



Quartz tuning fork, a low-cost orthogonal measurement tool for the characterization of low-volume liquid reagents

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ABSTRACT

Generally, expensive specimens that consume reagents in lesser quantities are characterized for use in multiple industries, specifically in those related to life sciences. In this study, we present quartz tuning fork (QTF) as a highly desirable sensor, which can perform the characterization of three liquid properties with a consumption of as low as 1 μ L. By simultaneously recording the resonance frequency, amplitude, and quality factor, we established trends between the different properties (density, viscosity, and concentration) of the liquids. Additionally, simple mathematical functions are also presented that enable the determination of one of the three properties of unknown liquid samples by simple calculations. The proposed QTF-based sensor was successful in the characterization of expensive liquid reagents and the results presented in this work clearly demonstrate the potential of using QTFs as sensing platforms in a wide range of applications including biomedical diagnosis, petrochemical analysis, and environmental surveillance.

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1. Introduction

Recent advances in micromechanical sensors have stimulated substantial interest in the development of novel and ultrasensitive sensors for use in the detection of a wide range of chemical, physical, and biological phenomena [1–3]. In particular, the detection of gases, chemical vapors, and heavy metal ions that impose serious health issues on both humans and the environment has been under intense investigation [4–6]. Surface acoustic wave, quartz crystal microbalance (QCM), and microcantilever sensors are some of the tools used in the sensing technology that have shown excellent accuracy [7–9]. The working principles of these methods rely on detecting the change in the resonance frequency in response to the interaction between the analyte molecules on the surface. Moreover, industries related to life sciences are continuously conducting research and development for the miniaturization of such tools that can ensure low volume consumptions. For applications in genetics, protein characterization, and DNA melting, owing to the high cost of reagents, there is a high demand for technologies that can measure smaller volumes in the range of micro and nanoliters [10,11]. In the last two decades, multiple research groups

have reported the development of various microfluidic platforms that ensured low volume consumption of reagents with high accuracy [12–15]. Thickness shear mode (TSM) resonators have also been used to measure viscosity of solutions at low volume [16,17]. Although these miniaturized sensors have high mechanical stability and small size, their frequency is the range of MHz making it difficult to compare their results with sensing systems operating in low-frequency regime [18]. Being operated in low frequency regime, QTF resonators are more suitable for measurements involving complex and non-Newtonian liquids but it is less resilient in a given fluid than high frequency resonators such as TSM [19].

Besides chemical and biological applications, research efforts have also been directed toward using sensing technology for the measurement of various properties in liquids such as density, viscosity, melting, and evaporation [13,19–21]. Among these properties, viscosity is one of the fluid characteristics that is considered important in multiple industries. In pharmaceuticals, the viscosity of different components defines the shelf life of a drug. In healthcare, the viscosity of plasma may directly relate to blood flow conditions, which may help physicians in the prediction of heart attacks. In the hydrocarbon industry, the viscosity of crude oil helps the decision makers to add appropriate quantities of diluents, which is directly related to the economics of the industry and the oil flow in a pipeline [22].

