



Research Article

Preparation and Characterization of Peo/Pvdf Polymer Blends at Varying Compositions

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Abstract

Polymer blending is an effective strategy for tailoring material properties by combining complementary polymers to achieve enhanced structural and functional performance. In this study, poly (ethylene oxide) (PEO) and poly (vinylidene fluoride) (PVDF) blends were prepared at weight ratios of 90/10, 80/20, 75/25, and 50/50 using the solution casting method, with N, N-dimethylformamide (DMF) as the solvent. Fourier Transform Infrared (FTIR) spectroscopy was employed to investigate composition dependent molecular interactions through the analysis of C–O–C stretching in PEO and C–F stretching in PVDF, while X-ray Diffraction (XRD) was used to assess crystalline phase structure and degree of crystallinity. The FTIR spectra revealed composition induced peak shifts and intensity variations, indicating intermolecular interactions and partial miscibility. XRD analysis demonstrated that PEO-rich blends exhibited higher crystallinity, with characteristic peaks at $2\theta \approx 19.82^\circ$ and 23.86° , while increased PVDF content reduced PEO crystallinity and enhanced PVD associated peaks. Results confirm that blend composition significantly influences molecular interactions, crystalline structure, and phase stability, underscoring the importance of compositional tuning for optimizing PEO/PVDF based solid polymer electrolytes.

Keywords: PEO, PVDF, polymer blend, FTIR spectroscopy, XRD.

1. INTRODUCTION

Polymer blending is a versatile approach used to engineer materials with combined or enhanced properties that are not easily achieved by individual polymers alone (Ahhbab et al., 2025). Polymer blending is a powerful approach to tailor material properties by combining complementary polymers. There are differences among various polymers as for the ability to form a homogeneous system in two or more blends, and the mutual solubility between polymers can be defined by the concept of compatibility (Liu et al., 2019). In particular, blends of poly (ethylene oxide) (PEO) and poly (vinylidene fluoride) (PVDF) are compelling where PEO contributes high ionic conductivity and flexibility, while PVDF offers mechanical robustness and chemical resistance and thermal stability. Together, these properties make PEO/PVDF blends promising candidates for solid polymer electrolytes in energy storage systems (Dirican et al., 2019).

Understanding the molecular and crystalline structure of such blends is essential for optimizing their functional performance. FTIR spectroscopy plays a pivotal role by identifying functional groups and detecting vibrational shifts (e.g., ether C–O–C in PEO and C–F in PVDF), offering insight into intermolecular interactions and miscibility. Meanwhile, XRD analysis informs on crystalline structure and degree of crystallinity valuable data especially in blends where crystalline behavior dictates mechanical and electrochemical behavior.

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- Most studies examine only a narrow band of blend ratios commonly around 70/30 or 60/40, There is a clear lack of research into blends with very high PEO content (>80 %) and equimolar blends (50/50) underexplored (Basappa et al., 2022).
- Qualitative rather than quantitative FTIR analysis: Many works present FTIR spectroscopic data without systematically analyzing peak shifts, intensity changes, or peak area variations factors essential for quantitatively assessing miscibility and interaction strength.(Riaz & Ashraf, 2015).
- Composition dependent vibrational mode evolution often neglected; Few reports detail how characteristic vibrational bands like the C–O–C in PEO or the C–F in PVDF evolve across different blend ratios, despite their potential to unveil functional group interactions (Riaz & Ashraf, 2015).
- Structure property relationships at extreme blend ratios unclear; Although PEO/PVDF blends are recognized for electrolyte applications, the molecular interactions and potential performance at extreme compositions remain largely undefined (Ushakova et al., 2020).
- Processing conditions seldom linked with FTIR findings the effects of solvent choice, film casting method, or drying parameters on molecular interactions and phase compatibility have not been rigorously explored via FTIR (Bao et al., 2025).

These gaps warrant more comprehensive study; thus, our work focuses on PEO/PVDF blends prepared at weight ratios of 90/10, 80/20, 75/25, and 50/50. We employ FTIR spectroscopy to monitor changes in vibrational modes indicative of inter-chain interactions, and XRD to assess crystallinity, phase structure, and potential polymorphic shifts. By bridging FTIR and XRD findings across a broad compositional range, this study endeavors to fill previous research gaps and deepen understanding of the interplay between blend composition, molecular interaction, and crystalline behavior in PEO/PVDF systems.

PEO provides segmental mobility and ion transport, whereas PVDF contributes mechanical robustness and thermal/chemical stability together enabling solid-electrolyte oriented blends. Recent studies reaffirm that tuning the PEO:PVDF ratio modulates amorphous content (beneficial for ion transport) while maintaining structural integrity (Concha et al., 2024).

