White Paper

Energy and Material Science Research

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Advancing Research of Lithium-Ion Batteries Using the Agilent Cary 630 FTIR Spectrometer

Lithium-ion battery studies published by global research groups

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Introduction

There is increasing demand for lithium-ion batteries (LIBs), especially from the electric vehicle sector. This uptake is partially due to government policies and legislation to reduce carbon emissions associated with the use of internal combustion engines. While commercially available LIBs with higher performance are becoming cheaper, there are still opportunities for development within the sector. Researchers around the globe are looking at ways to increase the energy capacity of batteries, reduce charging times, and find cheaper and safer alternative materials for LIB components.[1]

This white paper provides examples of how the Agilent Cary 630 FTIR spectrometer has been used by leading research groups for the analysis and characterization of LIB materials and components.

FTIR spectroscopy is a well-established and powerful analytical technique that provides information on the composition of materials. Even though it is a mature spectroscopic technique, advances in FTIR sampling interfaces are continuing to extend its flexibility and use. The quick and easy qualitative and quantitative analysis of polymers and electrolytes are example applications where sampling interfaces have broadened the scope of FTIR.

The Cary 630 FTIR spectrometer

The Cary 630 FTIR spectrometer is an ultra-compact, flexible, and high-performance benchtop FTIR instrument that includes many ease-of-use features to simplify operation by nonexpert users. The innovative Cary 630 FTIR can be configured with a range of interchangeable sampling modules that integrate with the optomechanical system of the instrument (Figure 1).

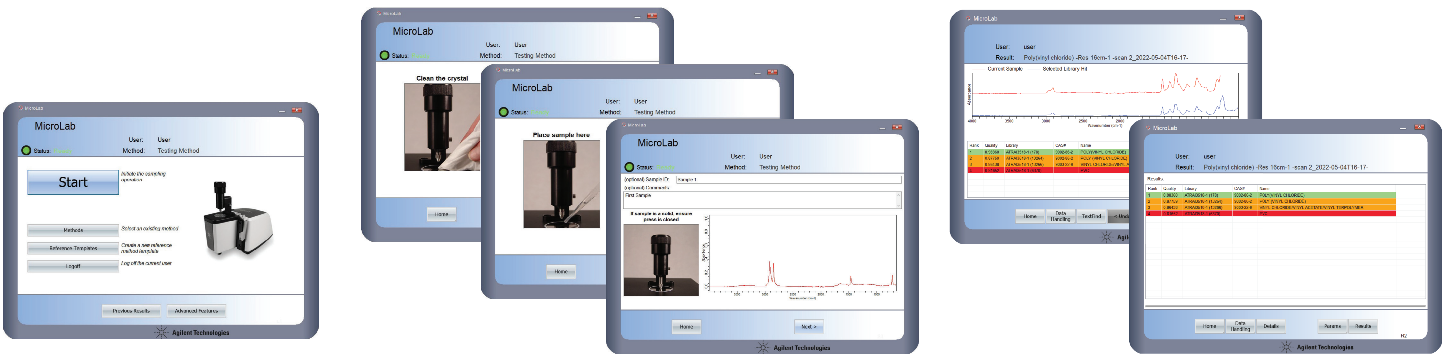
The versatile modular design means that the Cary 630 FTIR provides the configuration flexibility needed for the robust and reliable analysis of materials used in LIBs. Various materials are used within the main components of a LIB that include the anode, cathode, electrolyte (e.g., solvents and salts), and separator materials.

In a multi-user setting, a robust and reliable FTIR instrument is key to preventing downtime and reducing the risk of compromised data. A walk-up system that is easy to learn and that requires minimal training is an asset in a busy lab environment. The field-proven, robust optomechanical system of the Cary 630 FTIR has been shown to deliver outstanding performance and reproducibility.

The Cary 630 FTIR spectrometer is controlled by the powerful Agilent MicroLab software, which uses an intuitive pictorial interface to guide users through the steps of the analysis, from sample introduction to reporting. The software automatically detects which sampling accessory is installed, applies the required settings, and loads instructive images that are specific to the sampling accessory.

