Near-infrared and Raman spectroscopy for polymer analysis

An introduction

Metrohm

Discover the potential of near-infrared and Raman spectroscopy for more efficient polymer analysis

Preface

Versatility, durability, and cost-effectiveness make polymers indispensable. Their increased demand calls for more efficient manufacturing processes and quality control. Both imperatives have become key drivers for plastic production [**1**].

Quality control (QC) includes systematic processes and procedures that ensure specific quality standards in the final product. Effective QC in plastic manufacturing means monitoring and ensuring product quality at different stages of production. Raw materials inspection, monitoring the polymerization process, fine control of additives, and molding and finishing are the most important processes to consider [**2**].

Producers voluntarily implement more testing and quality practices because of:

- **Cost pressure.** Rapid feedback from the analysis of incoming raw materials and intermediates decreases manufacturing costs.
- **Increasing competition.** Quality practices can be used as a competitive marketing tool to raise brand value.
- **Scarcity of resources.** Laboratory processes and staff are valuable, so quality control methods that non-specialists can use are essential.
- **Confirmation.** Primary analytical methods are complex, time-consuming, and prone to human errors. Secondary methods like spectroscopic analysis can support primary testing methods without the added complexity and risk.
- **Safety.** Preventing human contact with manufacturing chemicals and avoiding product contamination as much as possible is desirable.

In the polymer industry, adhering to rigorous quality standards, optimizing production processes, and promoting innovation are more important than ever. To accomplish these objectives, it is imperative to employ advanced analytical techniques. **Near-infrared (NIR) spectroscopy** and **Raman spectroscopy** are recognized as powerful and complementary analytical tools that effectively address a wide range of requirements throughout the polymer lifecycle. These techniques are essential in verifying raw materials and conducting QC for final products. Understanding the strengths of each technique ensures their optimal use for specific applications.

Spectroscopy permits identification and quantification of different polymers for QC and for research and analysis purposes. Few spectroscopic techniques satisfy the need for fast, easy, accurate, nondestructive, and flexible testing. However, NIR and Raman spectroscopies top this list.

This e-book is intended to give an overview of various polymer applications where NIR and Raman spectroscopy excel. A short introduction is given for each technology followed by several application examples. Unique NIRS and Raman solutions are presented along with more information about the methodology.

Overview of NIR and Raman spectroscopy for polymer analysis

Raman and NIR spectroscopy are increasingly chosen for polymer analysis because they are both non-destructive, require no sample preparation, and give results in just seconds. Both techniques rely on the interaction between light and matter to provide insights about samples. Spectroscopy can be very easy on the user; non-technical operators can collect data on-site. It is also environmentally friendly – requiring no chemicals, solvents, or materials for sample preparation – and it generates no waste.

Near-infrared and Raman spectroscopy are complementary techniques that are highly sensitive to molecular structure and provide fast and facile identification and quantitative analyses.

Near-infrared spectroscopy
In general, near-infrared spectroscopy provides excellent determination of functional groups associated with polymeric composition, making it widely applicable for polymer identification and structural characterization. NIR spectroscopy is particularly suited for the rapid, nondestructive analysis of bulk materials. Its high sensitivity makes it an excellent choice for quantifying key chemical and physical properties in polymers, such as moisture content, hydroxyl number, acid number, density, and composition. NIR's ability to penetrate deeper into samples makes it indispensable for high-throughput environments like inline monitoring during extrusion or film production, where fast and reliable data collection is essential.

Raman spectroscopy
Raman spectroscopy excels at identifying molecular structures and functional groups with exceptional specificity. Its spectra contain signature «fingerprint» peaks that reflect functional groups, conformation, bonding, and extent of polymerization, for example. This technique is ideal for characterizing chemical composition, detecting additives and impurities, and analyzing multi-layered or composite materials. Raman's non-invasive and non-contact nature makes it particularly effective for examining complex polymer blends, coatings, and intricate microstructures.

Together, NIR and Raman spectroscopy complement each other – each addressing analytical challenges that the other cannot fully resolve. Both Raman and NIR spectroscopy are ideal for assessing bulk properties and large scale processes, though Raman provides an additional level of detail for defect analysis and advanced material characterization. By strategically combining both techniques, polymer manufacturers can achieve a deeper understanding of their incoming and finished materials, improve product quality, reduce waste, and enhance overall efficiency.

Why choose NIR spectroscopy for polymer analysis?

NONDESTRUCTIVE METHOD WITH NO SAMPLE PREPARATION REQUIRED

Liquid and solid samples are measured as-is with NIRS. Just add the sample to the corresponding vial or sample cup and start the measurement. Since NIRS is a nondestructive method, samples can be recovered and reused.

ACCURATE RESULTS FOR MULTIPLE PARAMETERS IN SECONDS

NIRS is a multicomponent technique. A single measurement provides information about several physical and chemical parameters. Therefore, NIRS can be used successfully at several different steps along the polymer production line and for the corresponding plastic products.

READY TO USE WITH PRE-CALIBRATIONS

Pre-calibrations guarantee the fast implementation of your application. You can be fully productive from day one. NIR measurements can be traceable to a recognized standard and often have the same level of accuracy and reproducibility as primary methods.

LOWER COSTS COMPARED TO WET CHEMICAL METHODS

No chemical reagents are required for analysis with NIRS. Aside from the cost savings, no chemical waste is produced, making this method extremely environmentally friendly.

NIRS IS STRAIGHTFORWARD AND EASY TO USE

The analysis itself can be performed by anyone – it is very simple.

SINGLE-SOFTWARE INTEGRATION WITH PRIMARY METHODS

OMNIS is an all-in-one spectroscopy software that combines everything from calibration to model development, measurement procedure definition, and routine analysis. It is also a complete platform, allowing the combination of multiple analytical technologies and automation possibilities.

Instrument shown:
OMNIS NIR Analyzer

Why choose Raman spectroscopy for polymer analysis?

TEST AQUEOUS SOLUTIONS

Unlike NIRS, Raman spectroscopy is insensitive to water, meaning that it can be used to analyze different quality parameters in aqueous solutions.

HIGH SPECIFICITY

Raman spectroscopy can distinguish between very similar materials. It is sensitive to functional groups, structure, configuration, extent of polymerization, and more. This makes it ideal for testing polymer materials throughout the production chain.

INCREASED EFFICIENCY

Raman spectrometers are rugged devices used for quick sampling in many locations. This means that more tests can be performed more quickly – by anyone – in or out of the lab.

CONSISTENT SAMPLING FOR CONSISTENT RESULTS

Raman systems can be adapted to the sample type. This reduces the impact of testing on a material, increases the accuracy of measurements, and even allows testing through thin barriers. Higher accuracy means less re-work and higher efficiency.

SUCCESSFULLY TEST MORE MATERIAL TYPES

Raman measurements of polymers have improved dramatically in recent years. Metrohm has multiple options to suppress interference from colored plastics, leading the field in polymer identification and verification.

Instruments shown (left to right): i-Raman Plus Portable Raman Spectrometer, NanoRam-1064 Handheld Raman Spectrometer, and Metrohm Instant Raman Analyzer (MIRA) P handheld Raman Spectrometer

Where are NIR and Raman spectroscopy best utilized during polymer production?

Both NIR and Raman spectroscopy have many fundamental qualities that surpass other technologies for polymer analysis. These include high material specificity, large libraries of known substances, the power to analyze mixtures, and the ability to sample plastics in myriad forms, including clear and colored composites, coatings, and adhesives. In many cases, more than one chemical or physical parameter can be determined. These characteristics mean that spectroscopy can be used successfully at several different steps along the production chain of polymers and the corresponding plastic parts.

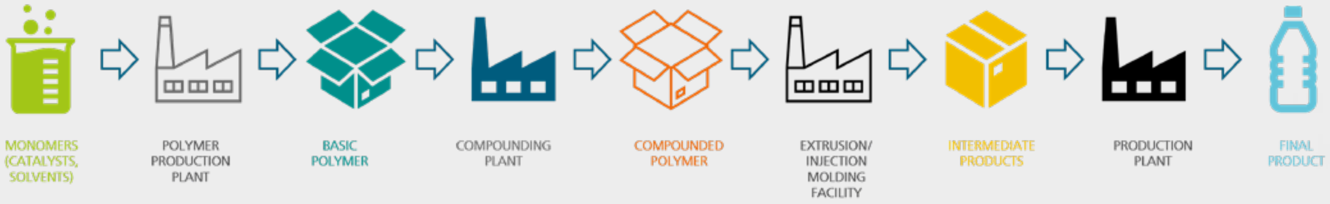
There are four major segments in the polymer process where spectroscopic analysis is beneficial:

1. Polymer producers
- Inspection of raw materials
- Quick parameter checks during chemical processes
- During research and development
- Final quality control

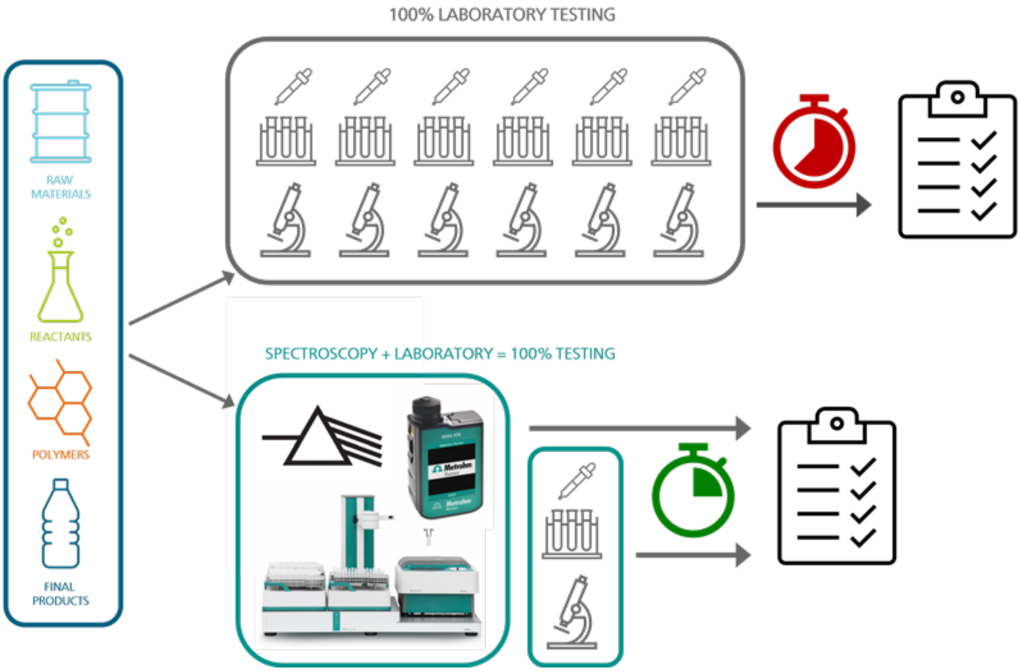
2. Compounders
- Verify the quality of the incoming polymers and additives/received or prepared masterbatches
- Quality control of the final compounded product

3. Polymer processors (e.g., injection molders and extruders)
- Confirm the quality of received polymers

4. Plastic parts producers
- Check the quality/consistency of final products



How NIR and Raman spectroscopy can impact the polymer industry



Efficient, low-resource methods like NIR and Raman spectroscopy enable rapid inspection of materials at any point in the production process, minimizing analysis time, production delays, and out-of-spec products.

Advances over the past 20 years have produced compact, sensitive instruments capable of polymer analysis across diverse settings, allowing nontechnical personnel to achieve reliable results both in and outside of the lab. The United States Pharmacopeia (USP) recently released General Chapter <858> Raman Spectroscopy [**3**] that provides guidelines on the practical application of Raman spectroscopy to the analysis of pharmaceuticals and excipients. The standards developing organization ASTM increasingly accepts Raman and NIR spectroscopy as an alternative fast technologies and has released several guidelines for its use [**4**]. The OMNIS NIR Analyzer and model development tools from Metrohm comply with these ASTM guidelines.

Spectroscopy makes 100% testing possible

A √(N)+1 sampling rate was once sufficient for quality control, but the shift to 100% testing demands faster methods like spectroscopy [**5**]. Traditional lab techniques (e.g., chromatography, mass spectroscopy, and wet chemical analysis) may require more than 14 hours to inspect 100 containers. By using NIR and Raman spectroscopy, √(N)+1 samples are fully lab-tested while the remaining containers can be screened in-situ. Raman spectroscopy alone can test 89 containers in less than two hours, reducing lab load and enabling faster turnaround. Over the course of a year, the savings are startling.

The benefits of using spectroscopy for 100% laboratory testing are considerable:

- Acquisition times in less than a minute with instant results enable high throughput.
- Reduced need for personnel, consumables, and lab resources lowers costs and workloads.
- Predefined workflows simplify and streamline sampling.
- Through-packaging (unique to Raman spectroscopy) and direct contact analyses eliminate aliquots, sample preparation, and physical exposure.
- Faster raw material release with 100% shipment testing, avoiding lab backlogs.
- Minimal reliance on specialized personnel due to simplified testing with NIR and Raman spectroscopy.

Applications – selected examples for the polymer industry

- Polymer identification/verification
- Polyamide (PA)
- Polyethylene terephthalate (PET)
- Polyolefins
- Polyvinyl chloride (PVC)
- Polyurethane (PU)
- Polystyrene (PS)
- Acrylonitrile butadiene styrene (ABS)
- Polyester (PES)
- Polymethyl methacrylate (PMMA)
- Polymer applications using specialized Raman technology

Polymer identification/verification

Raman spectroscopy – The easy way to analyze polymers

SUMMARY

Raman spectroscopy is a nondestructive method for materials testing. It is easily operable by non-technical users, requires no chemicals for operation and creates no waste.

Extensive libraries of known substances support quick and accurate analysis of polymers and mixtures in any environment. Most importantly, Raman spectroscopy promotes user safety with through-container testing – all this while maintaining high material specificity.

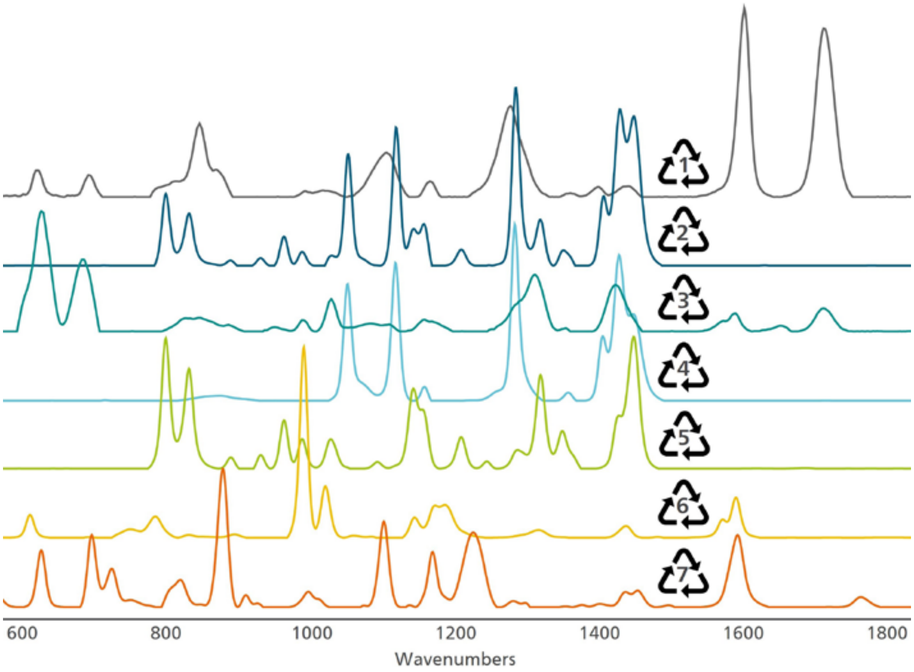
Most common plastics are excellent candidates for identification with Raman spectroscopy. The resulting fingerprint spectra of recyclable plastics permit quick, on-site identification.

EXPERIMENTAL

Handheld Raman delivers lab-quality results in non-traditional testing scenarios. Simply make contact with a material, and results are displayed within seconds. To improve plastic recycling and tackle global waste, sorting and identification of mixed and degraded materials must be addressed. Raman spectroscopy is a fast, easy, accurate, and incredibly effective solution.

RESULTS

Raman spectroscopy can characterize, sort, and determine the effects of long-term environmental exposure on plastic litter. The Raman spectra of major commercial plastics are easily distinguished, even with additives like dyes and after years of environmental exposure.



Distinct, high-resolution spectra of the most commonly recycled plastics permit easy identification with Raman spectroscopy.

Polymer identification/verification

Fast and easy polymer verification – Incoming material inspection

SUMMARY

Hauff-Technik GmbH & Co. KG in Hermaringen, Germany, is a leading manufacturer of sealing systems for cables, pipes, and building entries. To meet their high-quality standards, they verify the identity of incoming polymer pellets from various suppliers using a MIRA handheld Raman spectrometer from Metrohm.

Previously, Hauff-Technik did not perform verification, relying instead on supplier certificates, which did not align with their philosophy.

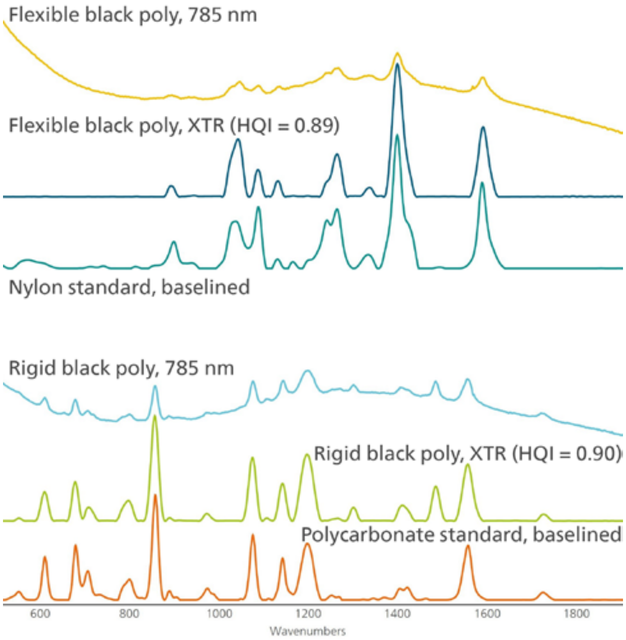
By choosing Raman spectroscopy over a costly chemical lab, they ensured fast, precise on-site knowledge of the materials they process.

EXPERIMENTAL

Colored samples sometimes cause fluorescence, which can overwhelm the Raman signal. The fluorescence extraction (or XTR®) capability of Raman spectrometers from Metrohm effectively separates the fluorescent and Raman signals, permitting accurate and reliable verification of colored pellets and fluorescent samples. The patented XTR algorithm transforms an apparently broad, featureless spectral background into clear and distinct fingerprint peaks that can then be matched to polymer spectra in the MIRA XTR library for immediate and unambiguous identification.