According to Cai et al, (2017) blending of PEO/PVDF systems, FTIR tracks composition dependent interactions primarily via the C–O–C stretching of PEO ($\approx 1100\text{ cm}^{-1}$) and C–F/skeletal bands of PVDF (notably ≈ 840 and $\approx 1275\text{ cm}^{-1}$ for β -phase indicators). Authors stress moving beyond qualitative “peak present/absent” toward quantitative peak-shift/area analysis to infer miscibility and interaction strength, FTIR also helps distinguish PVDF polymorphs: α , β , and γ phases show characteristic band sets that correlate with XRD signatures; unified protocols to identify phases from IR have been proposed and validated across processing methods (Cai et al., 2017).

Blends intended for solid polymer electrolytes show that increasing amorphous content (from PEO or plasticization) can enhance ionic transport, but mechanical strength and phase stability depend on PVDF’s crystalline framework; studies that jointly analyze FTIR (molecular interactions) and XRD (phase/crystallinity) provide the clearest structure–property links (Basappa et al., 2022).

In another research by Yasar et al,(2024) investigated PVDF phase content ($\alpha \leftrightarrow \beta \leftrightarrow \gamma$) is highly sensitive to processing (solvent, temperature, stretching, electrospinning, additives), which FTIR and XRD detect via band/peak evolution; recent reports map β -phase optimization routes and $\gamma \rightarrow \beta$ transformation in solution-processed films. Such sensitivities imply that reporting processing conditions is crucial for reproducibility and cross-study comparison (Yasar et al., 2024).

MATERIAL AND METHOD

MATERIAL

Poly(ethylene oxide) (PEO) analytical grade, average molecular weight $\approx 600,000\text{ g/mol}$, purchased from Sim best scientific & chemicals (Minna Niger State Nigeria opposite FUT Minna), used without further purification, Poly(vinylidene fluoride) (PVDF) analytical grade, average molecular weight $\approx 534,000\text{ g/mol}$, obtained from Sim best scientific & chemicals (Minna Niger State Nigeria opposite FUT Minna) used as received, Solvent analytical grade *N,N*-dimethylformamide (DMF) ($\geq 99.5\%$ purity, Sim best scientific & chemicals (Minna Niger State Nigeria opposite FUT Minna) was used as the common solvent for blending.

PREPARATION OF PEO/PVDF BLENDS

Using solution casting method, PEO and PVDF were weighed to obtain four weight ratios: 90/10, 80/20, 75/25, and 50/50 (w/w). Each polymer was separately dissolved in DMF at room temperature with continuous magnetic stirring for 2 h to ensure complete solvation. The solutions were then combined according to the target ratios and stirred for an additional 3 h to promote homogeneity. The homogeneous polymer solutions were cast into clean glass Petri dishes and the resulting films were vacuum-dried at $50\text{ }^{\circ}\text{C}$ for 24 h to remove residual solvent. The dried films were stored in a desiccator until

further analysis. FTIR spectra were recorded using a (ATR-FTIR) Perkin Elmer FTIR spectrometer in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1} and 32 scans per sample. Films were directly mounted on the sample holder without further preparation.

The spectra were analyzed to monitor the C–O–C stretching vibration of PEO (~1100 cm⁻¹) and C–F stretching vibration of PVDF (~840 cm⁻¹). Shifts in peak positions, changes in intensity, and peak broadening were examined to evaluate intermolecular interactions and miscibility. While XRD patterns were collected using an AV-2a diffractometer operating at 40 kV and 30 mA with Cu K α radiation ($\lambda = 1.5406 \text{ \AA}$). Diffraction patterns were recorded over a 2θ range of 5°–60° at a scan rate of 2°/min. The degree of crystallinity was calculated using the ratio of the integrated crystalline peak area to the total scattering area, following the method described by Segal et al. (1959). Special attention was given to the Characteristic peaks of crystalline PEO (~19.1° and 23.3°) and PVDF α - and β -phase peaks (~18.5° and ~20.6°). Peak position shifts and changes in intensity were used to assess structural modifications induced by blending.

