

PREPARATION AND DIELECTRIC PROPERTIES OF PVA AND P(VDF-HFP) ALTERNATING MULTILAYER FIBERS FOR ENERGY STORAGE APPLICATIONS

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Abstract

Dielectric polymer capacitors with high energy storage density are becoming increasingly important for the current advancements in electrical power applications. Energy storage performance in dielectric materials not only depends on the dielectric breakdown strength but also dielectric constant. This study focuses on the dielectric properties of polyvinyl alcohol (PVA) and poly(vinylidene fluoride-co-hexafluoropropylene) (P(VDF-HFP)) alternating multilayer fibers with different layers. Alternating multilayer structures layer by layer were prepared by using an electrospinning technique with suitable conditions, and the thickness of multilayer fibers was controlled by time. Alternating multilayer of P(VDF-HFP)/PVA structures can bring impressive the dielectric performance and energy storage efficiency. The morphology of obtained fibers was investigated by SEM. The dielectric properties and conductivity of multilayer fiber were investigated by using an LCR meter. The dielectric constant and conductivity of PVA and P(VDF-HFP) alternating multilayer fibers increase with the increasing number of layers. The dielectric constant of alternating multilayer fibers (8 layers) at a frequency of 20 Hz is observed to increase by 38.7% compared to PVA fibers. It appears that the integrated PVA and P(VDF-HFP) fibers, through an alternating multilayer structure, influence the interfacial polarization and the dominant charge carriers in each fiber layer. Additionally, the energy storage density of alternating multilayer fibers (8 layers) at 20MV/m is enhanced by 34.0% compared to PVA fibers.

Keywords: Alternating multilayers; Dielectric properties; Electrospinning technique; P(VDF-HFP); PVA

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Introduction

Energy demand refers to the consumption of energy by human activity, driving the entire energy system. Energy storage is a crucial component of energy demand for utilizing electricity and is divided into two main essentials: ultrahigh power density (ultrafast charging) and ultrahigh energy density. Examples of energy storage devices include lithium-ion batteries, dielectric capacitors, and supercapacitors (Hao, 2013). Batteries exhibit high power density, while dielectric capacitors possess high energy density. Supercapacitors (Electrochemical capacitors) offer relatively high power density and energy density, but they have a limited cycle life, meaning they can only be charged and discharged a certain number of times before their performance degrades. Moreover, supercapacitors containing environmentally unfriendly chemicals can have negative impacts on the environment if not disposed of properly. Energy storage density can be improved in two ways: by increasing the dielectric constant and enhancing the electrical breakdown strength.

Numerous researchers have delved into polymer/polymer dielectrics and discovered that employing a multi-layer structure appears to be an effective approach for enhancing energy storage properties (Mackey *et al.*, 2012; Carr *et al.*, 2013; Tseng *et al.*, 2014). Besides, the combination of the advantages of two polymers with multilayer structures can reduce the leakage current and increase the breakdown strength. Previously, Zhu *et al.* asserted that the layered structure provided the polarization hysteresis decreased with decreasing layer thickness due to significantly reduced dielectric losses (Tseng *et al.*, 2014). To maintain high energy storage performance, it is necessary to maintain low losses to reduce the discharged energy density of the capacitor. Moreover, the layered hetero-structure and interfaces between layers provided the space charge interfaces from passing from one layer to the next.

This study focuses on two materials, they are P(VDF-HFP) and PVA. P(VDF-HFP) is a ferroelectric material, characterized by its semi-crystalline thermoplastic fluoropolymer nature. This highly flexible material is easily processed and boasts a high dielectric constant. Moreover, P(VDF-HFP) is a highly non-reactive thermoplastic fluoropolymer, displaying resistance to the effects of temperature and various chemicals. On the other hand, PVA is known for its biocompatibility and biodegradability and readily dissolves in polar solvents such as water (Jambaladinni and Bhat, 2021). It exhibits high electrical breakdown

strength. The linear polymers of PVA, forming closely arranged polymer chains, create a barrier that impedes the penetration of an electric field (Vatanpour *et al.*, 2023). Electrospun membranes of these materials with a large surface area to volume ratio exhibit high interfacial polarization through electrospinning processes. Moreover, alignment of the polarities of nanofibers can be created by using electrospinning technique (Tohluebaji *et al.*, 2021; Khao-iam *et al.*, 2023).

