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# " Optimizing Solution Blow Spinning of PVDF Nanofibers: A Study on Morphology, Crystalline Phases, and Piezoelectric Performance"

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# Abstract

This study investigates optimizing the fabrication of polyvinylidene fluoride (PVDF) nanofiber (NF-PVDF) mats produced via the solution blow spinning (SBS) technique, with a focus on characterizing the morphological, crystalline, and piezoelectric response properties for energy harvesting applications. NF-PVDF mats were fabricated with varying PVDF solution concentrations with values of 12 wt.%, 15 wt.%, and 18 wt.%, solution feeding rates of 3 mL/hr. and 7 mL/hr., and the distance between the spraying nozzle and collecting roller with 25 cm and 50 cm values. The resulting nanofibers were characterized using Scanning Electron Microscopy (SEM) to assess fiber diameters, fiber agglomerations, and the presence of beads. X-ray diffraction (XRD) and Fourier-transform infrared Spectroscopy (FTIR) were employed to determine the crystalline structure, specifically analyzing the crystallite size and beta phase content. The piezoelectric response of the NF-PVDF mats was evaluated by measuring the piezoelectric coefficient d33 and the generated output voltage under applied cyclic forces of 0.1, 0.2, 0.3, 0.4, and 0.5 N at a frequency of 1Hz. The results demonstrated that the fabrication parameters significantly influence the nanofibers' morphology and crystalline structure, which in turn affect the piezoelectric properties. Factors such as finer nanofiber diameters and minimal beads and agglomerations presences influence the piezoelectric performance. The study provides critical insights into the relationship between SBS processing parameters and the functional properties of PVDF nanofibers, paving the way for the development of high-performance piezoelectric materials for use in energy harvesting applications.

Keywords: Nanofibers; PVDF; Solution Blow Spinning; Morphology; Crystalline Phases; Piezoelectricity; Energy Harvesting

### 1. Introduction

The growing demand for portable and wearable electronic devices has spurred research into self-powered systems that harness environmental energy. Piezoelectric materials have emerged as promising due to their ability to convert mechanical energy into electrical energy through the piezoelectric effect. The integration of piezoelectric materials into textiles has led to the development of piezoelectric textiles, which can harness energy from mechanical stimuli, paving the way for self-powered wearable electronics, sensors, and energy-efficient systems [1]. Piezoelectric textiles have demonstrated significant potential in energy harvesting, with wearable nanogenerators harnessing energy from human movements. Additionally, fabrics that generate energy from wind have been used for outdoor and environmental applications [2]. They have been used also in pressure sensors for healthcare and structural health monitoring of composite materials, showcasing their versatility in various fields [3]. Polyvinylidene fluoride (PVDF) is a versatile material with high chemical resistance, thermal stability, mechanical strength, and purity [4]. It is ideal for harsh chemical environments, and semiconductor industries, and it has good electrical properties. It has low water absorption, enhancing its stability in moist environments. PVDF is also UV and radiation-resistant, extending its lifespan in outdoor and high-radiation applications [4]. It is biocompatible, making it suitable for medical and biomedical applications [5]. PVDF also has piezoelectric and pyroelectric properties, making it useful in sensors, actuators, and energy harvesting [6, 7]. PVDF and its copolymers are also popular for their high piezoelectric coefficients, flexibility, and ease of processing with five crystalline phases ( $\alpha$ ,  $\beta$ ,  $\gamma$ ,  $\delta$ , and  $\epsilon$ ) [8]. The  $\beta$ -phase is more desirable for its strong piezoelectric response so various studies have focused on enhancing PVDF's β-phase content through processing techniques and post-treatment [9, 10]. It can be processed using techniques like extrusion, injection molding, electrospinning, and solution blow spinning [11, 12]. Previous studies investigated PVDF material for energy harvesting applications using different techniques as demonstrated in

#### Table 1.

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Material Used	Fabrication Technique	Key Findings	Application	Reference
PVDF	Melt extrusion and	Piezoelectric output:	Self-powered sensor for	[13]
	leaching	3.1 V- 25nA	sports wearables	
		High sensitivity:	_	
		0.092 V/N		
		Short response time:		
		70 ms		
PVDF	Electrospinning and 2D	1V voltage output	Real-time online damage	[14]
	braiding	Sustains long-term	monitoring in 3D textile	
		cycles at 4Hz	composites	
PVDF	Solution blow spinning	Tensile strength: 12.6	Energy harvesting,	[15]
	(SBS)	MPa	footstep generation	
		Voltage output: ~2.5		
		V		
PVDF,	Weaving	Highest sensitivity:	Large-scale pressure	[16]
Polyethylene		83 mV N	sensing, sports practice	
terephthalate (PET)*			monitoring	

Table 1	Examples of	of previous	investigations	for PVDF in ene	ergy harvesting	g applications

One promising technique for fabricating PVDF fibers with enhanced piezoelectric properties is solution blow spinning (SBS). This method is versatile, scalable, and cost-effective, combining the principles of electrospinning and solution spraying to produce micro- and nanofibers with diameters ranging from 50 nm to 5  $\mu$ m. It involves ejecting a polymer solution into a high-velocity gas stream, producing solid polymeric fibers. This technique is suitable for large-scale industrial applications, offering rapid production, simple setup, and versatility with various polymer solutions. Its simple structure enhances mechanical properties and is environmentally friendly, reducing solvent use and minimizing waste [13, 14]. SBS parameters like solution concentration, solution flow rate, and collecting distance influence significantly the morphology and crystalline structure of PVDF nanofibers (NF-PVDF), affecting their piezoelectric performance [17, 18].

