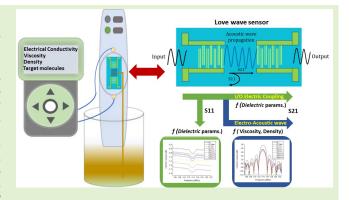


Love-Wave Acoustic Sensors Behavior in Complex Liquids: Multiparameter Sensing Using Acoustic and Electrical Signals

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Abstract—Love-wave (LW) acoustic sensors are promising devices for biochemical detection in liquid media. However, their application for in situ biochemical detection especially in the turbid liquid medium is not yet explored. Turbid liquids are complex in nature with both mechanical and electrical characteristics. These characteristics could be reliably estimated with LW acoustic sensor response, where some of the dedicated sensors based on electrochemistry or optical principles show limitations. This article presents experimental responses of the LW acoustic sensor to turbid liquids based on formazin solutions and its comparison to the spectrophotometer response. Analysis of the sensor's electro-acoustic response is used to better characterize the sensor performance with turbid liquids. Furthermore, this article demonstrates the feasibility of the LW acoustic sensor



as a multiparameter sensing unit by estimating the influence of electro-mechanical parameters of the turbid liquids on the overall response of the sensor.

Index Terms—Complex liquids, formazine solutions, love waves (LWs), multiparameter probes, multiphysical response, optical transduction, turbid waters.

I. INTRODUCTION

OWADAYS, advanced sensing technologies are crucial to monitor various environmental conditions as they play a critical role in analyzing and understanding the environment.

Environmental sensors are one of the effective solutions to surveil the water bodies and monitor water pollution.

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Especially, in the case of water bodies that are infested with harmful biochemical compounds, it is important to detect target molecules and monitor the quality of water. However, classical methods that involve the collection of in situ samples to be analyzed in the laboratory are limited in terms of realtime monitoring. The quality of test samples collected from the field is a very important factor, as the collected samples may get compromised by inaccurate manual handling during transportation. Moreover, the time delay between the sample collection from the field and the analysis in the lab could alter the biochemical compounds in the samples to be analyzed. Also, in some cases, difficulty in accessibility to the field adds to the challenges of required sample collection. To overcome these challenges in the effective analysis and the detection of target biochemical molecules, in situ test and monitoring techniques are promising as reliable alternatives. Hence, our goal is to develop an acoustic sensor-based multiparameter probe sensing unit for in situ biochemical target molecules detection as well as physical characterization of the sample to be analyzed. Surface acoustic wave (SAW) sensors, especially love-wave (LW) devices for sensing applications in liquid media have already demonstrated their usefulness, as they provide high sensitivity and reliable results for biochemical detection and liquid "mechanical" parameters estimation [1],

[2], [3], [4], [5], [6]. Moreover, as LW sensors are based on acoustic transduction generated by the piezoelectric phenomenon, both electrical and mechanical physics can be used for sensing purposes [7], [8]. Indeed, the knowledge of a large set of environmental physical data during the sample collection can improve the quality of the studies. The LW sensor allows an estimation of the change in input electrical impedance of the interdigitated transducers (IDTs) [7]. This change in electrical impedance is particularly due to the variations in the electrical parameters of the liquid samples contributed by the total dissolved solids (TDSs) and total suspended solids (TSSs) in the turbid solutions [7], [9], [10].

In this article, we explore the feasibility of an LW sensor dedicated to biochemical detection in turbid liquids which are identified as complex liquids [11]. Formazine-based turbid solutions are used as models of complex liquids. Our experiments of LW sensor with formazine-based turbid solutions (cf. [11]) using classical method (cf. [12], [13]) have demonstrated the stable response and no saturation even for highly turbid solutions up to 4000 FTU (formazine turbidity unit), where optical devices can reach a limit. To validate this, a comparative study of the response of an optical sensor (a spectrophotometer) and a commercial multiparameter probe (based on the electrochemistry principle) to the complex liquids of various turbidity is investigated. Lastly, the feasibility of the LW sensor as a multiparameter probe for sensing in turbid liquids based on the piezoelectric principle will be explored by analyzing both the acoustic and electrical response signals of the device. Thus, this article is organized as follows: First, the materials and methods section presents the LW device and the materials used in the study. It also describes the measurement methods for turbid liquids carried out with the LW sensor and the spectrophotometer. Section II presents the data and results obtained from the above-mentioned measurement methods. Section III discusses the comparison between the behavior of LW sensors for multiparameter sensing versus classical sensing based on electrochemistry in turbid liquid. Finally, a possibility to design the LW sensor as a multiparameter probe allowing biochemical detection along with the estimation of physical parameters of the liquid sample is demonstrated. Following this, the conclusion section gives remarks on the experiments in liquid media with short- and long-chamber polydimethylsiloxane (PDMS) chips.

