



# Precision density and viscosity measurement using two cantilevers with different widths



O. Cakmak<sup>a,\*</sup>, E. Ermek<sup>b</sup>, N. Kilinc<sup>c</sup>, G.G. Yaralioglu<sup>d</sup>, H. Urey<sup>b,\*\*</sup>

<sup>a</sup> Department of Mechanical Engineering, Koç University, Istanbul, 34450, Turkey

<sup>b</sup> Department of Electrical and Electronic Engineering, Koç University, Istanbul, 34450, Turkey

<sup>c</sup> Department of Mechatronics Engineering, Nigde University, Nigde, 51245, Turkey

<sup>d</sup> Faculty of Engineering, Ozyegin University, Istanbul, 34794, Turkey

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## ABSTRACT

We introduce a novel method for fast measurement of liquid viscosity and density using two cantilevers with different geometries. Our method can be used for real-time monitoring in lab on chip systems and offer high accuracy for a large range of densities and viscosities. The measurement principle is based on tracking the oscillation frequencies of two cantilevers with a phase-locked loop (PLL) and comparing with reference measurements with a known fluid. A set of equations and a simple algorithm is developed to relate the density and the viscosity to the frequency shifts of the cantilevers. We found that the effect of the density and the viscosity can be well separated if cantilevers have different widths. In the experiments, two Nickel microcantilevers (widths 25  $\mu\text{m}$  and 100  $\mu\text{m}$ , length: 200  $\mu\text{m}$ , thickness: 1.75  $\mu\text{m}$ ) were fully immersed in the liquid and the temperature was controlled. The actuation was using an external electro-coil and the oscillations were monitored using laser Doppler vibrometer. Thus, electrical connections to the cantilevers are not required, enabling measurements also in conductive liquids. The PLL is used to set the phase difference to 90° between the actuator and the sensor. Calibration measurements were performed using glycerol and ethylene glycol solutions with known densities and viscosities. The measurement error with the new method was lower than 3% in density in the range 995–1150  $\text{kg/m}^3$  and 4.6% in viscosity in the range 0.935–4  $\text{mPa}\cdot\text{s}$ . Based on the signal-to-noise ratio, the minimum detectable difference in the viscosity is 1  $\mu\text{Pa}\cdot\text{s}$  and the density is 0.18  $\text{kg/m}^3$ . Further improvements in the range and the accuracy are possible using 3 or more cantilevers with different geometries.

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## 1. Introduction

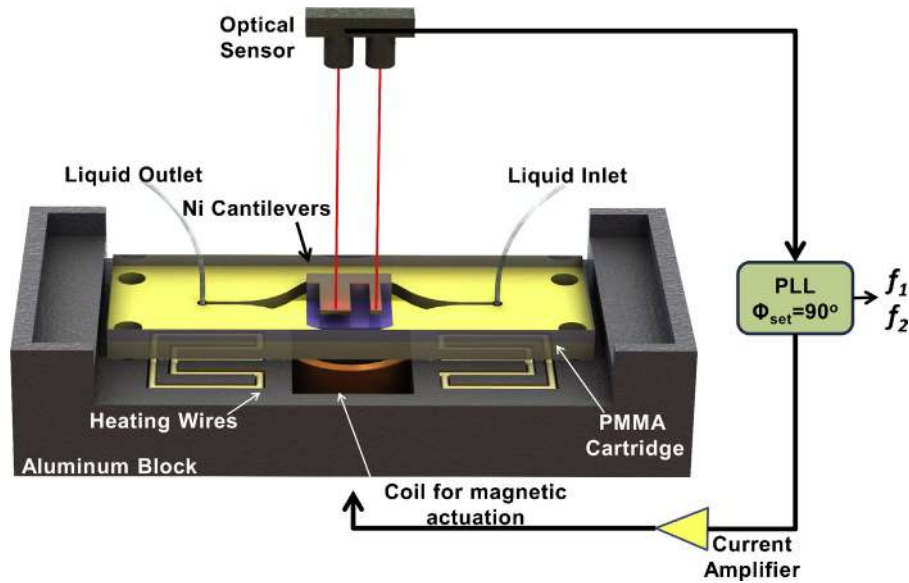
The ability to monitor viscosity and density with low sample volumes is important for various industrial applications and medical applications. Industrial producers of oil and ink use viscosity and density measurements for quality control purposes. In health care, blood viscosity is monitored for early diagnosis of diseases. MEMS based sensors may provide versatile tools meeting the requirements of the large range of applications. Microcantilevers are widely used for viscosity and density sensing applications due to their simple microfabrication [1–10] as well as being well suited for applications where measurements from small volumes are

