



Analysis of Polymers and Plastics

- Quality Control & Failure Analysis



• Quality Control for Cost-Efficiency

Plastics are used in countless products such as automotive parts, packaging materials, home appliance, electric devices or textiles.

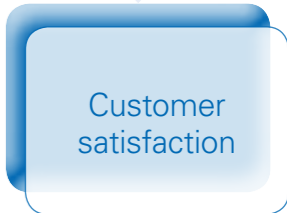
Many of today's plastics are complex multi component systems made from various compounds like different polymers, fillers and additives. The systematic selection and blending of these constituents in appropriate mixtures results in materials with optimized properties.

As a high product quality at low price is a basic requirement in modern industry, reliable quality control is mandatory. An essential step for a trouble-free manufacturing process without waste is to verify the correct identity of the incoming raw materials. Later on, the correct composition in the final plastic product has to be checked to ensure the right properties.

Reliable quality control is essential to achieve a cost-saving production of high quality plastic products. Bruker's ALPHA II provides a FTIR spectroscopy based solution for powerful routine quality control of incoming goods, intermediates and final products.

In case of any product defects an effective failure analysis is required to understand the source of error. The FTIR microscope LUMOS is a valuable analytical tool for trouble-shooting as it allows to determine the chemical composition of smallest structures.

- **Incoming goods inspection**
 - Plastics
 - Auxiliaries
- **Characterization of unknown samples**
 - Bulk
 - Multilayer products (laminates, paint chips and varnish)
- **Universally applicable**
 - Pellets
 - Thermoplastics, elastomers, rubbers
 - Monomers
 - Fillers
 - Additives
 - Plastic products (films, fibers, parts)
- **Quantification of fillers and additives**
- **Failure analysis on plastic components**
 - Wrong composition
 - Inclusions
 - Blooming
 - Contaminations



Fast and Easy Identification

Chemical Fingerprint of the Sample

Using Bruker's Fourier Transform Infrared (FTIR) spectrometer ALPHA II (Fig. 1) the identity and correct composition of any plastic product or raw material can be checked in less than one minute.

The IR-spectrum of any sample reflects its molecular composition – just like a chemical fingerprint (Fig. 2). Both organic and inorganic chemical components contribute to the sample spectrum. Therefore the IR-method is very suitable to identify as well pure compounds as complex materials. Furthermore the quantification of individual components inside the analyzed material is feasible.

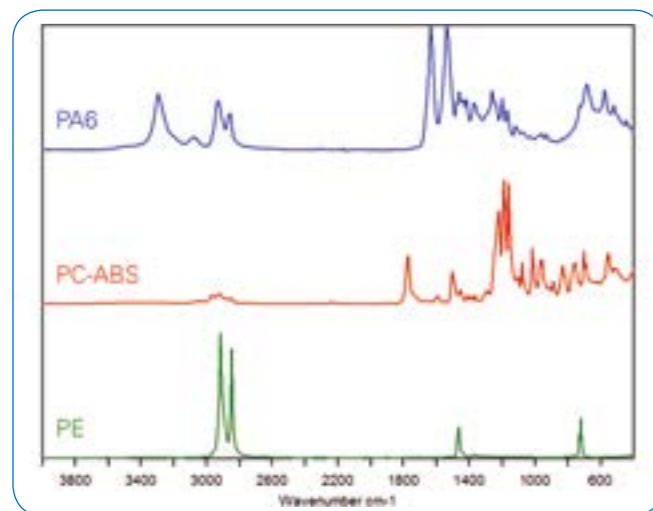


Fig. 2: The IR-spectrum is a chemical fingerprint that allows polymer identification.

Simple and Fast Analysis

The measurement interface of the ALPHA II is a diamond ATR crystal. Its mechanical and chemical robustness allows as well the analysis of reactive liquid samples like monomers as of very hard plastics like polycarbonate. The ATR (Attenuated Total Reflection) technique is very comfortable and fast as there is virtually no sample preparation required. No matter if the analysis has to be performed on pellets, films, plastic parts, powders or liquids: For recording the IR-spectrum the sample just has to be brought into contact with the ATR crystal.

