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► **To cite this version:**

Sylvain Treguier, Sébastien Cuq, Valérie Lemetter, Axel Canado, Marielle Pagès, et al.. Vibrational spectroscopy as a tool for fast and on-line measurement of ozone concentration in water inside a reactor. 24th International Ozone Association World Congress, 2019, Nice, France. <hal-04996917>

HAL Id: hal-04996917

<https://ut3-toulouseinp.hal.science/hal-04996917v1>

Submitted on 26 Mar 2025

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Vibrational spectroscopy as a tool for fast and on-line measurement of ozone concentration in water inside a reactor

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Food and water industries are highly sensitive to micro-organism contaminations. Nowadays, chlorine is the main treatment to provide clear water, but some alternative exist, such as UV treatment or ozone dissolved in water [1]. Ozone is well-known for its efficiency as a disinfecting agent. Current methods for evaluating levels of dissolved ozone in water involve a two-step analysis. These methods are indirect, time-consuming and prone to errors related to manipulation.

Spectroscopy in the near infrared (NIR) and UV-visible (UV-Vis) regions combined with chemometric methods are fast, repeatable and easy-to-use tools with many applications on water-based matrices. The work presented here details a method for on-line measurement of concentration of ozone dissolved in water by NIR and UV-Vis spectroscopy [2], [3].

MATERIALS AND METHODS

The reactor specifically designed for the experimentation allows to monitor the water ozonation using two methods (Figure 1). On one hand, a quartz finger is immersed right inside the reactor and allows us to follow the reaction from the inside with the UV-Vis spectrometer probe. On a second hand, a sampling aperture gives the opportunity to collect water and analyze it by a reference method and by a near-infrared spectrometer. Both acquired reflectance spectra.

Two different cases were studied, one consists in bubbling ozone in water up to saturation, then following the degassing process through sampling and spectra acquisition every 5 minutes for 2 hours. The other one consists in following both the ozone addition and the degassing phase, through sampling and spectra acquisition every 3 minutes for 2 hours and half. In total, 75 samples of water were analyzed by the indigo method and both UV-Vis and NIR spectrometers.

Then, partial least squares (PLS) regression models were developed. The performance criterion used to select the best PLS model is the ratio of performance to deviation (RPD). The higher is the RPD, the more accurate is the model. In fact, a RPD > 2 is commonly considered to justify the use of the model for prediction in routine analyses to quantify a component. For RPD between 1.4 and 2, classification models are preferred. All statistical analyses were done by using Matlab 2018.a (Mathworks, Natick, MA, USA) installed with the SAISIR Toolbox [4].