RESULTS

The benefits of Raman spectroscopy and the handheld MIRA XTR are very straightforward. Sample preparation and measurement requires less than two minutes. Easy operation allows even non-technical users to receive clear results. Instant chemical analysis and accurate results offer polymer processors like Hauff-Technik the ability to preserve the integrity of their products without investing in a separate QC lab.



Two examples show how XTR improves the Raman spectra of black polymer materials. EXTRacting the Raman signal from the broad fluorescent background improves spectral resolution and permits much more accurate library matching for both Nylon (top) and polycarbonate (bottom) materials.

Polymer identification/verification

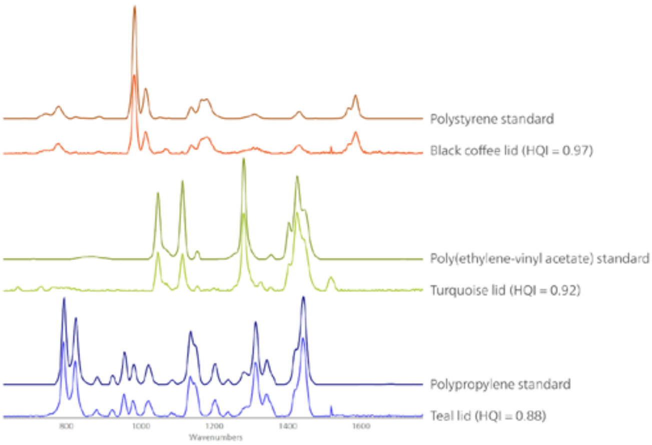
Rapid library ID of colorful polymers with Raman spectroscopy

SUMMARY

Historically, Raman spectroscopy was considered a poor technique for analysis of strongly colored materials. The fluorescence signal from dyes overwhelms the Raman response, contributing a broad, featureless background that obscures signature Raman peaks. This issue turns some users of 785 nm Raman toward 1064 nm Raman, which has its own issues regarding the analysis of colored materials.

Metrohm offers unique Raman solutions that overcome the problem of fluorescence while retaining all the benefits of 785 nm interrogation – fast, accurate, nondestructive identification of colored polymers.

This application describes the Raman identification of polystyrene (PS), polypropylene (PP), and poly(ethylene vinyl acetate) or PEVA; however this application can be easily expanded to include other common polymers like acrylonitrile butadiene styrene (ABS), polyethylene (PE), polyethylene terephthalate (PET), and polyvinyl chloride (PVC).



EXPERIMENTAL

All spectra were measured using a handheld Raman spectrometer in a point-and-shoot auto acquisition mode where integration times were determined automatically. A laser wavelength of 785 nm was used. An extensive collection of polymer standards and samples of different colors was used to build a comprehensive library within Mira Cal software. This library is used for library-correlation identification of samples. As the polymer samples in this study were very thin, spectra were recorded with the Short Working Distance (SWD) Smart Attachment from Metrohm – suitable for direct contact sampling.

RESULTS

The Raman spectra of various colored items of unknown polymer composition were collected and checked against the library. Results, including the name, CAS number, and HQI (Hit Quality Index) – a measure of the strength of correlation between sample and library spectra – display on the device within seconds. These results demonstrate the accuracy of data collected from even strongly colored samples, by comparing sample and library spectra.

A direct comparison of experimental and library spectra demonstrate the accuracy and resolution of both spectra and results. Each plastic sample matched to a single library reference with high HQI values, indicating good correlation between experimental and library spectra.

Polyamide (PA)

Quality control of PA – Multiparameter analysis with NIR spectroscopy

SUMMARY

Functional group and viscosity analysis (ASTM D789) of polyamides can be a lengthy and challenging process due to the material's limited solubility.

This study demonstrates the feasibility of using NIR spectroscopy for the simultaneous determination of key quality parameters like amine, carboxylic, and moisture content as well as the relative viscosity in polyamides.

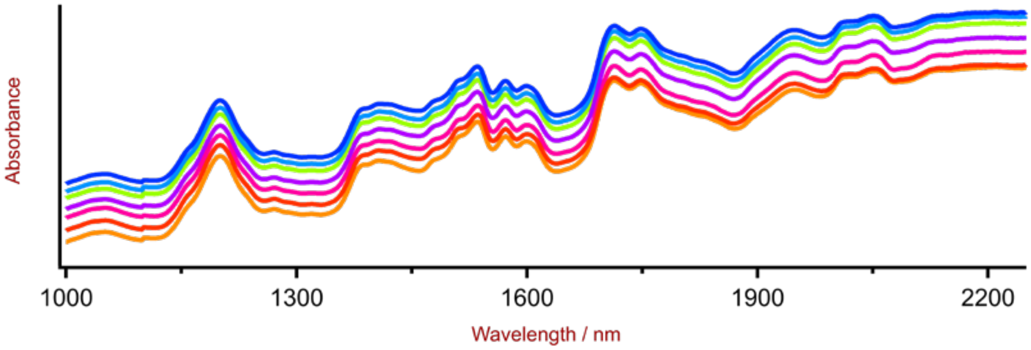
In comparison to wet chemical methods, the time to result is a major advantage of NIRS, since all parameters are determined in a few seconds.

EXPERIMENTAL

Neither sample preparation nor chemicals are needed. Polyamide pellets were measured in reflection mode over the full wavelength range (1000–2250 nm) with a Metrohm NIR Analyzer. A rotating large cup was employed to overcome the inhomogeneous distribution of the particle sizes and chemical components thanks to the multipoint measurement. Metrohm software was used for all data acquisition and prediction model development.

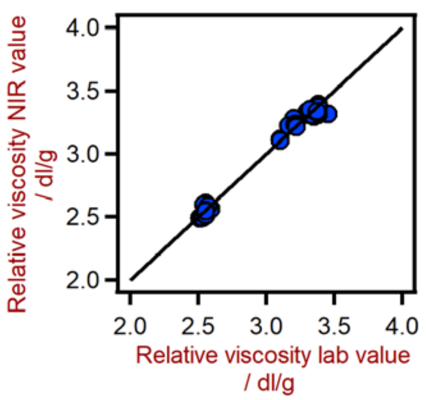
RESULTS

The obtained NIR spectra (shown below) were used to create prediction models for quantification of all mentioned polyamide QC parameters. The quality of the prediction models was evaluated using correlation diagrams which display the relationship between NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

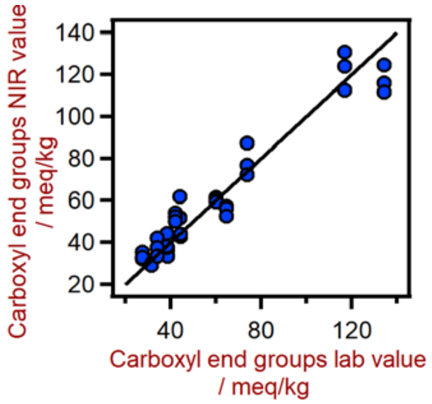


Polyamide NIR spectra. A spectra offset was applied for display reasons.

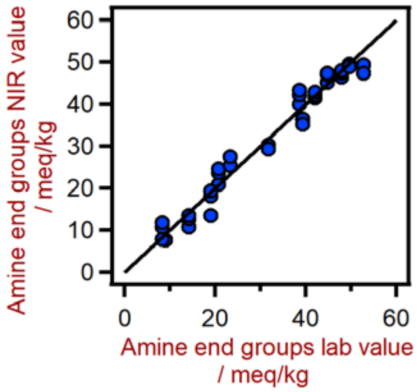
Correlation diagrams and figures of merit (FOM) for the prediction of relative viscosity, carboxyl end groups, amine end groups, and moisture in PA.



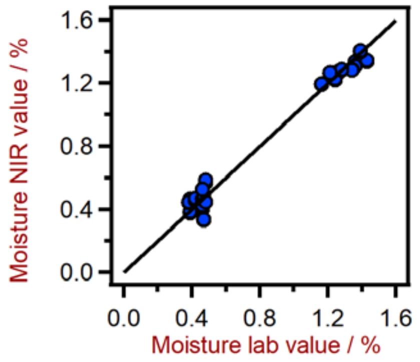
R²	SEC	SECV
0.986	0.046 (dL/g)	0.055 (dL/g)



R²	SEC	SECV
0.972	6.1 meq/Kg	11.1 meq/Kg



R²	SEC	SECV
0.981	2.5 meq/Kg	4.5 meq/Kg



R²	SEC	SECV
0.991	0.041%	0.067%

Time to result overview for different parameters using standard methods.

Parameter	Procedure	Time to result
Relative viscosity	Viscometry	~90 min (preparation) + ~1 min (viscometry)
Carboxyl end group	Titration	~90 min (preparation) + ~20 min (titration)
Amine end group	Titration	~90 min (preparation) + ~20 min (titration)
Moisture	Karl Fischer titration	~25 min (preparation) + ~5 min (Karl Fischer titration)

Polyamide (PA)

Nylon raw material ID with Raman spectroscopy

SUMMARY

Recycled plastic is increasingly utilized in the textile industry for manufacturing fashion and furniture items. Due to its ease of use and nondestructive nature, Raman spectroscopy is often used to identify and characterize recycled plastic fibers.

This Raman-based method provides detailed information about the chemical composition and structural properties of polymer fibers, enabling precise identification of polymer types such as Nylon, polyester, and polybutylene.

Sample	Component
1	Nylon – Type 6
2	Nylon – Type 66
3	Nylon – Type 6
4	Nylon – Type 6
5	Polybutylene
6	(Fluorescent)
7	(Fluorescent)
8	Nylon – Type 6
9	Polyester Elastomer
10	(Fluorescent)
11	(Fluorescent)
12	Nylon – Type 6
13 (Black)	Nylon – Type 6
13 (Gold)	Nylon – Type 6
13 (Green)	Nylon – Type 6
14	Nylon – Type 6

EXPERIMENTAL

Due to the variety of fiber colors, an i-Raman laboratory Raman system with a 1064 nm laser wavelength was used to measure all Nylon samples. This reduces the effects of fluorescence, which can overwhelm Raman signal.

The fiber samples were seated in aluminum pans and placed under the probe shaft for measurement. BWID® Identification software was used for analysis. Each sample was compared against a polymer library containing ~50 spectra.

RESULTS

Of the 14 samples measured, 10 were identified as Nylon type 6, Nylon type 66, polyester, or polybutylene. The other four samples that were not identified were black polymer fibers which did not generate any Raman scattering that could be used for identification. The results table is shown on the left.

Polyamide (PA)

Raman spectroscopy for quality assurance and quality control of Nylon production

SUMMARY

Nylon, a synthetic polyamide produced through the condensation polymerization of petrochemicals, is a strong, durable, and versatile material widely used in textiles, industrial applications, and consumer goods.

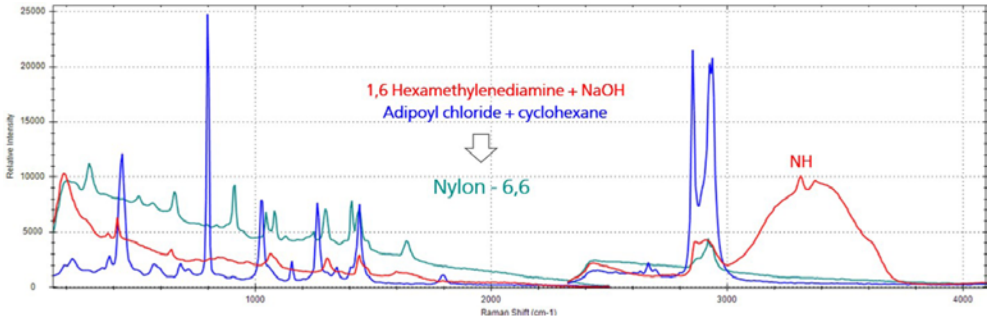
Raman spectroscopy is frequently employed in the Nylon manufacturing process to monitor polymerization, assess material crystallinity, and detect additives or impurities – ensuring consistent quality. Its ability to analyze samples without extensive preparation or destruction makes Raman spectroscopy a convenient and efficient tool for QA and QC purposes.

EXPERIMENTAL

Nylon 6,6 is synthesized through the condensation polymerization of a dicarboxylic acid (e.g., adipic acid) and a diamine (e.g., 1,6-hexamethylenediamine). This reaction produces long polyamide chains with water as a byproduct. The reaction can be safely analyzed by placing a Raman probe directly against the outside wall of a beaker containing the reactants. The final product can also be measured nondestructively to evaluate its properties.

RESULTS

Nylon is a synthetic polymer characterized by the presence of repeating amide groups (-CONH-) within its molecular structure. The first reactant, 1,6-hexamethylenediamine, exhibits distinct Raman peaks at approximately 650 and 3300 cm^{-1} (N-H vibrations), and at 1300 cm^{-1} (C-N stretching). Adipoyl chloride, the second reactant, contains peaks around 500–750 cm^{-1} (C-Cl vibrations) and 1790 cm^{-1} (C=O stretching) in the Raman spectrum. Nylon exhibits characteristic Raman bands for N-H, C-N, and C=O which shift due to changes in the molecular environment, chemical bonding, and intermolecular interactions. These shifts permit effective assessment of crystallinity, impurities, and batch-to-batch consistency for quality control.



Raman band of the reaction mix (blue and red) and Nylon 6,6 (green).

Polyethylene terephthalate (PET)

Quality control of first-use (virgin) PET with NIR spectroscopy

SUMMARY

Determining the diethylene glycol content (DEG), isophthalic acid content (IA), intrinsic viscosity (IV) (according to ASTM D4603), and the acid number (AN) in polyethylene terephthalate is a lengthy and challenging process. This is not only due to the sample's limited solubility, but also because of the need to use different analytical methods (e.g., HPLC-MS, HPLC, viscometry, titration).

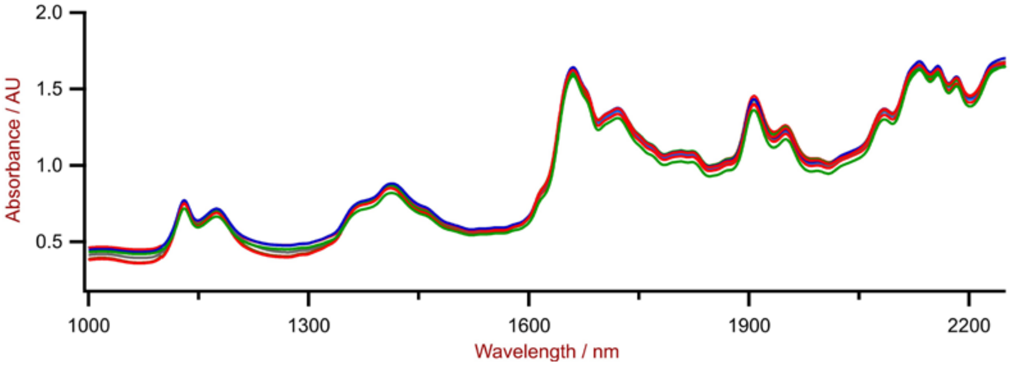
NIR spectroscopy provides a fast, cost-efficient solution for the simultaneous determination of these different quality control parameters in virgin PET. Unlike other wet chemical methods, NIR spectroscopy can analyze PET samples in seconds without sample preparation or using chemical reagents.

EXPERIMENTAL

PET pellets were measured with a Metrohm NIRS Analyzer in reflection mode over the wavelength range of 1000–2250 nm. A rotating large cup was employed to compensate for the distribution of varied particle sizes and chemical components. Samples were measured without any preparation step. Metrohm software was used for all data acquisition and prediction model development.

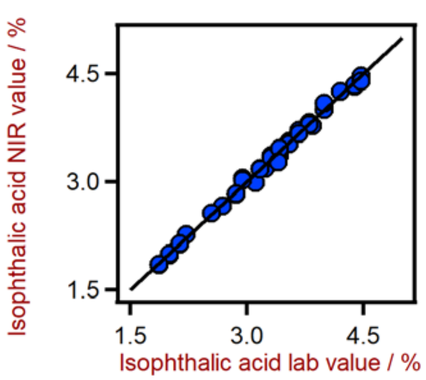
RESULTS

The obtained NIR spectra were used to create prediction models for quantification of DEG, IA, IV and AN in virgin polyethylene terephthalate. The quality of the prediction models was evaluated using correlation diagrams which display the correlation between NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

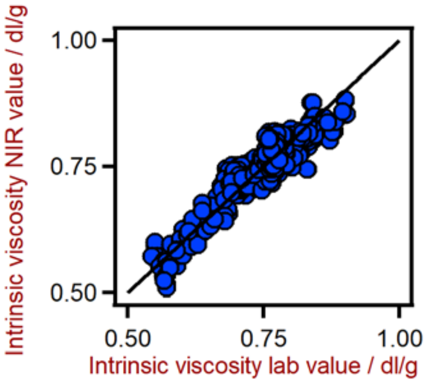


Polyethylene terephthalate NIR spectra. A spectra offset was applied for display reasons.

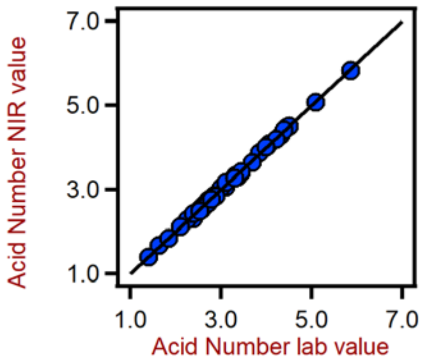
Correlation diagrams and figures of merit (FOM) for the prediction of isophthalic acid, intrinsic viscosity, acid number, and diethylene glycol in PET.



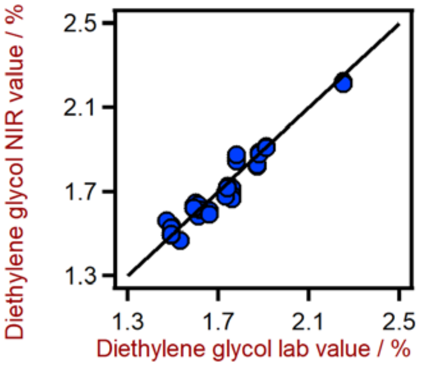
R^2	SEC	SECV
0.995	0.059%	0.085%



R^2	SEC	SECV
0.873	0.0236 dL/g	0.0238 dL/g



R^2	SEC	SECV
0.991	0.093	0.143



R^2	SEC	SECV
0.931	0.052%	0.066%

Time to result overview for different parameters using standard methods.

Parameter	Method	Time to result
Diethylene glycol	Extraction + analysis HPLC-MS	~45 min (preparation) + ~40 min (HPLC)
Isophthalic acid	Dissolve + HPLC	~45 min (preparation) + ~40 min (HPLC)
Intrinsic viscosity	Dissolve + viscometry	~90 min (preparation) + ~1 min (viscometry)
Acid number	Dissolve + titration	~90 min (preparation) + ~10 min (titration)

Polyethylene terephthalate (PET)

Intrinsic viscosity (IV) determination of recycled PET with NIR spectroscopy

SUMMARY

Determining the intrinsic viscosity of recycled polyethylene terephthalate (rPET) is a time-consuming and challenging process. This is especially true if the sample is highly crystalline and needs to be dried before the analysis with a glass capillary as mentioned in ASTM D4603. The drying process often takes several hours until a constant weight is reached, and high crystallinity hinders solubility.

