MICROWAVE RESONANT SENSOR FOR REAL-TIME CONTINUOUS-FLOW MEASUREMENTS OF MICROFLUIDIC SYSTEMS
D. J. Rowe 1, 2, J. Naylon 1, A. Porch 1, D. A. Barrow 1 and C. J. Allender 2
1School of Engineering, Cardiff University, Cardiff CF24 3AA, United Kingdom,
2School of Pharmacy, Cardiff University, Cardiff CF10 3NB, United Kingdom

ABSTRACT
An improved sensor (based on a coaxial resonator) is presented for high-precision dielectric permittivity measurements of fluids in microfluidic systems at GHz frequencies. Accurate, high-speed measurements of complex permittivity for low solute concentrations in a continuous-flow microfluidic system (e.g. acetic acid in toluene, with resolution of 40 ppm at 2.5 GHz) have been demonstrated using a novel digital readout system. The sensor has a higher sensitivity and shorter response time than others reported [1-4], and is easier to integrate into complex microfluidic devices. The high stability of temporal measurements, coupled with the robustness of the design, make this sensor ideal for process control in the pharmaceutical [1],[5], printing and food processing industries [6]. It is also non-invasive, and so can be added in-line to existing microfluidic systems.

KEYWORDS: microwave resonator, dielectric measurement, compositional analysis, industrial microfluidics.

INTRODUCTION
Microwave resonator permittivity measurements offer great potential for accurate, low-cost liquid compositional analysis, but are typically unsuitable for fast and repeatable continuous-flow measurements. Here, we present a measurement system that suits these requirements, which is integrable into any flow system using standard fittings. The demonstrated results show the probe can discern very small compositional changes between two liquids of similar permittivity, but it is equally appropriate for accurately measuring far greater differences in both volume fraction and permittivity. This versatility makes the sensor appropriate for a wide range of microchemical applications.

PERTURBATION THEORY
Resonator perturbations [7],[8] can be used to relate changes in complex resonant frequency to the permittivity of a dielectric sample through consideration of the electromagnetic fields according to an expression derived from Maxwell’s equations. It is possible to extend the perturbation technique to coaxial resonators [9],[10], when open circuit end conditions allow the propagation of evanescent fields that can be perturbed with a dielectric sample. The work presented here uses an empirical simulation-based method rather than approximated admittance models.

If the resonant frequency of the fundamental resonance of the coaxial line is given by \( f_0 \) (\( = \omega_0 / 2\pi \)) and half-power bandwidth by \( f_b \) (\( = f_0 / 2Q_0 \)), then a complex resonant frequency \( \tilde{\omega}_0 \) can be expressed as

\[
\tilde{\omega}_0 = \omega_0 \left( 1 - \frac{j}{2Q_0} \right) = 2\pi \left( f_0 - j \cdot f_b \right)
\]

(1)

A normalised change in this complex frequency can be related to volume integrals of the resonator fields [11]. If the fields occupy a region of complex permittivity \( \varepsilon \) and complex permeability \( \mu \), and are perturbed by an isotropic sample giving a change in permittivity, \( \Delta \varepsilon \), and change in permeability, \( \Delta \mu \), the frequency shift can be expressed by Equation 5. \( \tilde{\mathbf{E}}_0 \) and \( \tilde{\mathbf{H}}_0 \) are the electric and magnetic vector fields, respectively; \( V_0 \) is the volume occupied by the fields; an asterisk denotes the complex conjugate, and the subscript \( S \) denotes a perturbed quantity (i.e. by a material sample).

\[
\frac{\tilde{\omega} - \tilde{\omega}_0}{\tilde{\omega}} = -\int_{V_0} (\Delta \varepsilon \tilde{\mathbf{E}}_0 \cdot \tilde{\mathbf{E}}_0^* + \Delta \mu \tilde{\mathbf{H}}_0 \cdot \tilde{\mathbf{H}}_0^*) \, dv
\]

(2)

If a non-magnetic sample is assumed (i.e. \( \Delta \mu = 0 \)), and the \( \Delta \varepsilon \) term is taken outside of the integral, the remaining integrals can be treated as constants if changes in the fields are assumed to be small. The real and imaginary components of change in permittivity are therefore proportional to the real and imaginary components of change in frequency, which can be expanded in terms of \( f_0 \) and \( f_b \), as shown in Equations 3 and 4, respectively.

\[
\Re(\Delta \varepsilon) = f_{0s} - f_0 \left( 1 + \frac{f_{BS}^2}{f_{0s}^2} \right)^{-1} + f_{BS} - f_{B} \left( 1 + \frac{f_{0s}^2}{f_{BS}^2} \right)^{-1}
\]

(3)

\[
\Im(\Delta \varepsilon) = \frac{f_{0s} - f_{0} \cdot f_{BS}}{f_{0s}^2 + f_{BS}^2} \left( 1 - \frac{f_{0s}^2}{f_{BS}^2} \right)^{-1}
\]

(4)
EXPERIMENTAL DETAIL

To enable inversion of resonator measurements into dielectric properties, simulation of the electromagnetic fields of the resonator was first performed using COMSOL Multiphysics. The simulation included in Figure 1 shows the interaction of the electric field of the resonator with a microfluidic flow. These simulations were used to relate changes in half-power bandwidth and resonant frequency to real and imaginary parts, respectively, of the complex permittivity. The nomogram used for the data inversion is shown in Figure 2.

