Rapid Identification of Nylons by Temperature-rising Direct Analysis in Real Time Mass Spectrometry (TR-DART-MS)

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Introduction

Nylon is well-known polymer material as an aliphatic polyamide, mainly used in clothing. There are several types of nylon such as nylon-6, nylon-6,6, nylon-6,10 and nylon-6,12. Recently, the necessity of quality control for foods and daily commodities has been discussed. Some problems (e.g. contamination) may be caused by clothing fragment. So, identification of contaminants is important. Though many analysis methods are used for identification, e.g. FT-IR, NMR, DSC, TG-DTA and pyrolysis-GC/MS, it is difficult to distinguish the nylon types easily due to their structural complexity.

DART-MS is one of the powerful methods for the rapid analysis of mixtures. However, this method is not suitable for polymer analysis since the polymer is difficult to volatilize. To overcome this weak point, we developed the Temperature-rising device combined with DART-MS system (TR-DART-MS). The nylon types were determined rapidly by their pyrolysis profiles.

Methods

Four kinds of nylon were used as analysis samples. The nylon samples were cut into small pieces (0.5 mm x 0.5 mm) using a razor, and placed in crucibles. The samples were then heated linearly from room temperature to 600℃ in 6 min. The vapor phase obtained from heating of samples was introduced in front of DART orifice, and the components in the vapor phase were ionized. DART-MS (IonSense, Inc., USA) ion source and microTOF-QIII (Bruker Daltonics, Inc., Germany) mass spectrometer were used. Helium gas flow was set to 3 L/min at 400℃.

As a results of analysis, the pyrolysis products of samples (monomers and dimers) were observed over 200℃. Nyons-6 and nylon-6,6 were analyzed by TR-DART-MS. As a results of analysis, the pyrolysis products of samples (monomers and dimers) were observed over 200℃. The distinction between nylon-6,10 and nylon-6,12 by focusing on their pyrolysis products of nylon (monomers, dimers, trimers and so on). The distinction between nylon-6 and nylon-6,6

as a results of analysis, the pyrolysis products of samples (monomers and multimers) were observed over 200℃. As a results of analysis, the pyrolysis products of samples (monomers and dimers) were observed over 200℃.

Table 1: Nylon samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Elemental composition</th>
<th>Theoretical mono isotopic mass [M+H]+</th>
</tr>
</thead>
<tbody>
<tr>
<td>nylon-6</td>
<td>C_{11}H_{18}N_{0.5}O_{0.5}</td>
<td>m/z 114.0913 m/z 227.1754</td>
</tr>
<tr>
<td>nylon-6,6</td>
<td>C_{11}H_{22}N_{0.5}O_{0.5}</td>
<td>m/z 227.1754 m/z 453.3435</td>
</tr>
<tr>
<td>nylon-6,10</td>
<td>C_{12}H_{22}N_{0.5}O_{0.5}</td>
<td>m/z 340.2680 m/z 681.5360</td>
</tr>
<tr>
<td>nylon-6,12</td>
<td>C_{12}H_{22}N_{0.5}O_{0.5}</td>
<td>m/z 565.4617 m/z 1130.9234</td>
</tr>
</tbody>
</table>

Fig.2 TR-DART-MS system

Fig.3 Analysis of condition of temperature-rising system.

(a) nylon-6

As a results of analysis, the pyrolysis products of samples (monomers and dimers) were observed over 200℃.

(b) nylon-6,6

As a results of analysis, the pyrolysis products of samples (monomers and dimers) were observed over 200℃.

(c) nylon-6,10

As a results of analysis, the pyrolysis products of samples (monomers and dimers) were observed over 200℃.

(d) nylon-6,12

As a results of analysis, the pyrolysis products of samples (monomers and dimers) were observed over 200℃.

Conclusion

DART-MS is the powerful methods for the rapid analysis of mixtures. In order to identify the nyons, we used a temperature-rising device combined with DART-MS. We could distinguish between those nyons by focusing on their pyrolysis products of nylon (monomers, dimers, trimers and so on).