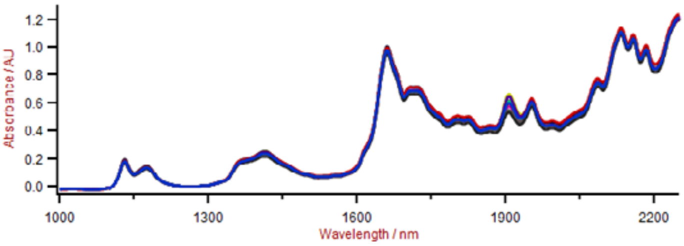
With near-infrared (NIR) spectroscopy, the intrinsic viscosity of rPET can be determined in a few seconds without any sample preparation.

EXPERIMENTAL

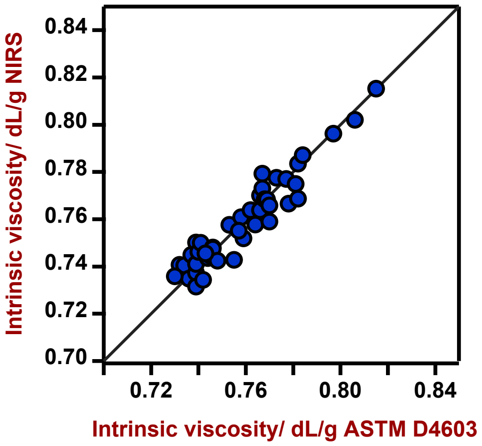
48 samples of recycled PET with varying IV were measured on a Metrohm NIR Analyzer, as well as with a glass capillary viscometer according to ASTM D4603. All NIRS measurements were performed in reflection mode using the large cup and multipoint measurement to reduce the influence of the particle size distribution of the polymer pellets. Data acquisition and prediction model development were performed with Metrohm software.

RESULTS

All 48 measured NIR spectra were used to create a prediction model for quantification of intrinsic viscosity in rPET. The quality of the prediction model was evaluated using correlation diagrams which display a high correlation between the NIR prediction and the reference viscosity values. The respective figures of merit (FOM) shown below display the expected precision and confirm the feasibility of using NIRS during routine analysis.



Recycled polyethylene terephthalate NIR spectra.



R^2	SEC	SECV
0.9061	0.0068 (dL/g)	0.0084 (dL/g)

Polyethylene terephthalate (PET)

Inline QC of bio-PET pellets with NIR spectroscopy

SUMMARY

Inline quality control using robust spectroscopic technologies and predictive models allows manufacturers to accurately measure and control moisture, melt flow, and melt index during bio-PET production.

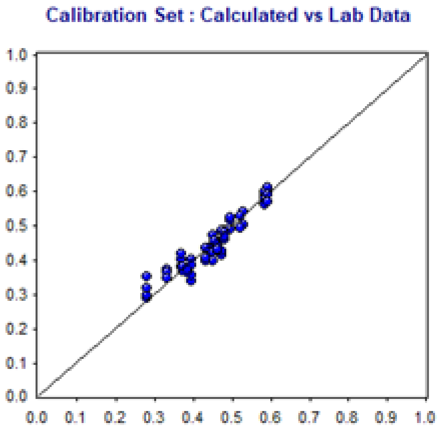
By replacing slow offline testing methods with real-time insights, producers can minimize waste, enhance quality, and reduce operational costs, resulting in a more efficient and sustainable manufacturing process.

EXPERIMENTAL

The sample is measured prior to bagging while the pellets fall down via gravity. Inline analysis is possible using a NIR spectrometer with a specially designed probe for polymer pellets. The probe's measuring area is sufficiently large to analyze a representative sample.

RESULTS

NIR model for inline moisture determination in bio-PET pellets.



Parameters to monitor in an industrial PET reactor with inline NIR spectroscopy.

Parameter	Concentration	Precision
Moisture	0.2–0.7 wt%	± 0.05 wt%
Melt flow	2–15 g/cm^3	± 0.5 g/cm^3
Melt index	1–1.3 g/10 min	± 0.005 g/10 min

Polyolefins

Density of polyolefins as measured by near-infrared spectroscopy (NIRS)

SUMMARY

Determination of the density of polyethylene (PE) as per ASTM D792 is normally a challenging procedure due to reproducibility difficulties. Since this parameter is most frequently used to determine the PE type, alternative methods which are sensitive to molecular structure (such as FT-IR spectroscopy) are also common. However, measurement via FT-IR can also be problematic when larger sample sizes must be analyzed due to sample inhomogeneity.

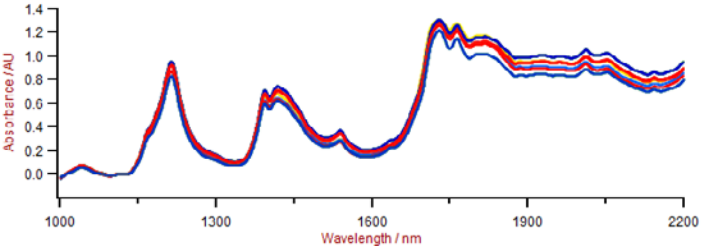
This study shows the feasibility of NIR spectroscopy for the analysis of density in polyethylene granulates. Compared to the standard method, NIRS analysis shows a lower prediction error when air bubbles are present in the polymer pellets. In addition, sample handling with NIRS is easier to perform and therefore less error-prone.

EXPERIMENTAL

Polyethylene samples (n = 29) with varying density were measured in reflection mode over the full wavelength range (1000–2250 nm) as well as with the buoyancy method described in ASTM D792. Samples were measured as-is without any preparation. A rotating large cup was employed to overcome the distribution of varied particle sizes and chemical components. This allowed automated measurements at different sample locations for a reproducible spectrum acquisition. Metrohm software was used for all data acquisition and prediction model development.

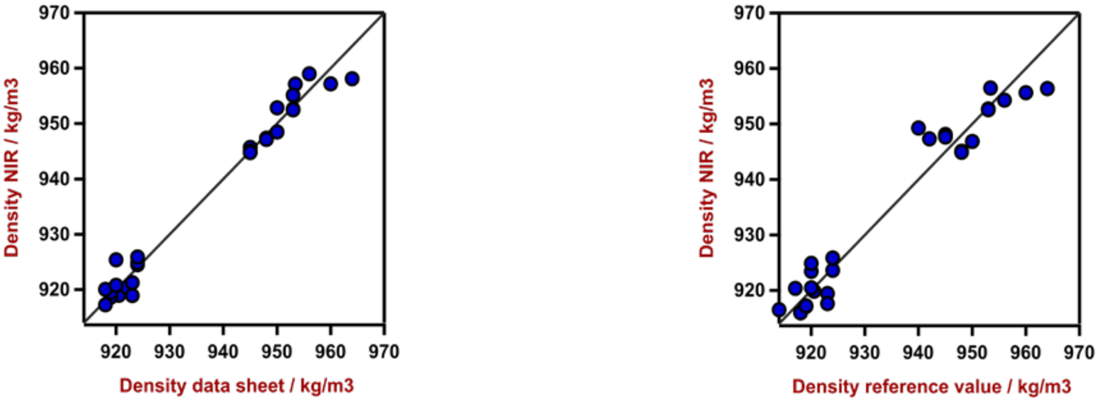
RESULTS

The obtained NIR spectra were used to create a prediction model for quantification of the density of PE pellets. The density of the PE pellets was also measured with a density balance in the laboratory. Compared to the NIRS data, these results deviated even more from the reference values given by the supplier. This is due to the appearance of air bubbles in some of the polymer pellets. To verify the quality of the prediction model, correlation diagrams were created which display the correlation between the NIR prediction and primary method values received from the supplier. The respective figures of merit (FOM) are also given for both correlation diagrams.



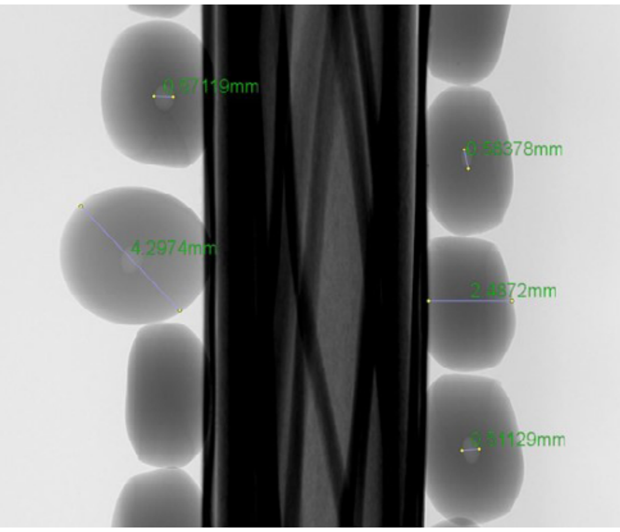
NIR spectra of several polyethylene (PE) samples with varying density.

Correlation diagrams and figures of merit (FOM) for the prediction of density in polyethylene using NIR spectroscopy.



R²	SEC	SECV
0.979	2.48 Kg/m³	3.42 Kg/m³

R²	SEC	SECV
0.948	3.95 Kg/m³	6.00 Kg/m³



A computerized tomography (CT) scan of polyethylene pellets showing air bubbles inside some of the polymer granulate.

Comparison of density values in PE from the producer vs. prediction with NIRS and with a density balance according to ASTM D792.

Sample	Density – producer	Density – lab balance	Density – NIRS	Air bubbles present
1	953 kg/m³	941 kg/m³	952 kg/m³	Yes
2	950 kg/m³	935 kg/m³	953 kg/m³	No
3	918 kg/m³	917 kg/m³	915 kg/m³	Yes

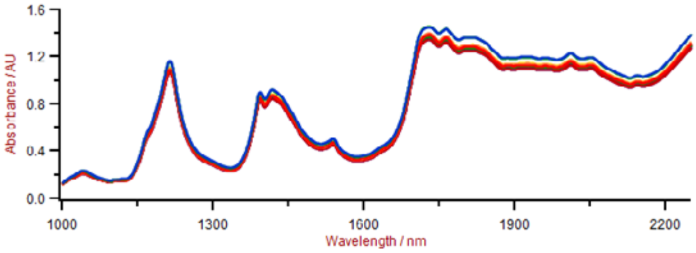
Polyolefins

Ash determination in polyethylene using NIR spectroscopy

SUMMARY

In order to improve certain characteristics in polymers, various fillers are added to resins. One important filler used is ash, which mainly consists of silicon-, magnesium-, and iron oxide. The ash content can vary in the product (depending on the particle size and the desired properties) from 0.01 wt% up to 10 wt%.

The standard test method for ash content analysis is thermogravimetric analysis (TGA). Although TGA is easy to perform, it is time-intensive (approximately two hours) and requires the use of nitrogen gas. With near-infrared spectroscopy (NIRS), polyethylene ash content can be measured in just a few seconds.



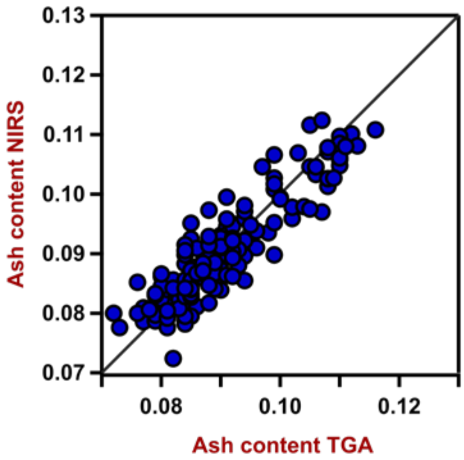
Near-infrared spectra generated from various polyethylene (PE) samples in this study.

EXPERIMENTAL

154 polyethylene (PE) samples with varying ash content (0.07% to 0.12%) were analyzed on a Metrohm NIR Analyzer equipped with a large cup. All measurements were performed in rotation to average the subsample spectra. This setup reduces the influence of the particle size distribution of the polymer pellets. Metrohm software was used for all data acquisition and prediction model development.

RESULTS

The obtained NIR spectra were used to create a prediction model for ash content in PE samples. To verify the quality of the prediction model, a correlation diagram (shown below) was created to determine the correlation between the NIR prediction and primary method (TGA) values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis of ash content in polyethylene.



R^2	SEC	SECV
0.77	0.0055%	0.066%

Polyolefins

Polyethylene content determination by NIRS in polypropylene pellets

SUMMARY

Recycling polyolefins is problematic, since it is challenging to separate polyethylene from polypropylene using sink float separation. Increasing polyethylene (PE) content in polypropylene (PP) leads to a poor interfacial bond strength which may negatively impact the mechanical properties of the final molded product.

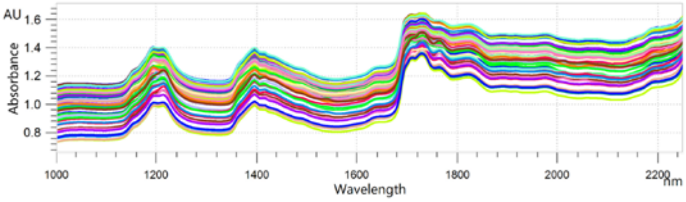
Differential Scanning Calorimetry (DSC) is the standard method of analysis. However, the DSC method can be challenging to perform and takes approximately one hour per sample. In contrast, the analysis of PE content in PP using near-infrared spectroscopy is performed in just seconds.

EXPERIMENTAL

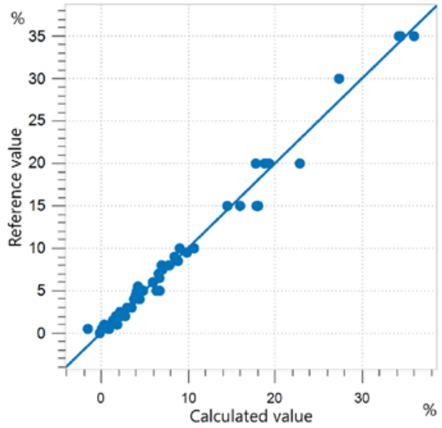
Different polymer blends (n = 54) with varying polyethylene (PE) content from 0.5–35% were produced in a compounder. The PE content was determined by weight before the polymer blends were produced. All measurements on the OMNIS NIR Analyzer Solid were performed in rotation using a large cup to average the subsample spectra. Metrohm's OMNIS software was used for all data acquisition and prediction model development.

RESULTS

The obtained NIR spectra were used to create a prediction model for PE content in PP. The correlation diagram (shown below) displays the relation between the NIR prediction and the reference values in the range of 0.5–35.0% PE content. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.



NIR spectra from various polymer blends tested in this study. A spectra offset was applied for display reasons.



R^2	SEC	SECV
0.987	0.94%	1.10%

Polyolefins

Quality control of polypropylene – Melt flow rate determination with NIRS

SUMMARY

As a general-purpose resin, polypropylene (PP) is widely used during electronics manufacturing and construction, as well as in packaging materials due to its insulating and processing properties. PP resins must first be melted in order to be formed into the intended shape, and therefore flow properties are important characteristics which affect the production process.

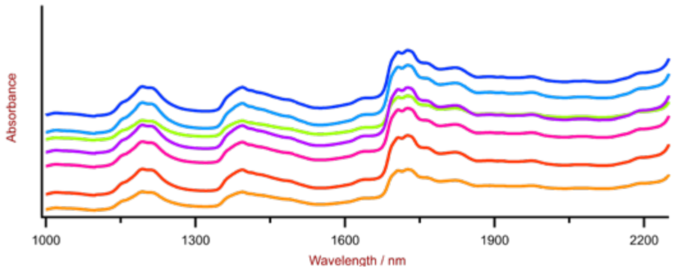
One parameter that describes the flow characteristics is the melt flow rate (MFR). MFR is a measure of the mass of material that extrudes from the die over a given period of time. The standard procedure (ASTM D1238) requires a significant amount of work as the sample must be packed, preheated, and cleaned. This takes approximately 20 minutes. Reduction of analysis time and workload is a major advantage of using NIR spectroscopy for MFR analysis.

EXPERIMENTAL

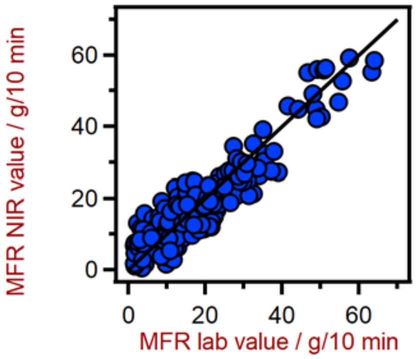
PP pellets were measured with a Metrohm NIR Analyzer in reflection mode over the wavelength range of 1000–2250 nm. To minimize particle size effects, a rotating large cup was employed. This accessory enables an automated measurement at different sample locations for a reproducible spectrum acquisition by averaging the spectra. Samples were measured without any sample preparation. Data acquisition and prediction model development were performed with software from Metrohm.

RESULTS

The obtained NIR spectra were used to create a prediction model for quantification of MFR in PP. The quality of the prediction model was evaluated using a correlation diagram (shown below) which displays the correlation between NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.



Polypropylene (PP) NIR spectra. A spectra offset was applied for display reasons.



R^2	SEC	SECV
0.865	4.99 g/10 min	7.00 g/10 min

Polyolefins

Identification of HDPE, LDPE, and PP with NIR spectroscopy

SUMMARY

Identification of individual polymers with FT-IR spectroscopy can be a challenge due to sample inhomogeneity, especially when larger sample sizes need to be analyzed.

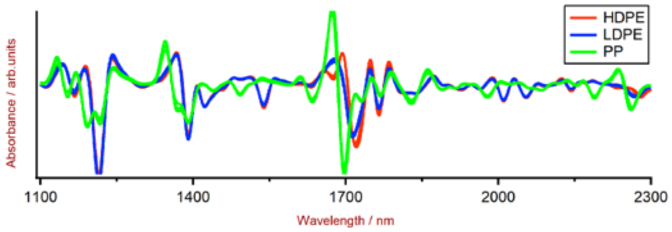
NIR spectroscopy provides a reliable and fast solution for the identification of high-density polyethylene (HDPE), low-density polyethylene (LDPE), and polypropylene (PP). With no sample preparation or chemicals needed, NIR spectroscopy allows the identification of larger inhomogeneous sample amounts in just seconds.