Figure 1. Interchangeable sampling modules for the Agilent Cary 630 FTIR facilitates lithium-ion battery research.

The software allows analysts to identify unknown compounds by automatically comparing the FTIR spectrum of an unknown sample with a library of spectra of known compounds. The Cary 630 FTIR spectrometer can also provide quantitative information (e.g., salts concentration in electrolyte solution). The software automatically performs all required calculations and provides the analyst with the final answer. The results are color-coded to help analysts interpret the data and take appropriate action (Figure 2). The FTIR spectrum of a sample provides valuable insights for the characterization of LIB components, which are useful for research studies.



1 Start the analysis

2 Follow picture-driven software guidance

3 Instantly receive color-coded, actionable results

Figure 2. Three simple steps using Agilent MicroLab software and an Agilent Cary 630 FTIR make performing an analysis straightforward and decision making easier. The picture-driven software also reduces training needs and minimizes the risk of user-based errors.

Figure 3. Analysis of a sheet-material using an Agilent Cary 630 FTIR with ATR sampling module.

The Cary 630 FTIR spectrometer with the MicroLab software form an ideal solution for the analysis of diverse material types. Once a sampling module has been selected for the 630 FTIR, a method can be tailored to the application. As an example, Figure 3 shows the Cary 630 FTIR equipped with the attenuated total reflectance (ATR) module for the analysis of materials in sheet-form.

Agilent also offers advanced FTIR spectroscopy software for the Cary 630 FTIR; MicroLab Expert provides a higher level of flexibility and spectral visualization for more sophisticated data processing.

Atmospheric moisture and oxygen can significantly affect the characterization of LIB components such as electrolyte salts, e.g., lithium hexafluorophosphate ($LiPF_6$). So, it is recommended that such experiments should be performed in an oxygen and moisture-controlled environment. The small footprint of the Cary 630 FTIR, ease-of-use features, such as changing modules, and its robustness, make it ideal for operation in a glovebox-controlled environment, as represented by Figure 4.

Lithium-ion batteries research applications using FTIR

Research-groups located around the world have used Agilent FTIR instrumentation as an integral solution for the analysis and characterization of LIB components, as summarized in the following examples:

Highly aligned graphene oxide for lithium storage in lithium-ion battery through a novel microfluidic process: The pulse freezing[2]

Yifan Liu and coworkers reported a new method to fabricate vertically aligned graphene oxide (GO) films as free-standing carbon lithium hosts for LIBs with enhanced performance. This process led to increased levels of microstructure porosity and vertical alignment of the GO films. The alignment and porous microstructures increase both electron and ion transfer capabilities across the prepared film. To characterize these GO films, the Cary 630 FTIR was used to assess the C–OH groups removal based on thermal treatment. The removal of the C-OH groups was confirmed by a significant decrease in the peak intensity at 3,429 cm^{-1} on thermally treated GO films compared to non-treated GO films.

Figure 4. Agilent Cary 630 FTIR spectrometer inside a glovebox in which the moisture or oxygen level can be controlled. An ideal setup for LIB research applications.

Lithiating magneto-ionics in a rechargeable battery[3]

Yong Hu and team reported that the reversible lithiation/delithiation in a molecular magneto-ionic material (i.e. the cathode in a rechargeable LIB) accurately monitors its real-time state of charge through a dynamic tunability of magnetic ordering. In the study, the Cary 630 FTIR was used to study compound changes due to lithiation. The system was used to study the vibrational shift of the compound in pristine condition. A peak at 2,108 cm^{-1} corresponding to the C≡N bond was observed, but upon lithiation, the peak disappeared, and two new peaks emerged at lower wavenumbers of 2,075 and 2,012 cm^{-1}.

ZIF 67 derived Co–Sn composites with N-doped nanoporous carbon as anode material for Li-ion batteries[4]

Sheeraz Ashraf *et al.*, synthesized a composite of SnO_2 with nanoporous carbon using ZIF-67, a 2-methylimidazole cobalt salt. ZIF-67 creates a framework composed of a Co–Sn alloy and Sn–C network that is responsible for the enhanced structural stability of the composite material. In the study, the Cary 630 FTIR was used to analyze the type of bonding present at the molecular level between the three synthesized composites.

