



Morphology and Structure of Amino-fatty Acid Intercalated Montmorillonite

Larry Reyes (1,2) and Florentino Sumera (1,2)

(1) Materials Science and Engineering Program, College of Science, University of the Philippines-Diliman, Quezon City, Philippines (lqr03@yahoo.com), (2) Polymer Materials Laboratory, Institute of Chemistry, College of Science, University of the Philippines-Diliman, Quezon City, Philippines

Natural clays and its modified forms have been studied for their wide range of applications, including polymer-layered silicate, catalysts and adsorbents. For nanocomposite production, montmorillonite (MMT) clays are often modified with organic surfactants to favor its intermixing with the polymer matrix. In the present study, Na⁺-montmorillonite (Na⁺-MMT) was subjected to organo-modification with a protonated 12-aminolauric acid (12-ALA). The amount of amino fatty acid surfactants loaded was 25, 50, 100 and 200% the cation exchange capacity (CEC) of Na⁺-MMT (25CEC-AMMT, 50CEC-AMMT, 100CEC-AMMT and 200CEC-AMMT). Fatty acid-derived surfactants are an attractive resource of intercalating agents for clays due to their renewability and abundance.

X-ray diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR) were performed to determine the occurrence of intercalation of 12-ALA and their molecular structure in the clay's silicates. XRD analysis revealed that the interlayer spacing between the aluminosilicate layers increased from 1.25 nm to 1.82 nm with increasing ALA content. The amino fatty acid chains were considered to be in a flat monolayer structure at low surfactant loading, and a bilayered to a pseudotrayered structure at high surfactant loading. On the other hand, FTIR revealed that the alkyl chains adopt a gauche conformation, indicating their disordered state based on their CH₂ symmetric and asymmetric vibrations. Thermogravimetric analyses (TGA) allows the determination of the moisture and organic content in clays. Here, TGA revealed that the surfactant in the clay was thermally stable, with T_d ranging from 353°C to 417°C. The difference in the melting behavior of the pristine amino fatty acids and confined fatty acids in the interlayer galleries of the clay were evaluated by Differential Scanning Calorimetry (DSC). The melting temperatures (T_m) of the amino fatty acid in the clay were initially found to be higher than those of the free amino fatty acid, but decreased with increasing surfactant loading. This suggested that the amino fatty acid may be tethered to the clay structure via ionic interaction and/or ion-dipole attraction. Significant changes in the clay morphology, particle size and surface charge were observed after organo-modification. Scanning electron microscopy (SEM) revealed that the organo-clays have a disordered and flaky morphology, while the unmodified MMT appeared to be dispersed spherical grains. The effective (Z) diameter of Na⁺-MMT was found to be ~520 nm, but increased up to ~937 nm upon intercalation of 12-ALA. The zeta potential (ξ) of the clay materials, on the other hand, ranged from -33 mV for unmodified MMT to -16 mV (200CEC-AMMT clay).

The possible occupational hazards of working with nanoclays should also be explored. Presently, the MTT-dye reduction assay was performed to determine cell viability of mouse monocyte-macrophages (J774A.1) after direct exposure to the clays. The cytotoxicity of the clays exhibited a chemistry and dose dependent response, with unmodified Na⁺-MMT as the most cytotoxic while the organo-clays exhibited low toxicity. These results demonstrated the successful intercalation of the surfactant for the production of organophilic clay materials for a wide range of applications.