The use of NIR spectroscopy in the paper industry

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Introduction

The use of NIR spectroscopy for the goods receipt control has been established in many industrial areas. Also paper manufacturers, who have to handle large amount of liquid and solid additives and auxiliary substances, which are delivered on a daily basis, use this technology more and more. The substances can be analyzed much more time- and cost-effective than with conventional, often wet chemical or physical analysis methods. By using fiber optic probes, the time for taking the sample to the laboratory can be saved and moreover the cost for sampling materials. By an unambiguous identification of the received substance or a precise determination of the concentration of ingredients the amount of faulty batches can be reduced and a consistent product quality and a more constant production flow can be achieved.

The large amount of different and highly specialized products in the paper and paper processing industry need tools to ensure a constant product quality. From teabags, tissue paper, beyond photocopying paper to heavy cardboards at first it is necessary to determine the physical parameters like thickness, grammage, wet extension and moisture content. These parameters are relevant for the quality control and for further processing steps. In addition, various detectable filler materials like clay and talc or paper coatings like silicone need to be investigated.

Conventionally, from every batch roll a paper-strip of some meters length is cut off and investigated with different mechanical testing equipment which analyzes one parameter after the other offline in the laboratory. This procedure is often regarded as sufficient because the production process itself is well established and reliable. However, the FT-NIR spectroscopy is an alternative analytical method – all calibrated parameters can be determined within approx. 30 seconds instead of in ten minutes or more.

Today's paper and paper processing machines are working at very high speed and the width of the paper web is permanently increasing. If the analysis results from the laboratory are not within the specification a lot of paper will be wasted. Online measurement systems like the MATRIX-E based on the FT-NIR technology are able to detect the standard physical parameters and also very low concentrations of silicone coatings (0.2-2.3 g/m^2) at a maximum working speed of the coating machine of about 600 m/min.

Experimental

Goods receipt control of auxiliary materials for the paper production

At the paper plant of UPM-Kymmene in Augsburg, Germany, about 40 different chemical auxiliary materials ranging from sodium hydroxide, hydrogen peroxide, brightener, stabilizer and retention agents to cationized starches are analyzed by NIR spectroscopy.[1] These substances are mainly liquids, but also very viscous liquids or powders. To be able to analyze all delivered substances, an FT-NIR spectrometer, the VECTOR 22/N-F (Bruker Optik GmbH, Ettlingen, Germany) with an integrating sphere with sample rotator was used (Figure 1, D).

The fiber ports were connected to a transmission probe with 1 mm optical path length (A), a reflectance probe (B) and an external cuvette holder (C). The liquids were measured using the transmission probe. The more viscous liquids were filled in disposable cuvettes, for easier handling. The powders were measured with the reflection probe or the integrating sphere with sample rotator.

All experiments are carried out with resolution 8 cm^{-1} and 16 scans measuring time. The Bruker software OPUS-Lab helps the operator with the daily routine analysis by pre-defined interfaces for all the substances to carry out the work easier, safer and faster. With OPUS-IDENT the hierarchical libraries with different used spectral ranges and data pre-processing are set up.

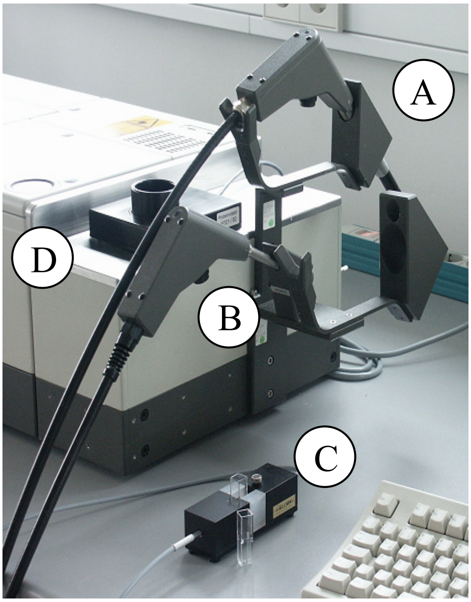

Figure 1. FT-NIR spectrometer VECTOR 22/N-F equipped with a transmission probe (A), a reflectance probe (B), an external fiber optic connected cuvette holder (C) and an integrating sphere (D)

Analysis of physical paper parameters in the laboratory

The sample set contains five different paper types from calandared up to double coated samples. Of each type of paper, between 7 and 13 samples with area weights of 60 to 300 g/m^2 were measured (altogether 53 different samples). All experiments were carried out with a VECTOR 22N FT-NIR spectrometer from Bruker Optik with an integrating sphere. The paper samples were measured three times each side in diffuse reflectance. A spectral resolution of 4 cm^{-1} in the spectral range from 3,600 to 12,000 cm^{-1} and 16 scans measuring time was used. The regression models were calculated by PLS using the Bruker Optik OPUS software. Because of the limited number of samples a full cross validation (one sample left out) was performed leading to the root mean square error of cross-validation (RMSECV). The rank represents the number of selected PLS factors.

