Talanta 188 (2018) 91–98

Contents lists available at ScienceDirect

Talanta

journal homepage: www.elsevier.com/locate/talanta

Near-infrared chemical imaging used for in-line analysis of functional finishes on textiles

Gabriele Mirschel[a], Olesya Daikos[a], Tom Scherzer[a,*], Carsten Steckert[b]

[a] Leibniz-Institut für Oberflächenmodifizierung e.V. (IOM), Chemische Abteilung, Permoserstr. 15, D-04318 Leipzig, Germany
[b] LLA Instruments GmbH, Justus-von-Liebig-Str. 9/11, D-12489 Berlin, Germany

ARTICLE INFO

Keywords:
Hyperspectral imaging
Impregnation
Application weight
In-line monitoring
Homogeneity
Process control

ABSTRACT

This paper demonstrates for the first time that near-infrared (NIR) chemical imaging can be used for in-line analysis of textile finishing processes based on impregnation. In particular, it was shown that this analytical method is sufficiently sensitive for the quantitative determination of the application weight of rather thin layers of finishing chemicals. Quantitative analysis of the data recorded by a hyperspectral camera (1320–1900 nm) was based on chemometric approaches using the partial least squares (PLS) algorithm. In this work, a flame retardant and a polyvinyl acetate-based stiffening agent applied to polyester or cotton fabrics, respectively, were studied with application weights in the range between about 1 and 50 g m^{-2}. For both systems, the prediction error (RMSEP) was found to be about 1.5–2 g m^{-2}. Averaging of the predicted individual values of the application weight of the finishes across the complete surface of the fabric resulted in a very close correlation with the corresponding reference values obtained by gravimetry. Furthermore, NIR chemical imaging was used for the detection of remaining traces of a size (a processing agent) after washing, which had to be washed-out before subsequent processing steps. Results of the present investigations prove that even for very thin size layers between 0.4 and 5.5 g m^{-2} the application weight can be predicted with a precision of about 0.4 g m^{-2}.

Apart from the quantitative determination of the application weights, the use of NIR chemical imaging for the analysis of finished textiles was mainly directed towards the investigation of the spatial distribution or the homogeneity of the applied colorless finishes across the surface of the fabrics. It was shown that this method is able to detect and visualize various inhomogeneities on the finished textiles resulting for instance from processing defects or from various technical effects that may influence the drying process and consequently the spatial distribution of the finish. Moreover, the distribution of traces of size that had been sprayed purposely on a washed polyester fabric could be detected.

All measurements in the present study were carried under conditions that were very similar to those in typical technical processes (e.g. with respect to line speed). Therefore, the outstanding performance of the method opens an immense potential for application in process and quality control.

1. Introduction

The permanently increasing demands on product quality and homogeneity pose a continuous challenge not only to the design of the production process itself, but also to measuring and process control technologies that are indispensable to meet these requirements. A broad spectrum of analytical methods is used for the wide range of specific problems to be considered in process monitoring [1,2]. Nevertheless, in case of special requirements, new analytical methods and instruments have to be developed in order to be able to monitor the process or the product quality with adequate accuracy, speed and extent.

In the past, the compliance of the specified value of certain parameters within a tolerance range and intervention into the process in case of deviations was the main focus of most process control approaches. However, in case of the production or processing of web-like or planar materials such as paper, polymer films, textiles, plates, sheets etc., the homogeneity of the material and the *spatial distribution* of quantitative values of parameters resulting from finishing steps such as coating, impregnation, varnishing, lamination, etc. become more and more important. Typical parameters of interest include for example color, gloss, the thickness of substrates or coatings, the application weight of finishing layers, the absence of defects etc. This is the domain of imaging technologies. Whereas techniques working in the visible spectral range mainly provide information about the optical appearance

* Corresponding author.
E-mail address: tom.scherzer@iom-leipzig.de (T. Scherzer).

https://doi.org/10.1016/j.talanta.2018.05.050
Received 2 February 2018; Received in revised form 8 May 2018; Accepted 13 May 2018
Available online 17 May 2018
0039-9140/

of the material, substantial information about chemical or physical parameters can be obtained by imaging in the near-infrared (NIR) range.

NIR spectroscopy has been used for several decades as a versatile and powerful analytical tool for quality and process control [3–6]. In particular, NIR reflection spectroscopy as a noncontact method is widely used. It allows measurements with an excellent precision, if chemometric approaches are applied for quantitative evaluation of the spectral data [17]. NIR process spectrometers are robust and compact and can be separated from their probe head by connecting them via glass fiber, which simplifies integration of the equipment in complex production lines. Consequently, NIR spectroscopy found numerous applications in many areas ranging from agriculture and food production to chemical engineering, e.g. in pharmaceutics, polymer production, textiles, and waste sorting [7–14]. The advent of large hyperspectral cameras working in the NIR range considerably broadens the potential of NIR-based methods for process control. Such instruments can scan surfaces with a field of view up to the range of a square meter, which makes them much better suited for process control than imaging techniques based on MIR microspectroscopy [15,16]. NIR hyperspectral cameras use one dimension of their sensor for spatial resolution (similar to a line camera), whereas the other one serves for spectral resolution. Two-dimensional images are obtained by moving the sample below the camera, either by mounting the camera above a conveyor belt or above web-like material in a converting plant. In combination with chemometric algorithms hyperspectral cameras may constitute a mighty tool for large-scale chemical imaging. One of the first applications of NIR chemical imaging was the sorting of plastic waste [18–20]. Further studies mainly dealt with the characterization of various foods such as meat, fish, vegetables, fruits and many others [21–27]. In contrast, only very few studies were focused on applications in chemistry or chemical engineering. Several papers were dealing with applications in polymer processing. Especially, they were targeted on the thermal curing of embedding resins, on extrusion and crystallization processes as well as on the control of the industrial processing of natural rubber [28–31]. Recently, the use of hyperspectral imaging for on-site discrimination of synthetic fibers has been reported [32].

