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Fourier Transform Infrared Spectroscopy (FTIR) Services

Fourier Transform Infrared Spectroscopy (FTIR) is an effective analytical technique for quickly identifying the "chemical family" of a substance.

FTIR is a spectroscopic technique based on the absorption of infrared photons that excite vibrations of molecular bonds. A spectrum of characteristic bands representing the specific functional groups present in a molecule is produced that can be used as a fingerprint to help identify and characterize the sample. FTIR analysis can be conducted in transmission or reflection modes. The specific FTIR analysis mode, as well as the sample preparation method, is selected based on the particular characteristics of the sample. For transmission FTIR analysis, samples must be placed onto an infrared transmissive substrate and thinned to between 0.1 to 10 µm prior to analysis. The ideal sample thickness for reflection FTIR analysis is in the same range and additionally reflection analysis only applies to samples on a reflective substrate (e.g., on polished metals). Attenuated total reflection (ATR) mode is a special form of reflectance FTIR where the IR radiation makes several passes through a high refractive index ATR crystal and the evanescent waves produced from total internal reflection extend into the top surface of the sample that is in direct contact with the ATR crystal, facilitating a surface sensitive mode for FTIR analysis.

The analytical spot size in FTIR can range from several millimeters in diameter down to ~50 µm with the use of a microscope attachment. For the detection of a minor component in a mixture of compounds, the component should be present at levels of at least a few percent by weight, though the detection limit can be higher or lower, depending on the molecular structures of the particular compounds. Because the fingerprint spectra of many organic compounds are unique, FTIR is most commonly used to provide qualitative compound identification. Quantitative FTIR can be a powerful technique when the appropriate calibration samples are available. FTIR is applicable to solids, liquids, and gases.

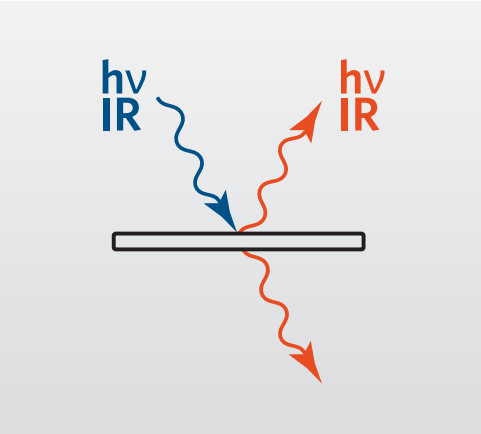

Figure 1: General schematic of how FTIR works

Strengths

- Capable of identifying organic functional groups and specific organic compounds
- Extensive spectral libraries to aid in compound identification
- Ambient analysis conditions (no vacuum required so suitable for volatile compounds and liquids)
- Large sample size can be accommodated
- Mostly non-destructive (ie the analysis itself does not cause damage)
- Minimum analysis area: ~30-50 micron (substrate dependent)
- Semi-quantitative in certain cases

Limitations

- Limited surface sensitivity
- Limited inorganic information
- Limited application for reactive samples (e.g., air/moisture sensitive materials)
- Qualitative information only unless calibration samples are used

Common Applications

- Identification of unknown organic and some inorganic materials, often mixtures and often microscopic
- Detection and characterization of organic and some inorganic additives in polymers at levels as low as a few percent
- Characterization of changes in the chemical structure of organic materials as a function of polymer cure, sterilization, heat treatment, plasma treatment, etc.
- Quantification of O_i in Si wafers, and H in SiN, SiON, and a-Si thin films (Si-H vs. N-H)
- Characterization of total non-volatile residue (NVR) for evaluation of cleanliness

Industry Sectors and Technologies

- Medical devices
- Pharmaceuticals
- Semiconductor
- Consumer electronics
- Industrial goods
- Aerospace & Defense

Case Study: Identification of Unknown Liquid Droplets

Here we have a case study showing the application of FTIR analysis for the identification of unknown liquid droplets on a substrate. An optical photo of the liquid droplets is provided in Figure 2, showing the size range of the droplets to be ~50 to 150 µm.

A representative FTIR spectrum obtained from the liquid droplets is provided in Figure 3, together with assignment of major functional groups, such as CH, C=O and C-O.

