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Validation of an in-line non-destructive headspace oxygen sensor☆

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ABSTRACT

Measurement and control of the gas composition of packaging headspace in situ is required by food and packaging industries. Contactless non-destructive Tunable Diode Laser Absorption Spectroscopy (TDLAS) gas sensors have been recently developed to enable real time in-line headspace characterization in a wide range of sealed food packaging.

The objective of this work is to validate a new optical method versus existing destructive oxygen sensing methods that are standard for the industry. For this purpose, oxygen concentration measurements have been carried out in the same packaging samples using both the TDLAS gas sensor and a calibrated, intrusive sensor. The intrusive sensor has been assumed to estabilish the reference oxygen concentration data. Different types of packaging have been used for the validation measurements. The measurements have been implemented with different data integration times.

The obtained results show that the TDLAS optical sensor is capable of non-destructive measurements of oxygen concentration in such common samples as flow-pack bags with an accuracy of 0.2 vol.% during one second.

1. Introduction

In the food packaging industry, the use of gases other than air in the process of manufacturing and sealing of food items for distribution to the consumer chain (supermarkets, retail points, etc.) has progressively grown (Lee, Piergiovanni, & Yam, 2008). It follows that the precise measurement and control of the gas composition of packaging headspace represent a requirement in the food and packaging industries. An European Union financed project aimed in the realization of new contactless non-destructive laser gas sensors. The utilized sensing method is the Tunable Diode Laser Absorption Spectroscopy (TDLAS). TDLAS works by evaluating the transmission of wavelength-scanned laser light through an absorbing sample. As this technique addresses narrow gas absorption lines, it can be made mostly immune to broadband features typical of solids and liquids, so a great variety of package materials can be considered compatible with the measurement.

TDLAS is a well-known optical method to measure gas absorption with great sensitivity and accuracy. An extensive review of the technique may be found in (Werle, D'Amato, & Viciani, 2008). Many examples of application of this technique are found in industrial sensing instruments (Linnerud, Kaspersen, & Jæger, 1998)

The device validated in this work, developed by teams from the Institute of Photonics and Nanotechnologies of CNR (Italy) and LPro Srl (Italy), is a laboratory unit with manual sample feed, allowing for maximum flexibility in sample choice in order to test the sensor performance on different products before its intended in-line application.

The design of the instrument was primarily tailored for testing mozzarella bags with a slightly diffusive envelope, but the device provides means to easily adapt to different sample types as well. Latteria Soligo is a partner of the project as an end user and produces the mozzarella bags object of the tests. Geometrical parameters of the package can be adjusted freely, as well as calibration of the output measurement. In this way the best

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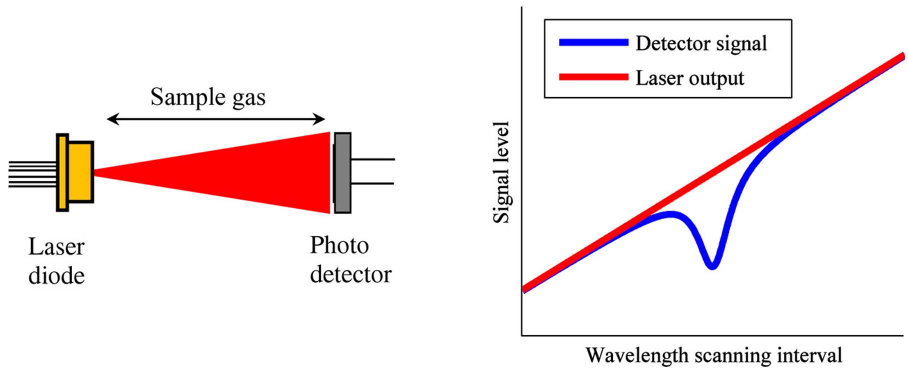

Fig. 1. (Left) Working principle of a Tunable Diode Laser Absorption Spectroscopy setup. Light is propagated through the sample gas (red area) where the package under inspection will be placed. (Right) Arbitrary units representation of laser emission wavelength being linearly scanned over a molecular absorption feature (a Lorentzian line in this case). Laser power output is likely to change during the wavelength scanning process. The ratio between detector signal and laser output (as a function of wavelength) gives the absorption profile.

approach for measurement on a given package type can be obtained.

