Application Note

Energy and Materials

Material Identification of Lithium-Ion Battery Separators Using FTIR Spectroscopy

Rapid analysis of new and used separators using the Agilent Cary 630 FTIR Spectrometer

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Introduction

Lithium-ion batteries (LIBs) have emerged as indispensable power sources in a range of applications, including electric vehicles, portable electronics, and renewable energy storage systems. Comprising four primary components – cathode, anode, separator, and electrolyte – rigorous quality control (QC) of all battery raw materials and components is needed to ensure safety, performance, and durability of LIBs.

One crucial component in the assembly of LIBs is the separator – a thin, porous membrane that physically separates the anode and cathode. This component plays a pivotal role in battery safety and performance by preventing direct electrode contact, and avoiding potential short-circuits and thermal runaway. It also facilitates the flow of ions between electrodes during charging and discharging processes.

Polymer materials, such as polyethylene (PE) or polypropylene (PP), are often used in the fabrication of LIB separators. These materials are selected for their robust mechanical strength, chemical stability, and capacity to enable the flow of ions between electrodes. Fourier Transform infrared (FTIR) spectroscopy is proven for its ability to differentiate materials by their distinct FTIR spectra, making it well suited for polymer analysis.[1]

This study focuses on the use of the Agilent Cary 630 FTIR spectrometer for material identification of LIB separators, in addition to its role in QC testing of salts and solvents used in LIB components.[2,3] Due to the ultra-compact design of the instrument, the analysis of potentially hazardous materials can be performed in a moisture-controlled environment within a glove box if necessary.[1] The Cary 630 FTIR is also widely used for research and development studies focused on advancing and improving battery materials.[4]

Experimental

Instrumentation

The Agilent Cary 630 FTIR, equipped with a single reflection diamond attenuated total reflection (ATR) interface was used for the quick and easy material identification (ID) of both new and used LIB separators (Figure 1).

Figure 1. Agilent Cary 630 FTIR spectrometer equipped with an ATR sampling module used for the identification of polymers and thin films.

Software

The intuitive Agilent MicroLab software (Figure 2) and an Agilent-generated polymer spectral library were used to identify two separators by applying the Similarity search algorithm. Instrument operating parameters are shown in Table 1.

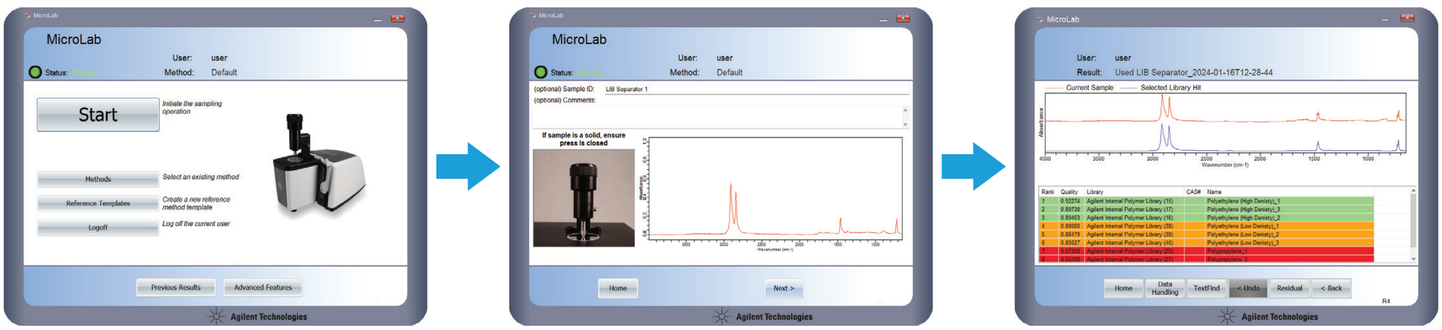
Samples

Two LIB separators were analyzed in this study:

- A new multilayer separator
- A used separator extracted from a LIB recycling facility

Table 1. Agilent Cary 630 FTIR–ATR operating parameters.

Parameter	Setting
Method	Library search
Library Used	User-generated polymers library (Agilent Internal Polymer Library)
Spectral Range	4,000 to 650 cm^{-1}
Sample/Background Scans	32
Spectral Resolution	4 cm^{-1}
Background Collection	Air
Color-Coded Confidence Level Thresholds	Green (high confidence): > 0.90 Yellow (medium confidence): 0.80 to 0.90 Red (low confidence): < 0.80



1 Start the analysis

2 Follow picture-driven software guidance

3 Instantly receive color-coded, actionable results

Figure 2. Agilent MicroLab software uses a picture-driven interface that reduces training needs and minimizes the risk of user-based errors.

