SPLIT RING RESONATOR TECHNIQUE FOR COMPOSITIONAL ANALYSIS OF SOLVENTS IN MICROCAPILLARY SYSTEMS

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ABSTRACT

A 3.4 GHz split ring resonator has been developed for highly sensitive remote compositional analysis of PEEK microcapillaries containing mixtures of acetonitrile in toluene. As little as 0.1% vol. acetonitrile can be measured in an active volume of around 50 nl. The method is fast (< 0.1 s per measurement) and is adaptable to compositional analysis where fluid flow and/or chemical reactions are occurring.

KEYWORDS: Microwave resonator, dielectric properties, solvent composition

INTRODUCTION

Microwave resonators enable precise, non-invasive and fast compositional analysis of liquid mixtures for a variety of industrial, analytical and quality control applications. Previously, we have demonstrated successfully a hairpin resonator for in-situ analysis of a microcapillary containing a mixture of polar solvents [1]. Here, the use of a split ring resonator has enhanced significantly the measurement sensitivity.

THEORY

The presence of a sample in the high electric field region in the resonator's gap (see Figure 1) will reduce both the resonant frequency f_0 and quality factor Q owing to polarization. These can be expressed using resonator perturbation analysis, giving

$$\frac{\Delta f_0}{f_0} \approx -\frac{\mathrm{Re}(\alpha)}{2V_{\mathrm{eff}}} \qquad (1) \qquad\qquad \Delta\left(\frac{1}{Q}\right) \approx -\frac{\mathrm{Im}(\alpha)}{V_{\mathrm{eff}}} \qquad (2)$$

where α is the sample's electric polarizability (defined by the induced electric dipole moment $p = \alpha\varepsilon_0 E_0$, E_0 being the applied electric field) and V_{eff} is the volume occupied by the electric field energy, essentially the volume of the gap. The split ring resonator is a lumped element structure [2] that can be designed to have a small V_{eff}, resulting in high sensitivity to the sample's dielectric polarization and power loss.

EXPERIMENTAL

The split ring resonator of Figure 1 consists of a copper ring having a length, thickness and inner radius each of 3 mm. The gap was machined mechanically to a width of 400 μm, yielding a resonant frequency of ≈ 3.4 GHz. Transmitted microwave power was measured in the frequency domain using a vector network analyzer. The empty resonator has a high quality factor ($Q \approx 1400$) for such a small $V_{\mathrm{eff}} \approx$

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$0.4 \times 3.0 \times 3.0\ mm^3 \approx 3.6\ \mu l$. A liquid-containing PEEK microcapillary (inner radius 75 μm) was passed through the gap, where the microwave electric field is uniform and perpendicular to the microcapillary's axis, and the liquid composition varied. The liquid volume is approximately 50 nl in each case. At resonance, less than 10 μW of microwave power is dissipated in the sample, so heating effects are negligible.

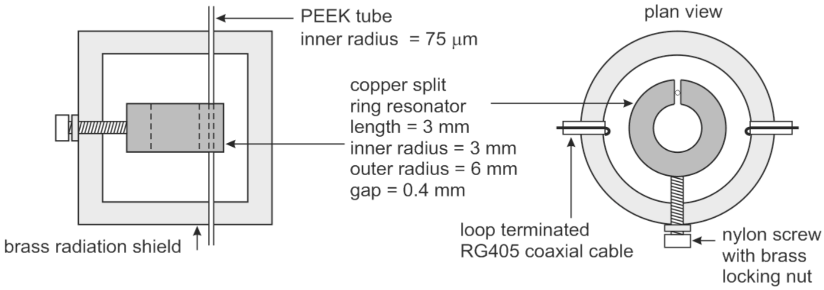

Figure 1. Schematic diagram of the 3.4 GHz split ring resonator package.

