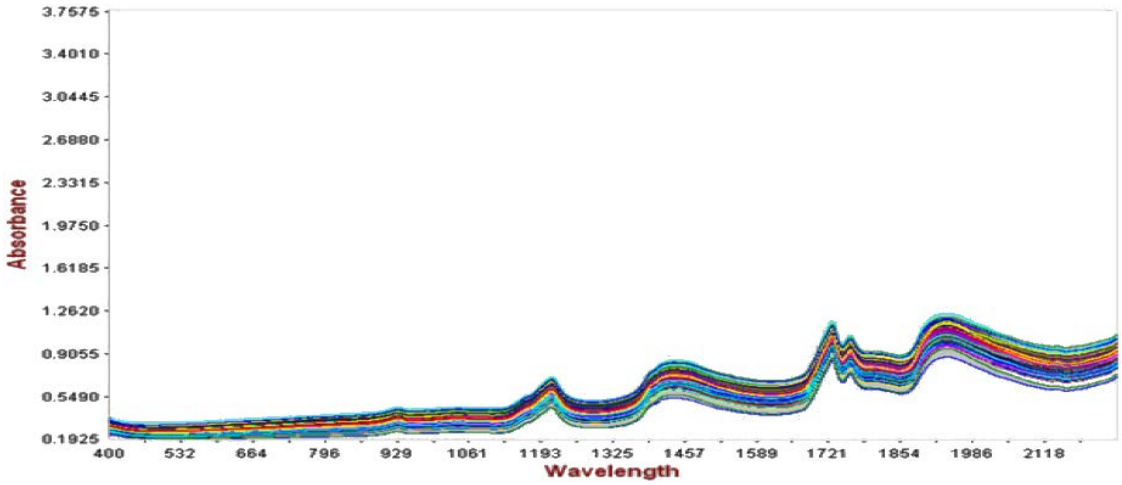
NIR Application Note NIR–51

Simultaneous determination of Total Fatty Matter, Iodine Value, and C8–C14 in soap noodles by Vis-NIRS



Near-infrared spectroscopy (NIRS) was used as an analysis method for quality control of soap noodles. Quantitative models for the determination of Total Fatty Matter, Iodine Number, and C8–C14 were developed, enabling fast and reliable quality control.

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Method description

Introduction

Soap is a widespread product in daily life. Its industrial production is a complex process that starts with saponification, which is followed by purification to remove impurities and byproducts of saponification. The soap is dried in the next step and compacted into pellets or noodles, which are used for soap finishing like converting into a soap bars. The specification of the finished product depends on the quality of the used soap noodles. Typical quality parameters that need to be determined are:

Total fatty matter (TFM): this parameter is defined as the total amount of fatty mater, which can be separated from a soap after reaction with a mineral acid. Soaps with low TFM content grasp water from the skin and cause it to become dry. This parameter is usually determined by titration.

Iodine number or value (IV): This parameter is defined as the amount of iodine in grams consumed by 100 g of sample. Low IV indicates that less unsaturated fatty acids are present in the soap and as a result the soap bar is harder. This parameter is most commonly determined by titration.

C8-C14 content: this is a sum parameter of different 8 to 14 carbon chain fatty acids, which act as surfactants. This parameter can be determined by chromatographic techniques.

An alternative for the quality control of soap noodles is the use of Vis-NIR spectroscopy. This analytical technique has the advantage that all of the above mentioned quality parameters can be simultaneously determined in less than one minute without any sample preparation, as shown in the current Application Note.

Experimental

The set of customer samples used consisted of 46 soap noodle samples. Near-infrared spectra were acquired using Metrohm XDS SmartProbe Analyzer in reflection mode (Table 1 and Figure 1). The probe was directly positioned on the sample zip lock bag and spectra were collected over the full Vis-NIR wavelength range of 400–2500 nm. The software package Vision Air 2.0 Complete was used for data acquisition, data management, and development of the quantification methods.

Tab.1: Used equipment and software.

Equipment	Metrohm order code
NIRS XDS SmartProbe Analyzer – 2 m Fiber	2.921.1610
Vision Air 2.0 Complete	6.6072.208

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

The spectra were pre-treated using a 2nd derivative and a Partial Least Squares Regression (PLS) was performed over the specific spectral regions. Typical pretreated spectra are shown in the Figure 2.

