Microwave Heating and Dielectric Characterization for Flow Chemistry

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Abstract*—We introduce a novel scalable microwave-assisted reactor setup that combines four Complementary Split Ring Resonators (CSRRs) operating at multiple frequencies (2, 4, 6, and 8 GHz) with a microfluidic cell, achieving high-temperature uniformity and precise in-situ temperature measurements for microwave-assisted organic synthesis. The reactor achieves high-temperature uniformity according to both COMSOL simulations and temperature measurements using temperature-dependent fluorescent dye Rhodamine B. Finally, the scalability of the proposed setup was investigated using a MW Single Pole, Double Throw (SPDT) switch with reactors operating at the same and various frequencies for a multistep synthesis or double the throughput. In addition, we introduce a highly sensitive microwave dielectric sensor IDC-CSRR for determining glucose concentration. By utilizing a digitally controlled step capacitor, we were able to achieve impedance matching at the two distinct frequencies of 3.69 GHz and 4.38 GHz while conserving the resonant mode of the IDC-CSRR. Both matched frequencies were then used for glucose concentration measurements, resulting in a maximum sensitivity of 8.727 dB/mg/ml and 53.01 kHz/mg/ml and principal component regression (PCR) was used to predict glucose concentration with mean average error (MAE) of only 0.045 mg/ml.

I. Scalable Frequency Selective Microwave-Assisted Reactor Setup for Organic Synthesis [1]

THE chemical industry lags in the transition to greener practices due to old-fashioned and inefficient reactors. For that, four reactors were designed to operate at 2, 4, 6 and 8 GHz.

To determine the temperature uniformity inside the MW reactor, multiphysics COMSOL (5.2) simulations were conducted where frequency domain simulation is coupled to heat transfer in solids. Figure 1 displays the temperature distribution for each of the four heaters when 1 W MW heating power is applied to the water-filled microfluidic cell. For precise temperature readout, the thermocouple (with a 0.25 mm radius) is positioned in the middle of the reactor, slightly influencing temperature distribution as shown in Figure 1. For CSRR1, the temperature around the sensor is 60°C compared to 58°C inside the channel. Nevertheless, the temperature distribution shows excellent uniformity with small hot spots when the sample is positioned above the CSRR's gaps. The highest temperature deviation is present when using CSRR4, where the small part of the sample above the CSRR's gap exhibits 15.2°C higher temperature compared to the temperature in the channel next to the thermocouple. However, temperature uniformity can be further improved by adjusting the shape of the microfluidic cell to avoid positioning the microfluidic channel above CSRR's gaps.

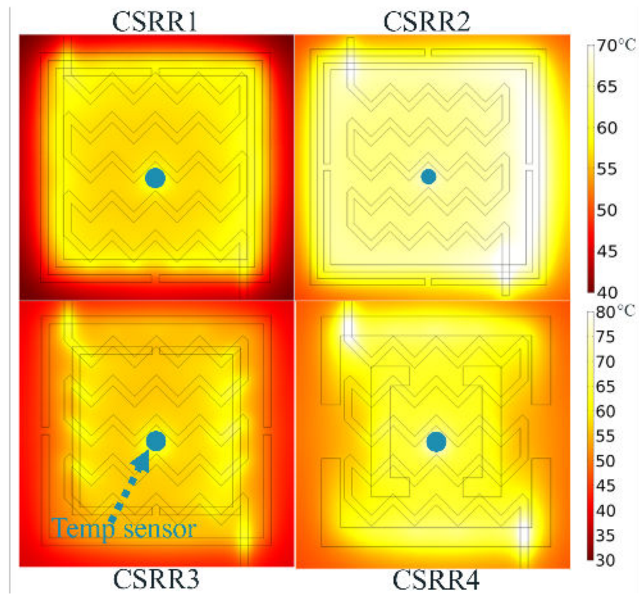

Fig. 1. COMSOL MW heating simulation of designed CSRRs loaded with microfluidic cell filled with water.

A. Scalability Utilizing Microwave SPDT switch

A setup with two reactors in parallel are connected to a voltage-controlled MW SPDT switch, as shown in Figure 2, with voltage supplied and controlled using a power supply (Tenma, 72-13330). Reactor 1 (CSRR1) is working at 2.04 GHz, and Reactor 2 is working at 2.08 (CSRR1) or 3.41 GHz (CSRR2). Due to the possibility of heating only one reactor when using an MW SPDT switch, two distinct set temperatures can be achieved when working with reactors at the same frequency. In addition, when working at different frequencies, it is possible to achieve the set temperature of 60°C when using CSRR2 (Figure 3). However, an additional voltage-controlled source is needed, increasing the cost of the setup.

II. Highly Sensitive Impedance-Matched Microwave Dielectric Sensor [2]

Many different resonant structures were designed for improved sensitivity and accuracy, yet all of them inherently suffer from significantly decreased sensitivity when working

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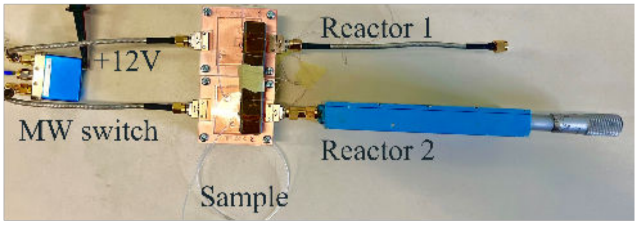

Fig. 2. Setup using an MW SPDT switch where impedance matching was achieved using an open-ended coaxial cable for Reactor 1 and a phase shifter for Reactor 2.

