APPLICATION NOTE

Batteries – Thermogravimetry – FT-IR

Identification of Separator Material by Means of TGA-FT-IR

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Introduction

Separators play a crucial role in lithium-ion batteries as they physically separate the anode and cathode while allowing the passage of lithium ions between the electrodes. Different materials and technologies are used for separators to meet different safety, performance and cost requirements. One commonly used group of separators in lithium-ion batteries are polyolefin separators, as they are chemically resistant to electrolytes, easy to produce and relatively cost effective.

In the case of competitive studies, the characterization and identification of separators can be essential in ensuring quality and improving the performance of a battery.

Two different separator foils were investigated by the TGA-FT-IR technique to determine the decomposition behavior and to identify the composition.

Measurement and Discussion

The measurement conditions are detailed in table 1.

Table 1 Measurement parameters

Instrument	NETZSCH TG *Libra*® coupled to the Bruker FT-IR *INVENIO*
Temperature program	RT to 850°C
Heating rate	10 K/min
Purge gas	Nitrogen, 40 ml/min
Crucibles	Al_2O_3, 85 µl, open

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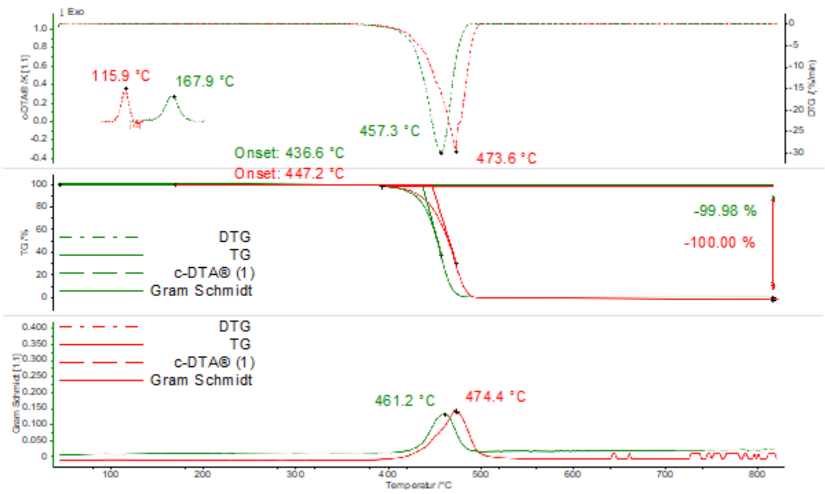
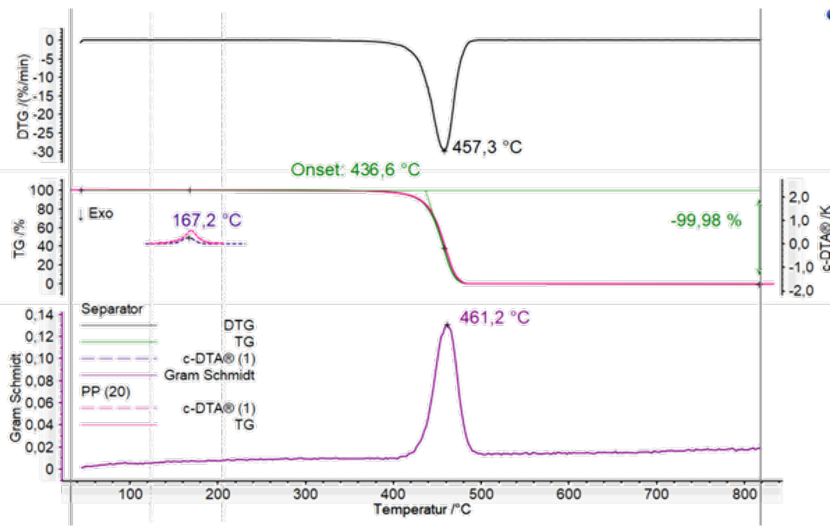
Figure 1 shows a comparison of the TGA curves (sample A: green; sample B: red) of two separator foils. Both samples were heated to 850°C under an inert atmosphere, resulting in complete pyrolysis. As a result, no pyrolytic carbon or ash content could be identified. However, a slight deviation in the decomposition onset temperature (437°C for sample A vs. 447°C for sample B) was observed. It is thus likely that two different materials were used for these separator foils. With the help of the *c-DTA*® function, the melting points of these two samples could also be determined. Again, a significant difference of 116°C vs. 168°C was detected.

