Analysis of Contaminants on Paper by ATR Microscopy

Field
Plastics, Polymer, Forensic, Food, Pharmaceutics, Electronics

Equipment
FTIR-8000 series
AIM-8800

Necessary Accessories
ATR Objective

Necessary Software
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Analysis of extraneous materials is very important in Quality Assurance procedures. Infrared Microscopy is widely used for QC purposes because of the ease of operation and short analysis time involved. ATR Microscopy requires very little sample preparation (non-destructive); and is free from the influence of interference fringes in Transmittance mode, as well as any possible reflection and/or absorption by substrate surfaces.

In ATR Microscopy, the sample is placed directly onto the stage. The stage is moved up and good sample contact can be ensured between the sample and the prism. This condition is extremely important because it will directly affect the quality of the spectra obtained. In most cases, the measurement of soft samples on hard flat substrates can be done quite easily, while the measurement of softer samples on rough surface substrates can be a bit more difficult. Below, is an example of an application involving the measurement of contaminants on/in paper products.

A. Measurement of Contaminants on Paper

ATR Microscopy requires adequate sample pressure in order to obtain spectra. When the sample itself is softer than the substrate, precise sample contact can be achieved rather easily. In cases where the "substrate" is very soft, the sample can actually be imbedded in the surface of the substrate. In this instance, the obtained spectrum can include peaks from both the sample of interest and the substrate; providing inconclusive spectral results.

It is recommended that a spectrum of both the sample and substrate material be analyzed separately, thus providing the user with pure spectra of each material free from any influence from the other compound during the measurement procedure.

Fig.1 shows a microscopic photograph of contaminants on paper. Fig.2 contains a spectrum of both the contaminant and paper substrate overlayed together. In this case, the contaminant was large and easily attached to the prism; providing a good sample spectrum without any influence from the paper substrate.

The spectrum contains a secondary amide compound, possibly originating from a piece of skin.
B. Measurement of a Fiber on Filter

ATR Microscopy measures all sample areas which come in contact with the prism. The maximum/minimum measurement area depends largely on the shape and size of the prism, as well as the dimensions of the sample itself.

When the Slide-On Ge ATR objective is used to measure a flat plastic sample, the minimum analysis area is roughly 20 or 30um in diameter. Now, if a smaller sampling area is used, the spectrum obtained might include unwanted spectral peaks of the other material present.

If the sample measurement area can be sufficiently isolated from the background substrate, a good sample spectrum can be obtained free of any substrate influence.

Fig.3 shows a microscopic photograph of a fiber contaminant on a nitrocellulose filter. The fiber was over 100um long, but only 10um wide. Fig.4 shows both the spectra of the fiber and filter together. The spectrum of the fiber contained polyethylene telephthalate, and did not include any peaks attributable to the filter substrate.
C. Measurement of a Stain

It is very difficult to measure samples imprinted, stained, or smeared on paper products without the influence of the paper itself on the measurement. Usually most paper influences can be removed through spectral subtraction. In some cases if adequate pressure is applied, the sample image maybe retained on the prism surface and can be analyzed separately using a gold mirror as the background when measuring. At this time, only the sample impression on the prism is measured: and the paper influences are completely removed.

Fig.5 shows the spectra of the paper substrate (red line) and the stain (green line), respectively.

Fig.6 shows the spectra of the stain (purple line), and the stain with gold mirror (red line) background.

Fig. 5 ATR Spectrum of Paper (Brown line) and Stain (Purple line)
Fig. 6 ATR Spectra of a Stain impression deposited on a Prism