

## PIEZOELECTRIC TWISTED YARN AS AN ENGINEERING PLATFORM TO SCALE-UP STRETCHABLE AND BREATHABLE ENERGY HARVESTERS

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### ABSTRACT

A highly stretchable piezoelectric nanofibrous yarn of PVDF/ZnO composite was electrospun through a one-step electrospinning method to facilitate the fabrication process of wearable nanogenerator device in a desired weave patterns and mechanical properties. Electrospun yarns of different counts, twists per meter, and fibers fineness were fabricated on a modified electrospinning setup and then their piezoelectric and mechanical properties were evaluated. Results showed that an increase in twists number and take up speed increase the output voltage of the fabricated strip of yarns. In spite of this, by increasing the amount of ZnO nanoparticles from 5% to 15% in the yarn nanofibers, the output voltage increased significantly which is contributed to the nature of strong piezoelectric properties of ZnO piezo-ceramic materials. It is believed that the whole piezoelectric device prepared by piezoelectric yarns is attractive for a variety of wearable applications, such as wearable generator to collect energy from human movement as a building block of energy- harvesting textiles, and in self-powered biomedical applications. The results of this work can be utilized as a platform to scale-up towards the fabrication of flexible and stretchable energy harvesting devices.

**KEYWORDS:** piezoelectric nanofibrous, fabrication, ZnO piezo-ceramic materials

### 1. INTRODUCTION

Energy harvesters can convert the ambient energy ubiquitously presents in the environment into electricity as a continuous, sustainable and portable power supply. Among the recently developed energy harvester, piezoelectric nanogenerators has gained lots of attention in recent years [1, 2]. Piezoelectric materials are substances that can convert mechanical stress into the electrical energy. This property can be used to generate, measure, and transmit energy. One type of piezoelectric compounds, are piezopolymers. In recent years, various polymers have been identified with piezoelectric properties. Among them Polyvinylidene fluoride (PVDF) and its copolymers are mostly studied organic piezoelectric materials with high piezoelectricity compared to other polymers. Unlike the ceramic materials such as PZT considered due to their good piezoelectric properties, polymeric compositions are non-toxic and high flexible. Therefore, these materials can be employed to produce many smart textile products [3, 4]. PVDF with molecular formula of -(C<sub>2</sub>H<sub>2</sub>F<sub>2</sub>)<sub>n</sub>-, melting temperature of 177 °C and density of 1.78 g/cm<sup>3</sup>, is one of the piezopolymers that is considered in the past two decades due to its good piezoelectric and pyroelectric behaviors [5-7].

Textile-based harvester with outstanding performance in terms of their flexibility and stretchability are highly imperative for wearable device development. Textiles are one of the most suitable substrate materials, which can effectively accommodate to mechanical deformations induced by body movement [1]. Today, the production of piezoelectric materials, due to a variety of applications, is in much interest. For example, researchers are trying to produce flexible and wearable sensors with piezopolymers which can control the vital signs of human in certain circumstances [5, 8, 9]. Conventional electrospinning can be used to synthesize and pattern an array of PVDF fibers. Generally, fibers produced with electrospinning method are collected in a

nonwoven layer shape without orientation which limits the application of these fibers. Up to now, many researchers have tried to fabricate the nanofiber based energy harvesting devices in different forms and structures. For instance, Ji et al [10] investigated wearable core-shell piezoelectric nanofiber yarns based on BTs and PVDF-TrFE with external electrodes. Maity et al [11] designed and constructed an all organic piezoelectric nanogenerator (OPNG) based on multilayer structure of PVDF NFs mats followed by PEDOT coating. A three-layered piezoelectric nanogenerator in which PVDF nanofibrous mat was sandwiched between two PVDF-rGO membranes was another structure produced using the electrospinning [12]. The piezoelectric membrane exhibited an energy output as high as  $18.1\mu\text{W}/\text{cm}^2$  under a compressive force of 35 N at 10 Hz. It is important to know that the energy harvesters based on the nanofiber structures are not versatile and convenient to be incorporated into textiles [13]. Traditionally, nanofibers are collected as nonwoven mats, although in many applications, nanofiber assembly in the form of a yarn could be in more interest over mats, since yarns can be incorporated into a textile conveniently by weaving or knitting methods [1, 8, 9]. If the nanofibers produced in this way are collected in the form of yarn, not only it can provide a good platform to develop applications of electrospun nanofibers, but also can create new applications for fibrous structures. One of these applications is producing the piezoelectric yarns that have so many applications in flexible electronic devices. The polymeric yarn and coils were fabricated by the twisting process to further increase the strength of the fiber sample [14, 15]. Different nanoyarn fabrication methods have been reported using electrospinning technologies. Enlong et al. [15] developed a plied nanofibrous piezoelectric yarns using a non-continuous method. Xue et al. [16] developed twisted polymer fibrous electrospun yarns using a typical electrospinning device with a rotating spinneret. Recently Gao et al. [17] developed a method of nanostructure yarn fabrication based on the touch-spinning technique which filament nanofibers spinning was provided. At the same time they succeed to manipulate yarn deposition on a conductive core, with different yarns structure including thickness, nanofiber diameter, and their alignment. However, all aforementioned researches were failed to provide a fabrication method in which the yarn count and twist level can be controlled in a large scale. Fabrication of a wearable piezoelectric device of excellent flexibility, lightweight with comfort related properties needs to have yarns with engineered properties as well as strong piezoelectric properties. For this purpose, the production of an engineered structure in a form of textile yarn can be considered as a big step in producing the self-charging wearable electronic devices [9]. Therefore, the search for nanofibrous yarns that possess good conversion efficiency, as well as great mechanical and environmental stability, easy to scale-up might push these researches to meet the requirement of a practical application.