required. The dynamic behaviour of a cantilever depends on the viscosity and the density of the liquid medium it operates in [1]. Hence, by tracking the resonant frequency and the damping (Quality factor), significant data about the density and viscosity can be gathered. The effects of density and viscosity on both resonant frequency and quality factor are coupled to each other [1–3]. Some groups worked on analytical methods to separately obtain the viscosity and density values from experimental data [3–5]. Although the derived equations are applicable for a large viscosity range, they require precise vibration amplitude measurements, to obtain high accuracy. A general approach to separate viscosity and density effects experimentally is to relate quality factor changes with viscosity and resonant peak shifts with density [6–9]. This approach is also used with other types of MEMS structures like Suspended microchannel resonators (SMR) [11,12] and piezoelectric MEMS resonators [13–16] and suspended microcapillary resonators [17]. With this approach in order to attain high sensitivity in a broad dynamic range, frequency sweeps with high frequency resolution

\* Corresponding author. Tel.: +90 2123381474.

\*\* Corresponding author.

E-mail addresses: [cakmakon@gmail.com](mailto:cakmakon@gmail.com) (O. Cakmak), [hurey@ku.edu.tr](mailto:hurey@ku.edu.tr) (H. Urey).



**Fig. 1.** The schematic representation of the measurement principle. actuation and sensing is conducted with an external electro-coil and laser doppler vibrometer (LDV), respectively. two cantilevers are immersed in the liquid and have different widths (25  $\mu\text{m}$  and 100  $\mu\text{m}$ ) and the same thickness (1.75  $\mu\text{m}$ ) and length (200  $\mu\text{m}$ ).

is necessary. This is generally a slow process and network analyzers should be employed to speed up the process [8]. To the best of our knowledge, in the published literature measuring both the frequency and the quality factor is necessary to separate the effect of viscosity and density.

In our previous works we utilized nickel cantilevers to measure viscosity of blood plasma and serum [18,19]. We achieved an accuracy of 6% and a viscosity sensitivity of 0.01 mPa.s but the density measurement was not possible and it was measured with another equipment. Our magnetic actuation and optical read-out scheme enables simultaneous monitoring of different assays in parallel channels [20,21].

In this paper, we introduce a novel empirical method for simultaneous measurement of viscosity and density by using cantilevers with different widths. The resonant frequency and quality factor is dependent on the hydrodynamic force acting on the cantilever. This hydrodynamic force is a coupled function of both viscosity and density. The effect of viscosity and density will differ depending on the cantilever geometries [4,22]. We fabricated cantilever designs with varying widths while keeping the length and the thickness same. Calibration liquids with water solutions of ethylene glycol and glycerol were prepared [6,7]. We use a commercial PLL to track the frequency at constant phase, record the frequency change in these different liquids with respect to DI water. For each cantilever design the same procedure is repeated and calibration constants are determined. After the calibration step, the viscosities and densities of different solutions are determined with the proposed method and the results are compared with reference measurements.

## 2. Materials and methods

### 2.1. Measurement principle and fabrication

Fig. 1 shows the schematic representation of the measurement setup. The nickel cantilevers are subjected to an AC magnetic field provided by an external electro-coil. Two permanent magnets are placed to right and left sides of the coil to generate DC magnetic field and saturate magnetization of the nickel cantilevers. (Not visible in Fig. 1) [19]. The read-out is conducted with a LDV. This optical read-out scheme can be altered with an interferometric read-out for easier packaging and multiplexing capability which