Results and discussion

Using the Similarity algorithm to search the user-generated internal spectral library, both separators were identified with a hit quality index (HQI) between 0.90 and 0.99 (green) confidence level threshold.

The new separator was identified as polypropylene (PP) with an HQI of 0.94064 (Figure 3A). The HQI reflects the separator's multilayer structure and likely inclusion of additives to the PP compared to the reference material used to generate the library spectrum. However, to improve the HQI using a better matched reference material, spectral libraries can be updated or created in a few seconds using the MicroLab software. Spectra can be added to the library, either at the time of creation or at any other time, directly from the results screen.

The used separator material was identified as polyethylene (PE) with an HQI of 0.92274 (Figure 3B). As shown in Figure 4, the visual presence of contaminants from other LIB components could have an impact on the HQI result.

The HQI, which is automatically calculated for each library item by the software, indicates how well the measured spectrum and the library spectrum match. The result is often used as pass/fail criteria in material identification and confirmation workflows based on HQI-threshold settings, which are user-adjustable in the MicroLab software. In this study, results with an HQI above 0.90 were color-coded in green.

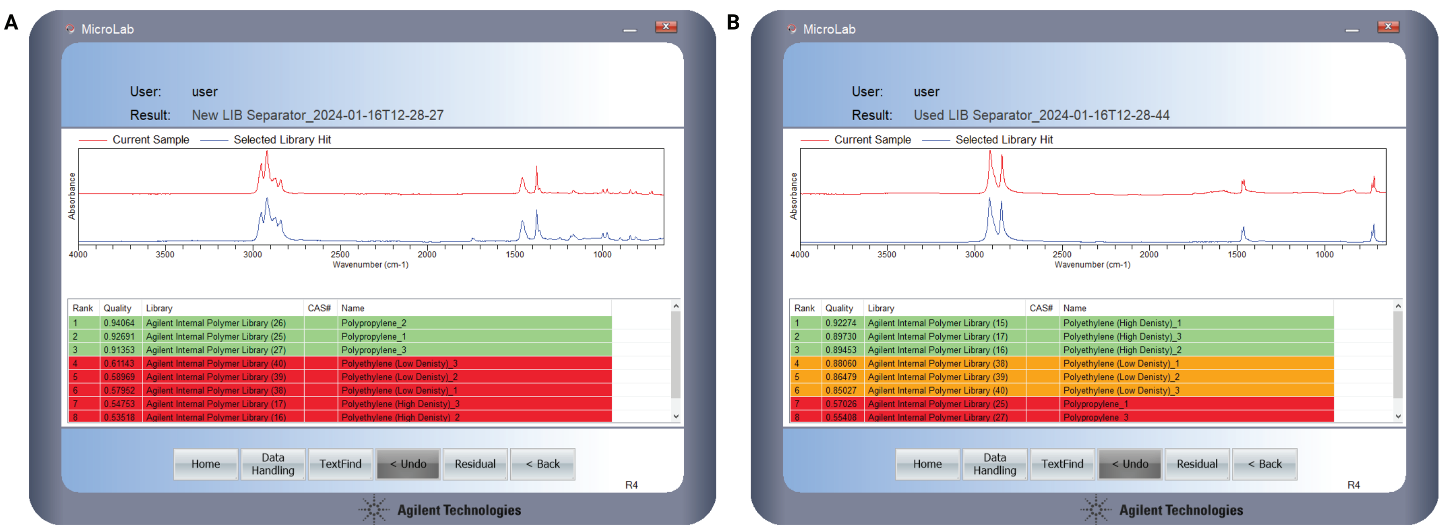


Figure 3. The Agilent Cary 630 FTIR spectrometer identification analysis of LIB separator samples (red traces) and library hits (blue traces). The table shows the quality (HQI) of the result, the library used, and the hit name of the material of the unused separator (A) and used separator (B).

Color-coding the results turns the Cary 630 FTIR system into an easy-to-use, turnkey solution that enables quick decision-making. Once the sample has been measured, the MicroLab software shows the final answer directly on screen, without any input needed by the user.

The software automatically performs the library search and provides the operator with the final color-coded results, reducing the complexity of the analysis and providing confidence in the identification of the material.

