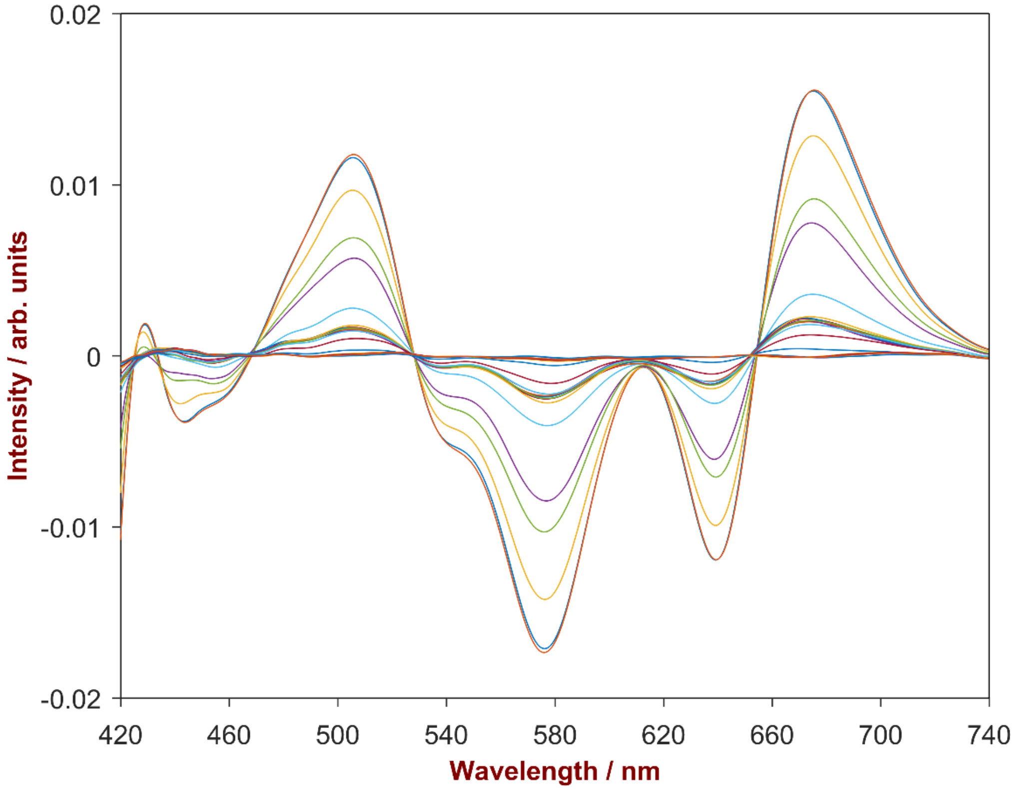
NIR Application Note NIR–058

Quantification of color intensity of diluted textile dye by visible near-infrared spectroscopy



This Application Note shows that the visible range of the Metrohm Vis-NIR analyzer can be used to quantify the color intensity of dyes, providing comparable results to the reference analysis UV-Vis. The NIR region can be used in addition to distinguish between different dye types or different suppliers and can identify impurities in the undiluted dyes during raw material control. The combination of the visible and near-infrared wavelength ranges offer the benefit that only one analyzer is required to receive results on multiple parameters in a 30-seconds-scan results on both cost and time saving.

Metrohm NIRSystems

Method description

Introduction

Colors give textiles a special meaning: The fashion world wouldn't be the same without colors.
Colorants, which can be either dyes or pigments, are used to give objects a desired appearance. Dyes are soluble colored organic compounds that are usually applied to textiles from a solution in water. They are designed to bond strongly to the molecules of the textile fiber. Coloring of textiles is called dyeing and is the process of adding color to textile products like fibers, yarns, and fabrics. Pigments are insoluble compounds used in paints, printing inks, ceramics and plastics. [1]
The color intensity is the most important property of dyes and the dyeing process, as it has influence on the color intensity of the finished textile. The parameter color intensity describes the coloring power of a colorant. [2]
Commonly, the color intensity is determined by UV-Vis spectrometry.

Experimental

Identification of impurities in dye:

The application purpose was threefold:

- Identify different dye types
- Distinguish between same dye types provided by different suppliers
- Detect impurities in dyes during raw material control

In total 25 dye samples of 3 different suppliers were provided. Out of these, 3 samples were contaminated with three different substances. The samples were filled into disposable glass vials with flat bottom and scanned in reflection on a NIRS DS2500 Analyzer (**Fig. 1, Tab. 1**) over the full wavelength range (400–2500 nm).

Tab. 1: Used equipment and software for identification analysis

Equipment	Metrohm number
NIRS DS2500 Analyzer	2.922.0010
NIRS DS2500 Iris	6.7425.100
NIRS DS2500 sample cup holder	6.7430.040
Vision Air 2.0 Complete	6.6072.208

Fig. 1: The NIRS DS2500 Analyzer was used for spectral data acquisition over the full range from 400 nm to 2500 nm.