Novel polymer coating for chemically absorbing CO_2 for safe Li-ion battery[5]

Jean-Christophe Daigle *et al.*, reported on the utilization of a mixture of polymers that can chemically absorb CO_2, including the coating of aluminum foils, which serve as trapping sheets. The authors concluded that the coating method is economically viable, industrially applicable, and permits the fabrication of safe high-power lithium-ion batteries based on large format cells with significant cycle life potential. In the study, the Cary 630 FTIR equipped with an ATR was used to measure the trapping sheets at different times and temperatures to monitor degradation and the conversion rates of epoxy groups.

A versatile method for grafting polymers onto $Li_4Ti_5O_{12}$ particles applicable to lithium-ion batteries[6]

Jean-Christophe Daigle *et al.*, reported a novel and versatile method of grafting polymers onto lithium titanium oxide (LTO) by dispersion mediated interfacial polymerization. The method can produce thin and homogenous polymer films that are useful for battery applications. The Cary 630 FTIR system was used to characterize the polymeric shells by monitoring the appearance of the polymer's -CH_2-CH characteristic signals at 2,900 cm^{-1}.

A new method for determining the concentration of electrolyte components in lithium-ion cells, using Fourier transform infrared spectroscopy and machine learning[7]

L. D. Ellis and coworkers introduced a new method for determining unknown concentrations of major components in typical LIB electrolytes. A quick, cheap, and accurate method was generated by utilizing the Cary 630 FTIR equipped with a germanium attenuated total reflectance (ATR) and machine learning. Machine learning techniques were used to match features of the FTIR spectrum of an unknown electrolyte to the same features of a database of FTIR spectra with known compositions. The researchers reported that using the method, the concentration of $LiPF_6$ could be determined by FTIR with similar accuracy and precision as an inductively coupled plasma optical emission spectrometry (ICP-OES) method.

High performance solid polymer electrolyte with graphene oxide nanosheets[8]

In this study, Mengying Yuan and co-authors introduced two-dimensional graphene oxide (GO) sheets with a high surface area and excellent mechanical properties into a solid polyethylene oxide/lithium salt electrolyte. The GO sheets improved ion conductivity and increased the tensile strength of the polymer electrolyte and appeared to significantly enhance the performance of the lithium-ion battery. To measure the lithium salt dissociation fraction, the Cary 630 FTIR system with MicroLab software was used. The dissociation fraction was obtained as the ratio of the respective areas under the peaks located in two specific ranges: the 620 to 624 cm^{-1} range, representing the dissociated "free" ClO_4^{-1} ions, and the 630 to 635 cm^{-1}, range representing the ion-pair $LiClO_4$.

User-friendly freeware for determining the concentration of electrolyte components in lithium-ion cells using Fourier transform infrared spectroscopy, Beer's Law, and machine learning[9]

Sam Buteau *et al.*, refined the model based on FTIR measurements[7] to determine the concentration of electrolytes including $LiPF_6$, ethylene carbonate (EC), ethyl-methyl carbonate (EMC), dimethyl-carbonate (DMC), and diethyl-carbonate (DEC). In the study, the Cary 630 FTIR equipped with a germanium ATR was used to characterize the composition of the electrolytes. The model could be applied to the fast determination of the composition of unknown electrolyte samples, with a specified set of components.

Conclusion

The Agilent Cary 630 FTIR is an effective spectrometer for the characterization of various materials of interest to researchers working on lithium-ion batteries. It can be fitted with a range of fully-interchangeable sampling technologies, ensuring that the best sampling technique can be used for the application.

The summaries of the research papers have shown the flexibility and usefulness of the Cary 630 FTIR in expanding knowledge of materials that are needed to improve the performance and safety of LIBs.

References

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Further information

- Agilent Cary 630 FTIR Spectrometer
- Agilent MicroLab Software
- Agilent MicroLab Expert Software
- FTIR Analysis & Applications Guide
- FTIR Spectroscopy Basics - FAQs
- ATR-FTIR Spectroscopy Overview

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