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# Optical sensor combined with chemometric methods for the spray deposits characterization

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#### ABSTRACT

Pesticide use became one of the main matters in the agriculture field, and that is due to the off-target spray particles causes environmental, health and economic problems. Spraying quality could be optimized by assessing off-target volumes, and thus help farmers to improve their farming performances. This work aimed to develop an optical sensor based on a RIB waveguide design in order to characterize the quantity and distribution of a liquid spray deposit. We assumed that the light guidance properties of these sensors would undergo a modification when spraying droplets are deposited on their surface. This phenomenon results from partial evanescent wave absorption by the water present in the pesticides which leads to a decrease in the transmitted intensity at the waveguide output. Using different droplet volumes (from 0,1 to 2,5 µl), we studied on an optical bench the influence of a range of deposits at the guide center. As expected, the result was a gradual decrease in the output intensity signal due to the water absorption of the evanescent field, and this proportionally to the droplet volume; the bigger the droplet, the greater the loss in output intensity. We tested the waveguide on a spectral analysis bench in order to highlight the light absorption at the specific water wavelength using the output spectrum. The result showed an absorption at 1450µm wavelength wich is one of the water wavelength absorption. These first results demonstrate the potential of RIB waveguide sensors to accurately quantify droplet deposits. Our future work should focus on improving the waveguide architecture in order to estimate deposited volumes and also the number of deposited droplets.

#### **INTRODUCTION**

As part of the French government's EcoPhyto plan to reduce and improve plant protection product use, Many research aims to find an alternative products or developing decision support tools to reduce doses of applied pesticides. Thus, spraying quality improvement can be a solution implemented in order to optimize the phytosanitary use on field. As a way of estimating the spraying quality, spray deposition characterization could deliver an indication of the locally received dose by the various organs of a treated area. Thereby it provides valuable information for input use optimization. Therefore, farmers and agricultural consultants highly need innovative, quick and accurate decision support tools to measure spray deposition in field condition. Many methods to quantify spray deposition have been developed [1-4]. One of the most common methods is based on the use of Water Sensitive Paper (WSP) cards. However, these methods are costly and time consuming. Automation of spray deposition measurement would allow the acquisition of more indications on the spray deposition. These data could be used by sprayer manufacturers to optimize their machines, and by agricultural consultants to define an optimal plant treatment dosage. This could lead to a significant plant protection products use reduction. Several works that aim to respond to this demand already exist [5,6]. Recently an electronic, resistance-based sensor array and data acquisition system was developed to measure spray deposition from hydraulic nozzles [7-8].

However, it was not enough sensitive to fine droplets. In these days, Optical sensor platforms based on integrated waveguides are well present in the actual research area. The need of a quick, automated and precise devices applied in a biomedical, healthcare, pharmaceuticals, environmental monitoring [9,10] led to a development of these measurement tools. Nonetheless, these tools have never been used to measure a spray deposition. The detection process is based on the interaction between the liquid deposited on the surface of a waveguide and the evanescent wave that escapes from it. So we expected that the light injected will undergo an intensity variation due to the water absorption [11].

In this work, we investigated on the feasibility of an optical sensor to estimate the quantity, distribution and composition of a spray deposit by analyzing the spectra of the injected light in the RIB waveguide at the output before and after a spray deposition. To do so, rib waveguides, composed of Ge25Se65Te10 and Ge25Se55Te20, and an operating range from 1 to  $17\mu m$ , were designed and manufactured. The spectra that we obtained were analyzed by using chemotric methods as we aimed to detect and predict the droplet volume deposition and the liquid composition.

#### MATERIALS AND METHODS

#### Waveguide manufacture & design

Ge25Se65Te10 and Ge25Se55Te20 films were deposited by thermal coevaporation using a PLASSYS MEB 500 device. The three sources were placed in a configuration that allows the deposition of films with uniform composition and thickness over a surface of about 4 cm in diameter [12,14]. The deposition rates and thicknesses of the three elements were automatically controlled with precalibrated quartz crystal monitors. When the elemental deposition rates were stable, the film deposition was initiated. The deposition was then stopped when the aimed film thickness was reached. The guiding structures were elaborated by stacking the Ge25Se65Te10 and Ge25Se55Te20 films successively onto a silicon wafer. The geometry of the upper film constituting the core layer was then modified by ion beam etching, after preparation of the surface by a photolithographic step. For photolithography, a mask comprising bands of different widths (between 4 and 42  $\mu$ m) and the positive photo-resist AZ4533 from MicroChemicals were used. Subsequent ion beam etching was performed using a set-up provided by PLASSYS. A Ge25Se65Te10 cladding layer and a Ge25Se55Te20 core layer (n= 2.55 and n= 2.65 at 1.55  $\mu$ m wavelength, respectively) [15] were then chosen, which corresponds to  $\Delta n = 0.1$ .