The melting of a separator foil is an important safety feature for batteries. In modern batteries, many separators have a so-called "shutdown function". This means that when overheated, the separator melts or closes its pores, stopping the current flow and thus protecting the battery before dangerous thermal runaway occurs. The Gram Schmidt curves show the total IR intensities. They are in good correlation with the TGA and DTG curves.

The identification of the separator material is exemplarily executed for sample A. The *Identify* feature of *Proteus*® contains several thousands of measured data sets for different thermal analysis methods and different classes of materials which can be compared with the current data. Here, the TGA curve and the melting point determined by *c-DTA*® of sample A are compared with the polymer library data. They show high similarity to polypropylene (pink curves); see figure 2.



1 Temperature-dependent mass change (TGA), mass change rate (DTG), the calculated DTA curves (*c-DTA*®) and Gram-Schmidt curves of separators A (green) and B (red).



2 Temperature-dependent mass change (TGA), mass change rate (DTG) and Gram-Schmidt curve of separator A compared with the *Identify* result for polypropylene.

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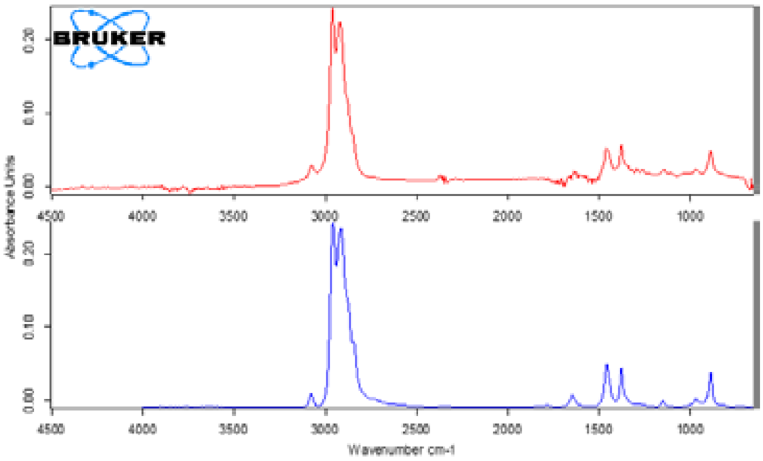
As additional proof, the gas phase spectra detected by FT-IR at 462°C were compared with those in the TGA-FT-IR database of polymers, which contains the pyrolysis spectra of all typical polymers. Again, high similarity to polypropylene was found; see figure 3.

Prior to a TGA-FT-IR measurement, an ATR-IR spectrum may also be helpful for identification. The separator foil was put on the ATR diamond crystal and an IR spectrum was taken of the solid material; see figure 4. Comparison of the spectrum with the library also yielded high similarity to polypropylene, as shown in figure 5.

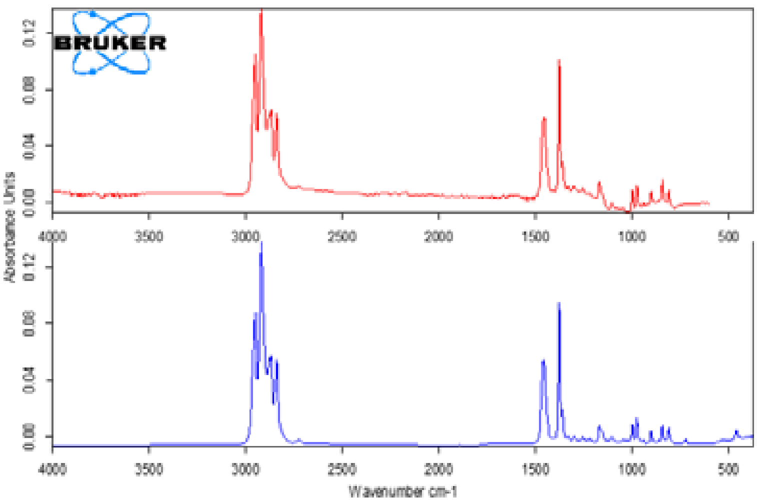
Summary

The combination of a thermobalance (TGA) and an FT-IR system provides a complementary set of measurement data – such as melting point, decomposition behavior, ash content, filler content and the identification of the gases released – from just one sample measurement. In this example, the thermal stability and the material of separator foils could be identified using the ***Identify*** library, the TGA-FT-IR database of polymers, and the ATR spectrum of the solid compound with only one instrument setup.



3 Comparison of the measured FT-IR spectrum of sample A at 462°C (red) with the pyrolysis spectrum of polypropylene (blue).

4 Bruker *INVEN*IO spectrometer equipped with an ATR unit.



5 Comparison of the measured ATR spectrum of sample A (red) with the ATR database spectrum of polypropylene (blue).

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