



Application Note AN-NIR-076

Quality control of polyvinyl alcohol

Improved polymer quality control with NIR spectroscopy

Polyvinyl alcohol (PVA) is used in a variety of medical products (e.g. eye drops) due to its low toxicity, low protein adhesion, and film forming properties. PVA is a linear polymer, which forms copolymers of vinyl acetate and vinyl alcohol. The degree of alcoholysis is the percentage of hydroxyl functional groups compared to the total functional groups accessible in the molecule. It is an important index for the water solubility, viscosity, and adhesion of the product. Conventional alcoholysis determination requires each

sample to be weighed, dissolved, heated, cooled, and titrated. This procedure can take up to six hours per sample. Compared to the primary method, analysis with near-infrared spectroscopy (NIRS) only takes one minute. The following application note describes the determination of the degree of alcoholysis by NIRS. Aside from alcoholysis, additional quantification methods for sodium acetate and volatiles can be established.

EXPERIMENTAL CONDITIONS

54 spectra from 18 different sample batches were collected using a Metrohm DS2500 Solid Analyzer in combination with the Vision Air Complete spectroscopy software. To overcome sample inhomogeneity, the measurement was performed with a large sample cup in rotation. The reference values were obtained by titration. Outlier detection was performed on pre-processed spectra (2nd derivative) using a maximum distance in wavelength space algorithm. The NIRS prediction model was created with the settings described in the following table, and validated using cross validation.



Figure 1. The DS2500 Solid Analyzer was used to collect the spectra of PVA polymer.

Pre-processing	Algorithm	Validation type
2 nd derivative	PLS	Cross-validation

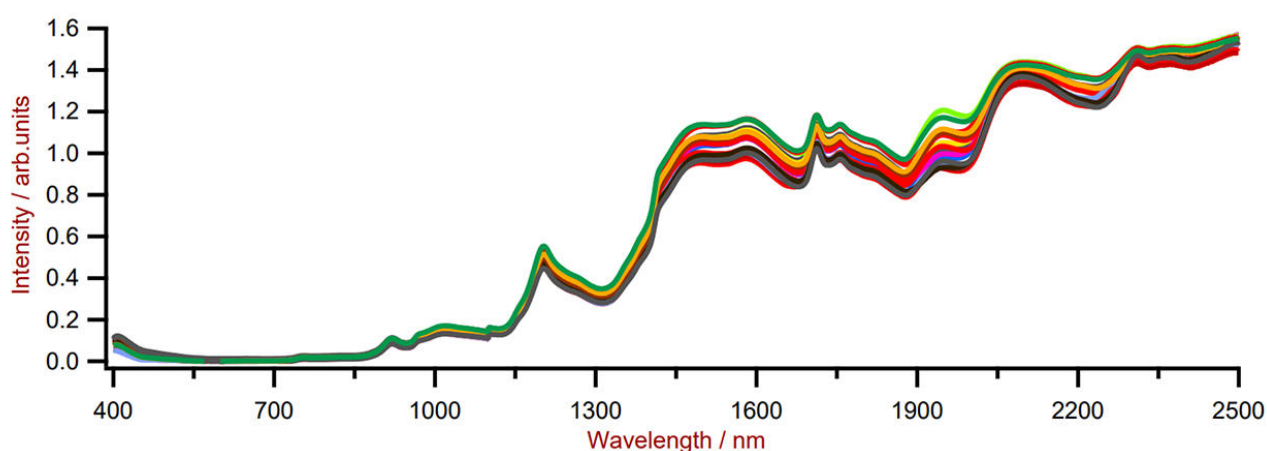


Figure 2. Selection of polymer spectra with varying degrees of alcoholysis.

RESULT & CONCLUSION

The obtained correlation graph displays a high correlation ($R^2 = 0.98$) between alcoholysis degree predicted by NIRS and the primary lab method. A

close ratio between SEC and SECV ($< 10\%$), proves that the method is valid.

# Factors	R^2	SEC	SECV
3	0.98	0.24%	0.25%

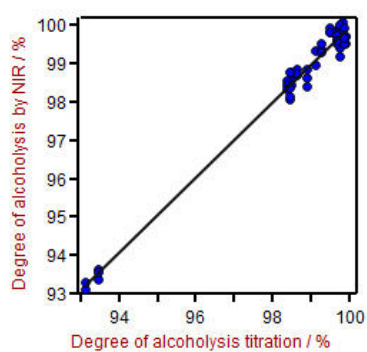


Figure 3. Correlation graph for alcoholysis degree predicted by NIRS vs. lab method.

CONTACT

Metrohm Brasil
Rua Minerva, 161
05007-030 São Paulo

metrohm@metrohm.com.br