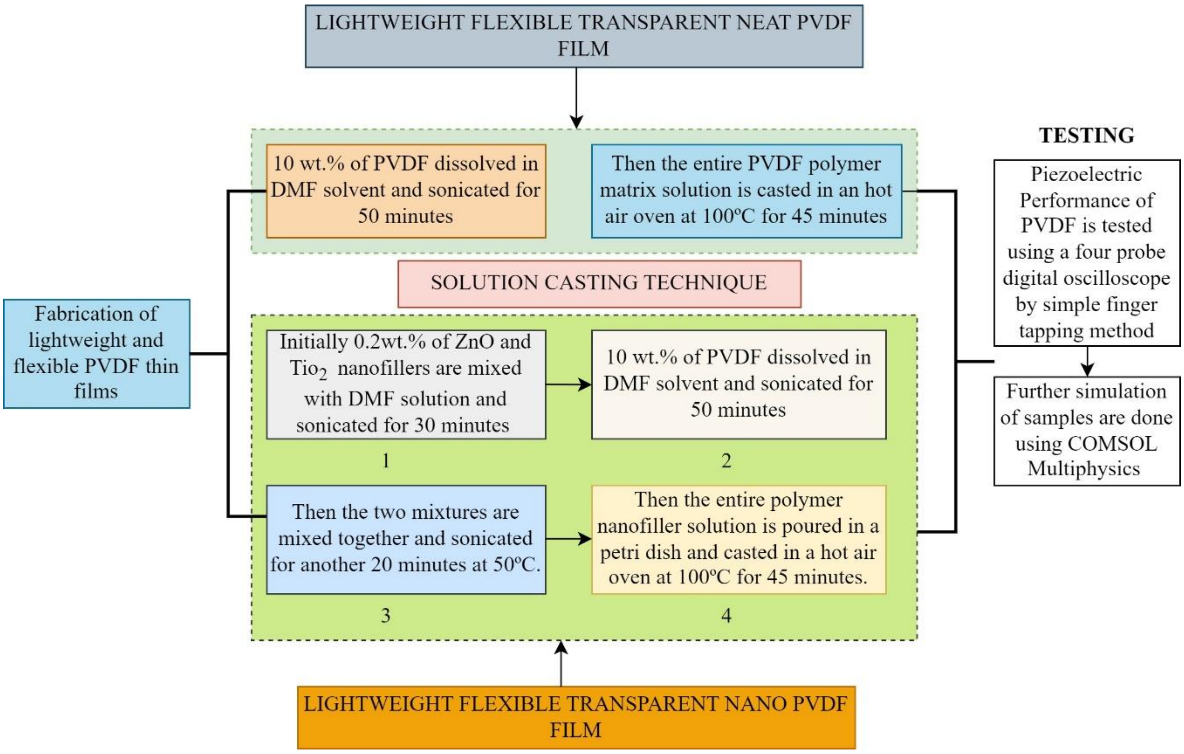

Figure 2. The entire methodology carried out in the research work

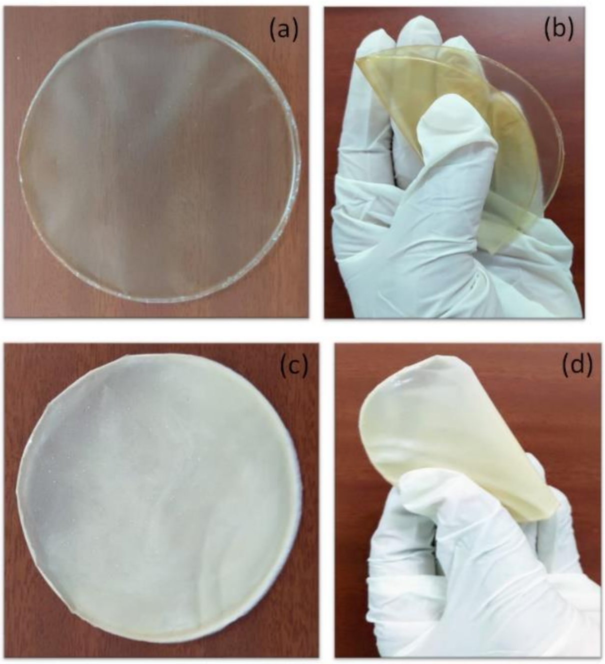

Figure 3. (a) and (b) Images of PVDF neat film and twisting of the film to confirm its, (c) and 3(d) Images of PVDF Nano film containing 0.2wt.% of ZnO and $TiO_2$and twisting of the film to confirm its flexibility.