EXPERIMENTAL

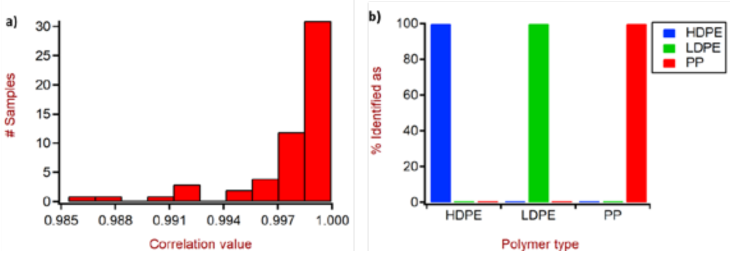
HDPE, LDPE, and PP pellets were measured in reflection mode with a Metrohm NIR Analyzer. A rotating large cup was employed to overcome the distribution of varied particle sizes and chemical components. This allowed automated measurements at different sample locations for a reproducible spectrum acquisition. Samples were measured as-is without any preparation steps. Metrohm software was used for all data acquisition and prediction model development.

RESULTS

A correlation algorithm was applied to the measured NIR spectra to create a prediction model for the identification of the individual polymer types. The quality of the prediction was evaluated using built-in statistical tools, which display the correlation value distribution between NIR prediction and product class. The high correlation values of 0.985 and above, together with the fact that no polymer was misidentified, highlight the feasibility of NIRS for identification of different polymer types.



Selection of HDPE, LDPE, and PP NIR spectra (pretreated with a 2nd derivative). Clear differences for the different polymer types are visible in the wavelength region around 1700 nm.



(a) Correlation value histogram for the identification of the individual polymer types. (b) All polymer types in the sample set were identified correctly.

Polyolefins

Moisture content and rheology determination of fluorinated polyolefins using NIRS

SUMMARY

Ethylene tetrafluoroethylene (ETFE) is a partially fluorinated copolymer of ethylene and tetrafluoroethylene. It is a melt processable polymer, designed to have high corrosion resistance and strength over a wide temperature range. Key quality parameters to monitor during the production of fluorinated polyolefins include melt flow rate (MFR) and moisture content.

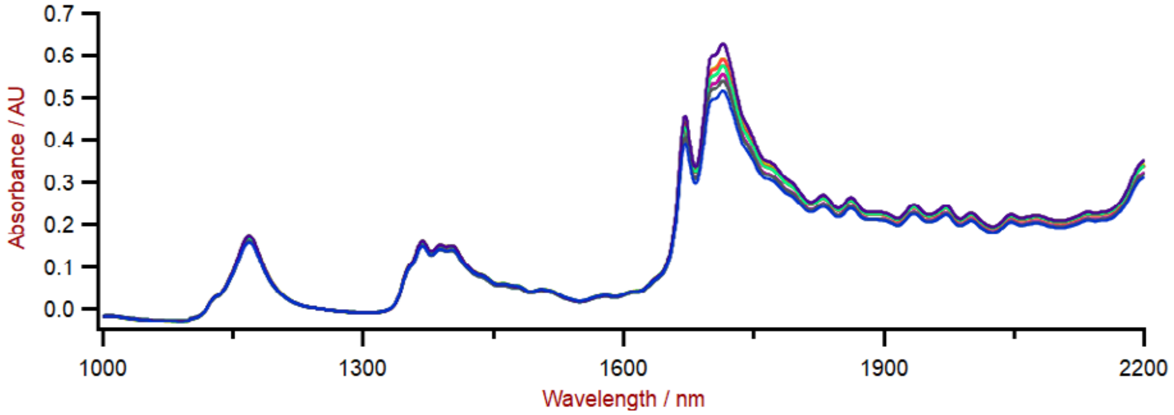
The measurement of MFR and moisture in ETFE is usually time-consuming and requires chemicals. Compared to wet chemical methods, running costs are significantly lower when using near-infrared spectroscopy (NIRS). NIRS offers rapid and reliable prediction of multiple parameters simultaneously without any sample preparation or cleaning procedures.

EXPERIMENTAL

ETFE pellet samples (n = 273) with varying MFR and moisture content were measured on a Metrohm NIR Analyzer in reflection mode. All measurements were performed in rotation using a large cup to reduce the stray light effect caused by varying particle sizes. Metrohm software was used for all data acquisition and prediction model development.

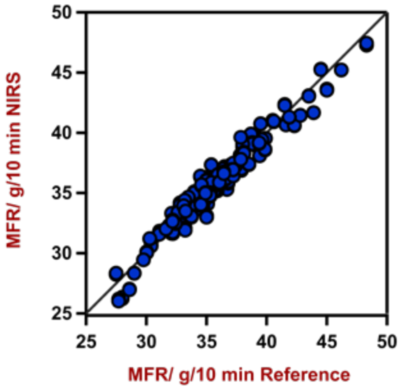
RESULTS

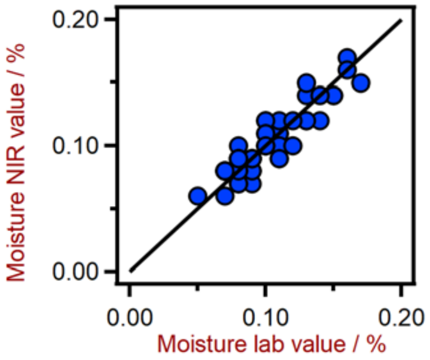
The obtained NIR spectra were used to create prediction models for quantification of all quality control parameters mentioned in this study. The quality of the prediction models was evaluated using correlation diagrams which display the relationship between NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.



NIR spectra of the ETFE samples measured in this study.

Correlation diagrams and figures of merit (FOM) for the prediction of melt flow rate (MFR) and moisture content in ETFE.





R^2	SEC	SECV
0.9413	0.96 g/10 min	0.98 g/10 min

R^2	SEC	SECV
0.840	0.011%	0.012%

Time to result overview for different parameters using standard methods.

Parameter	Method	Time to result
Melt flow rate	Melt flow indexer	~20 min (measurement and cleaning)
Moisture	Karl Fischer titration	~3–5 min

Polyolefins

Raman identification of multicolored polypropylene with fluorescence-rejection

SUMMARY

Approximately 10% of Raman-active materials fluoresce when examined with 785 nm Raman [6]. This climbs to nearly 100% for deeply colored materials. Fluorescence overwhelms the Raman signal and can prevent positive identification of the target substance. Often, weak Raman features can still be observed in fluorescent materials, but fluorescence mitigation is crucial for accurate library matching.

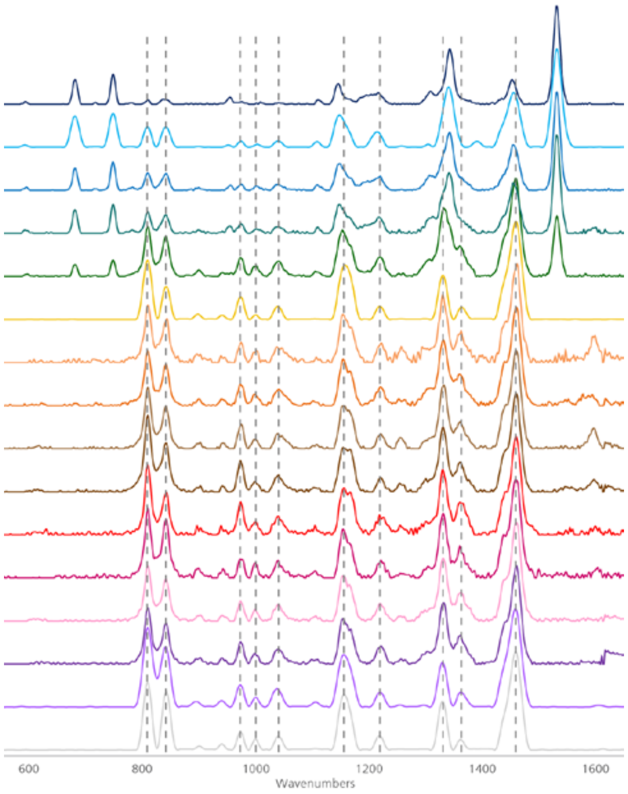
The ability of the XTR® algorithm to remove the fluorescent background and reveal a high-resolution spectrum of colored plastics is particularly impressive, since the spectra of uncolored polymers exhibit some inherent fluorescence, as many hydrocarbon-based materials often do.

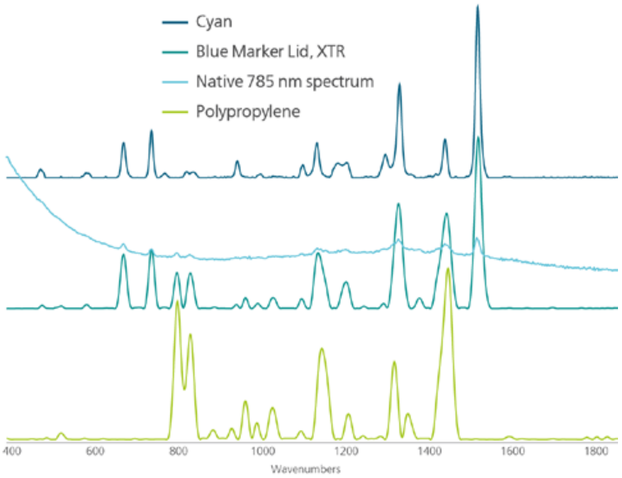
EXPERIMENTAL

A series of strongly colored polypropylene (PP) marker casings were directly tested at the surface with a 785 nm handheld Raman spectrometer equipped with the XTR algorithm. Similarly to Sequentially Shifted Excitation (SSE), XTR uses multiple shifted spectra that are generated by internal algorithms during the experiment to distinguish the Raman shift from fixed fluorescence, permitting the fluorescence component to be isolated and extracted [**7**]. In a secondary automated process in real time, the Raman data is optimized through an iterative process. After identification and elimination of the fluorescence component, only a pure, unobstructed Raman spectrum remains.

RESULTS

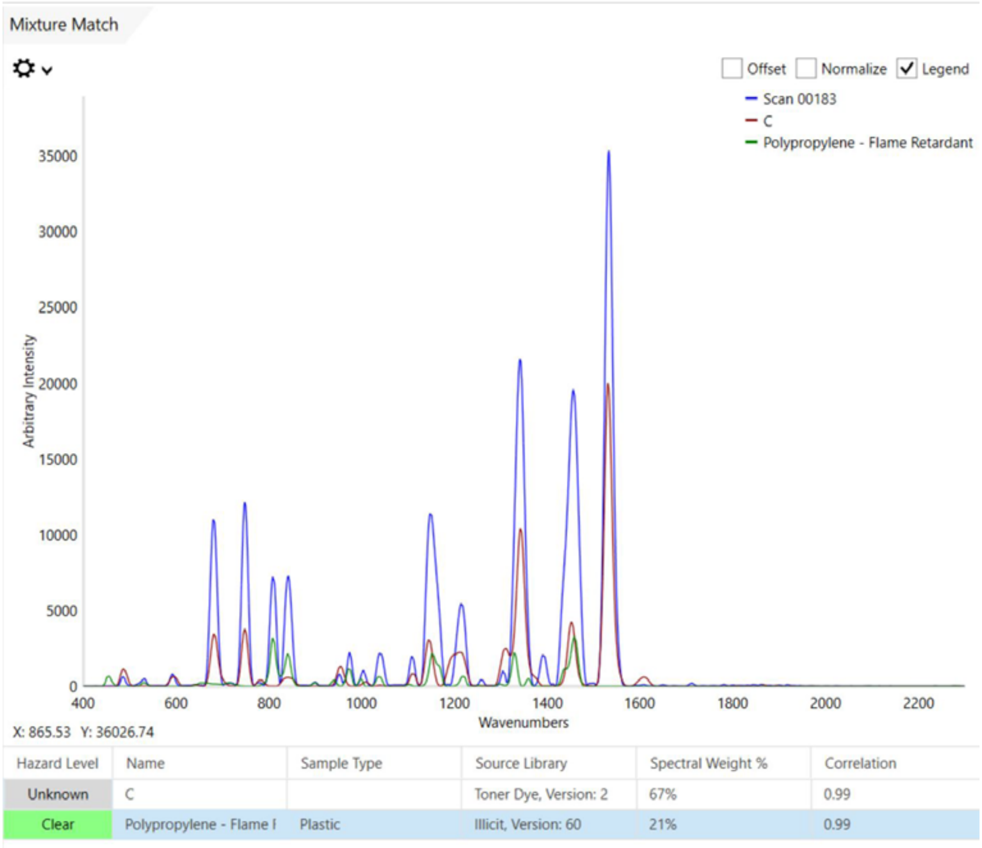
The ability of XTR to return baselined spectra containing the signature Raman fingerprint peaks for a given substance is demonstrated across a variety of colors. The polypropylene spectrum (indicated by dashed lines in the figure on the next page) is obvious in each of the samples measured. Only very saturated colors containing cyan pigment showed strong spectral contribution from the dye. Interestingly, the cyan signal was the main spectral contributor for only the very dark blue polypropylene. Despite significant contribution from both the dye and high levels of fluorescence, XTR permitted identification of both material and colorant.





Despite significant spectral contribution from cyan pigment, XTR produces a baselined, high-resolution spectrum that contains peaks from both dye and polymer. The native Raman spectrum reveals the striking benefits of XTR.

The color of the XTR spectra here coordinates with the color of the PP sample tested. The signature Raman peaks for polypropylene are indicated by dashed lines.



☐ Offset ☐ Normalize ☑ Legend

X: 865.53 Y: 36026.74

Hazard Level	Name	Sample Type	Source Library	Spectral Weight %	Correlation
Unknown	C		Toner Dye, Version: 2	67%	0.99
Clear	Polypropylene - Flame I	Plastic	Illicit, Version: 60	21%	0.99

XTR permits confident identification of both colorant and polymer, despite significant spectral contribution from the dye and high levels of fluorescence.

Polyvinyl chloride (PVC)

Quality control of PVC foils – Foil thickness determination by NIRS

SUMMARY

PVC (polyvinyl chloride) foils with a PVDC (polyvinylidene chloride) coating are often used for high performance films like pharmaceutical blister packs or in food packaging. In multi-layer blister films, the PVC serves as the thermoformable backbone structure, whereas the PVDC coating acts as a barrier against moisture and oxygen. The Water Vapor Transmission Rate (WVTR) and Oxygen Transmission Rate (OTR) are influenced by the composition and the thickness of the coating.

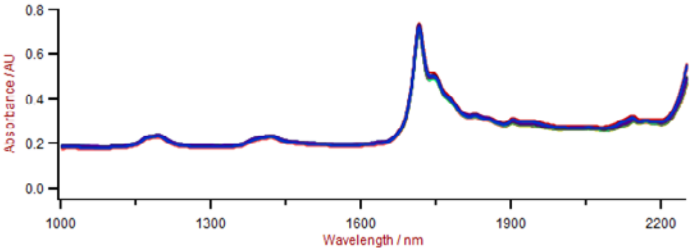
A fast way to monitor PVDC coating thickness is with near-infrared spectroscopy (NIRS). Results are provided in a few seconds, indicating when adjustments are necessary in the polymer production process. NIRS enables fast, reliable results without any sample preparation, and therefore represents a suitable method to measure PVDC layer thickness.

EXPERIMENTAL

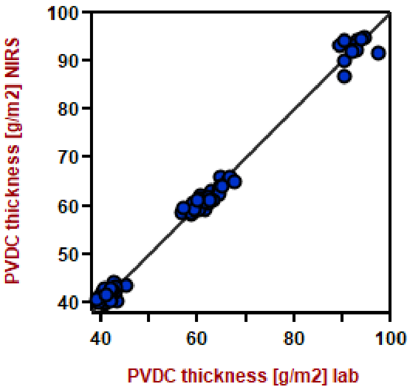
Several 250 µm PVC foils coated with a PVDC layer of varying thickness (40 g/m^2, 60 g/m^2, 90 g/m^2) were measured on a Metrohm NIRS Analyzer. The measurements were carried out in transflection mode using a diffuse reflector with 1 mm pathlength. This ensures that the spectral pathlength is constant while enhancing the spectral signal. Data acquisition and prediction model development were performed with Metrohm software.

RESULTS

All 68 measured NIR spectra were used to create a prediction model for quantification of PVDC layer thickness. The quality of the prediction model was evaluated using a correlation diagram (shown below) which displays a very high correlation between NIR prediction and the reference values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.



PVC NIR spectra from this study.



R^2	SEC	SECV
0.992	1.7 g/m^2	1.9 g/m^2

Polyvinyl chloride (PVC)

Quality control of PVC granulate – NIRS molecular weight determination

SUMMARY

Compared to other olefin-derived plastics that only contain carbon and hydrogen atoms in their chemical structure, PVC (polyvinyl chloride) has unique properties. Some features of PVC include increased chemical and mechanical stability as well as fire retardant properties. The molecular weight of the polymer significantly influences these properties. Molecular weight is defined in this case as the average weight of the molecules that make up a polymer, and this value gives an indication of the length of the polymer chains. To monitor the PVC quality, it is important to measure the molecular weight during the production process.

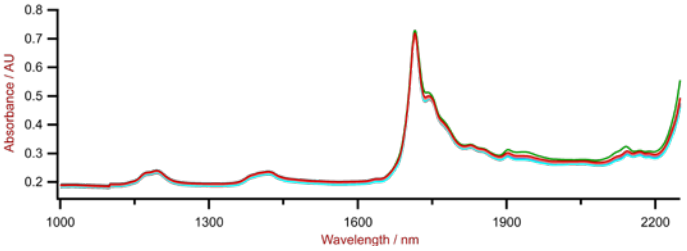
Size-exclusion chromatography (SEC) is the standard method to determine PVC molecular weight. However, SEC is time-intensive and requires trained personnel to perform. Near-infrared spectroscopy is a faster and cheaper alternative method and has proven itself suitable to measure PVC molecular weight.

EXPERIMENTAL

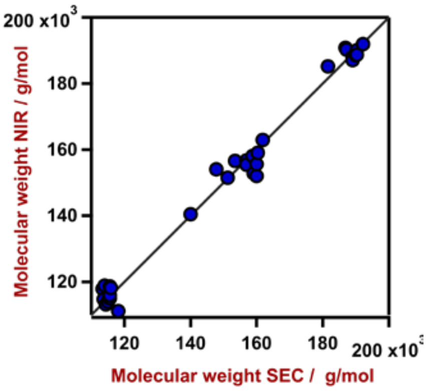
PVC samples (n = 33) with varying molecular weights from 113,000–192,000 g/mol were measured on a Metrohm NIR Analyzer. All NIR measurements were done in reflection mode using the large cup and multipoint measurement to reduce the influence of the particle size distribution of the granulates. Data acquisition and prediction model development were performed with Metrohm software.

RESULTS

All 33 measured NIR spectra were used to create a prediction model for quantification of molecular weight. The quality of the prediction model was evaluated using a correlation diagram (shown below) which displays a high correlation ($R^2 = 0.988$) between the NIR prediction and the reference molecular weight values. The respective figures of merit (FOM) display the expected precision and confirm the feasibility during routine analysis.



NIR spectra of the PVC samples measured in this study.



R^2	SEC	SECV
0.988	3640 g/mol	5375 g/mol

Polyvinyl chloride (PVC)

Determining phthalates in plastic toys with Raman spectroscopy

SUMMARY

Phthalates are toxic substances that may cause harm to reproductive organs. They are sometimes added to PVC (polyvinyl chloride) as softeners, especially in plastic toys meant for children.

