



Near-infrared chemical imaging used for in-line analysis of inside adhesive layers in textile laminates



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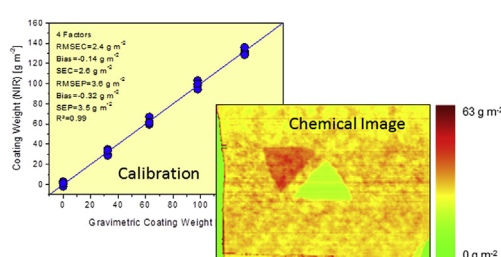
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HIGHLIGHTS

- Hyperspectral imaging was used for monitoring of textile lamination processes.
- The coating weight of adhesive layers inside fabric laminates was determined.
- The prediction error (RMSEP) was found to be in the order of 2–6 g m⁻².
- Even the homogeneity of adhesive layers inside black laminates could be monitored.
- Large-scale NIR chemical imaging is suited for process control in field-scale.

GRAPHICAL ABSTRACT



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ABSTRACT

This paper demonstrates for the first time that near-infrared (NIR) chemical imaging can be used for in-line analysis of textile lamination processes. In particular, it was applied for the quantitative determination of the applied coating weight and for monitoring of the spatial distribution of hot melt adhesive layers using chemometric approaches for spectra evaluation. Layers with coating weights between about 25 and 130 g m⁻² were used for the lamination of polyester fabrics and nonwovens as well as for polyurethane foam. It was shown that quantitative data with adequate precision can be actually obtained for layers applied to materials with significantly heterogeneous surface structure such as foam or for hidden layers inside fabric laminates. Even the coating weight and the homogeneity of adhesive layers in composites consisting of black textiles only could be quantitatively analyzed. The prediction errors (RMSEP) determined in an external validation of each calibration model were found to range from about 2 g m⁻² to 6 g m⁻² depending on the specific system under investigation. All calibration models were applied for chemical imaging in order to prove their performance for monitoring the thickness and the homogeneity of adhesive layers in the various textile systems. Moreover, they were used for the detection of irregularities and coating defects. Investigations were carried out with a large hyperspectral camera mounted above a conveyor. Therefore, this method allows large-area monitoring of the properties of laminar materials. Consequently, it is potentially suited for process and quality control during the lamination of fabrics, foams and other materials in field-scale.

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1. Introduction

Quality and process control becomes increasingly important in more and more branches of industry. This development is driven by economic and ecologic requirements such as high demands on quality, which have to be met on a constant and guaranteed high level, and the efficient use of raw materials, energy and other production media [1,2]. Spectroscopic methods belong to the most powerful and most widely used approaches in process control, since they can provide a very broad range of information without any significant time delay and can be operated in a contact-free mode without any impact on the material studied. In particular, near-infrared (NIR) reflection spectroscopy is an efficient and versatile analytical technique, which became one of the common methods in in-line process control in the last years [3–9]. It allows measurements with excellent precision, in particular when chemometric methods are used for quantitative evaluation of the spectral data. Equipment is compact, robust and rugged and may be installed at a distance, which is helpful for installation in a production environment. Moreover, it may be rather cost-efficient in comparison to other methods.

NIR spectrometers may be separated from their probe head, which can be linked to the main unit by an optical fiber. This allows installation of the probe head at positions, which are difficult to access or subject to safety regulations or where only limited space is available. On the other hand, the design and the small size of most NIR probes strongly limit the sample area, which can be recorded. Typically, the measuring spot is in the order of few millimeters only. In case of moving materials such as layers on a paper or polymer web, textiles, materials on a conveyor etc. only a small strip of the sample can be monitored, which might be linear or sinusoidal, when the probe is see-sawed on a traverse. For many applications this may be sufficient. However, in some applications the spatial distribution of the parameter of interest is important, for example in order to be able to control the homogeneity of the material. As far as only the optical appearance of the materials is relevant, the corresponding information can be obtained by a conventional camera working in the visible range. But if chemical properties or information about hidden layers that are not visible from outside are required this approach is not any longer sufficient. This is the domain of hyperspectral chemical imaging.