After the measurement the sample is identified by automatic comparison of its spectrum against spectral data from reference materials.



Fig. 1: Measurement of a plastic spoon using the ALPHA II with ATR measurement interface.

Prepared for Your QC Demands

The ALPHA II is a very compact system with a small footprint (A4 format) and light weight (<7 kg). Being insensitive to vibrations it can be placed almost anywhere and moved easily to relevant places. The option to operate the ALPHA II with an integrated touch panel pc and powered by a battery allow its use even outside the laboratory, e.g. in the warehouse or near production sites.

All hardware components are continually monitored for correct functionality. Fully automated instrument test procedures for Operational and Performance Qualification (OQ/PQ) are performed to ensure permanent instrument operation within specification. Furthermore the OPUS software is fully compliant to cGMP regulation.

IR-Advantages

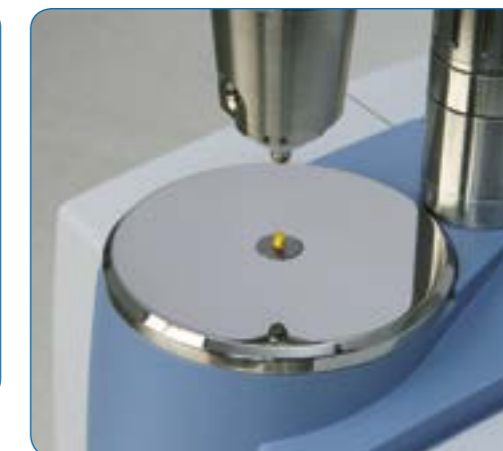
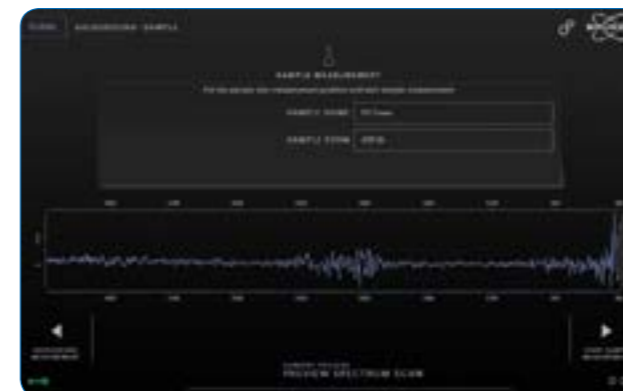
For most samples the FTIR analysis is performed without sample preparation and without the need of any consumables. Measurement times are typically below 1 minute. Therefore, FTIR spectroscopy saves your time and cost when being compared to classical wet chemical methods.

The ALPHA II and LUMOS are products that are designed to be used for many years. Both instruments utilize modern high quality optical components with a guaranteed long lifetime. Adding the low energy consumption these outstanding characteristics result in minimal running costs.