![Nomogram](image)

**Fig. 1:** A schematic view of the resonant sensor, incorporating a resonant length of RG401 coaxial cable. Also shown is the electric field distribution emanating from the end embedded into a grade 316 stainless steel tube, used to evaluate the dielectric properties of the fluid within the tube.

RG401 cable (OD = \( \frac{3}{4} \) inch), grade 316 stainless steel capillaries (OD = \( \frac{5}{16} \) inch) and Swagelok tube fittings were used to construct the flow cell shown schematically in Figure 1. Frequency domain measurements were taken at a rate of 200Hz with a National Instruments PXI-based RF signal generator/analyser, and used to estimate the resonator power transfer function for the fundamental frequency at 2.5 GHz. The PXI approach to a digital readout system was chosen over a Vector Network Analyser because of the extremely high speed at which it can be used to take measurements.

The experiments used HPLC grade glacial acetic acid dissolved in toluene (99.85%), obtained from Acros Organics. HPLC pumps supplied periodically varying concentrations of acetic acid to the flow cell at a rate of 5ml/min. Mixing of the liquids occurred at a stainless steel T-piece and was enhanced with an Agilent HPLC mixer. Permittivity as a function of acetic acid concentration is shown in Figure 3. A combination of simulation and measurement was used to estimate the sensor parameters shown in Table 1.

**RESULTS AND ANALYSIS**

The lag between resonator response and acetic acid concentration supplied is due to the time taken for the solution to reach the resonator, and the curvature of each step can be attributed to dispersion between the different concentration phases, which was also exaggerated with larger changes in concentration.

It can be seen that step changes in concentration yield differing changes in permittivity. This showed no correlation with temperature change, which varied erratically but did not exhibit any general upward or downward trend. The unit was sealed, and care was taken to prevent any bubbles from forming. It is not thought that this effect is due to solvents remaining in dead volumes within the T-pieces, as the additional solvents used to clean the system were invariably of higher permittivity than either toluene or acetic acid. The PTFE spacer is sufficiently hydrophobic to prevent ingress of solution between the layers of the coaxial cable. However, this effect could be due to the inner geometry of the T-pieces, which could disturb flow in a way that prevents complete circulation of fluid within the flow cell. Also, no specific effort was made to maintain back pressure in the fluidic circuit, so it cannot be assumed that the pumps behaved perfectly and mixed solutions to the programmed concentrations. Further experimentation is underway to verify whether this could explain the observed irregularities.
Fig. 3: A plot illustrating the change in sample permittivity, $\varepsilon$, as the volume fraction of acetic acid in toluene varies with respect to time.

Table 1: Sensor parameters: resolution ($R$), limit of detection ($\varepsilon_{LOD}$), dynamic range ($\varepsilon_{DR}$), sensitivity to changes in centre frequency ($\delta\varepsilon/\delta f_c$) and bandwidth ($\delta\varepsilon/\delta f_{bw}$) at the limit of detection, uncertainty ($u$) and measurement period ($t$).

<table>
<thead>
<tr>
<th>$R$ (ppm)</th>
<th>$\varepsilon_{LOD}$</th>
<th>$\varepsilon_{DR}$</th>
<th>$\delta\varepsilon/\delta f_c$ (GHz$^{-1}$)</th>
<th>$\delta\varepsilon/\delta f_{bw}$ (GHz$^{-1}$)</th>
<th>$u$ (%)</th>
<th>$t$ (ms)</th>
</tr>
</thead>
<tbody>
<tr>
<td>40</td>
<td>1.00011</td>
<td>20-38</td>
<td>46.18</td>
<td>42.14</td>
<td>±1.7</td>
<td>5</td>
</tr>
</tbody>
</table>

CONCLUDING REMARKS
Owing to the ease of integration and simplicity of operation once characterised, this sensor offers great potential for multi-sensor fusion. This could be for an ‘electronic tongue’ device, which would incorporate different sensing technologies to identify fluids. The architecture of the digital readout system is suitable for implementation with low cost, mass-produced components designed for the telecommunication industry and thus the complete sensor system is viable for large-scale, portable and low-cost applications. By measuring multiple harmonics (e.g. at $n \times 2.5$ GHz, $n = 1, 2, 3$ etc.), accurate dielectric spectra of microfluidic mixtures can be determined, thus providing a “fingerprint” of the test solution for multi-sensor applications, together with the benefits described above.

Further work is required to reduce the observed errors. This will initially involve verifying pump operation and solvent mixing. A complete, self-contained device is also being considered: passive microfluidic mixers could be used as an alternative to stainless steel T-pieces for the sake of sensor characterisation, in order to prevent irregular flow and dead volumes at the fluid-probe interface.

REFERENCES