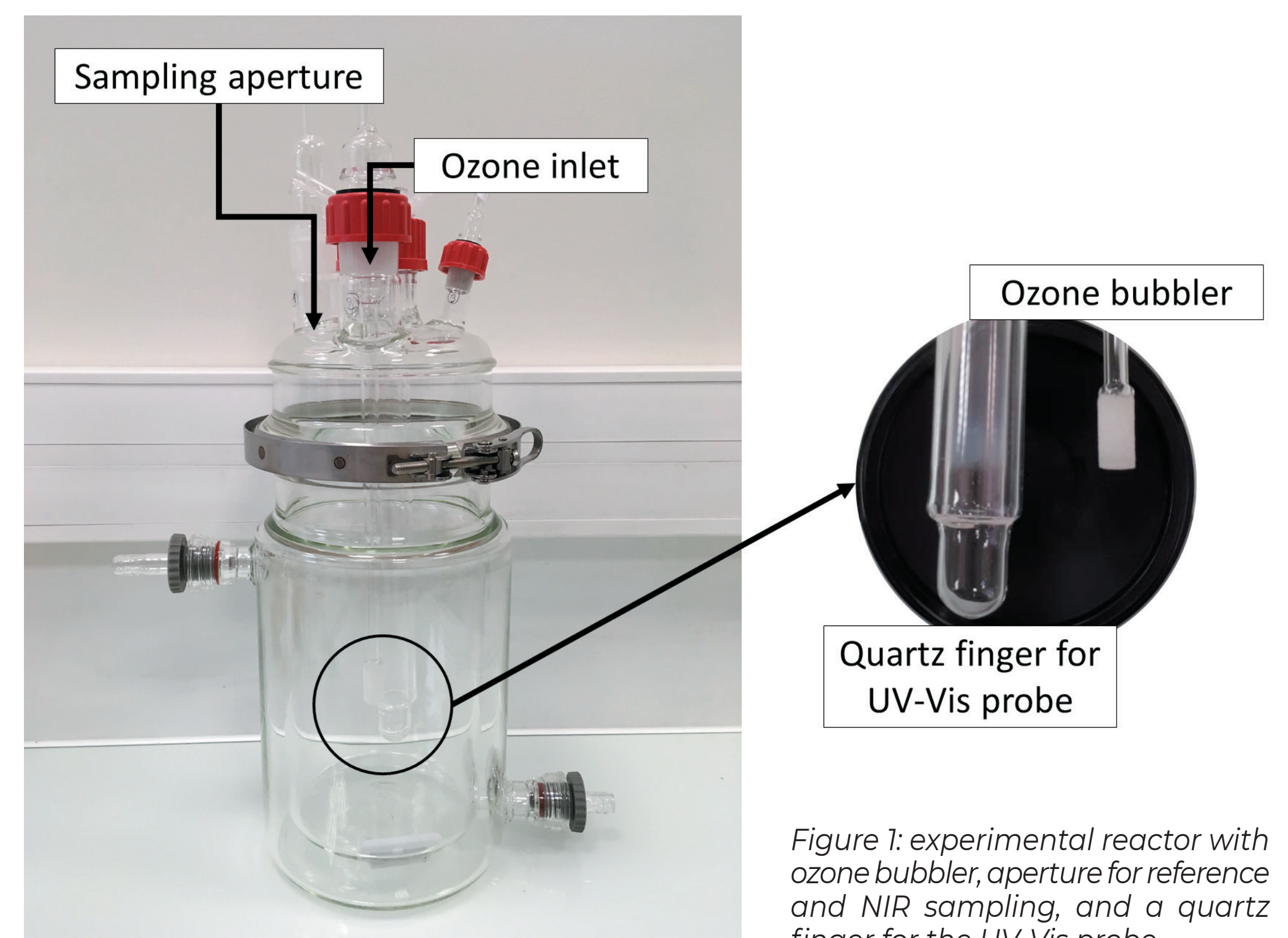


Figure 1: experimental reactor with ozone bubbler, aperture for reference and NIR sampling, and a quartz finger for the UV-Vis probe.

Results obtained with NIR spectrometer

Pre-processing	r ² Pred	RMSEP	RPD Pred
Raw	0.82	1.93	2.28
MSC	0.80	2.26	1.94
SNV	0.80	2.11	2.08
D1	0.82	1.98	2.21
D2	0.71	2.66	1.65

Table 1: Performance of each model stemming from the NIR spectrometer, depending on the pre-process method. Raw (no mathematical pre-treatment), multiple scatter correction (MSC), standard normal variate (SNV), first order derivative (D1), second order derivative (D2).

