Use of Secondary, Non-Saturating IR Spectrum Information for the Precise Measurement of Polyethylene Films Thickness Using Encoded Photometrics InfraRed (EP-IR) Spectrometry.

An EPIR spectrometer manufactured by Aspectrics, Inc. was tested for its capacity to accurately measure the thickness of films of polyethylene to the thousandth of inch using secondary, non-saturating infrared spectral information without loss of accuracy.After deliberately excluding the main absorption wavebands (saturating) from the useable spectral information, a quantitative method was developed using a Principal Components Regression calibration approach model and only secondary absorption regions. The method allowed calibration for the 0.6 to 5.9 1/1000th inch range with an accuracy defined by anSEC = 0.07 1/1000th inch and an SEP = 0.07 1/1000th in.

The use of diagnostic spectra (variance, correlation and mean spectra) allowed for the rapid identification of spectral regions enabling the development of a satisfying chemometrics model despite the strong (saturating) absorption of the samples of polyethylene.

The technique proved the concept of fruitful use of secondary bands when the primary absorption wavebands are saturated and marks the technique as a possible quality control tool for the manufacturing of such polyethylene films.

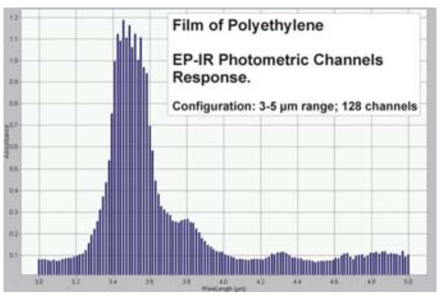

Fig. 1a: 128-channel actual EP-IR response for a film of polyethylene

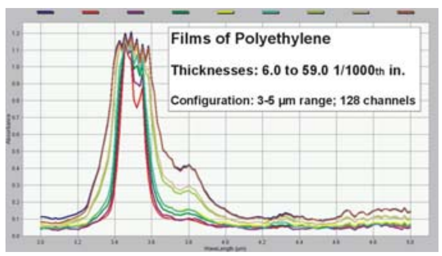

Fig. 1b: 128-channel EP-IR response for films of polyethylene with thicknesses varying from 0.6 to 5.9 1/1000th inch. "Lines" graphic representation of photometric channel response.

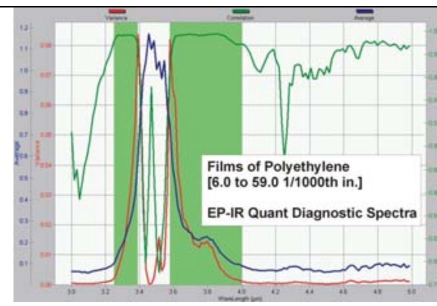

Fig. 2: Quant Diagnostic spectra (correlation, variance and mean spectra) for the identification of the spectral regions best fit to develop a quantitative PCR-based model.

INTRODUCTION

EP-IR spectrometry is a new, rugged, yet analytically accurate infrared spectrometry technology specifically designed for the simultaneous measurement of multiple chemical compounds at high speed (up to 100 scans per seconds) in industrial environments.

EPIR spectroscopy is based upon the use of an encoder disk onto which all wavelengths in a given region of interest are uniquely modulated. The final spectral intensities are computed via a Fourier transform providing access to the entire spectral information.

Current technology allows up to 256 user-defined micro-regions of the spectrum to be measured simultaneously by a single detector (multiplexing advantage), hence providing the opportunity for simultaneous quantitative analysis of several components as well as the implementation of complex, multivariate chemometrics methods.

MATERIALS & METHODS

An Aspectrics, Inc. Spectral Engine configured for the 3 – 5 µm region providing access to 128 photometric channels (each channel being 10 nm wide and with center-wavelength equally distributed every 15.7 nm in the wavelength domain) was used for this experiment. A glow-bar source and a PbSe (lead selenide) detector with single stage thermoelectric cooling were used.

The instrument was set to collect 30 spectra per second and was equipped with a sampling bench and a custom-made film sample holder.

Each sample of film of polyethylene was solidly placed in a slide holder enabling the placement of the film at an angle of 90° when compared to the beam in the sampling bench for measurement in transmission.

Principal Component Regression calibration equations (chemometrics methods) were developed using the SID, Inc. Real-Time Spectroscopy (RTS) software application, including automated calculation of diagnostic spectra and PCR quantitative models.

18 calibration and 18 validation spectra were collected, each resulting of the normalized co-addition of 75 spectra (~ 2 ¼ seconds collection time).

Reference thickness of the samples of films of polyethylene were provided by the manufacturer.