2.3. Fabrication of PVDF nanofilms

For the fabrication of PVDF nanofilms, initially,0.2wt % of ZnO and TiO_2 fillers were dissolved separately in DMF solvent and sonicated for 30 minutes at 50 ºC. The required amount of ZnO and TiO_2 to be added to a solution of PVDF in order to obtain a nanocomposite with 0.2 wt.% of nanofillers is 0.02 g (0.01g of ZnO and 0.01 of TiO_2). Further, the nano-mixed solution is mixed into a previously prepared PVDF matrix solution, where PVDF is dissolved in a DMF solution. Then the mixture is sonicated for 40 minutes to prepare a homogeneous nano matrix solution. Finally, this PVDF nano matrix solution is poured into a Petri dish and cured at 100ºC for 2 hours to get a flexible PVDF/ZnO/TiO_2 nanofilm. The same procedure is followed for various samples of 0.4wt%,0.6wt% and 0.8wt% of ZnO and TiO_2 nanofilms, as mentioned in Table 1. Figure 3(c) and 3(d). shows the image of PVDF/ZnO/TiO_2nanofilms and the twisting of the film to confirm its flexibility.

Table 1. Composition of PVDF samples fabricated via solution casting technique

S.No	Sample Composition	PVDF wt.%	ZnO and TiO_2
S1	Neat 10wt.% PVDF	10wt.% (1-gram PVDF and 9 gram of DMF)	-
S2	10wt.% PVDF + 0.2, wt.% of ZnO and TiO_2	10wt.% (1-gram PVDF and 9 gram of DMF)	0.2wt% of ZnO and TiO_2
S3	10wt.% PVDF + 0.4, wt.% of ZnO and TiO_2	10wt.% (1-gram PVDF and 9 gram of DMF)	0.4wt% of ZnO and TiO_2
S4	10wt.% PVDF + 0.6, wt.% of ZnO and TiO_2	10wt.% (1-gram PVDF and 9 gram of DMF)	0.6wt% of ZnO and TiO_2
S5	10wt.% PVDF + 0.8, wt.% of ZnO and TiO_2	10wt.% (1-gram PVDF and 9 gram of DMF)	0.8wt% of ZnO and TiO_2

3. Material Testing and Morphological Characterisations

3.1. Scanning electron Microscopy (SEM)

The microstructure of PVDF powder ZnO, TiO_2nanofillers and final fabricated samples of neat PVDF films and PVDF/ZnO/TiO_2 nanofilms were analysed using ZEISS EVO 18 scanning electron microscopy. This characterisation ensures the microstructure, size and dispersion of fillers in the nanofilms composites.

3.2. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis is used to obtain an infrared spectrum of absorption or reflection of solid liquids. Neat PVDF and 0.8wt.% of PVDF nanofilms were analysed for FTIR spectroscopy to identify and distinguish different crystalline structures of PVDF. TIR was carried out in a range of 400-4000 cm^{-1}. Also, this analysis is used to determine the nanofillers used.

3.3. X-Ray Diffraction (XRD)

XRD analysis were carried out for all the fabricated PVDF films by Bruker advanced diffraction instrument and the data were recorded from 2θ.

3.4. PiezoelectricPerformance Testing using fourprobes Digital Oscilloscope

To test the piezoelectric performance of the films, PVDF neat films and PVDF nanofilms with various weight percentages of fillers were chosen. All the fabricated samples were pasted with silver paste on both sides, acting as electrodes to which the wires are connected to the four-probe digital oscilloscope (DS1054Z), as shown in Figure 4. The entire setup has been laminated to avoid external influences. The output voltage generated from neat PVDF and PVDF nanofilm samples was recorded by simple finger tapping[32] over the centre of the film. Finally, the readings were recorded, and time Vsvoltage graphs was plotted for all the samples respectively.