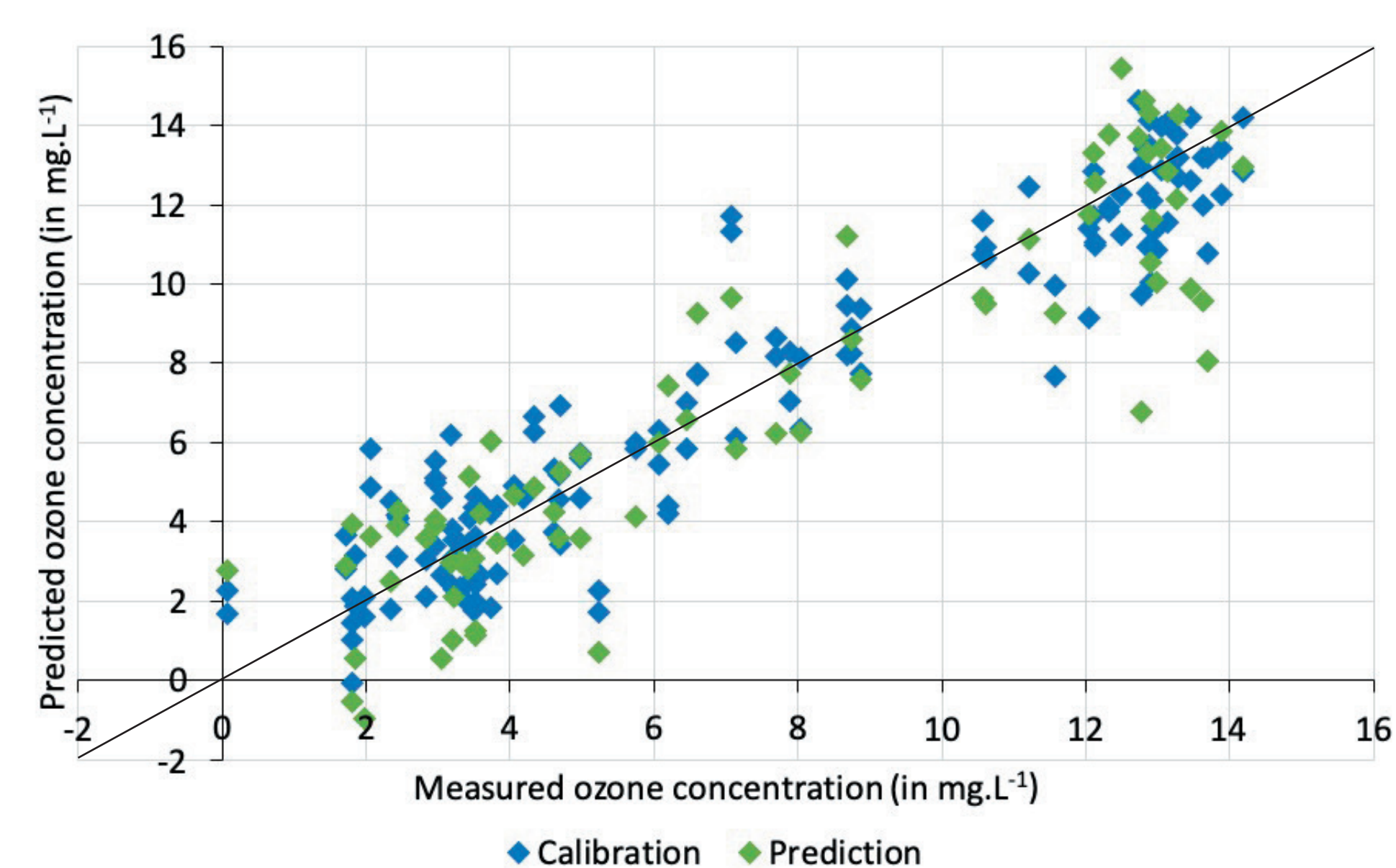


Figure 2: Scatter plot of measured vs. predicted ozone concentration, obtained with the model built on raw data stemming from the NIR spectrometer.

Results obtained with UV-Vis spectrometer

Pre-processing	r ² Pred	RMSEP	RPD Pred
Smoothing and Baseline	0.92	8.96	0.22
Smoothing, Baseline and MSC	0.82	0.89	2.27
Smoothing, Baseline and SNV	0.83	0.91	2.20
Smoothing, Baseline and D1	0.88	8.72	0.23
Smoothing, Baseline and D2	0.86	11.23	0.18

Table 2: Performance of each model stemming from the UV-Vis spectrometer, depending on the pre-process method. Smoothing, baseline for linear baseline correction, multiple scatter correction (MSC), standard normal variate (SNV), first order derivative (D1), second order derivative (D2).