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Despite the impressive advancements in sensing systems, there is still a need for the development of more adept micromechanical sensors that can meet the ongoing environmental, clinical, and industrial demands of miniaturized, inexpensive, reliable, and ultrasensitive micromechanical sensors. An alternative sensing tool that has recently gained popularity for this purpose is the quartz tuning fork (QTF). Its appealing characteristics such as low cost (\$5), high thermal and mechanical stability, fast response time, and simple operating electronics makes it a potential candidate to meet the requirements of novel chemical sensors mentioned above. A typical QTF consists of two vibrating prongs and has a high-quality factor (of approximately 10,000 in air) [23]. Owing to its low power consumption and high stability, it has been used as a reliable timing tool in small devices such as watches [24]. In addition, the use of QTFs in humidity, temperature, and gas sensing has been reported [25,26]. One of the earliest studies on the use of QTFs in sensing measurements was conducted by Bous-saad et al., who demonstrated that the adsorption of organic vapors on a polymer wire attached between the two prongs of the QTF resulted in a change in its resonance frequency [27]. Using a similar concept of attaching a sensitive polymer wire across the two fronts, Ren et al. demonstrated the detection of water, ethylnitrobenzene, and ethanol vapors using an array of QTFs [24]. In a recent study presented by Kim et al., electrospun PMMA wires placed across the tides of QTFs were used as a moisture sensor and enhanced sensitivity was achieved by increasing the number of PMMA wires [28]. The majority of previous studies that used QTFs as micromechanical sensors focused on attaching a polymer wire between the two prongs of the QTF and then measuring its resonance frequency upon the adsorption of gas molecules. QTF has also been demonstrated as a density/viscosity sensor but researchers had to limit its application to nonconductive liquids where the complete sensor was immersed into the liquid container [29].

However, the use of such methods for detection in conductive liquid environments has not yet been reported. The limitations associated with the latest highly sensitive but costly technologies (cantilever sensors and QCM) emphasize the need of adopting alternative tools such as QTFs that require relatively simpler instrumentations and obtain a higher quality factor (Q) value even in ambient conditions. In this study, we explored the orthogonality (simultaneous measurements of multiple parameters) of this low-cost sensor that can characterize conductive liquids with a droplet, which is much smaller than the overall volume of the sensor itself. In this work, we employed the sensor to systematically measure the density, viscosity, and concentration by recording the resonance frequency, quality factor, and vibration amplitude (at resonance), simultaneously. In this method, the two prongs of the QTF were immersed into 1 μ L droplet of the 11 solutions of water/glycerol mixtures. In response to this immersion, the resonance frequency of the QTF decreased owing to the change in surrounding media. To examine the effectiveness of this QTF sensing system, the resonance frequency change of QTF as a function of the properties of various water/glycerol mixtures was also investigated. The approach of taking a measurement on a droplet would make the present QTF system a feasible sensor in bio applications where the reagents are quite rare and thus, expensive.

2. Experimental details

2.1. Instrument

The measurements were performed using a commercial instrument, Quester Q10 (Fourien Inc., Canada). The instrument consists of an integrated impedance analyzer that can perform frequency

sweep at different amplitudes and measure the QTF's impedance response (real and imaginary components) at a rate of 0.5 million samples per second. Finally, the measured data is reported in a comma-separated value format, which can later be processed in various data analysis software such as MATLAB, Origin Lab, and Python. Additionally, the instrument can resonate the QTF at a fixed frequency. Using a technique called proportional integrated differential (PID), the integrated heater helps to maintain the temperature of the liquid sample.

2.2. Method

To perform the experiments with very low volume consumption, the QTF was directly immersed in a 1 μ L droplet placed on a glass slide. To make this study more systematic, 11 solutions of water/glycerol mixtures were prepared with 0% glycerol in Milli-Q water to 99% glycerol (CAS 56-81-5, VWR, USA), with 10% increments in the concentration of glycerol in water. For each experiment, a droplet was dispensed on a glass slide using a standard pipette. Then, using the on-board microscope and a three-axis translation mechanism, the QTF was aligned with the droplet. Next, the QTF was slowly brought near the droplet while continuously observing the live video through the microscope, which was connected to the computer. Fig. 1(a) provides a general schematic of the working of the instrument and the experiment. Internally, the impedance analyzer in the Quester instrument excited the QTF with a specific frequency sweep. Dependent on mass loading, the QTF resonated at a particular resonance frequency, exhibiting a relevant quality factor. The resonance frequency of the QTF depends on the density as well as the viscosity of the media around its two prongs. Viscosity which results from the interactions between fluid molecules with the surface leads to energy dissipation [30]. However, for Newtonian fluids as in fluids used in this study, the viscous friction of the media may be considered constant. After the QTF was immersed into the droplet, the resonance frequency dropped by 63 Hz. To maintain the consistency, the QTF was immersed into the droplet with the same depth of 25 μ m in all 11 samples. The response of the QTF was recorded by the impedance analyzer while the result of the sweep was reported on the computer screen.

