STRUCTURE AND CRYSTALLIZATION KINETICS IN SODIUM MONTMORILLONITE-CONTAINING POLYPROPYLENE NANOCOMPOSITES: INTEGRATION OF SAXS AND DSC ANALYSES OF DISPERSION, FRACTAL STRUCTURE, AND INTERFACIAL PROPERTIES

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Sodium montmorillonite-based PP nanocomposites are used as model systems in the fields of polymer physics, surface phenomena, rheology, and nanomaterials. Research conducted on them seeks to answer fundamental questions such as the self-assembly of low-dimensional fillers in a polymer matrix, the influence of interfacial interactions on properties, and the dynamics of polymer chains under nanoconfinement. For this reason, they have become a classic example in both fundamental and applied aspects of materials science. That may provide high performance at low filler content. From a scientific perspective, they reveal fundamental mechanisms such as nanoconfinement, interfacial interactions, and self-assembly of fillers. They also serve as a superior alternative to conventional polypropylene in industries requiring lightweight properties, mechanical strength, thermal resistance, barrier performance, and flame retardancy—including automotive, packaging, electronics, construction, and agriculture. Thus, this material contributes to deepening scientific understanding to providing the lightweight resources needed by industry [1-4].

This study investigates the effect of sodium montmorillonite (Na-MMT) incorporation on the supramolecular structure, thermal behavior, and crystallization kinetics of syndiotactic polypropylene (sPP) using a multi-method SAXS and DSC approach. Three complementary analyses – low-q power-law, high-q broad-peak modeling, and one-dimensional correlation function – were applied to neat sPP and a series of **sPP + %Na^+ MMT** composites (0–7 wt% Na-MMT). For neat sPP, the low-q region ($q<0.046$ Å^{-1}) power-law exponent was p=4.07, indicating a diffuse interface (fuzzy boundary) of large morphological entities such as spherulites. Upon Na-MMT addition, pp decreased monotonically from 4.01 (2 %) to 3.78 (7 %), i.e. the surface fractal dimension $D_s=6-p$ increased from 2.00 to 2.22. This demonstrates that at low clay loadings the aggregate interfaces remain relatively smooth, while higher loadings lead to progressive roughening due to particle agglomeration.

For neat sPP, the broad-peak model applied to the high-q region (**$q>q_{peak}$**) revealed a mass-fractal structure with exponent p=1.48 (fractal dimension D_m=1.48), indicating an open, branched lamellar organization. The correlation function gave an average long period of 7.83 nm, a crystalline lamellar thickness of 2.76 nm, an amorphous layer thickness of 5.07 nm, an interphase thickness of 0.82 nm, and a local crystallinity of 35.2 %.

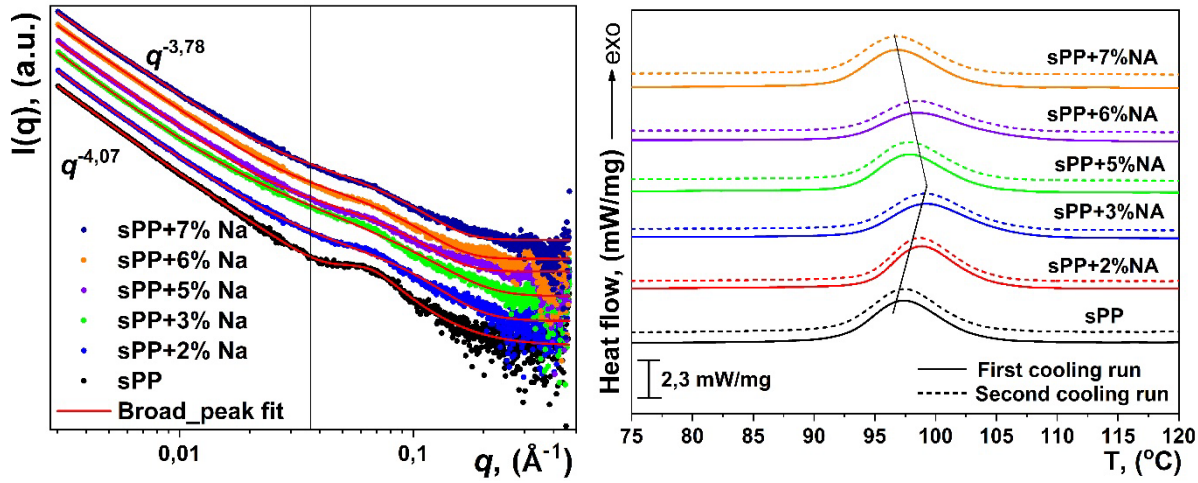

Figure 1. SAXS plots (left) and DSC thermograms (right) of PP+Na^+MMT nanocomposite films

From another hand results of DSC showed that the melting temperature (T_m) increased from 138.6 °C (0 %) to 140.9 °C (7 %). The crystallization temperature (T_c) first rose from 97.4 °C to a maximum of 99.3 °C at 3 % and then decreased to 96.9 °C at 7 %. Consequently, the supercooling degree ($\Delta T = T_m - T_c$) initially decreased from 41.2 °C to 39.5 °C (3 %) and later increased to 44.0 °C (7 %). This non-monotonic behavior reflects the dual role of Na-MMT: at low loadings (≤ 3 %) it acts as a heterogeneous nucleating agent, facilitating chain ordering and reducing supercooling; at higher loadings (≥ 5 %) particle agglomerates create a physical confinement effect that hinders chain mobility, increasing supercooling.

We may conclude that, the low-q power-law analysis for SAXS results, captures the progressive roughening of large-scale structures (spherulites/aggregates) with increasing Na-MMT content, in full agreement with the DSC-derived transition from nucleation-dominated to confinement-dominated crystallization. The high-q and correlation function results provide the detailed lamellar parameters of neat sPP, while the composites exhibit a clear shift from mass to surface fractal behavior on both size scales. This integrated SAXS/DSC approach offers a comprehensive framework for correlating hierarchical structural changes – from the lamellar to the aggregate level – with the crystallization kinetics and nucleation efficiency in sPP/clay nanocomposites, enabling tailored design of their mechanical and barrier properties.

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