Herein, we introduce a facile method to construct piezoelectric device based on electrospun yarn by involving one-step continuous electrospinning method. This piezoelectric device demonstrates high flexibility in its nature and can be utilized in production of knitted or woven textiles. The electrode membrane prepared by mixed electrospinning consists of interpenetrating networks of PVDF nanofiber in two electrospun nozzles placed in front of each other in a modified electrospinning set up. The lightness, flexibility, breathability and mechanical property of piezoelectric device demonstrate that it has the capability for either wearable application itself, or in the fabric structure with a desired weave pattern and structure. This approach has high scale-up capability and can improve the content of the piezoelectric active  $\beta$ -phase with no needs of additional poling steps. Moreover, the approach of continuous electrospinning for making piezoelectric yarn is a promising technology for massive construction of piezoelectric device with reasonable mechanical properties.

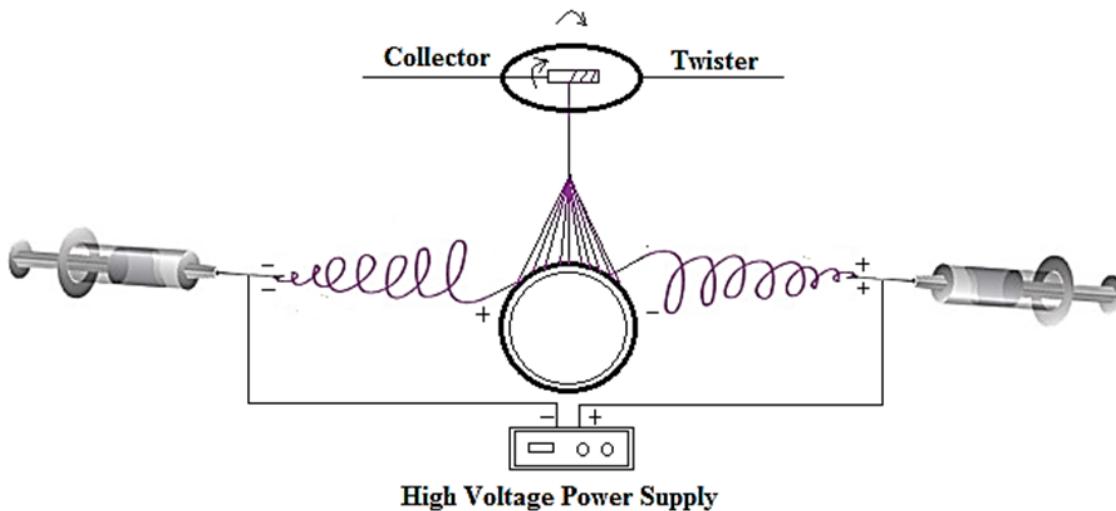
## 2 EXPERIMENTAL

### 2.1 MATERIALS

Polyvinylidene fluoride polymers granules with an average molecular weight of 107,000 g/m were purchased from Sigma-Aldrich. The desirable PVDF solution, leads to obtaining a uniform bead-free nanofiber sample. Homogenized solutions were obtained by solving PVDF pellets in Dimethylformamide (DMF) and acetone solvent in 6/4 ratio. Nano Zinc Oxide (ZnO) particles with an average particle size of 10 to 30 nm purchased from US Research Nanomaterial Inc.

