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CHROMATIC DISPERSION MEASUREMENT OF SYNTHETIC MODELS OF BEVERAGES USING LONG PITCH GRATINGS IN AN OPTICAL FIBRE

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RESUME

The chromatic dispersion of model drinkable liquids was studied using long pitch fibre gratings (LPG). The wavelength change of the LPG bands versus species and concentration of some chemical compounds found in commercial beverages show that the analytes have distinct chromatic signatures, and accessible limits of detection.

MOTS-CLEFS : *Optical fibre ; long pitch grating ; chromatic dispersion ; chemical analytes.*

1. INTRODUCTION

Food and drink industry necessitate strict control of manufacture process. Particularly, aromatic and aliphatic quantification is a challenge in beverage industry. Usually, random sampling is performed and the chemical analysis is done out of the production chain. On-line monitoring or fast checking in samples would help manufacturing. Development of optical technologies for continuously measuring in the bulk and online is necessary but is a major challenge. In particular, measuring of the chromatic dispersion of refractive index in liquids may provide very interesting insight in the progress of the manufacture (in line process) or to evaluate samples (spot measurement). Refractive index measurements are usually obtained using a refractometer; in-line process refractometers are commercially available. However, these typically operate only at a specific wavelength [1], and do not produce a dispersion relationship. To measure the chromatic dispersion of liquids, researchers use a rainbow or wedge-cell technique [2,3] or a liquid-prism surface plasmon resonance sensor [4] or the more conventional method of ellipsometry [5]. However, all these require free space optics, and are difficult to align and are costly to maintain.

Optical fibre based sensors are mainly used on physical measurands, e.g. strain and temperature. Fibre long period gratings (LPGs) have been routinely used to measure the changes in refractive index of various liquids in which they are immersed, using a single attenuation band with various configurations. Over the last couple of decades, many implementation of this technology have been demonstrated, from simple uniform LPG to specifically coated LPGs to yield index changes [6,7]. In addition, there are other configurations of LPG that utilise the dispersive properties of the optical fibre itself to produce dual-peak attenuation band resonance. This occurs where the phase-matching of the cladding mode to the core mode is at a dispersion turning point of the fibre [8]. These strategies have been employed to measure the refractive index of the surrounding medium at one or over a very small wavelength range, typically across a few nanometres. However, LPGs usually have several attenuation bands over a given spectral range. Each band is associated with the matching of

the core mode to the different cladding modes, the latter interacting with the external medium. Therefore LPGs can be used to estimate the dispersion relationship of the medium into which they are immersed.

In this work, we show for the first time to the best of our knowledge, the first results of the principle of online, simultaneous, refractive index measurement of a complex liquid over up to nine different and independent wavelength channels spanning across the near-infrared region. The aim of this study is to evaluate the sensitivity to usual chemical compounds found in beverages, and estimate their respective limit of detection.

2. EXPERIMENT AND RESULTS

The LPG were manufactured using femtosecond laser direct inscription in a standard optical fibre (type G.652). In order to increase the number of channels within the transmission range 1150-1750 nm, two 2.7 cm-long LPGs were inscribed side by side in the fiber, with periods of 307 μm and 550 μm , respectively. The LPGs have been annealed. The transmitted spectrum from a fiberized tungsten lamp (Ando AQ4305) across the LPGs was collected by an optical spectrum analyser (Yokogawa AQ6370D). The LPG was positioned in a metallic V-groove, of which the temperature was measured in order to correct the transmitted spectra from temperature slow drifts. Up to ten bands, from 1244 to 1642 nm, were detectable. The spectral position of each band was evaluated by its centroid (first momentum). The minimal detectable wavelength shift was 20 pm, limited by the equipment. The transmission of the LPG bands is shown in Fig. 1(a). They were calibrated in function of temperature and also refractive index using calibrated index fluids (Cargille Labs), except for channel #0, due to equipment limitation. Channel #2 had a non-monotonic behaviour and was not used in this study. The sensitivity of each attenuation band to refractive index was extracted (Fig. 1(b)). Note that the bands behave independently from each other with regards of the refractive index.