Chemical imaging using spectroscopic methods such as mid-infrared (MIR), NIR or Raman spectroscopy was typically carried out in microscopic setups in the past using either motorized x-y tables or two-dimensional detectors (e.g. focal array plane detectors). Numerous investigations have been carried out in medical, biological, agricultural, and food applications as well as in pharmaceuticals [10–14], polymer science and many other areas [15]. Although the potential of this method is extremely high, it is obvious that this approach is not suited for quality and process control in field-scale, for example in continuous processes such as coating, lamination, finishing of materials, plastic waste sorting or for large individual samples such as fish, meat, fruits, vegetables etc.

The development of large stand-alone hyperspectral cameras working in the NIR range in recent years opened new analytical possibilities for large-scale spectroscopic chemical imaging, which finally resulted in new application areas of this method, in particular in quality and process control [16]. In contrast to chemical imaging in microscopes coupled to Fourier transform spectrometers, where spectral resolution is obtained by scanning the moving mirror and consequently both coordinates of the two-dimensional detector may be used for spatial resolution, only one axis of the detector is used in NIR cameras for spatial resolution, whereas the other one is required for spectral resolution. Laminar resolution is

achieved by the motion of a conveyor, above which the camera has to be mounted. This allows continuous monitoring of web-type materials or large objects that run through the field of view of the camera. Quantitative data are usually obtained in a similar way as in conventional NIR spectroscopy: calibration models are established on the basis of the spectra of well-defined calibration samples by application of chemometric approaches [17]. During real measurement in process monitoring, these calibration models are used for in-line prediction of the chemical or physical parameter of interest from the recorded spectra and the subsequent compilation of chemical images of the spatial distribution of this parameter across the surface of the sample.

Although large-scale NIR hyperspectral imaging is still a rather new analytical method, a number of applications have already been reported. One of the earliest and still most prominent applications is in-line classification of synthetic polymers for plastic waste sorting [18,19]. Numerous investigations dealt with the characterization of diverse foods such as fish [20–24], meat [25–29], fruits [30,31], vegetables [31], mushrooms [32] many and other food-stuffs and biomaterials [33,34]. In contrast, only a few studies were directed towards applications in chemistry, most of them in the field of polymer processing. Lillhonga et al. studied the curing of a phenol formaldehyde resin used as backing material for sandpapers [35]. Bulk properties of natural rubber were analyzed by da Silva and Pasquini [36]. Other investigations were focused on quality control in polymer extrusion processes [37,38] or monitoring the crystallization of polylactic acid and its concentration in blends with another biodegradable polymer [39]. Moreover, the concentration of detergent and dispersant additives in gasoline was determined after application of the samples to filter paper [40]. Due to their usually small size, pharmaceutical systems are not subject of investigations by NIR hyperspectral imaging using camera systems. In fact, they are studied preferably by microspectroscopic approaches of chemical imaging [10–12].

The present paper deals with a new application area of NIR hyperspectral imaging. It was the intention of this work to apply this powerful spectroscopic method qualified for in-line monitoring for process control in textile processing technology. This includes processes such as the finishing of textiles by wet chemistry processes or the lamination of various textile fabrics resulting in composite materials. In the latter case, a thin layer of an adhesive, e.g. a hot melt, has to be applied to the fabrics (as powder, melted mass or melt-blown web) forming a continuous hidden layer inside the laminate. The thickness and the homogeneity of this layer significantly determine the final properties as well as the durability of the textile laminates. However, there is no analytical method capable for in-line inspection to date that allows monitoring of the quality of such layers, so that quality control is still mainly based on laborious visual inspection. NIR-based chemical imaging is expected to be the adequate tool for this kind of process monitoring.

Textiles fabrics and fibers have already been studied by conventional NIR spectroscopy in the past. An important application is the classification of materials for the sorting of used textiles and carpets [41–43]. Most applications in textile production processes are focused on the fabrication of fibers. In particular, the characterization and continuous monitoring of the specific fiber quality during the processing of natural fibers is one main objective of investigations by NIR spectroscopy in textile technology. For example, the purity, the fiber quality and the content of impurities of cotton, flax, alpaca wool and other natural fibers have been studied [43–47]. Furthermore, the degree of mercerization of cotton or its modification with polycarboxylic acids was monitored [43,48]. During the production and processing of synthetic fibers and yarns, NIR spectroscopy was found to be a helpful tool as well. The spinning of acrylic fibers was controlled by monitoring the

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