

Using chemometric methods and NIR spectrophotometry in the textile industry

E. Cleve*, E. Bach, E. Schollmeyer

Deutsches Textilforschungszentrum Nord-West e.V. (DTNW), Institut an der Gerhard-Mercator-Universität Duisburg Adlerstraße 2, 47798 Krefeld, Germany

Abstract

A quantitative and qualitative technique for identification of textiles, moisture measurements, textile coatings and process control was developed, using near infra-red spectroscopy (NIR) in combination with chemometric methods. These applications demonstrate by the use of computer assisted data processing the possibility of identifying textile fibers, not only for quality control but also for online textile recycling processes. In this study, seven various textile fibers (cotton, polyester, viscose, silk, wool, polyacrylonitrile, acetate) were used and all combinations of two factor blends were qualitatively identified using NIR spectroscopy and the chemometric PLS2 method for the calibration.

A quantitative analysis of textile moisture can also be performed with this technique. Water content above 50% does not deliver good results for a calibration set to determine the dampness of fibers. But to measure residual moisture from ≈ 0.05 up to 50%, the NIR technique is particularly good. Furthermore, the examination shows that the NIR method and chemometric methods can be used in quality- and product-control during the industrial production of upholstery fabrics. With this technique it will be possible to identify nylon flocks and to measure the residual moisture of the flocks and fabric, too. © 2000 Elsevier Science B.V. All rights reserved.

Keywords: Near infra-red spectroscopy; Chemometric methods; Identification of textiles; Textile moisture; Textile coating; Process and quality control

1. Introduction

The extent of the work in quality- and product-control in the textile finishing industry has grown rapidly in the last few years. For the classification of quality within the framework of quality assurance measures, quality planning and quality checking are necessary.

At first, internal production standards must be defined and then put into practice. This means that the supply of goods for the textiles, dyestuffs and textile finishing agents must be tested, and during

each production step the finishing results must also be controlled with the defined standard.

The first step is to determine the analytical method or the measuring technique which will deliver the best results for quality assurance and this must be checked to see whether it can measure the deviation from the defined quality. A very easy measuring technique for product and quality control in the textile finishing industry is the near infra-red (NIR) spectrophotometry. This can be used for many different applications, such as the analysis of fibers [1–9] and their properties, for example, the degree of maturity and sugar content of cotton fibers [10–14] and for on-line monitoring of different textile coatings [15–20], for example, measurements of durable resin on fabrics [21].

* Corresponding author. Tel.: +49-2151-843-155;
fax: +49-2151-843143.
E-mail address: cleve@dtnw.de (E. Cleve).

A good evaluation of the analytical data is possible by the use of modern chemometric methods for planning the experiment, evaluation and description of the data [22–23]. This must be carried out to collect maximum information from the experimental results. The problem in this field is not the evaluation of the NIR spectra or the data recording for the calibration set, but the determination of the influence of the brightness, color intensity, dampness and the surface of the textile fabric and finishing agents on the NIR spectra. To determine the standards, these influences must be taken into consideration in the calibration set [24].

This work shows a variety of different applications in the textile area for the use of the NIR spectroscopy in combination with chemometric methods for the description and calibration of the experimental data.

2. Experimental

2.1. NIR measurements

The NIR remission spectra were recorded with an on-line NIR diode indium gallium arsenide array spectrometer with a spectral range between 1200 and 2200 nm [25]. The textiles were illuminated with a cold light source and for the detection of the diffusely reflected NIR signal from the textile fiber a sensitive sensor was developed. The NIR spectra can be taken within milliseconds with the NIR sensor connected with a light fiber to the spectrometer. All textile fibers were measured in diffuse remission and as a reference barium sulfate (BaSO_4) was used for the white standard.