II. MATERIALS AND METHODS A. LW Sensor for Sensing in Liquids

LWs are horizontally polarized waves generated using a piezoelectric substrate with IDTs deposited on it. Due to the shear motion of substrate particles to the direction of wave propagation, they are also known as shear horizontal (SH) waves [2], [12]. A thin guiding layer (of a few micrometers) added onto the surface of the piezoelectric substrate allows trapping of the acoustic energy, close to the medium of detection [12]. The SH polarization allows optimum propagation of the LWs inside the guiding layer. When in contact with liquids, the enhanced density of acoustic energy trapped into the thin guiding layer and localized close to the surface, makes

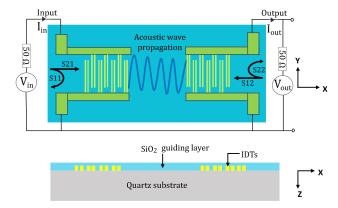


Fig. 1. Principle of open-loop measurement system of LW sensors. VNA allows the S_{21} and S_{11} measurements (top). Two-dimensional cross section of the LW acoustic sensor (bottom).

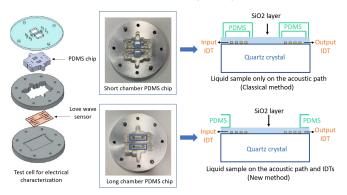


Fig. 2. Experimental test cell (left) and short- and long-chamber microfluidic PDMS chips for liquid sample localization on sensor surface (right) [11].

LW structures very sensitive to the surface phenomena [14]. In this study, the LW device used is a well-known delay line structure that consists of an AT-cut quartz piezoelectric substrate of 500 μ m thickness, with 44 split fingers IDTs (40 μ m wavelength) made of 150-nm gold-titanium layers both at the input and at the output. Finally, a SiO₂ guiding layer of about 4- μ m thickness is deposited on the quartz substrate and the IDTs by plasma-enhanced chemical vapor deposition (PECVD) with localized etching for open electrical contacts. The detailed description of this device is presented in [15] and is shown in Fig. 1.

B. Experimental Setup

For sensing in aqueous solutions, measurements are done using microfluidic PDMS chips with open cavities, namely "short-" and "long-"chamber PDMS chips as shown in Fig. 2 and used in [11]. Liquid samples are localized by micropipette (drop casting method) on the surface of the sensor. The "short-chamber" PDMS chip allows the localization of the liquid sample only on the acoustic path, as for classical methods [13]. The "long-chamber" PDMS chip localizes the liquid sample on the acoustic path as well as on the input and the output IDTs that are covered by the SiO_2 guiding layer. As in [8], the protocol for measurements with turbid liquid samples is as follows: turbid solution samples are localized on the sensor surface using short- (volume 75 μ L) and long- (volume 150 μ L) chamber PDMS chips. As soon as the liquid sample is localized on the sensor surface, the response of the LW

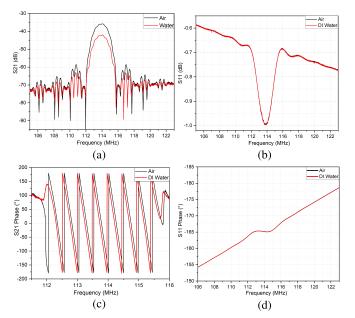


Fig. 3. LW sensor frequency-domain response with short-chamber PDMS for air and DIW. (a) S_{21} magnitude response. (b) S_{11} response. (c) S_{21} phase response. (d) S_{11} phase response.

sensor is recorded with a vector network analyzer (VNA, Copper Mountain C1049). After every liquid test, a complete cleaning step with deionized water (DIW), isopropyl alcohol, and acetone followed by drying with nitrogen spray is realized to prevent the contamination of the sensor surface. Fig. 1 (top) presents typical S_{21} and S_{11} measurements with open-loop readout instrumentation (i.e., VNA in our case). Typically, for this LW sensor, the resonance frequency of each IDT, at which the propagated acoustic energy is optimal, is 114 MHz [8]. The LW sensor experiments were carried out in a clean room for controlled relative humidity (RH) between 30% and 40% and temperature at 21 °C.