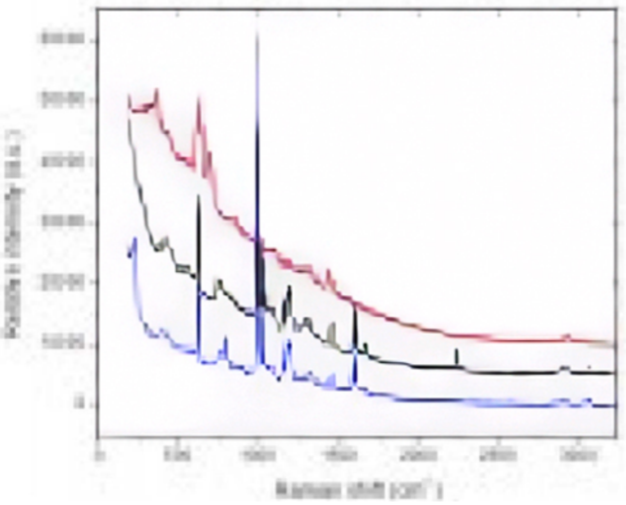
Since phthalates and PVC are mixed together rather than chemically bonded, these toxic compounds can leach into liquids or evaporate over time. The phthalate concentration in PVC can reach 30%. This is a major concern for the safety of small children who put toys in their mouths. Raman spectroscopy can be used to identify toys laced with phthalates and determine the concentration when present.

EXPERIMENTAL

A high-performance Raman spectrometer was used to analyze phthalates in plastic children's toys. Three different toys from three different manufacturers were tested.

RESULTS

The common Raman spectral peaks indicate the existence of phthalates in two of the tested toys. Further analysis proved that the Raman system used in this study can measure concentrations of phthalates as low as 0.5% in plastic toys.



Raman spectral overlay of three different toys from three different manufacturers. The common Raman peaks for phthalate at 1040 cm^{-1} and 1729 cm^{-1} are observed in two of the Raman spectra (black and blue), indicating existence of phthalates in these two toys. The phthalate Raman peaks are not apparent in the third Raman spectrum (red), indicating that the toy represented by the red spectrum has little to no phthalate content.

Polyurethane (PU)

Quality control of polyols and isocyanates with NIR spectroscopy

SUMMARY

Test methods for polyurethane (PU) raw materials (i.e., isocyanates and polyols, ASTM D7252) are challenging procedures due to the reactivity of isocyanates with atmospheric moisture, as well as their toxicity. HPLC, which is typically used for this kind of analysis, requires sample preparation and chemical reagents with each measurement taking up to 20 minutes to complete. In addition, toxic and corrosive chemicals such as *p*-toluenesulfonyl isocyanate (TSI) and tetrabutylammonium hydroxide are used for the analysis of hydroxyl number (OH number) in polyols by titration according to ASTM D4274.

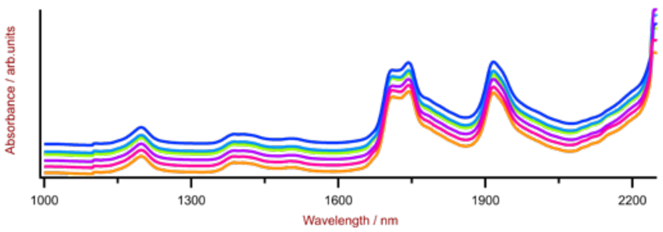
Near-infrared spectroscopy provides a faster, safer, more cost-efficient solution for the determination of free isocyanate content (NCO content) in isocyanates and hydroxyl (OH) number in polyols. Analysis is complete in a few seconds without sample preparation or chemicals.

EXPERIMENTAL

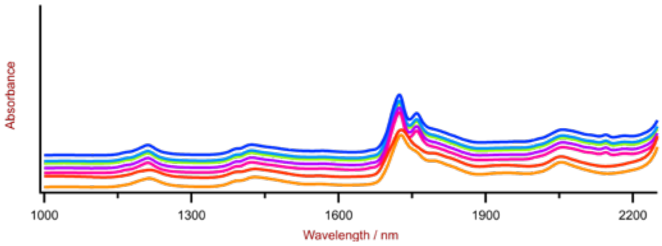
Isocyanate samples were measured with a Metrohm NIR Analyzer in transmission mode at 30 °C using 8 mm disposable vials over the wavelength range of 1000–2250 nm. Polyol samples were measured under the same conditions with the exception of using 4 mm disposable vials to avoid saturated spectra. Data acquisition and prediction model development were performed with Metrohm software.

RESULTS

The obtained NIR spectra of isocyanate samples were used to create a prediction model for quantification of NCO content, and the obtained NIR spectra of polyols were used to create a prediction model for quantification of OH content. The quality of the prediction models was evaluated using correlation diagrams which display the correlation between NIR prediction and primary method values. The respective figures of merit (FOM) display the expected precision of a prediction during routine analysis.

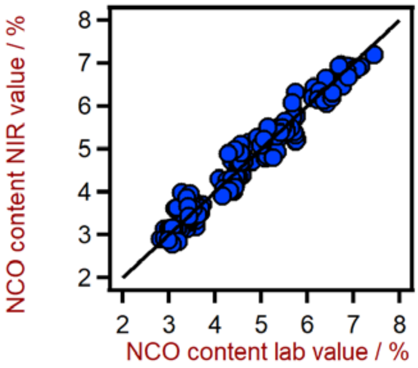


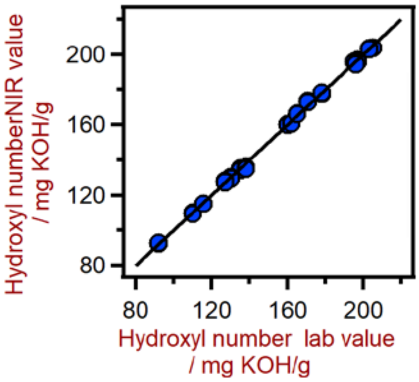
Isocyanate NIR spectra obtained in this study. For display reasons a spectra offset was applied.



Polyol NIR spectra obtained in this study. For display reasons a spectra offset was applied.

Correlation diagrams and figures of merit (FOM) for the prediction of NCO content in isocyanates and hydroxyl number in polyols.



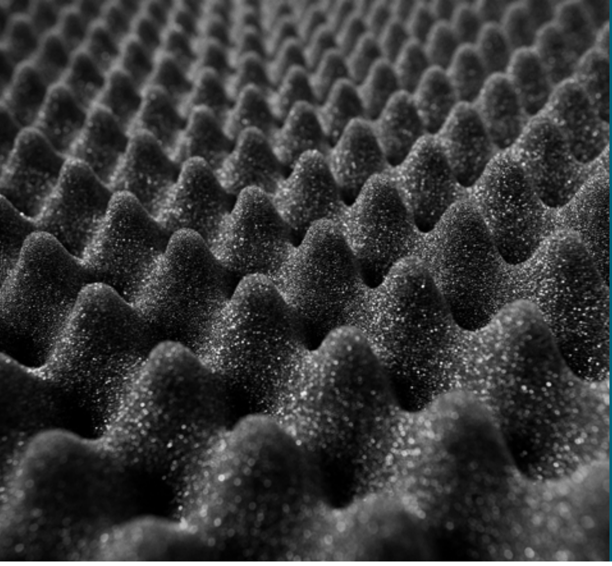


R^2	SEC	SECV
0.968	0.24 (%)	0.26 (%)

R^2	SEC	SECV
0.998	1.28 mg KOH/g	1.42 mg KOH/g

In comparison to wet chemical methods, running costs are significantly lower when using NIR spectroscopy. Additionally, there is no need to use dangerous chemicals for the analysis with NIRS as with ASTM D4274.

	Lab method	NIR method
Number of analyses (per day)	10	10
Cost of operator (per hour)	$25	$25
Costs of consumables and chemicals OH number	$6	$1
Time spent per analysis	5 min	1 min
Total running costs (per year)	$18,188	$2,063



Polyurethane (PU)

Inline monitoring of free isocyanate content in polyurethane with NIRS

SUMMARY

Polyurethanes (PU) are synthetic polymers used to create a wide range of products, from flexible or rigid foam structures to safety features in cars. The physical properties of PU products can vary widely.

PU is formed by reacting liquid di/polyisocyanates and polyols with a catalyst and additives. Therefore, determining the free isocyanate content (%NCO) in the manufacturing process is a critical parameter to optimize production.

Generally, spot samples are taken and analyzed in a laboratory by titration methods which are slow and generate waste. Inline monitoring of %NCO in real time with near-infrared spectroscopy is a safer and more efficient way to monitor and improve PU production and reduce waste.

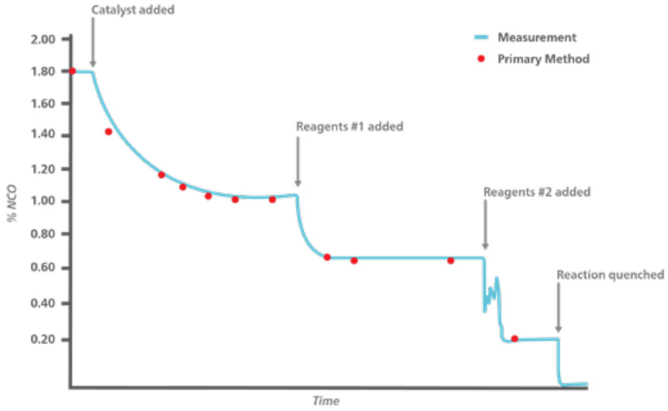
The micro interactance immersion probe used in this application study.

EXPERIMENTAL

Wavelength range used: 1950–2080 nm. Inline analysis is possible using the properties of transflectance and the micro interactance immersion probe (shown below). The sample flows through the gap between the probe body and high-energy mirror tip, and adjusting the mirror tip defines the pathlength (equal to two times the gap) for analysis.

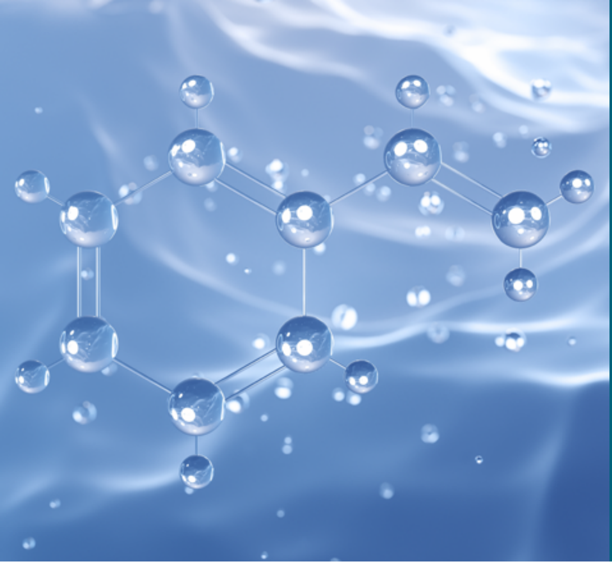
RESULTS

This chart illustrates how different mixing steps in the PU production process can be shortened. Spot sampling (red dots) can miss certain events, while constant inline process analysis with NIRS (blue line) illustrates where time savings can be gained.



Parameters to monitor in an industrial PU reactor with inline NIR spectroscopy.

Parameter	Concentration	Precision
%NCO	0–30%	0.1%



Polystyrene (PS)

Quantitative analysis of functionalized polystyrene with Raman spectroscopy

SUMMARY

Raman spectroscopy enables fast and accurate determination of chemical composition and structure. Characteristic Raman bands can provide details about chain conformation, easy discrimination of polymeric systems, and even quantitation of species over the course of a reaction [8].

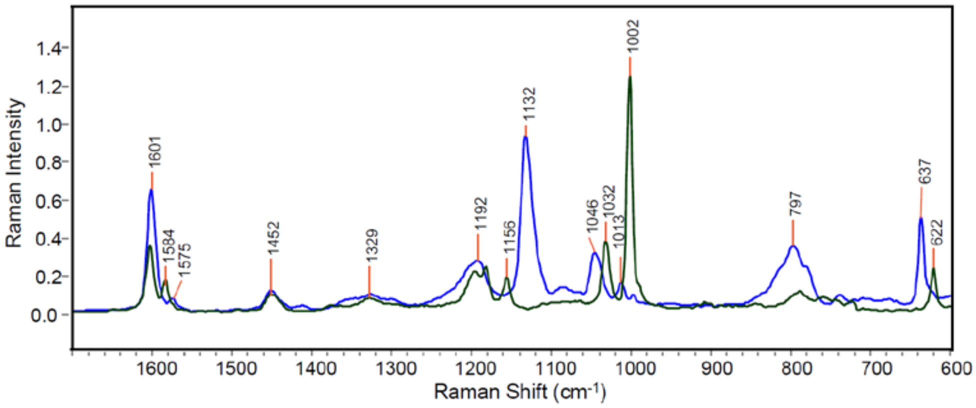
For example, Raman spectroscopy can track functionalization of polystyrene during the creation of a water-soluble polymer. Simple quantitative measurement of the final aqueous polymer determines the extent of reaction. Raman, using a partial least squares (PLS) chemometric model, enables simple, robust quantitation of the extent of polymer functionalization with excellent linearity, accuracy, and precision.

EXPERIMENTAL

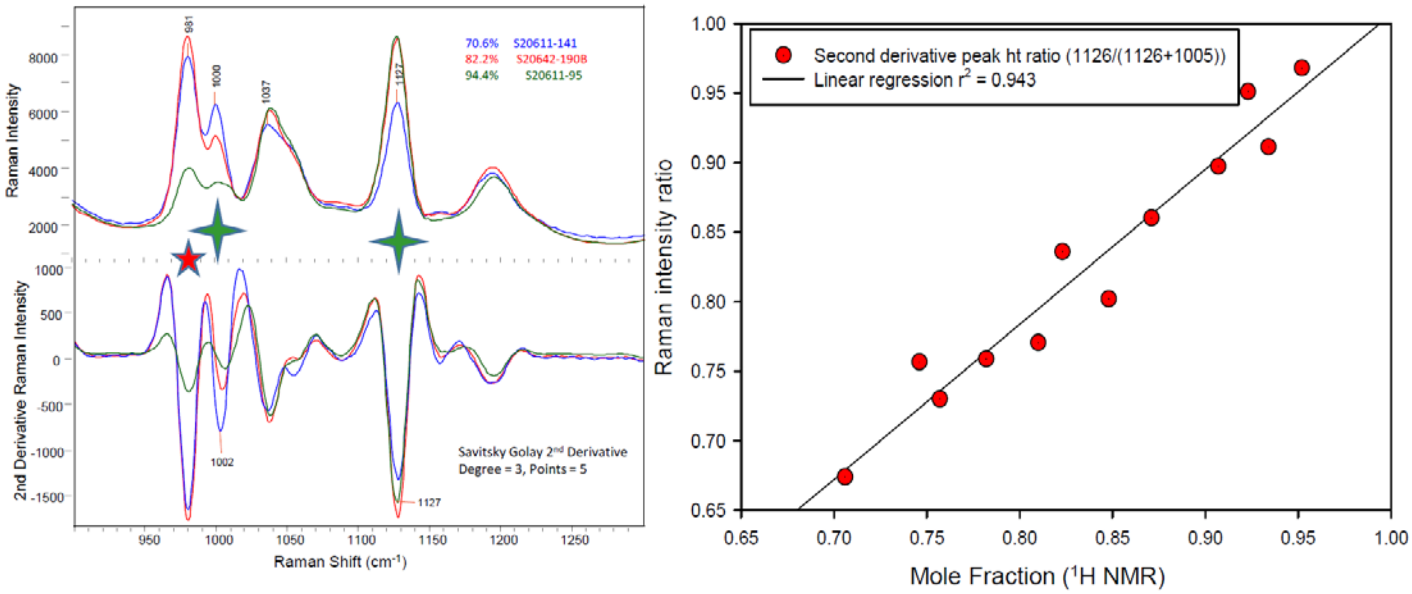
Raman spectra were acquired using a 1064 nm laboratory instrument. In an initial evaluation of starting and final materials, spectra show a clear distinction between the reactants and product. A chemometric model was developed to determine the extent of functionalization. Sixty different samples were investigated, varying from 65–98% functionalization as determined by ^{1}H NMR spectroscopy. A simple peak height ratio analysis was used to determine the extent of polymer functionalization. A quantitative model tested linearity between the predicted (PLS) and assayed (^{1}H NMR) values.

RESULTS

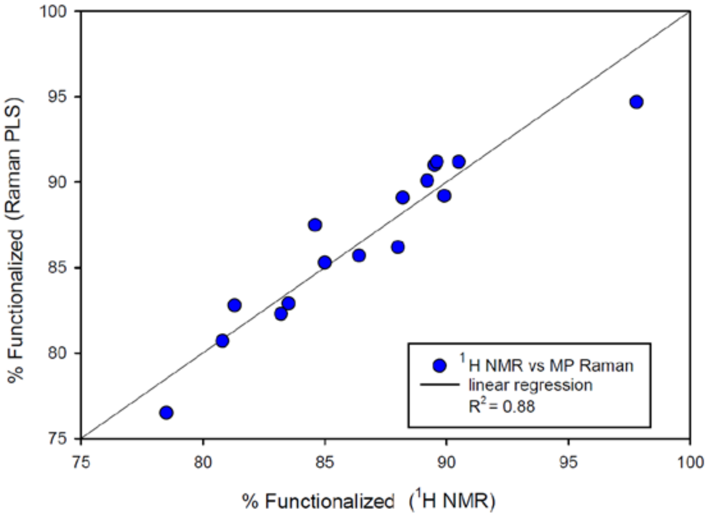
The Raman spectra of the polystyrene starting material and the fully functionalized polymer exhibits excellent specificity in the 900–1300 cm^{-1} region. The band ratio analysis resulted in very good linearity, confirming the suitability of the Raman spectral data to develop a robust quantitative PLS model. The final PLS model, used to develop a linear model between the Raman spectral data and reference values, provided good linearity ($R^2 = 0.88$) between the predicted (PLS) and assayed (^{1}H NMR) values.



Raman spectra of the two pure polymer components: starting material polystyrene (in green) and the fully functionalized polymer (in blue) measured with 1064 nm excitation.



Left: The spectral overlay shows the Raman spectra of three different polymers representative of low, medium, and highly functionalized systems. The spectral feature at 981 cm^{-1} (red star) derives from an undiagnostic inorganic species. The highly diagnostic bands at 1002 cm^{-1} and 1132 cm^{-1} are identified with a green star. **Right:** The band peak ratio plot shows the Raman vs. ^{1}H NMR determined extent of functionalization.



Correlation diagram plotting Raman-predicted % functionalized polymer versus NMR-determined value.

Acrylonitrile butadiene styrene (ABS)

Quantitative analysis of brominated flame retardants in ABS with Raman spectroscopy

SUMMARY

Brominated flame retardants (BFRs) are the largest group of additives used in the plastics industry. However, most BFRs are extremely toxic and are slowly being phased out in many parts of the world.