In-line analysis of silicone content

A MATRIX-E FT-NIR spectrometer from Bruker Optik was installed at a paper coating machine at Herma (Herma GmbH, Filderstadt, Germany). The spectrometer was developed for the contact-less measurements in diffuse reflectance especially in process environments. In the focus of the four light sources a measurement spot of 25 mm diameter is generated. The distance of the instrument to the paper web is approximately 20 cm. Every ten seconds a spectrum is recorded. For the silicone coating, one glassine paper as well as one coated Kraft paper was used. For more details on parameter settings and reference analysis to built-up the quantification methods see.[2] Today, the coating machine runs at an average speed of 600 m/min, but in exceptions with 400 or 800 m/min. The resulting changes in the NIR spectra have been accounted for in the respective quantification methods. The spectrometer is meanwhile mounted on a cross beam, enabling the measurement of different positions of the paper web or outside the web for test purposes.

Results and discussion

Goods receipt control of auxiliary materials for the paper production

Over a period of several months NIR spectra were recorded of all delivered chemical auxiliary materials – parallel to the normal goods received control. With additional information on usability of the products from the production line, identification libraries were built-up which are today used as the only form of goods receipt control. Now, erroneous deliveries are recognized much earlier and time- and cost-intensive subsequent work can be avoided.

The display of the results with all substances would be too complex. Therefore an example of six different liquids is shown in Figure 2, which describes a cluster analysis (a, Euclidian distance) as well as a scores plot (b, factorization). Both calculation modes show an unambiguous separation of the single substances. In practice, all substances are identified after vector normalization in different spectral ranges and by calculation of the Euclidian distance.

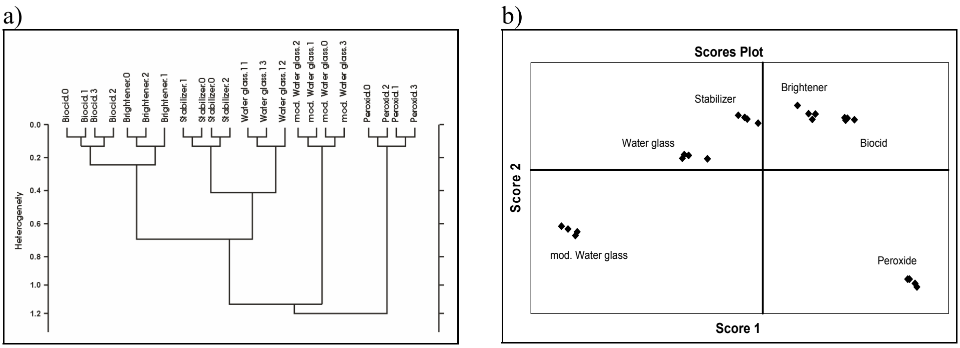

Figure 2. Separation of six different delivered liquid substances, a) cluster analysis, b) scores plot

Paper parameter analysis in the laboratory

The aim of these experiments was to show the determination of physical paper parameters by means of an optical instrument. Thickness, grammage, moisture and wet extension are necessary parameters for the paper manufacturer and for the paper fabricator, and each parameter has to be analyzed with a dedicated analytical instrument. With FT-NIR spectroscopy only one measurement is necessary to determine all calibrated paper parameters at once.

The PLS calibration results are represented in Figure 3 and summarized in Table 1. The property ranges of the single parameter were chosen with regard to the practical requirements of the paper manufacturer. Very useful calibrations were received although the determined parameters show no dedicated signal in the NIR spectrum (except moisture) and paper is an extremely complex medium. However, for this reason the calibrations need 6 to 8 PLS factors.

