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Multi-Probe Sensor for Water Content Analysis of Liquid Biofuels

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Abstract—A multi-probe sensor for water content analysis, in liquid biofuels, by using reflection and transmission measurements in microwave frequencies range, is proposed in this letter. As preliminary step and for a better understanding, the measurements were carried out with ethanol/water mixtures, which mimic bioethanol applications, at room temperature. In order to study water/alcohol mixtures, each of them was characterized using classical techniques like open ended coaxial probe or reflection/transmission coaxial line, before being tested in multi-probe sensors. At the end, the multi-probe sensor aims to be implemented in-line production in order to perform diagnosis of water in liquid biofuels.

1. INTRODUCTION

Liquid biofuel, such as bioethanol, is a sustainable source of energy that can be used in truck engines, heating systems, *etc.* To optimize the energy efficiency [1] of biofuel combustion and to limit greenhouse gas emissions [2], it is important to know the water content in these biomaterials. Ideally, this measurement should be done in-line, in real time, and continuously, enabling a perfect monitoring of a production line [3]. Within the framework of the European ***BIOFMET***[†] project, based on the study of solid and liquid biofuels in an industrial environment, this microwave method for the in-line determination of the water content of liquid biofuels is therefore developed. This sensor uses several probes located around a metal tube filled with the **M**aterial **U**nder **T**ests (**MUT**). It is possible with this geometry to mimic a production line in which biofuels circulate.

The main objective of this study is to find the threshold of the water content in biofuel like mixtures [4]. The tested materials are mixtures of ethanol and water [5]. With this multi-probe sensor, electromagnetic waves are emitted through the MUT. It is also possible to analyze the reflection on each probe and the transmission between two selected probes. A difference in reflection and transmission responses between the mixtures [6] is due to the dielectric contrast between the permittivities of water and ethanol [7, 8]. Therefore, before testing the multi-probe sensor, it is necessary to verify the existence of a dielectric contrast in these mixtures. A series of dielectric measurements were carried out on different ethanol/water sample mixtures to evaluate this dielectric contrast [9, 10]. This dependence between the permittivity value and the water content justifies the study of our multi-probe sensor.

2. DIELECTRIC PERMITTIVITY MEASUREMENTS OF LIQUID MIXTURES

Dielectric permittivity is an intrinsic value that defines the interaction between electromagnetic waves and a material. Among classical techniques used in the literature [11] to measure dielectric parameter, an open ended coaxial probe (*SPEAG DAK 3.5*[‡]) and a coaxial propagation line (*EpsiMu*[§]coaxial

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† *www.biofmet.eu*
‡ *www.speag.swiss*
§ *www.epsimu.com*

cell) are used in this work. Both cells are used with a **V**ector **N**etwork **A**nalyzer (VNA, ANRITSU MS2038C).

The open-ended coaxial cell is based on a capacitive model [11]. When the probe is in contact with a material, it changes the overall capacitance value of the equivalent circuit. The open ended coaxial probe is set directly inside the liquid to be tested [12]. The permittivity values are calculated from the reflection coefficient measurement (Fig. 1(b)). For the second method, the coaxial propagation line, the sample material under test (MUT) is positioned in a sample holder between two dielectric washers (Fig. 1(a)) [14, 15]. From the S-parameters (reflection and transmission) measurements, the method of *Nicolson and Ross* [13] is applied to calculate the permittivity value.

