SII NanoTechnology Inc.

TG/DTA AS PROBLEM-SOLVING TOOL: ANALYSIS OF MULTI-COMPONENT MATERIALS USING AUTO STEPWISE TGA

Problem

A customer in a thermal analysis laboratory for a major tobacco company is having difficulty in the characterization of the tobaccos since the samples exhibit multiple and severely overlapping weight loss events by TGA (thermogravimetric analysis). The customer desires an easy means of separating or resolving the successive mass loss events to better help in the characterization studies of the tobacco products. This is necessary in order to aid in the optimization of the processing parameters utilized in the production of the tobacco end products, particularly cigarettes.

Solution

The Seiko TG/DTA6000 instrument provides high sensitivity and the ability to perform TGA experiments in the auto stepwise isothermal mode. The use of the auto stepwise mode gives the highest possible degree of resolution between successive decomposition events, such as those associated with complex, multi-component materials, including tobacco. With this technique, the sample is heated at a constant rate until the instrument detects amount of weight loss due to the evolution of volatiles or a decomposition product. The TGA will then automatically hold the sample under isothermal conditions and permits the given decomposition event to take place until the rate

of weight loss is insignificant or less than the isothermal exit threshold value. The instrument will automatically resume heating the sample at a constant rate until the next weight loss event is detected. Using this approach, severely overlapping decomposition events can be fully separated or resolved.

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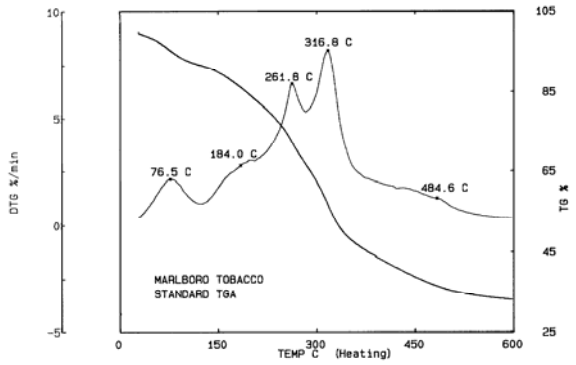

Figure 1

Displayed in Figure 1 are the TGA results obtained by heating a sample of Marlboro tobacco at a rate of 20°C/min. The plot shows the % mass along with the derivative (d%/dt) as a function of sample temperature. The derivative trace shows the occurrence of numerous, overlapping decomposition events associated with the tobacco lamina. It is difficult to obtain quantitative compositional results from the % mass signal from standard TGA because of the overlapping.

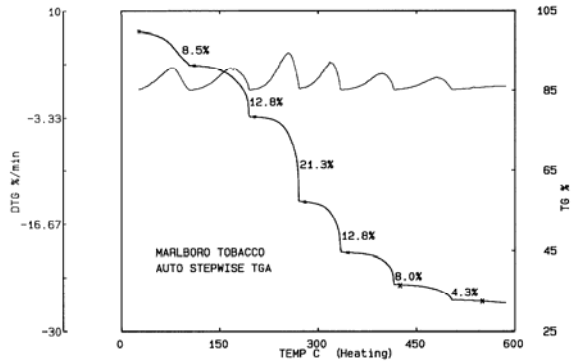

Figure 2

The Marlboro tobacco sample was analyzed using the auto stepwise mode of analysis featured with the Seiko TG/DTA. The sample was heated at a rate of 40°C/min under a nitrogen purge (200 mL/min) and with an auto stepwise entrance threshold value of 225 µg/min and an exit threshold value of 10 µg/min. Shown in Figure 2 are the TGA results obtained for the Marlboro tobacco sample using the auto stepwise mode of operation. Excellent resolution of the

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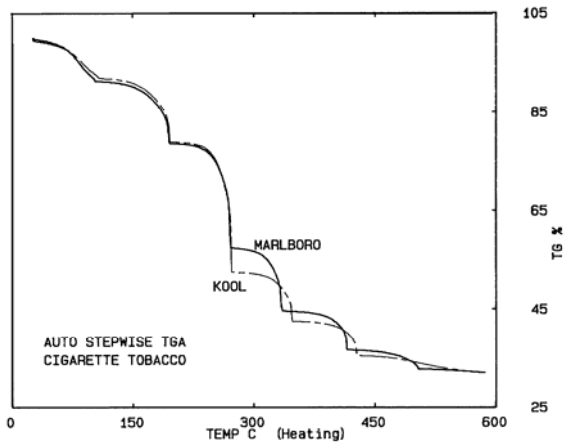

Figure 3

successive thermal decomposition events is obtained with this approach. The first weight loss at 100°C corresponds to the evolution of moisture, and the auto stepwise approach permits an accurate assessment of the moisture content in the tobacco. The complex nature of the tobacco lamina is clearly evident in the auto stepwise TGA results.

A direct comparison of the auto stepwise TGA data for Marlboro and Kool tobacco samples is displayed in Figure 3. The two tobacco samples yield essentially equivalent decomposition profiles up to 280°C. At 280°C and above, the Kool tobacco yields a significantly different thermal decomposition response due to its different composition and formulation.

Summary

The Seiko TG/DTA6000 provides the auto stepwise isothermal mode of analysis which gives the highest possible resolution between successive decomposition events. Complex, multi-component materials are better characterized using the auto stepwise TGA mode.

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