C. Measurements With Deionized Water

The sensing and biochemical detection mechanism using an LW sensor with an open-loop readout system (cf. Fig. 1), is mainly based on the measurement of S_{21} response from input to output IDTs. An LW sensor with a specific sensitive layer, when in contact with the liquid sample containing target molecules, changes its response in terms of shift in resonance frequency and the phase due to "mass loading effect" [16]. Based on this mass loading effect and also in terms of insertion losses, indications about the mechanical and physicochemical interactions on the surface of the acoustic path can be noted, which is a marker of biochemical detection. As an example, Fig. 3 shows LW sensor response with short-chamber PDMS chip to the presence of DI water on the acoustic path compared to the response of the LW sensor to air. From Fig. 3(a), it can be observed that, in the presence of the liquid on the acoustic path, the magnitude of the S_{21} response of the LW device decreases, mainly due to the mechanical parameters of the liquid sample, as the effect of electrical parameters is negligible in this case, as explained in [7] and [8]. Also, a phase shift to the lower frequencies occurs due to the decrease of the LW phase velocity as in Fig. 3(c). Because the use of the short chamber does not allow liquid to be on the input and output IDTs, there is no change in the S_{11} response which is similar to the response in air as shown in Fig. 3(b) and (d). To validate the proposed feasibility of LW sensors for multiparameter sensing, in this preliminary work, we focus mainly on the S_{21} and S_{11} magnitude responses of the LW sensor for this demonstration. Indeed, the change in amplitude of the LW propagating along the acoustic path is the key parameter of interest which indicates the availability of the sensor to operate in the complex liquid medium. Typically, we can consider that, with a noise level of around -70 dB in S_{21} magnitude response, a biochemical detection is possible with a minimum of insertion losses of around -55 dB at the resonance frequency with open-loop readout electronic system such as in [8].

D. Measurements With Complex (Turbid) Liquids

1) Liquid Samples Preparation: To investigate the response of the LW sensor in complex liquids, the choice of turbid solution as a "complex" liquid was done, mainly because the liquid media of focus for further in situ biochemical detection are turbid river waters, more specifically of Amazonian region (French Guyana, South American continent). By definition, turbidity is the cloudiness or haziness caused by the large number of suspended biochemical particles in the liquid. Turbidity is an important parameter to be measured, as it can be a sign of poor water quality, especially in the case of drinking water supplies. According to the World Health Organization (WHO), the turbidity level in drinking water supplies should not exceed 1 NTU (nephelometric turbidity unit) [17] where 1 NTU = 1 FTU. In the case of Amazonian water bodies, the classical turbidity level is 200 FTU or more [18] depending on the seasons due to an increase in the suspended dissolved organic, and inorganic matter. Hence, turbidity measurement is a key test of water quality in Amazonian waters. For preliminary experiments, the "formazine"-based solution is chosen as turbid liquid, as it is fairly easy to prepare it in various concentrations in the laboratory. So, according to the protocol given in [19], "formazine"-based "mother solution" is prepared by mixing 5 g of hydrazine sulfate in 50 mL of DI water and 50 g of hexamethylene tetramine in 50 mL of DI water. By diluting this mother solution in different proportions, various concentrations of turbid solutions called "daughter solutions" were prepared as shown in Fig. 4.

2) Optical Characterization of Turbid Liquid Samples: To compare the acoustic sensing with the LW sensor to the optical sensing, measurements of turbid samples were done using "ONDA Visible Spectrophotometer V-10 Plus." Calibration with a standard sample (DI water) was done as mentioned in the user manual of the spectrophotometer. Measurements of transmission and absorption across the cuvette containing the liquid sample were done at three selected wavelengths, viz. 450 nm (as recommended in [19]), 880, and 940 nm as infrared (IR) wavelengths would allow measurements that fall outside the visible range. For every measurement, each turbid liquid sample is vigorously shaken just before introducing it in a 3.5-mL transparent cuvette. The light transmitted through the cuvette is then received by the photodetector positioned behind the cuvette placed at right angles to the direction of

