

# Characterization of sizes on textiles by in-line NIR chemical imaging

Gabriele Mirschel<sup>1</sup>, Olesya Daikos<sup>1</sup>, Carsten Steckert<sup>2</sup>,  
Katja Heymann<sup>1\*</sup>, Tom Scherzer<sup>1</sup>

<sup>1</sup> Leibniz Institute of Surface Modification, Chemical Department,  
Permoserstr. 15, D-04318 Leipzig, Germany  
<sup>2</sup> LLA Instruments GmbH,  
Justus-von-Liebig-Str. 9/11, D-12489 Berlin, Germany

**Abstract** In the last years, demands on high quality products increases dramatically, e.g. in textile industry. In order to produce high functional textiles a numerous of chemical agents have to be applied to the fabrics, in several wet chemical treatments, in the production process. One auxiliary agent is the size. Sizes are colorless, water soluble substances which improve the mechanically stiffness of threads during weaving process. Usually, sizes have to be wash out of the fabric before further processing since they may affect the following production steps. Up to now, offline process control methods e.g. extraction of the size are the state of the art of process control in textile industry. It is obvious, that the time consuming and punctual analytical methods are no more seasonable for an optimal process control. Thus, alternative process control methods were on demand.

This paper presents a study of the potential of NIR hyperspectral imaging for in-line analysis in textile technology. Application weights and spatial distribution of sizes on polyester fabric are investigated by NIR hyperspectral imaging. In a preliminary study a calibration to the application weight of the size was prepared and a PLS model was established. This PLS model was applied for the quantitative monitoring of the colorless size across the fabrics. Additionally, contaminants on the textile were visualized by NIR chemical imaging. Thus, NIR hyperspectral imaging is presented as a fast, precise and powerful analytical method which also fulfills the requirements of textile industry.

**Keywords:** Process control, in-line monitoring, technical textiles, chemical treatment, sizing, homogeneity.

## 1 Introduction

In textile technology, fabrics or threads were usually treated with various chemical agents in order to improve stiffness of the threads or to obtain a specific functionality of the refined textile. These chemical substances can be divided into two main categories: finishes and auxiliary agents. Finishes are responsible for the final characteristic of the textiles and remain on the fibers, whereas auxiliary agents such as sizes are employed to improve the mechanical stiffness of the threads during weaving process and have to be washed out before further processing.

Both finishes and sizes are usually water soluble, colorless substances, which are applied to the fabrics or threads in a wet chemical process. Both fabrics and threads are pulled through a bath, in which the specific agent is dissolved. Afterwards, excess water is squeezed out and fibers are allowed to dry before further processing. Commonly, fabrics or threads pass several impregnation or washing steps until a finished functional textile is engendered. After each dipping step not only the quantitative amount of chemical substances at the fibers is essential for the final product quality, but also its homogeneous distribution is a basic requirement of a high quality product.

It is obvious, that a visual inspection of the distribution of the agents is hardly possible due to the colorless character of the finishes and auxiliary agents. Up to now, the common method for process control is a cost-intensive and laborious work, in which random samples are cut out of the fabric and weighted. Additionally, the specific substance may be also extracted from the textile in analytical laboratories in order to confirm the determined application weight. In the last decades, this kind of off-line process control was the state of the art and was thus considered as a sufficient method.

Nowadays, the requirements regarding the quality of specific products permanently increase. Therefore, powerful methods for process control become more and more important. It is nowhere near enough to determine merely the application weight, but the spatial distribu-

tion of the substances is also of great interest for the textile industry. Furthermore, contaminations of the textile web may occur during processing, when liquids, e.g. finishes, sizes, greases or machine oil, drop down from other parts of the machine. Again, these contaminants are either colorless or have pale color and are hence, invisible or at least difficult to detect by the eyes. Furthermore, the blots may strongly affect further processing such as printing, finishing or lamination. Thus, detection and possibly identification of the contaminants as well as monitoring of their distribution on the textile web is essential for an optimal process control.

In general, spectroscopic methods are commonly used for process control, since they are practicable, fast and mostly contact-free analytical methods. In particular, NIR spectroscopy is employed for product control in agriculture, food and chemical industries for the last three decades [1–6]. With the development of NIR multispectral cameras, the scope of NIR spectroscopy was enlarged. Up to now, NIR hyperspectral cameras may be used as process control instruments, since the monitoring of the spatial distribution of compounds or parameters of interests is a great benefit. On the basis of chemometric approaches powerful calibration models can be developed, which enable both the identification of different compounds as well as quantitative monitoring of the distribution of specific agents across the web. Nowadays, research areas employing NIR chemical imaging as analytical technique cover a wide range. Among others, monitoring of foods, medical or chemical products should be mentioned [7–11].