There is a need for a systematic analysis of the morphological, structural, chemical, and piezoelectric response properties of NF-PVDF, specifically for textile-based energy harvesters. This study aims to examine the impact of fabrication parameters, including PVDF solution concentrations, feeding rates, and collecting distances, on the morphological, crystalline, and piezoelectric properties of NF-PVDF mats produced through the SBS technique. The goal is to optimize these parameters to improve the nanofibers' performance for energy harvesting applications. Characterization techniques such as scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier-transform infrared spectroscopy (FTIR) were employed to analyze the morphological and crystalline properties of the fabricated NF-PVDF mats. The piezoelectric response was assessed under different mechanical stimuli, including applied forces using a piezoelectric testing setup and the measurement of the piezoelectric coefficient d<sub>33</sub>. These analyses provide crucial insights for optimizing the design of efficient piezoelectric textile energy harvesters.

### 2. Experimental Work

# 2.1. Materials

- Poly (vinylidene fluoride) (PVDF) powder (Kynar® 761, King of Prussia, PA, USA) from ARKEMA.
- N, N-Dimethylformamide Solvent (DMF anhydrous, 99.8%, Sigma Aldrich, Taufkirchen, Germany).
- Aluminum sheets (Conductivity of ~3.33 x 10<sup>7</sup> S/m Resistivity of 2.65 × 10<sup>-8</sup> Ω·m) to work as electrodes for NF-PVDF.

# 2.2. Preparation of NF-PVDF

Twelve samples of NF-PVDF were fabricated using the SBS technique. The process involved preparing the PVDF polymeric solution and fabricating the nanofiber through SBS with optimized parameters of PVDF solution concentrations, solutions feeding rates, and collecting distances.

#### 2.2.1. Solution Preparation

PVDF solution was prepared by dissolving different concentrations of PVDF powder (12 w.%, 15 wt.%, and 18 wt.%) in 15ml of DMF solvent. Then the mixture was heated and stirred using a magnetic hot plate at a temperature of 75 °C for 180 min, then the mixture was cooled for 20-25 min at  $20\pm2$ °C and relative humidity of  $65\pm5$ % before fabrication. The SBS setup, as shown in **Error! Reference source not found.**, used a 20ml plastic syringe with a 23-gauge needle filled with 15 ml of the PVDF solution and pumped using syringe Pumpat at feeding rates of 3 and 7 mL/h. The syringe is connected to the opened left end of the SBS concentric nozzle, so the solution passes through it and exits from the right end.



Fig. 1. A schematic diagram of the SBS setup for fabricating nanofiber mats

As shown in Error! Reference source not found., the concentric nozzle consists of 5 sections, the polymer solution inlet at the left side of the nozzle, the solution chamber, and the air inlet in the middle (~4 mm diameter) which transfers the pressured air into the air chamber, the last section is the spraying hole (~1 mm diameter) at the right end of the nozzle.



Fig. 2. Sections of solution Blow Spinning Concentric Nozzle

The pressured air in the air chamber is  $\sim 0.4-0.6$  bar supplied through the air compressor. A drum collector provided with a mesh length of 30 cm and outer diameter of 3 inches (7.62 cm), rotating with a speed of 1.4 rpm, is set at different distances of 25 and 50 cm from the spraying nozzle and the time of fabricating each sample ranged from 50-75 min according to processing conditions.

Table 2 and

 Table 3 illustrate the NF-PVDF spinning parameters and samples` specifications.

Parameter	Parameter Values			
<b>PVDF Concentration (wt. %)</b>	12 wt.% 15 wt.% 18		18 wt.%	
Collecting Distance (cm)	25 cm 50		50 cm	
Feeding Rate of Polymer Solution (ml/hr.)	3 ml/hr. 7 ml/hr.		7 ml/hr.	
Air Pressure (bar)	~ 0. 4-0. 6 Bar			

# **Table 2.** The parameters of processing the nanofiber mat samples

Table 3.	NF-PVDF	sample s	pecification
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Sample no.	Sample Code	<b>PVDF Concentration (wt.%)</b>	Feeding Rate (ml/hr)	Collecting Distance (cm)		
1	A1	12	3	25		
2	A2	15	3	25		
3	A2	18	3	25		
4	A4	12	3	50		
5	A5	15	3	50		
6	A6	18	3	50		
7	A7	12	7	25		
8	A8	15	7	25		

1		1			
	9	A9	18	7	25
	10	A10	12	7	50
	11	A11	15	7	50
	12	A12	18	7	50

#### 2.3. Characterization of PVDF Nanofiber Samples

# 2.3.1. Morphological Characterization

The surface morphology of the PVDF nanofiber mats was analyzed using scanning electron microscopy (SEM). The preparation involved cutting small sections from each film and sputtering them with a thin layer of gold by Quorum Q150, (UK) for enhanced conductivity and image clarity. The SEM images were captured using a TESCAN VEGA3, (Brno, Czech Republic) microscope at an acceleration voltage of 10 kV, with magnifications up to 20K x, allowing for detailed observation of fiber diameter, uniformity, agglomerations, and bead formation. Fiber diameters for each sample were measured using ImageJ software (Madison, WI, USA). A quantitative analysis of fiber diameter average and distribution was conducted at different imaging scales (10  $\mu$ m, 5  $\mu$ m, and 2  $\mu$ m). Fiber morphological experts qualitatively assessed beads` presence and fiber agglomerations.