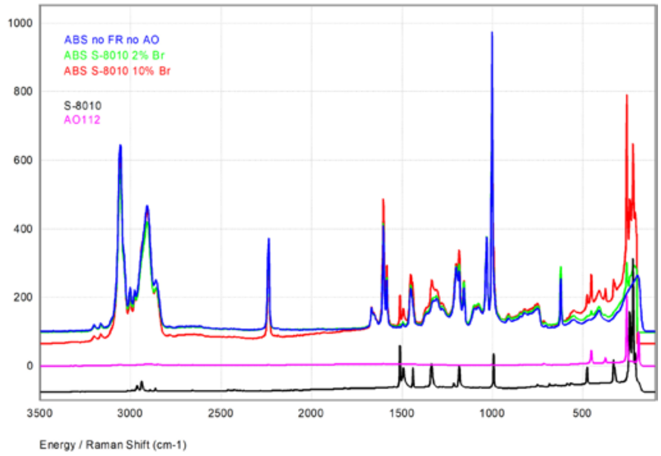
Analysis of these compounds is very time-consuming, requiring lengthy sample preparation that destroys the original sample. High-resolution Raman spectroscopy is nondestructive, requires no sample preparation and only takes a few minutes to fully characterize a material. These capabilities are very attractive to the plastics industry, particularly for masterbatchers and compounders who create the exact properties and coloring for the desired application. Raman spectroscopy meets the need for a rapid and cost-efficient measurement technology that ensures product performance, minimizes waste, and reduces production delays.

EXPERIMENTAL

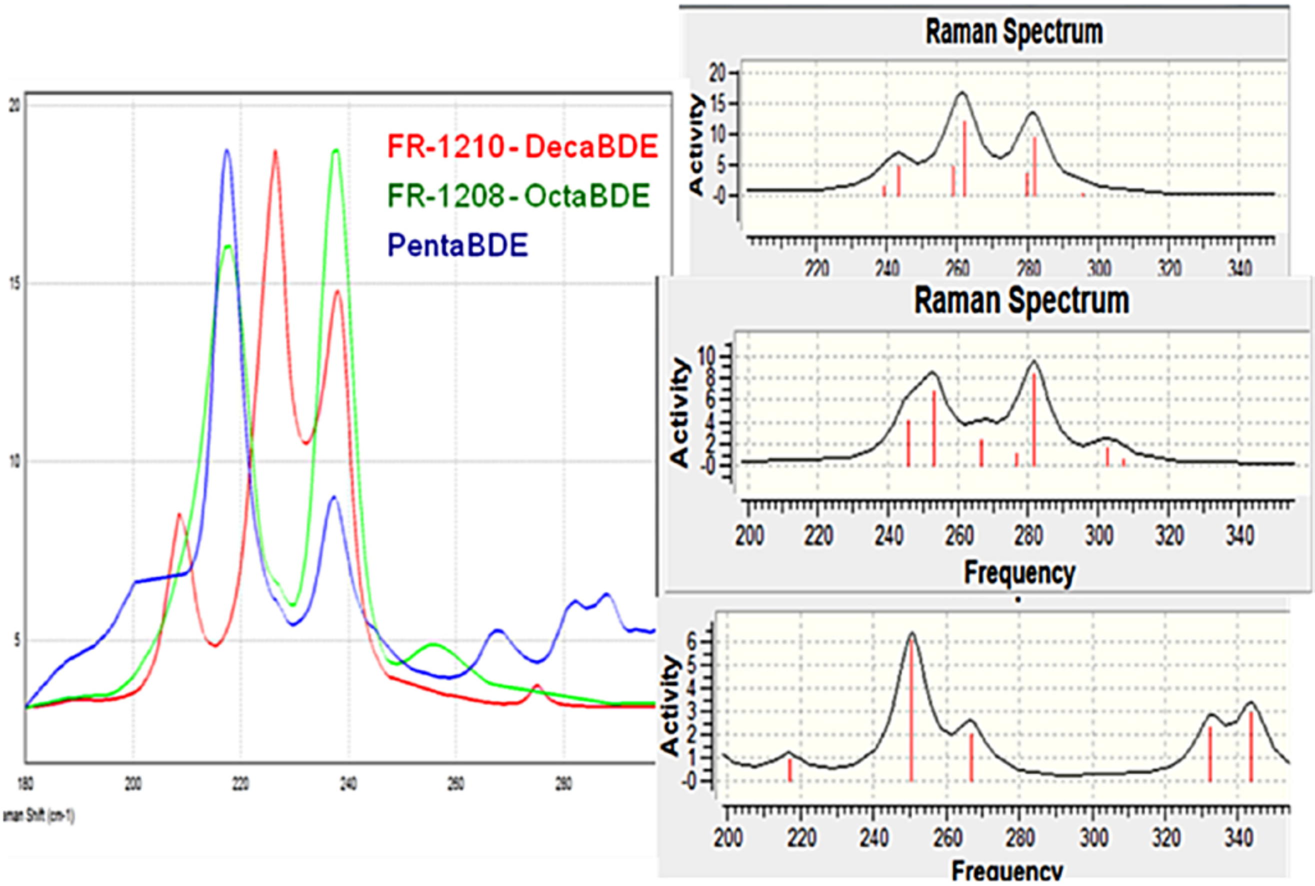
A laboratory i-Raman system was used to detect differences between very similar types of compounds with BFR additives including penta-, octa-, and decabromodiphenyl ether (BDE). Sampling was accomplished with a fiber optic probe accessory. Liquid samples were measured in a glass vial and solid samples were measured by direct reflectance from the sample surface. A range of acrylonitrile butadiene styrene (ABS) polymer materials with varying amounts of BDE present (0%, 2%, and 10% bromine) were tested to determine if their BDE concentrations could potentially be quantified.

RESULTS

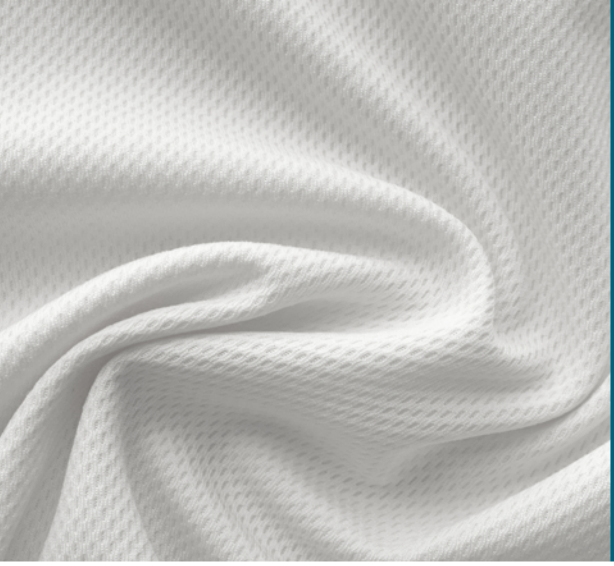
Raman spectral data for three BFRs were matched against reference library spectra. The distinct spectral differences between each compound forms the basis of identification. BFR agents at differing concentrations in ABS plastic were also measured. Aromatic peaks of interest below a Raman shift of 500 cm^{-1} were used to measure samples with varying bromine content. The three ABS compounds with different amounts of BDE present (0%, 2%, and 10% bromine) show subtle but distinct differences in their Raman spectra.



Raman spectral data for a range of ABS polymer materials with varying amounts of BDE (0% – blue, 2% – green, and 10% bromine – red), together with two commercial ABS polymer materials (S-8010 – black, AO112 – pink). Note: the brominated aromatic bands of interest are mostly seen below 500 cm^{-1}.



Raman spectral data for three brominated flame retardants, displayed together on the left and separately on the right: deca- (top), octa- (middle) and pentabromodiphenyl ether (bottom). These spectra clearly show the distinct differences between the intensity and frequency of the brominated aromatic molecules of each compound.



Polyester (PES)

Inline monitoring of PGS synthesis using Raman spectroscopy

SUMMARY

Polyol glycerol sebacate (PGS) is a biodegradable polyester with versatile properties that make it increasingly popular for applications in biomedicine, packaging, and 3D printing.

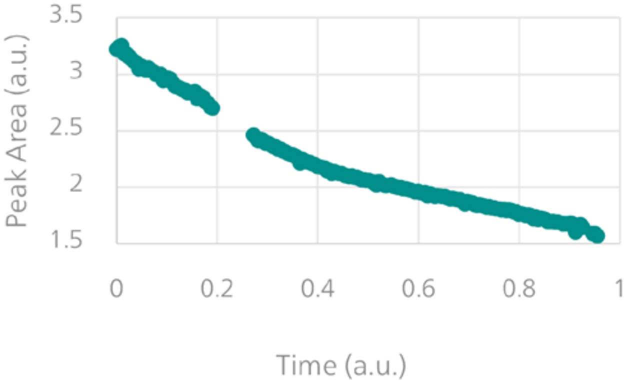
Inline Raman spectroscopy is employed to monitor – in real time – PGS production in an aqueous medium. Incorporating the Raman probe directly into the reactor offers manufacturers immediate understanding of the polymerization process. Unlike some quantitative methods, this setup focuses on tracking reaction trends and ensuring consistent quality rather than generating absolute concentration or purity values.

EXPERIMENTAL

A Raman probe is installed directly into the reactor vessel, enabling continuous inline monitoring of the PGS reaction. This also provides an added layer of quality assurance for raw materials before and during the synthesis. The probe's design is optimized for operation in aqueous environments and offers rapid, noninvasive measurements. This arrangement not only helps identify deviations in the reaction but also ensures the compatibility and purity of feedstock materials, ultimately streamlining production and enhancing overall process control without the need for frequent offline sampling.

RESULTS

This Raman trend chart illustrates how the carboxylic acid groups are consumed over the course of the reaction.



Parameters to monitor in an industrial reactor with inline Raman spectroscopy.

Parameter	Peak area	Pretreatment
Carboxylic group consumption	1420–1460 cm^{-1}	NPS+SNV

Polymethyl methacrylate (PMMA)

Online QC of acrylic acid feedstocks for the production of PMMA

SUMMARY

When synthesizing PMMA (polymethyl methacrylate), the purity of acrylic acid feedstocks directly impacts polymerization efficiency and final product quality.

By implementing online quality control for acrylic acid feedstocks – monitoring quality control parameters like acrylic acid, formic acid, acetic acid, and water – manufacturers can ensure the optimal conditions for PMMA production.

Rapid, real-time spectroscopic measurements reduce downtime, increase yield, and produce a higher-quality polymer, all while cutting costs and minimizing environmental impact. Online near-infrared process analyzers offer this and much more.

Parameters to monitor in an industrial reactor with online NIR spectroscopy.

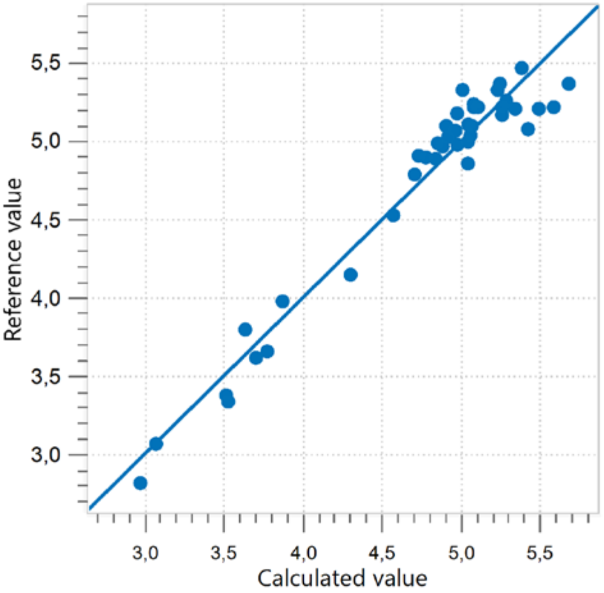
Parameter	Concentration	Pretreatment
Water	2–6%	0.15%
Acrylic acid	75–99%	0.2%
Acetic acid	0–15%	0.2%
Formic acid	0–1%	0.1%

EXPERIMENTAL

The process stream is measured online via a fast-loop sampling system meant to withstand hazardous environments. Multiple sampling points are connected in parallel, allowing sequential analysis of various process streams without interrupting overall flow. Using a NIR process analyzer designed with explosion-proof or intrinsically safe components suitable for the environment and a high-pressure sample loop ensures efficient, real-time transfer of the sample material to the measurement cell. This configuration enables rapid, automated measurements of critical parameters across different points in the process, delivering comprehensive monitoring in a single integrated setup.

RESULTS

NIR model for online moisture analysis in acrylic acid.



Specialized Raman technology

Raman microscopy for rapid identification of microplastics

SUMMARY

Microplastics are categorized as primary or secondary. Primary microplastics include small, manufactured items such as fibers and beads; secondary microplastics are fragments formed by a combination of physical, chemical, and biological processes.

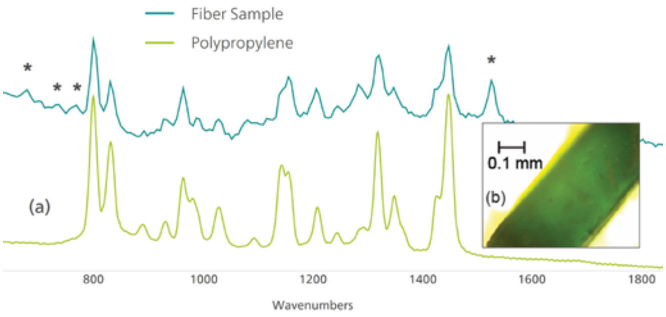
Metrohm's laboratory Raman solutions include micro-sampling accessories that enable the analysis and identification of microplastics as small as 1 micron in diameter.

EXPERIMENTAL

Water samples containing microplastics were collected from the surface water of Delaware Bay (USA). Drying, wet peroxide oxidation, and density separation processes isolated microplastics from digested organic material. These samples were examined with a 1064 nm laboratory i-Raman system using a laser equipped with a video microscope with an objective lens of 50X magnification. Laser power was kept below 50% max (<165 mW) to avoid sample burning. BWID® software was used for identification of the microplastics against a reference library of plastics spectra.

RESULTS

Several microplastic samples were analyzed and identified. A summary of the microplastics measured in this study indicates that the samples were mainly composed of polyethylene, polypropylene, or polystyrene. BWID software successfully matched the sample spectrum for both primary and secondary microplastics using its extensive spectral library. Spectra from a colored fiber (shown below) contain peaks that are consistent with chlorinated copper phthalocyanine green pigment, useful in determining the fiber's origin.



(a) Raman spectrum of a colored fiber (top) compared to a reference spectrum of polypropylene (bottom), and (b) microscope image of the colored fiber sample. The asterisks denote peaks that can be attributed to the colorant used in the plastic.

Specialized Raman technology

Orbital Raster Scan (ORS™) – Safer, more representative Raman sampling

SUMMARY

Raman spectroscopy is used for many different applications in industries where high-quality data is essential, including polymer manufacturing. The highest quality data supports the most accurate material identification. ORS is Metrohm's proprietary way of overcoming low resolution, poor sensitivity, and sample degradation while still interrogating a large sample area.

All MIRA systems from Metrohm use low 785 nm laser powers to collect high-resolution spectra. The combination of low laser power and ORS is ideal for the interrogation of sensitive materials, such as highly colored and volatile substances, in addition to highly inhomogeneous materials.

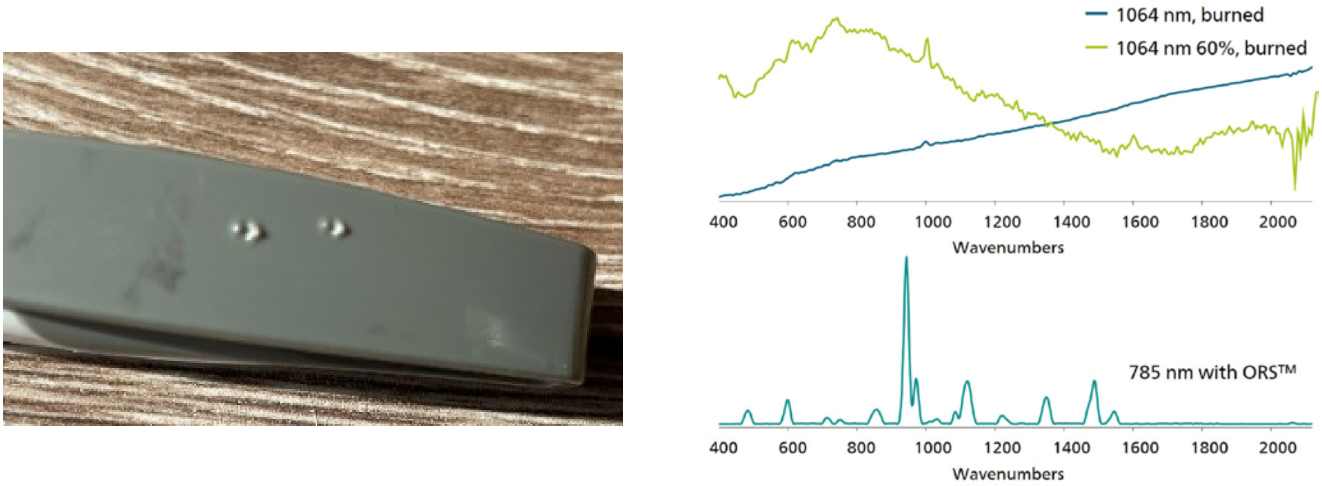
EXPERIMENTAL

Raman spectroscopy is a simple point-and-shoot, nondestructive analysis technique that allows for fast and safe analysis with no sample preparation. In some cases, samples can even be analyzed in their original packaging. A comparison of conventional 1064 nm Raman (which requires higher laser powers to collect high-resolution data) and 785 nm Raman systems was performed to demonstrate that low laser powers and ORS have clear advantages when examining colored plastics.