3.5. Analysis and Stimulation of fabricated samples using COMSOL Multiphysics software

Initially, the 3D CAD model of the fabricated samples was designed for neat PVDF film and PVDF nanofilms using this software and specifies the Multiphysics model of piezoelectric applications. Assign initial and boundary conditions. Here, solid mechanics and electrostatics are taken to study the piezoelectric effect. As for the boundary condition for the mechanics, when we apply the force, the load is applied to the top of the film while the lower part is kept fixed. For the boundary conditions for electrostatics, the positive terminal is at the top, and the ground is at the bottom. The mesh of the 3D model is done by free tetrahedral. Solve the integration test, and then post-process the film for stimulating results.

4. Results and Discussion

4.1. Scanning Electron Microscopy

Scanning electron microscopy (SEM) was evaluated for PVDF, ZnO and TiO_2. Figure 5(a),5(b,) and 5(c) show the microstructure of PVDF powder and ZnO, TiO_2 nanofillers. Figure 5(d) shows the morphology of pure PVDF films, which indicates that PVDF has dissolved uniformly in DMF solvent, and we could notice that the films are free from pores. The surface is transparent with an even surface, and we could see no agglomeration. Figure 5(e) depicts the morphology of 0.8wt.% of ZnO and TiO_2nanofilms.This image shows that the surface has few irregularities and small agglomerations. Still, it is also noticed that nanofillers are evenly dispersed in large amounts in the PVDF matrix solution.

4.2. FTIR Analysis of PVDF Films

The effect of ZnO nanofiller on crystalline phase and piezoelectric properties was investigated using FTIR spectroscopy. Figure 6(a) &(b) represents FTIR analysis of PVDF neat and PVDF nanofilms—pure PVDF and 0.8 wt.% ZnO and TiO_2 with PVDF nanofilms are identified between 400-1600cm^{-1} was identified. Figure 6(a) represents transmittance (%) of pure PVDF films, which means, after annealing, the formation of the electroactive phase or the β-phase with vibration peaks at 1401,1275 and 1172 cm^{-1}. Also, peaks at 1070 and 873 cm^{-1} were noticed, corresponding to α-phase. This identifies that pure PVDF films can achieve β-phase after annealing without adding nanofillers. Similarly, Figure 6(b) represents PVDF nanofilms incorporated with 0.8wt.% of ZnO and TiO_2. FTIR analysis confirms the presence of TiO_2 and ZnO nanofillers at peaks 483 and 1390 $cm^{-1,}$ and also we can observe an additional peak at 1236, denoting the semi-polar γ-phase which is the nucleation of β-phase respectively. This indicates that the addition of ZnO and TiO2 interaction with PVDF changed its original crystalline structure and other groups, resulting in the enhancement of the β-phase. And as it was noticed that as ZnO content increases, some bands of PVDF gradually decreased.

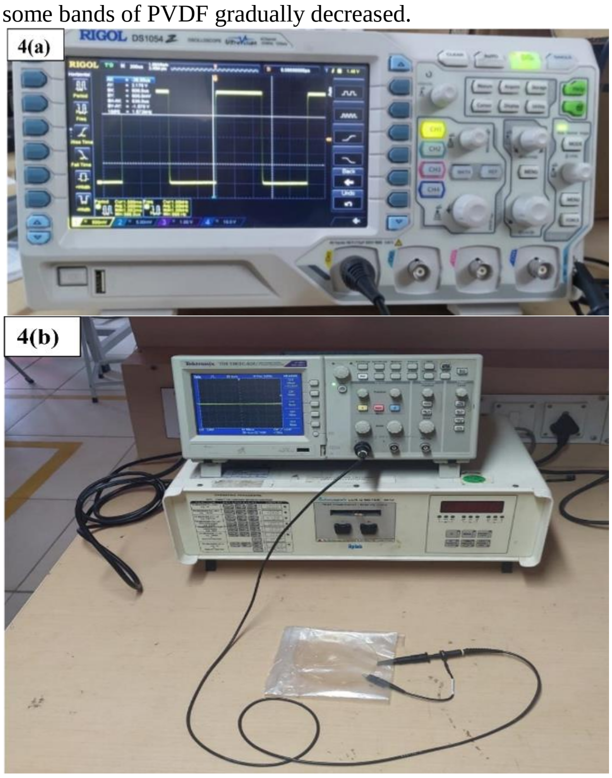

Figure 4. (a)Four-probe Digital Storage Oscilloscope (**DS1054Z**), (b) PVDF connected to Digital Storage Oscilloscope.