In textile industries, NIR spectroscopy was occasionally applied for the analysis of textile blends or for the determination of the concentration of some processing additives [12–14]. In a previously published paper, in-line analysis of textile laminates, especially the monitoring of the homogeneity of adhesive layers between the textile fabrics, by NIR chemical imaging was described [15]. However, the distribution of auxiliary agents on textile fabrics has never been analyzed before by NIR spectroscopy or hyperspectral imaging. The present paper is focused on the analysis of sizes as well as the detection of contaminants on textile webs by NIR chemical imaging.

## 2 Experimental

**Sample preparation** Polyester fabrics investigated in this study were kindly provided by a textile finishing company. Sizes were applied to the fabrics by padding the fabrics in a size-containing solution in a laboratory foulard (HVF 58401, Werner Mathis AG, Oberhasli, Switzerland). Subsequently, fabrics were spanned in a tentering frame (LTE-S 54101, Werner Mathis AG) and were dried at 100°C. The size of the fabrics was approximately 100 x 170 mm. The application weights of the size were determined by a textile laboratory (CHT/Bezema, Tübingen, Germany) by extracting the size from the fabric with petrol ether. These application weights were used as reference data for chemometric calibration models.

**Hyperspectral camera and chemical imaging** A NIR hyperspectral camera system KUSTA1.9MSI (LLA Instruments GmbH, Berlin, Germany) was employed for recording NIR spectra. The NIR camera was installed above a conveyor with a black polyurethane belt with a width of 500 mm (Axmann Fördersysteme GmbH, Zwenkau, Germany). The sensor of the camera is based on an InGaAs photodiode array (192 x 96 pixels) with Peltier cooling. It covers a spectral range from 1320 – 1900 nm. A NIR objective F2.0/15 mm purchased from Specim (Oulu, Finland) was used for imaging. The line speed of the conveyor belt was set to 10 m min<sup>-1</sup>. With this setup, a lateral resolution of 2.6 mm per pixel is achieved. Further details about the measuring system are described in [15].

The partial least squares (PLS) algorithm was employed for chemometric calculations. The software package KustaSpec 16.6.3 was provided by the manufacturer of the camera system. Details about the creation of the specific PLS model are described in the Results and Discussion section. Chemical images were generated on the basis of the NIR spectra by applying the optimized PLS model.

### 3 Results

As it was mentioned in the Introduction section, the control of the impregnation or washing steps in textile industry is usually done by gravimetric determination of the application weight or by extraction of the agents from the textile, respectively. Both procedures are laborious and cost intensive. Therefore, there is great interest in an alternative process control method, which is easier, faster, cheaper and which can be used in-line. NIR spectroscopy in combination with chemometric approaches usually fulfills these requirements with respect to fast and powerful in-line measurements of various parameters of interest. However, the prediction of the application weight of size on polyester fabrics by NIR hyperspectral imaging was expected to be a very challenging task, since the absorbance of NIR light by the fabric is commonly much stronger than that of the size. Thus, this paper presents a preliminary study on the determination of the application weight of size by NIR hyperspectral imaging. Results of a more comprehensive study of the determination of the application weight of e.g. several auxiliary agents by NIR hyperspectral imaging will be published elsewhere.

Samples were prepared by padding polyester fabrics in size solution in a foulard. In a second step, some of the sized samples were washed in a desizing solution also in the foulard. After drying, application weights of both sized and desized fabrics were determined by an analytical laboratory. The application weight of the sized and the washed fabrics were determined to be  $\sim 19 \text{ g m}^{-2}$  and  $\sim 2 \text{ g m}^{-2}$ , respectively. For spectroscopic investigations, approximately 26000 NIR spectra per sample were collected with a NIR hyperspectral camera. Subsequently, the samples were divided into 10 rectangular regions by defining a grid of 5 columns  $\times$  2 rows. The spectra in each rectangle ( $\sim 2600$ ) were averaged. This procedure resulted in 10 averaged spectra that were assigned to the application weight of the sample. In this way, an unintended but unavoidable variation of the size on the fabric is included in the calibration, which leads to a more stable PLS model than without inclusion of the variance. The PLS model was calculated on the basis of the test set calibration method. Samples were split into two independent sets, a calibration set and an internal test set. In order to optimize the PLS model, different preprocessing methods were applied to the spectral data such as baseline correction, differentiation

or normalization. For further processing, a PLS model with a high coefficient of determination (0.94) and a low root mean square error of prediction (RMSEP) of  $1.3 \text{ g m}^{-2}$  was chosen. This model was based on baseline corrected spectral data, and the spectral range was limited to 1325–1841 nm. The optimum number of eigenvectors was found to be three.

