Flow rate influence of the peristaltic-based pumps on the QCM sensor

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Abstract. The liquid injection is one of the crucial roles in the used of QCM sensors in a liquid application. Liquid sample injection should be controlled. The injection of the liquid sample to the sensor surface must be controlled in term of its volume as well as its flow rate. A syringe pump injection system for a single shot sample injection has been developed. However, the system lacked a continuous sample injection. In this study, a peristaltic-based pump has been developed. A motor stepper and appropriate motor driver allowed the system to works from a micro-step to full step speed. The injection system allowed one to control the volume as well as the flow rate. The developed system was able to feed a liquid sample to the QCM sensor surface with a wide range of flow rate. It was observed that the flow rate injection affected the QCM sensor to respond. Careful selection of the liquid flow rate was required to avoid an unintended effect on the QCM sensor. Using the developed system, we show that a minimum effect on the QCM sensor respond caused by the flow rate of the liquid injection can be achieved.

1. Introduction

The Quartz Crystal Microbalance (QCM) has a broad application range, including chemical and biosensors. The resonance frequency shifts of the quartz crystal were caused by some interaction of the sensor with its environment [1]. The change in frequency is proportional to the increase in mass on the surface as long as the mass is firmly attached to the deformation of the crystal oscillation. The advantages of QCM sensors are high sensitivity, high resolution, and low cost, therefore they have been widely used in the fields of chemistry, biology, physics, and medicine, especially in the analysis of gas or liquid composition [2]. The high sensitivity of the QCM sensor is highlighted by its ability to detect changes in mass in the order of micrograms to nanograms. This high sensitivity causes research on QCM sensors in a variety of applications increasingly rapidly, especially in its application as a biosensor [3].

In its use as a biosensor, the sample in water solution is injected to the sensor surface. Therefore, the liquid injection system is important. In previous studies, it was known that the flow rate effect of the syringe pump injection system affected the frequency response of the QCM sensor [4]. However, there were some shortcomings in the syringe pump; among others, the flow rate range was limited. Besides, the syringe pump is unable to flow the liquid to the sensor surface continuously. In this study, a peristaltic-based pump is used. Peristaltic pumps are used because these pumps can flow liquid continuously. Peristaltic pumps are also able to produce a higher flow rate range.

QCM sensor works in general as part of an oscillator circuit. When the electric power is applied to a pair of electrodes, mechanical force is generated via piezoelectric effect. With this effect, quartz crystal will resonate at its natural frequency by positive feedback through oscillator circuit [5]. One surface of the sensor directly comes in contact with the liquid containing the biomolecule sample [4] or other chemical substance to be detected. The mass of the biomolecule sample or the substance deposition on the sensor surface affects the resonance frequency of the sensor [6]. The Sauerbrey equation described the frequency change to the mass change on top of the sensor surface [7]. While in contact with liquid, the resonance frequency of the sensor changes due to the viscosity and density of the liquid. Kanazawa and Gordon showed the relation fo the resonance frequency to the density and viscosity of the contacting liquid [8]. However, due to the thin plate of the QCM sensor, for example a 10 MHz sensor has a thickness of 167µm, pressure change caused by liquid flow fluctuation may also affect the sensor resonance frequency [9].

The peristaltic pump sucks up liquid using a vacuum technique created by cyclic compression movements along with its flexible hose. Changing the rotation rate of the peristaltic pump motor changes the flow rate [10]. The rotation of the peristaltic pump produces a cyclic compression along the flexible hose, and therefore change the liquid pressure. The change of the liquid pressure propagates to liquid on the QCM sensor surface. Therefore, the QCM sensor also experiences a pressure fluctuation. Depending on the pressure change, it may affect to the QCM vibration. This work shows the effects of the flow rate of the peristaltic pump to the stability of the resonance frequency of the QCM sensor in contact with water.

2. Experimental

In this study, a liquid injection system in the form of a peristaltic-based pump is used. The movement of the stepper motor was assisted by a TB6600 motor driver which was controlled by an Arduino Nano microcontroller. The stepper motor used was a bipolar stepper motor with 200 steps per 360 degrees which in this study used a micro-stepping mode (1/32) to obtain more accurate results. The retrieval of the QCM sensor response data was carried out in the form of a change in the resonance frequency of the observation time. Data retrieval was done by flowing some distilled water continuously on the surface of the QCM sensor with a flow rate of 5, 10, 15, 19, and 32 µl/s.