The objective of this work is directed towards the quantitative monitoring of continuous finishing processes applied to web-like materials such as paper, polymer films and textiles in order to gain comprehensive spatio-temporal information about the current state of the process and the quality of the product. Monitoring of such processes is a novel application area of NIR hyperspectral imaging with high technologic significance. One of the challenges of this approach is the typically rather low thickness or application weight of coatings or finishing layers applied to the substrate, which makes high demands on the sensitivity of the method. The present paper deals with the finishing of textiles by wet chemistry processes such as impregnation. Agents applied as finishes provide the textile fabric with special functional features. For example, such formulations include stiffening agents, optical brighteners, flame retardants, hydrophilic or hydrophobic agents, anti-static, and anti-microbial finishes etc. Depending on the special substrate, agent, and intended application they may be applied with application weights in the range from less than 1 g m^{-2} to several tens of g m^{-2}. However, finishes have to be applied in compliance with the specified value and with a high extent of spatial homogeneity for further processing of the finished textiles as well as for optimum application properties. In addition to these functional finishes there are special processing agents such as sizes, which are applied to textile yarns before weaving to improve their processability and machinability. Sizes have to be removed in a washing step before further processing such as lamination or printing in order to avoid adhesion or wettability problems.

Although the technical processes used for wet-chemical finishing of textiles are roughly the same as one century ago, the quality requirements regarding the applied quantity and homogeneity have been considerably increased since that time, which requires careful in-line monitoring of the finished products. Most finishes are colorless solutions or emulsions, which form colorless layers on the fabric. Therefore, visual inspection after drying is not possible. On the other hand, there was also no analytical method capable for in-line inspection so far that allowed monitoring of the quality of such layers. Consequently, quality control is still based on off-line tests of local areas. In order to get quantitative information about the applied coating weight, random samples have to be cut out of the fabric and analyzed by extraction in a laborious and time consuming process. Evidently, this method is neither suited for direct process control nor can provide information about the spatial distribution of the finish on the textile.

In this study, we will show that NIR chemical imaging using a large hyperspectral camera is the adequate tool for a highly efficient process monitoring in textile impregnation. This technique is able to provide precise quantitative data on physical and chemical parameters and their spatial distribution in very large areas. In our previous paper [33], we have demonstrated that NIR chemical imaging can be used for in-line monitoring the thickness and the uniformity of inside adhesive layers in textile laminates as well as on foamed plastics. The thickness of the adhesive layers was in the range between 20 and more than 100 g m^{-2}. In comparison to that study, the application weights of the finishing layers in the present work are about one order of magnitude lower. In case of sizes, the required sensitivity is even higher since weights of the remaining traces on the fabric after desizing are well below 1 g m^{-2}. Nevertheless, we will show that the sufficient precision of the measurements can be achieved by using appropriate chemometric approaches.

2. Experimental

2.1. Materials and sample preparation

All samples investigated in this study correspond to finished textile products, which are used as technical textiles in quite different applications. They were either taken from production processes or prepared in a similar way using the same textiles and finishing agents as in these processes. In this work, three textile materials were used for finishing: a natural-colored cotton fabric (240 g m^{-2}) and two pale beige polyester fabrics (200 or 125 g m^{-2}). Samples made in the laboratory were usually prepared by impregnation of the textiles with aqueous solutions of the finishes in a padder (HVF 58401, Werner Mathis AG, Oberhasli, Switzerland). For comparison, some samples were prepared simply by soaking them in a glass dish. Afterwards, wet fabrics from the padder (also known as foulard) were fixed on a tentering frame (LTE-S 54101, Werner Mathis AG) and dried at 150 °C (cotton) or 180 °C (polyester), respectively, whereas samples made manually were dried in a drying oven (at the same temperatures). All samples that were impregnated in the padder were kindly prepared by the TITV Textile Research Institute (Textilforschungsinstitut Thüringen-Vogtland e.V., Greiz, Germany), whereas simple specimen were made at IOM.The thick polyester fabric (420 × 280 mm) was finished with a flame retardant based on an aqueous mixture of equal parts of methylphosphonic acid and amidino urea (Aflammit MSG; Thor GmbH, Speyer, Germany), which was applied with application weights roughly from 5 to 50 g m^{-2}. The cotton fabric (420 × 280 mm) was impregnated with a stiffening agent consisting of an aqueous dispersion of polyvinyl acetate (PVAc) (Eurovac D 60 NV; Cebra Chemie, Bramsche, Germany) resulting in application weights between about 1 and 20 g m^{-2}. The thin polyester fabric was treated with a size, i.e. an agent that improves the processing of textile fabrics, which is composed of hydrocarbons and fatty acid ethoxylates (Torsinol ZSB; Zschimmer & Schwarz, Burgstädt, Germany). Samples (105 × 145 mm) with different application weights between 0.4 g m^{-2} and 5.5 g m^{-2} were provided by Thorey Textilveredelung (Gera, Germany). The subsequent washing-out of the size was carried out with a commercial aqueous desizing solution. After desizing, the samples