So, this work aims to enhance the energy storage density of environmentally friendly dielectric materials used in dielectric capacitors with alternating multilayers. The PVA and P(VDF-HFP) alternating multilayer fibers were prepared by using the electrospinning process. Moreover, this work studies the energy storage performance on the dielectric and ferroelectric properties and microstructure of PVA and P(VDF-HFP) alternating multilayer fibers with different numbers of layers.

Experiments

Preparation of alternating multilayer fibers

Polyvinyl alcohol (PVA) purchased from Chem-Supply Pty Ltd, Australia, was dissolved in DI water. Polyvinylidene fluoride-hexafluoropropylene (P(VDF-HFP)) powder, Solef 11010/1001, purchased from Solvay Solexis, Belgium, was dissolved in DMF. The PVA solution was stirred at 50°C and 200 rpm for 6 h. The P(VDF-HFP) solution was stirred at 50°C and 200 rpm for 4 h. The solutions were stirred to ensure homogeneity. Afterward, they were stored at 35°C for two hours to remove air bubbles. Alternating multilayer 1-, 2-, 4- and 8-layer fibers were obtained by the electrospinning process, as illustrated in Figure 1. The multilayer fiber mats were fabricated by electrospinning technique. The solution was loaded into a 20 mL plastic syringe connected to a stainless-steel needle (gauge 20.5) and fed with a syringe pump (Nz1000 NEWERA Pump, NY, USA) at a flow rate of 1.0 mL/h. The rotation of the collection is 300 rpm. Moreover, the voltage of this experiment was chosen is 14 and 18 kV. An electric field was generated by a high DC voltage supply (Trek model 610E, New York, NY, USA). The positive pole was connected to the steel. Due to the equal quantities of PVA and PVDF-HFP solution at the collector, it is assumed that their thicknesses are approximately equal. The thickness of multilayer fibers was controlled by time as shown in Table 1. The solutions were dried in the oven for 30 minutes at 120°C to remove the solvent.

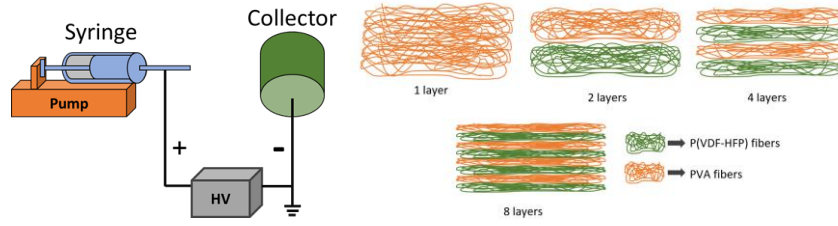


Figure 1. Schematic illustration of the fabrication process of PVA and P(VDF-HFP) alternating multilayer fibers via electrospinning process

Table 1. Time for forming alternating multilayer fibers

Layers	Time (min)/layer	Thickness of samples (mm)
1	120	80±10
2	60	65±15
4	30	70±10
8	15	70±10

Characterization

The morphology of P(VDF-HFP) fibers and PVA fibers were characterized by scanning electron microscopy (SEM, TM3030Plus, Hitachi). Further analysis using the ImageJ program was conducted on the SEM images to determine the diameters of the fibers. The diameters of 100 fibers were measured from each image. The underlying crystal structure of P(VDF-HFP) fibers and PVA fibers was determined by X-ray diffraction (XRD, PNAanalytical, Empyrean, Netherlands). XRD was used to identify crystal phase diffraction on semi-crystalline PVDF material. The diffraction peak was observed from $2\theta = 10^\circ$ to $2\theta = 40^\circ$. The functional groups of P(VDF-HFP) fibers and PVA fibers were identified through Fourier-transform infrared spectroscopy (FTIR, VTR, Vertex70, Bruker, Germany). FTIR was used to characterize functional groups of the sample's light absorption from 400 cm^{-1} to 1350 cm^{-1} , the resolution of it is 4 cm^{-1} . Subsequently, the acquired information was compared with the database. It can provide the dielectric properties of the materials by LCR meter in the frequency range of $20\text{-}10^5\text{ Hz}$. The dielectric constant (ϵ_r) and conductivity (σ) were calculated by equations (1) and equations (2), respectively.