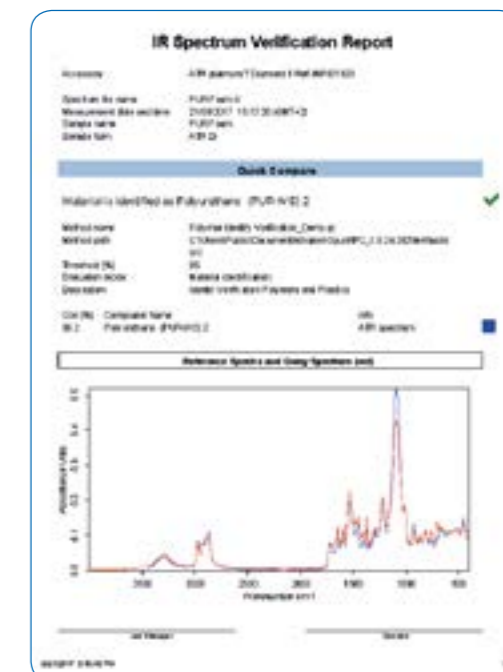
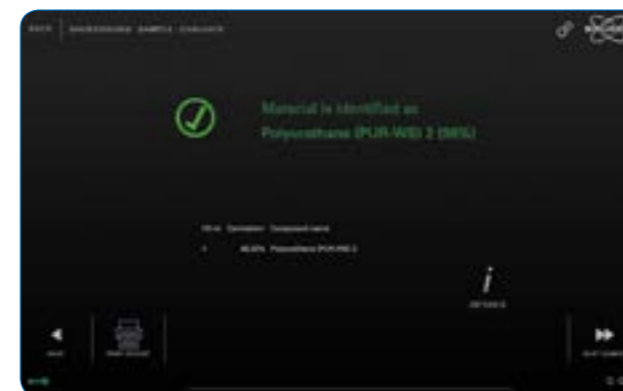
Intuitive Workflow

The ALPHA II user interface enables even untrained operators to verify the identity of a known sample or to identify unknown samples. Guided by the software the user performs measurement, evaluation and reporting in a few steps:

- 1 Measure background spectrum of the empty ATR measurement interface. Enter sample information.
- 2 Place sample on ATR measurement interface and apply pressure clamp. Start measurement with a single click.



- 3 Verify the identity of the measured sample by automatic comparison of the sample spectrum against references.
- 4 Generate a meaningful analysis report by a single click before proceeding with the next sample.



• Failure Analysis

The failure of polymer and plastic materials often is caused by the inhomogeneous distribution of the used components inside the polymeric material. Also contaminations like particles, fibers or inclusions may be the reason for its failure. In case of composite materials defect layers or a layer made from the wrong material will have a negative impact on the product properties.

As such defects are often extremely small they are hard or even impossible to analyze by a macroscopic measurement. However, a successful failure analysis includes the chemical analysis of the faulty region in the sample.

The FTIR microscope LUMOS (Fig. 3) is a powerful tool for failure analysis: It allows to obtain IR-spectra anywhere on the sample with high lateral resolution and thereby to reveal the chemical composition of this particular area of the sample.

Due to its outstanding ATR-performance the LUMOS is capable to analyze the vast majority of samples without any sample preparation.

Fig. 4: Example for the analysis of a product defect with LUMOS: A batch of polyethylene pellets contains unwanted brown inclusions.

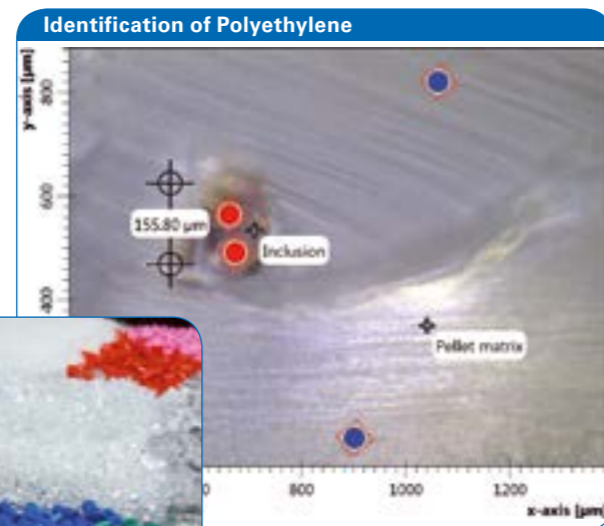


Fig. 5: The microscopic visual image of a polyethylene pellet with brown inclusion is shown together with the positions of the IR-analysis.