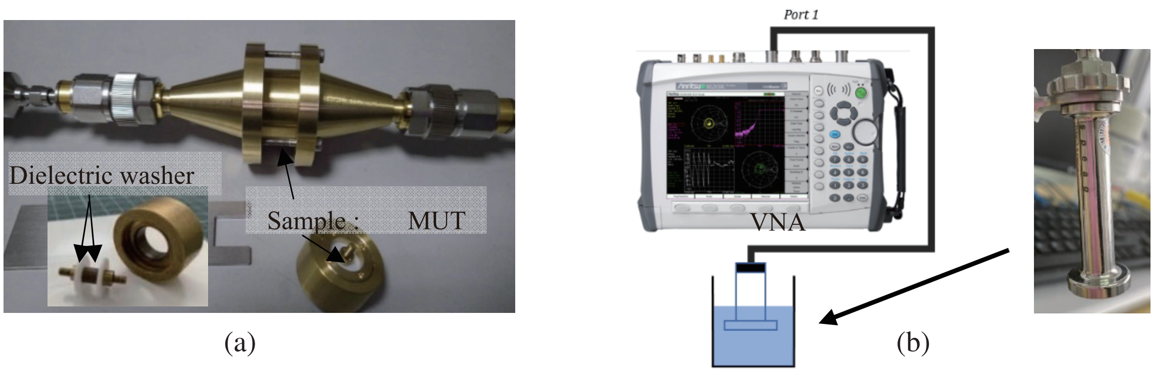

Figure 1. (a) Coaxial reflection/transmission cell (*EpsiMu*), (b) open ended coaxial probe (*SPEAG DAK 3.5)* and schematized measurement set-up, including the VNA.

With these two methods, we characterize some liquids: pure demineralized water, pure ethanol, or any mixtures of these liquids. It is important to be able to use two different technics for ensuring the reliability of our dielectric measurement.

For safety and health reasons, we test our approach with liquids such as demineralised water and ethanol (MERCK, Ethanol 96%, EMSURE® Reag. Ph Eur) instead of using liquid biofuels such as bioethanol. The water content of the different mixtures is between 4% and 20% with steps of 4%. The measurements were carried out at room temperature (25°C) and over a frequency band between 100 MHz and 5000 MHz. The real dielectric permittivity measurements of the different mixtures obtained with the two measurement techniques are shown in Fig. 2.

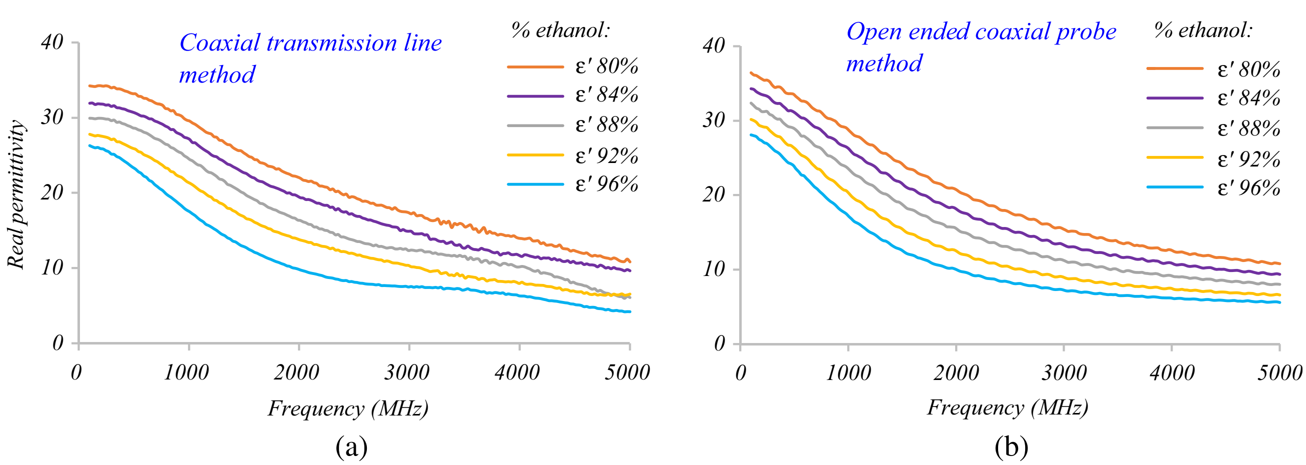

Figure 2. (a) Dielectric measurements with coaxial transmission line method, (b) dielectric measurements with the open-ended coaxial probe method.

Both results confirm the dependence of the real part of the permittivity of the ethanol/water mixture on the water content [16]. Thus, the propagation conditions of the electromagnetic waves in the sensor will be modified as a function of the water content: on one hand for the propagation between two probes and also on the other hand for the adaptation of a probe to the material.

3. MULTIPLE PROBE SENSOR: METHOD AND VALIDATION

3.1. Measurements Setup

The principle of this multi-probe sensor (Fig. 3) is based on a circular metal tube (50 mm diameter) around which several coaxial open ended probes are positioned on its periphery. Thanks to his design, it could be almost directly inserted into a production line.

