EFFICIENT MICROWAVE HEATING AND DIELECTRIC CHARACTERIZATION OF MICROFLUIDIC SYSTEMS

J. Naylon*, S. Gooding, C. John, A. Morgan, O. Squires, J. Lees, D.A. Barrow and A. Porch

School of Engineering, Cardiff University, Cardiff, CF24 3AA, United Kingdom

ABSTRACT

This paper presents a 2.45 GHz microwave cavity resonator with the novel dual function of both sensitive dielectric characterisation and directed, volumetric heating of fluids in a microfluidic chip. This method is shown to have a higher efficiency (>90 % for methanol), higher rate of heating and more accurate control than previous microfluidic microwave heating methods [1], [2]. The system is simple, robust and does not require on-chip integration of waveguide structures. Furthermore, this method can be combined with miniaturised resonators such as split rings [3] for localisation of heating within a microfluidic chip.

KEYWORDS: Microwave resonator, dielectric heating, industrial microfluidics, microwave-assisted extraction

INTRODUCTION

Compared to traditional microfluidic heating methods such as Joule heating via embedded wires or micro Peltier junctions, microwave heating has several advantages. It is efficient, non-contact, volumetric, selective (*i.e.*, only polar liquids heat appreciably) and immediate, with no thermal transport, so heating begins on a ns time scale.

With our single mode resonator and novel control system, high precision permittivity measurements on a ms sample time can also be made, allowing high speed temperature control without the need for additional temperature sensors. Compared to non-resonant microwave excitation, this method has a much greater efficiency and around 1000 times greater sensitivity, and it does not require the integration of lossy waveguide structures on chip, which also simplifies fabrication.

The combination of microwave heating in a multiphase system, particularly with a solid phase consisting of strongly absorbing particles, has great potential for high efficiency separation and synthesis. Complex permittivity is also a useful diagnostic measurement which has been proven for microfluidic compositional analysis [3] and is a good additional input to multisensor fusion systems owing to its high sensitivity and low noise. Previous applications of microwave heating which could all benefit from this resonant technique include on-chip reactors, microwave-assisted extraction (MAE) [4], DNA amplification [5], and polymer bead synthesis [6].

Additionally, since rapid heating is possible with only a few Watts, low voltage power supplies and miniaturised components designed for mobile communications can be used, allowing the entire system to be scaled into compact, portable devices and facilitating truly miniaturised microsynthesis systems and scalable industrial microfluidics.

THEORY

Dielectric heating in fluids is caused by Joule heating through the movement of ionic charge carriers and the viscous damping of dipole movement induced by a time-varying electric field. The time-averaged heating power ‹*P*› within a sample volume V_S produced by a uniform applied electric field *E* at frequency *ω* is given by:

$$\langle P \rangle = \tfrac{1}{2}\omega\varepsilon_0\varepsilon_S'' V_S \cdot \left| \frac{\varepsilon_M}{\varepsilon_M + N(\varepsilon_S - \varepsilon_M)} \right| \cdot |E|^2 \quad (1)$$

Heating is governed by ε''_S, the sample's dielectric loss factor. Since ε''_S is material-specific, selective and efficient heating is possible. The term immediately preceding $|E|^2$ accounts for the depolarization of the sample; ε_M is the permittivity of the surrounding matrix and ε_S is the sample permittivity. Depolarization occurs when the electric field crosses the sample boundary, the factor *N* then depends on sample geometry; for a spherical droplet, *N* = 1/3, and for a cylindrical channel with the field perpendicular to its axis, N = 1/2. Only when the field is applied along the axis of a channel is *N* = 0, the term is then 1 and the geometry is said to be non-depolarizing. Depolarization can make heating inefficient; *e.g.*, the heating power of a water droplet, $\varepsilon_S \approx 80 - 10i$, in a low loss fluorocarbon oil, $\varepsilon_M \approx 2$, is about 1/20 of a water column in a non-depolarizing geometry.

Microwave resonators have unparalleled sensitivity to changes in permittivity. The measurement is based on the dependence between the resonant frequency and sample permittivity, and between the change in the 'sharpness' or *Q* of the resonance and the sample loss. If the sample perturbs the stored energy only slightly then this dependence is linear.

