Thermal Degradation Studies of Trialkyl-Imidazolium Salts and Their Application in Nanocomposites

Walid H. Awad1, Jeffrey W. Gilman1, Marc Nyden1, Rick D. Davis2, Richard H. Harris Jr.1, Thomas E. Sutto1, John Callahan1, Paul C. Trulove3, Hugh C. Delong4

1Fire Research Division, Building and Fire Research Laboratory, National Institute of Standards and Technology, Gaithersburg MD.
2Naval Research Laboratory, Washington, DC
3Air Force Office of Scientific Research, Arlington, VA
4Chemistry Department, US Naval Academy, Annapolis, MD.

Polymer-layered silicates (PLS) nanocomposites have attracted a great deal of interest from researchers, since they exhibit dramatic improvements in mechanical, thermal, barrier, and flame-retardant properties compared to the neat or traditionally-filled resins. The efficiency of the montmorillonite (layered silicates) in improving the properties of the polymer materials is primarily determined by the degree of its dispersion in the polymer matrix. However, the hydrophilic nature of the montmorillonite surface impedes their homogenous dispersion in the organic polymer phase. To overcome this problem it is often necessary to make the surface organophilic prior to its use. This is done by ion exchange reactions involving the exchange of organic cationic surfactants for the interlayer cations. However, as elevated temperatures are necessary for nanocomposite processing, it is essential that the onset of the thermal decomposition of the organic modifier is below the polymer processing temperature. Therefore it is of prime importance to study the thermal stability of the organically modified layered silicate (OLS) and its implications on the nanocomposite processing and properties. In our efforts to pursue this objective we focused on imidazolium molten salts due to the well known higher thermostability of the delocalized imidazolium cation compared to the alkylammonium and pyridinium cations. Several trialkyl imidazolium Molten salts were prepared and characterized by thermogravimetric analysis to find out the effect of different parameters such as the counter ion, the chain length and structural isomerism on the thermal stability of the imidazolium salts. Imidazolium treated montmorillonite were prepared by ion exchange of the imidazolium salts with Na-montmorillonite. These imidazolium treated clays were then characterized by TGA-FTIR, XRD and mass spectra and compared to the conventional quaternary alkyl ammonium montmorillonite.

Results indicate that the counter ion has a potential effect on the thermal stability of the imidazolium salts, and that imidazolium salts with BF4 and PF6 anions are thermally more stable than the halide salts (fig. 1). A relationship was observed between the chain length and the thermal stability of the imidazolium salts, indicating that the thermal stability decreases as the chain length increases from propyl, butyl, decyl, hexadecyl, octadecyl to eicosyl. The results also show that the imidazolium treated montmorillonite has greater thermal stability as compared to the imidazolium salt (fig. 2). Analysis of decomposition products by FTIR provides an insight about the decomposition products which are basically water, carbon dioxide and hydrocarbon.

References

Figure 1

![Figure 1](image1)

Figure 2

![Figure 2](image2)