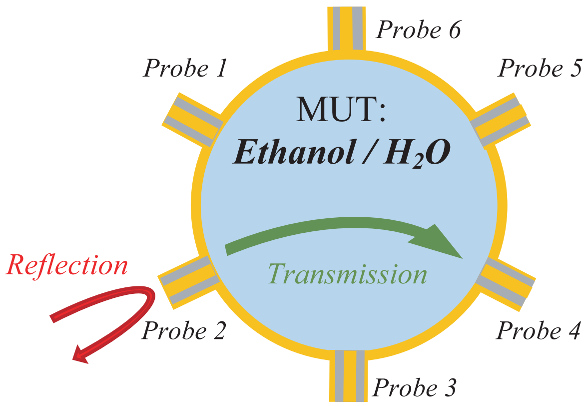

Figure 3. Schematic multi-probe sensor.

In order to enable all probes combination measurements, a switching matrix (*Mini-Circuits Switch Matrix ZTVX 8-12*) and a VNA are coupled to the multi-probe sensor as shown in the figure below (Fig. 4).

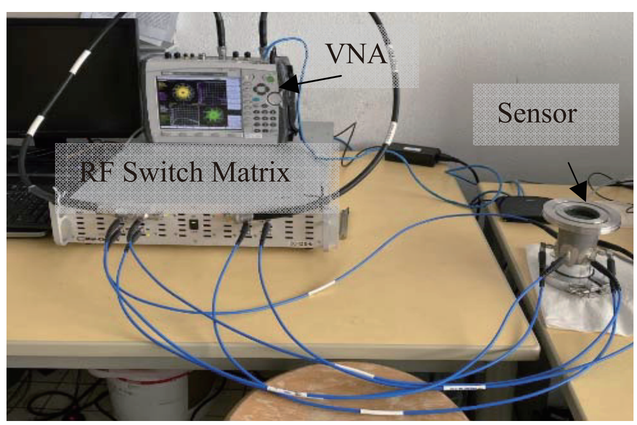

Figure 4. The complete measurements setup with the sensor.

3.2. Samples Preparation and Treatments of Measurements

All ethanol/water mixtures were made from an initial ethanol solution: ethanol 96% (Ethanol 96% EMSURE® Reag. Ph Eur). Thus, all results were obtained with the same mixture for the two technics of dielectric characterization (the coaxial line (Fig. 2(a)) and the open coaxial probe (Fig. 2(b)) and the

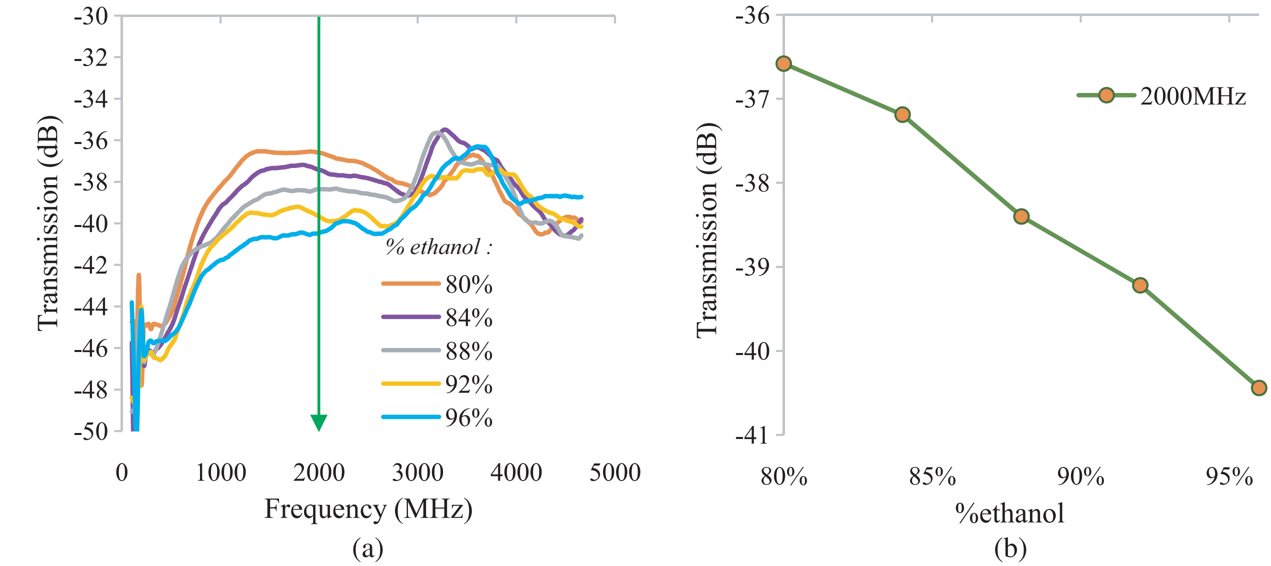

Figure 5. (a) Transmission between two close probes, (b) transmission with ethanol content at 2000 MHz.

multi-probe sensor technic (Figs. 5(a)/(b)). The frequency range is [100 MHz, 5000 MHz]. The water content range is between 4% and 20%.