Fig. 1(b) presents an image of the experimental setup. The QTF can be moved in x, y, and z-axis to accurately align with a droplet, as shown in the top view in Fig. 1(c). The droplet can be directly placed on the copper plate that has two resistive heaters mounted at the bottom. Alternatively, to avoid cross contamination, the droplets can also be dispensed on a glass slide, which is disposed after every experiment. To avoid foreign contamination through the circulating air, the experimental setup is enclosed in a transparent enclosure, as shown in Fig. 1(d). The QTF used in this experiment (part number QS32, Fourien Inc.) was not fully coated with an electrode. Instead, the free ends of its vibrating prongs were free from the thin metal film electrode (Fig. 1(e)), which enabled the immersion of the sensor in water/glycerol droplets without electrically shortening them.

3. Results and discussion

To test the QTF, it was first immersed in a 1 μ L water droplet. The time duration for each measurement was around four seconds thus there was a minimal chance for the water droplet to change its volume caused by evaporation where all of the measurement were done at consistent conditions of ambient temperature, pressure and humidity Using the software control of the instrument, the frequency was swept from 32.6 to 33 kHz, as shown in Fig. 2 (a). The resonance frequency of the QTF was found at 32.837 kHz

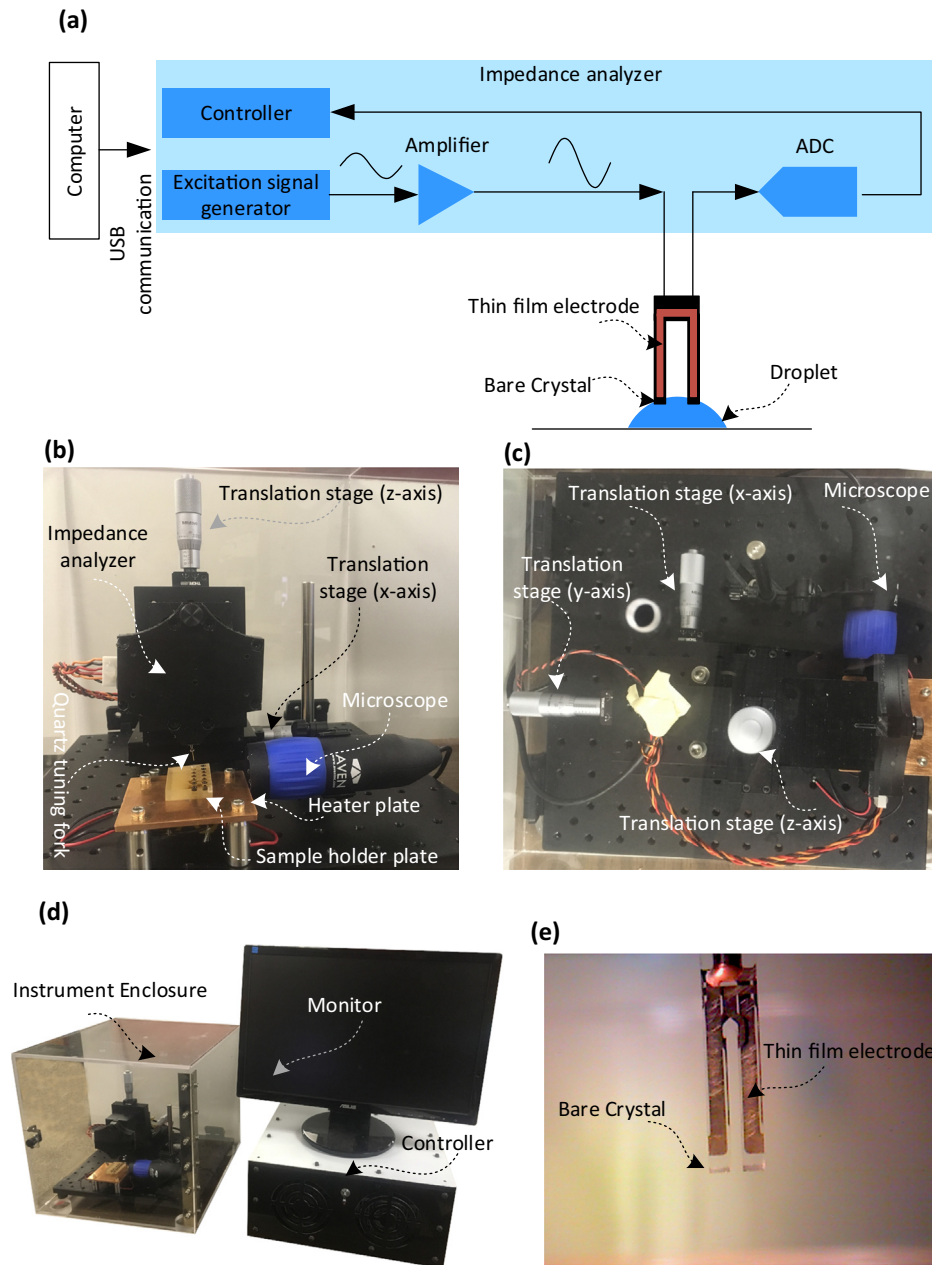


Fig. 1. (a) Experimental setup with the QTF directly immersed in a droplet. (b) Front view of the three-axis translation control for the alignment of QTF with the droplet. (c) Top view of the three-axis translation control. (d) Complete view of the instrument. (e) Close up of the QTF mounted on the holder.