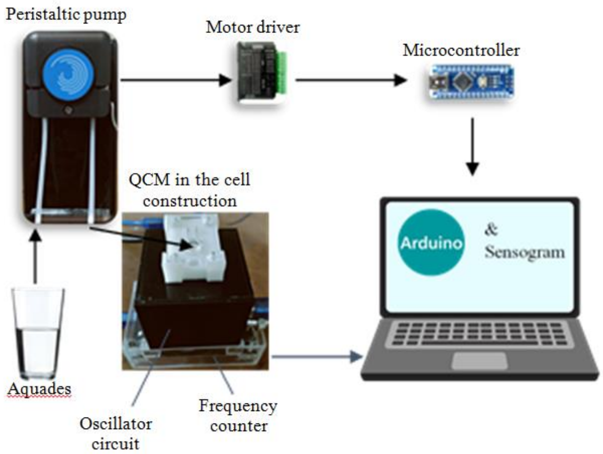


Figure 1. Experimental setup diagram.

Figure 1 shows the experimental setup of the experiment. One end of the peristaltic pump was placed in an open-air water container, and the other end was inserted into the QCM reaction cell. The QCM sensor was placed on a cell construction which was connected to an oscillator circuit. The resonance frequency of the sensor was recorded in every second using a frequency counter. The data was displayed as a sensogram. In the same time the frequency data was stored for further processing.

3. Result

Figure 2 shows the frequency change of the sensor to the initial frequency in contact with air (f_0). The frequency is the frequency difference between the resonance frequency at the measuring time to the initial frequency. It can be seen that the resonance frequency of the sensor change around -6KHz. Higher water flow rate leads to a bigger frequency change.

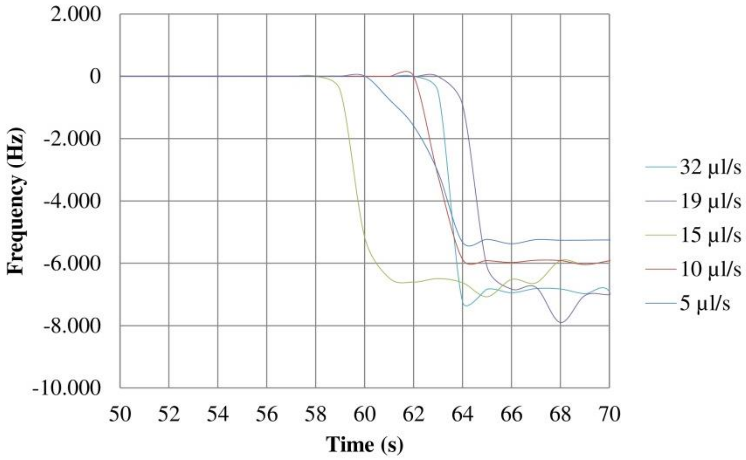
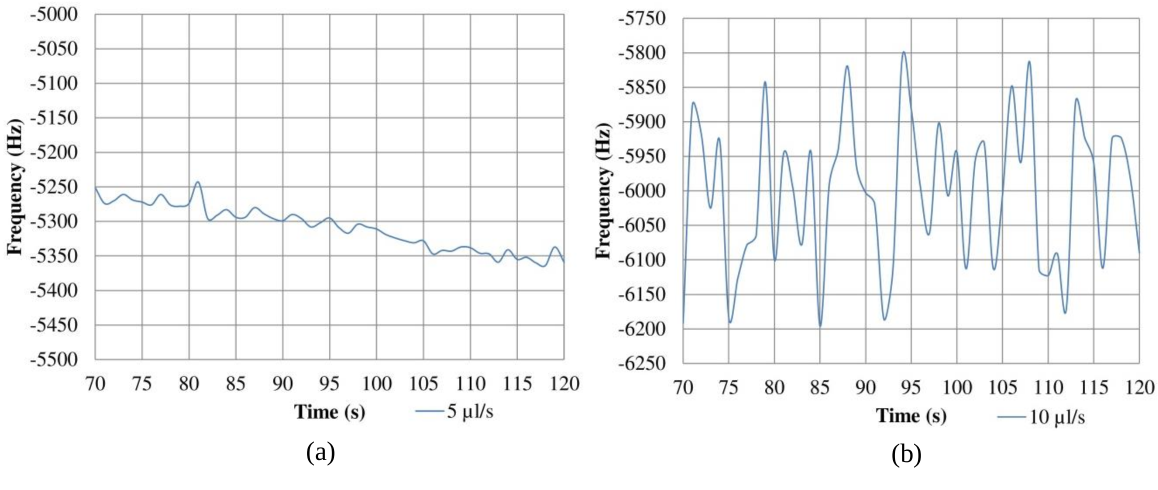
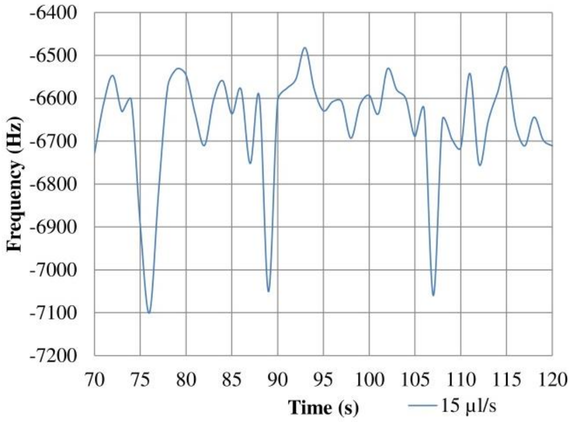