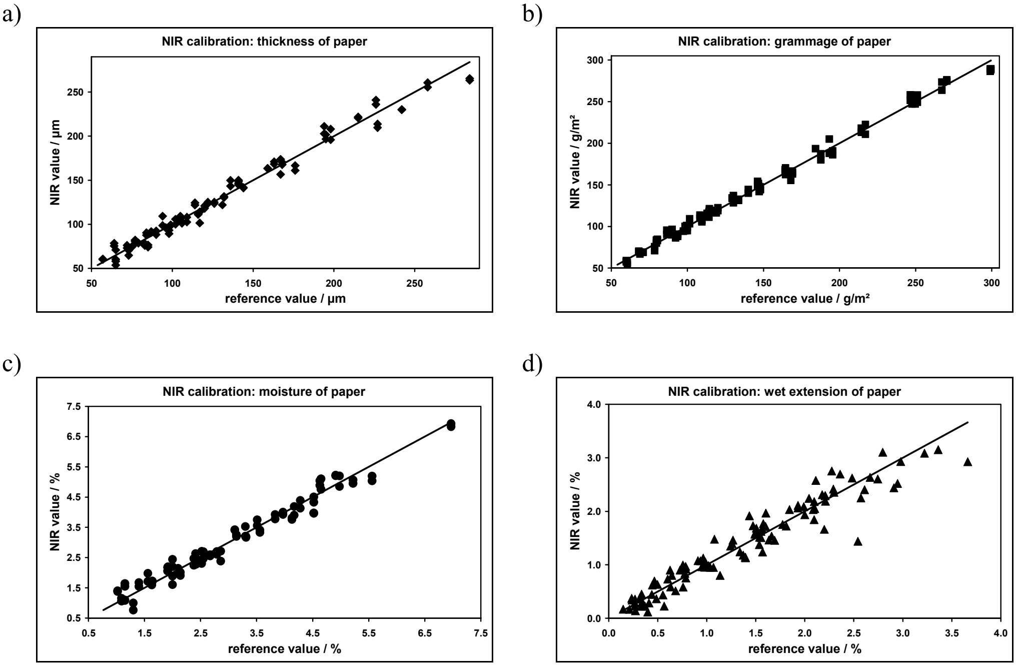

Figure 3. NIR calibration of a) thickness, b) grammage, c) moisture, and d) wet extension of different papers (53 samples)

Table 1. Calibration results of the physical paper parameters determined in the laboratory

	R^2	Range	RMSECV	Rank
Thickness	99.21	55 – 295 µm	± 7.6 µm	6
Grammage	99.36	60 – 300 g/m^2	± 5.08 g/m^2	6
Moisture	96.89	0.7 – 7 %	± 0.25 %	8
Wet Extension	92.22	0.15 – 3.66 %	±0.23 %	8

In-line analysis of silicone content

Figure 4 shows the PLS calibration results of the determination of the silicone content on glassine paper. This method contains meanwhile more than one hundred spectra and has proven to be very robust and reliable in the day-to-day routine analysis. The silicone concentrations measured in this application are already at the detection limit of the NIR technology. Still, it has been shown that only the NIR technology is able to deliver these kinds of results. Spectra with around 2,000 data points provide a good base to perform fast and precise measurements for this analytical task.

The in-line analysis of the silicone content is carried out as follows: The spectrum measured by the MATRIX-E is taken to identify the carrier material, either glassine or coated Kraft paper. Depending on the result, the respective quantification method is selected; the silicone content is calculated and handed over to the process control system to adjust the amount of silicone coating applied on the machine.

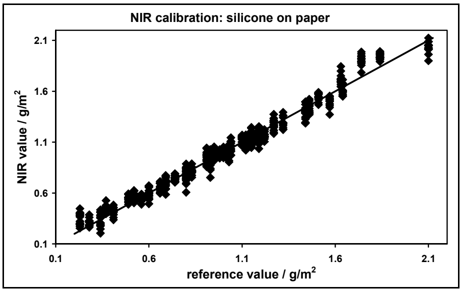

Figure 4. NIR calibration: determination of silicone content on glassine paper (R^2=98.23, 0.2-2.1 ± 0.07 g/m^2, rank=10)

Conclusion

The presented applications of FT-NIR spectroscopy for the paper industry show that this technology can accompany every step of the production. From the goods receipt control of the delivered substances and the off-line analysis of physical properties in the laboratory up to a complete in-line control of the separate production steps including an interactive communication with the process control system, everything is possible. The FT-NIR technology is a competitive alternative to the known analytical techniques, often being more time- and cost-effective.

In the future, at UPM-Kymmene (Augsburg, Germany) further substances will be added to the identification library to further extend the fields of application of FT-NIR spectroscopy.

In the laboratory the physical paper parameters are well determinable by FT-NIR spectroscopy. The next step will be to transfer this knowledge to process FT-NIR instruments for the in-line process analysis.

Acknowledgement

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