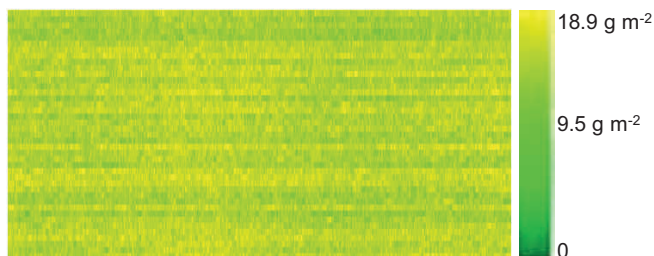
The prediction power of the PLS calibration model was tested with independent test samples, which were prepared in analogy to the calibration samples. The application weight of the size was predicted from the NIR spectra using the PLS calibration. All values of the predicted application weight (26000) of each sample were averaged. Data obtained by the PLS model and the application weights determined by the external laboratory were compared. Results are shown in Tab. 15.1.

**Table 15.1:** Comparison of the application weights determined by an external laboratory and the values of the application weight predicted by the PLS model

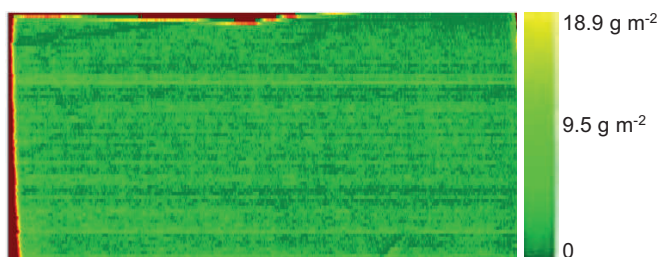
Sample	State of the fabric	Reference data [ $\text{g m}^{-2}$ ]	Predicted application weight from NIR [ $\text{g m}^{-2}$ ]	Difference [ $\text{g m}^{-2}$ ]
1	sized	18.9	18.1	-0.8
2	sized	18.9	18.5	-0.4
3	desized	2.0	2.4	0.4
4	desized	2.0	2.6	0.6

It is obvious that the predicted values and the reference data of the application weight show a close correlation. This result clearly demonstrates the feasibility of the determination of the application weight of the size on sized or desized polyester fabrics by NIR hyperspectral imaging in combination with powerful chemometric models. Chemical images of two samples (one sized and one desized polyester fabric) were calculated on the basis of the NIR spectra by applying the PLS model. In Fig. 1.1 and 1.2 the chemical images of the two fabrics are presented.

A homogenous distribution of the size on the fabric can be easily observed in the chemical images of the sample. Monitoring of the spatial distribution of a parameter of interest such as the local concentration



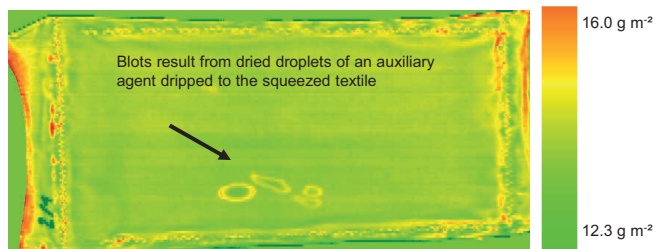
**Figure 15.1:** Chemical image of a polyester fabric before washing off the size. The application weight of the size was determined to be  $\sim 19 \text{ g m}^{-2}$ .



**Figure 15.2:** Chemical image of a polyester fabric after washing off the size. The remaining application weight of the size was determined  $\sim 2 \text{ g m}^{-2}$ .

of sizes or other finishing agents is a great milestone for the textile industry. Up to now, off-line process control methods, e.g. extraction of the substances, provide only punctual, time-delayed as well as cost intensive results. Thus, using NIR hyperspectral cameras in combination with powerful PLS models provides a powerful tool for in-line process control in textile industry.

Nevertheless, NIR hyperspectral imaging does not only enable the monitoring of the spatial distribution of an auxiliary agent etc., but allows also – after a specific calibration procedure – the detection of impurities on the fabric. Fig.1.3 presents an example of a fabric contaminated with blots of an auxiliary agent dripped to the washed and



**Figure 15.3:** Contaminations on a dried fabric. Blots of a colorless auxiliary agent may affect further processing of the textile, e.g. finishing or lamination.

squeezed textile web. The blots are not visible to the eyes, that is, the fabrics show a homogenous surface. NIR hyperspectral imaging for process control enables fast as well as spatially resolved detection of impurities or inhomogeneity on the textile web. Thus, contaminants may be detected efficiently. In this way, waste or products with insufficient quality will be reduced or avoided.

