Food Packaging and Shelf Life 17 (2018) 91–98

Contents lists available at ScienceDirect

Food Packaging and Shelf Life

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

Design and evaluation of an in-line system for gas sensing in flow-packed products

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ARTICLE INFO

Keywords:
Tunable Diode Laser Absorption Spectroscopy
In-line oxygen sensing
Flow packaging
Modified atmosphere packaging

ABSTRACT

A method for the in-line application of Tunable Diode Laser Absorption Spectroscopy (TDLAS) for headspace oxygen sensing in flow-packed products is presented. This is one of the first automated, industrial application on food packaging samples of a well known spectroscopic technique.The developed instrument is discussed starting from its sensor design with details on the optical working principles to point out the peculiarities and application issues linked with this particular technique. A production line implementation of the sensor is then presented, based on a conveyor belt for automatic sampling. This device was also integrated with line automation in order to enable measurement on the full production. Both in-line and off-line instruments were validated against traditional, industry-standard invasive measurement techniques. The accuracy of the measurement applied to regular samples from production line was found better than 0.4% vol. O_2.

1. Introduction

An important aspect in the quality control of food packaging is the evaluation of gas composition inside packages which are not under vacuum, but sealed in modified atmosphere (MAP, Modified Atmosphere Packaging). Most food products' shelf life can be extended substantially if packaged in a vacuum or modified atmosphere, even without the use of additives (Robertson, 2012). However, vacuum is not a viable option for porous foods or fragile food items where shape must be preserved. The presence of O_2 as a residual component from atmospheric air is linked with faster spoilage and mold growth and has to be avoided accurately during the packaging process. In order to ensure this, food is flushed with proper gas mixtures in the headspace (mainly nitrogen and carbon dioxide in variable concentrations) during the packaging process before sealing the container (Ghidelli & Pérez-Gago, 2016; Lee, Yam, & Piergiovanni, 2008; McMillin, 2008; Sandhya, 2010; Sivertsvik, Rosnes, & Bergslien, 2002).

This process has to be done at quite high speeds in an automated line, and many parameters can influence the residual amount of atmospheric components in the headspace of the sealed package. Among those parameters, some are linked to the flushing process, while others can be related to product shape and porosity (trapped gases) or even equilibrium unbalance with gases dissolved in the food.

A lot of effort is done at the food manufacturing plants to be able to maintain tight enough tolerances on food packaging headspace composition to ensure a long shelf life while still minimizing the amount of time and flushing gas required during the packaging phase. Control of gas composition is currently accomplished by randomly sampling the packages followed by measurements with invasive techniques. In this case, samples have to be discarded and only a minute fraction of the product will actually be tested.

The industry standard measurement techniques are either electrochemical (requiring gas sampling and flow through a sensor cell) or optical, based on fluorescence quenching (Hodgkinson & Tatam, 2013; Lakkis, Younes, Alayli, & Sawan, 2014). Although the latter technique is considered noninvasive, it requires a fluorophore dot which must be in contact with the gas inside the package; it can be excited with a short wavelength (usually blue light) to measure its fluorescence at a longer wavelength through a sufficiently transparent packaging material.

Tunable Diode Laser Absorption Spectroscopy (TDLAS) techniques are already mature enough and used throughout the process industry to monitor process gas composition in a completely non-invasive way (Werle, D'Amato, & Viciani, 2008). This technique involves propagation of light from a laser source through the sample gas and recording of the

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https://doi.org/10.1016/j.fpsl.2018.06.006
Received 27 October 2017; Received in revised form 4 June 2018; Accepted 15 June 2018

light output while the source wavelength is scanned over one or more gas absorption features, usually in the near or mid infrared. The laser sources are essentially low-power, robust single-mode laser diodes such as VCSELs or DFBs, similar to those used in the telecommunication industry. The addressed gas spectral lines are by nature very narrow (about a few GHz) and definitely narrower than any absorption feature due to solid or liquid matter in the optical path. For this reason, the method can be adopted quite successfully for measurements through packaging materials, even though they can be scattering, absorptive or with variable optical properties from sample to sample (Cocola et al., 2016). Depending on the package and content nature, light propagation inside the samples can be more or less direct, sampling different gas path lengths. The TDLAS technique applied to a very scattering media (i. e. where equivalent path length is several times the geometrical source-detector distance) is called Gas in Scattering Media Absorption Spectroscopy (GASMAS) and usually requires a secondary, reference path length measurement to give a calibrated gas concentration output (Li, Lin, Zhang, Svanberg, & Svanberg, 2017; Lundin, Cocola, Lewander, Olsson, & Svanberg, 2012; Mei, Somesfalean, & Svanberg, 2014).