### 2.3.2. Crystalline Structure Characterization

The crystalline structure and phase composition of the fabricated nanofibers were tested using an X-ray diffractometer (XRD) by BRUKER D8 ADVANCE (Massachusetts, USA) with Cu Ka (1.54059 Å=0.154059 nm) radiation with 2 $\theta$  angle range from 5° to 80°, scanning rate of 3°/min, and step angle of 0.05°.

#### 2.3.3. Crystalline Phases Characterization

The Fourier transform infrared (FT-IR) was operated in ATR mode to calculate the  $\beta$  phase content using Bruker VERTEX 80 (Germany), combined Platinum Diamond ATR, which comprises a diamond disk as that of an internal reflector in the range of 4000–400 cm<sup>-1</sup> with resolution of 4 cm<sup>-1</sup>, and refractive index 2.4.

#### 2.3.4. Piezoelectric characterization

The piezoelectric response of the NF-PVDF samples was conducted by cutting the nanofiber mat into a size of  $(15 \times 40 \text{ mm}^2)$  and then sandwiched between 2 aluminum electrodes (~33.3 x  $10^6 \text{ S/m} - 2.65 \times 10^{-8} \Omega \cdot \text{m})$  connected to 2 copper electrical wires as shown in **Error! Reference source not found.** The piezoelectric setup measured the output voltage of the 12 samples, which is shown in **Error! Reference source not found.** (Copyrights© to Center of Smart Materials, Nanotechnology and Photonics (CSMNP), Alexandria University, Egypt). A frequency generator application was used to interface with an amplifier, which in turn was connected to a speaker system designed to deliver vibrational cyclic forces of 0.1, 0.2, 0.3, 0.4, and 0.5 N. A movable digital balance precisely regulated these forces. The vibrational forces were applied on each sample via a pressing head with a diameter of 1 cm, attached to the speaker (vibrating head) at a constant frequency of 1 Hz. The output peak-to-peak voltage was measured using a high-impedance oscilloscope (Tektronix MDO3014, Massachusetts, USA). For each sample, three measurements were recorded per applied force value. The average output voltage for each applied force was subsequently calculated. The thickness of each sample was also measured by Digital Micrometre film 4000DIG, Baxlo (Barcelona, Spain).



Fig. 3. A schematic diagram of a sandwich of PVDF nanofiber layer and 2 aluminum electrodes

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Fig. 4. A schematic of Piezoelectric Characterization Setup (Copyrights© to Center of Smart Materials, Nanotechnology and Photonics (CSMNP))

The  $d_{33}$  test was performed using the wide-range  $d_{33}$  meter (YE2730, APC, USA), as shown in **Error! Reference source** not found., assessing the piezoelectric coefficient  $d_{33}$ . This coefficient reflects the material's capacity to convert mechanical stress or strain into electrical voltage and vice versa. Multilayers of approximately 1 x 1 cm<sup>2</sup> in size, with a total thickness of approximately 300 µm, were prepared from each PVDF nanofiber sample to determine the  $d_{33}$  piezoelectric coefficient. A force of  $0.25 \pm 0.01$  N was applied at a frequency of 110 Hz to each sample. To ensure accuracy, about 20 measurements were taken for each sample, and the average  $d_{33}$  coefficient was calculated based on these measurements.



Fig. 5. The wide-range d<sub>33</sub> meter (YE2730, APC, USA) for conducting piezoelectric coefficient

### 3. Results and Discussion

The performance of the 12 NF-PVDF mats was evaluated through different characterization techniques including morphological characterization by SEM, crystalline characterization by X-ray diffraction (XRD), Fourier transform infrared spectroscopy (FTIR), and piezoelectric response characterization by piezoelectric coefficient d<sub>33</sub> and force-voltage tests.

#### 3.1. Morphological Analysis

The morphological characterization of polyvinylidene fluoride (PVDF) nanofiber mats was conducted using SEM, providing detailed insights into the nanofibers` surface morphology and structural integrity for 12 samples. Key parameters such as fiber diameters, beads formation, and fiber agglomerations were conducted.

#### 3.1.1. Fiber Diameter

The fiber diameter average for each sample was analyzed at different imaging magnifications (10  $\mu$ m, 5  $\mu$ m, and 2  $\mu$ m). Fiber diameter measurements revealed a range of diameters for each sample, with averages and standard deviations presented in **Error! Reference source not found.** Histograms were generated to visualize the range and frequency of diameters, providing insights into fiber production uniformity. The fiber diameter averages varied from 142 ± 4 nm (A10) to 364 ± 9 nm (A6) across all samples as shown in **Error! Reference source not found.** to Error! Reference source not found.. Samples A3, A6, A9, and A12, fabricated with 18 wt.% PVDF concentration, displayed the widest fiber diameter range, from 150 nm to 516 nm. These samples also had the highest average fiber diameters, ranging from 273 nm to 364 nm.

Samples fabricated with a higher feeding rate exhibited finer fiber diameters (e.g. samples A10, A11, and A7) compared to the samples fabricated with a low feeding rate. Samples fabricated with a longer collecting distance exhibited finer fiber diameters (e.g. samples A10, A11, and A5) compared to the samples fabricated with a shorter collecting distance.

