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Online measurement of mass density and viscosity of pL fluid samples with suspended microchannel resonator

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ABSTRACT

Physical characterization of viscous samples is crucial in chemical, pharma and petroleum industry. For example, in the refining industry of petroleum, water percentage is verified by measuring the density of a sample. In this article we present a suspended microchannel resonator (SMR) which uses 5 pL of a fluid sample and measures its density with a resolution of 0.01 kg/m^3 and a sensitivity of 16 Hz/kg/m^3. The resonator can also simultaneously measure viscosity of the solutions with an accuracy of 0.025 mPa s. The SMR is part of a system which contains packaging and tubing to deliver samples to the resonator. The system can easily handle multiple viscous fluids to measure their densities and viscosities. The SMR is transparent, facilitating visual inspection of the microchannel content.

1. Introduction

Density and viscosity are known as important physical properties of fluidic samples in pharmaceutical, chemical and petroleum industries. For example, in pharmaceutical plants the concentration of a solution is determined by its mass density. Since drug samples are generally quite expensive, only small amounts are available for characterization. In petroleum refineries as well as in the automotive industry, oil analysis requires very accurate density measurements to ensure required lubrication and water percentage. For characterization purpose, oil samples are available in large quantities but they are viscous and difficult to handle. Microfluidics is already finding applications in analysis of crude oil at microscale [1,2].

For a variety of applications, density sensors have been reported, dealing with fluids like; alcohols [3,4], water/propanol mixtures [5], sucrose/water solution [6], glycerol [7], olive oil [8], slurries [9], various alkane compounds [10], sulphuric acid [11] and single biological cells [12]. All of these works present sensors that either; need large volumes of a sample, cannot handle a variety of samples or require to be fully immersed in a fluid to measure its density or viscosity. Immersing for example a cantilever sensor into a fluid highly degrades the performance of the device.

Along with the density, knowledge of viscosity is also of great interest for applications such as lubricating oils [13], rheological behaviour of paint [14], analysis of polymers [15] and milk analysis [16]. Some of the reported micro viscosity sensors are; AFM cantilevers [17], double clamped beams [18] and plain cantilevers [19,20]. Recently Livak-Dahl et al. reported a nanoliter viscometer [21] where the viscosity of a sample is measured by observing a flow rate of a sample. In this approach, prior information like surface tension and miscibility of a sample are required.

Burg et al. have reported a suspended microchannel resonator (SMR), which is basically a cantilever resonator with an integrated microchannel, and used it as a density [22] and viscosity sensor [23]. However, in this earlier work, the SMR was only used to characterize a single type of viscous liquid and the microchannels were opaque impeding visual inspection. Using SMR, the density of liquid is extracted from a change in resonant frequency of the SMR. This principle of measuring density has already been demonstrated in the form of resonating U-tubes [24] and suspended resonator plates [25]. The resonating tubes are also currently being used in commercial instruments for density measurement [26]. Macro sized U-tubes provide very precise density results. But they might not be economical in industries where even small quantities of samples are relatively expensive. The SMR and suspended resonator plates have presented promising results but their complicated fabrication makes them expensive at a commercial scale. Also, in most cases a new sensor needs to be used for every viscous sample, since it is not possible to empty the sensor between measurements. This might introduce errors due to small variations in sensor performances.

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Here we report a SMR with a transparent microfluidic channel designed to handle multiple viscous samples. The SMR has a structural mass of 12 ng and a sensitivity of 16 Hz/kg/m^3. We have observed that the transparency of the microchannel provides an advantage of visual inspection for the presence of air bubbles, blocked channels and remains of previous samples in the channel. The SMR can provide a cost effective solution for repetitive measurements by employing a single chip to be used for multiple samples.

In our experiments we evaluated the ability of a single SMR to handle viscous sticky samples of crude oil which are often tricky to handle in microfluidic devices. To measure density and viscosity, four samples of interest were identified; Isopar-L, Isopar-V, Oil-SA and Oil-D [27]. The later two samples were completely unknown with no prior information available on their physical properties. In addition, we tested water, isopropanol and ethanol. With a volume of 5 pL, the SMR measured the mass density of the loaded liquid with a resolution of 0.01 kg/m^3. It was fabricated using MEMS technology and made of silicon nitride. A measurement chip containing a single SMR, was packaged in such a way that the sample of interest was introduced from the bottom of the chip while vacuum and optical access were provided from the top. This technique allowed a quick exchange of chips and fast pumping of an integrated small vacuum chamber.