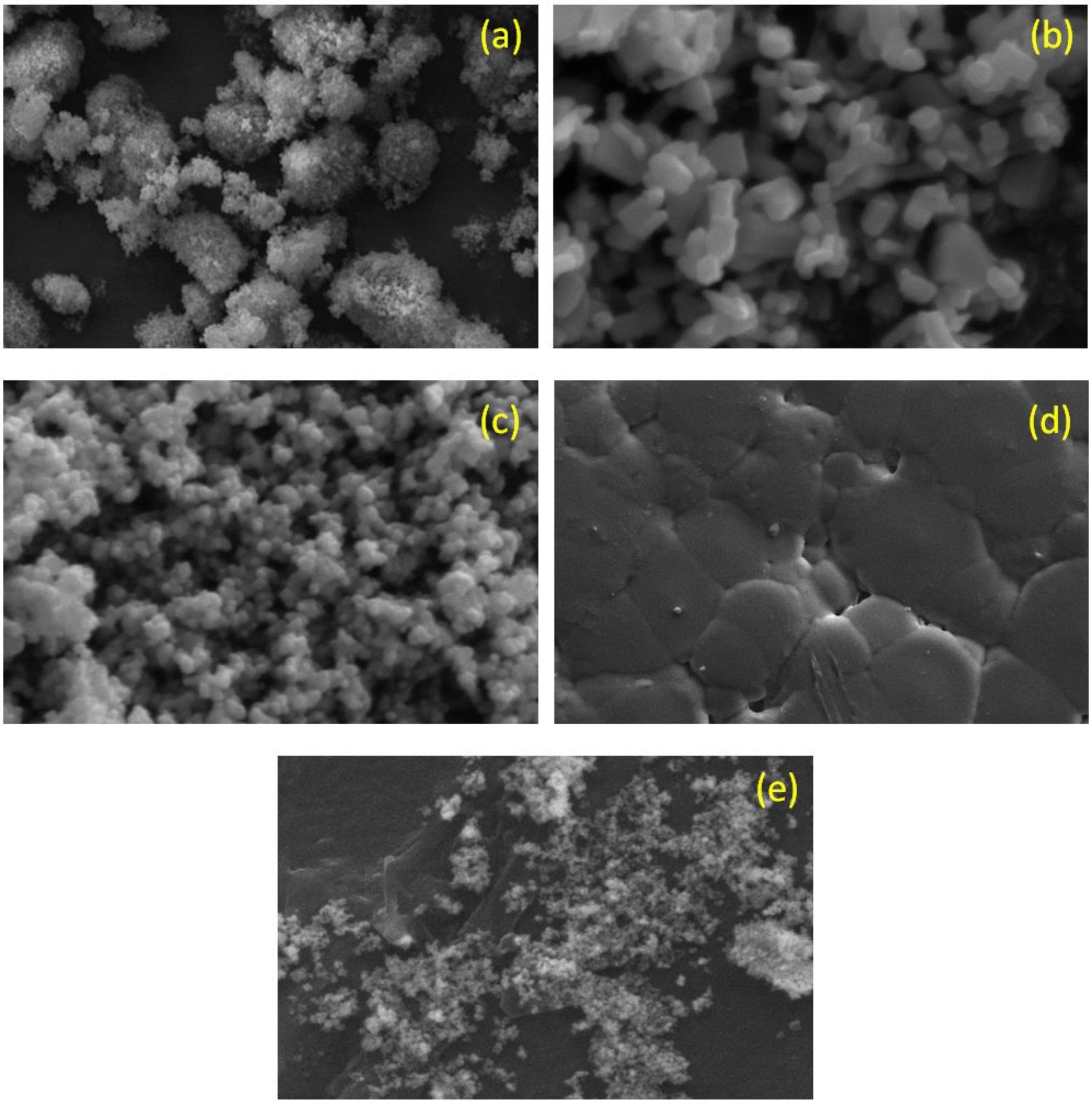

Figure 5. (a) 5(b) and 5(c) SEM images of PVDF, ZnO and TiO_2 nanofillers, (d) and 5(e) SEM images of neat PVDF films and 0.8wt.% ZnO and TiO_2 PVDF nanofilms

