



## Analysis method

## Multivariate image analysis for inspection of multilayer films



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## ABSTRACT

The potential and effectiveness of near-infrared (NIR) hyperspectral imaging was investigated for rapid and nondestructive spectral-spatial inspection of functional multilayer films. A series of film samples having different combinations of layers were used to demonstrate the sensor performance. Multivariate Image Analysis (MIA) was applied to NIR hyperspectral images of unprinted multilayer films with different compositions to distinguish them regarding absence/presence of particular layers. Since printing of commercial brands on the films may interfere with product inspection, the ability of MIA to detect the presence of the functional layer (barrier) in spite of film surface printing was also assessed. The results show that the proposed MIA approach is successful at classifying multilayer films, even in the presence of printing.

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## 1. Introduction

The largest market for polymer resins is the packaging industry [1]. Plastic packages are extensively used to protect food products against physical, chemical, biological and environmental factors [2]. Functional barrier films are used to reduce migration of authorized substances below the specific migration limit (SML) and/or the migration of non-authorized substances into food [3]. Several articles describing the off-line development of new barrier films involving various material combinations and their properties can be found in the literature [3–6]. However, literature on methods for real-time inspection of these multilayer films once put into production is not as common. Fluctuations in raw material properties caused by lot-to-lot variability and supplier changes, slight variations in the formulations and processing conditions are expected in the film manufacturing industry and all of these disturbances may affect the film properties. In spite of the high production rates, film quality control is often performed by

off-line inspection and testing in the laboratory using destructive and time-consuming analytical techniques applied to a limited number of samples per lot. The film quality attributes measured in this context may not be representative of the entire lot and some of the more subtle film defects may go undetected because of the small percentage of inspected surfaces. This is an important limitation, especially for food and medical applications, which require higher inspection rates to prevent health hazards and reduce economic losses.

In addition to physical defects (e.g. spots, contamination and gels) and product dimensions (e.g. film thickness), chemical composition of multilayer films (e.g. presence/absence of a layer) needs to be monitored because of its impact on the functionality of final products. Very few articles on the subject of on-line quality control of plastic films can be found in the literature. A camera and special pattern-recognition software have been proposed to detect different physical defects such as spots, burns and gel particles on running cast or blown films [7]. Wide-angle X-ray diffraction (WAXD) has been applied for real-time structural studies on low-density polyethylene (LDPE) blown films [8,9], but this technique is limited to pilot or lab scale experiments [8,10]. Raman spectroscopy is a convenient technique and has been used for molecular

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orientation evolution of LDPE and microstructural measurement of polypropylene (PP)/LDPE blown films [11,12]. However, Raman instrumentation is more expensive compared to infrared (IR) since it requires signal amplification and fluorescence suppression devices, and the lasers used for excitation heat the samples and may change or damage them [13–15]. A commercial on-line Fourier transform infrared (FT-IR) spectroscopy system (APLAIRS) has been proposed by Optical Control System GmbH (OCS) for blown and cast films, but this system is based on point-scan and can only be applied to extract morphology and chemical information concealed in the spectra. Thus, no information about spatial features can be obtained by this system [16]. Alternatively, Gosselin et al. proposed visible (VIS) hyperspectral imaging to predict LDPE/polystyrene (PS) film properties and provided real time 2D estimation of blend composition [17]. In order to monitor crystallinity spatial distribution of polyethylene (PE) and PP films, a machine vision approach based on NIR imaging was proposed [18]. Recently, the crystallinity and crystal evolution of polylactic acid (PLA) films and their concentration in PLA/poly-(R)-3-hydroxybutyrate (PHB) blends has been investigated using a new high-speed two-dimensional NIR camera (Compovision) [19].

