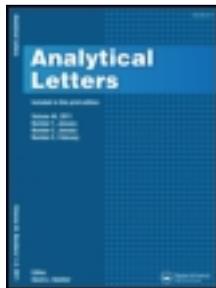


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Imaging Microscopy

DETERMINATION OF CHEMICAL HOMOGENEITY OF FIRE RETARDANT POLYMERIC NANOCOMPOSITE MATERIALS BY NEAR-INFRARED MULTISPECTRAL IMAGING MICROSCOPY

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Polymer nanocomposites containing layered double hydroxide (LDH) additives offer great potential for improving polymer physical properties. Of particular interest is the possibility of improving the fire retardancy and thermal stability of polymers using low loadings of this emerging class of nano-additives. Understanding the relationship between the quality of additive dispersion in the polymer matrix (i.e., chemical homogeneity) and selected flammability properties is a key question for optimizing LDHs for use in fire retardant formulations. We have demonstrated, for the first time, that the near infrared multispectral imaging (NIR-MSI) microscope can be successfully used to characterize the chemical homogeneity of a model system containing a magnesium aluminum hydroxide LDH modified with interlayer undecenoate anions mixed with poly(ethylene). The NIR-MSI is suited for this task because it can simultaneously record spectral and spatial information of a sample with high sensitivity (single pixel resolution) and high spatial resolution (~0.9 $\mu\text{m}/\text{pixel}$). At 20% added, LDH was found to be distributed inhomogeneously in a poly(ethylene) nanocomposite sample on the micron scale.

Keywords: Chemical homogeneity; Fire retardant; Imaging; Nanocomposite materials; Near-infrared; Polymer; Spectroscopy

Polymer nanocomposites have been widely investigated and shown to result in significant improvement in many polymer physical properties, including reduction in the flammability of synthetic polymers (Kashiwagi et al. 1998; Manzi-Nshuti et al. 2009). For example, polymer nanocomposites that utilize cationic clays

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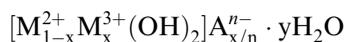
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improve selected flammability properties of polymers at much lower loadings compared to conventional fire retardants (FR) such as magnesium hydroxide (Gilman 1999; Costa, Wagenknecht, and Heinrich 2007). One of the most studied classes of polymer/clay nanocomposites has been material that incorporates organically-modified montmorillonite (MMT) clay. Improvement in the peak heat release rates, as measured by cone calorimetry, has been correlated with the quality of the dispersion of the clay in the polymer; it is relatively easy to achieve an exfoliated or intercalated system resulting in good dispersion of the MMT in the polymer matrix.

Anionic clays are starting to receive recognition as potential FR additives, and in this class, layered double hydroxides (LDHs) are the most studied (Costa, Wagenknecht, and Heinrich 2007; Manzi-Nshuti, Hossenlopp, and Wilkie 2008; Nyambo, Wang, and Wilkie 2009; Manzi-Nshuti et al. 2009). The ability to systematically alter the composition of the metal hydroxide layer, as well as the ease of modifying the interlayer organic anions makes LDHs and related anionic clays attractive as potential polymer additives. The LDH layers have a brucite $\{Mg(OH)_6\}$ type structure where magnesium ions are surrounded by six hydroxide ions in an approximately octahedral geometry (Evans 2006). The general formula of LDH can be represented as



where M^{2+} is a divalent cation, M^{3+} is a trivalent cation and A^{n-} is an exchangeable charge compensating anion of charge n and controls the interlayer spacing (Manzi-Nshuti, Hossenlopp, and Wilkie 2008). Although LDHs are difficult to exfoliate because of the high charge density of the layers, they have been shown to significantly improve the flammability of polymers (Albiston et al. 1996; Manzi-Nshuti, Hossenlopp, and Wilkie 2008). Studies with MMT have shown that there is correlation between the dispersion of the clay particles in the polymer and selected FR properties. Good dispersion in the polymer matrix results in greater improvements in flammability and thermal stability of polymers as compared to filler which form aggregates (Zhang and Wilkie 2003; Lim, Choi, and Jhon 2003). However, in LDHs, dispersion does not seem to play as significant a role, poorly dispersed materials do, in some cases, results in significant improvements in flammability properties (Nyambo, Wang, and Wilkie 2009). Understanding the relationship between dispersion and polymer nanocomposite thermal/fire properties is thus critical for designing the next generation of effective, environmentally benign FR formulations.