2.2. NIR measurements for textile identification

The NIR measurements for textile identification were carried out with seven different fibers: cotton (CO), polyester (PES), viscose (CV), silk (TS), wool (WO), polyacrylonitrile (PAN) and acetate (CA) and all combinations of two factor blends. The fibers were ground into a powder using liquid N_2 and the powders of two fibers were mixed in equal amounts (50/50), giving 21 combinations. The NIR remission spectra of the seven fibers and 21 combinations were mea-

sured in the range of 1450–1850 nm because in this wavelength range the spectrum is not influenced by dampness [24]. The first derivative of all the spectra was calculated, to minimize the influence of brightness, color intensity and the structure of the textile fabric [24].

2.3. NIR measurements of textile dampness

The amount of water for the analysis of residual moisture content of a hydrophobic (PAN) and a hydrophilic fiber (CV) was measured with a balance and correlated with the NIR signal at the wavelength of 1928 nm (absorption band of water in the NIR range).

2.4. On-line measurements to determine the textile dampness during a drying process

The equipment for the on-line determination of dampness during a textile drying process consists of a padding unit for wetting the PES fiber and an oven heated to 160°C for drying the textile. The NIR sensor is fitted at the end of the oven and a spectrum was taken every 10 s during the drying process. From the spectrum the residual moisture content of the fiber was calculated at the wavelength of 1928 nm after calibration.

2.5. Quality control in industrial production of upholstery fabrics

The NIR spectra of nylon flock (PA 6.6) with different degrees of fineness (0.9 and 2 dtex (1 dtex is the weight of 10,000 m in grams)) for quality control in the industrial production of upholstery fabrics were measured in the range of 1300–2000 nm.

3. Results and discussion

3.1. Identification of textiles for quality control and textile recycling processes

One important application of NIR spectroscopy in the textile finishing industry is the identification of fibers and the qualitative detection of components of fiber blends [1–9]. During these determinations, it is

not only possible to use this for quality control, but also to sort and separate the fibers for textile recycling automatically.

For textile recycling, the chemometric PLS2 method was used to evaluate the data for the calibration of the system to identify the fibers automatically from their NIR spectra [26]. To work out the calibration set, we used seven different fibers and all combinations of two factor blends. The inverse Lambert–Beer law was used for the calibration task.

$$C = E \cdot Q^2 \quad (1)$$

This is formulated by a regression of a matrix of dependent properties C (standards) to a matrix of independent variables E (derivative spectra of the NIR remission data). Q is the unknown parameter matrix.

The first task is to calculate the unknown parameter matrix with the chemometric PLS2 method by the use of the defined matrix of standards which is shown in Fig. 1a schematically.

Fig. 1a shows which of the fibers were used in each of the samples. If the fiber was used, it is described in the diagram with the value 1 and if not with the value 0; for example, sample 1 contains only CO and sample 28 contains PAN and CA.

After calculation of the parameter matrix, unknown fabrics were identified from the measured NIR data.

3.2. Determination of textile dampness

During a lot of textile production processes a constant water content of the fibers is required. It is known from literature that the determination of the moisture of solid, powdered and liquid samples is a main field of NIR spectroscopy [27–34] too.

The object of our research is to determine the maximum water content and minimum residual moisture content of different fibers for which we can still obtain good NIR signals for the qualitative analyses of textile dampness.

In the textile finishing industry, a distinction is made between a film of water (layer of water on the textile) and residual moisture content (fixed water contained within the fibers).

Fig. 2 shows the spectra of CO under atmospheric conditions (a) and after drying at 100°C (b). In the NIR wavelength range, there are two absorption bands of water. The first at 1450 nm and the second at 1930 nm. The main variation can be seen at ≈1928 nm. Here the fiber contains between 8.5 and 10.5% water.

Two different fibers, a hydrophobic (PAN) and a hydrophilic fiber (CV) were used for the analysis of textile dampness. The measured intensity at the wavelength of 1928 nm in relation to the water content measured with a balance is presented in Fig. 3.