Figure 2. Time is needed when the sensor surface is in contact with air until when the entire sensor surface has been covered with water.

The fluctuation of the resonance frequency of the QCM sensor in contact with flowing water pumped using the peristaltic pump is presented in Figure 3. Small frequency fluctuation is observed in Figure 3a.





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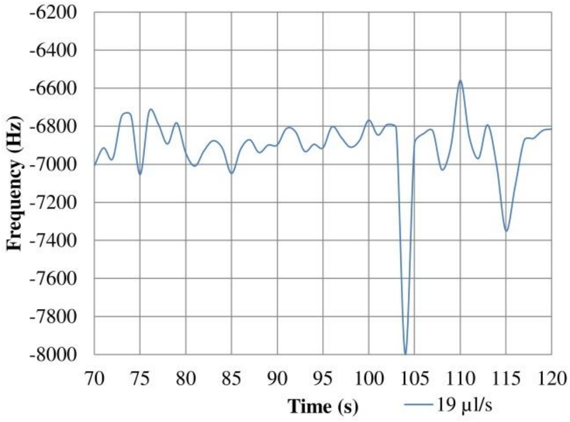

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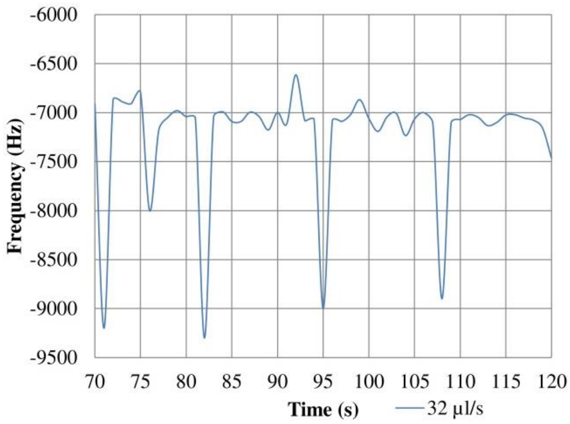
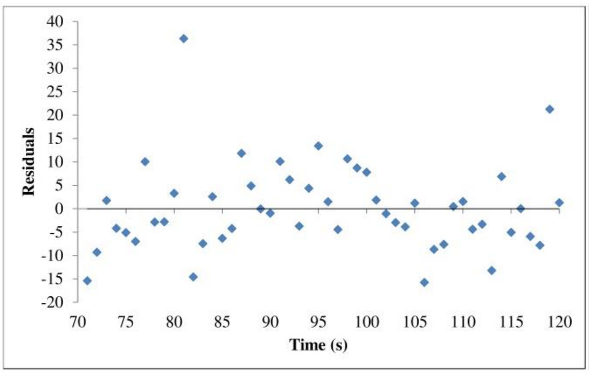


