

Investigation on the mass sensitivity of quartz crystal microbalance gas sensor using finite element simulation

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ABSTRACT

The increasing global trends in healthcare priorities towards improving the effectiveness of diagnostic procedure by utilizing a non-invasive method which is breath analysis. This will benefit in increasing treatment efficiency and also reducing healthcare costs. Breath is a simple technique where the sample are easily obtained and can be provided immediately. The most popular method that had been used in hospital are urine and blood. Contradict with breath, urine and blood take too much time to analyse the disease and a painful process. The detection technique of breath analysis is done by using electroacoustic wave sensor from piezoelectric substrate. This acoustic wave sensor has been used to detect the changes in the frequency where it will be used to detect the disease. Breath analysis is a technique where it uses an electronic nose (E-nose) as a device. E-nose consist of Quartz Crystal Microbalance (QCM) sensor in order to differentiate odor in human breath. QCM is a sensitive weighing device which have a high efficiency. AT-cut quartz was chosen as the piezoelectric material and aluminum as the electrode. The objective of this paper is to design and simulate a QCM sensor for breath analysis application. Other than that, it also to determine the important key parameters that influence the performance of breath analysis which is sensitivity and resonant frequency. QCM sensor is being simulate by using COMSOL Multiphysics software. This is to evaluate the behavior of QCM sensor in terms of Eigen frequency analysis. The simulated QCM sensor with quartz radius of 166 um resonates at 8.871 MHz. The sensitivity of the sensor is 0.167 MHz/ng after exposed to acetone gas which act as the breath marker for detection of diseases in exhaled breath. Hence, the proposed design can be used as a non-invasive approach for early detection of disease through breath analysis.

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1. INTRODUCTION

According to the World Health Organization (WHO) in 2015, the majority of death are caused by the four main noncommunicable disease (NCD) which are cardiovascular disease, cancer, chronic respiratory disease and diabetes. The highest cause of death among all these four diseases is cardiovascular disease which is 17.7 million deaths (45%) [1]. NCDs caused 70% of deaths globally where the highest statistic of death is in high-income countries which is 88 % while only 37% from low income countries [2]. Measuring the reason of the death and the statistic is important as this can help health authorities to take health action.

The first measure that can be taken by health authorities is by diagnosing the patient's health. However, diagnoses in today's clinical solution is quite costly and it takes too much time to be done. People in high-income countries will avoid cumbersome procedure as it will takes too much of their valuable time. Other than that, it is also a hurtful procedure by using blood as they need to draw blood from patient's body.

Thus, researchers have been seeking for a solution to solve this problem. As the technology in clinical diagnosis moved forward, a solution have been found by researchers to improve the effectiveness of diagnostic procedures by introducing a non-invasive method which is breath analysis. Breath is the most applicable method for disease detection as all human beings need to breath in order to live. Breath analysis functioning by detecting volatile organic compound (VOC) from patient's breath [3]. Illness can be diagnosed by detecting the differences in the gas components concentration. In order to diagnose a disease, breath markers have been discovered from the human's breath. Different breath markers will detect different disease. Nowadays, in the medical world there are a lot critical diseases that need a high efficiency device to help medical team to analyze diseases with a short of time. In today's clinical solution, the patient's need to use blood and also urine to diagnose the disease. As the problem with today's clinical solution takes too much time to analyze the disease and also painful to draw blood from the patient's body, researchers took initiation to solve this problem. Thus, new devices that only using breath have been introduced, called electronic nose (E-nose) and also gas chromatography [4]. In gas chromatography, the reason why it is not preferable as the device has a limitation, bulky and also expensive. It is also a complex instrument that consume too much time to detect the disease. Thus, E-nose has been chosen as the most efficient device to replace the invasive method which is blood and urine to non-invasive method which is by breath odor [5].