2. Device design, fabrication and packaging

A schematic drawing of the sensor chip is shown in Fig. 1a. The SMR is located in the center of the chip. The cantilever resonator is 20 μm wide and 200 μm long with fluidic-channel dimensions of 4 μm × 3 μm. It is connected by larger sample delivery channels (SDC) to access-through holes connecting the backside of the chip to macro sized fluidic tubing. Each SDC is 150 μm wide and approximately 2300 μm long.

The fabrication of the SMR was done by a combination of surface and bulk micromachining of a silicon wafer. Low-stress silicon-rich silicon nitride was used as a structural material while polysilicon was used as a sacrificial material to create the hollow microchannel. Both materials were deposited by chemical vapour deposition, patterned by contact photolithography, and structured by dry and wet etching. Most of the fabrication is explained in our previous work [28]. In the current devices, the etching of the access holes and sacrificial material was done simultaneously by an 18 h KOH-etch. The release of the SMR, etching of SDCs and chip boundary was achieved in a single step of KOH etching. A single 4-in. wafer contains 52 sensor chips with a size of 10 mm × 10 mm. Fig. 1b shows the topview of a finished sensor chip. In Fig. 1c a fully etched silicon nitride microchannel is depicted.

The packaging consisted of a polyetheretherketone (PEEK) fixture at the bottom and an aluminium lid at the top of the chip. A schematic of the packaging is shown in Fig. 2. The PEEK fixture provided microfluidic connections, a piezo-actuator, and a temperature sensor. The aluminium top lid provided vacuum and contained a window for an optical readout. The sealing between the PEEK fixture and the sensor chip was provided by a 300 μm thick polydimethylsiloxane layer (PDMS). The PDMS-seal was prepared by a 10:1 ratio of elastomer and curing agent from "Sylgard 184 Silicone Elastomer Kit". Afterwards, a CO^2-laser was used to pattern the PDMS-seal into square pieces equal to the size of the sensor chip. The patterning by laser created a lot of particles which got stuck on the seal. Therefore, the PDMS-seal was sonicated in an ethanol solution for 30 min to remove particles. Later it was dried for 48 h at room temperature. The fluidic samples were provided to the SMR chip by Teflon tubing (inner diameter: 1 mm). The sealing between the chip and the aluminium lid is provided by an o-ring