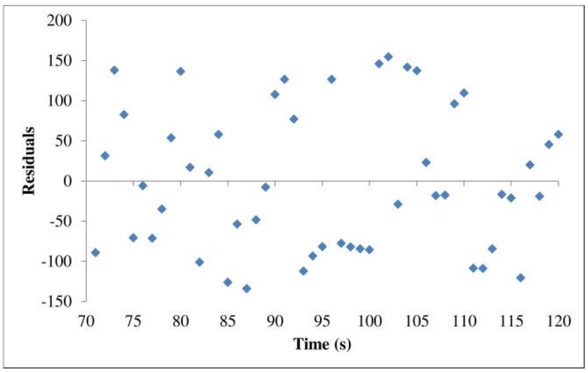
(e)

Figure 3. The relationship between the change of frequency and observed time after 70 s for (a) 5 µl/s, (b) 10 µl/s, (c) 15 µl/s, (d) 19 µl/s, and (e) 32 µl/s.

Residual analysis was applied to the resonance frequency at the fluctuating regime. Figure 4 shows the residual analysis of the resonance frequency under different water flow rate injection using a peristaltic pump.



(a)



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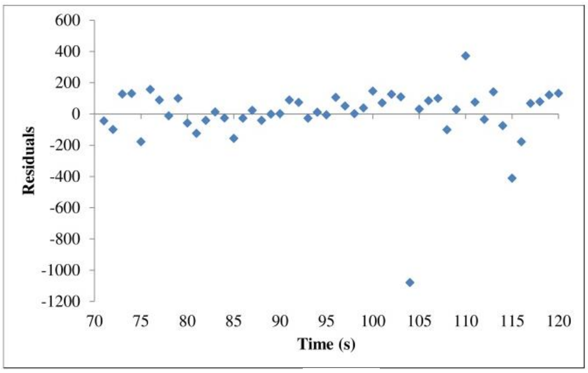

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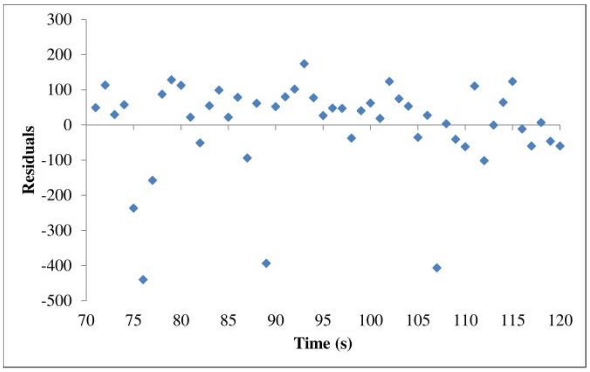

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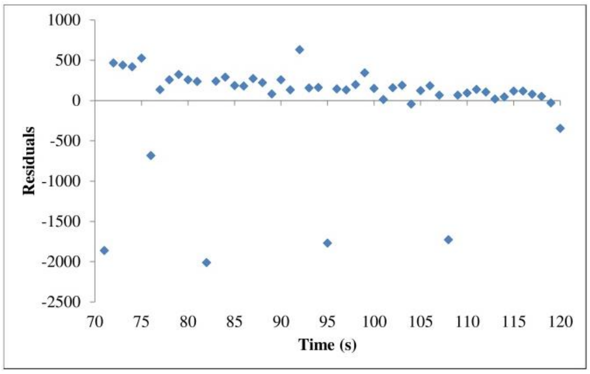

(e)

Figure 4. Residuals plot of the observation time for (a) 5 µl/s, (b) 10 µl/s, (c) 15 µl/s, (d) 19 µl/s, and (e) 32 µl/s.

Based on the residual plot in Figure 4, the ripple frequency was taken. Figure 5 shows the ripple frequency dependency on the water flow rate. The ripple frequency at a flow rate of 5µl/s is 9Hz, 89 Hz at a flow rate of 10µl/s and increased significantly at 589Hz when the flow rate was 32µl/s. The ripple frequency was higher compared to the spike frequency occurred when the water was injected using a continuous syringe pump as reported [4].

