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### ACCEPTED MANUSCRIPT

# Infrared spectroscopic studies on crystalline phase transition of PVDF and PVDF/Hyperbranched polyester blend ultrathin films

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#### ABSTRACT

In this study, as cast (AC, at 30 °C) and annealed (AN, at 130 °C, 3h) samples of polyvinylidene fluoride (PVDF) and PVDF/hyperbranched polyester (HBP) (90/10) blend ultrathin films were subjected to heating-cooling  $(30 \rightarrow 210 \rightarrow 30 \text{ °C})$  cycle, and studied for their changes in crystalline phase transition behavior using *in-situ* Fourier transform infrared-transmission spectroscopy (FTIR-TS) and grazing incident reflection absorption spectroscopy (FTIR-GIRAS) techniques. Factor analysis was employed to extract the pure crystalline and amorphous spectra as well as the percentage content of ferroelectric crystallinity for both the samples. Irrespective of the thermal treatment (AC or AN) and spectral measurement (FTIR-TS or GIRAS) techniques, neat PVDF sample exhibited irreversible phase transitions during heating-cooling cycle associated with the transformation from ordered  $\beta$ -crystalline (1276 cm<sup>-1</sup>) into disordered amorphous (1234 cm<sup>-1</sup>) form. Interestingly, annealed PVDF/HBP blend sample measured using FTIR-GIRAS exhibited reversible crystalline phase transition behavior similar to a 'dipole memory effect' even after heating to 210  $^{\circ}$ C (>T<sub>m</sub>) and then cooled to 30  $^{\circ}$ C. Compared to neat PVDF, higher ferroelectric crystallinity and reversible phase transition in PVDF/HBP blend may be attributed to (i) the existence of H-bonding between HBP (C=O and OH groups) and PVDF (-CH<sub>2</sub> and -CF<sub>2</sub>) and/or (ii) HBP acting as a nanoparticle in PVDF matrix.

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