was shown in our earlier studies [18,20]. The PLL module of the commercial lock-in amplifier (Zurich Instruments HF2LI) is utilized for frequency tracking. The nickel cantilevers are fabricated with a simple one-mask microfabrication process [18,23]. After the chips are fabricated they are placed inside a PMMA channel [18,21]. This channel is consisting of three different PMMA layers. The bottom layer houses the chip; channel geometry is patterned into the mid-layer by laser cutting and the top layer is used a lid. The thickness of each layer is 1 mm. A circular geometry is required to obtain a perfect liquid exchange and avoid liquid residues from the previous sample. The chips are bonded to the bottom layer by double sided tape. Finally, all of the layers are bonded to each other by using chloroform. A temperature controller with 0.1  $^{\circ}\text{C}$  precision is used [21]. The sample volume required to make measurement is 100  $\mu\text{l}$  and it can be further reduced to 10  $\mu\text{l}$  with optimized chip and channel design.

### 2.2. Theoretical background

When operating in a liquid medium a hydrodynamic force is applied to the cantilever. This hydrodynamic force per unit length,  $F_{\text{hydro}}$ , includes two separate terms [4]:

$$F_{\text{hydro}} = -g_1 \dot{\omega} - g_2 \ddot{\omega} \quad (1)$$

$\omega = \omega(x,t)$  is the deflection function of the beam axis. According to Sader's theory [1], the liquid mass accelerated with the cantilever vibration can be assumed as a cylinder with mass per length equal to. The and parameters can be expressed as [5]:

$$g_1 = M_L(2\pi f_L) \left( b_1 \frac{\delta}{w} + b_2 \left( \frac{\delta}{w} \right)^2 \right) \quad (2)$$

**Table 1**

Densities and viscosities of the reference solutions for calibration experiments conducted at 23  $^{\circ}\text{C}$ .

Solution	Density (kg/m <sup>3</sup> )	Viscosity (mPa.s)
DI H <sub>2</sub> O	997	0.935
G8.6 (8.6% glycerol)	1021	1.175
E10 (10% ethylene glycol)	1010	1.175
G5 (5% glycerol)	1010	1.060