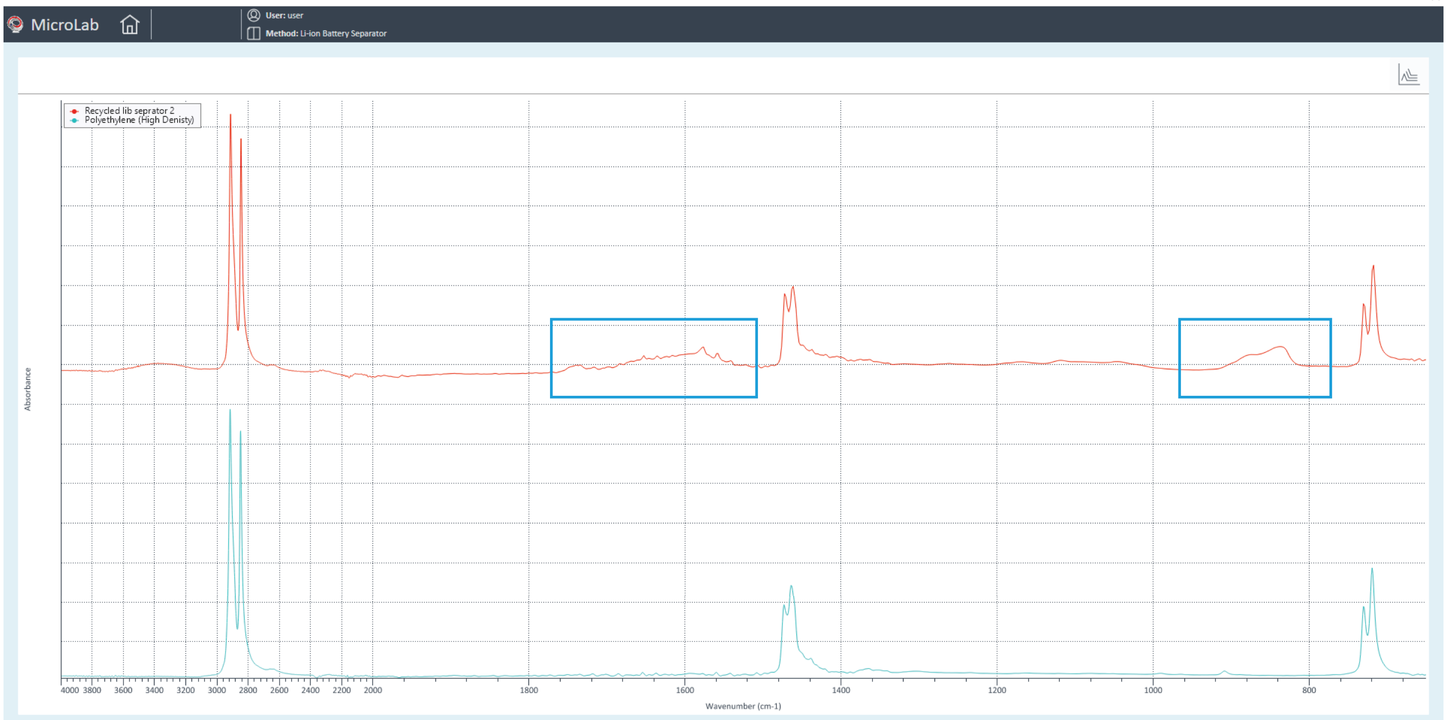


Figure 4. Overlay of spectra of the used LIB separator (red trace) and library hit (blue trace) in a compressed view. The wavenumber scaling factor within the Agilent MicroLab software enables a more detailed view of the spectral range of interest. The black boxes used to highlight regions of the top spectrum indicate potential contaminants from other LIB components.

Conclusion

The Agilent Cary 630 FTIR spectrometer and Agilent MicroLab software rapidly provided accurate material-identification results for a new and a used lithium-ion battery separator.

Using an existing user-generated spectral library for polymers within the MicroLab software enabled the Cary 630 FTIR to identify the two separator samples as polypropylene and polyethylene. Confidence in the result could be further improved by adding more representative reference spectra to the library. Spectral libraries can easily be maintained and managed in the MicroLab software, and a new library can be created within seconds. The MicroLab software applied color-coding to the identification results of the new and used separator samples based on the HQI, making it quick and easy to review the quality of the data. Both samples were identified with a high level of confidence.

This study has shown the effectiveness of the Cary 630 FTIR fitted with the ATR sampling module for QC testing of materials as required by LIB manufacturers. The methodology is also suitable for R&D groups working to develop or improve battery materials.

References

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2. Alwan, W.; Babu, S.; Zieschang, F. Quick and Easy Material Identification of Salts Used in Lithium-Ion Batteries by FTIR, *Agilent Technologies application note*, publication number 5994-6243EN, **2023**.
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Further information

Agilent Cary 630 FTIR Spectrometer

MicroLab FTIR Software

MicroLab Expert

FTIR Analysis & Applications Guide

FTIR Spectroscopy Basics – FAQs

ATR-FTIR Spectroscopy Overview

www.agilent.com/chem/cary630

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