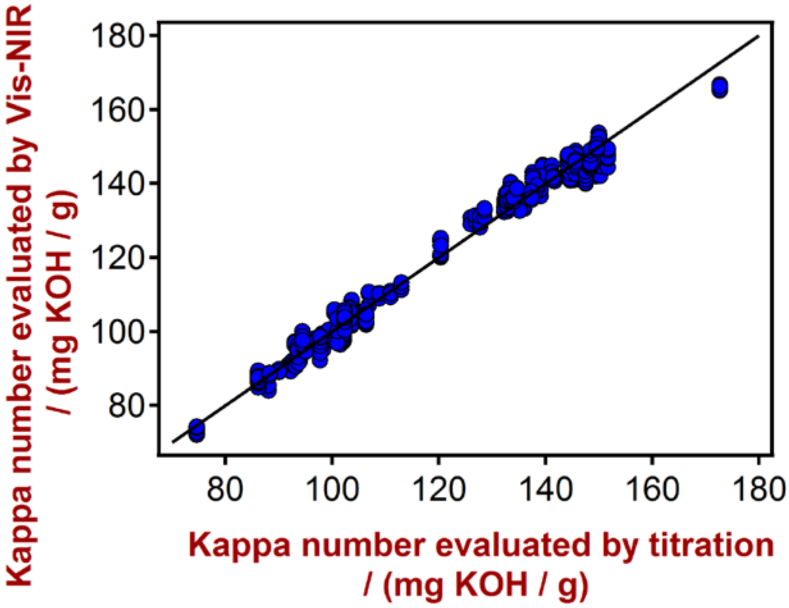
NIR Application Note NIR-39

Vis-NIR spectroscopic analysis of wood pulps for multiparameter prediction



This Application Note shows that visible near-infrared spectroscopy (Vis-NIRS) can simultaneously determine six pulp parameters (kappa number (titration), applied density (densimeter), pulp freeness (CSF), breaking strength (SCT), buckling strength (RCT), and tensile strength (tensometer)) in wood pulp. It is an alternative to conventional lab methods, resulting in a significant reduction in both time to result and chemical waste.

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Method description

Introduction

The pulp and paper industry, especially the production of office paper, tissues, and paper-based packaging material, is one of the largest industrial sectors worldwide. Around 400 million tons are produced per year. There are several chemical and mechanical pulping methods for separating wood fibers. The most common method is the Kraft pulping. Kraft pulping involves a cooking process where the chips are mixed with "white liquor" (a solution of sodium hydroxide and sodium sulfide) and heated to increase the reaction rate. The wood chips are then disintegrated into fibers by subjecting them to a sudden decrease in pressure. Once the pulp is produced, the quality should be controlled. Usability and quality of wood pulps is depending mainly on the following parameters, see Tab.1.

Tab.1: Parameter for quality determination of wood pulp and the corresponding used lab methods:

Parameter	Method
Kappa number	Titration
Pulp freeness	CSF - Canadian Standard Freeness
Breaking strength	SCT - Short Span Comprehensive Strength Test
Buckling strength	RCT - Ring Crush Test
Tensile strength	Tensometer
Applied density	Densimeter

These important parameters can be determined simultaneously in one measurement using visible-near-infrared spectroscopy (Vis-NIRS) as a much faster alternative to conventional lab methods.

Experimental

A sample group of 236 standard paper hand sheets were provided with reference values for the following six parameters, see Tab.2 and Fig. 1.

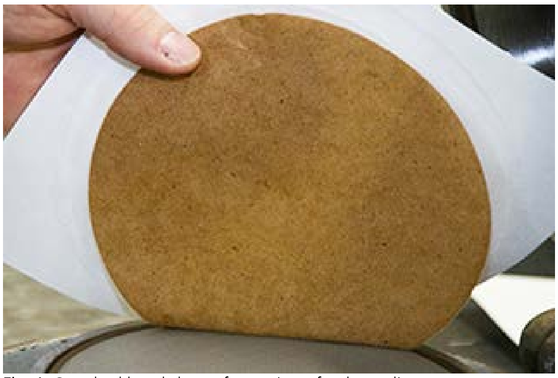

Fig. 1: Standard hand sheets for testing of pulp quality

Tab.2: Reference value range for the six different parameter for the paper sheets.

