Sensors and Actuators B 257 (2018) 745–752

Contents lists available at ScienceDirect

Sensors and Actuators B: Chemical

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

Development and validation of a diode laser sensor for gas-phase CO_2 monitoring above champagne and sparkling wines

Anne-Laure Moriaux, Raphaël Vallon, Clara Cilindre, Bertrand Parvitte, Gérard Liger-Belair, Virginie Zeninari*

Equipe Effervescence, Champagne et Applications, Groupe de Spectrométrie Moléculaire et Atmosphérique (GSMA), UMR CNRS 7331, Université de Reims Champagne-Ardenne, B.P. 1039, 51687 Reims Cedex 2, France

ARTICLE INFO

Article history:
Received 16 June 2017
Received in revised form 6 October 2017
Accepted 27 October 2017
Available online 4 November 2017

Keywords:
Champagne
Sparkling wines
Effervescence
CO_2 sensor
Diode laser spectrometry

ABSTRACT

Champagne and sparkling wines are multicomponent hydroalcoholic systems supersaturated with dissolved carbon dioxide (CO_2). Under standard tasting conditions, CO_2 progressively invades the headspace above glasses, thus progressively modifying the chemical headspace perceived by the consumer. Monitoring in situ, and as accurately as possible the level of gas-phase CO_2 above liquid is therefore a challenge of importance aimed at better understanding the close relationship between the release of gas-phase CO_2 and a collection of various parameters such as glass-shape, and champagne temperature, for example. The development and validation of an instrument which combines two infrared diode lasers coupled with an optical fiber and devoted to real-time monitoring of gas-phase CO_2 above sparkling beverages are reported. A first set of data showing the impact of liquid phase temperature on the release of gas-phase CO_2 found in the headspace of champagne glasses is presented.

1. Introduction

Despite the huge body of research (initiated by Louis Pasteur in the 19th century) aimed at progressively unlocking wine science in general, only quite recently much interest was devoted to depict each and every parameter involved during champagne and sparkling wines tasting. Charters noted that drinkers little involved in wine appreciation find it even more difficult to evaluate a sparkling wine than a flat wine [1].

From a strictly chemical point of view, Champagne wines are multicomponent hydroalcoholic systems with a density close to unity, a surface tension $\gamma \approx 50\,mN{\cdot}m^{-1}$, and a viscosity about 50% larger than that of pure water mainly due to the presence of 12–13% v/v ethanol [2–4]. Champagne wines mainly differ from flat wines as they undergo a second fermentation process (called *prise de mousse*). Actually, during this second fermentation process which occurs in cool cellars, the bottles are sealed, so that gaseous CO_2 cannot escape and progressively gets dissolved into the wine (an application of Henry's law which states that the concentration of a given gas specie dissolved into a liquid phase is proportional to its partial pressure in the vapour phase). A standard 75 centilitres champagne bottle typically holds about 9 g of dissolved CO_2, which corresponds to a volume close to 5 L of gaseous CO_2 (under standard conditions for temperature and pressure). For a recent overview of how Champagne wines are elaborated in minute details, see the recent article by Liger-Belair [5], and references therein.

In sparkling beverages in general, and in Champagne wines in particular, the level of dissolved CO_2 found in the liquid phase is indeed a parameter of huge importance since it is responsible for the very much sought-after effervescence through non-classical heterogeneous nucleation of CO_2 bubbles [5]. Under standard tasting conditions, bubbling therefore forces gas-phase CO_2 to progressively invades the headspace above glasses, thus progressively modifying the chemical headspace perceived by the consumer. Actually, it is worth noting that dissolved and gaseous CO_2 acts on both trigeminal receptors [6–10], and gustatory receptors, via the conversion of dissolved CO_2 to carbonic acid [11,12]. Otherwise, a link has been evidenced between carbonation and the release of some aroma compounds in carbonated waters [13,14]. Following these highlights, better understanding the complex mechanisms at play during champagne tasting progressively became a challenge for sommeliers, champagne elaborators, and glass makers.

Abbreviations: TDLAS, tunable diode laser absorption spectroscopy; FP, Fabry–Pérot; FSR, free spectral range; L1, Laser # 1; L2, Laser # 2; D1, Detector # 1; D2, Detector # 2; TDMS, Technical Data Management Streaming; VOC, volatile organic compound; FPGA, field-programmable gate array; DDS, direct digital synthesizer.

* Corresponding author.
E-mail address: virginie.zeninari@univ-reims.fr (V. Zeninari).

https://doi.org/10.1016/j.snb.2017.10.165
0925-4005/

Table 1
Technical characteristics of the two lasers.

Parameters	Laser #1 (L1) @ 2.004 μm	Laser #2 (L2) @ 2.682 μm
Supplier	Eblana	Nanoplus
Mount	Thermoelectric cooler plate	Integrated thermoelectric cooler TO8
Forward current (mA)	30	50
Optical output power (mW)	3 @25 °C	2 @20 °C
Spectral range (cm^{-1})	1 @25 °C and I: 0–110 mA	1 @20 °C and I: 0–87 mA
Single mode spectral range	4989–4995 cm^{-1}	3726–3733 cm^{-1}

In recent years, glass-shape was found to play a key role concerning the rate at which dissolved CO_2 escapes from champagne, under standard tasting conditions. The role of both temperature and bubbling intensity were also closely examined, and found to be key parameters as concerns the progressive losses of dissolved CO_2 from champagne glasses [15–17]. From the sensory analysis point of view, several studies about the crucial influence of the vessel on the perception of alcoholic or non-alcoholic beverages were also reported [18–22]. Recently also, the release of gas-phase CO_2 above beer glasses was monitored by a CO_2 sensor based on the solid electrolyte cell principle [23]. The volume fraction of gas-phase CO_2 measured by this instrument ranged between 4×10^{-4} and 10^{-2}, which is nevertheless much lower than the volume fractions of gaseous CO_2 typically found in the headspace of glasses poured with Champagne wines [16,24]. Monitoring as accurately as possible the level of gas-phase CO_2 above champagne glasses is therefore a key step aimed at better understanding of the strong interplay between the release of gas-phase CO_2 and a collection of various parameters such as glass-shape, and champagne temperature, for example.