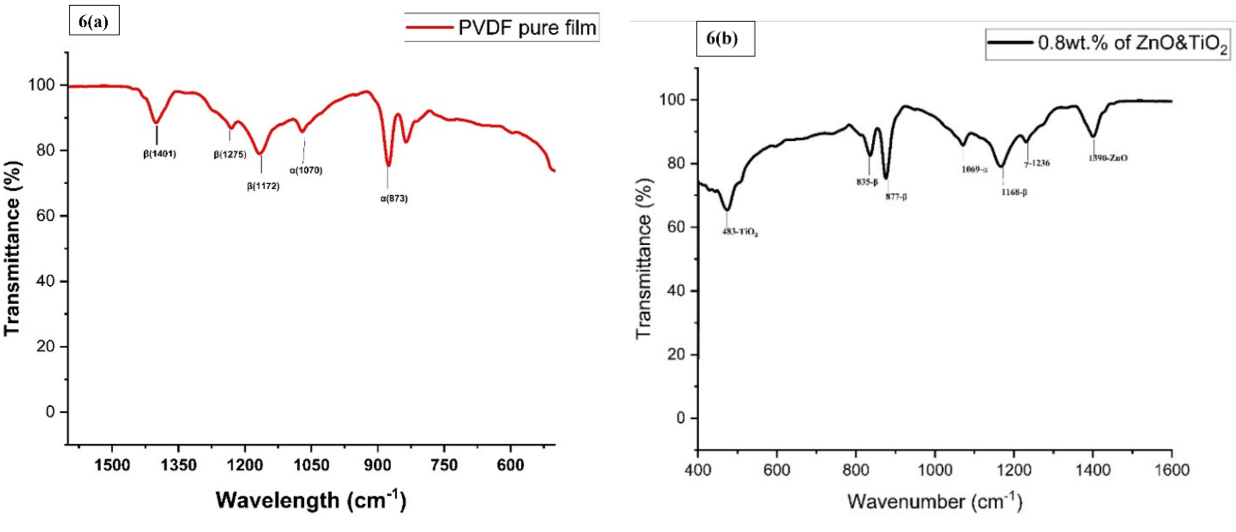

Figure 6. (a) FTIR spectra of PVDF film and (b) PVDF/0.8wt.% ZnO and TiO_2

4.3. X-Ray Diffraction (XRD) Analysis of PVDF Films

Figure7. represents the XRD analysis of nanofillers, pure PVDF films and of PVDF incorporated with 0.8wt.% of ZnO and TiO_2. The diffraction spectra peaks at 36.25°, 25° confirms the presence of ZnO and TiO_2 nanofillers. Also, peaks at 17.5°,19.7° indicate the presence of α-phase of PVDF, and peaks at 20° confirm the characteristics of β-phase both in pure and nano PVDF films. Additionally, peak at 19.3° indicates the presence of semi polar γ-phase.