Typically a combination of TEM images and x-ray diffraction data have been used to evaluate the dispersion of fillers in polymers (Manzi-Nshuti et al. 2009), but other spectroscopy methods, which are more readily available, have also been used to evaluate the homogeneity of the fillers in polymers (Marosfői et al. 2006). The need to evaluate heterogeneity on a range of length scales is also important for macroscopic samples, as has been demonstrated using confocal microscopy (Kashiwagi et al. 2007). It is important to note that effective polymer FR formulations will most likely involve more than one additive in order to obtain the desired combination of physical and chemical stability. It is, thus, of particular importance that a new

technique be developed for the determination of chemical homogeneity of compounds added to polymers to improve their fire retardant properties. The near-infrared (NIR) multispectral imaging microscope described herein offers a solution to this problem.

A multispectral imaging spectrometer is an instrument that can simultaneously record spectral and spatial information about a sample (Morris 1993; Tran 2000; 2001; 2003; 2005). Unlike conventional imaging techniques, which rely on recording a single image using either single or multiwavelength light for illumination, the multispectral imaging technique records a series of several thousand images, each image at a specific wavelength. That is, it measures absorption spectra of a sample not at a single position, as is the case for a conventional spectrophotometer, but simultaneously at many different positions within a sample (by using a focal plane array detector rather than a single channel detector) (Morris 1993; Tran 2000; 2001; 2003; 2005). Chemical composition and structure at different positions within a sample can be elucidated from such images. We have recently developed a novel near-infrared multispectral imaging (NIR-MSI) microscope that employs an acousto-optic tunable filter (AOTF) for rapid spectral tuning and a microscope for higher spatial resolution. The high sensitivity (single pixel resolution), fast temporal (milliseconds), and high spatial resolution ($\sim\mu\text{m}$) of this imaging microscope, make it possible for us to use this multispectral imaging microscope for studies and measurements that, to date, have not been possible using existing techniques (Fischer and Tran 1999a; Fischer and Tran 1999b; Tran 2000; 2001; 2003; 2005). These include photo induced changes of a single unit micron-size cell in temperature-sensitive liquid crystals as a function of time and wavelength, and the determination of molecular state and distribution of fullerenes entrapped in sol-gel samples (Fischer and Tran 1999a; Fischer and Tran 1999b; Khait, Smirnov, and Tran 2001; Tran 2000; 2001; 2003; 2005; Tran, Grishko, and Challa 2008).

The information presented in the present manuscript is indeed provocative and clearly demonstrates that it is possible to use the NIR-MSI microscope to determine concentration distribution of compound added to polymer to improve its fire retardant properties. Such considerations prompted us to initiate this study, which aims to: (1) synthesize a novel polymeric nanocomposite material which contains polyethylene substrate with added magnesium-aluminum layered double hydroxide (LDH) intercalated by undecenoate to improve its fire retardancy; and (2) use NIR spectrophotometric method to characterize the polymeric nanocomposite material and the NIR-MSI microscope to determine chemical homogeneity of the additive added LDH layer. The results of our initial investigation are reported herein.

EXPERIMENTAL

Materials

The $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ ($\geq 98\%$), $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ ($\geq 98\%$) and sodium hydroxide (flakes, 98%) were all obtained from Aldrich Chemical Co; 10-undecenoic acid (99%) and PE (LDPE) BPD 8063 were obtained from TCI and BP petrochemicals, respectively. All chemicals were used without further purification.

Preparation

The magnesium aluminum 10-undecenoate layered double hydroxide (MgAl-C11) was prepared according to literature procedure with minor modifications (Manzi-Nshuti, Wang, Hossenlopp, and Wilkie 2008). In a typical experiment, a solution of $\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (0.02 mol) and $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$ (0.01 mol) was prepared in deionized and decarbonated water (50 ml) and was drop wise added to a solution of 10-undecenoic acid (0.02 mol) and NaOH (0.02 mol) in 100 ml of deionized and decarbonated water under nitrogen atmosphere. The pH of the mixture was maintained at 10.0 by the addition of 1.0 M NaOH solution. The resulting slurry was aged at 60°C for 24 hours and washed several times with deionized, decarbonated water before drying at 50°C in vacuum.