Quantification of color intensity of diluted dyes:

19 aqueous solutions of the pure dye samples (without impurities) with color intensities ranging from 2780–5373 were provided to evaluate the correlation between spectral data and reference values. **Tab.2** shows the values of the color intensity *I* based on the color concentration *c* of the dye solution and the provided color strength *F* from reference values determined by UV-Vis spectroscopy. The calculation of the color intensity *I* was done as follows:

$$I = F * c$$

Sample 1-22 were used to build the prediction model, sample 23-28 were used for external validation.

Tab. 2: Sample overview. Listed are the supplier names, color concentration of the aqueous solutions, the color strength reference values from UV-Vis and the calculated color intensity used for identification and quantification.

No.	Supplier	Concentration / (mg/L)	Color strength / F / %	Color intensity / I
1	A1	47.98	111	5326
2	A1	47.44	106	5029
3	A1	47.44	100	4744
4	A1	46.70	96	4483
5	A1	45.84	91	4171
6	A1	46.60	60	2796
7	A2	46.94	105	4929
8	A2	46.52	98	4559
9	A2	47.08	95	4473
10	A2	46.58	90	4192
11	A2	47.12	59	2780
12	D	50.36	92	4633

Tab. 2: continued

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No.	Supplier	Concentr ation / (mg/L)	Color strength / F / %	Color intensity / I
13	D	51.40	90	4626
14	D	51.32	85	4362
15	D	50.54	80	4043
16	D	51.64	75	3873
17	D	51.30	70	3591
18	D	51.56	65	3351
29	D	51.80	60	3108
20	A1 + 1	53.70	93	4994
21	A1 + 2	56.80	94	5339
22	A1 + 3	57.16	94	5373
23	x	47.98	x	x
24	x	47.44	x	x
25	x	47.44	x	x
26	x	46.70	x	x
27	x	45.84	x	x
28	x	46.60	x	x

The samples were placed into 4 mm disposable glass vials and scanned in transmission on a NIRS XDS RapidLiquid Analyzer (**Fig. 2**, **Tab. 3**) over the full wavelength range (400–2500 nm) to quantify the color intensity of diluted dyes.

Tab. 3: Used equipment and software for quantification analysis

Equipment	Metrohm number
NIRS XDS RapidLiquid Analyzer	2.921.1410
NIRS disposable glass vials, 4 mm diameter	6.7402.010
NIRS XDS spacer for 6.7402.010	6.7403.010
Vision Air 2.0 Complete	6.6072.208

Fig. 2: The NIRS XDS RapidLiquid Analyzer was used for spectral data acquisition over the full range from 400 nm to 2500 nm.

In both cases, the software package Vision Air 2.0 Complete was used for data acquisition, data management and development of the identification library and quantification method respectively.

Results

Identification of impurities in dye:

An identification library was developed that can be used for three different purposes.

- Differentiate between different dye types
- Distinguish between the same dye types delivered by two different suppliers.
- Test the quality of the dye by identifying impurities.

Therefore, different wavelength ranges for the different purposes were chosen. The data were pretreated using the 2nd derivative to correct for baseline shifts.
The two different dye types display Spectral differences over the full wavelength range, but are especially prominent in the wavelength region 1570-1720 nm (see **Fig. 3**). **Fig.4** displays the overlay of the two same dye types delivered by two different suppliers. Especially, the region around 730-830 nm show significant differences between samples from supplier A1 and A2. **Fig. 5** shows the overlay of the pure and the contaminated dye samples. The region around 1750-1800 nm shows significant differences between pure and contaminated dye samples.

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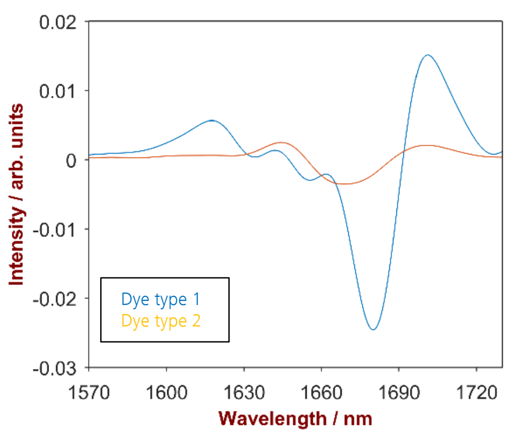

Fig. 3: 2^{nd} derivative spectra of two different dye types. The spectral difference in the wavelength range 1570-1720 nm is used to distinguish the two different dye types.

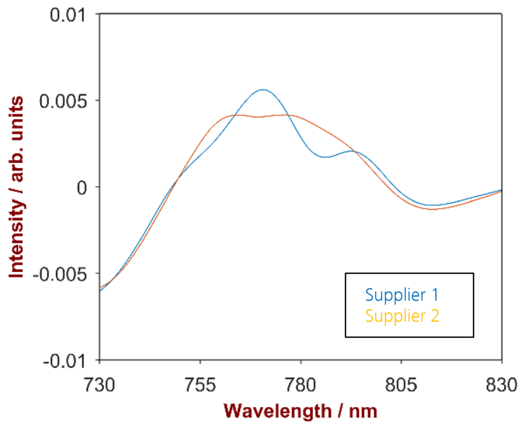

Fig. 4: 2^{nd} derivative spectra of two samples of the same dye type, but delivered from two different suppliers. The wavelength range 730-830nm shows obvious spectral differences that are used to distinguish the two different suppliers.