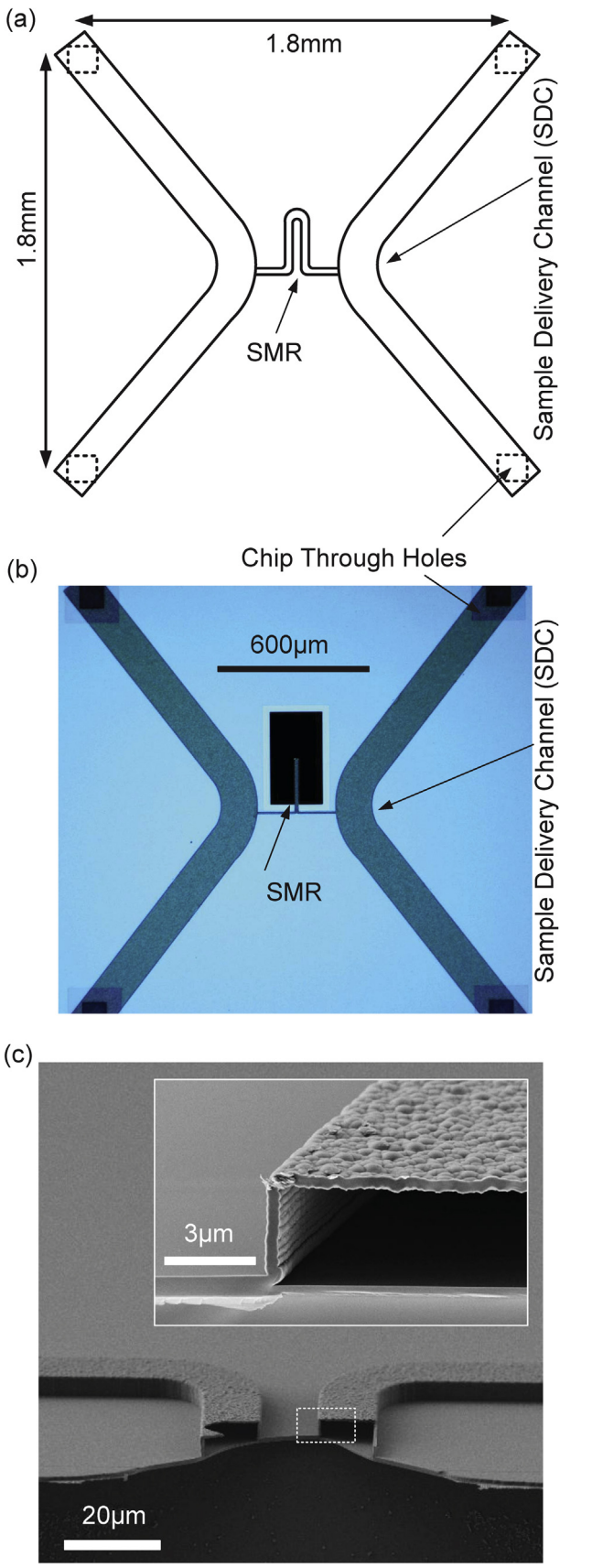

Fig. 1. (a) Schematic drawing of a chip with the suspended microchannel resonator in the center. (b) Optical microscope view from the top of sensor chip. (c) Scanning electron microscope image of microchannel cross section.

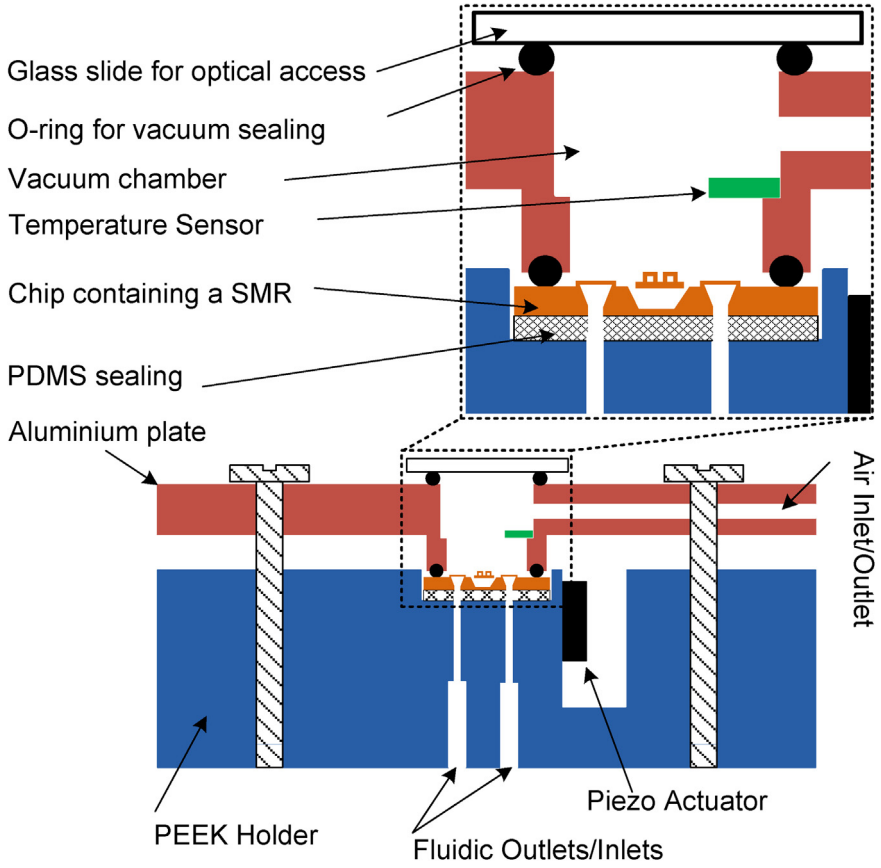

Fig. 2. Schematic of the packaging of the SMR chip. The measurements were performed at a pressure below than 1×10^{-5} mbar. Temperature inside the vacuum chamber was recorded by a sensor PT-1000. Multiple liquid samples were supplied from the bottom of the chip. SMR chips can be quickly exchanged in the packaging.