For water content above 50%, we do not obtain good NIR signals which are sufficient for a calibration set

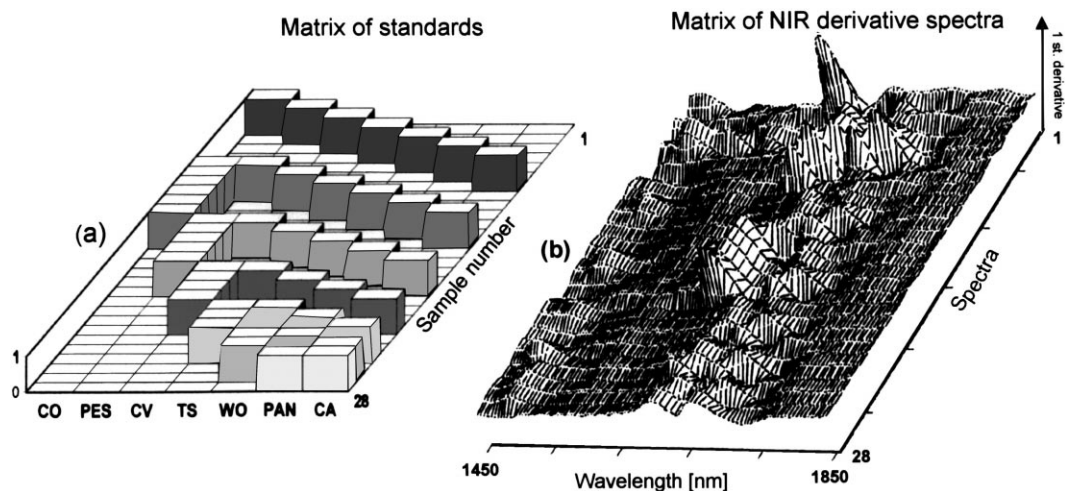


Fig. 1. Schematic representation of the matrix C , of standards (a) and the matrix E of the NIR derivative spectra (b) of the 28 textile samples.

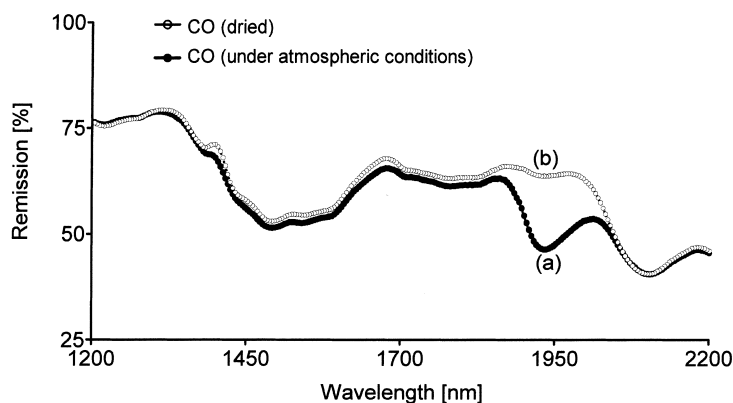


Fig. 2. NIR remission spectra for CO measured under atmospheric conditions (a) and CO dried at 100°C (b).

to determine the moisture of fibers. But for residual moisture from ≈ 0.05 up to 50%; this is an extremely good technique.

This method can also be used for on-line determination of dampness during a textile drying process. It is tested at a PES fiber after drying at 160°C. From the

measured NIR spectra during processing, the residual moisture content of 25% was calculated consistently after drying.

3.3. Using NIR spectrophotometry for quality control in the industrial production of furniture upholstery fabrics

The next investigations demonstrate that the NIR method can be used for quality- and product-control in the industrial production of upholstery fabrics. During the industrial production of upholstery fabrics the adhesive is applied to the back of the fiber using a spreader. Then nylon flock (PA 6.6) is put into an electrostatic field vertically onto the surface of the fabric and the adhesive dried and condensed.

In special cases, nylon flock with different degrees of fineness (0.9 and 2 dtex) were used which to the naked eye, look the same. For the production process it is important to differentiate between the two flocks. Also, during the electrostatic process the fabric and flock must have a defined constant dampness. Only then will the fabric have obtain a velvety look and be of good quality. Fig. 4 shows the spectra of different nylon flocks.