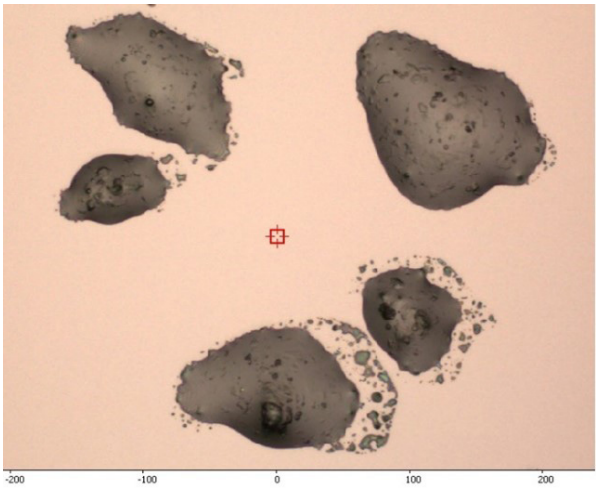

Figure 2: Optical photo of the liquid droplets on a substrate (scale at the bottom is in the unit of micron).

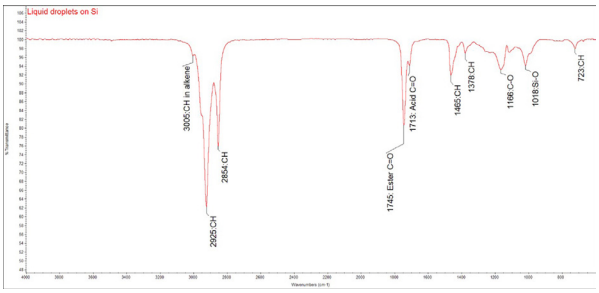

Figure 3: FTIR spectrum of the liquid droplets with peak assignments.

In addition to the functional group analysis, identification of the droplet composition is achieved by matching the sample spectrum against the extensive digital libraries containing ~350,000 reference FTIR spectra. The liquid droplets were characterized as a mixture of a fatty ester oil similar to palm oil together with trace amounts of magnesium silicate similar to talc, as shown in the overlay in Figure 4.

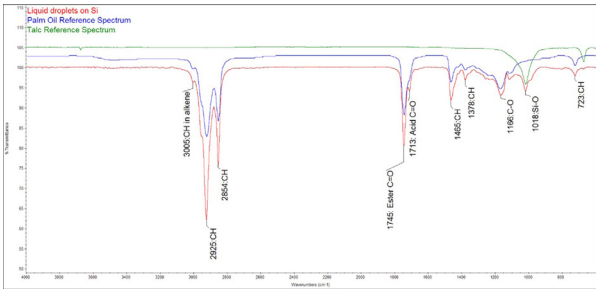

Figure 4: Overlay of droplet spectrum with library references of palm oil and talc.

Complementary Techniques

Other techniques complementary to FTIR Spectroscopy include Raman Spectroscopy, X-ray Photoelectron Spectroscopy (XPS), Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS), Gas Chromatography-Mass Spectrometry (GC-MS) and Liquid Chromatography-Mass Spectrometry (LC-MS). All of these techniques can be used to characterize organic materials to varying degrees.

Raman is also a vibrational spectroscopy technique that, in many cases, provides complementary information. Both techniques provide organic compound identification; and are not limited simply to functional group identification. The minimum spot size in Raman is 1 µm, significantly smaller than for FTIR. Certain samples analyzed by Raman will show interference from fluorescence. Also, in Raman spectroscopy, if elemental carbon and/or dyes are present, they can dominate and mask signals from organic compounds, making FTIR more appropriate in these cases.

XPS provides information on short-range chemical bonding of a sample while TOF-SIMS provides information on its molecular composition. The depth of analysis with XPS and TOF-SIMS is on the order of 10-100 Å so they can sometimes be too surface sensitive when it comes to characterizing the bulk of an unknown organic material, if the sample is not pristine and the surface has been contaminated with other impurities. GC-MS and LC- MS are commonly used tools in the characterization of organic compounds. Both techniques have the ability to separate mixtures of samples into their individual components and characterize or identify each of the constituents. These techniques typically require the sample to be vaporized or dissolved prior to analysis, complicating the sample preparation needed for these analyses.

FTIR at EAG

Our FTIR scientists are highly educated and knowledgeable and are leaders in the analysis of complex organic mixtures. We pride ourselves in skillful sample preparation, knowledgeable spectral interpretation and extensive experience with many different types of materials and applications. We provide accurate data, complete written reports, fast turnaround times, and person-to-person service.