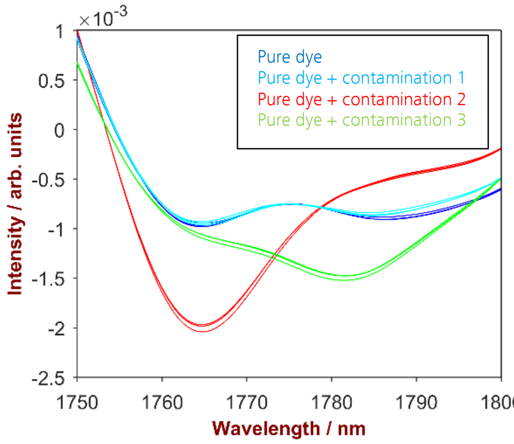

Fig. 5: 2^{nd} derivative spectra of 4 different dye sample types in the wavelength region of 1750-1800 nm. Pure dye (dark blue), dye contaminated with substance 1 (red), dye contaminated with substance 2 (green), dye contaminated with substance 3 (turquois).

<u>Quantification of color intensity of diluted dyes:</u>

A Partial Least Squares regression (PLS) was performed on 19 dye samples using a 2^{nd} derivative as data pre-treatment to correct for spectral baseline shifts. **Fig.6** shows the wavelength region of 420–740 nm, were the correlation between absorbance change and concentration change is obvious,. Internal cross-validation was applied to verify the performance of the derived quantification model.

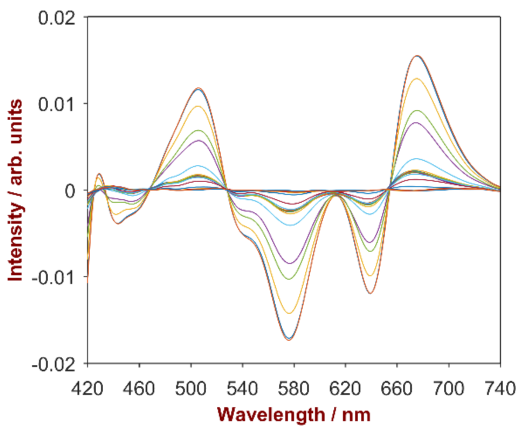

Fig. 6: 2^{nd} derivative spectra in the wavelength range of 420-740 nm. A high correlation between absorbance change and concentration change is obvious.

For the quantification of the color intensity of aqueous dye solutions, a 6-factor-model with a Standard Error of Calibration (SEC) of 108, a Standard Error of Cross Validation (SECV) of 126 and a Standard Error of

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Prediction (SEP) of 133 was developed. A high correlation between the provided reference values and the calculated values (R^2 = 0.9846) was achieved, see **Fig. 7**. Parameters used for method development and the Figures of Merit (FoM) are listed in **Tab.4**.

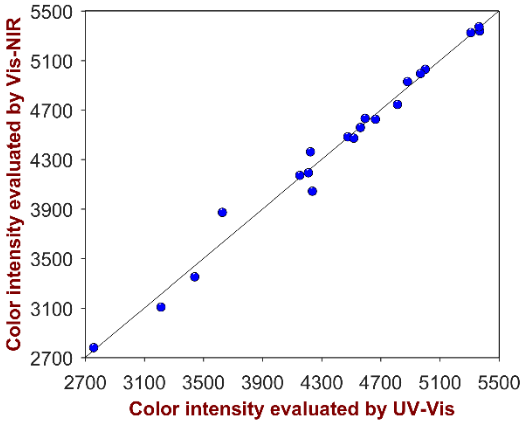

Fig. 7: Correlation plot of the predicted color intensity by NIRS versus the UV-Vis reference values. A high correlation is given.

Tab. 4: Results of the quantitative method development for color intensity.

Regression model	PLS, 6 factors
Pre-treatment	2nd derivative
Wavelength range	420-740 nm
R^2	0.9846
SEC	108
SECV	126
SEP	133

External Validation:

An external validation on an independent data set of 7 additional dye samples was applied. Samples were correctly identified with respect to dye type and supplier and were quantified in accordance to the reference analysis.

Summary

This application note shows that quantification of color intensity of aqueous dye solutions can be conveniently done with Vis-NIR spectroscopy. The calculated standard errors are similar to the UV-Vis reference method highlighting the suitability of Vis-NIR spectroscopy as an alternative that can be used for multi-purpose applications.

References

[1] http://www.essentialchemicalindustry.org/materials-and-applications/colorants.html
[2] https://en.wikipedia.org/wiki/Dyeing

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