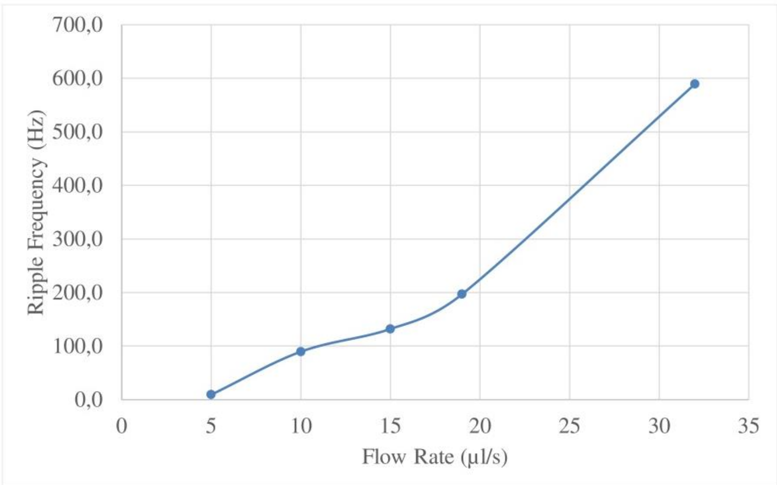

Figure 5. The relationship between ripple frequency and flow rate.

4. Discussion

Testing the response of the QCM sensor was carried out by flowing water onto the surface of the QCM sensor continuously. The test was carried out by flowing the water at a flow rate of 5, 10, 15, 19, and 32 μl/s. The flowrate was chosen between the minimum and maximum flowrate which can be achieve by the developed peristaltic pump with a maximum flowrate of 32 μl/s. . Before the water was injected into the reaction cell where the QCM sensor was inserted, the frequency of the sensor was recorded. After a constant resonance frequency in contact with air was achieved (f_0), the water was pumped to the reaction cell until the sensor surface was entirely covered with water. A new resonance frequency was reached.

Based on Figure 2, the time changes that occur when the sensor surface is still in contact with air and when the sensor surface is fully filled with water for flow rates of 5, 10, 15, 19, and 32 μl/s respectively are 5, 4, 3, 2, and 1 second. The time difference was as expected, as bigger flow rate means that the time to fill the same volume is shorter. After the surface of the sensor covered with water, the resonance frequency should be constant. However, we can see that the resonance frequency after the sensor in contact with water fluctuated.

The fluctuation at Figure 3 increased with an increasing flow rate. At a flow rate of 5μl/s and 10μl/s the fluctuation of the resonance frequency occurred similarly along with time. The fluctuation could be attributed to the cyclic pressure change as the peristaltic pump head rotated. However, a spike-like resonance frequency fluctuation was observed at higher flow rate. It seems that there was a release of accumulated pressure which was indicated by a big frequency change of the resonance frequency. Based on the data on Figure 3, the developed peristaltic pump is suitable to be used for a flowrate of less than 5 μl/s. High resonance frequency variation caused by the flowrate burdens the sensor signal respond.

Based on Figure 4, it can be seen that the frequency variation increases with the increasing flow rate. The residual value of the resonance frequency is bigger at a higher flow rate. At high flowrate it seems that the generated pressure from the peristaltic pump is not continuously delivered to the liquid and then to the sensor surface. The silicone hose preserve the generated pressure and released as an impulse pressure at a periodic time indicated by the spike in the sensor resonance frequency. The used of high flowrate is not recommended because of this effect.

Figure 5 shows that the ripple frequency of the QCM sensor increased with increasing water flow rate injected using the peristaltic pump. The pressure experienced by the sensor surface is very influential on the sensor frequency response was seen from the size of the ripple frequency that occurs. The smaller ripple frequency causes a more stable sensor response. Therefore it is important to control the water flow rate when the QCM sensor used with the peristaltic pump. Pressure cancelation or a low pass filter needs to be applied to minimize the pressure change caused by the peristaltic pump.

5. Conclusion

The frequency change of the QCM sensor in contact with air to water has a lag time. Higher flow rate has a shorter lag time. The pressure fluctuation of the water caused by the peristaltic pump injection mechanism affects the resonance frequency of the QCM sensor. The resonance frequency of the sensor was fluctuated according to the cyclic pressure change caused by peristaltic pump head rotation. Higher flowrate followed by a bigger ripple of the resonance frequency of the sensor. At high flowrate, the fluctuation does not have constant ripple amplitude but has spike-like fluctuation periodically.

Acknowledgments

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