A family of instruments based on this technique was developed in the context of the SAFETYPACK EU-project in the FP7 program to enable sensing of gas content (in particular oxygen and carbon dioxide) in flow-packed products using laser spectroscopy in a contactless and non-invasive way for in-line real-time application on 100% of the production (http://www.safetypack-project.eu/ (accessed on 2 February 2017)). The aim of the project was to develop sensors that could provide the food manufacturing industry with a control technology to perform in-line quality and safety control on a wide range of sealed food packaging. The sensors operate on packaging units (such as containers, bags, cups, etc.) whose optical properties vary from high transparency, which enables viewing of packed products, to almost opaque packages, which screen the content from daylight. Two pilot installations have been fully validated for in-line operation at the production plants of two end-users.

The instruments discussed in this paper have been developed having soft food bags as target packages. This is a very important difference from most spectroscopic instrument applications. In particular, the target application within the project is packaging of pre-cut mozzarella cheese for the manufacturing of pizza. This product has the appearance of cubed or shredded mozzarella pieces in flow-packed bags. The aim of the laser control is to measure the residual O_2 content inside sealed packages to assess the safety and quality of the MAP process. Two instruments have been developed and are here described; results of validation measurements are reported for an indication of the measuring performances compared and referenced with other industry standard methods. This performance comparison is provided as an indication of the suitability of the TDLAS method to soft, flow packed food samples.

The purpose of the paper is to present the results achieved within the in-line implementations for O_2 measurement on flow-packed food, with different levels of automation and integration.

2. Material and methods

The instruments were designed and constructed, and all measurements were performed for plastic bags of mozzarella cheese (Fig. 1) as the objects of this study.

2.1. Working principle and measurement issues

Gas detection with TDLAS is based on the same principles as any absorption spectroscopy measurement (Linnerud, Kaspersen, & Jaeger, 1998). The relation between gas concentration and the intensity of the detected absorption feature is given by the Lambert Beer law:

$$I = I_0 e^{-kcl}$$

where I is the detected light intensity, I_0 the source intensity and the absorption is proportional to the product of equivalent path length l and concentration c; the constant k is a property of the chosen absorption feature and can be found on spectroscopic databases or determined experimentally by calibration. It can vary widely depending on the nature of the absorption line. When the optical path is non-homogeneous, the attenuation term cl actually becomes $\int c(z)dz$ taken along the optical path. This means that only the integrated concentration can be measured. In the case of detection over an irregular optical path containing broadband absorbers and scatterers, I_0 must take into account all the losses involved and simply represents the light intensity at the detector when no gas absorption is found.

This very simplified theory immediately points out the main limits and challenges for the applicability of this technique when measuring O_2 in a flow-packed bag (Cocola, Fedel, Poletto, & Tondello, 2015):

- O_2 is an essential component of the atmosphere while is mainly avoided inside packaged food products. The measured optical path outside the sample must be minimized to avoid a large background absorption signal.
- I_0 may be a residual fraction, in the range from 1/10 to 1/1000, of the actual optical power emitted at the source due to variable absorption and scattering in the package material and content. Its value must be accurately measured to provide a normalization of the measurement. This can be done by applying an amplitude modulation over the laser signal.
- Optical path length must be kept constant and either known or calibrated to provide a measurement in concentration units. This is especially difficult on a soft sample such as the flow-packed bag, which does not have a fixed shape and headspace.
- Gas absorption intensity can be very low (absorption coefficient of a few 10^{-5}) due to the nature of O_2 spectral features in the region where single-mode tunable laser diodes are available. In order to overcome the difficulty of a reliable detection of weak absorption artifacts, Wavelength Modulation Spectroscopy (WMS) is used (Mei & Svanberg, 2015). This increases the signal to noise ratio due to the high-frequency lock-in detection of the signal, thus minimizing the $1/f$ noise component of the laser source.

The WMS implementations discussed in this work are based on a thermally stabilized Distributed Feed Back (DFB) laser operating around 760 nm with an emission power of 15 mW at the chosen wavelength. Scanning and modulation are applied to laser diode current.

In order to improve rejection of stray interference fringes arising from multiple scattering paths in the sample, the laser collimation assembly is mechanically dithered to destroy optical coherence. In this way the interference noise is washed out over long enough averaging of the photodetector signal.

A large area (5.6 mm × 5.6 mm) Si PIN photodiode with an adjustable-gain AC-coupled transimpedance amplifier is used as the receiver. Both the source and the detector signals are managed by a Data Acquisition (DAQ) card sharing the same clock throughout the digital-to-analog and the analog-to-digital converter blocks. In this way a fully software-defined lock-in detection is arranged (Svensson et al., 2008). The block diagram is shown in Fig. 2.

Fig. 3 shows all the signal processing stages needed for a WMS setup working on the 2f component, including the typical method for normalization based on the 1f component. An example of the photodetector signal with its Fourier transform is reported in Fig. 4. The DC component of the 1f signal (shown in Fig. 5) is essentially due to residual amplitude modulation, making it a good parameter for normalization of the absorption signal in conditions of very variable average sample transmission.

The demodulated and filtered signal is then processed through a fitting algorithm in order to provide maximum noise rejection on the measurement. A relevant noise component of the measurement is