The opto-geometrical parameters, of the waveguides were studied and referred on the following article [11]. A tradeoff between the design parameters had to be found and the optimal design values were estimated; Hclad=6 $\mu$ m, Hc=6 $\mu$ m and h= 4  $\mu$ m [11]. First study showed the impact of a droplet deposit on the light intensity injected in the waveguides and also the correlation between the volume deposited and the light intensity variation, the more the volume is the more light loss we obtain [11].

#### Droplet deposition and optical measurements

The straight waveguides made provided a multi-mode propagation solution with a RIB design using a supercontinuum laser (450 nm  $< \lambda < 2500$  nm). To minimize the coupling losses with optical fibers, we used a lens in such a way that the laser beam injected diameter was comparable to the waveguide one. In order to record the output spectrum, The ARCspectro UV-VIS-NIR spectrometer was used due to its large wavelength range 1000-2500nm. This spectrometer operates a FT-NIR Fourier-Transform scanning in the NIR spectrum range.

The initial spectrum was compared to the output spectrum measured after a droplet deposition to study the waveguide transmission and the influence of the droplet deposition.

Demineralized water was used in all the experiments and different volumes of droplets were tested. A micropipette was used to dispense 0,1 to 2,5  $\mu$ l droplets, in steps of 0,1  $\mu$ l, on the surface of the sensor. Between two droplet depositions, the sample was dried with dry air and we ensured that the transmission of the waveguide was not altered. These tests were performed three times. The droplet sizes range used in this work was quit large compared to the typical size of agricultural spray nozzles. these experiments aimed at checking the sensor sensitivity in controlled conditions.



Fig.1 Spectroscopic Analysis Bench (Absorption spectra)

#### Multivariate data analysis

In chemometrics, Partial Least Squares (PLS) regression is the most common regression method for multivariate data [16]. In spectroscopy, PLS is used in most cases for relating spectral data to measured variables [17]. In this study, PLS model was used as the multivariate linear calibration method for predicting droplet volume.

Before preprocessing, the data acquired in transmittance were transformed in pseudo-absorbance – log(I/I0). The spectral range was selected from 1200nm to 2000nm. This spectral range is optimal for all optical components used. A principal component analysis was performed to evaluate the main source of variance and to remove outliers. The quality of the prediction obtained from a PLS regression model is evaluated according to a block leave-out. For a given volume, all corresponding spectra are removed from the calibration set. In addition, pretreatments were tested to increase the quality of prediction (First Derivative, Detrending, Savitzky–Golay algorithm [17], Standard Normal Variate (SNV) [18], etc.).**RESULTS** 

The best results were obtained with 8 latent variables by combining Savitzky–Golay algorithm with SNV. Figure 2 shows the different absorptions from 1200 to 2000 nm after spectral treatment for each volume deposited. We can observe a strong absorption at 1450nm, matching to one of the wavelengths at which the water absorbs. If we analyze the spectra according to the volume, the hollow is more important when the volume increases, especially between 1300 and 1600nm range.

The determination coefficient  $R^2$  and the variance  $\sigma$  are presented in Figure 3. The results obtained ( $R^2 = 0.784$  and  $\sigma = 0.035$ ) shows a strong link between spectra modification and droplets deposited volumes.

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#### CONCLUSIONS

In this work, first bricks toward an optical micro sensor for pesticide droplet size characterization from agricultural spray nozzles were carried out. Straight RIB waveguides based on GeSeTe films were elaborated and tested on a spectral analysis bench (that we developed). Water droplets, that are the main component of phytosanitary inputs, were deposited onto the waveguides surface in order to investigate their influence on the light propagation. As expected, the water absorbs a part of the guided light via the evanescent field, leading to a decrease in the light intensity at the output of the waveguide wich causes a spectrum shape modification at 1450nm that is one of the water wavelengths absorption. The larger the droplet volume, the larger the effect, which is due to the extension of the interaction surface between water and evanescent field. The sensitivity of chalcogenide RIB waveguides to water droplet deposition has been demonstrated.

Further experiments will then be conducted with aqueous solutions in order to assess on the detection of other chemical components that are present in pesticides liquids using the previous approche. Furthermore, other waveguide architectures will be developed to study the impact on the light propagation for a bi-dimensional surface distribution detection; droplets positions and volumes. These structures could help us to solidify our approach to lead us the closest possible to a sensor that could be tested in field condition. Therefore, the present work opens the door to the development of sensors for the monitoring of spray deposits and also a tool able to analyze the influence of the liquid composition on the output spectrum when a droplet is present on the waveguides.

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