Application Notes AN R541

Analysis of Polymer Fillers and Additives by Raman

About Polymers, Fillers and Additives

Polymers have been around for more than a century and today, they are are a high-tech, well engineered products with multiple ingredients. Beside the polymer, copolymer or blend itself, additives and fillers play a vital role in the final mechanical and chemical properties of the product. While additives bond with the base material, fillers don't physically interact with the main substrate.

And while fillers might be added in high percentages, additives and plasticisers may only be present as ppm contributions. As all of them are essential for the proper mechanical and chemical properties of plastic products, reliable quality control is indispensable.

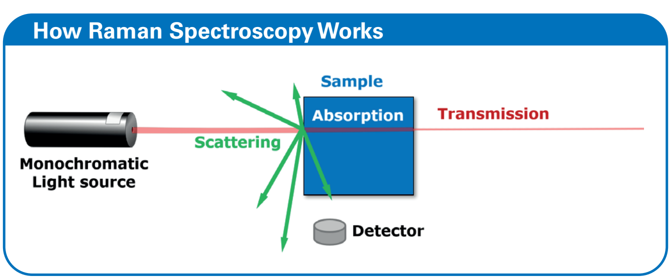

Figure 1: In Raman (micro) spectroscopy, a laser is directed at the sample and the light scattered off the surface is then collected. The light now carries molecular information which is analyzed.

Keywords	Instrumentation and Software
Polymers	SENTERRA II Raman Microscope
Additives	R-KIMW Polymer Library
Fillers & Dyes	OPUS/Search
Plasticisers	OPUS/3D

Raman Spectroscopy and Microscopy as a tool for polymer, additive and filler analysis and QA/QC

In the following three typical examples are described where Raman can help with a fast and reliable chemical analysis:

- **Inorganic Fillers**
  Raman spectra can tell you the type of polymer and any inorganic filler that might have been added in ~ 1 minute.
- **Pigments and Dyes**
  Usually, organic pigments are added in low quantities. Raman can trace and identify them (often in ppm amounts).
- **Flame Retardants and other functional additivess**
  Functional additives can have deteriorating effects to the material if not properly dispersed and Raman can evaluate the distribution of components using 2D and 3D imaging.

TiO$_2$ in Polyethylene

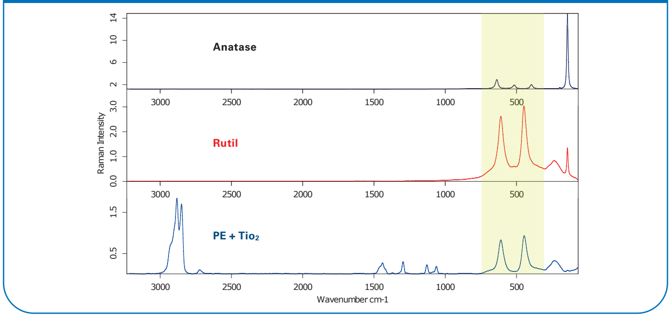

Figure 2: Raman spectra of Anatase (black), Rutil (red) and the TiO$_2$ filled polymer (blue). Region of interest highlighted in yellow.

Application #1: Carbon or TiO$_2$ filled polymers

A very broad spectral range allows Raman to easily access spectral information about inorganic components in polymers.

Figure 2 shows polystyrene filled with TiO$_2$. As the Anatase modification of TiO$_2$ promotes photocatalytic decomposition, the product specification demands the use of Rutil only. Fortunately, Raman spectroscopy is not only very sensitive to inorganic compounds in mixtures but also provides information about cristallinity and morphology.

Figure 3 shows a polymer composition with very distinct signals of carbon. Usually, this is applied either to verify the type of Carbon inside the polymer, or to check if the addition had any deteriorating effects on the sample.

Generally, Raman spectroscopy is extremely sensitive to all carbon allotropes and also analyzes additives based on graphene, fullerenes or other carbon modifications.

Carbon-Filled Polymer

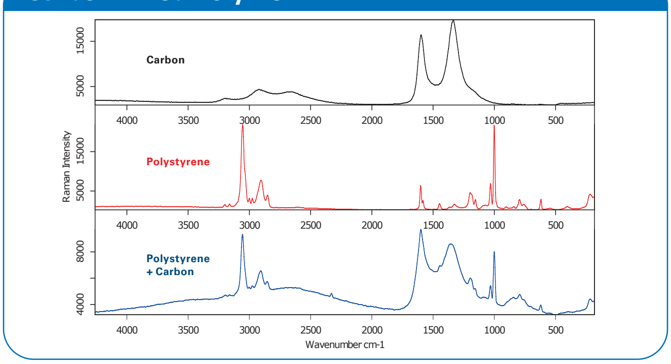

Figure 3: Raman spectra of pure carbon (black), pure polystyrene (red) and polystyrene filled with carbon (blue).

Colored Polyethylene

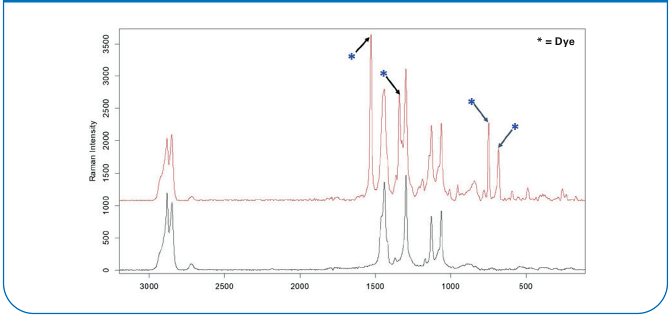

Figure 4: Raman spectra of pure polyethylene (black) and colored polyethylene (red). Raman bands of dye indicated by arrows.

Application #2: Pigments and Additives

Organic pigments are often very strong Raman active substances. That's why it is quite feasible to detect trace amounts of pigments or other additives in a polymer sample. *Figure 4* shows the spectra of the pure polymer and with the pigment added. The signals of the pigment are very pronounced making it easy to verify the use of the correct pigment during production.

Application #3: Flame Retardant

Flame retardants are key ingredients in polymers used for construction and engineering. They can make up to 30% of the polymer formulation in some materials! An incorrect distribution of such addtivies can lead to critical comprizing effects of the base material. *Figure 5* shows the chemical Raman image of the distribution of the halogen free flame retardant melamine cyanurate (MC) in a polyamide-6 matrix The homogeneity and size of particulates is easily evaluated.

Flame Retardant Distribution in Polymer

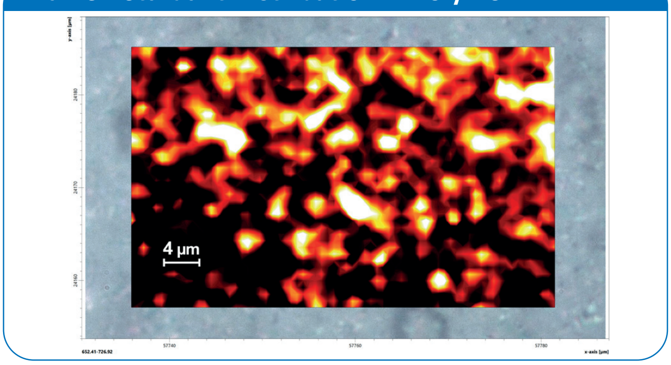

Figure 5: Chemical heatmap of melamine cyanurate (MC) in polyamide-6. The brighter the color, the more MC is incorporated into the matrix.

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