The polymer-LDH composite was prepared by melt blending for 10 minutes in a Brabender Plasticorder operated at 60 rpm at a temperature of 130°C. The percent loading of the LDH (MgAl-C11) in the polymer was 20, a reference sample of pure PE was obtained using the same procedure but without the MgAl-C11. Samples for NIR experiments were pressed into thin films on a Carver Laboratory hydraulic press with the temperatures of both top and bottom heating plates being 300°F.

Characterization

Fourier-transform infrared (FTIR) spectroscopy of the layered materials and the composites were obtained on a Perkin Elmer spectrum 100 FT-IR spectrometer operated at a 2 cm^{-1} resolution in the $4000\text{--}650\text{ cm}^{-1}$ spectral range, and the number of scan were 8. The IR spectra were recorded using a single reflection ATR accessory with a ZnSe prism (PIKE MIRacle™, from PIKE technology). Powder X-ray diffraction (PXRD) measurements were recorded on a Rigaku Miniflex II diffractometer using $\text{Cu K}\alpha$ ($\lambda = 1.54\text{ \AA}$) radiation source operated at 30 kV and 15 mA. The patterns were recorded in the 2θ range of $2.0^\circ\text{--}45.0^\circ$ for powders and $2.0^\circ\text{--}10^\circ$ for composites, data acquisition was performed using a step size of 0.083° per second. Powder samples were mounted on glass sample holders and polymer samples were compression molded into thin rectangular plaques and mounted on aluminum sample holders.

The NIR spectrophotometric measurements were performed using a home-built NIR spectrophotometer that uses an acousto-optic tunable filter (AOTF) as the wavelength selection device. The detailed description of this instrument has been given before in our previous publications (Duri, Molthen, and Tran, 2009; Tran and Kong 2000; Tran, Oliveira, and Grishko 2004). Each spectrum is an average of 2000 spectra taken at 2 nm intervals measured from 1300 nm up to 2200 nm.

The acousto-optic tunable filter (AOTF) based NIR multispectral imaging microscope used in this work is similar to those used in previous studies (Khait, Smirnov, and Tran 2001, Tran 2003; 2005; Tran, Grishko, and Challa 2008; Mejac et al. 2009) except for the replacement of the focusing microscopic lenses in the microscope with a pair of 0.58 N.A. A 15X reflecting Cassegrains to avoid possible aberrations in the NIR region. Images recorded by the NIR camera were grabbed and transferred to a PC by a frame grabber. A software written in C++ language was used to control the imaging microscope as well as to facilitate the frame

grabbing, saving and processing images. The 3-D images were calculated from recorded images by use of an image processing program. Recorded multispectral images were analyzed using MatLab. The spatial resolution of this NIR-MSI microscope was determined to be $(0.93 \pm 03) \mu\text{m}/\text{pixel}$ (Khait, Smirnov, and Tran 2001; Tran 2003; 2005; Tran, Grishko, and Challa 2008).

RESULTS AND DISCUSSION

The FTIR spectrum (spectrum not shown) of MgAl-C11 indicates that it is free of contaminants since bands due to carbonate and nitrate impurity at 1365 cm^{-1} and 1385 cm^{-1} are absent. Figure 1 shows the FTIR spectra of pure PE and of PE doped with 20% MgAl-C11. As illustrated, both samples exhibit several bands. The bands at 2917 cm^{-1} and 2849 cm^{-1} can be assigned to C-H stretching of methyl groups, the 1464 cm^{-1} -band is due to deformations of methyl or methylene groups, and the rocking motion of $(\text{CH}_2)_n$ groups is probably responsible for the band at 720 cm^{-1} (Lomakin et al. 2008). As expected, in addition to the aforementioned peaks, the PE sample doped with MgAl-C11 also exhibits a peak at 1567 cm^{-1} , which can be attributed to the carboxyl stretching mode of 10-undecenoate. The broad peak at about 3500 cm^{-1} may be due to moisture trapped during pressing the polymers into thin films.