$$\epsilon_r = \frac{Cd}{\epsilon_0 A} \quad (1)$$

$$\sigma = \frac{Cd}{A} \quad (2)$$

when C is the capacitance (F), d is the sample thickness (m), ϵ_0 is the permittivity of air ($8.853 \times 10^{-12} F/m$), A is the electrode area (m), and G is

the conductance (s) (Chooseng *et al.*, 2021). The hysteresis loops depicting polarization (P) versus electric field (E) were generated utilizing a high voltage power amplifier operating at low current. All samples underwent an electric field (E) of 20 MV/m . When the applied electric field (E) varies between two equal but opposite values, the dipole polarization (P_d) approaches the spontaneous polarization (P_s). Additionally, P_d is zero when the electric field equals the coercive field (E_c) and has a nonzero value at zero fields, known as remnant polarization (P_R). The saturated switching dipole polarization curve is assumed to have symmetric branches, as expressed by equation (3.1) - equation (3.3).

$$P_d^+(E) = P_s \tanh \left[\frac{E - E_c}{2d} \right] \quad (3.1)$$

$$P_d^-(E) = -P_d^+(-E) \quad (3.2)$$

$$d = E_c \left[\log \left(\frac{1 + P_R/P_s}{1 - P_R/P_s} \right) \right]^{-1} \quad (3.3)$$

when P_d^- and P_d^+ are negative and positive going polarization branches of the hysteresis loop respectively, E is applied electric field, P_d is the dipole polarization, P_s is the spontaneous polarization and $P_d > P_s$, P_R is remnant polarization, and E_c is the coercive field (Shrestha, 2012). The P-E loop of this experiment should relate to these equations. The Energy storage density (U_e) and %efficiency can be calculated by area of P-E loop that is related to equation (4) and equation (5), respectively.

$$U_e = \int_{P_R}^{P_s} E dP \quad (4)$$

$$\%efficiency = \frac{U_e}{U_e + U_l} \times 100\% \quad (5)$$

When P_s is the spontaneous polarization and P_R is remnant polarization, E is the electric field, U_l and U_e is energy loss and energy storage density of materials (Chooseng *et al.*, 2021).

Results and Discussion

To prepare alternating multilayer fibers comprising two different polymers, PVA and P(VDF-HFP), were employed in the electrospinning process. The resulting PVA and P(VDF-HFP) fibers can be observed in Figure 2. Further analysis via the image J program was conducted on the SEM images to determine the diameters of the fibers. The diameters of 100 fibers were measured from each image in Figure 2 and data was exported for analysis in Figure 3(a-d). Each mode of PVA and P(VDF-HFP) was counted and shown in Figure 3(e). The maximum counts of the P(VDF-HFP) and PVA fiber diameter modes significantly show at the electrical field of 14 kV and 18 kV, respectively. The maximum count and the diameters of PVA fibers at 18 kV are 44 and 400-500 nm, respectively. While, the maximum count and the diameters of P(VDF-HFP) fibers at 14 kV are 34 and 1500-2000 nm, respectively. The diameter of the fiber not only

depends on the flow rate, and intensity of the electric field but also the viscosity of the solution. In our previous work (Nawaka and Putson, 2020), it was found that the decrease in average fiber diameter was due to decreased viscosity and the surface tension of the electrospun solution, which tends to decrease the fiber diameter.

The XRD spectra of PVA and P(VDF-HFP) fibers were shown in Figure 4. PVA fibers consist of one peak at $2\theta \approx 19.5^\circ$, it corresponds to the reflection of (101) plane (El-Desoky *et al.*, 2020; Norouzi *et al.*, 2021). P(VDF-HFP) fibers consist of two peaks at $2\theta \approx 18.2^\circ$ (020) and $2\theta \approx 20.0^\circ$ (110)/(200), there are α phase and β phase, respectively (Durga Prasad and Hemalatha, 2019; Hussein *et al.*, 2019). Moreover, β crystalline phase refers to polar polymers.