EXPERIMENTAL

The assembly of the rectangular cavity is shown in Figure 1. The TE_{011} mode is used since there is no electric field depolarization within the vertical sections of the microfluidic channels, as shown in Figure 2, thus achieving a high efficiency and heating rate with µl samples (> 90 % efficiency for methanol). Microfluidic devices were fabricated from PTFE with micromilled channels, bonded using a commercial polyolefin-based bonding film to form a durable cartridge

(90×60×4 mm) which can be inserted into a slot in the cavity. For these experiments a simple ‘U-shaped’ channel of 500×500 µm was used. M6 finger-tight fittings fastened with a connector bar are used to interface to a flow system.

In our novel measurement system, shown in Figure 3, the resonator is characterized by passing a modulated signal through the resonator, demodulating it and analyzing the response digitally. The complex permittivity of fluids is thus determined in real time without requiring a vector network analyzer. Microwave power up to 2 W is provided by a synthesiser combined with a 35 dB power amplifier and is coupled into the resonator using a magnetic coupling loop. Both sensing and heating are controlled using a National Instruments (NI) RF signal generator (RFSG) and RF signal analyzer (RFSA), which allows a real time sampling period as low as 5 ms. Control of heating is achieved by varying the period of a heating pulse modulated by a PiN-diode switch (internal to the RFSG). The architecture of this system is similar to a simple communications front-end and thus may be implemented at 2.45 GHz very economically with commercially available and miniaturised electronics designed for the telecommunications industry.

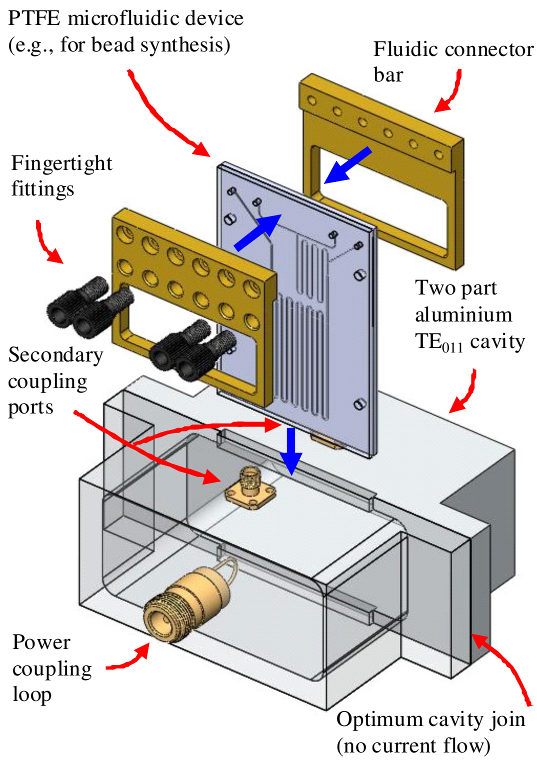

Figure 1: Assembly graphic of the microwave cavity-based microfluidic heating system.

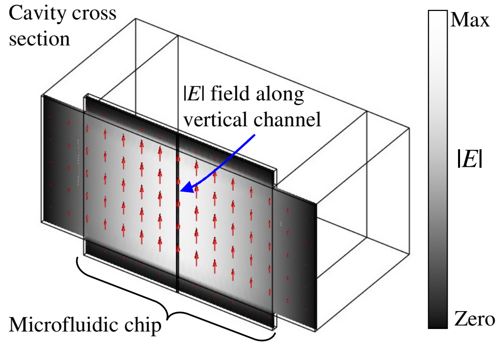

Figure 2: Finite element simulation of the cavity with chip consisting of a single channel. Arrows show E field direction.

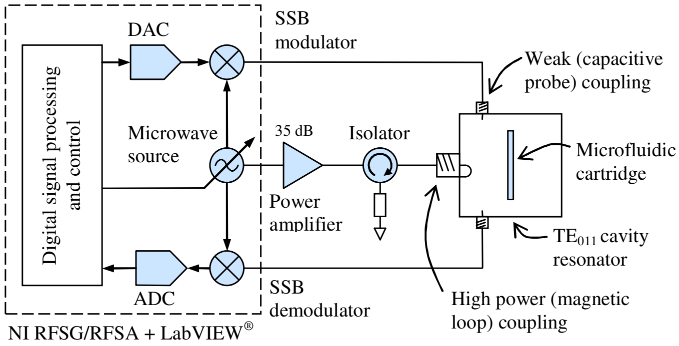

Figure 3: Microwave control system configuration.