Table 4. Results of fiber diameters analysis of the 12 NF-PVDF samples

Samples	Diameter Range(nm)	Diameter Average (nm)
A1	107-378	235 ±7
A2	122-432	259 ±8
A3	150-430	273 ±7
A4	153-426	289 ±7
A5	120-351	229 ±7
A6	161-516	364 ±9





Fig. 6. SEM images with fiber diameter distribution for 12wt.% PVDF samples (a) Sample A1 (b) Sample A4 (c) Sample A7 (d) Sample A10.



Fig. 7. SEM images for fiber diameter distribution for 15wt.%PVDF samples (a) Sample A2 (b) Sample A5 (c) Sample A8 (d) Sample A11.



Fig. 8. SEM images with fiber diameter distribution for 18wt. %PVDF samples (a) Sample: A3 (b) Sample: A6 (c) Sample: A9 (d) Sample: A12.

The study used One-Way ANOVA to analyze the effect of PVDF concentrations on fiber diameters. ANOVA results indicated a significant difference in fiber diameter among the three concentration groups of 12%, 15%, and 18% with a P value < 0.01, as detailed in **Table 5**. The NF-PVDF samples with a concentration of 18wt.% showed a substantially larger mean fiber diameter of 333 nm compared to the other two groups of NF-PVDF samples with 224 nm for 15wt.% and 215 nm for 12wt.%. This indicates a direct correlation between PVDF concentration and fiber diameter, as low polymer concentrations produce finer fibers due to less chain entanglement and viscosity. On the other hand, high viscosity reduces the fiber drawing effect, leading to thicker fibers [19]. Finer nanofibers offer increased surface area, flexibility, and faster response times, which is beneficial in enhancing piezoelectric properties and energy harvesting applications. In contrast, thicker nanofibers offer improved mechanical

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strength which is advantageous for applications requiring high wear and tear resistance, but less piezoelectric performance compared to finer nanofibers [20].

	Fiber Diameter					
	Sum of Squares	df	Mean Square	F	P Value.	
Between Groups	3435694.685	2	1717847.343	281.727	0.000	
Within Groups	7298774.446	1197	6097.556			
Total	10734469.132	1199				

Table 5. ANOVA statistical analysis of PVDF concentration effect and fiber diameter of the NF-PVDF samples

For feeding rates, ANOVA results indicated significant differences in fiber diameter among the two feeding rate groups (3ml/hr. and 7 ml/hr.) with a P value < 0.01, as detailed in **Error! Reference source not found.** The NF-PVDF samples with a feeding rate of 3ml/hr. showed a substantially larger mean fiber diameter with a value of 275 nm compared to NF-PVDF samples of 7ml/hr. with a value of 240 nm. This indicates a direct correlation between PVDF concentration and fiber diameter, as a high feeding rate produces finer fibers due to increased stretching forces that facilitate finer nanofiber formation [21].

Table 6. ANOVA statistical analysis of feeding rate effect and fiber diameter of the NF-PVDF samples

Fiber Diameter						
Sum of Squares df Mean Square F P Val						
Between Groups	387815.700	1	387815.700	44.904	0.000	
Within Groups	10346653.432	1198	8636.606			
Total	10734469.132	1199				

For collecting distance, ANOVA results suggest that the difference in collecting distances of 25 cm and 50 cm does not have a statistically detectable effect on the fiber diameter under the conditions of this experiment with a P value of 0.267, as demonstrated in **Error! Reference source not found.** 

Fiber Diameter					
	Sum of Squares	df	Mean Square	F	P Value.
Between Groups	11059.498	1	11059.498	1.236	0.267
Within Groups	10723409.633	1198	8951.093		
Total	10734469.132	1199			

# 3.1.2. Beads` Presence and Fiber Agglomerations

Based on the SEM in **Error! Reference source not found.** to **Error! Reference source not found.** beads presence and fiber agglomerations were evaluated qualitatively for all the 12 PVDF samples. Samples A4, A5, A6, A7, A10, A11, and A12 show minimal beads presence and fiber agglomerations. Samples A1, A2, A9 show moderate beads presence and fiber agglomerations. In contrast, Sample A3 shows the highest presence of beads and agglomerations. Samples with minimal bead and agglomeration formation exhibit more consistent piezoelectric behavior across the textile. In contrast, samples with high bead and agglomeration formation exhibit inconsistent and misleading piezoelectric responses as they can act as stress concentration points during mechanical deformation, causing apparent piezoelectric signals that may not accurately represent the material's true, uniform response [22].

#### 3.2. Crystalline Structure and Phases Characterization

The crystalline structure and phases characterization of the polyvinylidene fluoride (PVDF) nanofiber mats was analyzed using XRD and Fourier transform infrared spectroscopy (FTIR), providing detailed insights into the crystalline structure and the active groups of ( $\beta \& \alpha$ ) phases for the 12 nanofiber samples.

#### 3.2.1. X-Ray Diffraction

The XRD analysis provided insights into the PVDF nanofibers' crystalline structure and phase composition. As shown in **Error! Reference source not found.** and **Error! Reference source not found.** the 2 Theta ( $\theta$ ) angles for the samples ranged consistently from 20.33° to 20.92° which represents the diffraction peak at planes of 110 and 200, indicating a predominance of the  $\beta$ -phase in the PVDF nanofibers (commonly at 20.6°), which is known for its piezoelectric properties [23]. The slight variations in the 2 $\theta$  angles among the samples suggest minor differences in crystallite orientation and structural arrangement.