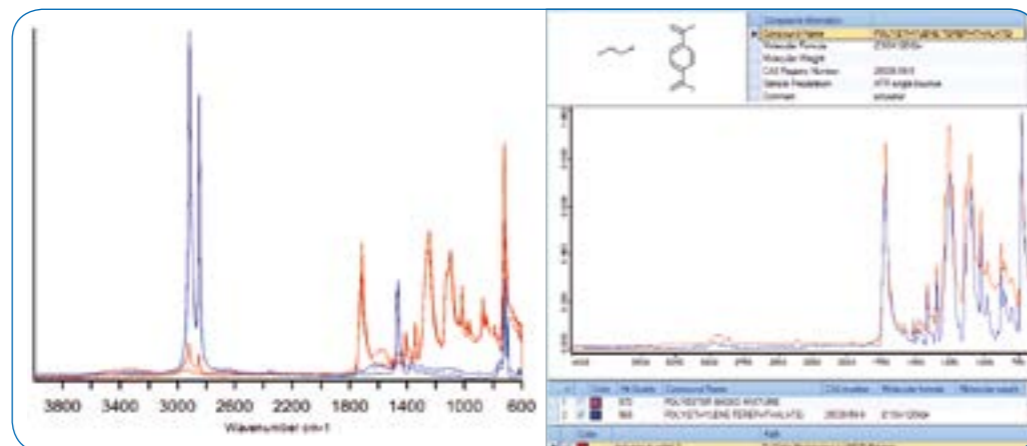


Fig. 6: The IR-spectra show clearly different characteristics on the PE pellet matrix (blue spectra) and on the unknown inclusion (red spectra). Search in a spectral library identifies the inclusion to be a polyester (PET).



Fig. 3: FTIR microscope LUMOS

• Avoiding Product Defects

The failure of polymer and plastic products is most effectively avoided by a good product design, correct material selection and an appropriate production process. During product development many variables influencing plastic properties have to be considered to ensure a high quality of the final product.

FTIR Microscopy Made Easy

LUMOS is a compact stand-alone system with full automation of all hardware components. The intuitive software of the LUMOS guides the operator step by step through the process of data acquisition. At each step the user interface only provides these functions appropriate to proceed. Although the LUMOS is designed to be operated by non-experts for routine applications, its exceptional sensitivity makes it also very suitable for high demanding applications.

Comfortable Sampling

The LUMOS provides plenty of space for the sample and a large working distance. In combination with the good accessibility of the sample stage a very convenient sample positioning is achieved.

The large field of view and the high visual quality eases finding the region of interest on the sample.

LUMOS generates precious information regarding product quality: Measurements with a local resolution in the micrometer range allow to characterize the composition of plastic materials. Mapping measurements on the sample reveal the distribution of individual components, e.g. the basic polymer, fillers and plasticizers. The impact of variations in the processing conditions on the material homogeneity can therefore be determined. Events of self-contamination (e.g. due to partly melted granules) are easily detected.

Moreover LUMOS is a helpful tool in reverse engineering as it provides information about the composition of products which are already in the market.