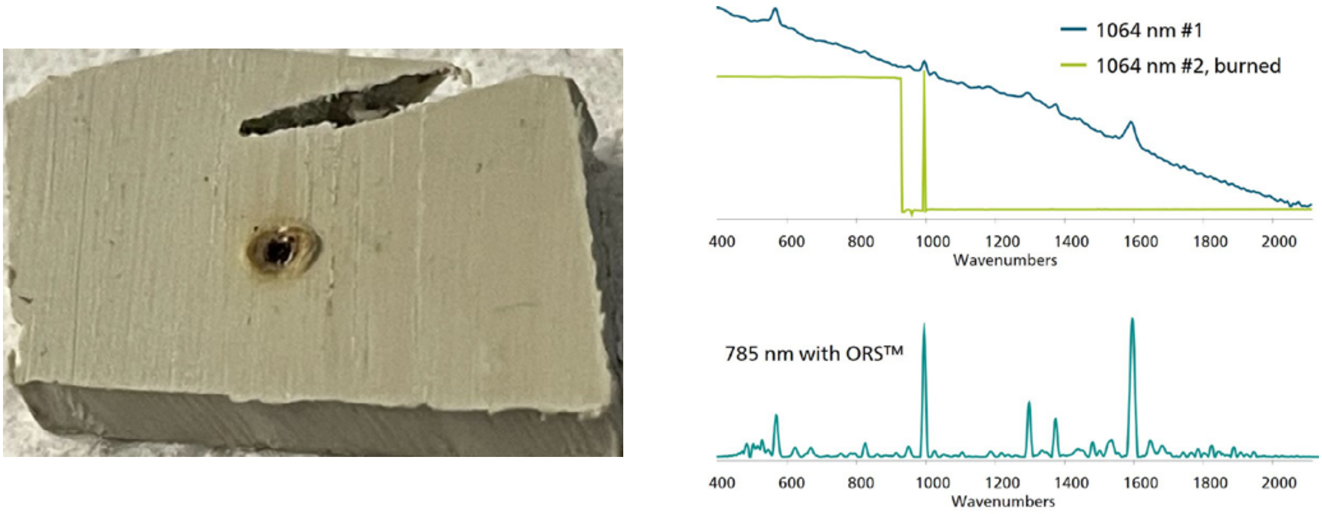
RESULTS

A MIRA handheld spectrometer collected high-quality data from the plastic barrel of a ballpoint pen with no sample damage and identified the plastic as polystyrene. A second examination was performed on a different colored polymer. Nondestructive sampling and positive material identification was possible by using a MIRA system and ORS.

ORS offers users three significant benefits:
- Confidence in sample preservation.
- Data consistency, even for heterogeneous samples.
- Best quality data collection.



Left: A fixed-laser 1064 nm Raman system at 100% power burned the sample, and a second test at 60% laser power gave the same result.
Right: Both tests failed to identify the material (top), which was eventually identified as polystyrene with 785 nm interrogation combined with ORS (bottom).



A comparison of Raman spectra collected from a colored polymer (left) with 1064 nm and 785 nm systems. MIRA easily identified this material, giving a positive identification of polyphenylene ether (PPE) with HQI (Hit Quality Index) = 0.91.

Specialized Raman technology

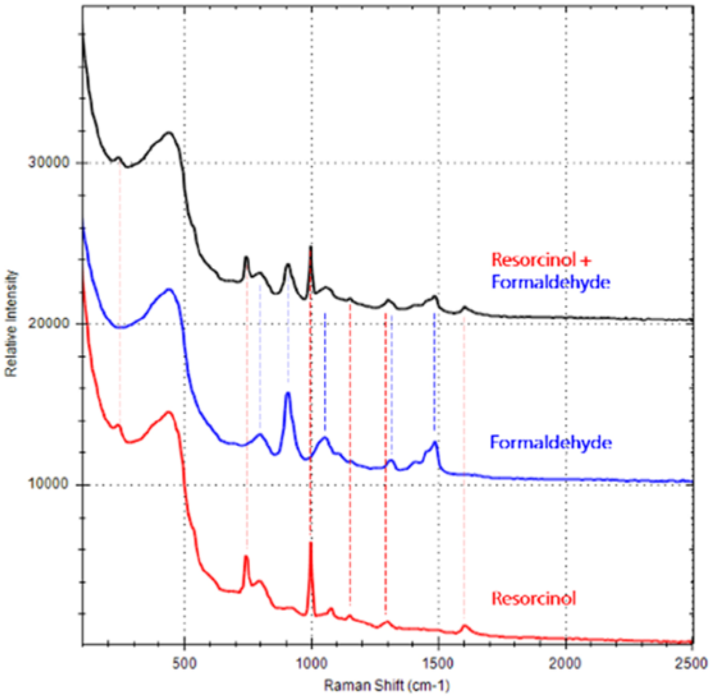
Inline real-time monitoring of the polymerization reaction

SUMMARY

Raman spectroscopy provides a nondestructive, in-situ, real-time method to monitor the polymerization process by tracking monomer consumption and polymer formation.

This technique allows researchers to observe spectral changes associated with functional groups, such as C-H and C=O bonds, providing insight into the reaction.

Furthermore, Raman spectroscopy eliminates the need for extensive sample preparation, making it a convenient and efficient tool for various industries.



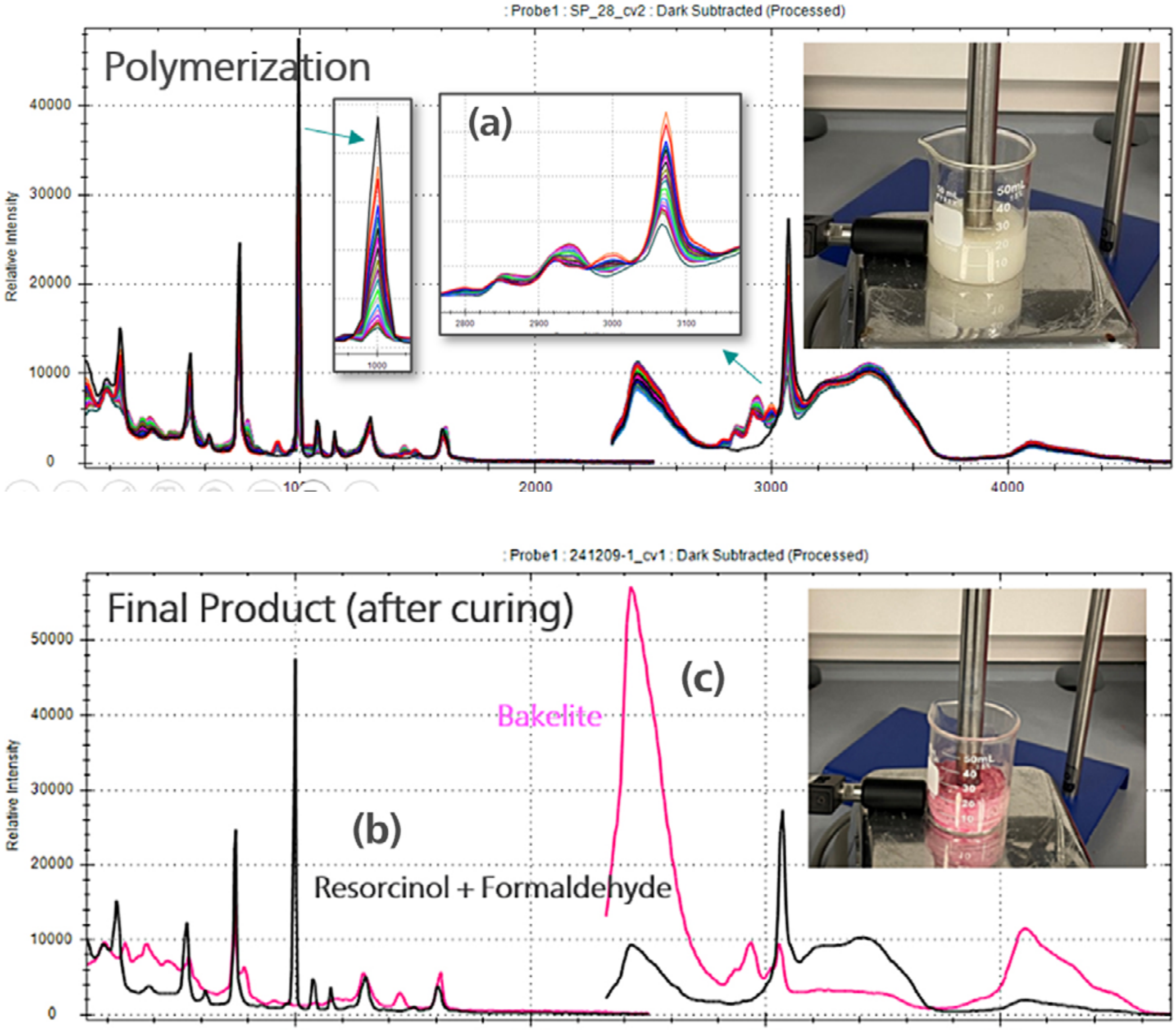
EXPERIMENTAL

Bakelite is a thermosetting plastic created through the polymerization of phenol (e.g., resorcinol) and formaldehyde. This reaction can be safely monitored by placing a Raman probe against the wall of a beaker containing the reactants, allowing real-time evaluation of changing Raman bands as the reaction progresses.

RESULTS

The Raman band at 1000 cm^{-1} (associated with the ring-breathing mode of phenol) diminishes as phenol reacts with formaldehyde to form a cross-linked polymer, while new bands emerge due to changes in the vibrational environment. The ability to measure higher Raman shift regions enhances monitoring of the reaction process by enabling the observation of changes in the C-H stretching vibrations of formaldehyde (2800–3000 cm^{-1}), ensuring a more comprehensive analysis and better assessment of the final product’s quality.

Carboxyl end groups are measured in the raw materials by Raman spectroscopy. These end groups can be monitored over the polymerization reaction once resourcinol and formaldehyde are combined.



Raman bands of the reaction mix: (a) change in Raman spectra during polymerization, (b) Raman profile of the starting materials, and of the final product (c).

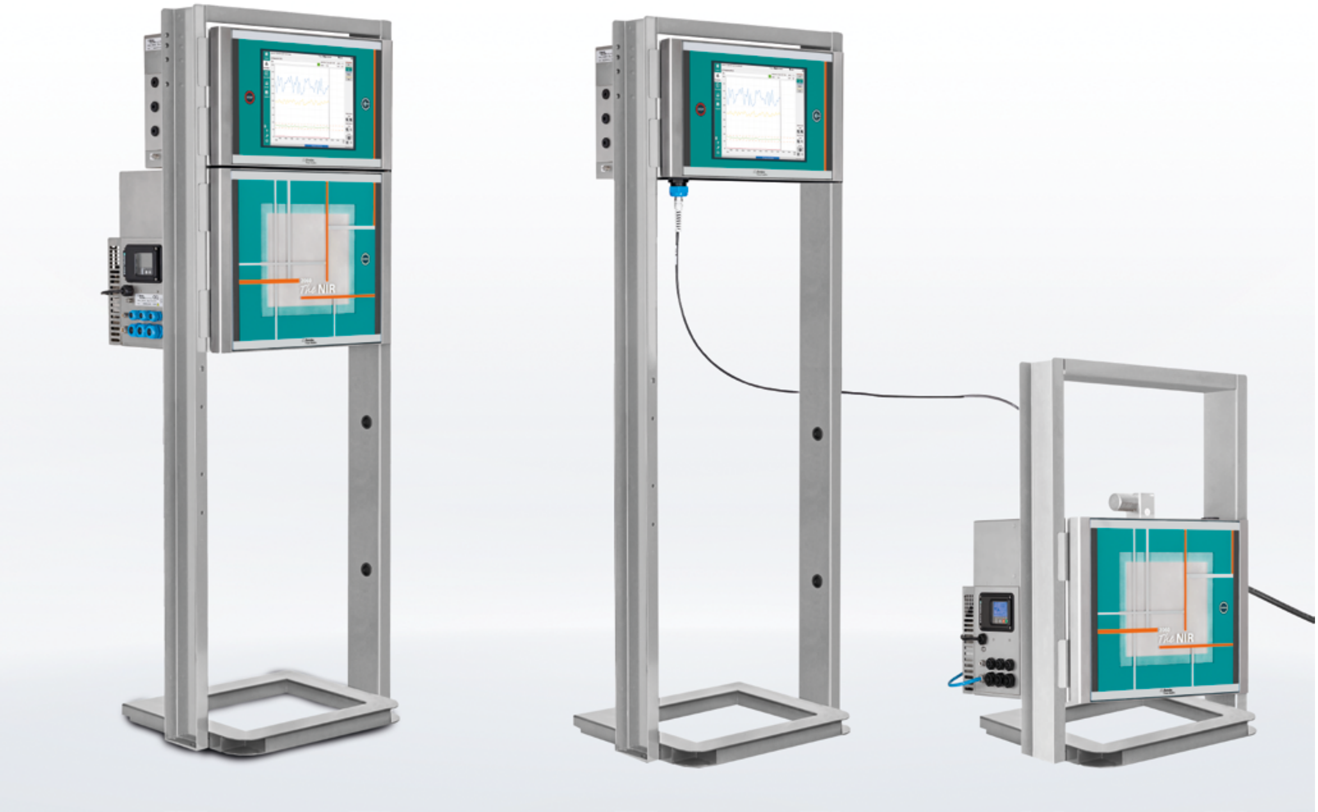
Unique NIR solutions from Metrohm

Metrohm offers a comprehensive range of NIRS analyzers for offline, atline, inline, and online use – all operated by OMNIS software for efficient data collection and analysis. Aside from the instruments themselves, we also provide many more unique solutions to make your analysis as easy as possible.

PRE-CALIBRATIONS

Our pre-calibrations eliminate the need for data acquisition, calibration set creation, or validation. These models allow immediate routine sample analysis at the push of a button, saving time and associated costs.

The NIR prediction models created are based on a diverse collection of real product spectra and are developed in accordance with ASTM E1655: Standard Practices for Infrared Multivariate Quantitative Analysis [**9**].



The 2060 *The* NIR Analyzer is the next generation of process spectroscopy instruments from Metrohm Process Analytics. Its innovative modular design with a separable human interface (HI) and multiple NIR cabinets provides maximal efficiency in cost savings along with improved production lines and final product quality.

OMNIS MODEL DEVELOPER (OMD)

When no pre-calibrations are available, the OMNIS Model Developer automates prediction model creation and validation. It generates five models ranked by predictive performance, with figures of merit provided for each. Advanced users can further refine model parameters if needed.

OMNIS NIR ANALYZER ROBUSTNESS

The OMNIS NIR Analyzer features a Swiss-made, IP54-rated design with no moving optical parts. This ensures durability, long-term measurement stability, and minimal maintenance.

OMNIS PLATFORM

The OMNIS platform is a modular, all-in-one software solution for data collection, prediction modeling, and routine analysis. It supports the combination of NIR analyzers with other techniques such as titration for enhanced data collection.

OMNIS – A universal platform to integrate your laboratory analytics.

For high sample throughput, the OMNIS Sample Robot enables automation possibilities. The OMNIS Software also provides comprehensive user management with granular access controls and optional Microsoft Active Directory and LIMS integration.

NON-CONTACT MEASUREMENT FOR POLYMER PELLETS WITH 2060 *THE* NIR

The 2060 *The* NIR Analyzer offers non-contact measurements for polymer pellets, ensuring accurate, repeatable data directly from the production line. The probe is designed to handle diverse pellet sizes and shapes, adapting seamlessly to the inherent variability in polymer materials. By eliminating the need for contact-based sampling and manual intervention, the 2060 *The* NIR Analyzer streamlines workflows, reduces human error, and accelerates decision-making processes.

SAMPLING SOLUTIONS FOR THE ENTIRE PRODUCTION CHAIN

Besides offering precise and robust analyzer solutions, Metrohm provides advanced sampling solutions for seamless integration into production environments. These solutions excel in high-temperature and high-pressure conditions without requiring O-rings or epoxy. Instead, compression fittings secure the optics, enhancing durability and minimizing maintenance requirements. Robust, user-friendly, and reliable – our instruments are ideal for both laboratory and production settings.

NIR SPECTROSCOPY PRE-CALIBRATIONS FROM METROHM AVAILABLE FOR THE POLYMER INDUSTRY

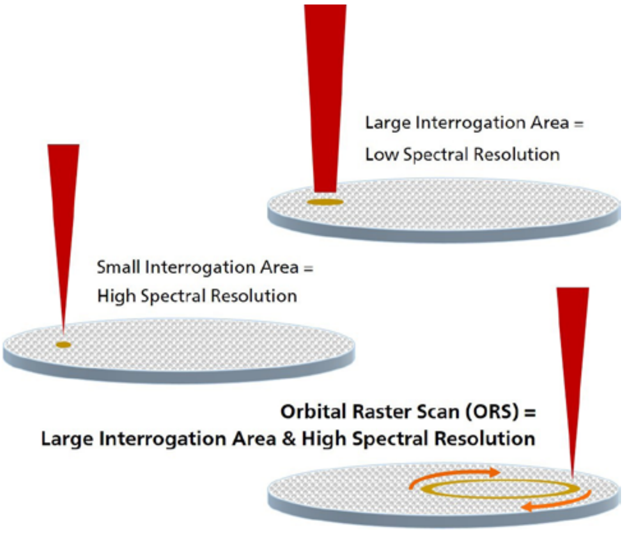
Pre-calibration	Selected important parameter(s)
Polyols	Hydroxyl number (ASTM D6342)
Isocyanates	NCO content
Polyethylene (PE)	Density, intrinsic viscosity
Polypropylene	Melt flow rate
Polyethylene terephthalate (PET)	Intrinsic viscosity, isophthalic acid content, diethylene glycol content
Polyamide (PA 6)	Intrinsic viscosity, amine end group, carboxyl end group

Unique Raman solutions from Metrohm

Metrohm means spectroscopy. We offer a wide selection of handheld, benchtop, and process Raman solutions. No matter what your application needs are, Metrohm is your trusted partner for polymer analysis.

ORBITAL RASTER SCANNING

ORS is ideal for analyzing inhomogeneous mixtures and sample preservation.



Quickly moving (rastering) a tightly focused laser over the sample to collect information from a large area is the principle behind the patented Orbital Raster Scanning (ORS™) method. ORS technology is Metrohm's proprietary way of overcoming low resolution, poor sensitivity, and sample degradation to provide highly accurate results.

> The European Pharmacopoeia specifically mentions ORS: «When using Raman spectroscopy […] care must be taken to ensure that the measurement is representative. This can be achieved by, for example rotation of the sample, performing multiple measurements on different preparations of the sample, using orbital raster scanning (ORS) […]» [**10**].

Learn more about ORS in our Application Note (AN-RS-034): Orbital Raster Scan (ORS™)

FLUORESCENCE REJECTION WITH XTR

XTR opens hundreds of new applications to Raman, including polymers. XTR is the next step in the evolution of Raman spectroscopy. It combines the smaller size, higher resolution, and lower power consumption of a 785 nm Raman instrument with patent-pending advanced algorithms to eXTRact Raman data, even from spectra that have strong fluorescence.

MIRA XTR is capable of producing excellent spectral resolution and flat baseline data with low laser power, short acquisition times, and all of the other excellent functionalities that users have come to expect from Metrohm's Raman devices.

Read our White Paper for more information about XTR (WP-072): Fluorescence-free 785 nm material ID with MIRA XTR DS

COMPREHENSIVE SPECTRAL LIBRARIES

Large, customizable libraries simplify material ID for Raman customers in diverse industries.

Metrohm has comprehensive, up-to-date, high-resolution spectral libraries developed with our own instruments. This results in a more trustworthy, more precise analysis of substances and mixtures. Metrohm library development is a continuous process, designed to give our users ultimate Raman capabilities.

Find out how to benefit from using our Raman libraries here: Raman Libraries

CUSTOMIZABLE RAMAN SOLUTIONS

Not only can Metrohm equip both laboratory and handheld Raman users – we also offer solutions for process analysis! Whatever the situation, Metrohm has the right tools for you.