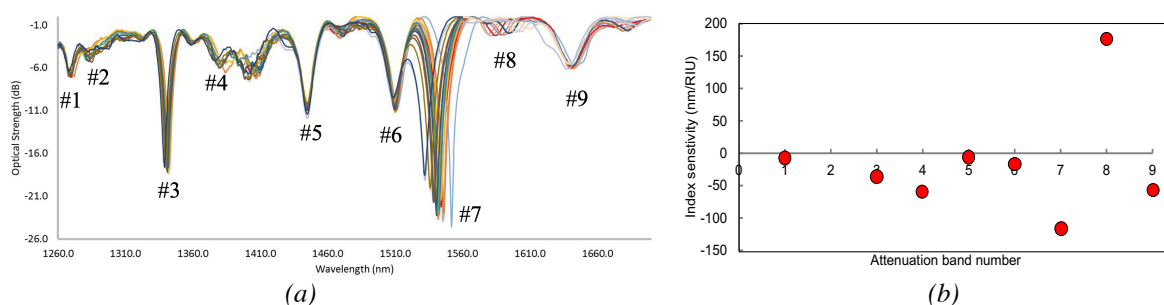
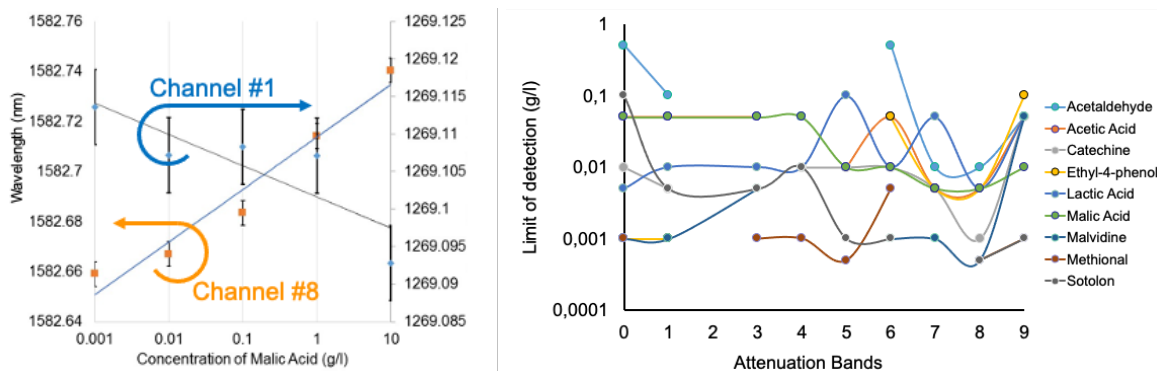


Figure 1 : (a) : Spectral transmission of the double LPG immersed into index liquids. Band #0 at 1244 nm is not shown. (b) : Sensitivity of each independent channel to change in immersion index liquids.

The base of all the synthetic solutions was a mixture of pure water, tartaric acid (5 g/L), ethanol (12%), and was adjusted at pH= 3.5 with NaOH. A set of chemical compounds have been selected because they may be found in several beverages. The compounds include malic acid, lactic acid, acetic acid, ethyl-4 phenol acid, methional, sotolon, acetaldehyde, catechin and malvidin. The span of concentrations for each compounds series was typically 0.001-100 g/L (e.g. malic acid) down to 10^{-5} -1 g/L (e.g. malvidin). A 0.2-ml drop of a solution was set on the LPG, and the wavelength shift of each LPG band was recorded vs. the compound concentration. Between two measurements, the LPG was rinsed in ethanol until all bands had recovered their initial position, both in ethanol and in air. The LOD was limited by the 20-pm wavelength resolution. An example of sensitivity of bands #1 and #8 to malvidin is given in figure 2(a). It is seen that some attenuation bands, for example channel #1 for malvidin, have a linear sensitivity to concentration (on a log scale), over five orders of magnitude. Also, each channel sensitivity is, by principle, demonstrated as independent from each other. For example channels #1 and #8 for malvidin show opposite wavelength shifts. Thereby, each chemical may be in principle identified by its chromatic dispersion curve, in other words, by its « chromatic fingerprint ». Fig 2(b) shows the « multi-channel LOD » for all chemicals and all LPG

bands. It strongly depends on the chemical composition and structure of the molecule, as well as the polarizability of the chemical bonds. The lowest (resp. highest) LOD in molar concentration are for methional (<10 $\mu\text{mol/L}$) and acetaldehyde ($\sim 230 \mu\text{mol/L}$), respectively. For five out of eight compounds, the LOD is well below the usual concentrations found in usual beverages such as wine. This will be discussed in more details during the conference.



(a) # 8 vs concentration of malvidin. (b) LOD for all chemical compounds and all attenuation bands.

CONCLUSION

The technology of LPG in standard optical fibres is sensitive to the effects of the chromatic dispersion of dilute chemical compounds in liquids. The sensitive fibre was not sensitized to specific compounds, i.e. label free detection would be possible. Hence, it shows promising application in the chemical survey of liquids, for example during the production of beverages, with a minimum footprint and a great versatility. Although the LPGs used here are not optimized in terms of sensitivity, several compounds have been detected with a LOD as low as 10 $\mu\text{mol/L}$. Lower LOD are, to the best of our knowledge, detectable with chemically-specific sensors. It shows that this simple, not yet optimized, technique to detect chemical compounds in liquids, has potential for the agri-food industry.

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