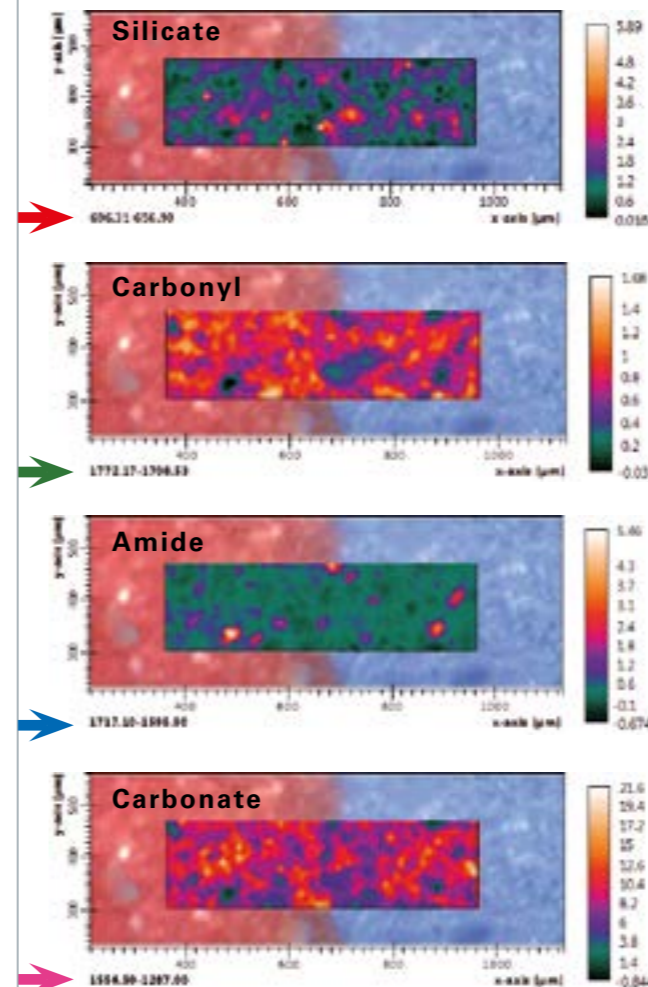
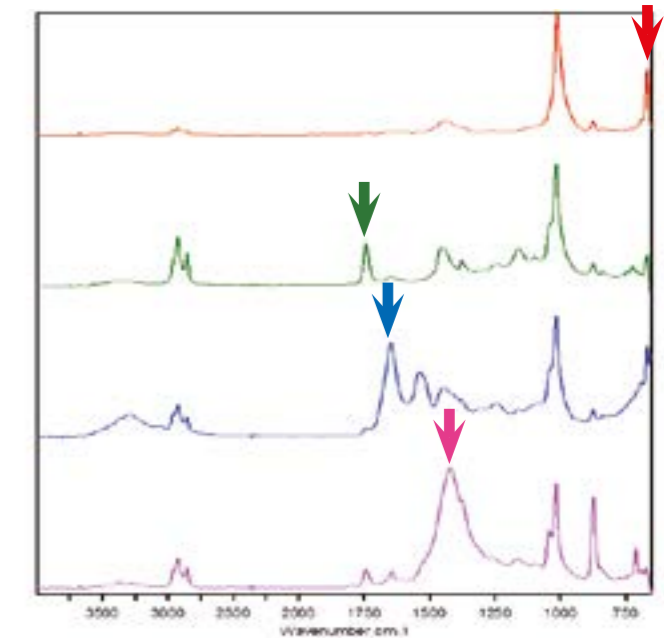


Fig. 7: Microscopic analysis of a rubber eraser. Chemical images on top of the visual sample image show the distribution of individual organic and inorganic components. The concentration is indicated by color coding (white/orange=high; black=low). The chemical images are generated by integration of spectral bands which are specific for a certain component of interest (see indications on selected sample spectra).



• Dedicated Applications

Quantification of Fillers, Additives and Blends

Modern plastics are multi component systems made from polymers, fillers, plasticizers and compatibilizers. To determine the correct composition is an essential part of the quality control. The IR-spectrum allows to quantify individual components on base of calibrations (Fig. 8).

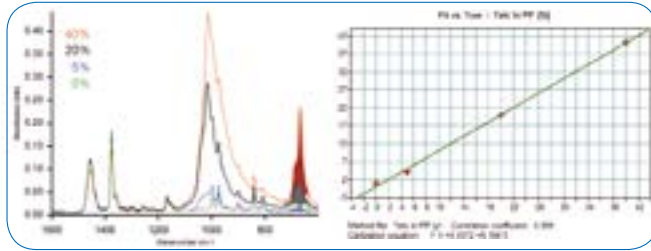


Fig. 8: Quantification of talc in polypropylene. Calibration by integration of the substance specific band at ~690 cm⁻¹.

Differentiation of Polyamides

Polyamides are thermoplastic polymers consisting of monomers joined by an amide bond. The polyamide group includes many different polymers with different chemical and physical properties. Despite a high similarity in their chemical structure IR spectroscopy allows to differentiate between various polyamides like PA6, PA6.6, PA10 and PA12.