Actually, laser diode spectrometry proved to be an effective tool to provide accurate gaseous CO_2 concentration measurements, by infrared absorption spectroscopy. In particular, a new generation of laser diodes emitting at room-temperature in the whole infrared appears promising for the future development of compact CO_2 sensors, especially well-adapted for atmospheric monitoring [25–27]. Thus, compact laser sensors that use new generation diode lasers emitting at 2.68 μm at room temperature, have been developed for the fine detection of gas-phase CO_2 [28,29]. The spectral emission properties of laser diodes make it particularly well suited for gas monitoring by infrared absorption spectroscopy, usually called tunable diode laser absorption spectroscopy (TDLAS) [28]. In this paper, the development and validation of a spectrometer which combines two infrared lasers coupled with an optical fiber and devoted to real-time monitoring of gas-phase CO_2 above carbonated beverages were reported. A first set of data showing the impact of temperature on the release of gas-phase CO_2 found in the headspace of champagne glasses is presented.

2. Instrument design

The instrument is based on TDLAS and has been designed and improved to perform measurements through the headspace of glass poured with a carbonated beverage and not above the edge of the glass (contrary to the previous setup, [30]). In this way, the level of gas-phase CO_2 above the liquid is measured in real tasting conditions. Furthermore, measurements are less sensitive to air movement in the laboratory than with the previous setup [30], and the absorption path-length is fixed by the dimension of the glass and accurately known. In order to improve the measurement accuracy of CO_2 concentration, two different lasers (operating at 2.004 μm and 2.682 μm) were sequentially used in this work. The previous setup [30] used a commercial laser diode emitting at 2.68 μm and allowed the measurement of CO_2 concentrations below 15%. As described by Cilindre et al. (2010) [31] and Liger-Belair et al. (2012) [32], much higher concentrations of CO_2 could be retrieved, through micro-gas chromatography, in the headspace of champagne glasses during the first minutes after the end of pouring. Therefore, a second laser emitting at 2.004 μm, monitoring weaker CO_2 absorption line, was added to our instrument in order to extend the CO_2 concentrations coverage range to higher values up to 100% for typical 10 cm optical path length probing. Indeed, both lasers emit respectively at 2.004 μm (Laser 1) and 2.682 μm (Laser 2) where carbon dioxide has strong vibrational–rotational transitions well suited for TDLAS with an optical path shorter than 10 cm.

The optimized setup, presented in Fig. 1, is composed of two parts. The first part consists in the laser sources and optical elements for collimation and control. The optical elements are mounted on a $46 \times 60\ cm^2$ board. Both lasers are monomode, tunable with precise selection of target wavelength, emitting at room temperature in continuous wave. The technical characteristics of the two lasers are given in Table 1. Both lasers are controlled by Wavelength Electronics LDTC 0520 (current and temperature controllers) and their emitted wavelength are swept over the selected molecular transitions using a sawtooth signal. The laser beam of L1 is collimated by an off-axis parabolic mirror (1″ diameter, 2″ focal length) and that of L2 by a tilted CaF_2 lens of 10 mm focal length, to avoid laser optical feedback which was more important with L1 than L2, this is why an off-axis parabolic mirror is used. The laser beams are then combined by a pellicle beam splitter (45/55, 3.0–5.0 μm wavelength range). The pellicle beam splitter used does not cause deflection of the laser beam, allowing the construction of a single path for the two lasers. The transmitted beam of L1 and the reflected beam of L2 are sent through a one-inch germanium Fabry–Perot etalon (Laser Components, model L5940, length about 2.54 ± 0.05 cm) and are detected with an InAs photodiode cooled by a double stage Peltier (D1 in Fig. 1, Judson model J12TE2-66D-R01M). The reflected beam of L1 and the transmitted beam of L2 are sent through the FC APC/PC fluorozirconium singlemode optical fiber (19 μm of core diameter), using a fiber coupler, to the glass holder. Both lasers are switched on during all the experiment in order to minimize thermal drift of the emitted wavelength. Pneumatic shutters allow to selectively obturate one of the beams. The optical elements are enclosed in a custom made PMMA box filled with gaseous nitrogen in order to limit the absorption by ambient CO_2. The ambient conditions, temperature and humidity are measured by two sensors, respectively TMP36-GZ (accuracy ± 1 °C) and HIH-4000-003 (accuracy ± 3.5%) and the data are recorded using a NI-USB-6009 device, from National Instruments.

The second part of the instrument is located outside the box. It consists in the fiber output coupler, a CaF_2 lens (10 mm focal length), a wine glass and an InAs photodiode (D2 on Fig. 1, Judson model J12TE2-66D-R01M). We observed a little deviation of the laser beam due to the glass wall but, no Fabry-Pérot effect. Moreover, a 10 mm focal lens was added in front of the photodiode which reduced its field of view only to the direct path of the laser beam. The optical path length is limited to the glass diameter, thus the measurement is made in the glass headspace only and the optical