4. RESULTS AND DISCUSSION

The main objective of this study is to define the water detection threshold with the multi-probe sensor. Five mixtures with water content between 20% and 4% with 4% steps have been tested. In other words, this corresponds to mixtures of 80%, 84%, 88%, 92%, and 96% of ethanol. The variation of transmission between two adjacent probes (Fig. 5(a)) is maximum around 2000 MHz (Fig. 5(b)).

For the reflection parameter (Fig. 6(a)), the variation is significative up 3000 MHz. Thus, for example, the variation of the reflection value versus ethanol content is shown at 4000 MHz (Fig. 6(b)).

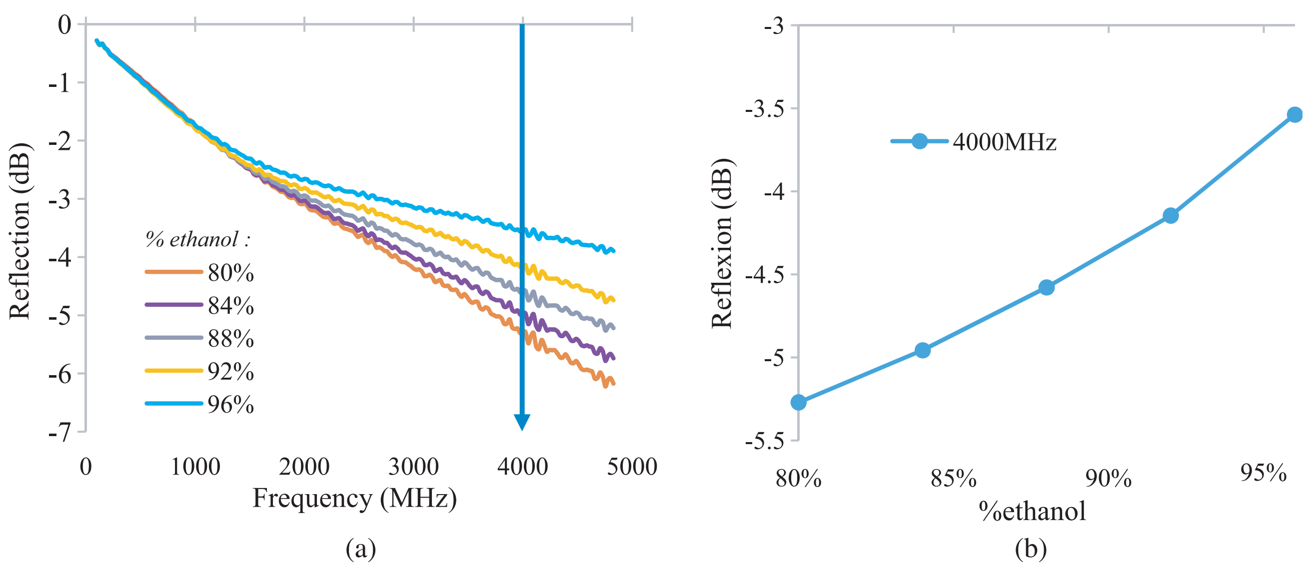

Figure 6. (a) Reflection on the probe number 1, (b) reflection with ethanol content at 4000 GHz.

With this multi-antenna system, in this case of 6 antennas, we have several determinations of the same parameter like reflection. This redundancy increases the accuracy or decreases the error bar, of the measurement of the target parameter: the water content of the biofuel.

In the same context, the transmission measured between two given antennas will increase the accuracy of this measurement, but also provide information about the homogeneity of the material under test.