Electronic nose is a device which is simple, low cost and also portable device. In electronic nose, it consists of Quartz Crystal Microbalance (QCM) sensor in order to differentiate odor in human breath. The significance of QCM sensor is that it can imitate the mammalian nose that could reenact mammalian olfactory responses to smells. QCM have been selected to use in this device instead of other sensor is that it is an ultrasensitive weighing device. It also have high efficiency compared to other sensor in Gas Chromatography. Breath is a non-invasive approach for analysis and favored over blood and urine specimens [6]. The problem with today's clinical solution such as blood and urine and invasive [7]. As the technology in clinical diagnosis moved forward, researchers have found a solution to improve the effectiveness of diagnostic procedures by utilizing non-invasive methods which is breath analysis. As the diagnostic based on breath analysis are being developed, definitely there are advantages and disadvantages towards this test In breath analysis, breathing can be analyzed as exhaled breathing gas or exhaled breathing condensate. Collection through gas sampling in a gas bag is substantially more popular than the breath condensate method [8]. The first step for the breath test is to collect exhaled breath from a patient. From the exhaled breath, the breath marker will detect the composition of volatile organic compound by the sensor in electronic nose to detect the specific disease. The next process will go through sample collection/preparation and analysis.

The analysis of volatile organic compounds are used as the biomarker for breath analysis for noninvasive disease detection methods. Sensors are chosen for breath analysis as they are smaller and portable compared to laboratory diagnostic equipments [9]. Many attempts also have investigated the volatile organic compound sensing using quartz crystal microbalance (QCM) sensors and thin film bulk acoustic wave resonators [10]. QCM is the best among sensors to identify a wide range of (VOCs) for the quick response and also excellent sensitivity [11-16]. Previous work reported the detection of blood glucose by sensing the acetone as the biomarker. 30 patients were tested to detect the blood glucose. In this experiment, patient's breath is detected by using electronic nose. An electronic nose mimic the animal's nose as it has the ability to identify volatile chemical in surrounding. This experiment is done by detecting blood glucose and HbA1c level in patient's odor to detect diabetes illness. Data for this study were collected using AT-Cut quartz piezoelectric material which the thickness is 166.1 μm . The resonant frequency for this device is 10 MHz. The type of electrode used in this experiment is gold plated with 3mm diameter and coated with phthalocyanine solutions. This study has shown that the changes in frequency is in the range 5484–7300 Hz for the glucose while 572–768 Hz for the HbA1c [14]. The present of acetone gas in exhaled breathing odor is sensed using QCM sensor to detect diabetes disease. The capability of QCM sensor to analyses the data in actual time making them been utilized in a wide range of applications [17-19]. From the composition of breath, patients that suffer with lung cancer can be detected by using electronic nose. This device that was used consist of 8 quartz microbalance (QMB) gas sensor cover with diverse metalloporphyrins. Metalloporphyrins were used as sensing element due to the magnificent ligands for metal ions. In this research, 60 people has participated. A total of 35 were suffering from lung cancer, 18 participants as reference and 9 of them were evaluated after surgical therapy. This test takes time about 5 weeks. As the result, the volatile compounds in the breath samples were not detected in the healthy participant [20].

Overall, these researches have shown that quartz crystal microbalance (QCM) has been gain interest as it widely employed in many areas due to the outstanding performance. From the previous studies, it has been shown that QCM has a very high sensitivity to the mass changes. Thus, it is proven that QCM is highly suitable to use as a sensor due to its sensitivity [21]. In this paper, the design and optimization of a QCM sensor for breath analysis using finite element simulation is presented. The thickness of piezoelectric material is affecting the frequency change in the fundamental oscillation frequency as it is inversely proportional. Other than that, the area and the shape of the electrode can also affect the performance of the sensor. This interesting phenomenon requires further investigation. Hence, the objective of this paper is to investigate the mass sensitivity of quartz crystal microbalance sensor. For this purpose, the finite element simulation was conducted. The rest of the paper is organized as follows. Section 2 explains an overview on design concept and performance. Section 3 gives the details on the finite element simulation of the QCM sensor. Section 4 presents the simulation results. Finally, a conclusion is given in Section 5.

2. DESIGN CONCEPT

Piezoelectricity is the capability of converting from electrical (voltage) into mechanical signal and the other way around. When voltage is applied, mechanical signal will be produced in piezoelectric device [22]. Consequently, mechanical signal will be converting backwards to voltage. The piezoelectric sensor used in gas sensing consists of two types, which are the quartz crystal microbalance (QCM) and the surface acoustic wave (SAW) device [21]. The QCM creates a wave that travels along the bulk of the sensor while the SAW device creates a surface wave that travels through the surface of the sensor [21]. Both types of sensors follow the principle that the mass changing of the piezoelectric sensor coating is due to the gas absorption, which results in a change of the resonant frequency of exposure to a gas.