Although the remissions of the two different flocks look very similar, the derivative spectra are significantly different at 1699 nm. At wavelength 1928 nm, the difference in the dampness can be seen.

Using the measured spectra you see it is possible to differentiate the flocks and measure the residual moisture content using the NIR technique.

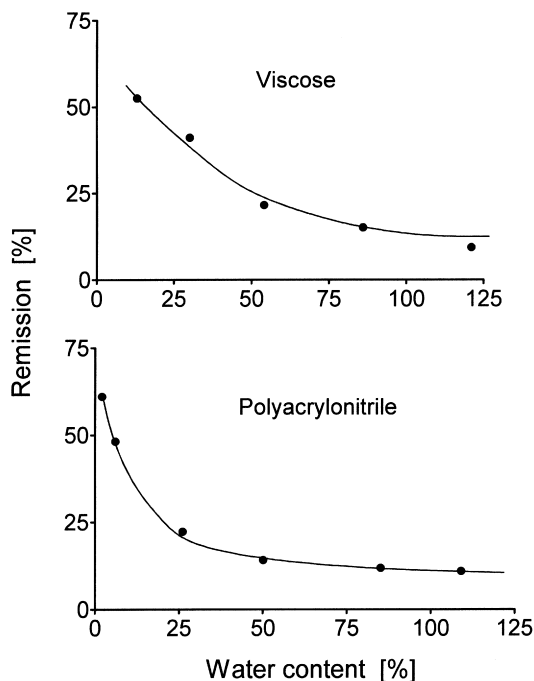


Fig. 3. NIR remission intensity of a hydrophilic (CV) and a hydrophobic fiber (PAN) at the wavelength of 1928 nm in relation to water content in percent.

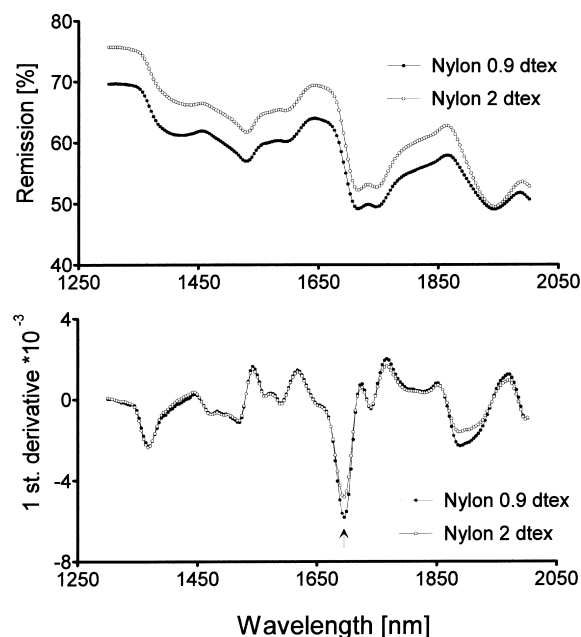


Fig. 4. NIR spectra of nylon flocks with different degrees of fineness and the calculated first derivative of the spectra.

Acknowledgements

We would like to thank the Forschungskuratorium Gesamttextil for their financial support of the research project AIF-No. 10583. This support was granted from the resources of the Federal Ministry of Economics by means of a supplementary contribution to the Association of Industrial Research Organization (Arbeitsgemeinschaft Industrieller Forschungsvereinigungen, AiF).