RESULT AND DISCOUSION

FOURIER TRANSFORM INFRARED SPECTROSCOPY (FTIR) ANALYSIS OF PEO/PVDF POLYMER BLEND

Figure 1 shows the FTIR spectra in the wave range $4000 - 500 \text{ cm}^{-1}$ of all PEO/PVDF polymer blend at different percentage (90/10, 80/20, 75/25, 50/50). The FTIR spectra show characteristic peaks corresponding to functional groups in the PEO and PVDF polymers. The relative intensity of the peaks changes as the composition of the blend varies, indicating interactions between PEO and PVDF in the blends. The spectra reveal the presence of chemical composition and shifting bonds in PEO/PVDF (90/10) at these vibrations a large broad band between 3620 cm^{-1} to 3288 cm^{-1} is observed (broad peak in all spectra), those band is associated bands of asymmetric corresponding to O-H stretching vibrations (alcohol) which may arise from moisture absorption or hydrogen bonding. Near 2900 cm^{-1} this peak represents C-H stretching vibrations, characteristic of PEO. Around $1455\text{--}1400 \text{ cm}^{-1}$ associated with CH_2 scissoring vibrations, typically present in both PEO and PVDF. Between $1200\text{--}1000 \text{ cm}^{-1}$ Strong peaks in this region are due to C-F stretching vibrations from the PVDF polymer. As the PVDF content increases (green spectrum, PEO 50/PVDF 50), these peaks become more prominent. Around $950\text{--}800 \text{ cm}^{-1}$ peaks here also represent C-F and CH_2 wagging vibrations from PVDF. Their intensity increases with higher PVDF ratios, the intensity of peaks associated with PVDF (e.g., C-F stretches) becomes more dominant. The bending small peak observed between 1094 cm^{-1} to 1450 cm^{-1} indexed to CF_2 - in PEO/PVDF blend, CH_2 deformation occurs at 1340 cm^{-1} , this peak slightly shifted towards the higher frequency wavenumber due to effect of increasing the percentage of PVDF and decreasing the percentage of PEO, the intensity of peaks associated with PVDF (e.g., C-F stretches) becomes more dominant. Whereas the peaks appear at 840 cm^{-1} , 957 cm^{-1} , 1090 cm^{-1} , and 1465 cm^{-1} related to the α -phase crystal of the PVDF, 1145 cm^{-1} , and 1245 cm^{-1} belongs to the β -phase crystal. vibrational and bending modes at 840 cm^{-1} indicate the host polymer matrix's existence in the PEO/PVDF blend complexation belong to CH_2 rocking vibrations of methylene groups and are related to helical structural group of PEO (Das & Ghosh, 2017).

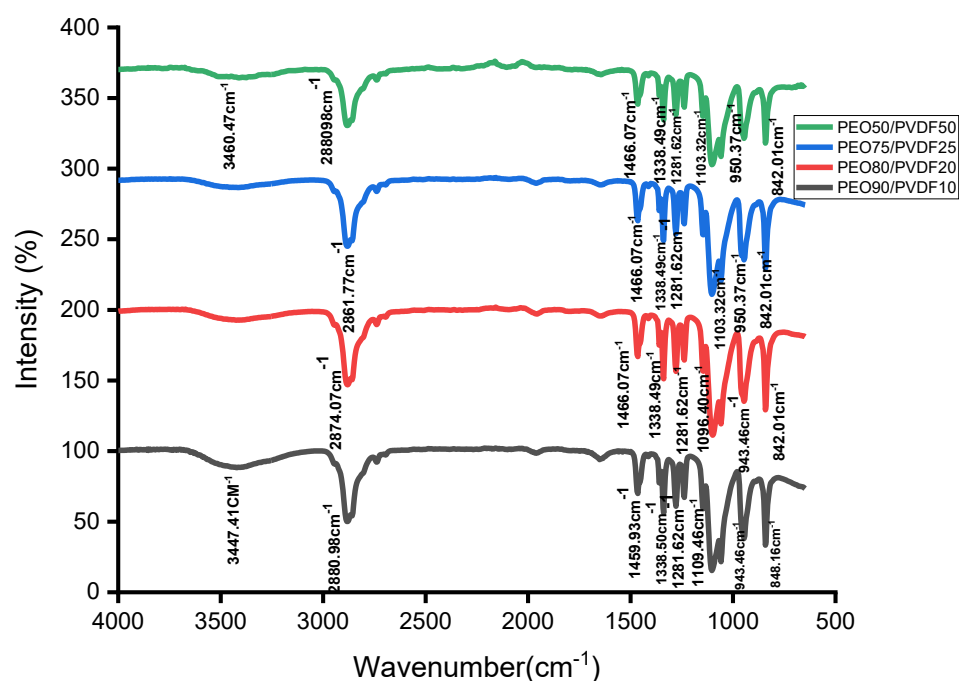


Figure 1: FTIR spectra of PEO90/PVDF10, PEO80/PVDF20, PEO75/PVDF25 and PEO50/PVDF50 for blend films