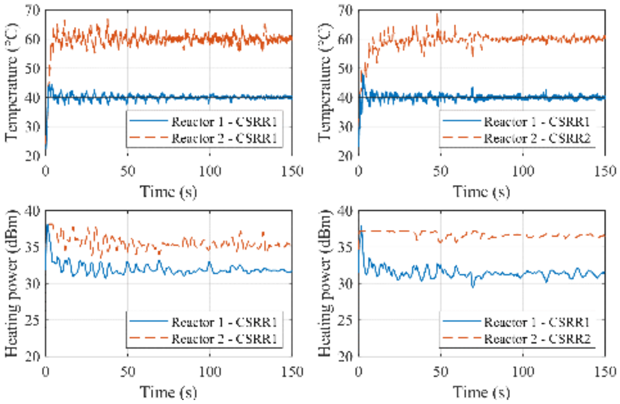

Fig. 3. Measured temperature and applied power using a MW SPDT switch of reactors working at (left) the same and (right) distinct frequencies. CSRR1 is a heater working at 2 GHz, whereas CSRR2 works at 3.41 GHz.

with lossy materials. Impedance matching might solve this issue.

In order to achieve impedance matching of the IDC-CSRR at both frequencies, we need an adjustable length l or an adjustable impedance at the second port. For that, we opted for a digitally controlled step capacitor. This way, it is possible to change the quality of impedance matching and the matched frequency in a stable and repeatable manner, resulting in dielectric sensing at two distinct frequencies.

In our case, the matching of -72.3 dB at 3.69 GHz and -46.2 dB at 4.38 GHz was achieved by altering step capacitor values while the sensor was loaded with water. To maximize the sensitivity of a sensor, it is important to achieve as low S_{11} value as possible with impedance matching, resulting in a large change in S_{11} magnitude with a small change in MUT's permittivity.

The $|S_{11}|$ minimum changes in the range of -78.7 dB to -49.1 dB with a frequency change of 600 kHz with different water-glucose samples (Figure 4 a). The resulting maximum sensitivity is 8.727 dB/mg/ml (a 6.5-fold improvement over interferometer setup) and 53.01 kHz/mg/ml when looking at the $|S_{11}|$ minimum change between Samples 1 and 2.

On the other hand, for the second sweep, the S_{11} magnitude decreases with the sample's concentration increase, with a total range of 9 dB as shown in Figure 4 b. The lower sensitivity of the second matched frequency of 1.27 dB/mg/ml can be attributed to a higher $|S_{11}|$ starting value.

We demonstrated the possibility of the reported setup to precisely distinguish low glucose-water concentration utilizing a principal component regression model with a minimum difference of 0.42 mg/ml between the two sample concentrations. The detection limit of the proposed setup is expected to be between 0.42 mg/ml and 0.045 mg/ml (MAE). The investigation of the detection limit is planned for future work together with the temperature stability improvements and transition to glass microfluidic cells. Such a setup can then be employed for dielectric sensing of the outcomes of chemical reactions.

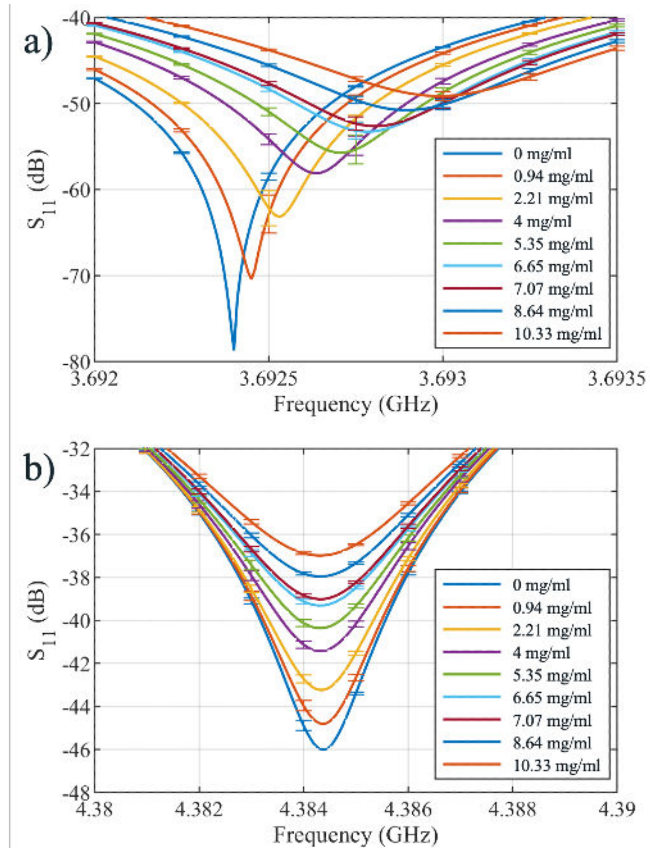

Fig. 4. Average of S-parameter measurements from three sets of measurements of all water-glucose samples for (a) the first and (b) second sweep with corresponding error bars representing maximum deviation.

III. MTT-S Award Implications

Lastly, I would like to thank MTT-s and IEEE for sponsoring our research. Even though I enjoyed the research, I am planning to continue my career in the industry. The knowledge and experience obtained during my PhD, shaped me as a better engineer. This, together with the IMS 2025 conference made possible by MTT-s, gave me experience and connections I will cherish throughout my professional career.

References

[1] M. Martinic, L. Y. Vázquez-Amaya, M. Mertens, U. K. Sharma, E. V. Van der Eycken, G. Vandenbosch, B. Nauwelaers, and T. Markovic, "Scalable frequency selective microwave-assisted reactor setup for organic synthesis," *Available at SSRN 4979533*.

[2] M. Martinic, M. Mertens, D. Schreurs, B. Nauwelaers, G. A. Vandenbosch, and T. Markovic, "Highly sensitive impedance-matched microwave dielectric sensor for glucose concentration measurements," *IEEE Sensors Journal*, 2025.