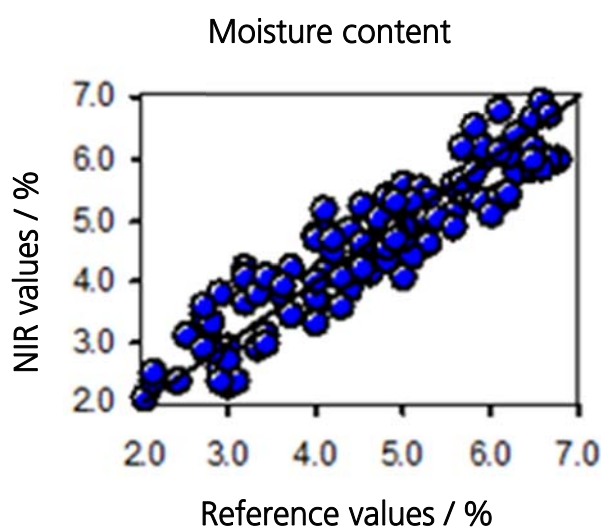# Purity, degree of substitution (DS), and moisture content of carboxymethyl cellulose (CMC)

Moisture content

This Application Note shows that Vis-NIR spectroscopy can be used to quantify three important parameters (purity, degree of substitution, and moisture content) for the production of carboxymethyl cellulose (CMC) in the pulp and paper industry. The determination of those parameters, commonly achieved with time-consuming conventional lab methods, can easily be accomplished by Vis-NIR spectroscopy. Vis-NIR spectroscopy provides results of multiple parameters within a single measurement operated by even untrained users.

Metrohm
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Method description

Introduction

Carboxymethyl cellulose (CMC) is the most important commercially used cellulose ether, prepared in the reaction of cellulose with monochloroacetic acid in the presence of sodium hydroxide. The market for CMC is estimated to show an increasing growth rate in the upcoming years. This is a result of the growth of the pharmaceutical, cosmetic and paint industry, where CMC is used e.g. as disintegrant in tablets, as thickener in creams or as binder in paints and coatings. The possible application fields depend on parameter like the level of purity, the degree of substitution (DS) or the moisture content.

The DS has a major influence on solubility and viscosity and represents the average number of carboxymethyl groups per anhydroglucose unit (approx. 0.5 - 1.5) which makes it the most important property of CMC. Moisture content (max. 8 %) and the level of purity (approx. 99 %) have to be determined to calculate the quantity of active polymer, which then affects the amount of CMC added to a certain formulation.

Selling into regulated industries (e.g. pharmaceuticals), makes a fast, but strict analytical method essential to insure high quality and product consistency.

The determination of these parameters, commonly achieved with time-consuming conventional lab methods (ASTM D1347 and ASTM D1439), can easily be replaced by Vis-NIR spectroscopy. Vis-NIR spectroscopy provides results of multiple parameters within a single measurement operated by even untrained users.

Configuration

Tab. 1: Used equipment and accessories

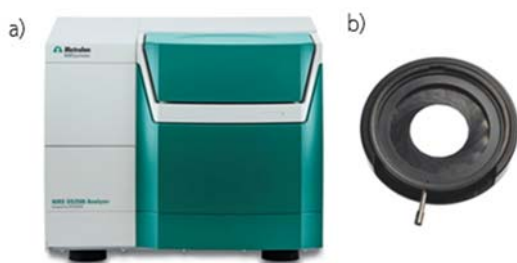| Equipment	Metrohm code
NIRS DS2500 Analyzer	2.922.0010
NIRS DS2500 Iris	6.7425.100
Vision 4.03 Software	6.6069.102

Fig. 1: a) A NIRS DS2500 Analyzer was used to record the spectral data. b) The NIRS iris adapter centered the sample glass vials above the measurement window.

Experimental

A NIRS DS2500 Analyzer (reflection measurement) was used to collect the spectral data of carboxymethyl cellulose samples containing different levels of purity and degree of substitution. An additional sample set was used to determine moisture content. The samples were placed in glass vials and were centered above the measuring window using the NIRS DS2500 Iris. The data were collected over the full Vis-NIR wavelength range (400 – 2500 nm).

Vision (Metrohm chemometrical software) with the algorithm of Partial Least Squares (PLS) was used to develop quantitative prediction models for moisture content, DS and purity, see **Tab.2**. The spectral data were pretreated using a 2nd derivative with a segment size of 10 nm and a gap size of 0 nm. External validation on an independent data set was applied to verify the performance of the derived quantitative models.

Tab. 2:

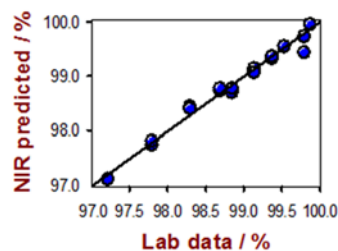| Parameter	Concentration range
Purity	97 – 100 %
Degree of substitution	0.5 – 0.9
Moisture content	2 – 7 %

Results

Purity:

Fig. 2: Correlation of NIR predicted values to lab data as a result of quantitative method development of CMC purity.

Regression model	PLS with 3 factors
Wavelength range	420-1080 nm, 1120-2480 nm
R^2	0.9836
SEC	0.1186
SEV	0.2038
SEP	0.1054
F-value	300.70
PRESS	0.7888

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Method description
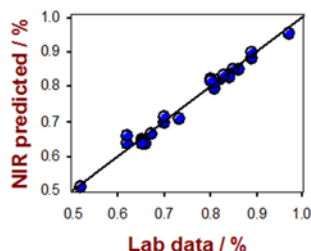
Degree of Substitution (DS):

Fig. 3: Correlation of NIR predicted values to reference data as a result of quantitative method development of the degree of substitution.

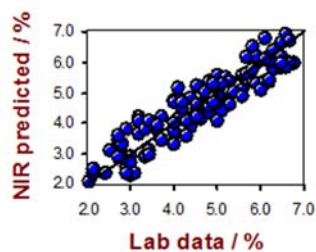| Regression model	PLS with 5 factors
Wavelength range	420-1080 nm, 1120-2480 nm
R^2	0.9817
SEC	0.0170
SEV	0.0247
SEP	0.0147
F-value	193.10
PRESS	0.0147

Moisture content:

Fig. 4: Correlation of NIR predicted values to reference data as a result of quantitative method development of moisture content.

Regression model	PLS with 3 factors
Wavelength range	420-1080 nm, 1120-2480 nm
R^2	0.8401
SEC	0.5083
SEV	0.5260
SEP	0.4992
F-value	190.89
PRESS	31.123

External validation:

	NIR value	Lab value	Difference
Purity	99,71 %	99,75 %	0,04 %
	99,43 %	99,40 %	-0,03 %
	98,74 %	98,71 %	-0,03 %
DS	0,65	0,65	0,00
	0,80	0,80	-0,01
	0,77	0,75	-0,02
Moisture content	5,2 %	5,0 %	-0,2 %
	4,4 %	4,2 %	-0,2 %
	6,6 %	6,5 %	-0,1 %

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