Samples	2θ° (For Beta)	<b>FWHM°</b>	Crystallite Size (A°)
A1	20.92	0.866	97.80
A2	20.68	0.835	96.71
A3	20.55	0.875	92.27
A4	20.78	0.866	93.27
A5	20.57	0.892	90.52
A6	20.61	0.808	99.93
A7	20.50	0.964	83.75
A8	20.36	0.977	82.61
A9	20.39	0.902	89.49
A10	20.55	0.933	86.54
A11	20.33	0.901	89.58
A12	20.45	0.964	83.74

Table 8. The crystallite size of the 12 PVDF nanofiber samples



Fig. 9. XRD pattern of the 12 PVDF nanofiber samples.

The Scherrer equation was used to estimate the crystallite size for each sample, using the following formula:

### $L = K\lambda/(\beta \cos\theta).$

Where "L" represents the crystallite size in nanometres (nm), "K" is the Scherrer constant, with an often-used value of 0.9, " $\lambda$ " represents the wavelength of the X-ray radiation used in the XRD experiment, in nanometres (nm), " $\beta$ " represents the full width at half maximum (FWHM) of the diffraction peak in radians ( This value is obtained by measuring the width of the peak at half its maximum intensity on the XRD pattern), and " $\theta$ " represents the Bragg angle of the diffraction peak in degrees.

The crystallite size of the nanofiber samples ranged between 99.9 A° and 82.6 A°, according to the crystallite size calculations, as shown in **Error! Reference source not found.** and **Error! Reference source not found.** . Samples A6, A1, A2, A4, A5, A11, A9, and A10 had larger crystallite sizes, indicating a highly ordered crystalline structure. This is beneficial for mechanical stability and piezoelectric performance. Smaller crystallite sizes, like those in A8, A12, and A7, indicate a more amorphous structure, increasing flexibility but potentially compromising mechanical strength [12, 17].

Samples with 18 wt.% PVDF showed the highest mean crystallite size of 91.36 A°, suggesting increased crystallinity with higher concentrations. Samples with 3mL/hr. feeding rate showed the highest mean crystallite size of 95 A°, indicating slower jet formation and elongation. Crystallite sizes were slightly larger at 50 cm collecting distance, with the largest mean crystallite size of 90.6 A°, possibly due to the extended time for solvent evaporation and solidification, which may hinder crystallization [24].



Fig. 10. The crystallite size of the 12 NF-PVDF samples

#### 3.2.2. Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR analysis of the PVDF nanofiber mats in ATR mode focused on the relative absorption intensities at the characteristic wavenumbers of 840 cm<sup>-1</sup> and 761 cm<sup>-1</sup>, which refer to specific vibrational modes of the PVDF, as shown in **Error! Reference source not found.** The peak at 840 cm<sup>-1</sup> is associated with the  $\beta$ -phase of PVDF. This phase is highly desirable for piezoelectric applications because it exhibits strong piezoelectric properties. The peak at 761 cm<sup>-1</sup> corresponds to the  $\alpha$ -phase of PVDF. This phase is non-polar and does not contribute to piezoelectricity.

The  $\beta$ -phase content was calculated for each sample from the absorption intensities, as shown in **Error! Reference source not** found., according to the following Beer-Lambert law-based equation for  $\beta$ -phase content:

$$\mathbf{F}(\boldsymbol{\beta}) \mathbf{\mathbf{6}} = \frac{A\boldsymbol{\beta}}{(1.26A\alpha + A\boldsymbol{\beta})} \times 100$$

Where " $\mathbf{F}(\boldsymbol{\beta})$ " is the  $\beta$ -phase content (%), " $\mathbf{A}\boldsymbol{\beta}$ " is the absorbance at the wavenumber corresponding to the beta phase (commonly around 840 cm<sup>-1</sup>), " $\mathbf{A}\boldsymbol{a}$ " is the absorbance at the wavenumber corresponding to the alpha phase (commonly around 764 cm<sup>-1</sup>), and "1.26" is a normalization constant used to adjust the contribution of the alpha-phase absorbance relative to the beta-phase absorbance.

A=2-log10(T%)

The percentage of light transmitted (T%) through the sample was converted into absorbance (A) by the following equation:



Fig. 11. FT-IR analysis for the 12 samples of NF-PVDF mats

According to **Error! Reference source not found.** and **Error! Reference source not found.**, the  $\beta$ -phase content varied significantly among the samples, with values ranging from 67.29% (A11) to 88.30% (A3). The higher  $\beta$ -phase content in samples from A1to A10 suggests these samples are more likely to exhibit superior piezoelectric properties as the  $\beta$ -phase is primarily responsible for the piezoelectric properties of PVDF. Lower  $\beta$ -phase content in samples A11 and A12 indicates a lower piezoelectric response, aligning with the lower crystallite sizes observed in these samples. Samples with 18wt.% PVDF showed the highest mean  $\beta$ -phase content of 82%, indicating that increasing polymer solution viscosity enhances beta-phase presence, leading to thicker NF-PVDF fabrication and crystallite size. Samples with a 3mL/hr. feeding rate (85%) and 25cm collecting distance (82%) also showed higher mean  $\beta$ -phase content [25].