(nitrile butadiene rubber) while the top side of the vacuum chamber is sealed by another o-ring and a microscope slide.

3. Theory

A linear resonator can be characterized by its eigenfrequency f_0 and quality factor Q. The fluid inside the suspended microchannel is, to a good approximation, constituting a homogeneous mass load of the cantilever. The continuum mechanical resonator can then be described as a linear lumped-model resonator. For low damping, the measured resonance frequency ω_r becomes approximately equal to the undamped eigenfrequency ω_0

$$2\pi f_r = \omega_r \approx \omega_0 = \sqrt{\frac{k}{m}} \tag{1}$$

where k is the spring constant and m is the mass. The mass is the sum of the mass of the empty cantilever m_c and the mass of the fluid in the microchannel m_f

$$m = m_c + m_f = V_c\rho_c + V_f\rho_f \tag{2}$$

with the volumes V and mass densities ρ. Under the assumption that the fluid in the microchannel does not change the cantilever stiffness k, Eq. (1) can be simplified to

$$\omega_r = \frac{A}{\sqrt{B + \rho_f}} \tag{3}$$

where A and B are constants that can be determined from resonance frequency measurements of two different fluids with well known mass densities, such as water and air. With A and B the mass density of a fluid inside the suspended microchannel can be determined by

$$\rho_f = \left(\frac{A}{\omega_r}\right)^2 - B. \tag{4}$$

The quality factor is defined as the ratio of energy stored W to energy lost ΔW over a cycle of oscillation. For a linear resonator it can be written as

$$Q = 2\pi\frac{W}{\Delta W} = \frac{m\omega_r}{c} \tag{5}$$

where c is the viscous damping coefficient. The total damping is the sum of the damping of the empty cantilever c_c and the fluid in the suspended microchannel c_f

$$c = c_c + c_f. \tag{6}$$

With (1) and (5) can be rewritten as

$$c = \frac{m\omega_r}{Q} \approx \frac{k}{\omega_r Q}. \tag{7}$$

The stiffness is again assumed not to change and k can be found from a measurement with air in the microchannel. The viscosity of air is significantly smaller than the viscosity of the fluids to be measured and the stiffness can be calculated by

$$k = c_c\omega_{r,air}Q_{air}. \tag{8}$$

It is now possible to write the ratio of the viscous damping coefficient of the fluid to the viscous damping coefficient of the empty cantilever

$$\frac{c_f}{c_c} = \frac{\omega_{r,air}Q_{air}}{\omega_f Q_f} - 1. \tag{9}$$

Similar to the density measurements, the intrinsic damping coefficient c_c could be determined from a sample with well known viscosity, such as water. But the measured viscous damping in a microchannel cantilever is non-monotonic [23] and it is therefore only possible to measure the viscosity of a fluid sample in a narrow viscosity range.

4. Experimental

Before measuring the density of a targeted fluid, the resonance frequency of the SMR was separately measured with air and water present in the microchannel. This was done for calibration purpose for later density calculations. The resonance frequency was measured with a laser Doppler vibrometer (MSA-500 from Polytec GmbH) with twenty times averaging. The resonator was driven by a piezo in the linear regime. The resonance frequency and quality factor were then extracted from the resonance peak.

After calibration, isopropanol and ethanol were measured first with the SMR. The microchannel was flushed with water in-between measurements for cleaning. Then individual oil samples (isopar-L, isopar-V, oil-SA and oil-D) were delivered to the system. In order to clean the microchannel from the oil, toluene was loaded into the system which remained inside for about 5 min. Finally, the channels were flushed with acetone. For temperature and flow stability, each sample was kept for 4 min in the microchannel before starting a measurement. The temperature was monitored with a temperature sensor (PT1000) inside the packaging (see Fig. 2). Between and during all sample exchanges, the SMR was visually inspected to ensure that the channels were cleaned, filled and emptied as planned.