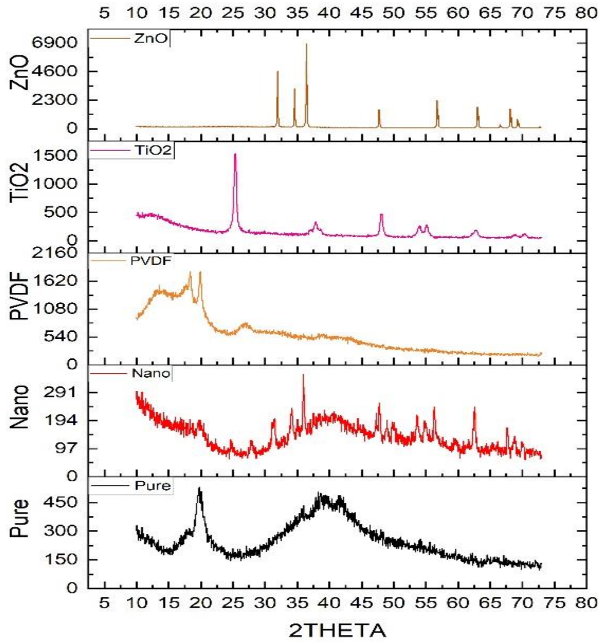

Figure 7. XRD spectra of nanofillers and PVDF nanofilms

4.4. Piezoelectric performance of PVDF neat films using Digital Oscilloscope

To test the piezoelectric performance of the polymer composites, PVDF neat films and PVDF nanofilms with various weight percentages are chosen. The output voltage generated from the samples was recorded by simple finger tapping over the film, and graphs were plotted for time Vs voltage during a time of 10 seconds with certain frequency calculated using a stopwatch. Figure 8. shows that the maximum peak voltage (V_{PP}) for neat PVDF films containing 10wt.% of PVDF was recorded as 58.1mV. Also, from figure8. it was noted that V_{pp} for 0.2wt.%,0.4wt.%. 0.6wt.% and 0.8wt.% of nanofilms was 70.9mV, 89.5mV, 110.1mV, 119.6mV respectively. From this, we can conclude that with the increase of nanofillers, the piezoelectric performance of the films also increases. This is due to the uniform dispersion and crystalline piezoelectric nature of ZnO, which enhance the piezoelectric performance of the film. As mentioned in various literatures, ZnO has been widely used for enhancing the β-phase of PVDF composites, which eventually increases the electro active properties making it more suitable for energy harvesting applications. As the weight percentage of ZnO increases the piezoelectric properties of PVDF nanocomposites increase. So that 0.8wt.% of ZnO and TiO_2 nanocomposites can achieve high electric potential with great thermal stability.

4.5. Stimulation analysis using COMSOLMultiphysics

The simulation result or the electric potential of pure PVDF and PVDF nanofillers are analysed using COMSOL Multiphysics. Figures9(a) and 9(b)showthe electric potential result for neat PVDF film and 0.8 wt. % of ZnO and TiO_2PVDF nanofilms. The diagram shows that the PVDF nanofillers with 0.8 wt.% have the highest voltage output, and the neat PVDF has the lowest result. The PVDF nanofillers with 0.8 wt.% produce a higher voltage than PVDF under the same loading. The voltage increases as the of nanofillers increases. The yellow colour region is where the output voltage is determined. Because the bottom structure is connected to the ground, the base voltage is usually zero. Table 2. Shows the experimental and statistical results of samples.

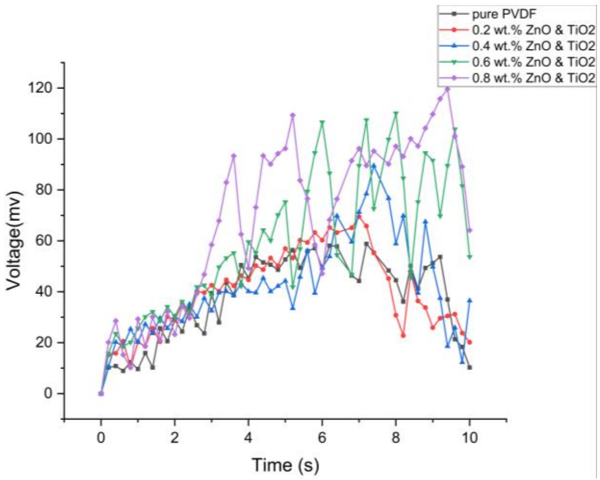

Figure 8. shows the Voltage Vs Time graph of neat PVDF film. and 0.2,0.4,0.6 and 0.8wt.% of ZnO and TiO_2, nanofilms.