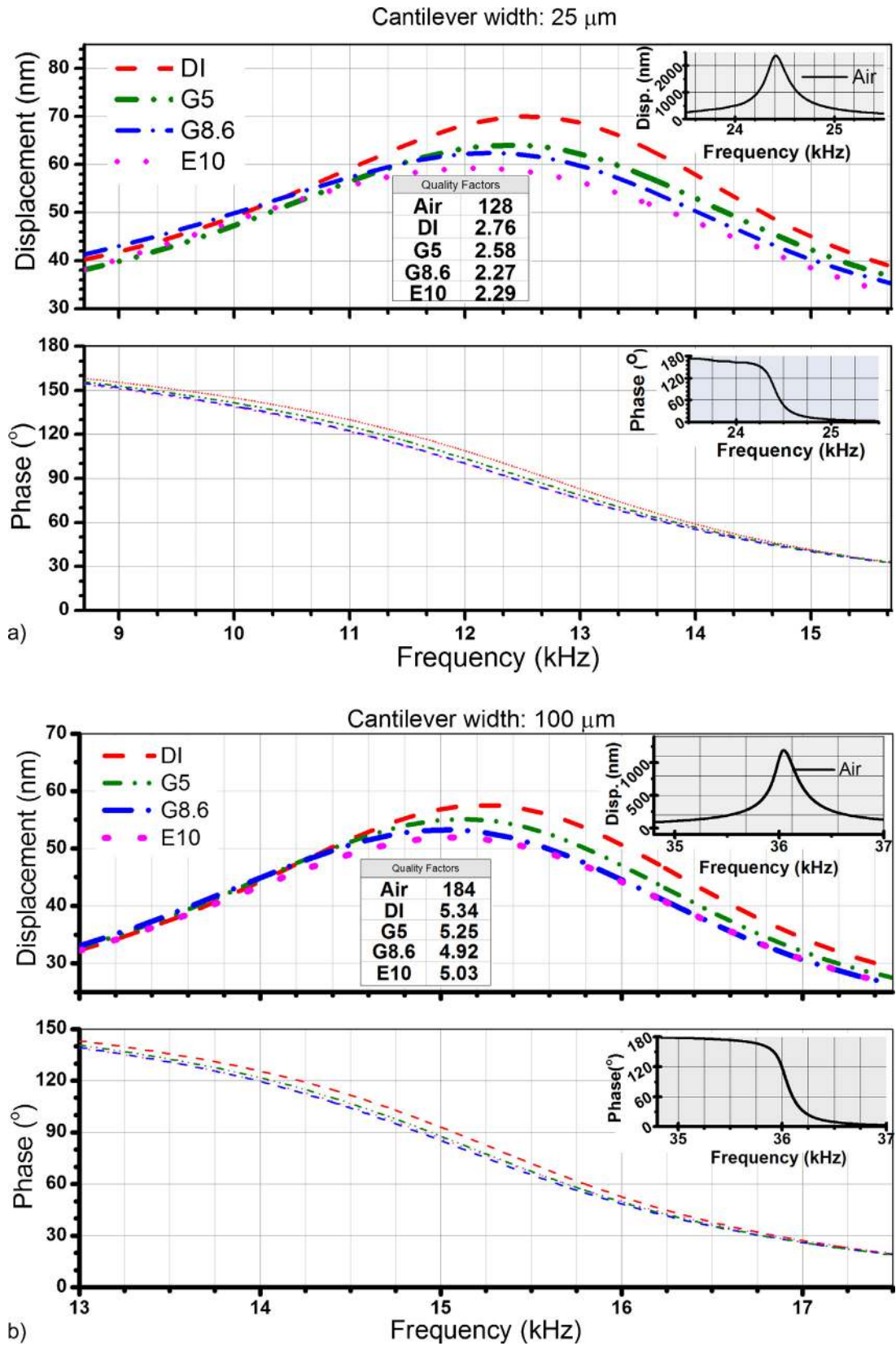


Fig. 2. Amplitude and phase spectrums of cantilevers with 25  $\mu\text{m}$  (a) and 100  $\mu\text{m}$  (b) widths in Air, DI water, G5, G8.6 and E10. PLL is set to 90° phase difference.

$$g_2 = M_L \left( a_1 + a_2 \frac{\delta}{w} \right) \quad (3)$$

where  $w$  is the width of the cantilever,  $f_L$  is the damped resonant frequency of the cantilever in liquid.  $a_1 = 1.0553$ ,  $a_2 = 3.7997$ ,  $b_1 = 3.8018$ , and  $b_2 = 2.7364$  are dimensionless “Maali’s parameters” for an infinitely thin beam [2].  $M_L = \pi \rho_f w^2/4$ , where  $\rho_f$  is the fluid density. The viscous layer,  $\delta$  is the fluid layer thickness where the fluid velocity drops by a factor of  $1/e$ .  $\delta$  can be expressed using the viscosity of the fluid ( $\mu$ ), as:

$$\delta = \sqrt{\frac{2\mu}{\rho_f(2\pi f_L)}} \quad (4)$$

The resonant frequency and Quality Factor (Q) with the hydrodynamic force effects in liquid can be expressed as:

$$f_L = f_0 \frac{1}{\sqrt{1 + Lg_2/m}} \sqrt{1 - \frac{1}{2Q^2}} \quad (5)$$

$$Q = 2\pi f_0 \sqrt{\frac{1 + Lg_2/m1}{Lg_1/m}} \quad (6)$$

$L$  and  $m$  are the length and mass of the cantilever respectively, and  $f_0$  is the undamped resonant frequency in vacuum, which is very close to the value in air. As can be seen from Eqs. (2) and (3), the ratio between the viscous layer,  $\delta$  and the cantilever width,  $w$  is a critical parameter. The effect of viscosity and density on resonant frequencies of cantilevers with different widths will significantly vary. According to this theory, when the density is constant the effect of viscosity change on the resonant frequency will be lower for wider cantilevers. Whereas for the constant viscosity case the added mass term will be dominant, which is highly dependent on the density changes. In the light of this assumption we define a frequency change as a function of viscosity,  $\mu$  and density,  $\rho$ :