## 4 Summary

In this study, it was demonstrated that parameters such as the application weight or the distribution of impurities can be visualized with high precision by NIR chemical imaging. In this way, in-line monitoring using NIR hyperspectral cameras has a great potential to open up a new area of process control in textile industry, but also in other branches.

## Acknowledgement

The authors thank the Arbeitsgemeinschaft industrieller Forschungsvereinigungen “Otto von Guericke” (AiF, Berlin, Germany) for financial support under grant no. KF 207 4011 KM3. Furthermore, we would like to thank Marcel Bäuml for the preparation of the samples.

## References

1. H. W. Siesler, Y. Ozaki, S. Kawata, and H. M. Heise, Eds., *Near-Infrared Spectroscopy: Principles, Instruments, Application*, Wiley-VCH, Weinheim, 2002.
2. D. A. Burns and E. W. Ciurczak, Eds., *Handbook of Near-Infrared Analysis*, 3rd ed., CRC Press, Boca Raton, 2008.
3. P. C. Williams and K. Norris, Eds., *Near-Infrared Technology in the Agricultural and Food Industries*, 2nd ed., Am. Assoc. of Cereal Chemists, St.Paul/MN, 2001.
4. G. Mirschel, K. Heymann, and T. Scherzer, "Simultaneous In-Line Monitoring of the Conversion and the Coating Thickness in UV-Cured Acrylate Coatings by Near-Infrared Reflection Spectroscopy," *ANALYTICAL CHEMISTRY*, vol. 82, no. 19, pp. 8088–8094, OCT 1 2010.
5. D. L. Chicoma, C. Sayer, and R. Giudici, "In-Line Monitoring of Particle Size during Emulsion Polymerization under Different Operational Conditions using NIR Spectroscopy," *MACROMOLECULAR REACTION ENGINEERING*, vol. 5, no. 3-4, pp. 150–162, APR 13 2011.
6. J. Simpson, S. Staunton, and M. O'Shea, "A review of NIR applications for process control purposes," *INTERNATIONAL SUGAR JOURNAL*, vol. 114, no. 1358, pp. 99–103, FEB 2012.
7. H. Martens and T. Næs, *Multivariate Calibration*, John Wiley and Sons, Chichester, 1989.
8. D. Wu and D.-W. Sun, "Advanced applications of hyperspectral imaging technology for food quality and safety analysis and assessment: A review - Part I: Fundamentals," *INNOVATIVE FOOD SCIENCE & EMERGING TECHNOLOGIES*, vol. 19, pp. 1–14, JUL 2013.
9. A. A. Gowen, C. P. O'Donnell, P. J. Cullen, G. Downey, and J. M. Frias, "Hyperspectral imaging - an emerging process analytical tool for food quality and safety control," *TRENDS IN FOOD SCIENCE & TECHNOLOGY*, vol. 18, no. 12, pp. 590–598, 2007.
10. C. Gendrin, Y. Roggo, and C. Collet, "Pharmaceutical applications of vibrational chemical imaging and chemometrics: A review," *JOURNAL OF PHARMACEUTICAL AND BIOMEDICAL ANALYSIS*, vol. 48, no. 3, pp. 533–553, NOV 4 2008.
11. A. Kulcke, C. Gurschler, G. Spock, R. Leitner, and M. Kraft, "On-line classification of synthetic polymers using near infrared spectral imaging," *JOURNAL OF NEAR INFRARED SPECTROSCOPY*, vol. 11, no. 1, pp. 71–81, 2003.

12. S. Gosh and J. Rodgers, "NIR analysis of textiles," in *Handbook of Near-Infrared Analysis*, CRC Press, Boca Raton, 2008.
13. M. Blanco, J. Coello, H. Iturriaga, S. Maspoch, and J. Pages, "Use of near-infrared spectrometry in control analyses of acrylic fibre manufacturing processes," *ANALYTICA CHIMICA ACTA*, vol. 383, no. 3, pp. 291–298, MAR 15 1999.
14. M. Blanco, J. Coello, J. Fraga, H. Iturriaga, S. Maspoch, and J. Pages, "Determination of finishing oils in acrylic fibres by near-infrared reflectance spectrometry," *ANALYST*, vol. 122, no. 8, pp. 777–781, AUG 1997.
15. G. Mirschel, O. Daikos, T. Scherzer, and C. Steckert, "Near-infrared chemical imaging used for in-line analysis of inside adhesive layers in textile laminates," *ANALYTICA CHIMICA ACTA*, vol. 932, pp. 69–79, AUG 17 2016.