with an impedance value of $2.45 \text{ M}\Omega$. Then, the QTF was lifted upwards and left for 20 min to be dried naturally. The experiment was repeated thrice, as shown in the overlaid data, with three different colors, in Fig. 2(a). To further test the change in the characteristics of the QTF as a function of density and viscosity, it was immersed in a droplet consisting of 50% water and 50% glycerol. The resonance frequency dropped to 32.768 kHz , as shown in the left side of Fig. 2(a). This indicates that the QTF sensed the density of the water/glycerol mixture with a sensitivity of approximately 0.5 Hz/kg/m^3 . Additionally, we observed that the resonance peak became wider, which is an indication that the QTF experienced higher energy dissipation owing to the higher viscosity of the mixture [31]. It is also observed that there is a negative value in the real component of the impedance as can be seen in Fig. 2(a). This negative impedance is attributed to the dominant capacitive

nature of the QTF before approaching the resonance. The capacitive behavior can be attributed to the presence of charge on the QTF surface as well as the liquid samples (under test)[32]. The effect of ambient charge on the QTF's resonance frequency was separately tested on three water samples. The results are presented in Fig. S1 in the Supplementary materials section. Fig. 2(b) shows the imaginary component of the impedance change in the QTF while separately being in water and glycerol droplets.

Next, the QTF was systematically tested for the complete set of water/glycerol mixtures, as shown in Fig. 3(a). First, the QTF was immersed in a Milli-Q water droplet with a volume of $1 \mu\text{L}$, dispensed on a glass slide. As shown in Fig. 2(a), the QTF exhibited a certain resonance frequency as well as quality factor. The resonance frequency was found to be significantly similar to the one observed before. After each measurement, the data were fit using