References

- [1] J.E. Rodgers, S. Lee, *Text. Res. J.* 61 (1991) 531.
- [2] R. Williams, E. Setzer, R. Taylor, in: *Proceedings of Conference on Near Infrared Spectroscopy*, Aberdeen, 1991, p. 501.
- [3] J.E. Rodgers, in: *Proceedings of the 47th Symposium of American Association of Textile Chemists and Colorists*, Cberlone, USA, 1990, p. 1.
- [4] J. Rodgers, S. Lee, *Text. Chem. Color.* 17 (1985) 200.
- [5] H.H. Ramey, *Text. Res. J.* 51 (1981) 20.
- [6] C.S. Suh, in: *ATC 95*, in: *Proceedings of the 3rd Asian Textile Conference*, Vol. 1, Textile Research and Development in the Pacific Rim, Hongkong, 1995, p. 105.
- [7] H.E. Howel, J.R. Davis, in: *Book of Papers of International Conference and Exhibition of AATCC*, Boston, USA, American Association of Textile Chemists and Colorists, 1990, p. 166.
- [8] H.E. Howell, J.R. Davis, *Text. Chem. Color.* 23 (1991) 69.
- [9] W.J. Jasper, E.T. Kovacs, *Text. Res. J.* 64 (1994) 444.
- [10] S. Ghost, in: *Proceedings of Beltwide Cotton Conference*, Vol. 3, Cotton Council of America, San Diego, USA, 1994, p. 1459.
- [11] B. Tennigkeit, T. Schneider, *Melliand Textilber.* 77 (1996) 18.
- [12] S. Ghosh, H.M. Ghorashi, in: *21st Internationale Baumwoll-Tagung*, Bremen, 1992, p. 89.
- [13] D.P. Thibodeaux, in: *21st Internationale Baumwoll-Tagung*, Bremen, 1992, p. 31.
- [14] J.G. Montalvo, S.E. Faught, B.T. Vinyard, S.M. Bucu, in: *Proceedings of Beltwide Cotton Conference*, Vol. 2 (of 3), National Cotton Council of America, New Orleans, 1993, p. 1155.
- [15] M.J. Drews, S.L. Harris, in: *Proceedings of the 47th Symposium of American Association of Textile Chemists and Colorists*, Charlotte, USA, 1990, p. 1.
- [16] S. Ghost, G.L. Brodmann, *Text. Chem. Color.* 25 (1993) 11.
- [17] N.M. Morris, S. Faught, E.A. Catalano, J.G. Montalvo, B. Andrews, *Text. Chem. Color.* 26 (1994) 33.
- [18] B.T. Knight, *J. Coated Fabrics* 21 (1992) 260.
- [19] E. Cleve, E. Bach, W. Kesting, E. Schollmeyer, *Textilveredlung* 30 (1995) 169.
- [20] J.E. Rodgers, *Text. Chem. Color.* 26 (1994) 19.
- [21] S. Ghost, G. Brodmann, in: *Proceedings of the International Conference A, Exhibition of AATCC*, Charlotte, USA, American Association of Textile Chemists and Colorists, 1991, p. 204.
- [22] M. Otto, *Chemometrie, Statistik und Computereinsatz in der Analytik*, Verlag Chemie, Weinheim, 1997.
- [23] H. Martens, T. Naes, *Multivariate Calibration*, Wiley, New York, 1993.
- [24] E. Cleve, E. Bach, E. Schollmeyer, *Textilveredlung* 9/10 (1998) 35.
- [25] IKS Optoelektronik Meßgeräte GmbH, Mannesmannstr. 161, D-47259 Duisburg, Germany.
- [26] Software Unscrambler EX 5.5, CAMO, Computer-aided modelling A/S, Olav Tryggvassonsgt. 24, N-7011 Trondheim, Norway.
- [27] H.W. Siesler, C. Paul, *Nachr. Chem. Lab.* 37 (1989) 171.
- [28] K. Molt, *GIT Fachz. Lab.* 2 (1992) 107.
- [29] K. Molt, *GIT Fachz. Lab.* 4 (1992) 353.
- [30] K. Molt, *Mol. Struct.* 349 (1995) 345.
- [31] K. Molt, *GIT Fachz. Lab.* 4 (1998) 386.
- [32] M.J. Hammersley, P.E. Townsend, G.F. Grayston, S.L. Ranford, *Text. Res. J.* 65 (1995) 241.
- [33] E. Setzer, R. Williams, R. Taylor, *Nat. Cotton Council Am.* 2 (1993) 1154.
- [34] K. Molt, *Mol. Struct.* 410/411 (1997) 565.