Figure 2 shows the PXRD pattern of MgAl-C11. Intense and equally spaced $00l$ reflections ($l=3$ to 9) are observed indicating that the material is layered and possess high range ordering at least to the third order in the c direction. The PXRD profile indicates an interlayer distance of $2.7 \pm 0.1 \text{ nm}$ and matches previously reported value (Manzi-Nshuti, Wang, Hossenlopp, and Wilkie 2008). The XRD

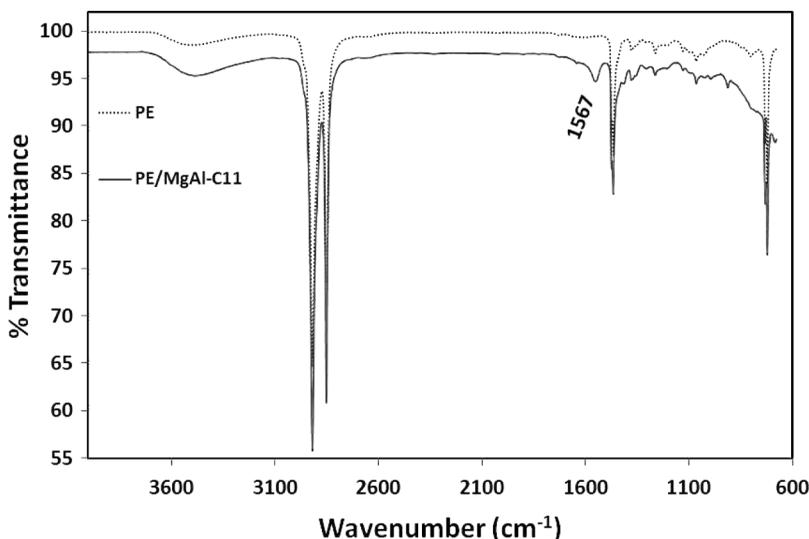


Figure 1. FTIR spectra of samples of pure PE (dashed red line) and PE doped with 20% MgAl-C11 (solid green line).

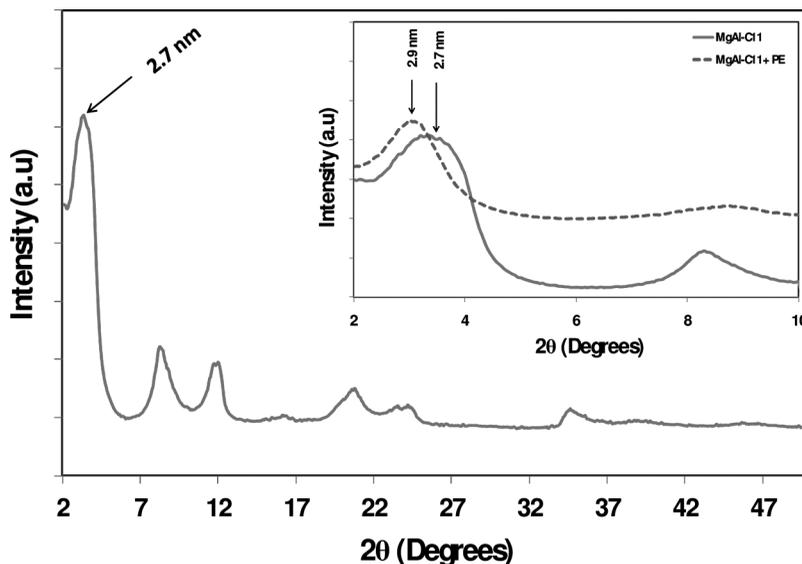


Figure 2. XRD profile of MgAl-C11 layered double hydroxide (red line) and PE doped with 20% MgAl-C11 layered double hydroxide (green line).

profile confirms the results from FTIR that there was no contamination from nitrate and carbonate since the expected reflections from nitrate and carbonate-containing LDHs which are typically observed at approximately 0.89 nm and 0.76 nm, respectively, are absent (Albiston et al. 1996). As illustrated in the insert figure, when MgAl-C11 was compounded with PE during melt blending, there is a slight shift of the first peak to lower 2 theta values with the interlayer distance increasing from 2.7 ± 0.1 nm for MgAl-C11 to 2.9 ± 0.1 nm for the composite. This swelling is consistent with some intercalation of the polymer into the LDH gallery.