XRD ANALYSIS OF POLYMER BLEND

Figure 2 indicate the crystalline regions in the blends which reveals a series of diffraction peaks corresponding to its semi-crystalline nature. The main peaks for PEO and PVDF are visible, highlighting both polymers maintain their crystalline structure at varying ratios. The reflection of X-ray is observed at different angles 2θ of 8.27° , 17.90° , 19.822° , 21.61° , 23.86° , 27.00° , 37.887° & 44.066° and so on. The values of angle of diffraction, hkl , d -spacing, FWHM (β), crystalline structure (\AA) and dislocation density (δ) are calculated for all major diffraction lines, as tabulated in table 1 International Centre for Diffraction Data (ICDD) is used to compare the diffraction data obtained with the database. These values provide a base to solve the pattern. If there are some diffraction lines due to impurities in material or due to other reasons, then it creates problems and needs extra skills to tackle them. Figure 2 shows the respective diffraction patterns of undoped polymer blends of different ratio PEO90/PVDF20, PEO80/PVDF20, PEO75/PVDF25, PEO50/PVDF50. The intensity of peaks in the XRD graph corresponds to the relative abundance of a particular crystalline phase in the sample. The intensity of the peak's changes across the samples, the most prominent diffraction peak appears at approximately $2\theta = 19.822^\circ$, which is characteristic of the crystalline phase of PEO. This peak corresponds to the (110) lattice plane and is a well-known feature of high-molecular weight PEO. A second, broader peak is observed around 23.86° (2θ) indicating additional crystalline order in the material. The 90/10 blend has stronger peaks compared to the 50/50 blend. This suggests a higher degree of crystallinity in PEO-rich blends, as PEO is highly crystalline. Some peaks from PEO and PVDF overlap, indicating partial miscibility or compatibility between the two polymers. However, distinct peaks suggest that they remain largely phase-separated. By comparing the intensity and position of the peaks in the four graphs, the crystalline phases are identified in the samples. The crystallinity of these polymers was significantly reduced and the peaks were broadened. As the PEO content decreases (from 90/10 to 50/50), the peak intensities decrease for PEO and increase for PVDF. This is expected as the contribution of each polymer to the overall crystallinity changes with composition.

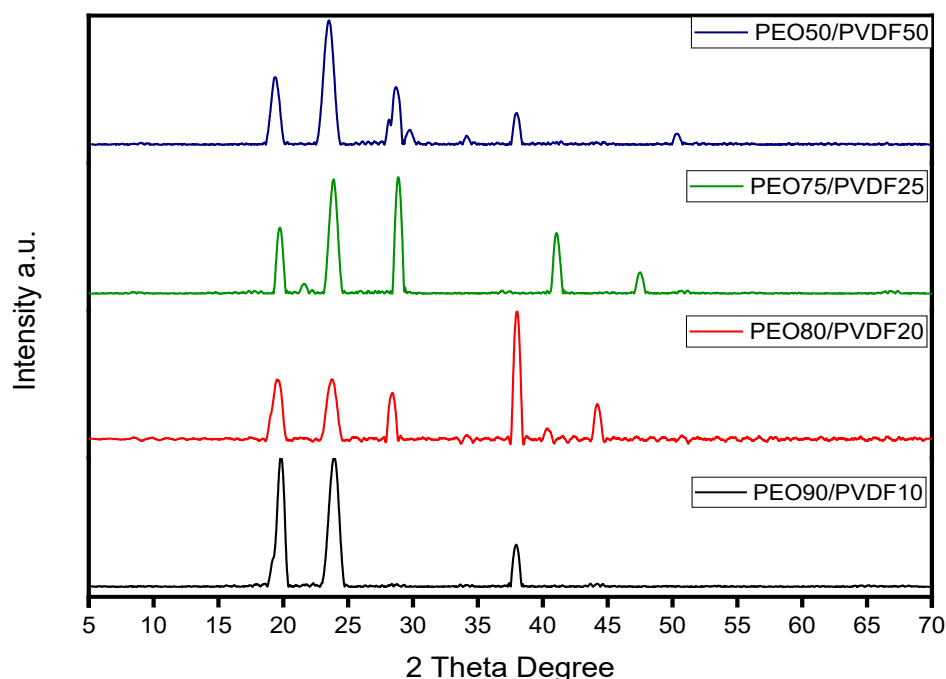


Figure 2: XRD curve of polymer blends of PEO90/PVDF10, PEO80/PVDF20, PEO75/PVDF25, PEO50/PVDF50