5. Results and discussion

Fig. 3a shows the resonance frequency for different samples with various mass density and viscosity. First, the SMR was calibrated according to (3) by measuring the resonance frequency when filled with water and air. The SMR has a sensitivity of 16 Hz/kg/m^3. With a frequency resolution of 0.16 Hz measured in a closed-loop oscillation for a time period of 20 s (data not shown), a mass density resolution of 0.01 kg/m^3 results. The measurements of all the samples have an accuracy of 0.762 kg/m^3. The measurement of Oil-D is treated as an outlier and was dismissed in the calculation of the accuracy. In Fig. 3b the mass density, determined with the calibrated SMR at 24.58 ± 0.05 °C, is compared to the mass density measured with a reference sensor at 24.99 ± 0.05 °C. From the correlation plot an offset to the reference sensor of +1.0 kg/m^3 is found. The mass density resolution and accuracy of the SMR of 0.01 kg/m^3 and 0.762 kg/m^3, respectively, is comparable to state-of-the-art mass density sensors (Anton Paar DMA-4100) with a repeatability and accuracy of 0.05 kg/m^3 and 0.1 kg/m^3, respectively. The offset of +1.0 kg/m^3 is mainly coming from the temperature difference between the two measurements of 0.4 °C. The small sample volume used in the SMR reduces the time required to reach thermal equilibrium and is small enough to enable real-time mass density measurements. The reference sensor typically needs 30 s for a single measurement.

In addition to the resonance frequency, the quality factor (Q) of the SMR was recorded simultaneously in order to extract the viscous damping of the sample. Fig. 4 shows the viscous damping of the sample compared to the damping of the empty SMR (9) versus the sample viscosity. The sample viscosity was measured with a reference sensor (Anton Paar AMV-200) with an accuracy of 0.5%. Except for the sample Oil-D, there is a clear trend of the measured viscous damping to the sample viscosity. It has been shown that the damping of an SMR is a non-monotonic function of the

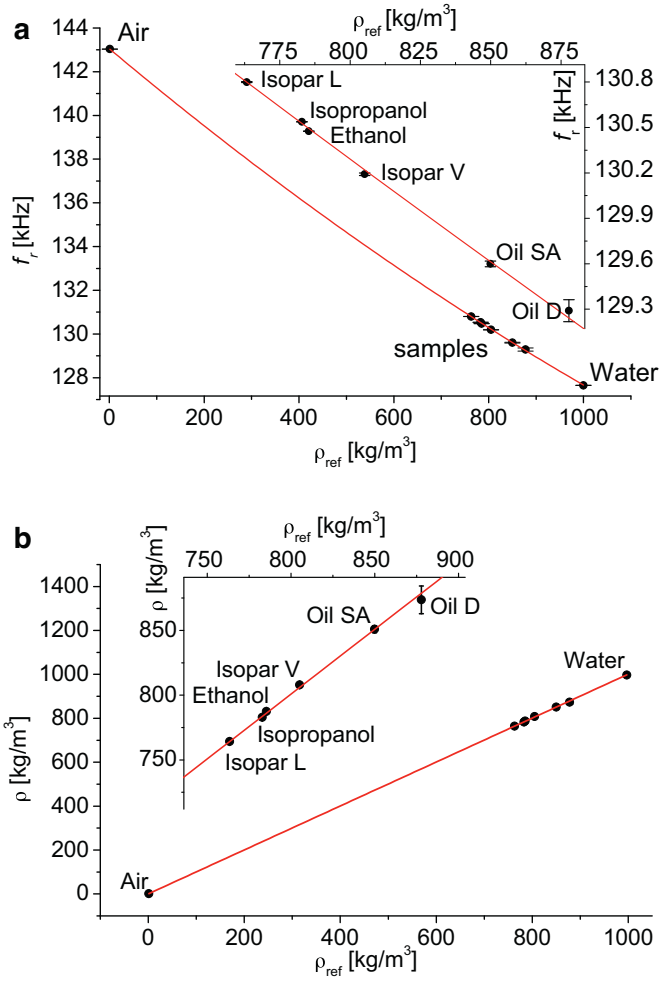

Fig. 3. Mass density measurements of different samples at 24.59 ± 0.001 °C. (a) Measured SMR resonance frequency f_r for different liquids. The red line is the calibration fit of (3) for the measurements of water and air. (b) Correlation plot of the mass density ρ based on (4) compared to the reference density ρ_{ref} measured with a commercial tool at 24.99 ± 0.05 °C (Anton Paar DMA-4100). The red line represents the unity correlation. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

