



Application Note AN-RS-008

Identification of monomers with Raman spectroscopy

Monitor the polymerization process from monomer to polymer

Polymers are comprised of macromolecules that are in turn made of numerous identical or similar structural units, referred to as monomers. The number of monomers, including additives or inhibitors such as benzoquinone – used to endow the polymers with specific properties – is enormous.

All polymer manufacturers use the same monomers and can profit from a quick raw material check before

feeding them into the polymerization process. Raman spectroscopy provides a nondestructive, in-situ, real-time analytical method to monitor the polymerization process by tracking monomer consumption and polymer formation. Ultimately, Raman spectroscopy is a convenient and efficient tool for various industries related to the polymer sector.

INTRODUCTION

This Application Note demonstrates the convenient identification of monomers within seconds using Raman spectroscopy. Monomers such as styrene, various alkyl methacrylates, vinyl acetate, ethylene glycol, phenol, terephthalic acid, and urea, as well as additives or inhibitors like benzoquinone, can be

identified rapidly and unambiguously.

A quick demonstration of the distinct spectra of different monomers and their respective polymer leads to an in-depth look at the polymerization reaction of Bakelite.

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 handheld 785 nm Raman device, featuring an automated workflow and Orbital Raster Scanning (ORSTM), was used to collect basic monomer spectra. The polymerization reaction of Bakelite was safely monitored by placing a laboratory Raman fiber-optic probe against the wall of a beaker containing the reactants, allowing real-time evaluation as the reaction progressed.



MONOMER SPECTRA

Figure 1 contains overlaid spectra of different monomers (and benzoquinone), demonstrating that Raman is both sensitive and high specific—it is very easy to distinguish the spectra of different materials.

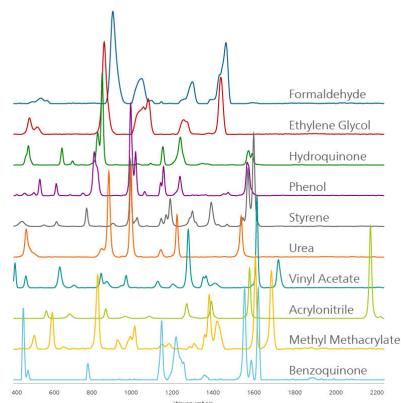


Figure 1. Overlay of Raman spectra from the analyzed monomers and benzoquinone.

MONOMERS AND POLYMERS

Monomers bind together to form polymers during the polymerization process. Real-time monitoring of the polymerization reaction with Raman spectroscopy is a powerful way to optimize and control the process and the resulting product. It is Raman's specificity that permits monomers to be easily distinguished from their respective polymers. **Figure 2** illustrates the spectral differences between polymer starting materials and products.

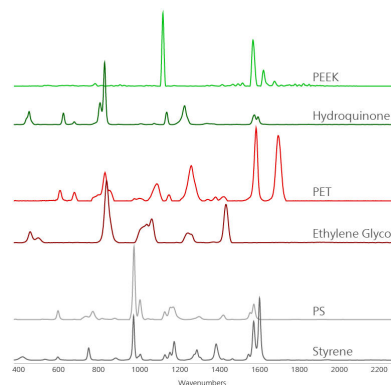


Figure 2. Overlay of Raman spectra from monomers with their respective polymers, including polyether ether ketone (PEEK), polyethylene terephthalate (PET), and polystyrene (PS).

POLYMERIZATION MONITORING WITH RAMAN

Bakelite is a thermosetting plastic created through the polymerization of phenol and formaldehyde. **Figure 3** shows how the Raman peaks of phenol diminish as it reacts with formaldehyde to form a cross-linked polymer, while new peaks emerge due to changes in the vibrational environment. High Raman shift regions permit the observation of changes in the C-H stretching vibrations of phenol (2000–4500 cm⁻¹), providing insight into this reaction.

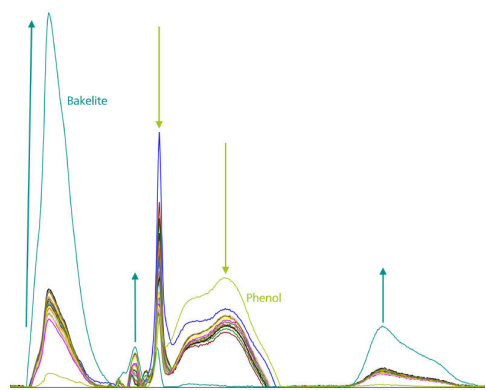


Figure 3. Evolution of Raman bands for phenol and Bakelite during the polymerization reaction.

CONCLUSION

Real-time, on-site analysis using Raman spectroscopy enables polymer manufacturers to maintain product integrity through quality checks at every stage, from

raw materials to final products. This ensures consistent quality, optimizes efficiency, and drives innovation in polymer manufacturing.

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CONFIGURATION

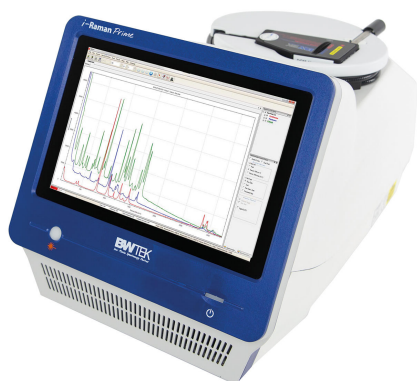


MIRA P Advanced

Le Metrohm Instant Raman Analyzer (MIRA) P est un spectromètre Raman portable performant qui s'utilise pour les déterminations rapides et non destructives et le contrôle des matériaux les plus divers, comme les principes actifs pharmaceutiques et les excipients. De très petite taille, le MIRA P est pourtant très robuste et dispose d'une structure de spectrographe haute efficacité, équipée de notre technologie « Orbital Raster Scan » (ORS) inédite. MIRA P satisfait aux prescriptions FDA 21 CFR partie 11.

Le Advanced Package comprend une lentille avec laquelle les matériaux peuvent être analysés directement ou dans leur conditionnement (classe de laser 3b), ainsi qu'un support de flacon pour analyser les échantillons dans des flacons en verre (classe de laser 1).

CONFIGURATION



Spectromètre Raman portable i-Raman Prime 785H

L'i-Raman[®] Prime 785H est un système Raman à faible bruit entièrement intégré à haut débit, avec une tablette PC intégrée et une sonde à fibre optique. Faisant appel à un détecteur à barrette CCD à haute efficacité quantique, refroidi par cryogénie thermoélectrique (-25 °C) et une gamme dynamique élevée, ce spectromètre Raman portable offre la possibilité de réaliser des analyses Raman de qualité recherche, incluant quantification et identification en temps réel. Le haut débit délivre des spectres Raman avec un excellent rapport signal/bruit, ce qui permet de mesurer des processus rapides, de même que les signaux Raman les plus faibles afin de détecter les différences les plus fines entre les échantillons.

Outre le concept d'appareil portable, l'i-Raman Prime 785H propose aussi la combinaison unique d'une vaste gamme spectrale et d'une haute résolution, qui permet des mesures de 150 cm⁻¹ à 2800 cm⁻¹. L'i-Raman Prime peut fonctionner sur batterie, ce qui facilite son transport. Ainsi, quel que soit le lieu, il est possible de réaliser des analyses Raman de qualité recherche, à la fois de haute précision et de haut niveau qualitatif et quantitatif. Le système est optimisé pour une application de notre technologie STRaman[®] pour des analyses au travers d'emballages non transparents.