Table 1: Diffraction lines and their corresponding angles taken from diffraction plot

S/No	Samples	2θ	hkl	d-spacing (β) (Å)	FWHM	Crystalline Structure $a=\sqrt{(h^2+k^2+l^2)}d^2$	Lattice stress (ε)
1.	A	8.27	002	10.7	0.9	228.98	0.345387
		17.90	004	4.952	0.47	4	-0.06039
		19.822		4.476	0.425		0.056158
		21.61		4.109	0.73		0.94601
		23.86		3.726	0.92		-0.16995
		27.00		3.299	1.94		0.655273
		37.887		2.3728	0.175		0.004122
		44.066		2.0533	0.19		0.001989
2.	B	8.42		10.49	0.77		0.350377
		19.64		4.515	0.66		0.068833
		23.69		3.752	0.92		-0.20228
		28.345		3.1461	0.11		-0.77798
3.	C	37.995	202	2.3663	0.186	6.692907105	0.00693
		44.179	461	2.0484	0.13	14.9125771	0.003207
		8.53	002 & 110	10.36	0.71	20.72	0.369916
				4.499	0.27	10.06006983	0.031385
		21.64	041	4.102	0.60	16.91297928	0.845542
							-0.13527
		23.87	230 & 023	3.724	0.74	13.42707295	
		26.13		3.407	0.28		0.038117
							0.044841
		26.700	006	3.336	0.18	20.016	-0.10391
		28.885	200	3.0884	0.131	6.1768	-0.08477
		36.94		2.4312	0.85	4.8624	-0.08477
		41.131	220 & 028	2.1928	0.19	4.3856	-0.32495
		47.49		1.913	0.32		-0.43213
		50.70		1.799	0.47		0.025937
		66.91	645	1.3973	0.46	8.947085497	-0.22736
4.	D	8.33	110	10.61	0.71		0.291213
		19.40	&	4.572	0.63	6.465784407	0.044476
		21.37	220	4.155	0.98	12.465	0.763333
		23.51	023	3.781		11.95657183	-0.2528
					0.96		
		26.59	006	3.349		20.094	0.321283
					1.44		
		28.264	150	3.1549		4.461702368	14.99907
					0.31		
		28.761	200, 028 &	3.1015	0.147	6.935164832	-0.14803
		40.82	422	2.209	1.05	17.80952737	25.355925.3559
		50.44		1.8077	0.37		0.008092

SUMMARY

The research investigates the structural and molecular behavior of PEO/PVDF blends across a broad compositional range, addressing gaps in prior studies which often focus on limited ratios and qualitative FTIR analysis. Four blend ratios 90/10, 80/20, 75/25, and 50/50 were prepared by dissolving each polymer in DMF, mixing under controlled stirring, and casting into films.

FTIR results showed that key vibrational bands (C–O–C of PEO and C–F of PVDF) varied in intensity and position with changing composition, reflecting changes in intermolecular interactions and phase compatibility. Peaks associated with

PVDF β -phase (1145 and 1245 cm^{-1}) and α -phase (840 cm^{-1}) were identified, and their relative prominence increased with PVDF content.

XRD analysis confirmed that PEO rich blends maintained strong crystalline peaks, notably at $2\theta \approx 19.82^\circ$ (110) and 23.86° , while increasing PVDF content reduced PEO crystallinity and shifted intensity toward PVDF-associated reflections. Peak broadening in blends with higher PVDF indicated reduced crystalline domain sizes and possible structural strain.

Overall, the results highlight the correlation between blend ratio, molecular interactions, and crystalline structure. This dual FTIR–XRD approach bridges the gap between molecular level bonding and macrostructural arrangement, offering insights for tuning electrolyte properties in energy storage applications (Dirican et al., 2019; Yasar et al., 2024).

RECOMENDATION

- 1) Future studies should investigate extreme compositions beyond 90% PEO and below 50% PEO to fully map structure property relationships across the entire blend spectrum.
- 2) Incorporating systematic peak-shift measurements, peak area ratios, and deconvolution in FTIR can yield more precise miscibility and interaction strength data.
- 3) Varying solvent systems, casting temperatures, and drying rates could reveal their influence on PVDF phase content (α , β , γ) and amorphous crystalline balance.
- 4) To complement the structural analysis, ionic conductivity and electrochemical stability measurements should be integrated, especially for applications in solid polymer electrolytes.
- 5) Employing scanning electron microscopy (SEM) or atomic force microscopy (AFM) can provide direct visualization of phase distribution and interfacial morphology in PEO/PVDF blends.

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