The objective of this work is to extend the application of hyperspectral imaging for spectral-spatial analyses and mapping chemical composition of multilayer barrier films in order to discriminate and classify products based on absence/presence of specified composition (layer). It is shown that NIR hyperspectral imaging associated with chemometric methods, such as multivariate image analysis (MIA), is a promising method for scanning the whole surface of samples, differentiate multilayer films and detect absence/presence of specific layers. Since printing may be an obstacle for product classification, the robustness of the method on printed multilayer films is also demonstrated. It is worth mentioning that the proposed methods can be considered and are applicable in a systematic preventive approach, to meet Hazard Analysis and Critical Control Points (HACCP) regulations, in which food safety is addressed through monitoring multilayer films used for food packages.

## 2. Material and methods

### 2.1. Description of film samples

The samples investigated are listed in Table 1. The films are to be applied on different types of plastic containers in order to obtain finished products with specific functions to meet market demand in various applications (e.g. food, pharmaceutical, etc.). These films were supplied by IPL Inc. in two sets. Set 1 consists of unprinted films (samples 1–4) with a different number of layers: polypropylene (PP), a primer (PR), a top coat (T), and a barrier layer (B). The layers were added sequentially in the same order as just listed to make films with 1–4 layers (e.g. PP/PR means PP first and then PR). The chemical composition of the layers is proprietary information and cannot be revealed. However, the objective of this work is to develop a film inspection scheme and not to present the development of a new material. Set 2 consists of two printed films (samples 6 and 7) with the

**Table 1**  
Multilayer film samples.

Set	No	Code	Layers	Dimensions	
				Width (mm)	Thickness ( $\mu\text{m}$ )
1	1	PP	PP film	50	56
	2	PP/PR	PP/primer film	50	58
	3	PP/PR/T	PP/primer/top coat film	50	58
	4	PP/PR/T/B	PP/primer/top coat/barrier film	50	60
2	6	Non-barrier	PP/top coat/printing	80	58
	7	Barrier	PP/top coat/barrier film/printing	80	63

same printed graphics but one of them has the functional (i.e. barrier) layer missing. The films in Set 2 have been manufactured similarly to those in Set 1, but no primer was used. Note that a limited number of samples were available for this study since the product was in the development phase at the time. Therefore, one film sample was investigated for PP, PP/PR and PP/PR/T (Set 1, number 1–3), while for the final product PP/PR/T/B (Set 1, number 4) three different films were investigated. For Set 2, two different films were analyzed for each sample as the training and validation sets.

Note that, initially, the inspection scheme was intended to be used by the food container manufacturer and was aimed at inspecting films that are cut and ready for application onto food containers. This explains the relatively small dimensions of the films samples shown in Table 1. However, if the approach was to be applied on films of larger dimensions, for example on the film manufacturer lines, the proposed approach could also be used but the imaging hardware may require a different combination of lenses and sensor technology and/or size to obtain the desired spatial resolution and field of view. Large scale and high speed applications using NIR imaging technology are already commercially available in different fields such as chemical and material sorting [20,21].

### 2.2. Hyperspectral image acquisition

A line-scan NIR imaging system composed of a NIR camera (Xenics XEVA USB-FPA), covering the 900–1700 nm spectral range integrated with a grating spectroscopy (Specim ImSpector N17E) were used to perform image acquisition. An image of resolution  $256 \times 320$  pixels along the spectral and spatial axes, respectively, is obtained at each acquisition (Fig. 1). The images have 0.4 mm/pixel nominal spatial and 3 nm/pixel nominal spectral resolutions. NIR images were captured in transmission mode from moving samples at 0.3 mm intervals and were then cropped separately for each sample. The NIR images were corrected for environmental and instrumental variability (e.g. scattering, uneven lighting, pixel-to-pixel differences in the charge-coupled device (CCD) array, etc.) using the standardization procedure proposed by Liu et al. [22]. This method was selected because it does not require using expensive calibration standards. The reader is also referred to Martens and Næs (314–356) [23] for more details on spectral data correction. Autoscaling was chosen as the

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