Parameter	Concentration range
Kappa number	74.6 – 174.6
Pulp freeness	130 – 740 mL
Breaking strength	7.6 – 29.8 MPa
Buckling strength	26.3 – 92.4 MPa
Tensile strength	8 – 68 MPa
Applied density	0.2280 – 0.6447 g / cm^3

The spectra were collected in reflection mode on a NIRS DS2500 Analyzer (Tab. 3 and Fig. 2) over the full wavelength range (400–2500 nm). The samples were placed directly onto the sample window and measured in triplicates.

Tab.3: Used equipment.

Equipment	Metrohm part number
NIRS DS2500 Analyzer	2.922.0010
Vision 4.03 Software	6.6069.102

Fig. 2: A NIRS DS2500 Analyzer was used to collect the spectral data of 236 samples as triple measurements in reflection mode over the full wavelength range of 400 – 2500 nm.

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Method development

The Vision, with its Partial Least Squares (PLS) algorithm, was used to develop six individual quantitative prediction models for kappa number, applied density, pulp freeness, breaking strength, buckling strength, and tensile strength. Absorption bands of the Vis– (400 - 780 nm) and the NIR (780 – 1370 nm, 1450 – 1870 nm, 1960 – 2480 nm) were selected for model development.
The typical bands for water bands in the region of 1400 and 1900 nm were excluded.
The raw spectra were pre-treated using a 2nd derivative in combination with a Standard Normal Variate (SNV) to reduce the effect of multiplicative baseline effects (e.g. spectral variation and scatter effects). Internal cross-validation was applied to verify the performance of the derived quantitative models. Parameter used for method development are summarized in **Tab. 4**.

Tab. 4: Results of the quantitative method development for Kappa number

Wavelength regions	420 – 1080 nm 1120 – 1370 nm 1450 – 1870 nm 1960 – 2480 nm
Regression model	Partial Least Squares (PLS)
Math pre-treatments	2nd derivative + SNV 10 nm segment size 0 nm gap size
Validation	Internal cross-validation with segment size 3

Results

The correlation plots, see Fig. 3 - 8, show good correlation between the parameters determined by reference analytical method (x-axis) and the predicted values (y-axis) from Vis-NIR spectroscopy. The good correlation results are confirmed by the Figures of Merit (FoM), see Tab.5 - 9.

Kappa number

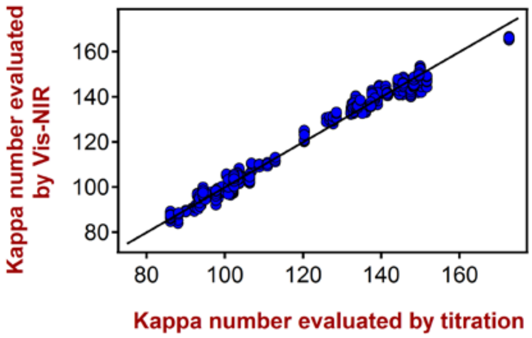

Fig. 3: Correlation plot of reference values from titration versus predicted values from Vis-NIR. The Kappa number varies between 74.6 – 174.6 mg.

Tab. 5: Results of the quantitative method development for Kappa number

Number of factors	5
R^2	0.986
SEC	2.9 mg
SECV	3.0 mg

Pulp freeness

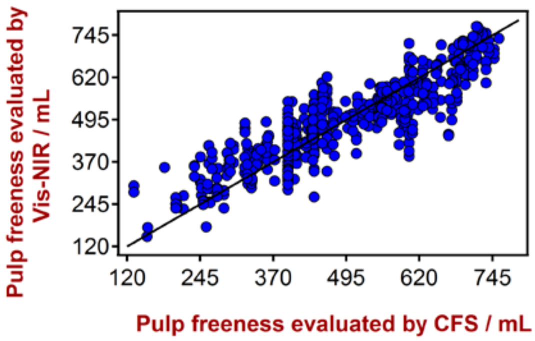

Fig. 4: Correlation plot of reference values from Canadian Standard Freeness (CSF) versus predicted values from Vis-NIR. The pulp freeness varies between 130 - 740 mL.