Samples	Transmittance% for α at (761 <sup>-1</sup> )	<b>Transmittance% for <math>\beta</math> at (840<sup>-1</sup>)</b>	β-phase content(%).
A1	96.376	74.859	86.16
A2	95.600	71.418	85.58
A3	95.936	67.387	88.30
A4	93.502	74.481	77.68

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95.266	70.485	85.13
95.255	71.903	84.34
88.387	62.016	75.44
88.679	65.427	73.70
91.995	62.259	81.84
87.266	62.151	73.48
87.249	70.218	67.29
85.832	63.044	70.56
	95.266 95.255 88.387 88.679 91.995 87.266 87.249 85.832	95.266         70.485           95.255         71.903           88.387         62.016           88.679         65.427           91.995         62.259           87.266         62.151           87.249         70.218           85.832         63.044



Fig. 12 The  $\beta$ -phase content % of the 12 NF-PVDF samples

# 3.3. Piezoelectric Response Characterization

The piezoelectric response of the polyvinylidene fluoride (PVDF) nanofiber mats was analyzed using the  $d_{33}$  test and voltage-force response providing detailed insights into the piezoelectric coefficient  $d_{33}$ , the output voltage at applied cyclic forces of 0.1,0.2,0.3,0.4 and 0.5 N with a constant frequency of 1Hz for the 12 nanofiber samples.

# 3.3.1. Piezoelectric Coefficient d33 Test

The  $d_{33}$  piezoelectric coefficient was conducted to evaluate the efficiency of the samples in converting mechanical energy into electrical energy using the wide range  $d_{33}$  meter, YE2730, APC, USA by applying a force of ~0.25 ±0.01 N with a frequency of 110 Hz on each one of the PVDF nanofiber samples. About 20 measurements were recorded to calculate the average of the  $d_{33}$  coefficient for each sample according to the following equation:

#### $d_{33} = Q / F$

Where " $d_{33}$ " is the piezoelectric charge coefficient (measured in C/N or pC/N), "Q" is the electric charge generated (measured in Coulombs (C) or Picocoulombs (pC)), and "F" is the applied force (measured in Newton (N)).

The d<sub>33</sub> values of the fabricated samples ranged approximately from 24.04 pC/N (A4) to 86.36 pC/N (A7), demonstrating a significant variation in piezoelectric response across the sample set, as shown in **Error! Reference source not found.** Samples A7, A5, A10, A9, A6, A3, A8, A2, A1, and A11 showed higher d<sub>33</sub> values, while samples A12 and A4 showed lower values which are still favourable for piezoelectric coefficient [26]. These results correlate with PVDF finer diameters, higher crystallite size, and beta phase content [25].

Table 10. The piezoelectric response for the 12 NF-PVDF samples according to d <sub>33</sub> and gen	rerated peak-peak output voltage under applied
cyclic forces	

0 1		0.4.43	7 14 4			(\$71)
Samples	Average of d33 Piezoelectric Coefficient(pC/N)	Output	voltage at 1	Applied Cy	velic Force	s (V/mm)
		0.1N	0.2N	0.3N	0.4N	0.5N
A1	~34 ±0.06	~88	~92	~105	~108	~118
A2	~35.6 ±0.02	~66	~73	~80	~84	~93
A3	~46.2 ±0.02	~33	~45	~70	~74	~78
A4	~24.1±0.02	~34	~43	~53	~60	~71
A5	~68.5 ±0.04	~73	~107	~128	~134	~140
A6	~48.8 ±0.02	~60	~68	~85	~90	~98
A7	~86.4± 0.06	~57	~69	~83	~97	~120
A8	~39.2 ±0.05	~25	~48	~52	~59	~65
A9	~49.2 ±0.05	~38	~51	~59	~63	~75
A10	~59.6 ±0.03	~48	~63	~69	~90	~99
A11	~28.5 ±0.03	~53	~67	~76	~90	~94
A12	~26.3 ±0.02	~21	~26	~45	~64	~73

For PVDF concentrations, ANOVA results suggested significant differences in the piezoelectric coefficient d<sub>33</sub> among the three concentration groups of 12%, 15%, and 18% with a P value of 0.005, as detailed in **Error! Reference source not found**.

The NF-PVDF samples with a concentration of 12wt.% showed a substantially larger mean piezoelectric coefficient  $d_{33}$  of 50.77 pC/N compared to the other two groups of NF-PVDF samples with values of 42.9 pC/N for 15wt.% and 42.8 pC/N for 12wt.%. This indicates a direct correlation between PVDF concentration and the piezoelectric coefficient  $d_{33}$ , as low polymer concentrations produce higher values of the  $d_{33}$ . This is due to the finer nanofiber diameters produced by lower PVDF concentrations which enhance the piezoelectric performance [20].

Piezoelectric Coefficient d <sub>33</sub>								
Sum of Squares df Mean Square F P Value.								
Between Groups	3337.076	2	1668.53	5.400	0.005			
Within Groups	73229.60	237	308.986					
Total	76566.68	239						

Table 11. ANOVA statistical analysis of PVDF concentration effect and the piezoelectric coefficient d<sub>33</sub> of the NF-PVDF samples

For feeding rates, ANOVA results indicated significant differences in the piezoelectric coefficient d33 among the two feeding rate groups of 3ml/hr. and 7ml/hr. with a P value of 0.010, as detailed in **Error! Reference source not found.**. The NF-PVDF samples with a feeding rate of 7 ml/hr. showed a substantially larger mean piezoelectric coefficient d33 of 48.5 pC/N compared to the other 3ml/hr. feeding rate group of NF-PVDF samples of 42.5 pC/N. This also indicates a direct correlation between the feeding rate and the piezoelectric coefficient d33, as a high feeding rate produces higher values of the d33. This refers to the finer nanofiber diameters produced by a higher feeding rate which enhances the piezoelectric performance [17, 19].