The NIR absorption spectrum of a polyethylene (PE) film is shown in Fig. 3. As illustrated, overtones and combination transitions of C-H groups can be seen at 1725 nm, 1775 nm, 1800 nm, and a broad band at around 1400 nm. Also, shown in the figure are spectra of PE films to which different amounts (5, 10, and 20%wt) of MgAl-C11 have been added. In addition to C-H bands, the LDH-PE films also exhibit additional broad band at around 1950 nm. This band can be attributed to combined overtone and combination transitions of O-H group (of magnesium-aluminum hydroxide) and COO^- group (of undecenoate) of the added LDH layer. As expected, absorbance of this 1950 nm band was found to be linearly related to concentration of added LDH (insert of Fig. 1 shows linear relationship ($R^2 = 0.9972$) between absorbance at 1950 nm vs. concentration of LDH added). This result clearly indicates that the concentration of added LDH can be determined by using the NIR spectrophotometer to measure absorption of the 1950 nm band.

As described previously, a multispectral imaging microscope is an instrument that can simultaneously record spectral and spatial information about a sample. That is, it measures absorption spectra of a sample not at a single position, as is the case for a NIR spectrophotometer, but simultaneously at many different

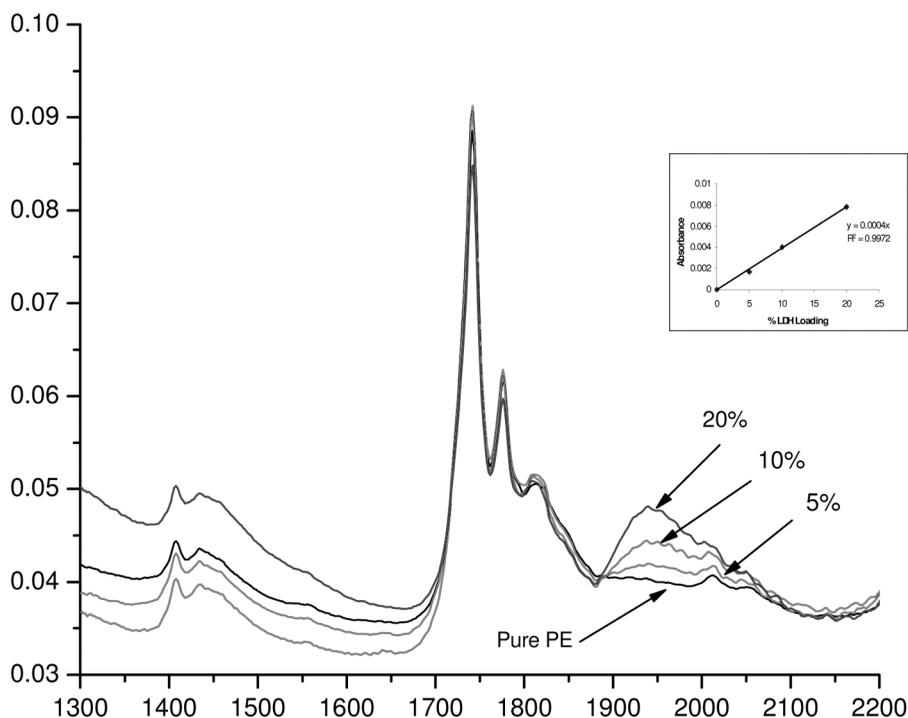
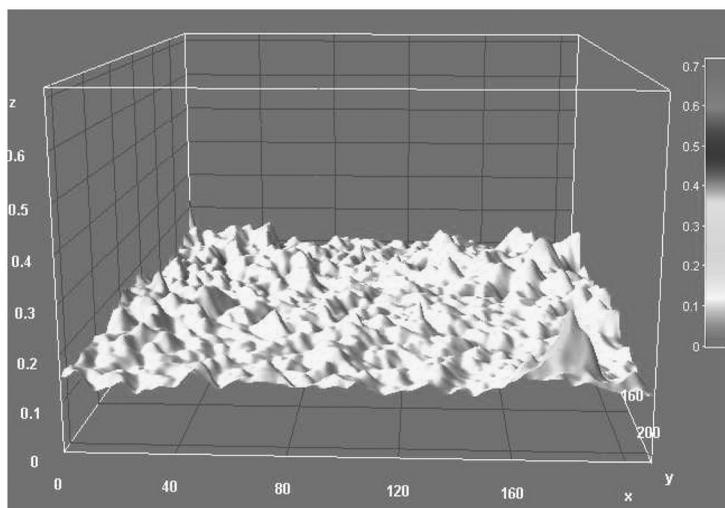