QCM is a simple device that has excellent sensitivity. QCM sensor contains a piezoelectric material which is quartz and sandwiched between two electrodes. When a voltage which is electrical signal that consist of electron is applied to the electrode, the piezoelectric effect will convert the electrical signal into mechanical vibration in terms of acoustic wave [22,23]. Then, the mechanical vibration will be converted back to the electrical signal. When a surface on QCM is being touched by a gas, the changes of mass will be occurred as the particle combined in the film. This will be effecting the changes in the frequency. In QCM sensor, the principle of the sensor is based on the changes in frequency to analyze the result of the experiment [12].

$$\Delta C_{gas} \Rightarrow \Delta m \Rightarrow \Delta f$$

where: ΔC_{gas} =concentration of gas

Δm =changes in mass (g)

Δf =frequency change (Hz)

The performance of a QCM sensor can be determined by resonant frequency, sensitivity and coupling coefficient.

2.1. Resonant frequency

Resonant frequency, f_r is defined as the specific frequency when acoustic wave oscillates at higher amplitude. The dimension of piezoelectric material will determine f_r . It is determined by using mathematical formula where v is velocity and d is the dimension in thickness of piezoelectric material as shown in (1).

$$f_r = \frac{v}{2d} \quad (1)$$

2.2. Sensitivity

The fundamental of QCM sensor is totally depending on the frequency shift because of the changes in mass. This sensitivity can be calculated using (2) where frequency change is denoted in Δf , c_f is the quartz mass sensitivity, A is sensitivity of area, Δm is the change in mass, and f_0 is resonant frequency of quartz [14].

$$s = \frac{\Delta f}{\Delta m} = -\frac{c_f f_0}{A} \quad (2)$$

From (2), it can be summarized as when there is a change in the layer, the frequency change will be affected. This is because the relationship between them is inversely proportional.

2.3. Coupling coefficient

Coupling coefficient is a no magnitude value that calculate the effectiveness of piezoelectric to convert from mechanical signal to electrical signal. The value of coupling coefficient is calculated using (2) and (3), where f_p is the parallel resonance frequency and f_s is the series resonance frequency.

$$k^2 = \left(\frac{\pi^2}{4}\right) \left(\frac{f_p - f_s}{f_p}\right) \quad (3)$$

3. RESEARCH METHOD

3.1. Before sensing

The proposed design was simulated using COMSOL Multiphysics. Frequency domain and eigen frequency analysis was done to compute the maximum displacement, resonance frequency and coupling coefficient. Eigen frequency analysis was performed to provide eigen values which shows the resonant frequency of the design. The electrode that been used in this project is AT-Cut quartz piezoelectric material. Therefore, the resonant frequency of this QCM sensor is 10 MHz. The device dimension of the proposed design is shown in Table 1. Figure 1 shows the cross section of the proposed design that consists of top electrode, quartz as the piezoelectric layer and gold bottom electrode. The 3D view of the QCM sensor and the mesh analysis were displayed in Figure 1(b) and Figure 1(c) respectively.

Table 1. Proposed design

Parameter	Value
Piezoelectric Material	AT Cut Quartz
Piezoelectric Thickness, t_p	166 μm
Electrode Material	Gold (Au)
Resonant Frequency, f_r	10 MHz
Acoustic wave velocity Quartz, v	3322 m/s
Length and width of AT cut quartz	1670 μm x 1670 μm
Length and width of AT cut quartz	1000 μm x 1000 μm
Electrode thickness	0.2 μm