5. CONCLUSION

In this work, a multi-probe sensor is studied for in-line and real-time determination of water content in homogeneous liquids as alcohol or other. A prototype has been developed and tested. This method is based on the measurement of reflection and transmission coefficients through probes. This electromagnetic characterization is related to the dielectric sensitivity of liquids as a function of water concentration. The first study was carried out with mixtures of water and ethanol in a frequency range between 100 MHz and 5000 MHz. The results obtained with liquid mixtures allowed not only to evaluate the detection thresholds of water content but also to diagnose the homogeneity of the material under test. As further steps, we plan to implement this sensor in a production line to have a means of diagnosis in line and in real time, of presence of water in a liquid biofuel.

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REFERENCES

1. Assaye, M., B. Tamirat, and B. Fekadu, "Ethanol concentration and calorific value of some local distilled Ethiopian alcohol (Areki): An energy potential assessment," *Cogent Eng.*, Vol. 8, No. 1, 1979444, 2021.
2. Rasool, U. and S. Hemalatha, "A review on bioenergy and biofuels: Sources and their production," *Braz. J. Biol. Sci.*, Vol. 3, No. 5, 3–22, 2016.
3. Liu, Y., Y. Deng, and Y. Li, "Experimental investigation of gas-oil two-phase flow using electrical capacitance tomography," *2017 IEEE International Conference on Imaging Systems and Techniques (IST)*, 1–5, 2017.
4. Meaney, P. M., C. J. Fox, S. D. Geimer, and K. D. Paulsen, "Electrical characterization of glycerin: Water mixtures: Implications for use as a coupling medium in microwave tomography," *IEEE Trans. Microw. Theory Tech.*, Vol. 65, No. 5, 1471–1478, 2017.
5. Middelburg, L. M., et al., "Multi-domain spectroscopy for composition measurement of water-containing bio-ethanol fuel," *Fuel Process. Technol.*, Vol. 167, 127–135, 2017.
6. Ellison, W. J., K. Lamkaouchi, and J.-M. Moreau, "Water: A dielectric reference," *J. Mol. Liq.*, Vol. 68, No. 2–3, 171–279, 1996.
7. Barthel, J. M. G. and R. Buchner, "High frequency permittivity and its use in the investigation of solution properties," *Pure Appl. Chem.*, Vol. 63, No. 10, 1473–1482, 1991.
8. Sudo, S., N. Shinyashiki, Y. Kitsuki, and S. Yagihara, "Dielectric relaxation time and relaxation time distribution of alcohol-water mixtures," *J. Phys. Chem. A*, Vol. 106, No. 3, 458–464, 2002.
9. Petong, P., R. Pottel, and U. Kaatze, "Water-ethanol mixtures at different compositions and temperatures. A dieletric relaxation study," *J. Phys. Chem. A*, Vol. 104, No. 32, 7420–7428, 2000.
10. Gregory, A. P. and R. N. Clarke, "Tables of the complex permittivity of dielectric reference liquids at frequencies up to 5 GHz," 2012.

11. Baker-Jarvis, J. R., et al., "Measuring the permittivity and permeability of lossy materials: solids, liquids, metals, and negative-index materials," Technical Note (NIST TN)-1536, Jan. 2005.
12. Blackham, D. V. and R. D. Pollard, "An improved technique for permittivity measurements using a coaxial probe," *IEEE Trans. Instrum. Meas.*, Vol. 46, No. 5, 1093–1099, 1997.
13. Nicolson, A. M. and G. F. Ross, "Measurement of the intrinsic properties of materials by time-domain techniques," *IEEE Trans. Instrum. Meas.*, Vol. 19, No. 4, 377–382, Nov. 1970.
14. Ba, D. and P. Sabouroux, "Epsimu, a toolkit for permittivity and permeability measurement in microwave domain at real time of all materials: Applications to solid and semisolid materials," *Microw. Opt. Technol. Lett.*, Vol. 52, No. 12, 2643–2648, 2010.
15. Georget, E., R. Abdeddaim, and P. Sabouroux, "A quasi-universal method to measure the electromagnetic characteristics of usual materials in the microwave range," *Comptes Rendus Phys.*, Vol. 15, No. 5, 448–457, 2014.
16. Ajaya, B. and S. S. Kumar, "Effects of concentration and relative permittivity on the transport properties of sodium chloride in pure water and ethanol-water mixed solvent media," *Res. J. Chem. Sci. ISSN*, Vol. 2231, 606X, 2011.

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