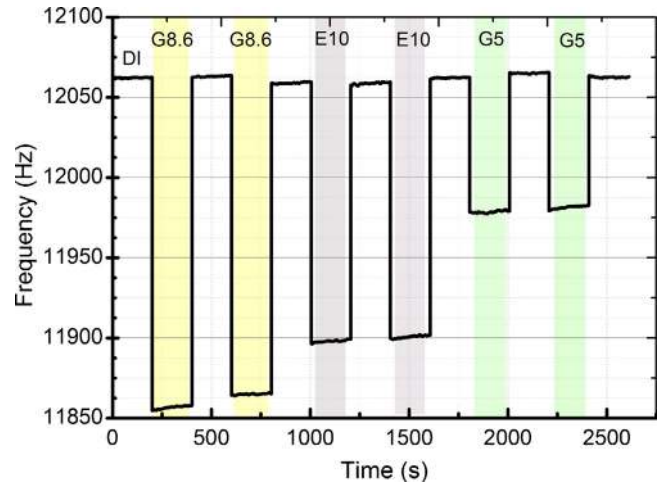
$$\Delta f_i(\mu, \rho) = a_i \mu + b_i \rho + C_i \quad (7)$$

The  $i$  indices show the cantilever design number associated with the width of the cantilever. The  $\Delta f$  represents the percent frequency change with respect to the frequency in DI water. The coefficients  $a_i$ ,  $b_i$  and  $C_i$  are obtained from the calibration measurements.

### 2.3. Calibration of the sensor

Special solutions with the same viscosity but different densities and the same density but different viscosities are prepared for this calibration step similar to the solutions used in recent studies [6,7,24–26]. This step should be conducted for only one time for each sensor geometry. After this one-time calibration only DI water is sufficient as a reference liquid. Glycerol and Ethylene Glycol solutions are used to obtain the viscosity and density solutions. The viscosities of these liquids were measured with Anton PAAR AMVn commercial viscometer whereas, the densities were measured using Anton PAAR DMA 4500M density meter. The viscosity and densities of these calibration liquids are shown in Table 1.

The amplitude and phase spectrums of different cantilevers in these calibration liquids are shown in Fig. 2. As expected the cantilever with 25  $\mu\text{m}$  width has a lower Q, which means that it is less immune to viscous damping. Also the pure density difference, which is the difference between the liquids G8.6 and E10, is more effective on 100  $\mu\text{m}$  width cantilever. However it is not easy to observe these differences clearly and practically with a frequency sweep. It is clear that to distinguish the resonant peaks a frequency sweep with very high frequency resolution is required [6,8]. Furthermore, calculating the Q-factor accurately requires a frequency sweep covering a broad range due to large damping. It should be



**Fig. 3.** The real time plot of a calibration experiment. The frequency in DI water is 12,060 Hz. The absolute frequency changes for G8.6, E10 and G5 are 200 Hz, 159 Hz and 83 Hz. The standard deviations of the frequencies at the last 100 s are less than 0.65 Hz for every liquid. The  $a_1$ ,  $a_2$ ,  $b_1$ ,  $b_2$  and  $C_1$ ,  $C_2$  values are 0.016, 0.033, 7.741, 3.409 and 23.321, 36.119, respectively.

carefully noted that we do not use these amplitude and phase spectrums in Fig. 2 to obtain calibration coefficients.

In this study, instead of conducting high resolution frequency sweeps we tracked the frequency at a certain phase with the PLL. The phase of the PLL was set to  $90^\circ$  for all the cantilevers. The experimental procedure is as follows; at first we introduced DI water in the microfluidic channel and recorded the frequency at  $90^\circ$  phase difference. After taking data for 200 s, the DI water was drained and the next sample was introduced into the channel. The frequency was again recorded for 200 s. In the last 100 s of this period the frequency got stabilized and the standard deviation of the frequency was less than 1 Hz for all cantilevers, which defines the noise floor of the system. Subsequently the sample was drained and DI water was introduced again. The data monitoring interval (200 s) can be reduced with more optimized PLL parameters for specific viscosity and density intervals. This procedure is repeated two times for each calibration solution. A real time plot of one experiment is shown in Fig. 3. The difference between the frequency in DI water and frequency in the sample liquid is divided to the frequency in DI water and the percent frequency change ( $\Delta f$ ) is calculated. The same procedure is repeated three times for each cantilever design and the calibration curves are obtained. All the experiments are conducted at  $23 \pm 0.1^\circ \text{C}$ .