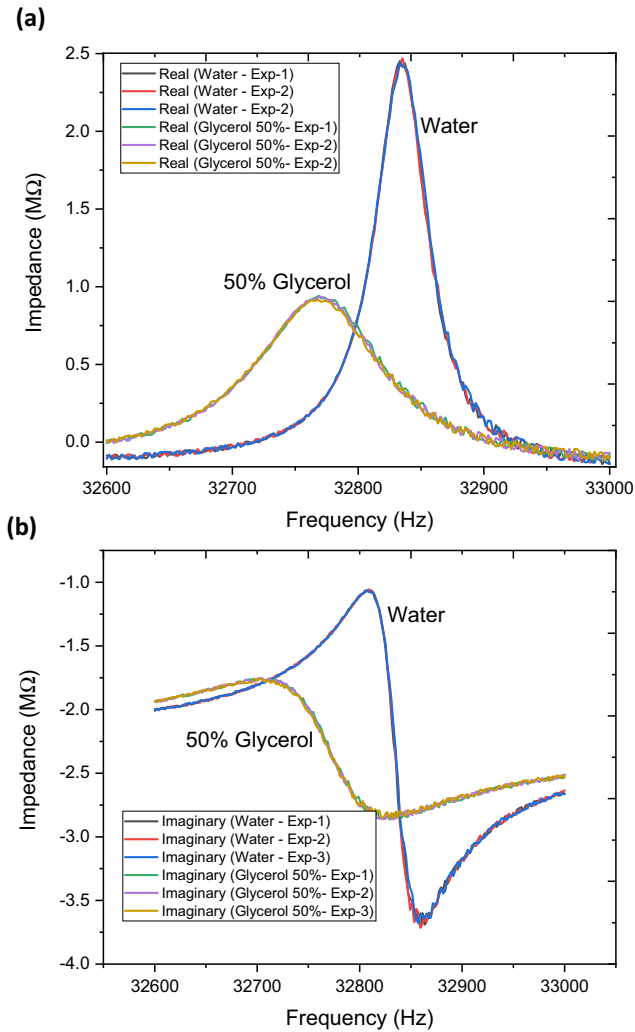


Fig. 2. (a) Real component of impedance change of QTF at resonance as a function of density and viscosity of pure water and glycerol/water mixture. (b) Imaginary component of impedance change of QTF at resonance with water and glycerol/water mixture.

the Lorentz function to calculate the resonance frequency at full width half maximum (FWHM). A fitting example for 60% glycerol solution is shown in Fig. 3(b).

$$y = y_0 + \frac{2A}{\pi} \frac{w}{4(x - x_c)^2 + w^2} \quad (1)$$

Eq. 1 shows the Lorentzian function where y is the real component of impedance plotted on y -axis, y_0 is an offset, A is the area, w is the difference between the values of the resonance frequency at FWHM, and x_c is the value of resonance frequency at maximum amplitude. For the water sample, y_0 , x_c , w , and A were calculated to be -143682 , 32.833 kHz, 51 Hz, and 2 , respectively. From this data, the quality factor was calculated to be 643 using Eq. 2.

$$Q = \frac{x_c}{w} \quad (2)$$

Once the QTF was dried, it was immersed in a droplet consisting of 10% glycerol and 90% water. It should be noted that this did not change the resonance frequency of the QTF. However, the amplitude of its impedance decreased along with a reduction in its quality factor. This demonstrates that the change in density from 0% to 10% glycerol is smaller than the sensitivity of the QTF but the change in the viscosity of the same solution is within the