### 2.2 FABRICATION AND CHARACTERIZATION OF NANOFIBROUS YARN

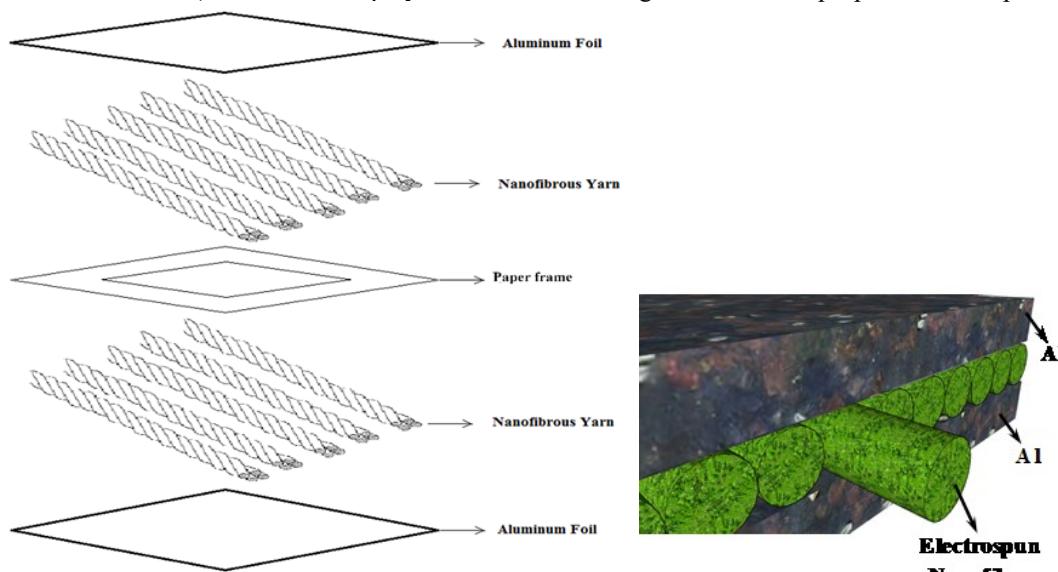
In electrospinning, by changing the collecting mechanism, nanofibers in a continuous form of nanofibrous yarn can be collected. In this study, conjugation electrospinning method was used. Two nozzles placed in front of each other and therefore, two jets with the opposite charges were formed. Nanofibers with the opposite charges are deposited on a neutral surface. Continuous bundle of nanofibers collected from this surface and then were stretched and twisted on a spool as shown schematically in Figure 1.



**Figure 1.** Schematic of employed conjugation electrospinning set up [18].

The polymer concentrations of 24 wt.%, voltage of 18 kV, feed rate of 1 mL/h and 26 cm the distance between two nozzles from each other, are the electrospinning parameters to produce piezoelectric yarns. In order to investigate the effect of ZnO nanoparticles, piezoelectric fibers with 5, 10, and 15 wt. % of ZnO to weight of PVDF were produced. Samples characteristics are shown in Table 1. In order to measure piezoelectricity property, samples were wrapped with two aluminum electrodes. Samples were laid in a parallel shape in contact with each other on the 2×2 cm aluminum electrodes. To avoid connection of two aluminum plates a paper frame was utilized (Figure 2).

To characterize the morphology and fiber diameter of samples, a scanning electron microscopy (SEM, model: XL30, PHILIPS Co.) was employed. All samples were gold coated (Bal-Tec SCD50 sputter coater) and their images were taken at the acceleration voltage of 25 kV. The fiber diameter was measured using image processing software (ImageJ, National Institutes of Health, USA). X-ray (EQUINOX 3000 model, INEL France Co.) device with characteristic wavelength (Cu  $\text{K}\alpha$ : 0/154) were used to study the crystalline structure of samples. The melting temperature ( $T_m$ ), and melting enthalpy ( $\Delta H_m$ ) of PVDF electrosprun nanofibers were measured with differential scanning calorimeter (DSC) (model: DSC 2010, TA Instruments.co) from room temperature to 200 °C with a heating rate of 5 °C/min. To analyze polymer crystalline structure and crystalline phases Fourier transform infrared spectrometer (FTIR) tests were performed by Spectrometer (model: NEXUS 670, Nicolet Co.) over a range of 400–4000 cm<sup>-1</sup>. Instron (TM-SM Model, Britain) device was employed in order to investigate mechanical properties of samples.