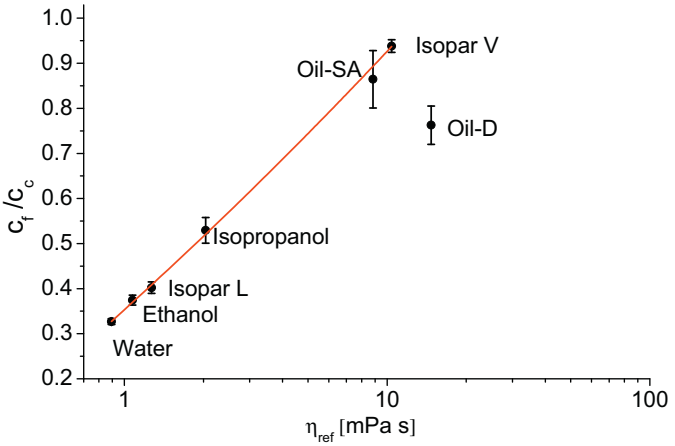

Fig. 4. Ratio of viscous damping of liquid to empty microchannel versus the sample viscosity, calculated based on (9). The measurements were performed at 24.58 ± 0.05 °C. The data was fit with a function $a+bx^c$ where $a = -5.18$, $b = 5.54$ and $c = 0.04$ are the fitting parameters. The sample viscosity η_{ref} was measured with a reference sensor at 25.00 ± 0.005 °C (Anton Paar AMV-200). c_f/c_c was calculated from the quality factor which was in the range of 15,000.