RESULTS AND DISCUSSION

A range of solvents were tested within the resonator; the resonator frequency response with each is shown in Figure 4. Maximum power transfer into the resonator occurs when so-called critical coupling is achieved. For simplicity in our device coupling was adjusted manually by rotating the power coupling loop. A reflection coefficient $|\Gamma| < -40$ dB can be achieved, however only with servo control can critical coupling be ensured for all samples and temperatures. Cavity perturbation theory states that sample heating efficiency is related to the ratio of the resonator’s Q with the sample to the Q with no sample (*i.e.*, air). Thus the overall heating efficiency in terms of amplifier output power is:

$$\frac{P_{sample}}{P_{amplifier}} = \left(1 - |\Gamma|^2\right) \cdot \left(1 - \frac{Q_{sample}}{Q_{empty}}\right). \tag{2}$$

This equation was used to estimate the heating efficiency for various solvents (Figure 6) and standard perturbation theory was used to estimate their permittivity (Figure 7). The uncertainty in absolute values of permittivity (~ 5 %) is dominated by the static uncertainty in the channel dimensions. However, the measurement deviation is just a few ppm, so differential comparisons are much more accurate. The measured response of water to a 2 W pulse is shown in the inset of Figure 4. The initial heating rate is approximately 10 K/s, which slows as the water is heated due to the loss of critical coupling (which could be prevented with dynamic coupling control) and the reduction in the loss factor of water.

The permittivity temperature dependence of a known material can be used to regulate heating. A controlled heating cycle based on feedback of permittivity measurements is shown in Figure 5. HPLC grade water was heated at a flow rate of 0.5 ml/min, the sampling rate was 50ms. The instability at the top of the heating cycle is thought to be due to bubble nucleation, as gas segments were seen at the outlet. A better control algorithm could reduce the magnitude of the additional fluctuations.

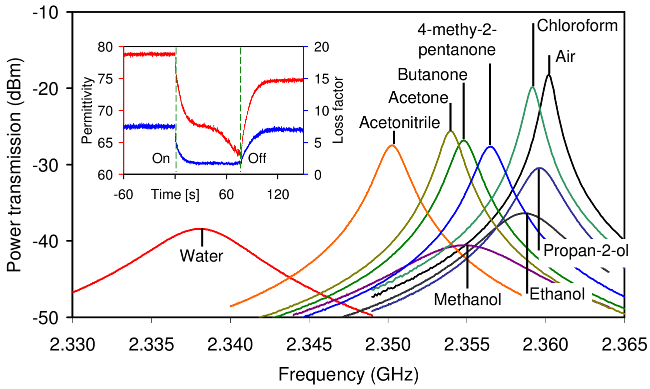

Figure 4: *Microwave transmission response of the cavity resonator with different solvents present in the microfluidic chip. Inset: Response of water to a 2 W pulse of microwave power. Axes are permittivity against time in seconds*

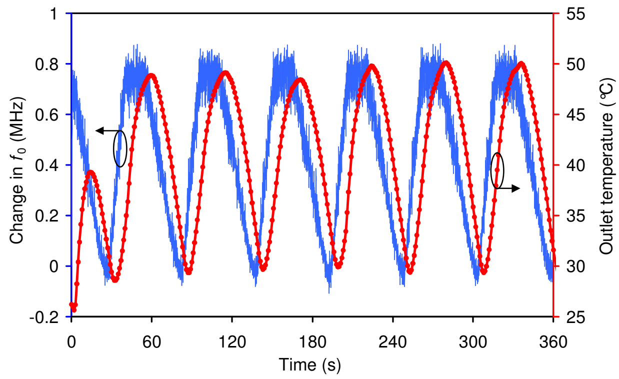

Figure 5: *Change in resonant frequency over an arbitrary controlled heating cycle (linear ramp, hold at 50°C and linear cool-down). Temperature measured by a thermocouple at the chip outlet is shown in red.*