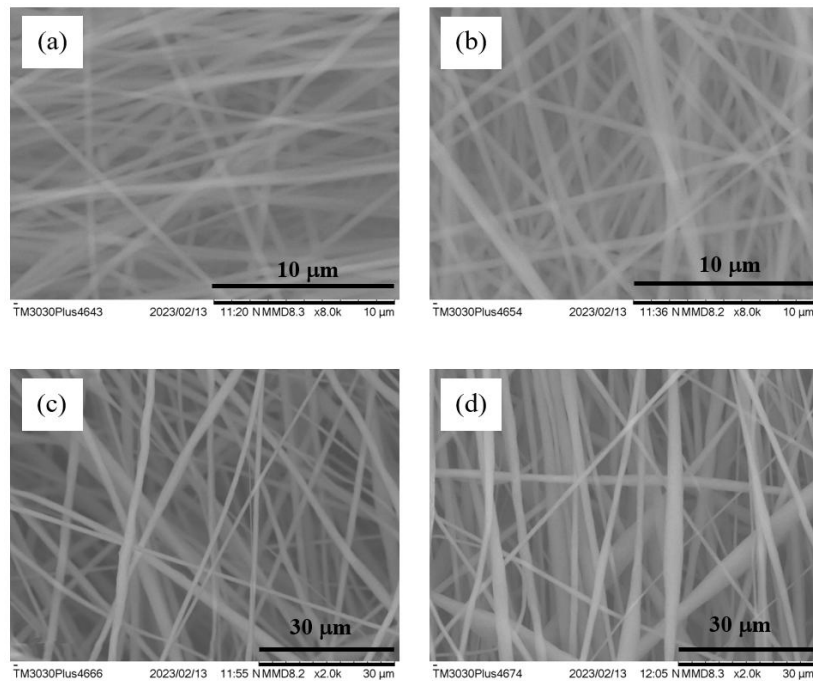


Figure 2. Surface scanning electron microscopy (SEM) images of PVA fibers at (a) 14 kV, (b) 18 kV, and P(VDF-HFP) fibers at (c) 14 kV and (d) 18 kV, respectively