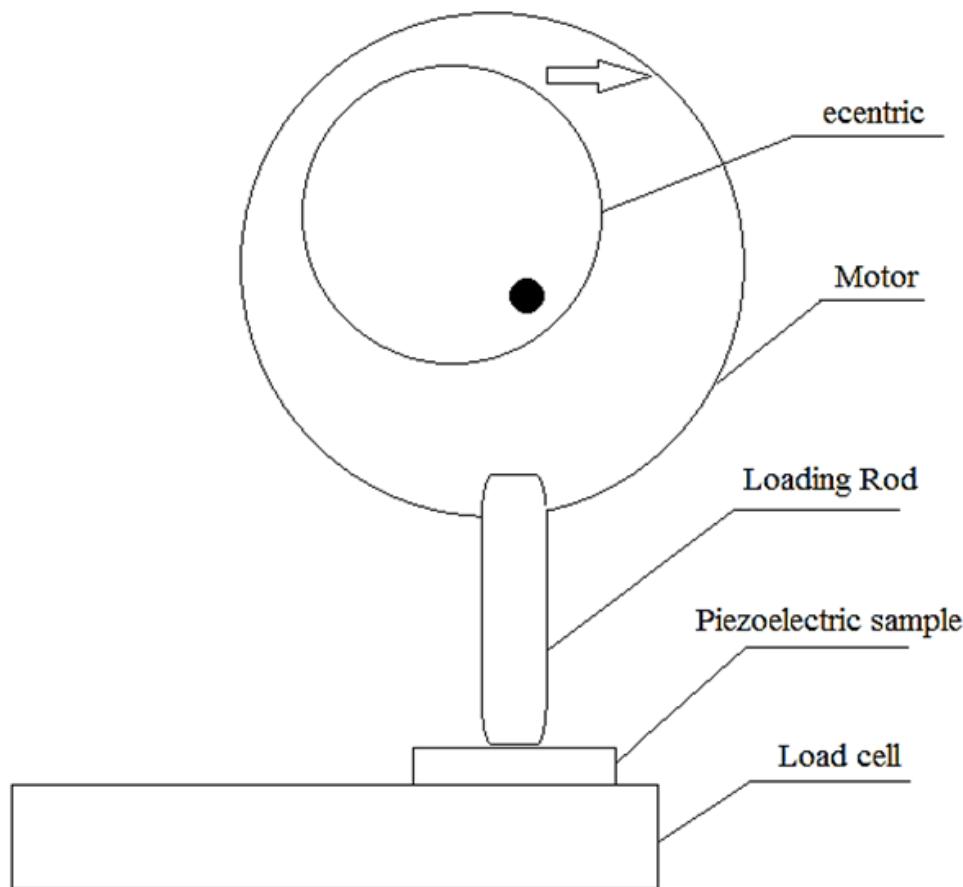


**Figure 2.** Sample preparation method for piezoelectricity measurements and the layers of each samples

**Table 1.** Samples characteristics

TPI	Take up roller speed (m/min)	ZnO wt.%	Samples
1800	0.04	-	1
1800	0.06	-	2
2600	0.04	-	3
1800	0.04	5%	4
1800	0.06	5%	5
2600	0.04	5%	6
1800	0.04	10%	7
1800	0.06	10%	8
2600	0.04	10%	9
1800	0.04	15%	10
1800	0.06	15%	11
2600	0.04	15%	12

The piezoelectric property of samples was measured by a device that is shown schematically in Figure 3. The device for applying force was an eccentric cylinder connected to the main shaft derived by a servomotor. Beating frequency has a direct effect to the amount of applied force. On the other hand, by changing eccentric radius without changing the motor speed, applied force to the samples can be changed. The intensity of the applied force was measured with a load cell which is in contact with sample. The generated signals by load cell and samples were magnified and their noises were filtered in which they could be characterized by an oscilloscope (see Figure 4).