In the present paper, the performance of this instrument is evaluated and validated on samples of different types made by the purpose with various oxygen content. The effective package filling was also tested with industry-standard, invasive techniques. Performance of the measurement was also evaluated against integration time, to acquire knowledge of the allowable measurement times for the following in-line deployment.

2. Material and methods

Absorption spectroscopy is based on the Beer–Lambert law, which states that the transmission of the light I/I_0 when crossing a sample decays exponentially as

$$\frac{I}{I_0} = e^{-\sigma(\lambda)NL}$$

where $\sigma(\lambda)$ is the line cross section that depends on the wavelength, N is the concentration of absorbing molecules and L is the optical path length inside the sample to be analyzed. The TDLAS principle is shown in Fig. 1. The beam emitted by a tunable diode laser passes through an absorption cell containing the gas of interest. The wavelength of the laser is tuned by a linear modulation of the driving current, in resonance with one absorption line of the molecules of the gas to be probed. The signal measured on the detector as a function of time is proportional to the absorption of the samples as a function of the wavelength. The line cross section $\sigma(\lambda)$ is in general known from the molecular database as e.g. HITRAN (Rothman et al., 2013). If the length L is known then N is derived by the measurement of the absorbed light. In the case of measurement of the oxygen concentration, the only absorption lines are in the 760-nm band.

Resolution and processing of gas absorption lines enables a detection which is intrinsically immune from broadband absorption features such the ones from solids and liquids as well as from absorption lines of other gases such as water vapour and carbon dioxide. A proper choice of the line minimizes the temperature dependence to negligible values.

However, these lines are very weak resulting in a very low absorbance for the normal percentage concentration in MAPs and absorption path length of few centimeters. As an example, for a typical package being at atmospheric pressure with 50 mm path length and 2 vol.% oxygen concentration, the expected absorbance is 1.3×10^{-4} (Cocola, Fedel, Poletto, & Tondello, 2015). For this reason, a variant of TDLAS known as Wavelength Modulation Spectroscopy (WMS) is employed. WMS is a well know technique (Svensson et al., 2008), consisting in superimposing on the linear wavelength scan, a sinusoidal modulation at a suitable higher frequency and synchronously demodulating the received signal. A more detailed description of the WMS method can be found in (Liang & Svanberg, 2015). The absorption information is contained in the higher order harmonics of the modulating frequency. We retrieve it by detecting the second harmonic: by shifting its signal to base band, an absorption artifact can be found with a shape similar to the second derivative of the gas absorption profile. A further feature of this technique is the ability to use the average component of the detector signal at the carrier frequency as a normalization factor to compensate for broadband light attenuation (i.e., due to absorption from packaging material, other non-gas absorption in the content or scattering losses). Average attenuation of laser light in the 760 nm band can vary widely depending on the package material: a factor of 300 was considered typical after a 50 mm path in a sample made of two white diffusive layers and a 5.8×5.8 mm photodiode. An attenuation factor of 10,000 is considered to be the design limit of the instrument.

When the gas to be measured is in a container like the headspace of a food package, which is not perfectly transparent due to some scattering from the envelope, the optical path length L needs some correction from the pure geometrical dimensions. This correction is easily performed through calibration as described below.

2.1. The instrument

The developed instrument is shown in Fig. 2. The laser head contains an Eagleyard single mode laser diode of the Distributed Feed Back (DFB) type enclosed in a thermally controlled. The laser and collimating optics enclosure is maintained oxygen free (with residual oxygen estimated to be well below 0.01 vol.%) thanks to a chemical oxygen scavenger. Operating center wavelength is 761 nm, with a scanning interval of roughly 0.3 nm. The laser is collimated with a lens and the beam of approximately 15 mW of power exits through a wedged window. The transmitted light is detected by a Hamamatsu silicon photodiode of $5.8 \times 5.8\ mm^2$.

The adopted Wavelength Modulation Spectroscopy (WMS) scheme has been implemented entirely in a digital, software-defined approach: all the required wavelength scanning, modulation and temperature control signals are synthesized by the embedded control computer through synchronous analog-to-digital and digital-to-analog boards and a digital lock-in algorithm is used for the demodulation of WMS artifacts.