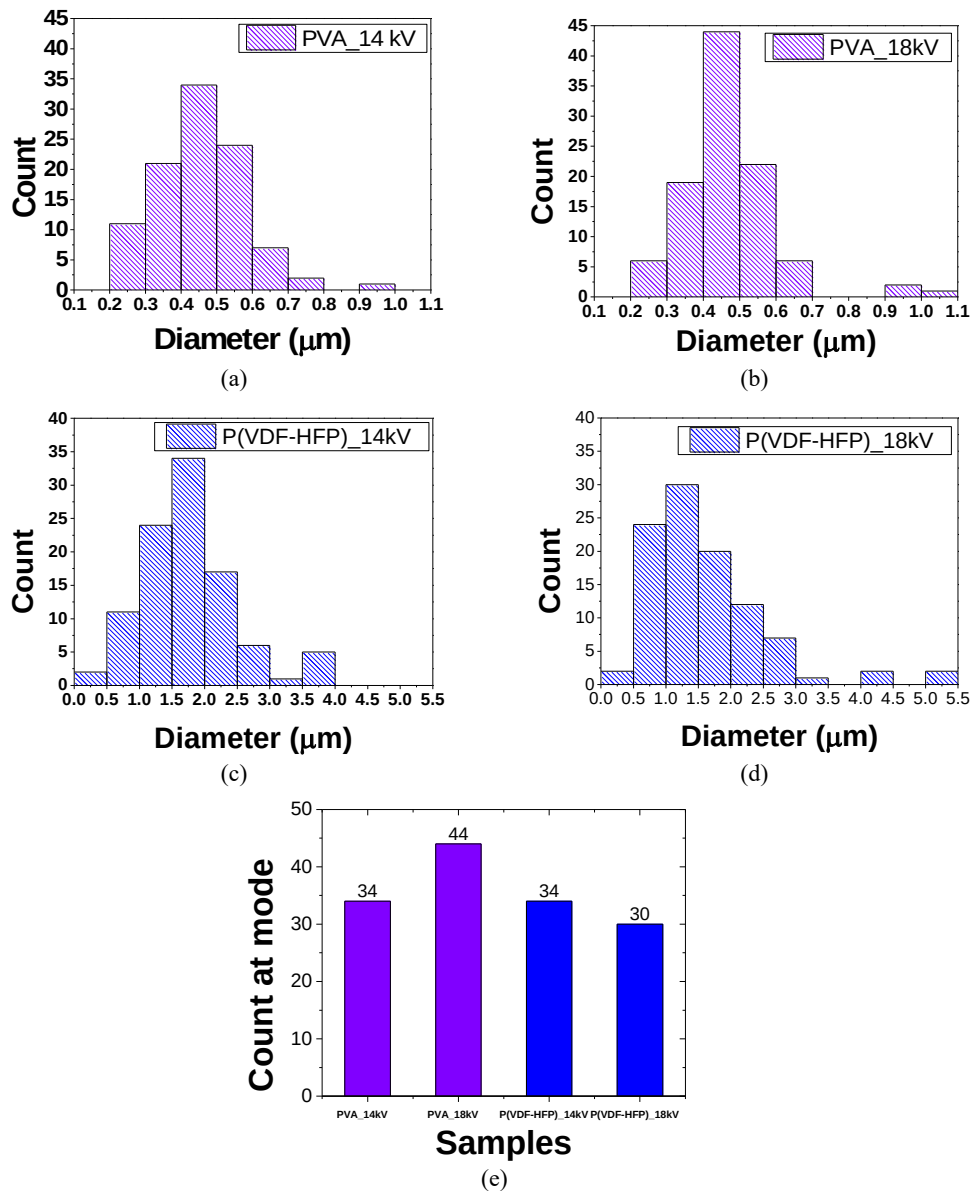


Figure 3. Fiber diameter analysis/Histogram of (a,b) PVA and (c,d) P(VDF-HFP) fiber diameter distribution at the applied voltage of 14 kV and 18 kV, respectively; and (e) count of mode in the histogram (a)-(d)

To identify the functional groups of fibers, one must examine the FTIR spectrum in Figure 5. In this work, the functional group of P(VDF-HFP) fibers and PVA fibers is shown in Table 2. They relate to the formula of PVA and P(VDF-HFP) which are $(-\text{CH}_2\text{CH}(\text{OH})-)_x$ and $(-\text{CH}_2\text{CF}_2-)_x[-\text{CF}_2\text{CF}(\text{CF}_3)-]_y$, respectively.

Dielectric testing was conducted to assess the impact of the number of layers on the dielectric properties of the PVA and P(VDF-HFP) alternating multilayer fibers. Figure 6(a-c). shows the dielectric constant, dielectric loss, and conductivity of PVA

and P(VDF-HFP) alternating multilayer fibers versus frequency. Figure 6(d). shows the dielectric constant versus the number of layers at 20 Hz and 1 kHz. In this work, we focus on the low number of fiber structures within 8 layers. The dielectric constant and conductivity of PVA and P(VDF-HFP) alternating multilayer fibers increase with the increasing number of layers. The dielectric constant of alternating multilayer fibers (8 layers) at a frequency of 20 Hz is observed to increase by 38.7% compared to PVA fibers. Previously, Feng *et al.* reported that the multilayer structure can