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OPTICAL MEASUREMENTS OF TURBID LIQUID SAMPLES WITH SPECTROPHOTOMETER AT THREE DIFFERENT WAVELENGTHS: TRANSMISSIO					
(TRANS) AND ABSORBANCE (ABS)					
	Samples	450 nm	880 nm	940 nm]

TARLE I

Samples	450 nm		880 nm		940 nm	
(FTU)	Trans (%)	Abs (dB)	Trans (%)	Abs (dB)	Trans (%)	Abs (dB)
2000	0.1	2.974	0.4	2.421	0.5	2.326
1000	0.4	2.410	1.6	1.800	1.8	1.745
400	3.9	1.411	13.3	0.875	14.8	0.830
280	7.2	1.147	20.2	0.692	22.15	0.655
200	16.0	0.790	33.7	0.474	35.6	0.441
94	48.2	0.318	65.5	0.184	68.2	0.167
47	67.1	0.174	78.7	0.105	79.7	0.099
7	83	0.08	90	0.046	90.5	0.044

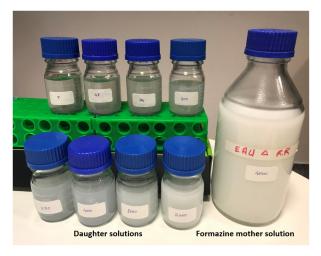


Fig. 4. Formazine-based turbid liquid samples concentration in FTU [19].

travel of light. The time delay between the introduction of the sample in the cuvette and the measurement is less than a minute. Thus, the amount of light transmitted, which is directly linked to the turbidity, is measured in "optical transmission" (in %) as the ratio of the received optical signal and the emitted optical energy. Also, the absorbance $(-\log_{10}[\text{transmission}])$ which represents the amount of optical energy absorbed by each liquid sample is measured as given in Table I. In the case of high turbidity liquids, which indicate high amounts of suspended organic matter, higher absorption of optical energy and lesser and lesser transmitted optical signal through the liquid sample is observed. Due to this, one of the principal drawbacks of the spectrophotometer technique for testing real samples, that is, for higher turbidity liquids, is that the output signal received by the photodetector is significantly lower and insufficient for further analysis as shown in Table I.

3) Measurements of EC and TDSs of Liquid Samples: Hanna HI 98129 electrical conductivity (EC) tester was used to measure the EC, as well as the TDSs of each turbid liquid sample. The measured values are presented in Table II.

Measurements of EC and TDSs are done by inserting a Hanna tester probe directly in a beaker containing the turbid liquid samples each of volume 100 mL. The calibration procedure was done as mentioned in the user manual of the HI 98 129 tester. After that, measurements of each sample are done at 20 °C. It can be observed from Table II that the EC of the samples increases with the increase in the

TABLE II
EC AND TDS MEASUREMENTS OF TURBID LIQUID SAMPLES WITH
HANNA (HI 98 129) TESTER

Liquid sample (FTU)	EC (µS/cm)	TDS (ppm)
2000	3999 (max limit)	2000 (max limit)
1000	3684	1877
400	1888	965
280	1198	675
200	914	459
94	368	188
47	253	129
7	117	59
DIW	0	0

value of turbidity. It is interesting to note that the Hanna tester is based on the classical impedimetric measurement and shows a saturation of 3999 μ S/cm for a liquid sample between 1000 and 2000 FTU and more.

III. LW SENSOR RESPONSE TO TURBID LIQUIDS A. LW Sensor Response With Short-Chamber PDMS

LW sensor experiments with short-chamber PDMS chips are the most conventional method used by the acoustic sensor community for sensing liquids. Fig. 5 presents the LW sensor's time-gated S_{21} and S_{11} responses to turbid liquids with this classical method. The postprocessing based on time-gating filtering of the time domain S_{21} measurements allows extraction of the pure fundamental LW acoustic propagation, by removing any electromagnetic or reflected acoustic signals. This improves an accurate analysis of the insertion losses in the S_{21} response back in the frequency domain [8].

It can be observed in Fig. 5(a) that the decrease of ≈ -1 dB from DIW up to 4000 FTU in S_{21} at the resonance frequency of 114 MHz, which is reasonable to achieve a biochemical detection of target molecules as compared to previous works [20]. In Fig. 5(b), S_{11} response shows nonsignificant decrease of ≈ -0.03 dB at 114 MHz. Hence, with a classical method based on mechanical effect, especially the mass loading effect [16], biochemical detection could be achieved even in highly turbid liquids.