By using the viscosity and density values for the calibration liquids shown in Table 1 and conducting the experiments for cantilevers with different width, the coefficients  $a_i$ ,  $b_i$  and  $C_i$  in Eq. (7) can be determined. Then the frequency changes of the two cantilevers with respect to the frequencies in DI are measured ( $\Delta f_1$  and  $\Delta f_2$ ) and inserted into the Eq. (8) below to solve for the unknown viscosity and density ( $\mu, \rho$ ) values:

$$\begin{Bmatrix} \mu \\ \rho \end{Bmatrix} = \begin{bmatrix} a_1 & b_1 \\ a_2 & b_2 \end{bmatrix}^{-1} \begin{Bmatrix} \Delta f_1 & -C_1 \\ \Delta f_2 & -C_2 \end{Bmatrix} \quad (8)$$

As shown in Eqs. (7) and (8), the approach is based on the linear relation between viscosity and frequency change. When the viscosity is further increased a quadratic relation occurs. Hence we added a  $\mu^2$  term to the right hand side of Eq. (7) and used a quadratic fit to achieve the new coefficients. For the viscosity range 0.6 to 2 mPa.s the linear assumption is used, whereas for the 2 mPa.s–10 mPa.s range the quadratic assumption is used. At first we tested our

system with two cantilevers (with widths 25  $\mu\text{m}$  and 100  $\mu\text{m}$ ). A third cantilever with 50  $\mu\text{m}$  is also calibrated and added to the matrix for further improvement in accuracy and reliability.

### 3. Results and discussion

To investigate the accuracy of the proposed method, water glycerol solutions G5.2 (5.2%), G13 (13%), G17 (17%), G25 (25%), G43 (43%) and G60 (60%) and water ethylene glycol solution E6 (6%) are prepared. All the experiments are conducted at  $23 \pm 0.1^\circ\text{C}$ . Also DI water at  $35 \pm 0.1^\circ\text{C}$  is used as another test liquid having low viscosity. Fig. 4 shows the graphical comparison of the results obtained with the proposed method using two cantilevers (25  $\mu\text{m}$  and 100  $\mu\text{m}$ ) and reference measurements.

Table 2 and Table 3 shows all the results, standard deviations and the accuracy of the proposed method for density and viscosity, respectively. Using three cantilevers (25  $\mu\text{m}$ , 50  $\mu\text{m}$  and 100  $\mu\text{m}$ ) instead of two (25  $\mu\text{m}$  and 100  $\mu\text{m}$ ) slightly improves the standard deviations which correspond to higher reliability. The accuracy of the density measurements is better than 3% for the density range 997–1150  $\text{kg/m}^3$  which is comparable with the recent literature with the microcantilever based sensors [8,10] and other MEMS devices [14] based on frequency sweep techniques. The accuracy of the viscosity measurements is better than 4.6% for 0.935–4  $\text{mPa}\cdot\text{s}$  range which is comparable [8,14] or better than [7] the published literature. The geometrical imperfections due to microfabrication (undercut and residual stress during electroplating), liquid exchange errors and local temperature instabilities due to laser heating are the main error sources. These errors can be reduced with further improvements on measurement setup and microfabrication processes. Another important point is when the viscosity is further increased to 9.99  $\text{mPa}\cdot\text{s}$  the accuracy in viscosity measurements decreases significantly. This can be explained with the dramatic change in the phase curves for the high viscosity liquids which results in a saturation [18]. The minimum detectable viscosity and density change can be calculated from the noise floor