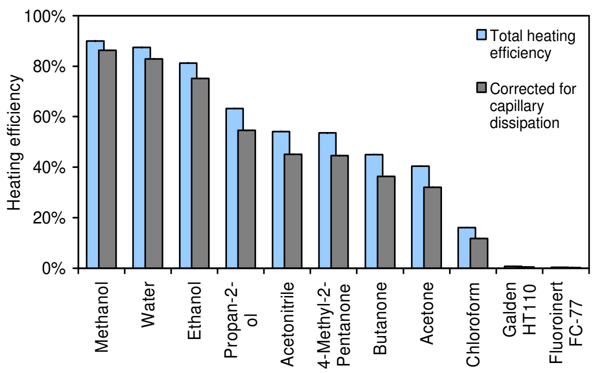

Figure 6: *Estimated heating efficiency based on resonator measurements of solvents. The second set of data are corrected for the dissipation in the chip itself.*

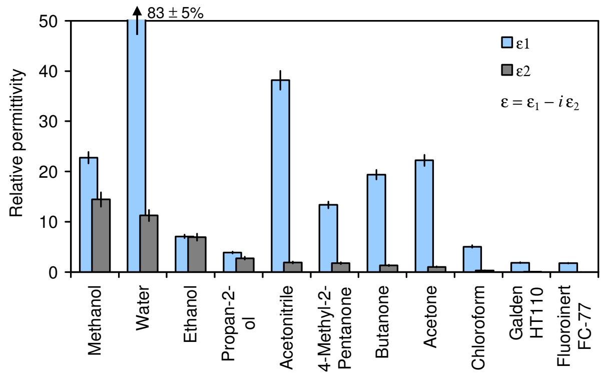

Figure 7: *Complex relative permittivity of solvents based on standard cavity perturbation theory, from the same experimental setup as the data in Figure 5.*

CONCLUSION

Using a resonator to provide microwave heating results in a high efficiency and combined with our measurement system also provides simultaneous permittivity measurement which can be successfully used for temperature control. Due to the non-depolarizing geometry and high thermal isolation, heating rates are close to the maximum possible with any heating method for high loss factor liquids such as water and methanol. Future work will be to incorporate servo-controlled coupling, localisation of heating using miniature resonators, and refinement of the heating control system.

REFERENCES

[1] J.J. Shah, S.G. Sundaresan, J. Geist, D.R. Reyes, J.C. Booth, M.V. Rao, and M. Gaitan, "Microwave dielectric heating of fluids in an integrated microfluidic device," *Journal of Micromechanics and Microengineering*, vol. 17, 2007, pp. 2224-2230.

[2] D. Issadore, K.J. Humphry, K.A. Brown, L. Sandberg, D.A. Weitz, and R.M. Westervelt, "Microwave dielectric heating of drops in microfluidic devices," *Lab on a Chip*, vol. 9, 2009, pp. 1701-1706.

[3] A. Masood, O. Castell, D. A. Barrow, C. Allender and A. Porch, "Split Ring Resonator Technique for Compositional Analysis of Solvents in Microcapillary Systems", *The Proceedings of MicroTAS 2008 Conference*, San Diego, pp.1636-1638, October 2008.

[4] C.S. Eskilsson and E. Björklund, "Analytical-scale microwave-assisted extraction," *Journal of Chromatography A*, vol. 902, 2000, pp. 227 - 250.

[5] A. Kempitiya, D.A. Borca-Tasciuc, H.S. Mohamed, and M.M. Hella, "Localized microwave heating in microwells for parallel DNA amplification applications," *Applied Physics Letters*, vol. 94, 2009, p. 064106.

[6] Y. Zhang, R. Liu, Y. Hu, and G. Li, "Microwave Heating in Preparation of Magnetic Molecularly Imprinted Polymer Beads for Trace Triazines Analysis in Complicated Samples," *Analytical Chemistry*, vol. 81, Feb. 2009, pp. 967-976.

CONTACT

*Jack Naylon; NaylonJ@cardiff.ac.uk