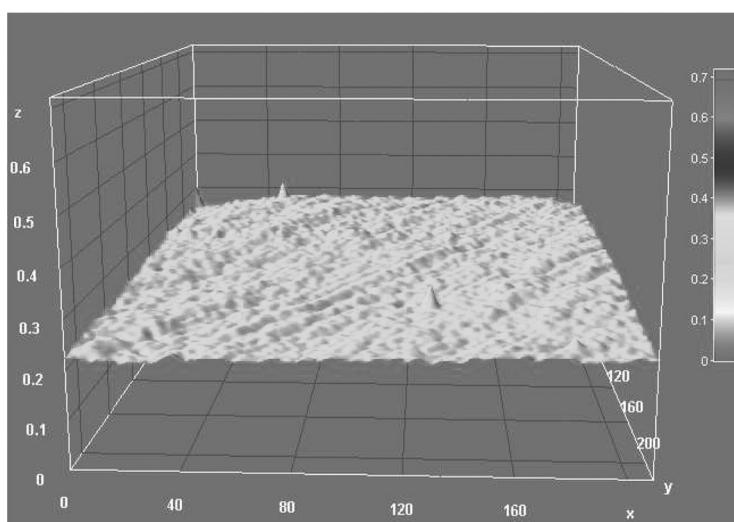
Figure 3. NIR absorption spectra of PE polymer without (black) and with 5% (red), 10% (green) and 20% (blue) added LDH. Insert is plot of absorbance at 1950 nm as a function of concentration of added LDH. See text for detailed information.

positions within a sample. The chemical composition at different positions within a sample can be elucidated from such images. As a consequence, by using NIR-MSI microscope to measure absorbance at 1950 nm at many different positions over an entire sample, it is possible to determine if added LDH is distributed homogeneously or inhomogeneously in the PE sample. Accordingly, the NIR-MSI microscope was used to record multispectral images of a sample of PE with 20% of added LDH. From recorded images, 3-D plot of absorbance at 1950 nm of the sample as a function of its dimension can be obtained. The result is shown in Figure 4a where units for x, y, and z axes of the plot are pixel, pixel and absorbance at 1950 nm, respectively, with one, corresponds to 0.93 μm , and the color of the plot denotes different absorbance at 1950 nm with scale shown in upper right-hand.

For comparison, the 3-D plot of a sample of pure PE polymer sample taken at the absorption of the C–H group at 1750 nm is also shown in Figure 4b. As described previously, absorbance at 1950 nm of doped PE sample is related to concentration of added LDH, whereas absorbance at 1750 nm of pure PE sample corresponds to its concentration or rather its thickness. As illustrated, the 3-D image shown in Fig. 2b is not smooth but has some contours which indicate that the PE film does not have the same microscopic thickness over its entire surface. This is hardly surprising considering the fact that the film was prepared using a mechanical



(a)



(b)

Figure 4. 3-D absorption images taken at (a) 1950 nm of a polyethylene sample doped with 20% LDH, and (b) at 1750 nm of pure PE film. Units for x, y, and z axes are pixel, pixel and absorbance at 1950 nm and 1750 nm, respectively (one pixel corresponds to 0.93 μm).

hot plate. Substantial differences in the absorbance at 1950 nm were observed for the PE sample doped with 20% LDH. Because the absorbance differences observed here is much larger than those observed for PE sample at 1750 nm, they cannot be account solely due to the difference in the thickness of the sample. Rather, they are due mainly to the differences in distribution of added LDH compound. That is added LDH is not homogeneously distributed over the entire the PE polymer film.

In summary, we have demonstrated for the first time that the NIR-MSI microscope can be successfully used to determine microscopic concentration distribution of LDH compound added to a polymer film. Use of LDHs with nonpolar polymers such as poly(ethylene) is known to be a challenging system for obtaining good nano-dispersion (Nyambo, Wang, and Wilkie 2009). Having the ability to monitor dispersion (chemical homogeneity) on the micron length scale will provide an excellent complement to other methods of characterization. The NIR-MSI offers the possibility of examining more chemically complex systems, such as those containing mixtures of additives. Future work will include measuring flammability properties in order to better understand the relationship between chemical homogeneity and nanocomposite performance as fire retardants.

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