Table 12. ANOVA statistical analysis of feeding rates effect and the piezoelectric coefficient d<sub>33</sub> of the NF-PVDF samples

Piezoelectric Coefficient d <sub>33</sub>							
Sum of Squares df Mean Square F P Value.							
Between Groups	2093.913	1	2093.913	6.692	0.010		
Within Groups         74472.770         238         312.911							
Total	76566.683	239					

For collecting distance, ANOVA results suggested significant differences in the piezoelectric coefficient  $d_{33}$  among the two collecting distance groups of 25 cm and 50 cm with a P value of 0.013, as detailed in **Error! Reference source not found.** The NF-PVDF samples with a collecting distance of 25 cm showed a substantially larger mean piezoelectric coefficient  $d_{33}$  of 48.4 pC/N compared to the other 50 cm collecting distance group of NF-PVDF samples with a value of 42.7 pC/N. This also indicates a direct correlation between collecting distance and the piezoelectric coefficient  $d_{33}$ , as a short collecting distance produces higher values of the  $d_{33}$ . This refers to the high  $\beta$  – phase content which is increased by a short collecting distance which enhances the piezoelectric performance [25].

Table 13. ANOVA statistical analysis of collecting distance effect and the piezoelectric coefficient d33 of the NF-PVDF samples

Piezoelectric Coefficient d <sub>33</sub>							
	Sum of Squares	df	Mean Square	F	P Value.		
Between Groups	1953.392	1	1953.392	6.231	0.013		
Within Groups	74613.291	238	313.501				
Total	76566.683	239					

#### 3.3.2. Force-Voltage Response

The piezoelectric response was measured by the output voltage under applied mechanical cyclic forces of 0.1, 0.2, 0.3, 0.4, and 0.5 N for the 12 NF-PVDF samples. A pressing head with a diameter of 1 cm was used, and the measurements were conducted at a frequency of 1 Hz. According to **Error! Reference source not found.**, and **Error! Reference source not** 

**found.**, there is also a direct correlation between the applied cyclic force and the output voltage. For example, Sample A5 exhibited a peak-to-peak voltage of  $\sim$ 73V/mm under an applied cyclic force of 0.1 N and reached  $\sim$ 140 V/mm at 0.5N.



Fig. 13. A graph of the output voltage of the 12 NF-PVDF samples under cyclic forces from 0.1 to 0.5 N (a) 12wt.%PVDF samples (b) 15wt.%PVDF samples (c) 18wt.%PVDF samples

According to Fig. 14, sample A5 exhibited the highest peak-to-peak voltage of ~140 V/mm generated under an applied cyclic force of 0.5N at a frequency of 1Hz. While sample A8 exhibited the lowest peak-to-peak voltage of ~65V/mm under the same cyclic force of 0.5N. Samples with low PVDF concentration, low feeding rate, and longer collecting distance had higher output voltage. Properties like finer fiber diameters, uniformity, beads-free, minimal fiber agglomerations, and piezoelectric coefficient d33 increased output voltage. Sample A3, despite having the highest  $\beta$ -phase content and moderate measurements, exhibited a lower output voltage of 78V/mm due to beads, fiber agglomerations, and lower fiber uniformity. This highlights the need to adjust the fabrication process of NF-PVDF mats.



Fig. 14. The output voltage of the NF-PVDF samples under an applied cyclic force of 0.5N

For PVDF concentrations, ANOVA results suggested significant differences in the generated output voltage among the three concentration groups of 12%, 15%, and 18% with a P value of 0.008, as detailed in

**Table 14.** The NF-PVDF samples with a concentration of 12wt.% showed a substantially larger mean output voltage of approximately 106 V/mm compared to the other two groups of NF-PVDF samples with values of roughly 98 V/mm for 15wt.% and 80 V/mm for 18wt.%. This indicates a correlation between PVDF concentration and the generated output voltage under applied cyclic force, as low polymer concentrations produce higher values of the output voltage. This aligns with finer fiber diameter and high values of d<sub>33</sub> produced by lower PVDF concentrations which enhance the piezoelectric performance.

Table 14. ANOVA statistical analysis of PVDF concentration effect and the output voltage of the NF-PVDF samples at 0.5N

Output Voltage at 0.5 N					
	Sum of Squares	df	Mean Square	F	P Value.

Between Groups	4315.732	2	2157.866	5.633	0.008
Within Groups	12641.486	33	383.075		
Total	16957.218	35			

For feeding rate, ANOVA results showed that the difference in feeding rates of 3mL/hr. and 7mL/hr. does not have a statistically significant difference in the output voltage under the applied mechanical cyclic force of 0.5N, under the conditions of this experiment with a P value of 0.059, as demonstrated in

Table 15.