HANDHELD AND LABORATORY RAMAN SPECTROMETERS

➔ Raman spectrometers

RAMAN ANALYZERS FOR PROCESS ANALYSIS

➔ Process Raman Analyzers

SAMPLING FLEXIBILITY

Metrohm's see-through capabilities support nondestructive analysis of samples through opaque and transparent packaging. Users get real-time identification of materials, which significantly reduces operational times.

Download our brochure for more information: Brochure: Metrohm TacticID-1064 ST: Transforming On-Site Chemical Identification with Precision and Speed

All Raman systems from Metrohm can be equipped with accessories that automate sampling in any situation from trace detection and through-container analysis to direct-contact or Laser Class 1 security.

HIGH-THROUGHPUT ANALYSIS AND PHASE TRANSITION MONITORING

Metrohm Process Analytics spectroscopy solutions are engineered for high-throughput analysis, giving reliable results in a fraction of the time required by traditional wet chemical methods. Whether you need to analyze large sample sets or monitor phase transitions in real time, our Raman instruments provide unmatched performance.

Phase transition monitoring is a critical polymer application where Raman spectroscopy excels. Whether observing crystallization during cooling or tracking the amorphous-to-crystalline phase shift, our Raman systems deliver real-time data that allow users to make immediate, informed decisions. This is especially valuable for research and development teams optimizing polymer formulations or production teams seeking to maintain tight process control.

Metrohm Process Analytics instruments combine speed with precision, offering high spectral resolution and sensitivity for even the most challenging applications. With user-friendly software (IMPACT) and advanced data processing capabilities, our solutions empower you to quickly interpret complex spectra and translate insights into action. For polymer manufacturers looking to stay ahead of the competition, our Raman spectroscopy technology provides the tools you need to achieve faster development cycles, improved product quality, and operational excellence.

The 2060 RISE (Raman Intelligent SEnsor) from Metrohm Process Analytics is suitable for inline process measurements.

Learn more about NIR spectroscopy for polymer analysis

The light-matter interaction is a well-known process most people have already encountered: a sunburn is just one example. However, contrary to UV light which causes sunburns, near-infrared light is lower in energy and therefore nondestructive.

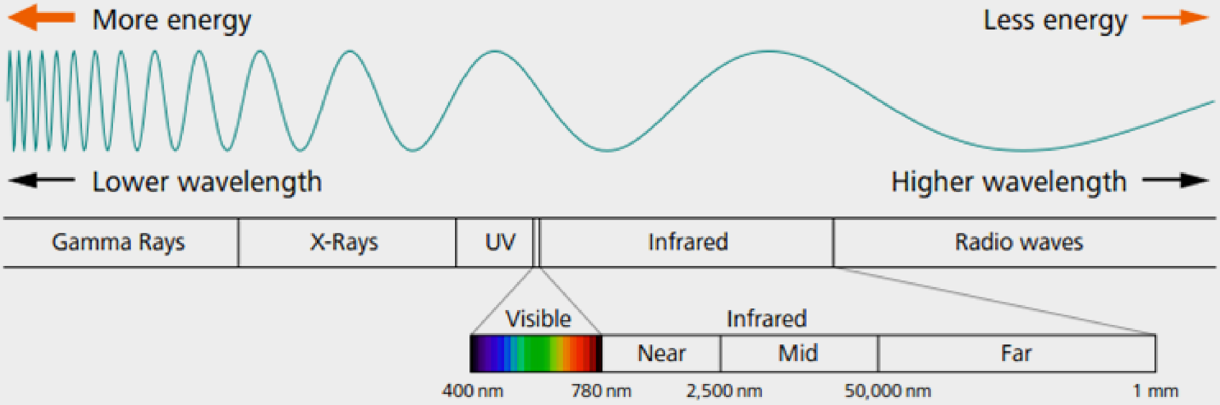
Compared to infrared (IR) spectroscopy, NIRS offers users several benefits:

1. **Deeper sample penetration depth.** The higher energy of NIR light compared to IR light results in deeper sample penetration. Therefore, NIRS provides users information about the bulk characteristics of a sample and not just the surface material.

2. **Lower band intensity, less saturation.** The absorption of NIR light (and therefore the band intensity in the spectra) is typically lower compared to IR. This allows, e.g., the analysis of aqueous solutions of up to 15% water with NIRS. In general, this eliminates the need for any sample preparation prior to the analysis – something which can still be required for IR spectroscopy. It further allows the usage of more convenient sample cells with larger optical path lengths compared to IR spectroscopy.

3. **High accuracy with quantitative measurements.** The lower absorption bands with NIRS offer better analysis of complex mixtures. Furthermore, the sensitivity of NIR bands to physical properties (e.g., particle size, density, or viscosity) can be used to determine these parameters as well, which is typically not possible with IR spectroscopy.

NIR spectroscopy is the analysis of the interaction between specific wavelengths of light in the near-infrared region and matter.



Regions of electromagnetic radiation. The near-infrared (NIR) region is adjacent to the region of visible light. NIR light includes wavelengths from 780 nm to 2500 nm.

For more than 30 years, NIRS has been used for determining multiple parameters in samples quickly without requiring any chemicals. Determination can be performed in liquids, pastes, and solids with this nondestructive analytical technique. It is a fast and reliable method to measure chemical and physical properties in solid and liquids.

HOW DOES NIR SPECTROSCOPY WORK?

The OMNIS NIR Analyzer operates in the wavelength range of 1100 nm to 2250 nm, and generates a spectrum based on the interaction between light in this range and the sample under investigation.

NIRS is a secondary technique. This means a prediction model must be created first. For this reason, a number of spectra must be generated with known concentrations or known values of parameters that were gathered from a primary method, such as titration. A prediction model is created from these spectra using chemometric software (OMNIS Software). Then, routine analysis of unknown samples can begin.

NIR spectroscopy is especially sensitive to the presence of certain functional groups including -CH, -NH, -OH, and -SH. Therefore, it is an ideal method to quantify chemical parameters such as water content (moisture), hydroxyl value, acid number, amine content, and much more.

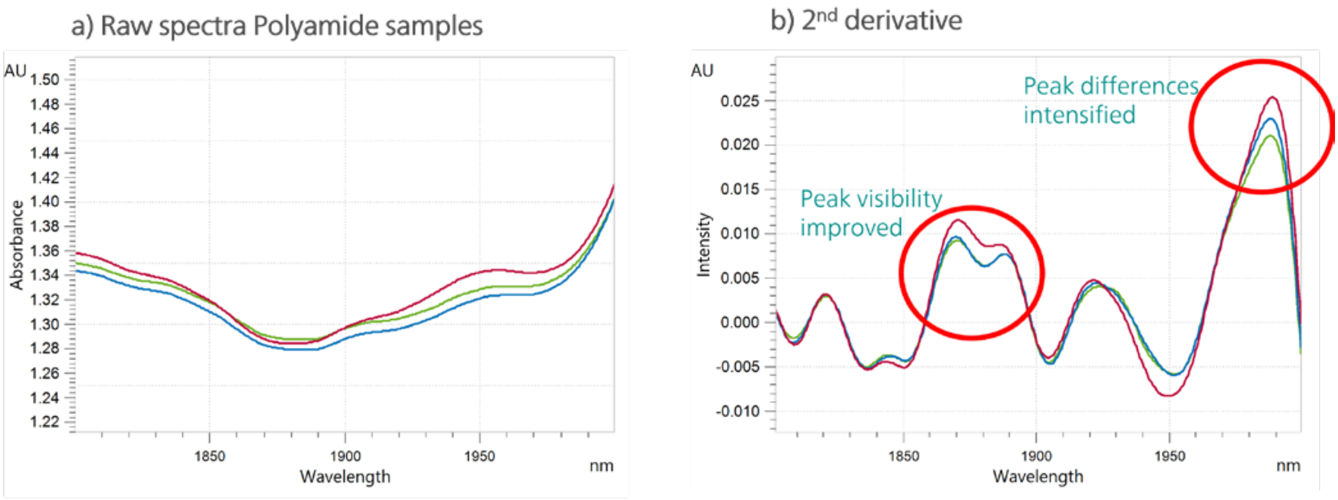
In addition, this interaction between light and matter is also dependent upon the matrix of the sample itself. This allows the detection of physical and rheological parameters such as particle size, density, intrinsic viscosity, and melt flow rate.

As mentioned, NIRS is a secondary technique; therefore the implementation of NIRS requires some steps.

1 – Create a calibration set

As NIR spectroscopy is a secondary method, this means that NIR analyzers require «training» with a set of spectra corresponding to parameter values sourced from a primary method. Take the example of moisture and intrinsic viscosity, where the primary methods are Karl Fischer titration and viscometry. For the NIRS method to be robust, the samples in the training set (referred to as the calibration set) must cover the complete expected concentration range of the parameters tested.

The ideal number of spectra in a calibration set depends on the variation in the sample (particle size, chemical distribution, etc.). However, to build a robust calibration set which covers all sample variations and ensures a reliable quantitative analysis, more sample spectra are required. Approximately 40–50 sample spectra provide a suitable prediction model in most cases.



a) Raw spectra Polyamide samples

b) 2nd derivative

Example of the intensifying effect on spectral information by using mathematical calculation: a) without any mathematical optimization and b) with applied second derivative highlighting the spectral difference and intensifying the peaks.

2 – Create and validate prediction models

Once the calibration set has been measured across the range of expected values, a prediction model must be created. This step is also known as NIR calibration model development.

First, visually inspect each NIR spectrum to identify regions that change with varying concentration. Often, applying a mathematical adjustment (such as the first or second derivative) enhances the visibility of the spectral differences.

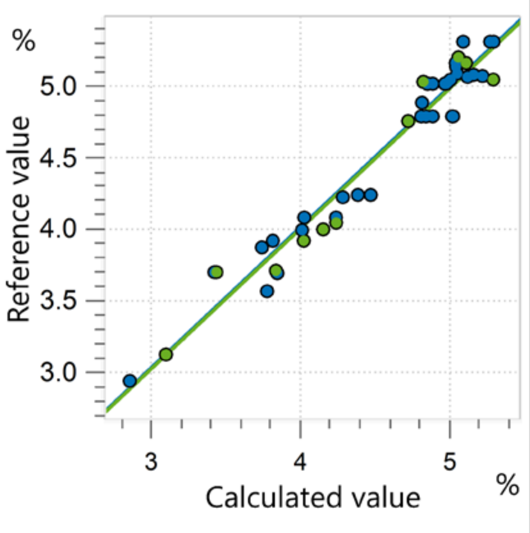
Once visually identified, the instrument software attempts to correlate these selected spectral regions with values sourced from the primary method. The result is a correlation diagram, including the respective figures of merit, i.e., the standard error of calibration (SEC, precision) and the correlation coefficient (R^2). After creating a prediction model, it must then be validated using the validation data set.

3 – Routine analysis

After creating and validating a prediction model, polymer samples with unknown moisture content and unknown intrinsic viscosity can now be analyzed in a few seconds without any sample preparation.

Looking for more information about the basics of NIRS? We've got you covered:

- ➔ White Paper (WP-094): OMNIS NIRS: An efficiency boost for your laboratory
- ➔ White Paper (WP-054): Boost efficiency in the QC laboratory: How NIRS helps reduce costs up to 90%
- ➔ Video: Basics of NIR spectroscopy – How does NIR spectroscopy work?
- ➔ Video: What are pre-calibrations for NIR spectroscopy?



R^2	SEC (%)	SECV (%)	SEP (%)
0.946	0.1021	0.1325	0.1610

Example correlation diagram and figures of merit (FOM) for moisture content analysis. The same steps must be taken when determining, e.g., intrinsic viscosity.

Learn more about Raman spectroscopy for polymer analysis

Plastic is ubiquitous, and Raman spectroscopy can be used anywhere it is located for identification, classification, and quality testing purposes. Raman spectroscopy is increasingly chosen for polymer analysis because it is nondestructive, requires no sample preparation, and gives results in just seconds. It is easy on the user; non-technical operators can collect data on-site. Raman is also environmentally friendly – requiring no chemicals, solvents, or materials for sample prep – and it does not generate any waste.

Discover how Raman spectroscopy is used for polymer analysis in our blog:

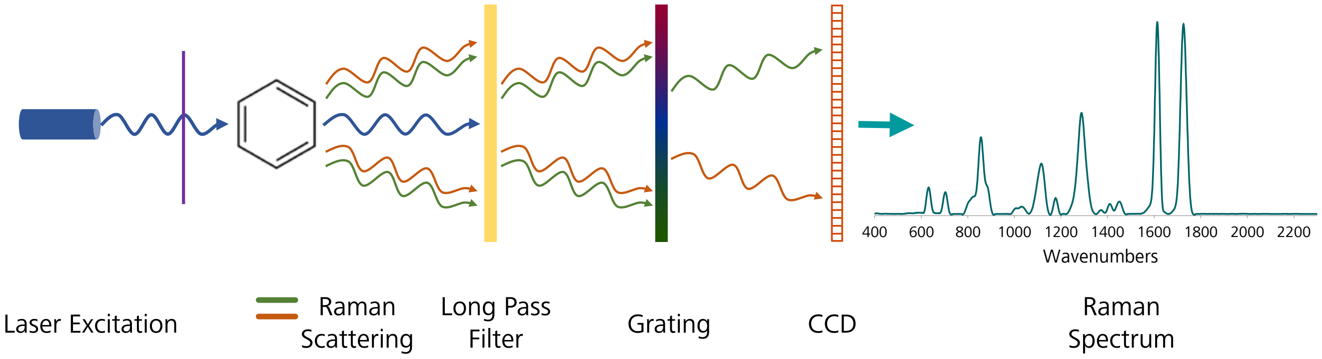
➔ Raman spectroscopy: The easy way to analyze polymers

WHAT IS RAMAN SPECTROSCOPY?

Raman is a form of molecular spectroscopy that is observed as inelastically scattered light when a sample is excited by a laser. While most scattering occurs elastically, one in a million photons will interact with a molecule and result in Raman-scattered light. Shifted by these molecular interactions, the detected Raman photons are processed into a spectrum characteristic of the unique bonds within a molecule, providing the user with an invaluable analytical tool for molecular fingerprinting. This «fingerprint» is used primarily for material identification and, increasingly, for quantification.

Read our blog posts to learn more about the theory and usage of Raman spectroscopy as well as some of its applications:

➔ Frequently Asked Questions (FAQ) about Raman spectroscopy: Theory and usage
➔ Frequently Asked Questions (FAQ) about Raman spectroscopy: Applications



Simplified illustration of how a Raman spectrum is generated.

Learn more about Process Analytical Technology for polymer analysis

The polymer industry is vital for many other sectors, including aerospace, automotive, and consumer products. Since it supplies crucial raw materials, the quality, consistency, and efficiency of polymer production processes significantly influence the performance and safety of downstream applications.

Continuous insight into these processes is essential. Polymerization reactions and processes are complex and valuable, yet even the smallest variations can lead to significant issues. To address this, polymer manufacturers must embrace a data-driven approach.

Integrating Process Analytical Technologies (PAT), such as advanced process analyzers, into polymer production facilities supports manufacturers with:

- **Enhanced reproducibility:** Minimized batch-to-batch variations leads to improved product quality and reduced rework.

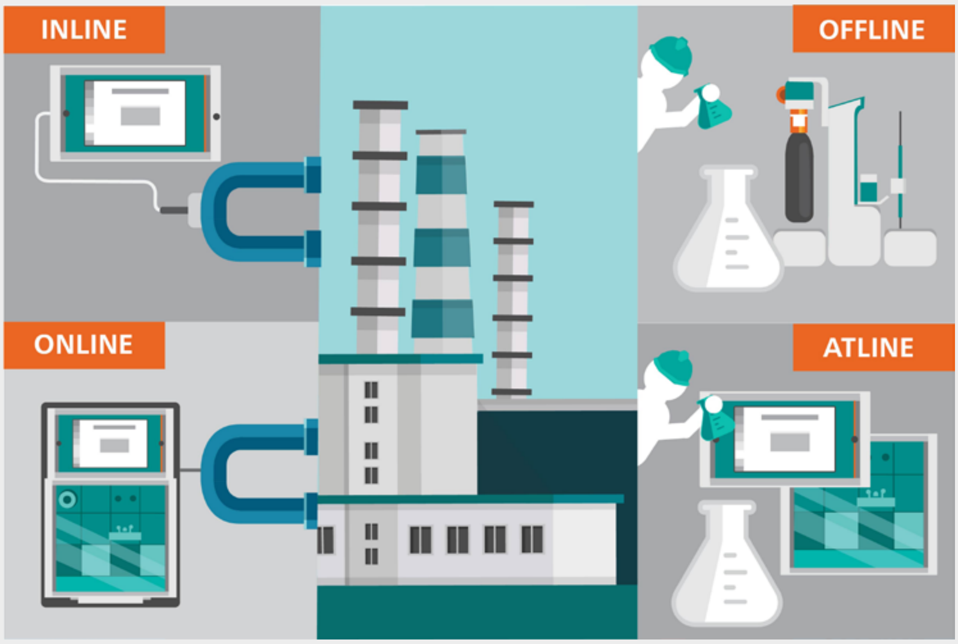
- **Significant cost savings:** Optimized process control minimizes off-spec material production, reducing raw material consumption and associated costs.

- **Increased productivity/efficiency:** Real-time process adjustments, optimized process parameters, and minimized downtime maximize productivity and throughput.

- **Enhanced safety:** Reduced human intervention, minimized exposure to hazardous materials, and early detection of safety hazards create a safer working environment.

Nondestructive and versatile, NIR and Raman spectroscopy provide critical insights into polymer composition and quality. These techniques offer rapid and accurate analysis, enabling real-time monitoring and control throughout the production process.

There are four primary ways to implement NIR and Raman spectroscopy in your polymer production workflow:



COMPARISON OF INLINE, ONLINE, ATLINE, AND OFFLINE ANALYSIS METHODS FOR POLYMER PRODUCTION

Feature	Inline	Online	Atline	Offline
Sampling	Direct measurement within process stream	Automated from process stream	Manual sampling from process to nearby analyzer	Manual sampling from process to remote lab
Automation	High	High	Moderate (manual sampling)	Low (manual sampling and transport)
Real-time data	Most real-time analysis	Continuous, real-time feedback	Some delay between sampling and analysis	Significant delay due to transport and analysis
Process control	Optimal for critical applications	Immediate process adjustments	Suitable for less critical applications	Limited process control due to delayed results
Labor requirements	Minimal	Minimal	Moderate (manual sampling)	High (manual sampling, transport, and lab analysis)
Cost	Higher initial investment, lower operational costs	Higher initial investment, lower operational costs	Moderate initial investment, moderate operational costs	Lower initial investment, higher operational costs
Suitable/ optimal for	Critical applications, immediate feedback	High-frequency analysis, continuous monitoring	Multiple sampling points, less frequent analysis	Infrequent analysis, detailed investigations

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