**Figure 3.** Schematic of mechanical load mechanism and force measurement.

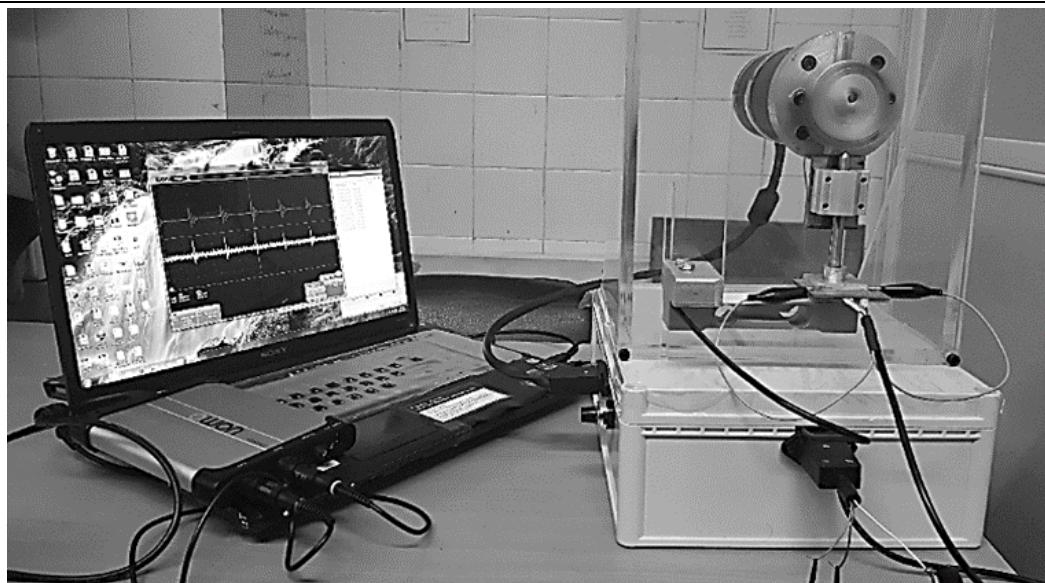
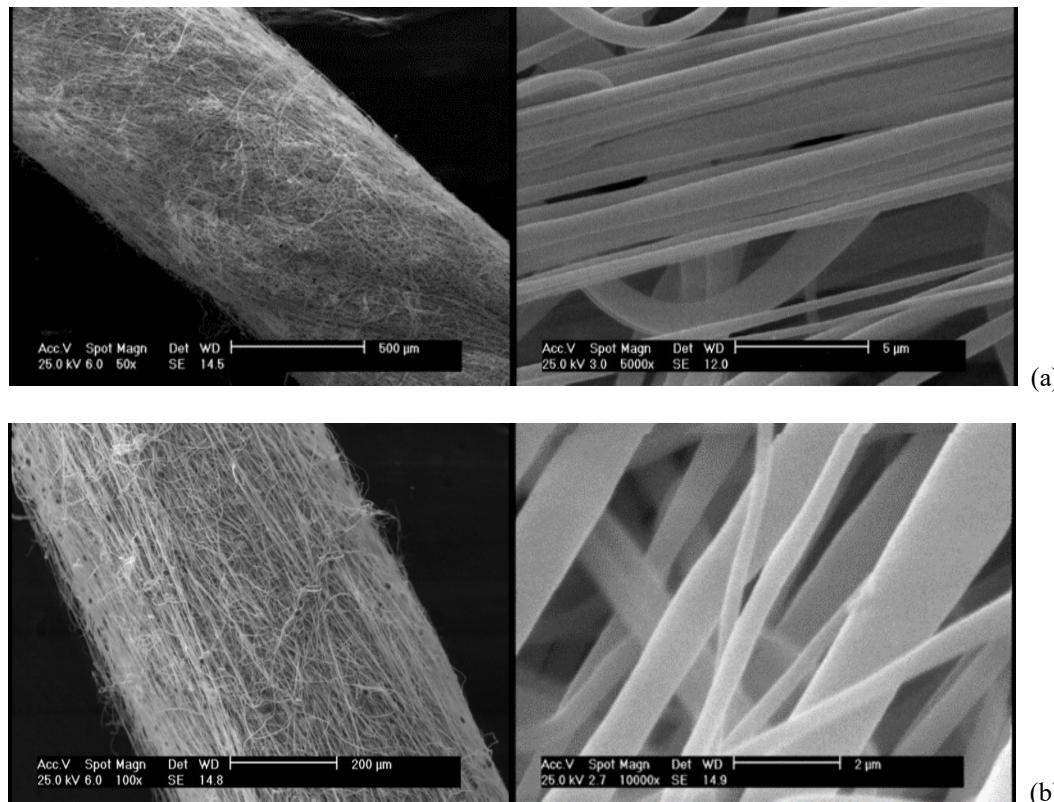


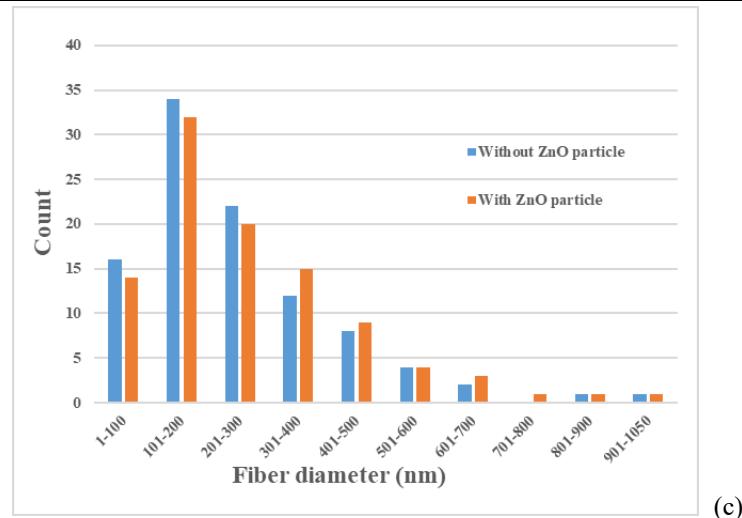
Figure 4. Piezoelectric property measurement device [26]

### 3 RESULT AND DISCUSSION

#### 3.1 SEM AND MORPHOLOGY ANALYSES

The SEM images show nanofibers and fabricated yarn structure. ImageJ software was utilized to estimate the average diameter of nanofibers. The fibers diameter average without ZnO nanoparticle was 245.5 nm with standard deviation of 166.4 but composite fibers containing ZnO nanoparticle had 262.0 nm average diameter with standard deviation of 175.8. Figure 5 shows histogram distribution of fiber diameter and SEM image of the nanofibrous yarn.