Another interesting point is that, for turbidity close to 200 FTU (typical turbidity value in Amazonian waters), around 55% of signal information is still available compared to the air (no liquid) response of the LW sensor. By comparison, the spectrophotometer response shows only 16% of the transmitted optical signal at the wavelength of 450 nm (84% loss of optical energy), the result is better at higher wavelengths

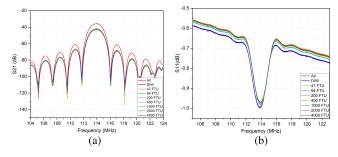


Fig. 5. LW sensor frequency-domain response for formazine solutions with a short-chamber protocol. (a) Time-gated S_{21} response. (b) S_{11} response.

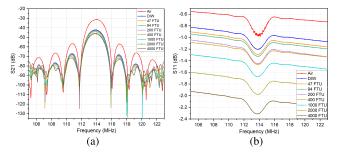


Fig. 6. LW sensor frequency-domain response to turbid liquids with the long-chamber protocol. (a) Time-gated S_{21} response. (b) S_{11} response.

for 200 FTU sample, but poor again when reaching 400 FTU, with less than 15% of the transmitted optical signal at 880 and 940 nm.

B. LW Sensor Response With Long-Chamber PDMS

LW sensor experiments with the "long-chamber" PDMS chip provide an enriched response of the sensor, compared to the conventional method with the short chamber as demonstrated in [7] and [8]. With liquid samples present on the acoustic path as well as on the IDTs, indications about both the electrical and mechanical parameters of the complex liquid samples can be studied. It allows measurements of the electrical impedance of the LW sensor by monitoring the reflected electrical signal (S_{11}) at the input IDT as well as the transmitted signal (S_{21}) from the input to the output IDT, with a particular focus on the acoustic resonance. However, due to the electromagnetic coupling between the input and the output IDTs, time-gating filtering is necessary to precisely estimate the insertion losses from S_{21} response of the sensor in the frequency domain.

Fig. 6 presents the S_{11} and time-gated S_{21} response of the LW sensor to the turbid samples with the long-chamber protocol. Two interesting observations can be pointed out. First, the LW sensor S_{21} response to turbid liquids [cf. Fig. 6(a)] shows that with the measured insertion loss of ≈ -43 dB at 200 FTU (less than -1 dB compared to DIW), a biochemical detection is fairly achievable. Also S_{21} of ≈ -46 dB at 4000 FTU (-4 dB decrease compared to DIW and quite good rejection ratio) still remains usable for biochemical target detection.

Secondly, S_{11} response of the LW device [cf. Fig. 6(b)] clearly shows a decrease in magnitude with an increase in turbidity. As explained in [7] and [8], S_{11} response is highly sensitive to the electrical parameters of the liquid sample on

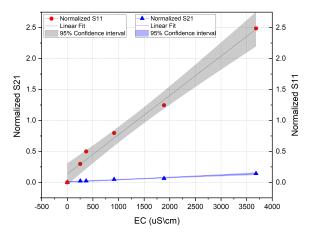


Fig. 7. Relative variation of S_{21} and S_{11} responses for the EC of the turbid solutions.

the input IDT. Hence, these parameters can be investigated from S_{11} response, mainly at frequencies lower than the acoustic resonance (50 MHz in our study) to facilitate the analysis with the least influence of the acoustic phenomena. In Fig. 7, normalized S_{21} and S_{11} responses (w.r.t. DIW, at 50 MHz) for an increase in EC of turbid solutions is shown. This graph highlights that the variation of S_{11} response is greater compared to that of S_{21} response for increase in the EC. This reaffirms that electrical parameters of the turbid liquid influence mainly the S_{11} response of the LW sensor. However, the S_{21} response of the sensor is mainly influenced by the mechanical parameters of the liquid. This validation will be useful for a possible decorrelation between the influence of electrical and mechanical parameters of the liquid on the LW sensor's response.