Determination of the Degree of Cure of a Varnish

The material properties of modern paints and varnishes are critically influenced by the curing process. FTIR spectroscopy quickly and reliably allows to measure the degree of cure of paints, varnishes and the like (Fig. 9). Within only a minute a quantitative conclusion about the curing state of the sample can be drawn.

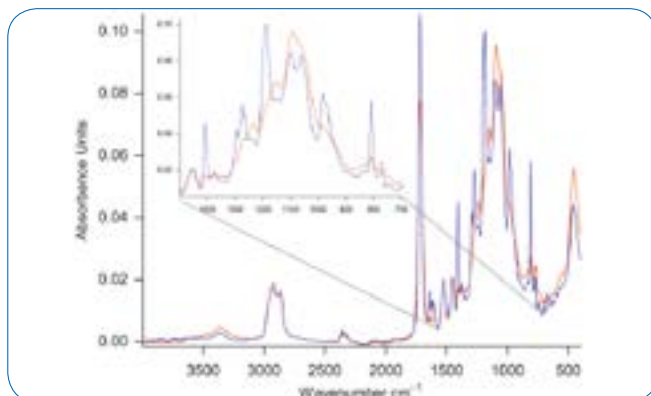


Fig. 9: IR-spectra of an acrylic varnish before curing (blue) and 100% cured (red).

Differentiation of HDPE and LDPE

Polyethylene (PE) is the most produced polymer in the world. High density PE is used for bottles, boxes whereas plastic films and bags are made from low density PE. The IR-spectrum contains information that allows the differentiation of low density and high density PE.

Analysis of Multi-layered Packaging

Multi-layer packaging is often required for maintaining product integrity, e.g. of food and pharmaceuticals. Different polymer films and other materials are combined to prevent the product from being exposed to oxygen, ultraviolet illumination, or other environmental factors. The design and fabrication of polymer films is typically a complex and costly process that can affect actual and perceived product quality. IR-microanalysis provides insight into the structure of such polymer laminates for quality control and for the analyses of found defects (Fig. 10).

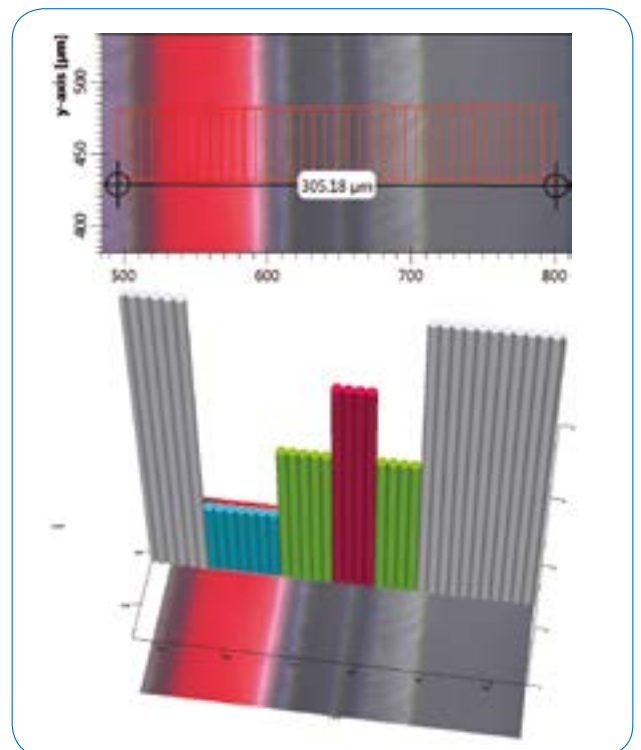


Fig. 10: Line map on the cross section of a packaging foil. Evaluation of the IR-spectra reveals six layers from four different materials. The result is visualized in a 3D column plot. Colors indicate the different materials: Grey: metal (no IR-spectrum); Blue: PET; Green: LD-PE; Red: PA 6

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