(c)

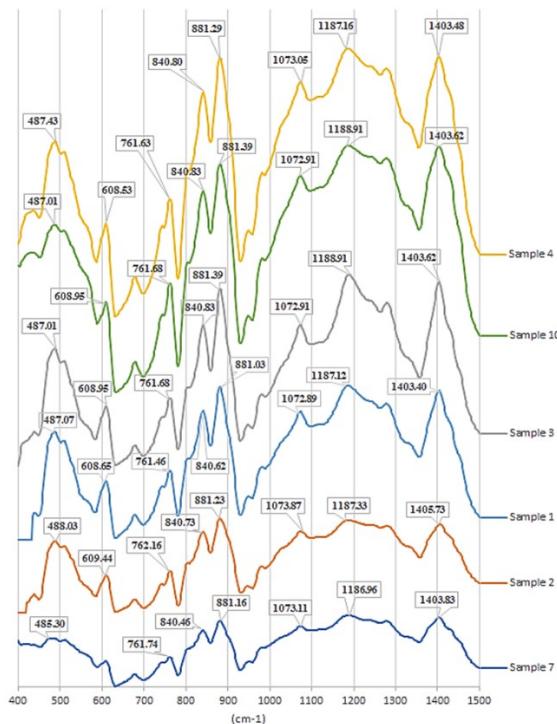
**Figure 5 a.** Nanofibrous yarn without ZnO nanoparticle. **b.** Nanofibrous yarn with 15 wt.% ZnO nanoparticle. **c.** Histogram distribution of nanofiber diameter.

### 3.2 FTIR ANALYSIS

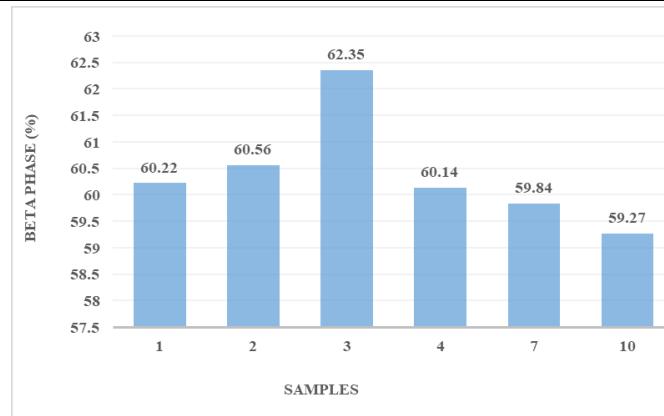
To determine the percentage of beta crystalline phase in each sample, the absorption peaks of alpha and beta phases were evaluated at the wavelengths of 762 cm<sup>-1</sup> and 841 cm<sup>-1</sup>, respectively. The percentage of beta crystalline phase is calculated from Equation (1).

$$f(\beta) = \frac{X_\beta}{X_\beta + X_\alpha} \times 100 = \frac{A_\beta}{1.26A_\alpha + A_\beta} \times 100 \quad (1)$$

Where A<sub>α</sub> and A<sub>β</sub> are absorption at 762 cm<sup>-1</sup> and 841 cm<sup>-1</sup> and X<sub>α</sub> and X<sub>β</sub> are the crystalline degree for the alpha and beta phases, respectively [19-23]. Figure 6 presents the FTIR diagram of samples.



**Figure 6.** FTIR diagram of samples.



**Figure 7.** Beta phase percentage of nanofibrous samples.

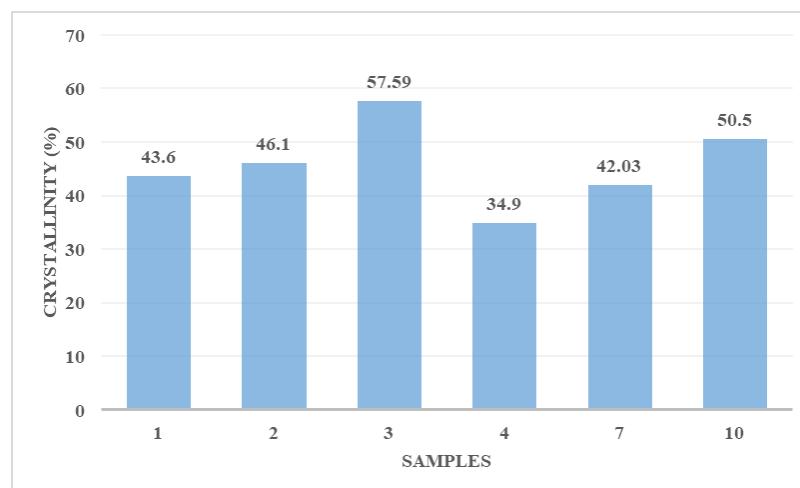
In general, ZnO reduces the percentage of beta phase crystalline. The reason can be attributed due to the presence of ZnO semiconductor nanoparticles. These particles lead to the dielectric coefficient reduction which can disrupt the electrospinning process. The presence of nano-particles in the solution also leads to the heterogeneity distribution of jet draft at the critical voltage and affects on the necessary conditions during the formation of beta phase within the fibers structure. As it can be seen in Figure 7, by increasing the take up roller speed and the amount of yarn twist, the degree of crystallinity increases significantly. This can be contributed to the increasing the tension draft of droplet during the electrospinning process, which is a positive factor in the formation of crystalline phases.