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Tab. 6: Results of the quantitative method development for pulp freeness

Number of factors	7
R^2	0.785
SEC	68 mL
SECV	71 mL

Tab. 8 Results of the quantitative method development for buckling strength

Number of factors	5
R^2	0.768
SEC	7.2 MPa
SECV	7.4 MPa

Breaking strength

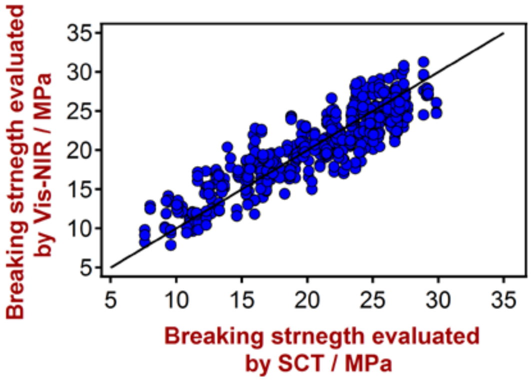

Fig. 5: Correlation plot of reference values from Short Span Comprehensive Strength Test (SCT) versus predicted values from Vis-NIR. The breaking strength varies between 7.6 – 29.8 MPa

Tab. 7: Results of the quantitative method development for breaking strength

Number of factors	6
R^2	0.803
SEC	2.5 MPa
SECV	2.6 MPa

Buckling strength

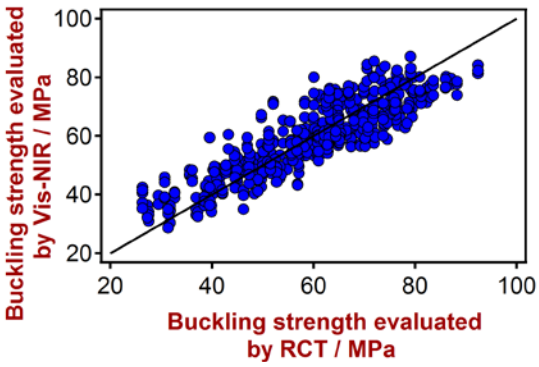

Fig. 6: Correlation plot of reference values from Ring Crush Test (RTC) versus predicted values from Vis-NIR. The buckling strength varies between 26.3 – 92.4 MPa.

Tensile strength

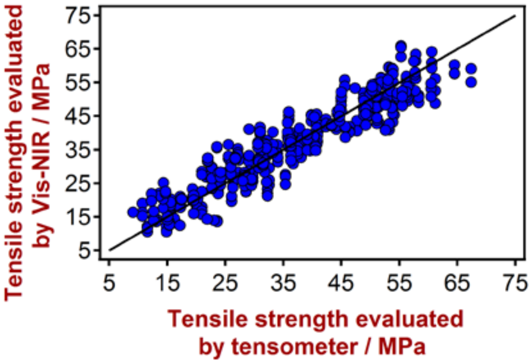

Fig. 7: Correlation plot of reference values from tensometer versus predicted values from Vis-NIR. The tensile strength varies between 8 – 68 MPa.

Tab. 9: Results of the quantitative method development for tensile strength

Number of factors	4
R^2	0.874
SEC	5 MPa
SECV	5 MPa

Applied density

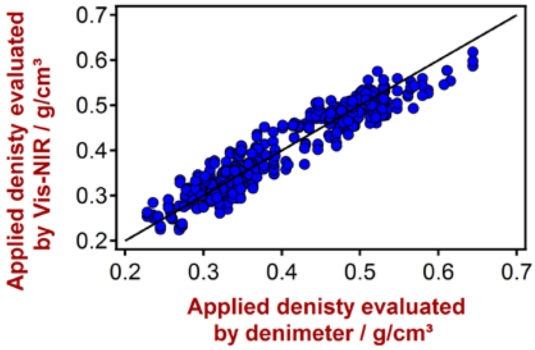

Fig. 8: Correlation plot of reference values from densimeter versus predicted values from Vis-NIR. The applied density values vary between 0.2280 – 0.6437 g / cm^3.

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Tab. 10: Results of the quantitative method development for applied density

Number of factors	5
R^2	0.903
SEC	0.0292 g/cm^3
SECV	0.0308 g/cm^3

Conclusion

Vis-NIR spectroscopy has been successfully applied to determine a number of useful parameters (Kappa number, applied density, paper freeness (CSF), breaking strength (SCT), buckling strength (RCT), and tensile strength) in wood pulp. These six parameters can be measured simultaneously in 30 seconds. Compared to the corresponding reference methods, Vis-NIR spectroscopy is much faster and does not produce any chemical waste.

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