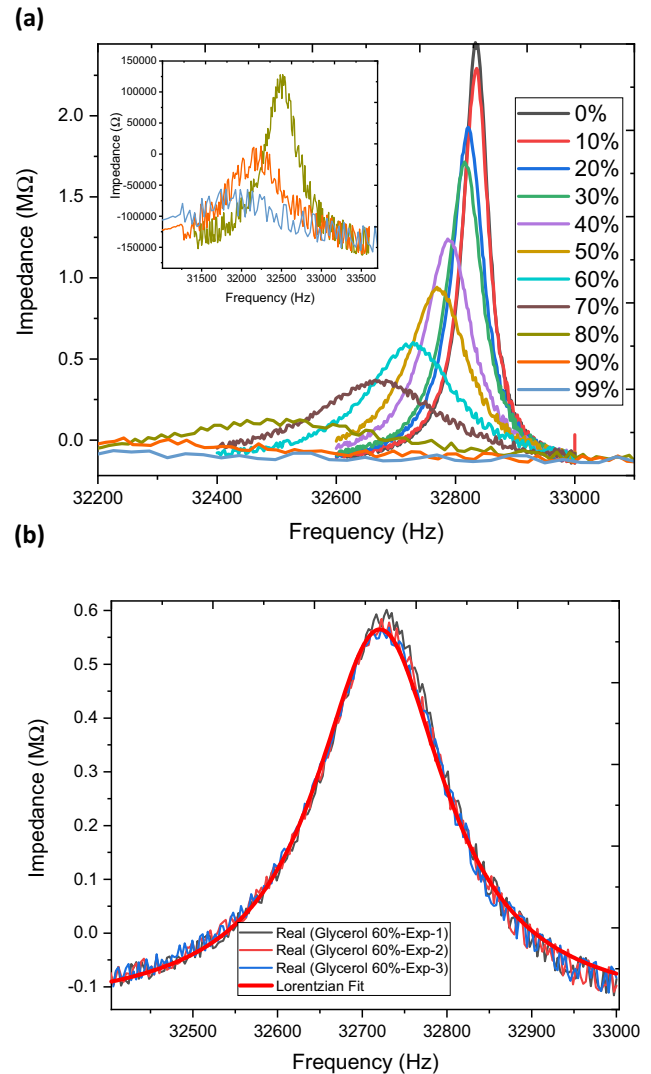


Fig. 3. (a) Resonance frequency change of QTF as a function of the properties of various water/glycerol mixtures. (b) Lorentzian function is fit with the data of 60% glycerol solution.

resolvable range. This is possibly because of energy dissipation which affected the quality factor more than the resonance frequency. The capability of QTF to deliver multiple signals makes it a highly suitable low-cost sensor because if one parameter (e.g. resonance frequency) is not altered owing to the orthogonality of the sensor, other parameters (e.g. amplitude and Q) would be responsive. Thus, it is possible to characterize a liquid reagent in different ways.

Once the 10% glycerol sample was measured, the QTF was immersed in Milli-Q water to remove any glycerol traces, which is important to avoid cross contamination from one sample to another. Next, the 20% glycerol sample was tested. Here, the resonance frequency, resonance amplitude, and quality factor decreased to 32.819 kHz, 512 , and 1.9 MΩ, respectively. For all subsequent measurements, the QTF sensor was rinsed with Milli-Q water in between every sample.

Fig. 3(a) shows that as the concentration of glycerol gradually increased, the three parameters (resonance frequency, resonance amplitude, and quality factor) decreased with different proportions. For concentrations higher than 80%, the amplitude at the resonance became very small. The inset shows a magnified view of the data for glycerol concentrations of 80%, 90%, and 99%.

Another noteworthy change, and possibly a fourth parameter, in the QTF was an increase in the measurement noise as a function of glycerol concentration. This phenomenon was more evident in the glycerol concentrations higher than 50%. This increase in noise was possibly due to the higher energy dissipation as a function of viscosity. Thus, it can also be an important parameter to be observed in detail. However, it is beyond the scope of this study.