significantly improve energy storage efficiency and enhance both breakdown strength and permittivity (Feng *et al.*, 2020). It was found that the breakdown strength increased with the number of layers (1-9 layers) which can be explained by single electron theory. Besides, when the layers grow to 11 layers, the breakdown strength decreases slightly. Nikrusong *et al.* purpose comparison between P(VDF-HFP) films and fiber membranes, the result shows that the dielectric constant of fiber membranes and thin films is equal at high frequency. However, the dielectric constant of fiber membranes with high surface and aspect ratio is higher than film at low frequency due to free electron density and mobile charge carriers (Tohluebaji *et al.*, 2019). The dielectric constant of alternating multilayer fibers is higher than that of PVA fibers. The investigation of polarization effects in the multilayered fibers extended to the analysis of orientation-related, ionic, and interfacial polarization. Various layer materials exhibited different interactions within the multilayer fibers, resulting in varying interphase fractions in the alternating multilayer fibers. The rise in dielectric constant is mainly attributed to interfacial polarization occurring at the surface area between the layers of nanofibers. Dielectric loss of polymer matrix decreases with frequency, as these are affected by the orientation of electric dipoles that can follow the low frequencies (Nawaka and Putson, 2020). At high frequencies, the dipoles don't have enough time to reorient themselves in the direction of the applied electric field. The conductivity and dielectric loss increase when the number of layers is increased. It may be attributed to the number of multilayer polymer structures with interface region content providing the quantity of mobile charge carriers increased, which increased the conductivity and the dielectric loss.

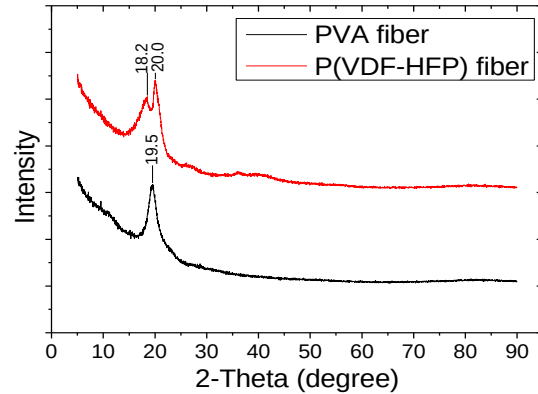


Figure 4. XRD patterns of PVA and P(VDF-HFP) fibers

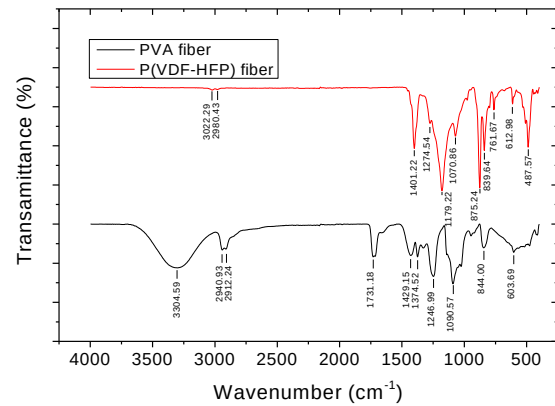


Figure 5. FT-IR spectrum of PVA and P(VDF-HFP) fibers

Table 2. The FTIR results of the PVA and P (VDF-HFP) fibers

Wave number cm^{-1}	observed wavelength	reference
PVA fibers		
844.00	C-C stretching	(Kharazmi <i>et al.</i> , 2015)
1070.86	C-O stretching	(Kharazmi <i>et al.</i> , 2015)
2912.24	CH_2 asymmetric stretching	(Gomaa <i>et al.</i> , 2018; Kharazmi <i>et al.</i> , 2015)
2940.93	C-H	(Gomaa <i>et al.</i> , 2018)
3304.95	O-H stretching	(Gomaa <i>et al.</i> , 2018; Kharazmi <i>et al.</i> , 2015)
P(VDF-HFP) fibers		
839.64	CF_3 rocking	(Yang <i>et al.</i> , 2012)
875.24	CF_3 rocking	(Yang <i>et al.</i> , 2012)
1274.54	CF_2 asymmetric stretching	(Yang <i>et al.</i> , 2012)
1401.22	CF_2 symmetric stretching	(Yang <i>et al.</i> , 2012)
2980.43	CF_2 symmetric stretching	(Yang <i>et al.</i> , 2012)
3022.29	CF_2 asymmetric stretching	(Yang <i>et al.</i> , 2012)