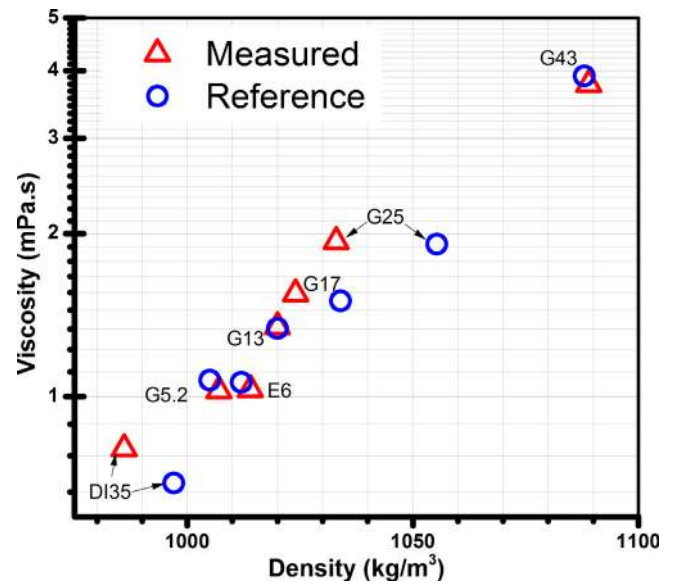


Fig. 4. The graphical comparison of measured and reference values of test liquids. The reference viscosity measurements are conducted with Anton Paar AMVn capillary viscometer. The reference density measurements of DI35, G5.2, E6 are conducted with Anton Paar DMA 4500M density meter which provides a standard deviation less than  $1 \times 10^{-1} \text{ kg/m}^3$ . The reference density measurements of other solutions are conducted with high precision balance and micropipettes.

of the frequency measurement (0.65 Hz) and the frequency difference between calibration liquids. Based on the signal-to-noise ratio, the minimum detectable differences in the viscosity and the density are 1  $\mu\text{Pa}\cdot\text{s}$  and 0.18  $\text{kg/m}^3$  respectively which is comparable with the recent work which reports 0.05  $\text{kg/m}^3$  minimum detectable density using microcapillary resonators [17].

Table 2

Viscosity results obtained from reference measurements and the proposed method. The standard deviation of the reference measurements are less than  $1 \times 10^{-4} \text{ mPa}\cdot\text{s}$ . Error is the percent difference between the reference and measured values.

Density	Reference	2 Cantilevers		3 Cantilevers	
		Mean(standard deviation)[ $\text{kg/m}^3$ ]	Mean(standard deviation)[ $\text{kg/m}^3$ ]	Error(%)	Mean(standard deviation)[ $\text{kg/m}^3$ ]
DI35	994	986 (0.3)	0.8	995 (0.6)	0.1
G5.2	1012	1014 (0.5)	0.2	1016 (0.8)	0.4
E6	1005	1007 (0.6)	0.2	1011 (0.4)	0.6
G13	1020 (13.8)	1020 (6.8)	0.0	1017 (4.5)	0.3
G17	1034 (23.4)	1024 (6.1)	0.9	1018 (4.4)	1.5
G25	1055 (11.2)	1033 (13.3)	2.1	1026 (6.0)	2.8
G43	1088 (14.3)	1089 (9.5)	0.1	1086 (8.0)	0.2
G60	1144 (16.4)	1178 (9.0)	2.9	1175 (10)	2.7

Table 3

Density results obtained from reference measurements and the proposed method. The standard deviation of 6 different measurements is shown. Error is the percent difference between the reference and measured values.

Viscosity	Reference	2 Cantilevers		3 Cantilevers	
		Mean[ $\text{mPa}\cdot\text{s}$ ]	Mean(standard deviation)[ $\text{mPa}\cdot\text{s}$ ]	Error(%)	Mean(standard deviation)[ $\text{mPa}\cdot\text{s}$ ]
DI35	0.720	0.801 (0.001)	11.2	0.793 (0.001)	10.1
G5.2	1.062	1.031 (0.006)	2.9	1.029 (0.006)	3.1
E6	1.072	1.025 (0.010)	4.3	1.022 (0.010)	4.6
G13	1.336	1.347(0.007)	0.8	1.350 (0.005)	1.0
G17	1.502	1.548 (0.007)	3.0	1.553 (0.009)	3.4
G25	1.911	1.934 (0.016)	1.2	1.940 (0.022)	1.5
G43	3.910	3.773 (0.242)	3.5	3.785 (0.234)	3.2
G60	9.990	5.660 (0.232)	43.3	5.670 (0.236)	43.2

