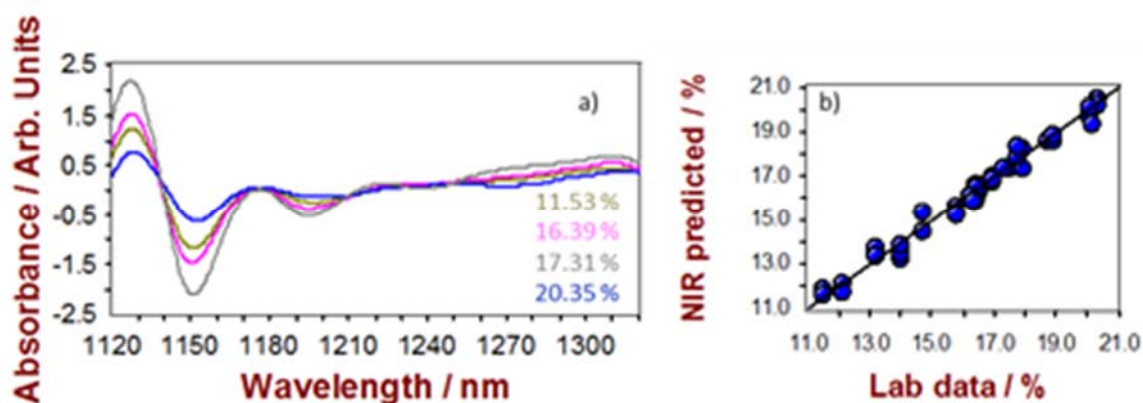# Determination of amine number and solid content of dipping paint

This Application Note shows that Vis-NIR spectroscopy can be used to quantify the amine number and solid content of electrophoretic coating material in the paint industry. The determination of those parameters, commonly achieved with time-consuming conventional lab methods, can easily be performed by Vis-NIR spectroscopy. With a single measurement, other parameters can be determined – also by untrained users.

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Method description

Introduction

Beautification and protection of polymeric materials or steal is an important field of paint industry. Therefore, special dipping paints, stoved at high temperatures into the material, were developed. The dipping paint is transferred onto the material in an electrochemical reaction. Multiple paint layers are produced by deposition of colloidal particles from an aqueous paint bath onto an electrically conductive surface, either in an anionic or cathodic electrophoretic deposition process (EPD). The type of EPD performed depends on the binder used, where the cathodic process is the most common one. Hereby, the electrophoretic coating material consists of a low-solvent (< 4 % organic alcohol e.g. butyl glycol), water-based paint containing resins (e.g. melamine) as binders, organic amines (e.g. diethyl amine) as neutralizer to stabilize the pH value, cross-linking agents (e.g. epoxy resin) to fasten the cross-linking reaction, and additives (e.g. pigments, plasticizers or foaming inhibitors) giving the paint a specific behavior or color.

The quality control of chemical and physical characteristics of finished products is of high importance to achieve consistency of product quality, not only of the paint itself, but also for the coated object. The most important parameters to be quantified in dipping paints are the amine number and the solid content. The amine value (DIN 53176) reflects the amount of free nitrogen, which can be protonated and has therefore an important impact on the paint hardening process. The solid content (DIN 53219) represents the amount of non-volatile constituents and therefore the layer thickness.

The determination of those parameters, commonly achieved with time-consuming conventional lab methods, can easily be replaced by Vis-NIR spectroscopy to give results of all parameters of interest within a single measurement operated even by untrained users.

Configuration

The following equipment was used (**Tab. 1 and Fig.1**).

Tab. 1:

Equipment	Metrohm article number
NIRS XDS SmartProbe Analyzer	29211610
Vision Software 4.0.3	66069102

Fig. 1: A NIRS SmartProbe Analyzer equipped with a transflection probe was used to record the spectral data.

Experimental

A NIRS SmartProbe Analyzer (transflection measurement) was used to collect the spectral data of white dipping paint samples of different solid content and amine number. The samples were placed in a beaker and stirred constantly to avoid sedimentation of pigment particles.

A Partial Least Squares algorithm in Vision (Metrohm chemometric software), was used to develop quantitative prediction models for solid content and amine number, see **Tab.2**.

Tab. 2:

Parameter	Concentration
Solid content	11.5–20.4 %
Amine number	37.5–48.3 mmol/kg

Spectral data pretreatment was done by a 2nd derivative with a segment size of 10 nm and a gap size of 0 nm. In addition Standard Normal Variate procedure (SNV) was also applied to correct for particle scattering influences in the spectra. An internal cross-validation was used to verify the performance of the derived quantitative models.

Method description

Results

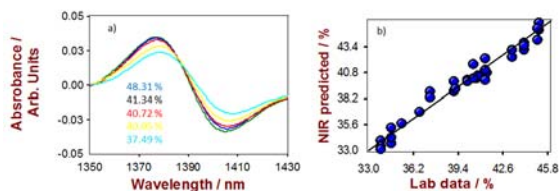### Amine number

Fig. 2: a) The pre-treated spectra of dipping paints (λ = 1350–1430 nm) with varying amine number from 37.5 mmol/kg to 48.3 mmol/kg. b) Correlation of the NIR predicted values with the reference data as a result of a quantitative method development of the amine number in dipping paint.

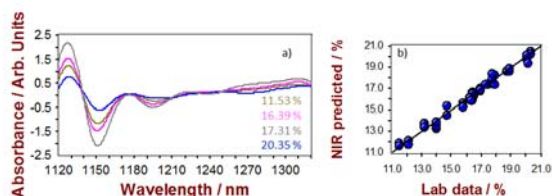| Regression model	PLS with 5 factors
Wavelength range	1120–1920 nm
R^2	0.9678
SEC	0.7345
SEV	2.9284
F-value	156.3
PRESS	274.4

Solid content

Fig. 3: a) Spectra of dipping paints (λ = 1120–1330 nm) with solid content from 11.5% to 20.4%. b) Correlation of the NIR predicted values with the reference data as a result of a quantitative method development of the solid content in dipping paint.

Regression model	PLS with 6 factors
Wavelength range	1120–1920 nm
R^2	0.9239
SEC	0.7484
SEV	0.8606
F-value	331.1
PRESS	19.81

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