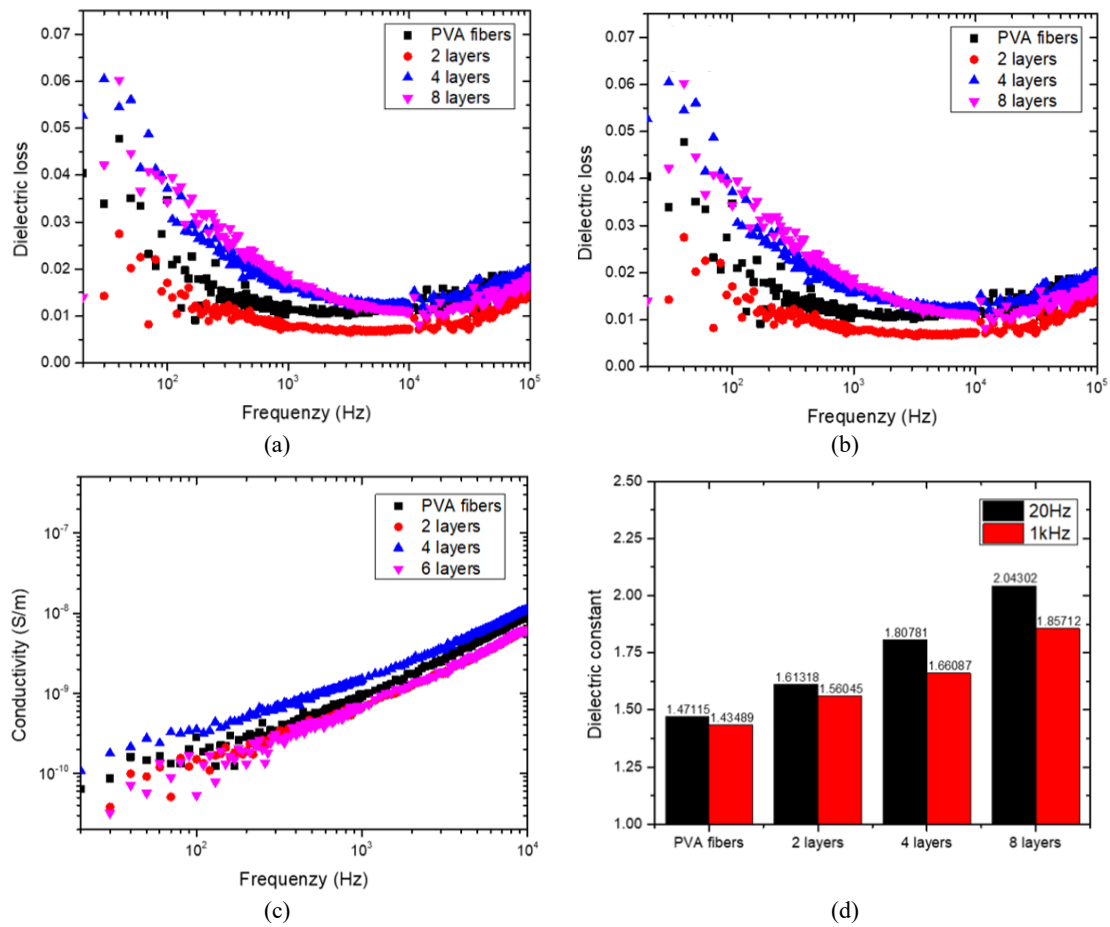


Figure 6. (a) Dielectric constant, (b) Dielectric loss, (c) Conductivity of PVA and P(VDF-HFP) alternating multilayer fibers and (d) Dielectric constant of alternating multilayer fibers at 20 Hz and 1 kHz.

The polarization of alternating multilayer fibers at different electric fields was shown in Figure 7(a). The polarization of alternating multilayer fibers increases as the number of layers increases. The energy storage density of fiber was calculated by the area of the P-E loop with Equation (4), shown in Figure 7(b). Consequently, the P-E loop of this work relates to Equation (3.1) and (3.2). The energy storage density of 1-, 2-, 4- and 8-layer fibers are 8.30, 9.66, 9.19, and 11.12 mJ/cm³ at 20 MV/m, respectively. The energy

storage density of alternating multilayer fibers increases as the number of layers increases. The integration of PVA and P(VDF-HFP) fibers via an alternating multilayer structure influences the interfacial polarization between layers. The efficiency of these fibers is higher as the number of layers increases. Furthermore, the remnant polarization and maximum polarization of PVA and P(VDF-HFP) alternating multilayer fibers in Figure 7(c) and Figure 7(d) are correlated with the P-E loop and energy storage density.