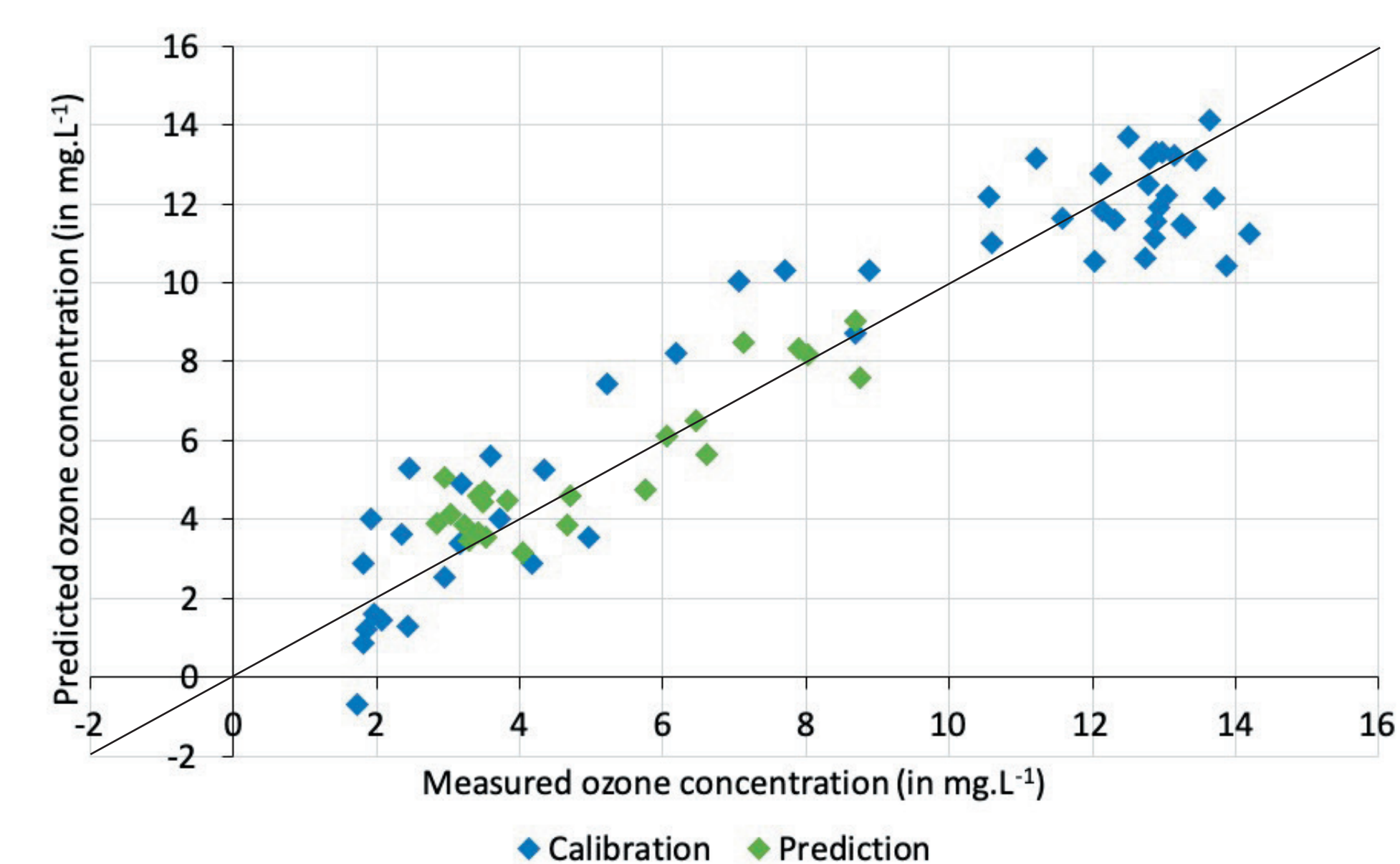


Figure 3: Scatter plot of measured vs. predicted ozone concentration, obtained with the model built on smoothed, baseline-corrected and MSC pre-treated data stemming from the UV-Vis spectrometer.

CONCLUSION

Those different results show the capacity of NIR and UV-Vis spectroscopy to monitor on-line the concentration of ozone dissolved in water. Even if performances with UV-Vis are better, NIR spectroscopy shows interesting results. In order to improve the performance of each model, more assays are needed. Also, the two spectrometers are used in two different configurations: the UV-Vis spectrometer is immersed right into the reactor through a quartz finger. This allows a following of the water ozonation from the inside. The NIR spectrometer needs to work with samples, but its size allows to move it easily and follows the ozonation of water at different points of the reaction (if different sampling apertures exist).

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