APPLICATION OF CURVE FIT AND DECONVOLUTION TO POLYMER ANALYSIS

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INTRODUCTION

A great wealth of information about a material is found in its infrared spectrum. For many years infrared was used not just for identifying compounds but also for obtaining semi-quantitative information about structural units. In more recent years other instrumental techniques, such as NMR, have been developed and have superseded infrared spectroscopy in the quantitative determination of structural information. Now, however, because of the availability of high signal-to-noise infrared spectrometers, and the computers associated with the spectrometers, structural information, which could not be accessed previously, can be extracted from the infrared spectrum. Techniques such as deconvolution and curve fit analysis provided by the computer, allow more structural information to be made visible in the infrared spectrum than was once thought possible. In the deconvolution process broad complex bands can be mathematically narrowed to yield an improvement in the observation of the overlapped component bands. The results

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obtained are often similar to those obtained from samples analyzed at low temperatures.

This paper will deal with two examples of deconvolution applied to polymer analysis with supporting data from a curve fit analysis. The first example deals with ethylene propylene copolymers and shows the enhancement of the infrared spectrum to give information about the length of the methylene sequences in the polymer. In the second example the technique is used to provide information about the crystallinity of polypropylene samples.

EXPERIMENTAL

The ethylene propylene copolymers used in this study were obtained from ICI, England. Carbon-13 NMR (C-13) spectra were obtained by Dr. Alan Bunn of ICI Research. Results of the C-13 analyses are given in Table 1 for four of the samples. These represent the number of respective sequences of CH\textsubscript{2} groups normalized to 100 carbon atoms.

<table>
<thead>
<tr>
<th>Sample</th>
<th>(CH\textsubscript{2})\textsubscript{3}</th>
<th>(CH\textsubscript{2})\textsubscript{5}</th>
<th>(CH\textsubscript{2})\textsubscript{11&gt;7}</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>100</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
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<td>9</td>
<td>0</td>
</tr>
<tr>
<td>3</td>
<td>82</td>
<td>11</td>
<td>6</td>
</tr>
<tr>
<td>4</td>
<td>78</td>
<td>8</td>
<td>14</td>
</tr>
</tbody>
</table>

Infrared spectra were obtained for thin self supporting films using the Perkin-Elmer Model 1800 Fourier Transform Spectrophotometer. Relevant operating conditions were 4 double beam scans at 2 cm\(^{-1}\) resolution, weak Beer-Norton apodization, and double sided interferograms. These conditions yield a peak to peak signal-to-noise in excess of 1500 which satisfies the requirements for the amount of resolution enhancement to be done. Spectra were stored on disk at 1 cm\(^{-1}\) data interval for the full range 4000 to 450 cm\(^{-1}\).

In order to improve the observability of the overlapping bands in the CH\textsubscript{2} rocking region near 730 cm\(^{-1}\), mathematical resolution enhancement was performed. The process is identical to the Fourier self-deconvolution [1-3] except that the mathematical operations are performed in the spectral domain rather than in the Fourier domain.

The Fourier self-deconvolution technique involves the enhancement of the high resolution components of a spectrum in the Fourier, or time domain by multiplication with an exponential function. The degree of the enhancement is related to the power...