Fig. 4 shows the synthesis of all data presented in Fig. 3(a). Various parameters are separately plotted as the functions of different properties of liquid samples, each measured with a volume consumption of 1 μL . Fig. 4(a) shows the quality factor plotted as a function of viscosity of water/glycerol mixtures. The viscosity (reference viscosity) values were acquired from the publically available online source [33] where the physical model developed by by N-S Cheng [34] and Volk and Kähler have been utilized [35]. The data were consistent with the second-order exponential function presented in Eq. 3.

$$y = y_0 + A_1 e^{-ax/t_1} + A_2 e^{-ax/t_2} \quad (3)$$

where y_0 is an offset, A_1 and A_2 are the amplitudes, t_1 and t_2 are the time constants, y is the quality factor, a is a constant and x is the viscosity of the samples. For this specific dataset, the calculated values

are $y_0 = 27.1$, $A_1 = 561.27$, $t_1 = 0.00275$ s, $A_2 = 224.7$, and $t_2 = 0.0354$ s. From these values and Eq. 3, the viscosity was calculated for all values of the quality factor. The inset in Fig. 4(a) shows a plot of calculated and reference viscosity. It is evident that at low viscosity, there is a higher mismatch between the reference and calculated values of the viscosity of the mixtures. This difference can be minimized by improving the fitting parameters of the exponential function.

Fig. 4(b) shows a trend between the resonance frequency and density of the binary mixtures. The data were fit with a first order exponential function, as shown in Eq. 4. The inset shows a linear trend between the density and concentration of glycerol.

$$y = y_0 + A_1 e^{-ax/t_1} \quad (4)$$

where y_0 is an offset, A_1 is the amplitudes, t_1 is the time constants, y is the resonance frequency, a is a constant and x is the density of the sample. Fig. 4(c) presents an exponential relationship between the impedance amplitude and reference viscosity of the mixtures. The inset shows a magnified view of a set of viscosity values. The data were fit with a second-order exponential function, as shown in Eq. 3. Fig. 4(d) shows the relationship between the impedance