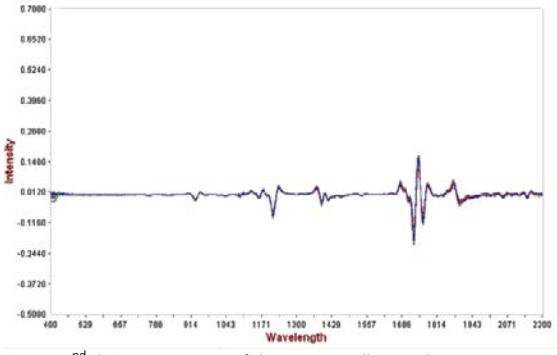

Fig. 2: 2nd derivative spectra of the soap noodle samples.

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Results

Total fatty matter

The concentration range of TFM was 78.5–80%. Fitting the model with 5 factors yielded a Standard Error of Calibration (SEC) of 0.10% and a Standard Error of Cross Validation (SECV) of 0.13% (see Figure 3).

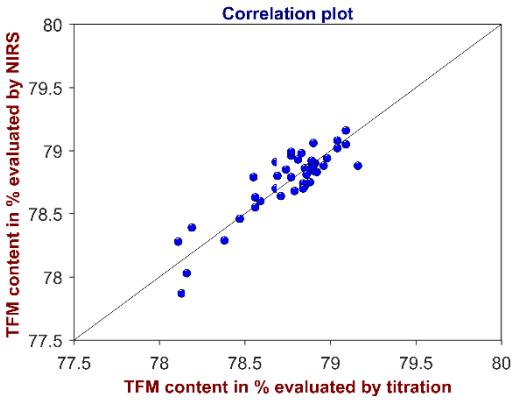

Fig. 3: Correlation plot of the total fatty matter content determined by titration and by NIRS.

Iodine value

The calibration range of iodine value was 36–40. Fitting the model with 6 factors yielded a Standard Error of Calibration (SEC) of 0.39 and a Standard Error of Cross Validation (SECV) of 0.45 (see Figure 4).

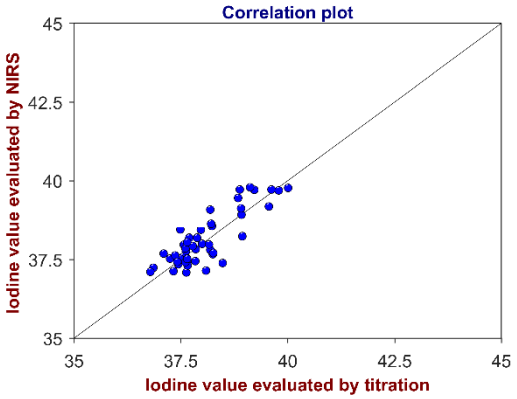

Fig. 4: Correlation plot of the iodine value determined by titration and by NIRS.

C8-C14 content

The concentration range of C8-C14 content was 1–17%. Fitting the model with 5 factors yielded a Standard Error of Calibration (SEC) of 0.17% and a Standard Error of Cross Validation (SECV) of 0.21% (see Figure 5).

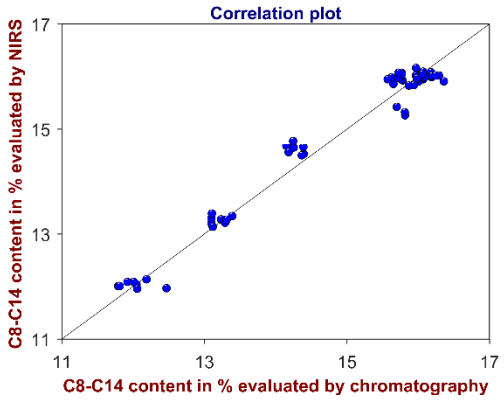

Fig. 5: Correlation plot of the C8–C14 content determined by chromatography and by NIRS.

These results show that NIRS is excellently suited to reliably determine different quality parameters in soap noodles and, thus, it can be used in quality control.

Summary

A Metrohm NIRS XDS SmartProbe Analyzer was used for the determination of total fatty mater content, iodine value and C8–C14 content in soap noodles. The calibration models of the Vis-NIR method provided results with acceptable standard errors and accuracy. It has been demonstrated that Vis-NIR spectroscopy is excellently suited for high throughput quality control analysis method of soap production steps. This approach constitutes a time- and cost-saving solution for routine analysis of soap noodles and in combination with the intuitive Vision Air Software it can even be successfully performed by untrained operators. In addition, it should be mentioned that Vis-NIR can be used for the determination of additional quality parameters in soap and soap noodles like moisture, acid value, or active detergents.

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