Table 15. ANOVA statistical analysis of the feeding rate effect and the output voltage of the NF-PVDF samples at 0.5N

Output.Voltage.at0.5N							
	Sum of Squares	df	Mean Square	F	P Value		
Between Groups	1714.794	1	1714.794	3.825	0.059		
Within Groups	15242.424	34	448.307				
Total	16957.218	35					

For collecting distance, ANOVA results showed that the difference in collecting distances of 25 cm and 50cm does not have a statistically significant difference in the output voltage under the applied mechanical cyclic force of 0.5N, under the conditions of this experiment with a P value of 0.400, as demonstrated in T

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able 16. ANOVA statistical analysis of the collecting distance effect and the output voltage of the NF-PVDF samples at 0.5N

Output.Voltage.at0.5N							
Sum of Squares         df         Mean Square         F         P Val							
Between Groups	354.746	1	354.746	0.726	0.400		
Within Groups	16602.471	34	488.308				
Total	16957.218	35					

Based on the previous results, the study highlights the statistically significant effects of PVDF concentration, feeding rate, and collection distance on the morphological, crystalline, and piezoelectric properties of NF-PVDF fibers. Morphologically, lower PVDF concentration (12 wt.%), higher feeding rate (7 mL/h), and longer collection distance (50 cm) resulted in finer fiber diameters, which enhance piezoelectric properties. Crystalline analysis revealed that samples with higher PVDF concentration (18 wt.%), lower feeding rates, and shorter collection distances exhibited larger crystallite sizes and higher  $\beta$ -phase content. However, even samples with finer fiber diameters demonstrated improved  $\beta$ -phase content, suggesting that finer diameters play a dominant role in enhancing piezoelectric performance. Piezoelectric response testing confirmed that low PVDF concentration, high feeding rates, and short collection distances lead to higher d<sub>33</sub> values, with finer fiber diameters and uniform fiber distribution contributing to better performance.

Overall, the optimal parameters for achieving high piezoelectric performance in NF-PVDF samples are as follows:

- **PVDF Concentration**: Lower concentrations (12-15%) consistently outperformed higher concentrations (18%). This is supported by the top-performing samples A7 and A10, which used 12% concentration, and A5, which used 15% concentration. The results align with previous observations that finer fiber diameters, typically produced by lower polymer concentrations, correlate with better piezoelectric performance.
- Feeding Rate: Both feeding rates of 3 mL/hr. and 7 mL/hr. yielded excellent results. However, a higher feeding rate (7 mL/hr.) was slightly more favorable when combined with optimal concentration and collection distance. This

suggests that higher feeding rates can enhance productivity without sacrificing performance, provided other parameters are optimized.

• **Collecting Distance**: Both 25 cm and 50 cm collecting distances produced top-performing samples. The top performer (A7) used a 25 cm distance, while the second and third performers (A5 and A10) used 50 cm. This indicates that the optimal collecting distance may vary depending on its interaction with other parameters, particularly the feeding rate.

These findings highlight the critical role that the interaction between PVDF concentration, feeding rate, and collecting distance plays in optimizing piezoelectric performance. To optimize NF-PVDF mats for energy harvesting applications, such as flexible self-charging wearable sensors, it is essential to balance factors like fiber uniformity,  $\beta$ -phase content, crystallinity, and mechanical stability. Achieving maximum energy conversion efficiency while ensuring durability can be accomplished by fine-tuning the fabrication parameters accordingly.

### 4. Conclusion

This study successfully fabricated polyvinylidene fluoride (PVDF) nanofiber (NF-PVDF) mats using the solution blow spinning (SBS) technique, systematically investigating the influence of key fabrication parameters, namely PVDF concentrations of 12 wt.%, 15 wt.%, and 18 wt.%, feeding rates of 3 mL/hr. and 7 mL/hr., and collecting distances of 25 cm and 50 cm, on the morphology, crystalline, and piezoelectric response properties of the produced mats. According to the SEM, the nanofiber diameters ranged from 142 nm to 364nm with minimal beads and agglomerations presence. Based on XRD and FTIR tests for crystalline characterization, the  $2\theta^{\circ}$  of  $\beta$  – phase ranged from 20.33° to 20.92° indicating a domination of  $\beta$  – phase in the NF – PVDF mats, the crystallite size was calculated and ranged from 82.61 Å to 99.9 Å, and beta content ranged from 67.29% to 88.3%. For the piezoelectric characterization, the piezoelectric coefficient d<sub>33</sub> ranged from 24.1 pC/N to 86.4 pC/N approximately. The generated output voltage was measured at different applied cyclic forces of (0.1,0.2, 0.3, 0.4, and 0.5 N) and ranged from 65 to 140 V/mm approximately per unit thickness. The results also demonstrated that, finer nanofibers with reduced bead formation and agglomerations, enhanced crystallinity, and increased beta phase correlate with piezoelectric coefficient d<sub>33</sub> and the output voltage at applied cyclic forces, achieving a superior piezoelectric performance. The optimal parameters for high piezoelectric performance include lower concentrations of PVDF (12-15%), feeding rate (3 mL/hr.), and collecting distance (25 cm and 50 cm). Lower concentrations yield better results, while higher feeding rates increase productivity without compromising performance. The optimal distance depends on its interaction with other parameters, particularly the feeding rate. This highlights the importance of fine-tuning SBS parameters for optimizing PVDF nanofiber mats for energy harvesting applications such as flexible self-charging wearable sensors, offering a promising pathway for developing highperformance piezoelectric materials.

### 5. Declaration of Conflicts of Interest

The Authors declare that there is no conflict of interest.

# 6. Funding sources

This research received no specific grant from any funding agency in the public, commercial, or not-for-profit sectors.

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