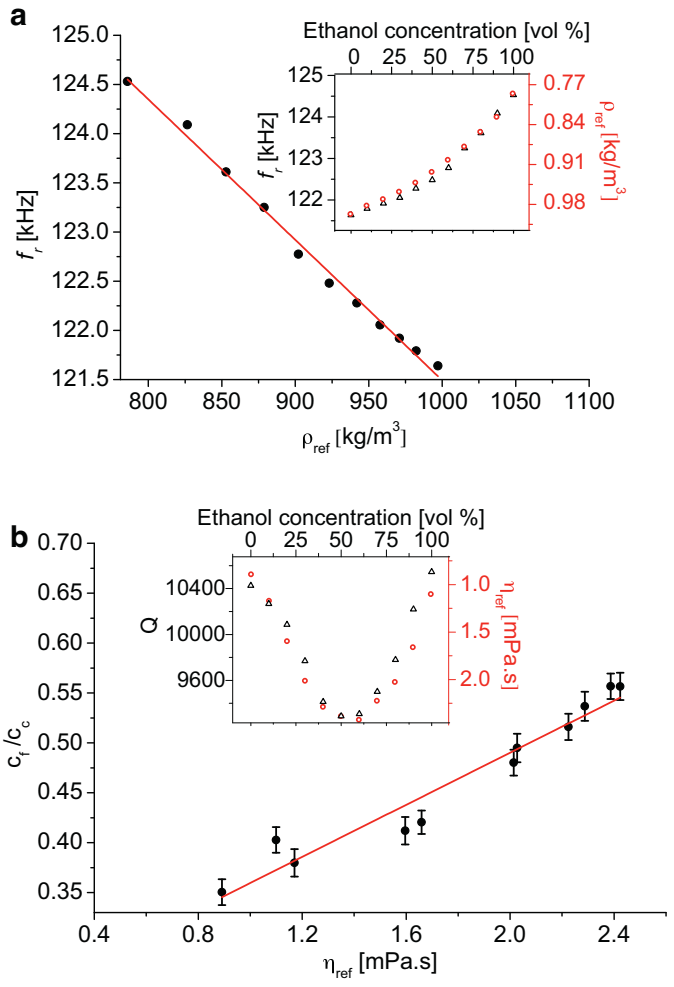

Fig. 5. Characterization of ethanol–water mixtures at 24.73 ± 0.04 °C. The measured values are compared to data taken from [30] measured at 24.85 ± 0.1 °C. (a) Measured resonance frequency compared to mass density. The red line represents a linear fit. The inset shows a comparison of the resonance frequency to the mass density for specific volumetric ethanol concentrations. (b) Ratio of viscous damping coefficient of filled microchannel to empty microchannel (9) versus viscosity. The inset shows the comparison of the measured quality factor to the viscosity for specific ethanol concentrations. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

sample viscosity inside the SMR [23,29]. Our measured increase in damping with increasing viscosity agrees with the finding of Sader et al. which shows that this is the case in the low viscosity regime [29]. The non-monotonic relationship between damping and sample viscosity concludes that viscosity measurement with SMR is only possible in a limited range of viscosity. In Fig. 4 it can be seen that the measured damping values are monotonic with viscosity and can be described by an empirical function. In the limited range, the viscosity of an unknown sample can be extracted using the function. In the given viscosity range an accuracy of 0.025 mPa s (0.45%) can be calculated. This is comparable to the accuracy of 0.5% of the reference sensor. The time needed for a viscosity measurement with the SMR is less than a second. This poses a significant improvement compared to the long measurement times of 3 min required for the reference sensor (rolling-ball viscometer).

In order to further test the accuracy of mass density and viscosity measurements, the SMR was used to test binary mixtures of water and ethanol, as shown in Fig. 5. Eleven mixtures of 99.9% ethanol and MilliQ water were prepared with an increment of 10% volumetric concentrations of ethanol. Fig. 5a shows the measured resonance frequency versus the mass density for all ethanol–water mixtures. The mass density data is reported by Khattab et al. [30]. After the measurement with each binary mixture, the resonance frequency of the SMR was measured to be 13, 7726 ± 2.88 Hz. With a sensitivity of 14.3 Hz/kg/m^3, the resonance frequency of the SMR reduced linearly as a function of the densities of the binary mixtures. This agreed well with the linear relationship (shown in Fig. 3a) between the respective resonance frequency of the SMR and the densities of ethanol and water. The insert shows the comparison of resonance frequency to mass density for the different concentrations. The data was recorded at 24.73 ± 0.04 °C and matched well with the results reported by Khattab et al. [30] measured at 24.85 ± 0.1 °C.

In Fig. 5b, the ratio of viscous damping coefficient of the filled microchannel to the empty microchannel (9) is compared to the viscosity for different ethanol–water concentrations. In the limited viscosity range, the damping viscosity relationship can be described by a linear function. With an accuracy of 0.002 mPa s, the data was simultaneously recorded while measuring the density (described above) of the binary mixtures. The insert shows the direct comparison of the quality factor (Q) to the viscosity for the different concentrations. Due to polar nature of ethanol and water molecules, the total volume of the mixture remains smaller than the sum of their individual volumes. This affects density as well as viscosity of the mixtures. This is evident from the measured data where the Q of the SMR linearly decreased and increased before and after the 50% volume of ethanol in the binary solutions.

6. Conclusion

Development of the SMR was an effort to add a better sensor in the family of other devices of similar specifications. The SMR helped in online characterization of multiple liquid samples. With a density sensitivity of 16 Hz/kg/m^3 we tested different samples ranging from very light solvents to very viscous and sticky crude oil samples. The transparent SMR, measures density from 5 pL volume of a sample with a resolution of 0.01 kg/m^3. The viscosity of the solutions were simultaneously measured with an accuracy of 0.025 mPa s. Results matched well with data achieved from commercial density and viscosity meters. Additionally, the SMR was used to characterize binary solutions of ethanol and water. The device is not only limited to oil and solvents but its unique transparent channels also provide an opportunity to visually analyze different biological species, polymers etc. The compact size of the whole system facilitates using it in arrays for measurements of critical process parameters (CPP) which are considered important in pharmaceutical industry.

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