#### 4. Conclusions

A novel method to obtain viscosity and density separately with two (or more) cantilevers is developed. The frequency shifts due to viscosity and density changes are used in the calculation. The PLL-based frequency tracking enables fast and accurate detection which makes the system more practical compared to frequency sweep based methods using network analysers. An important point to highlight is the achieved accuracies for viscosity and density are well suited for blood and blood plasma measurements [13,19]. Within the scope of this study we focused on proving the capability of the method to extract density and viscosity and did not force to achieve higher accuracy in a broader dynamic range. It is obvious that one can still employ our methodology to achieve higher accuracy and dynamic range by either choosing cantilevers which are optimized for the intended range or increasing the number of cantilever designs. Also the remote nature of actuation and sensing makes the system suitable for usage in single use and disposable cartridges, which is required for point-of-care applications.

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#### Biographies



**Onur Cakmak** received his B.Sc. degree in Mechanical Engineering from Istanbul Technical University and M.Sc. degree on Vibration Engineering from the same University in 2010. Between 2008 and 2010 he worked as an engineer in vibration & acoustics group at BEKO R&D Center, Istanbul, Turkey. Since May 2011, he is a PhD Candidate at Optical Microsystems Laboratory at Koç University, Turkey. He designed, fabricated and characterized MEMS based Point-of-Care device for blood analysis. He developed PMMA based microchannels to achieve multiplexed coagulation time and viscosity measurements from whole blood and blood plasma. He is also taking part in the commercialization of MEMS based coagulometer for PT and a PTT tests. His research interests are microcantilever based sensors, Lab-on-a-Chip systems, microfluidics and BioMEMS.



**Erhan Ermek** received his M.Sc. degree in Department of Biomedical Engineering from Baskent University in 2010. He worked as a senior research engineer in Optical Microsystems Research Laboratory at Electrical Engineering Department, Koç University, Istanbul between 2010 and 2015. He is currently working as a senior research engineer at Department of Mechanical Engineering, Koç University. His research interests are: mass sensitive transducers such as cantilever and QCM, biosensors.



**Dr. Necmettin Kilinc** received the B.Sc. degree from Marmara University, Istanbul, in 2003, and M.Sc. and PhD. degrees from Gebze Institute of Technology in 2006 and 2012, all in Physics, respectively. After his PhD, he started to postdoc at Optical Microsystems Laboratory, Koc University to research cantilever based biosensors. He is an assistant professor at Nigde University, Mechatronics Engineering Department, Nigde, Turkey. His research interests are fabrication of nanostructures and thin films of metaloxides and organic materials and structural and electrical properties of these materials and using these materials for bio-chemical sensor applications.



**Dr. Gökse G. Yaraloğlu** received his PhD degree from Electrical and Electronics Engineering Department, Bilkent University in 1999. Between 1999 and 2006, he worked as an Engineering Research Associate at Stanford University. During his post-doc, he worked on Micro electromechanical systems (MEMS), ultrasound, material characterization and Atomic Force Microscopy. After his post-doc studies, he joined a start-up company where he worked as a MEMS design engineer. In 2011, he joined Ozyegin University. Currently, he is an associate professor at the Electrical and Electronics Engineering Department, Ozyegin University.



**Dr. Hakan Urey** received the B.Sc. degree from Middle East Technical University, Ankara, in 1992, and M.Sc. and PhD. degrees from Georgia Institute of Technology in 1996 and in 1997, all in Electrical Engineering. After completing his PhD, he joined Microvision Inc.-Seattle as Research Engineer and he played a key role in the development of the Retinal Scanning Display technology. He was the Principal System Engineer when he left Microvision to join the faculty of engineering at Koç University in 2001. He was promoted to Associate Professor in 2007 and Professor in 2010. He published about 50 journals and 100 international conference papers, 7 edited books, 4 book chapters, and has more than 25 issued and several pending patents.

His research interests are in the area of optical MEMS, micro-optics and optical system design, 2D/3D display and imaging systems, and biosensors. He is a member of SPIE, IEEE, and OSA. He received an Advanced grant from the European Research Council (ERC-AdG) in 2013, Outstanding Faculty Award from Koç University in 2013, TÜBİTAK-Encouragement Award in 2009, Outstanding Young Scientist Award from the Turkish Academy of Sciences (TÜBA) in 2007, and Werner Von Siemens Excellence Award in 2006.