IV. DISCUSSION: PERSPECTIVES OF THE LW SENSOR AS A MULTIPARAMETER PROBE UNIT IN TURBID LIQUIDS

The results in the previous section can be used further in correlating the S_{11} response of the LW device with the long chamber, to electro-mechanical parameters of the liquid. Specifically, in the perspective of measurements of in situ samples, EC is a very useful parameter. Indeed, Thirumalini and Joseph [21] indicate that EC of a liquid is related to the total dissolved matters and as per [9], the EC of a solution is linearly correlated to the amounts of suspended matter, which is in agreement with the characterization results in Table II and is an indicator of the turbidity [10]. Thus, estimation of the EC of a solution based on the measurements of electrical impedance of IDTs [8], which is directly linked to the reflection scattering parameter S_{11} , can be correlated to the suspended matters and then to the turbidity of this solution, with a range, larger than that of the spectrophotometer (optical measurement) or even the Hannah EC tester. Hence, LW sensors with "long-chamber PDMS" protocol could be designed as a promising multiparameter probe unit allowing biochemical detection based on mass loading effect (by studying S_{21} response), even in highly turbid liquids, along with a physical characterization of liquid medium for in situ measurements (by studying S_{11} response for liquid electrical parameters estimation).

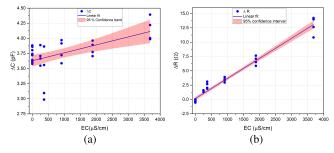


Fig. 8. Influence of EC of turbid samples on the LW sensor's input IDT impedance (from the S_{11} parameter). (a) Capacitance versus EC. (b) Resistance versus EC.

To go a bit further in this multiparameter probe projection, Fig. 8 presents the measurement of the relative capacitance and resistance of LW sensor input IDT, extracted from the long-chamber protocol S_{11} response with the reference taken in air. This method to characterize both resistance and capacitance from S-parameters of an electrical device is well known by the "radio frequency" community and is detailed in [7] and [8]. Again, both resistance and capacitance values are estimated at 50 MHz, lower than the resonance frequency, to avoid the influence of the liquid mechanical parameters on the S_{11} response of the sensor. From Fig. 8(a), we can observe that the relative capacitance of the input IDT does not show a useful response for EC estimation, mainly at low EC. On the contrary, the response in Fig. 8(b) exhibits a linear variation of the resistance with no distinguishable saturation even at high EC. Hence, it could be used to estimate the turbidity of the liquid.

Investigations on the insertion losses, the phase responses, and also on the shift in the LW resonance frequency in the transmission and the reflection losses measured both in time and frequency domains can give more information on the electrical and mechanical parameters of the turbid liquid medium. The influence of the static permittivity of the formazine solutions also needs to be investigated to evaluate its influence on the LW sensor response. Indeed, this large set of data which can become accessible with a single "low-cost" LW transducer needs specific analysis tools to decorrelate each phenomenon and related parameters from the LW sensor's enriched response, which can propose another paradigm in the use of environmental sensors.

V. CONCLUSION

In this article, we demonstrate the feasibility of an LW sensor for biochemical detection in turbid liquids while estimating its EC. This first approach needs to be studied further, to estimate the turbidity of a liquid by monitoring the S_{11} response of an LW device as compared to a classical Nephelometer. Both short- and long-chamber PDMS chips can be used for biochemical detection, depending on a biochemical target and the required precision of sensitivity.

 The short-chamber protocol can be used with a matching circuit strategy to minimize the insertion losses at the resonance frequency. This strategy is also compatible with the use of a large set of classical sensitive coatings [12], [20], which could contribute to more losses but also increase the sensitivity and the selectivity of

- the LW sensor. In that configuration, the S_{11} parameter of the LW device, according to the liquid sample is not accessible to enrich the response of the sensor.
- 2) The long-chamber PDMS protocol intrinsically generates more insertion losses at the resonance frequency due to electromagnetic coupling between input and output IDTs, together with a longer acoustic path interacting with the liquid. Thus, the sensitive coating to be used for biochemical detection, which generates additional insertion losses, may lead to a sensor with reduced signal-to-noise ratio and thus measurement resolution, compared to the use of a short-chamber protocol. Nevertheless, the LW sensor with the long-chamber protocol does allow estimation of the EC of liquid samples even for highly turbid liquids.

Currently, work on the second approach is in progress, which is very promising for the goal of biochemical detection in Amazonian rivers with a low-cost LW multiparameter probe unit.

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