### 3.3 DSC ANALYSIS

To evaluate the samples crystallinity, Equation (2) is used, in which the  $X_c$  indicate crystallinity degree,  $\Delta H_m$  represents melting enthalpy and  $\Delta H_{Lit}$  is melting enthalpy of 100% crystalline material in beta phase [22-24].

$$X_c = \frac{\Delta H_m (\text{Sample})}{\Delta H_{Lit}} \times 100, (\Delta H_{Lit} = 104.7 \frac{J}{g}) \quad (2)$$

Results showed that by adding the ZnO nanoparticles in the fiber structure, the crystallization of fibers is decreased comparing with the sample No. 1 and 4, but by increasing in the amount of nanoparticles from 10 to 15%, the crystallinity increased significantly. This could be due to the crystallization structure of nanoparticles. Also, the crystallinity of nanofibers increased with increasing the rotational speed of collector, as summarized in Figure 8.

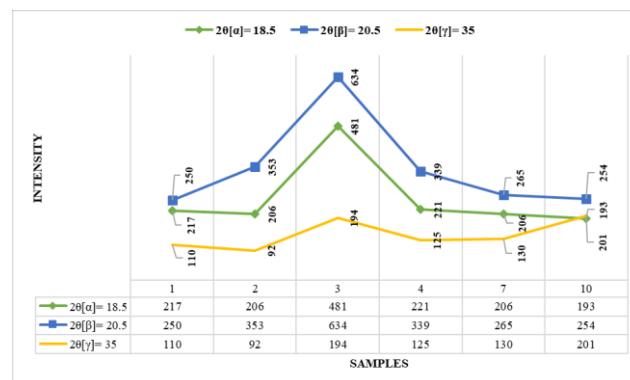


**Figure 8.** Summarized DSC results for samples

### 3.4 X-RAY DIFFRACTION ANALYSIS

The existence of peak at  $2\theta=20.5^\circ$ ,  $2\theta=18.5^\circ$  and  $2\theta=35^\circ$  angle in the X-ray diffraction pattern of samples indicates respectively the Beta, Alfa and Gamma phases in samples crystalline structure as it shown in Figure 9. It is believed that the Gamma phase is created in samples because of DMF presence in solution [22, 23, 25].

The diffraction intensity at  $20.5^\circ$  angle decreased by increasing the amount of ZnO nanoparticles. On the other hand, by increasing the amount of nanoparticles, the diffraction intensity at angle of  $35^\circ$  increases. This result is in consistent with the results of FTIR analysis. Nanoparticles reduce the dielectric coefficient, as it was mentioned, and therefore the fibers crystallinity is reduced because of low draft. Nano particles also create unbalance draft and the formation of beta phase will be interrupted. Twisting and higher take up roller speed cause increasing the draft of fiber, and as a result, crystalline phases increase too. As it can be observed in Figure 9, by adding ZnO nano-particles, beta phase is reduced in sample No. 10 and sample No. 3 have the most amount of beta phase comparing with others due to its higher draft during the production process.



**Figure 9.** X-ray diffraction patterns in  $\alpha$ ,  $\beta$  and  $\gamma$

### 3.5 TENSILE ANALYSIS

Tensile strength test is used to evaluate the mechanical properties of nanofibrous yarns. Tensile test was carried out using an Instron Universal Testing Machine (model 5566, USA) at the incremental speed of 10 mm/min and the gage length of 20 mm with the load cell of 2 kg to determine the mechanical properties of nanofibrous yarns similar to the guidelines of ASTM D 3822 standard. The average of five specimens for each sample was measured and reported. The results are shown in Table 2.