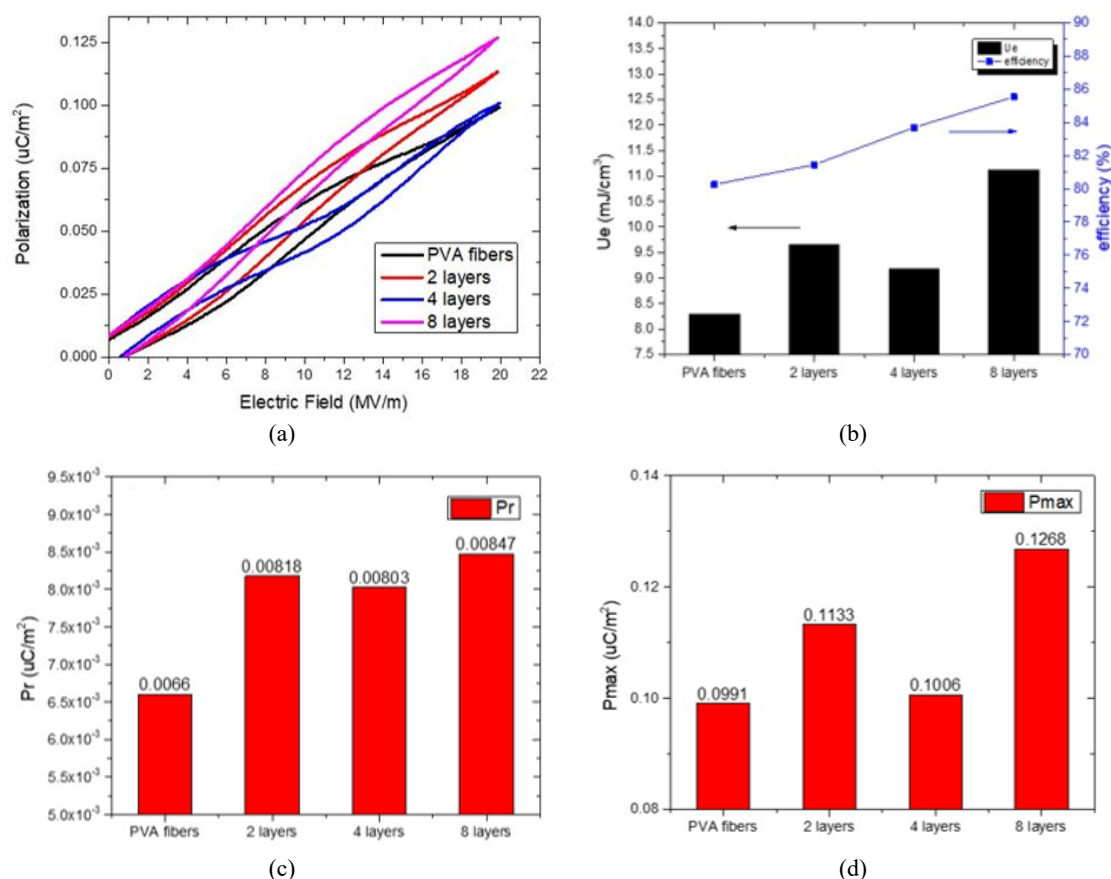


Figure 7. (a) P-E loop, (b) Energy storage density and efficiency, (c) Remnant polarization, and (d) Maximum polarization of PVA and P(VDF-HFP) alternating multilayer fibers at different numbers of layers

Conclusions

This work proposed the energy storage performances on the dielectric and ferroelectric properties of PVA and P(VDF-HFP) alternating multilayer fibers. All samples were prepared using the electrospinning process. The results showed that the dielectric constant, remanent polarization, and the energy storage density of alternating multilayer fibers increase when the number of layers increases. Integrated PVA and P(VDF-HFP) fibers via alternating multilayer structure significantly affect the interfacial polarization and the dominant charge carriers of each fiber layer.

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