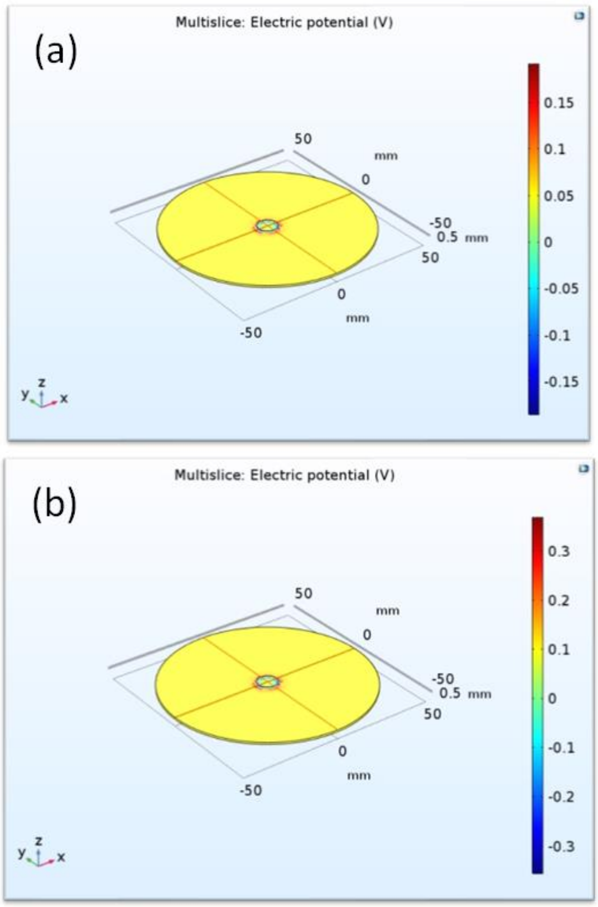

Figure 9. (a) and 9(b) images show the stimulated result for neat PVDF film and PVDF with 0.8 wt. % of ZnO and TiO_2 nanofillers

Table 2. Experimental and analysed voltage for the samples

S.No	Sample	Experimental voltage	Analysed voltage
S1	Neat 10wt.% PVDF	58.1mV	43-60 mV
S2	10wt.% PVDF + 0.2, wt.% of ZnO and TiO_2	70.9mV	58-76 mV
S3	10wt.% PVDF + 0.4, wt.% of ZnO and TiO_2	89.5mV	72-87 mV
S4	10wt.% PVDF + 0.6, wt.% of ZnO and TiO_2	110.1mV	90-108 mV
S5	10wt.% PVDF + 0.8, wt.% of ZnO and TiO_2	119.6mV	95-123mV

5. Conclusion

From the overall research work, pure PVDF and PVDF/ZnO/TiO_2 nanocomposite films with different weight proportions of nanofillers have been fabricated via the solution casting technique. Morphological characterisation, such as SEM, FTIR, XRD analyses revealed that the size of nanofillers and the dispersion of fillers were uniformly dispersed in the matrix solution, which played a crucial role in enhancing the piezoelectric performance. Also, FTIR and XRD analyses confirm the presence of nanofillers and formation of crystalline structure of PVDF and enhancement of β- phase by increase of ZnO nanofiller. From this work, the following conclusions were made:

1. 1. Fabrication of flexible PVDF and PVDF nanofilms with a thickness of 1mm were fabricated using the solution casting technique.
2. 2. The experimental piezoelectric performance of the fabricated films was tested using a digital four-probe oscilloscope by simple finger tapping at the centre of the film. Notably, the maximum output voltage of neat PVDF film was 58.1mV, whereas for 0.8wt.% of PVDF nanofilm was 119.6mV. This showed that an increase in nanofillers eventually increased the piezoelectric performance of the PVDF films.
3. 3.Further statistical analysis of the fabricated films was analysed using COMSOL Multiphysics software. From this, we observed that the electric potential of neat PVDF film was in the range of 43-60mV, whereas for 0.8wt.% of PVDF nanofilms was around 92-123mV. A comparative chart has been created for both statistical and experimental output results.
4. 4. It is concluded that these flexible PVDF nanofilms are portable and easy to use for pressure-sensing applications.

Conflict of Interest

This is to inform you that the research article has no conflicts of interest among the mentioned authors.

Nomenclature

1. PVDF- Polyvinylidene Fluoride
2. ZnO- Zinc Oxide
3. TiO_2- Titanium dioxide
4. mV- Millivolts
5. Hz- Hertz
6. wt.%- weight percentage
7. SEM- Scanning Electron Microscopy

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