RESULTS

The principal obstacle to developing a quantitative model was that the main absorption band for polyethylene is too strong, resulting in the saturation of the detector for thicknesses greater than approximately 1.5 1/1000th in. (See *fig. 1b*, with particular focus on the 3.48 – 3.58 µm where saturation of the detector as the thickness of the film increases is particularly visible.)

Study of the combined diagnostic spectra for the set of calibration standards confirm the impossibility of using this band to calibrate the instrument for the measurement of the thickness of the film (See *fig. 2*).

More specifically, it is observed that the 3.48 – 3.58 µm region shows very weak variance over the entire calibration range [red plot] and shows very low correlation [green plot] between the variation of the response of photometric channels in this range and the variation in thickness of the samples (despite maximum absorption of the mean spectrum which would finger this region as that with the best signal-to-noise ratio for the measurement).

Two secondary regions were however identified (3.25 – 3.38 µm and 3.58 – 4.00 µm) as highly correlated [see green plot on *fig. 2*] with the variations in thickness of the polyethylene film samples.

These regions were selected as source of optimal spectral information for the method.

Despite the fact that these regions showed less intrinsic variance and mean absorption (therefore less than optimal S/N ratio), when using these regions and a Principal Component Regression method, the following results were obtained:

PCR Calibrations Statistics			
n	R^2	Bias (1/1000th in.)	SEC (1/1000th in.)
18	0.9993	0.05	0.07

PCR Validations Statistics			
n	R^2	Bias (1/1000th in.)	SEP (1/1000th in.)
18	0.9993	0.05	0.07

Such results are proof that, despite the interference of some non-optimized analytical conditions (unknown precision for the reference measurement of the films thickness, unknown precision for the measurement of the incidence angle of the infrared beam onto the sample), a solid, validated quant model can be developed and used for the application, possibly even as a quality control tool in the plastic industry.

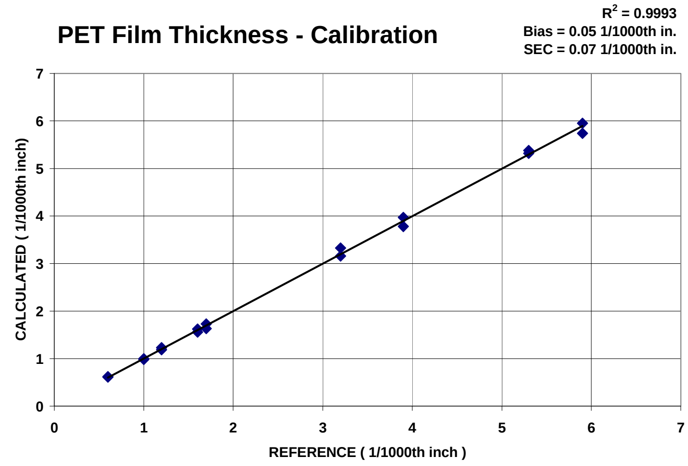

Fig 3a: Calculated film thickness vs. Actual (Reference) film thickness. Calibration plot using 18 standard spectra (each resulting of the normalized co-addition of 75 single scans)

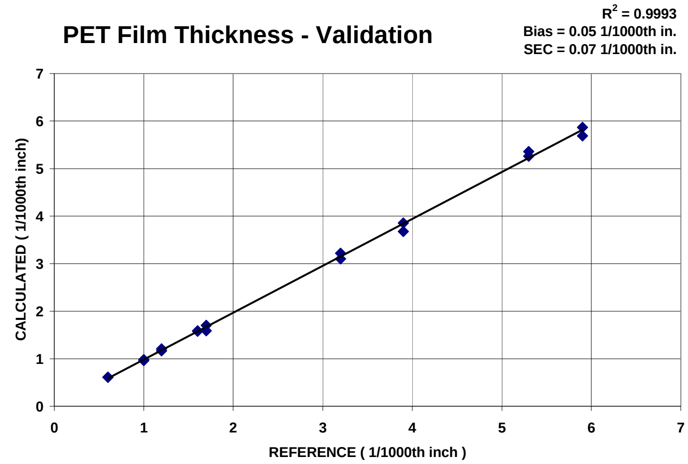

Fig 3b: Calculated film thickness vs. Actual (Reference) film thickness. Calibration plot using 18 standard spectra (each resulting of the normalized co-addition of 75 single scans)

Both calibration and validation chart further show that the thicknesses of the sets of calibration and validation standards were reasonably equitably distributed, thus adding to the ruggedness of the calibration by minimizing the differences in statistical weights for each data point.

CONCLUSION

The Aspectrics, Inc. EP-IR spectrometer clearly demonstrated that the technology can produce very accurate validated quantitative results, even when the instrumental response of some photometric channels is saturated by the presence of an excess of absorbing material.

This is very important as saturation of the instrumental response on some photometric channels cannot be construed as a limiting factor to developing satisfying quantitative models using the instrumental response of other photometric channels.

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