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Effect of smectite incorporation on structure and ionic transport in PEO/Li_3PO_4 composite solid electrolytes

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Smectite clay minerals can modify polymer structure and ion transport through their layered structure, high surface area, and interlayer cation exchange capacity. This study evaluated how smectite type and loading affect the microstructure and ionic conductivity of poly(ethylene oxide) (PEO)/lithium phosphate (Li_3PO_4) composite solid electrolytes. PEO was used as the polymer matrix, and Li_3PO_4 supplied lithium ions. Four smectite samples (SWy-3, STx-1b, WRK, and organoclay) were incorporated to examine the effect of layer charge, exchangeable cations, and surface chemistry on composite structure and ion transport. To prepare the composite material, PEO and Li_3PO_4 were separately stirred in ethanol for 6 h. Smectite was dispersed via ultrasonication for 10 min and then added to the mixture, followed by further stirring at 40 °C for 12 h. The resulting mixture was cast onto a glass slide and dried in a vacuum oven at 40 °C to form the composite film. Structural changes were characterized by X-ray diffraction (XRD), and microstructural homogeneity was evaluated using field emission scanning electron microscopy (FE-SEM) and energy-dispersive X-ray spectroscopy (EDS) mapping. Electrochemical impedance spectroscopy (EIS) was conducted to determine bulk resistance and calculate room-temperature ionic conductivity. Smectite incorporation disrupted the crystalline domains of PEO. Among the tested clay minerals, 5 wt.% SWy-3 sodium smectite produced the most pronounced reduction in crystallinity and the highest ionic conductivity. Calcium-type smectites (WRK and STx-1b) also enhanced conductivity but to a lesser extent, reflecting differences in interlayer cation type and surface charge density. The organoclay-added sample showed the lowest improvement due to partial recrystallization of PEO and reduced interfacial compatibility. The results indicate that the enhancement of ionic conductivity correlates directly with the extent of PEO crystallinity suppression and the uniform dispersion of smectite layers within the polymer matrix.