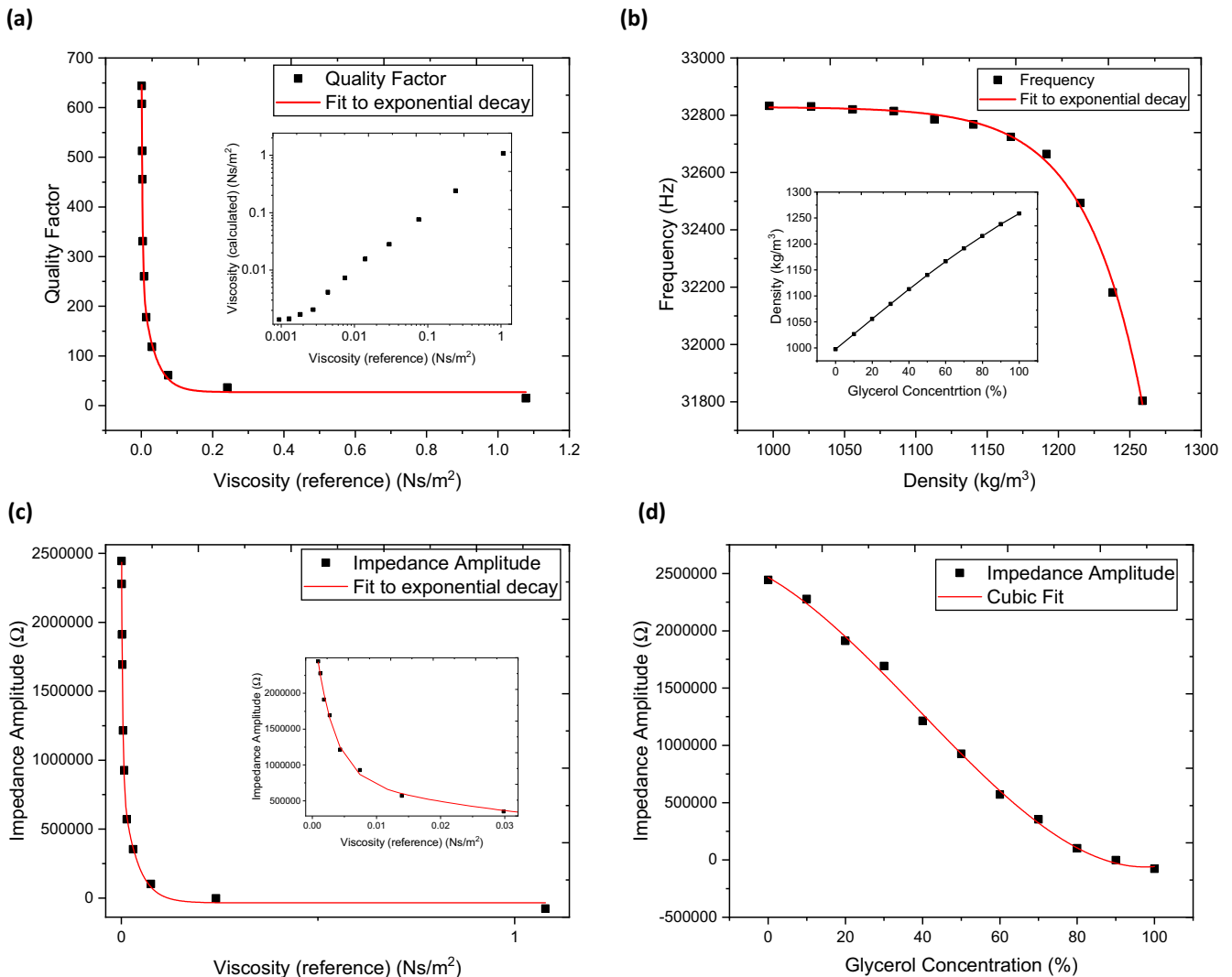


Fig. 4. (a) Exponential relationship between quality factor of QTF and viscosity of water/glycerol mixtures. (b) Exponential relationship between resonance frequency of QTF and density of water/glycerol mixtures. (c) Exponential relationship between impedance amplitude of QTF and viscosity of water/glycerol mixtures. (d) Impedance amplitude versus concentration of the mixtures fitted to a cubic function.

amplitude and concentration of the binary mixtures. Here, the data were fit with a cubic function, as presented in Eq. 5.

$$y = A + Bx + Cx^2 + Dx^3 \quad (4)$$

where A , B , C , and D are the fitting constants with values of 2.47, -19216, -402.9, and 3.42, respectively.

From the data presented in Fig. 4, we can conclude that the orthogonality of the QTF sensor is a very useful quality. Here, we can simultaneously record multiple parameters that help in the extraction of multiple properties (of liquid reagents) such as density, viscosity, and concentration. We also demonstrated that all relationships between the QTF parameters and liquid properties are significantly consistent with the mathematical functions. Therefore, with a certain margin of error, the characterization of unknown liquid samples can be performed.

4. Conclusions

In this work, a QTF was employed to perform the characterization of water/glycerol binary mixtures. During the measurements, the QTF was actuated with a frequency sweep, which helped in simultaneously recording the multiple parameters of the QTF as a response to the sensor. The resonance frequency, quality factor, and amplitude, and their relationships with respective liquid properties are presented in this paper. The sensor could characterize the liquid samples with a volume of as low as $1 \mu\text{L}$. QTF, being a low-cost sensor, can support various applications in several industrial fields. It should be noted that a fourth parameter, noise, may also provide important information about the liquid samples. The results presented herein ascertained the possibility of employing QTFs as sensitive, small, and cost-effective sensors in significant and intriguing applications including medical diagnosis, environmental monitoring, and detection of chemical host-guest interactions. Such employment of QTF sensors require further efforts to miniaturize the instrumentation to better develop QTF-based handheld devices. Furthermore, the QTFs should also be functionalized to target specific molecules, which would also help enhance their sensitivity. Further efforts will also be made to investigate the potential of using our QTF system in the characterization of non-Newtonian fluids such as blood which is a significant aspect in medical and biological applications.

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Declaration of Competing Interest

None.

Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.measurement.2019.107313>.

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