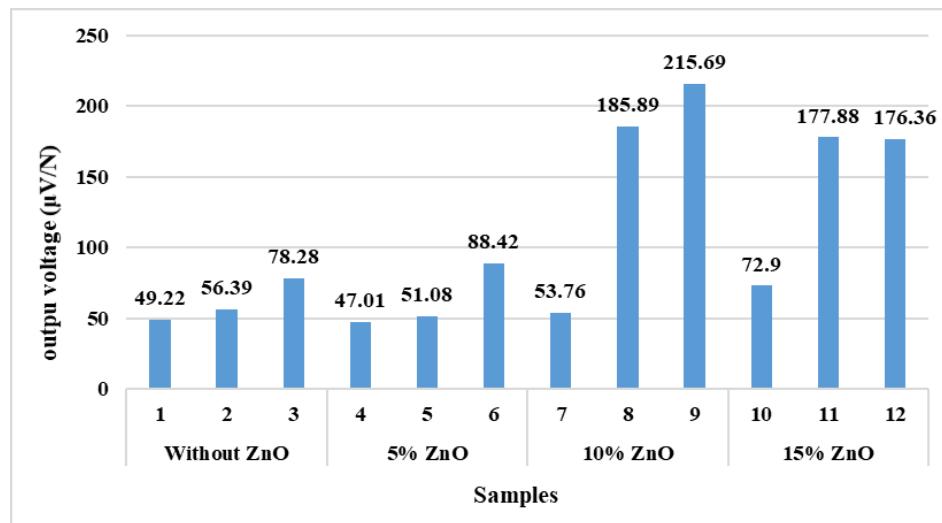
**Table 2.** Tensile properties of the nanofibrous yarns

Samples	Yarn count (tex)	Max load (N)	Strain (%)	tensile stress (cN/tex)	CV %
1	197	2.24	225.14	1.14	9.99
2	78	1.29	150	1.48	8.98
3	77	1.2	180.83	1.56	8.56
4	204	1.53	143.36	0.68	9.2
7	207	1.13	155.74	0.66	9.89
10	200	1.04	112.95	0.5	7.72

The nanofibrous yarns with ZnO nanoparticles showed less tensile stress value than the pure PVDF nanofibrous yarns. Increasing the amount of ZnO resulted in more decrease in the tensile strength of the yarns. This may be attributed to the reducing of crystallinity of samples with the presence of ZnO. Results also showed that the tensile strength of nanofibrous yarns increased by increasing the yarn twist and the speed of take up roller due to increase in their crystalline structure. Higher twist during nanofibrous yarn formation caused the higher amount of torsion. This resulted in a more compact structure of fibers and consequently, more forces were required for the yarn break. Furthermore, higher take up roller speed lead to more orientation in nanofibers. Accordingly, this leads to increase the mechanical properties of samples.

### 3.6 EVALUATION OF PIEZOELECTRIC PERFORMANCE

The piezoelectric performance of samples with different yarn twist, take up roller speed, and the percentage of ZnO nanoparticles were evaluated. The results summarized in Figure 10 indicate that, all samples output voltage increases with increasing the twist level of yarns. As it was mentioned, the increase in yarn twist caused more crystallinity and beta-phase crystalline structure and consequently the increase of the output voltage at a constant pressure.



**Figure 10.** Test results of the piezoelectric properties of fabricated samples.

The higher speed of the take up roller has the same effect but with a smooth trend, since it applies less draft in fibers comparing to the yarn twist. Furthermore, by increasing the amount of nanoparticles from 5% to 15% in the yarn nanofibers, the output voltage increased significantly which is contributed to the nature of strong piezoelectric properties of piezo-ceramic materials, i.e. ZnO, although the adding ZnO nanoparticles slightly reduced the output voltage up to 5%. The reason is due to reduction of beta crystalline phase in the presence of nanoparticles. By increasing the amount of nano-particles gradually the output voltage increases. That is because of the Gama crystalline phase of ZnO nanoparticle.

To investigate the effect of loading frequency on the output voltage, samples were impacted with different load frequencies. Results showed that by increasing the load frequency, the output voltage of samples increased significantly. Intensified frequency creates more instability in dipoles and causes the production of higher voltage.

### 4. CONCLUSIONS

The PVDF/ZnO nanofibrous yarns of various twist and ZnO proportions were produced in electrospinning system. The analysis of the fibers crystalline structure showed that the increase in fiber draft resulted by increase in both take-up roller and applied twist in yarn during the fiber formation in the electrospinning process leads to increasing the crystallinity degree. It can be extracted from FTIR analysis that the presence of nanoparticles causes a reduction in the degree of beta phase in the crystallinity of electrospun nanofibers. Further, due to the increase of the applied tension on nanofibers during fabrication and the increase in take-up roller speed which is significantly improved the crystalline structure and the molecular arrangement of nanofibers, their electrical outputs increased. The results of structural analysis are in accordance with the piezoelectric properties test. More twists increases the crystallinity of the fibers and thus the maximum output voltage swing can be seen. As an example, the sample with 10 wt.% ZnO nanoparticles has 216 mV output.

By increasing ZnO nanoparticles up to 10 wt.% the output voltage will increase but adding more ZnO nanoparticle will reduce the output voltage due to irregularities in the molecular structure of fiber caused by ZnO nanoparticles. Draft will increase the crystallinity of nanofibers while ZnO nanoparticles reduce the crystallinity. The adding more ZnO nanoparticles causes more reduction in the crystallinity and beta phase.

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