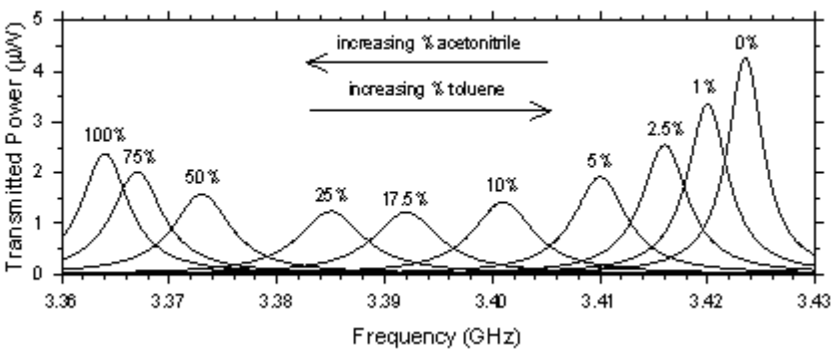

Figure 2. The transmitted power measured as a function of frequency for various acetonitrile:toluene solvent mixtures (volume % of acetonitrile is shown).

RESULTS AND DISCUSSION

Mixtures of acetonitrile in toluene were first measured using a coaxial reflectance probe [1] to establish the variation of complex permittivity with composition. Resonances perturbed by solvent mixtures are shown in Figure 2, from which the change of resonant frequency and unloaded Q factor were measured (Figures 3 and 4, respectively). The resonant frequency decreased monotonically as the polar nature of the solvent mixture increased. Also shown in Figure 3 is the theoretical prediction for a liquid-filled PEEK capillary, based on the measured dielectric properties of the component liquids and PEEK tube. The Q was minimized (i.e. microwave power loss maximized) for small proportions of acetonitrile ($\approx$ 18%). This is entirely predicable by theory (solid curve of Figure 4), its physical interpretation being the competition between the increased dielectric loss and the reduced internal electric field (by depolarization) within the solvent mixture as the proportion of acetonitrile was increased.

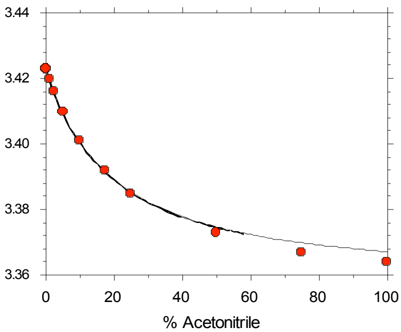

Figure 3. The resonant frequency as a function of fluid composition (the solid line is the theoretical prediction from Eqn. 1, based on the measured dielectric properties of the solvent mixture).

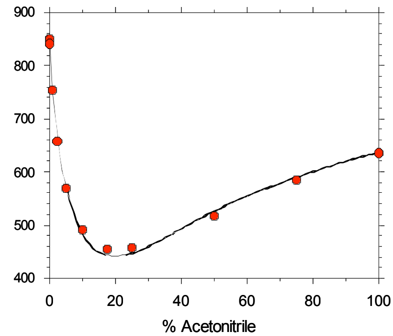

Figure 4. The unloaded quality factor as a function of fluid composition (solid line is the theoretical prediction from Eqn. 2). Note the sensitivity to small % volume of polar species.

CONCLUSIONS

There are numerous benefits of this split ring microwave resonator technique over other methods. Firstly, it is fast, with data accumulation on a timescale of less than 0.1 s, hence enabling compositional analysis in situations where fluid flow and/or chemical reaction are occurring. Secondly, being wavelength independent, the size of split ring resonator can be miniaturized to suit the system under test. Thirdly, the very small volume occupied by the microwave electric field energy (i.e. volume of the gap region) leads to very sensitive compositional analysis compared to resonator techniques where the field energy is distributed over a larger volume [1], [3]; here, less than 0.1% by volume of acetonitrile in toluene can be detected. We plan to further enhance the sensitivity by using microwave resonators whose electric field is along the microcapillary axis, thereby leading to negligible depolarization.

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