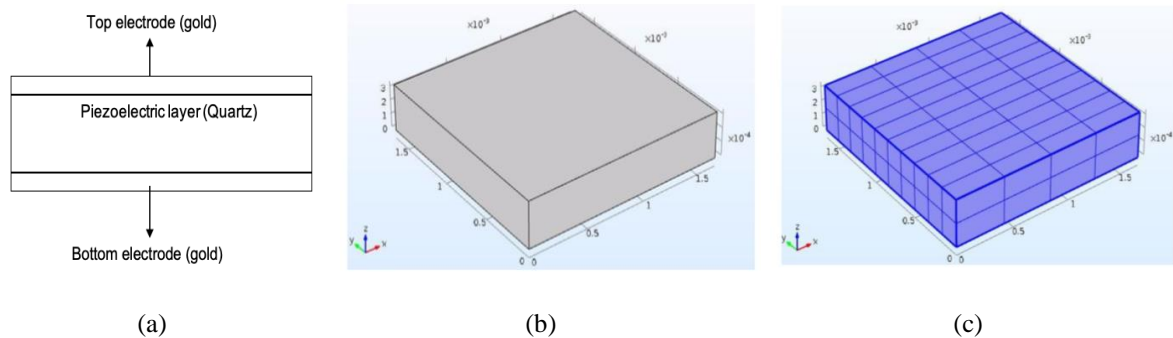


Figure 1. Model geometry of the COMSOL Multiphysics simulation, (a) Cross section of proposed design, (b) Geometry of square QCM sensor, (c) Mesh analysis with perfect matched layer

3.2. After sensing

For the sensing gas, this work is focused on the breath analysis and acetone gas was chosen as the sensing gas. This is because acetone gas is the potential biomarker for diabetes detection through breath. Current technique to diagnosis and monitoring blood glucose involve pricking fingers for blood test. This method is invasive and painful. Hence, excess amounts of acetone that is exhaled breath during are produced for patients with diabetes [24]. Figure 2 shows the model geometry after sensing. The performance of the QCM sensor is depends on the type of sensing films coated on the electrodes. Many type of sensing layers such as zeolites, polymer, carbon graphites and zeolites able to sense acetone vapors [25]. In this work, polymer was added on the QCM sensor to selectively adsorb the acetone gas as shown in Figure 2(a). Figure 2(b) shows the mesh analysis for the QCM sensor after sensing.

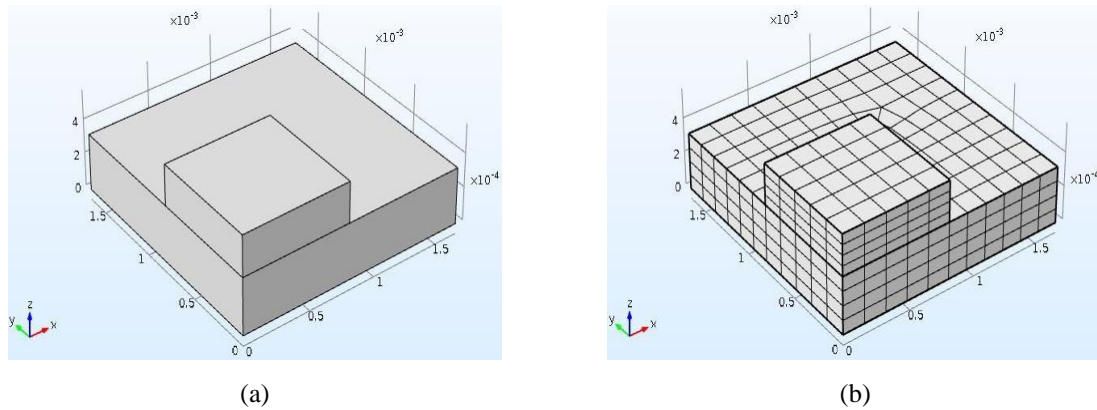


Figure 2. Model geometry after sensing, (a) Geometry of QCM sensor after sensing, (b) Mesh analysis with perfect matched layer

4. RESULT AND DISCUSSION

4.1. Before sensing

This section discussed the results obtained from the simulation of COMSOL Multiphysics. This simulation consists of two parts which are before sensing simulation and after sensing simulation. Simulation starts with the select geometry and square shape has been chosen for QCM sensor structure. Then, material can be selected at the Add Material toolbar to insert the needed material for QCM sensor. Next, electrostatics part is for the terminal and ground selection of the model. Lastly for mesh analysis, the model is divided into discrete components to get more accurate result in term of frequency. The analyzed results are displacement and figure for different thickness of quartz. In this simulation, there are 6 different thickness of quartz that varies from 50 μm to 400 μm . The contour plot for displacement of QCM sensor is shown in Figure 3 for different piezoelectric thickness of 50 μm , 100 μm and 250 μm . The resonance frequency shown is 32.475 MHz, 15.64 MHz and 6.495 MHz as predicted in the theory calculation from (1).

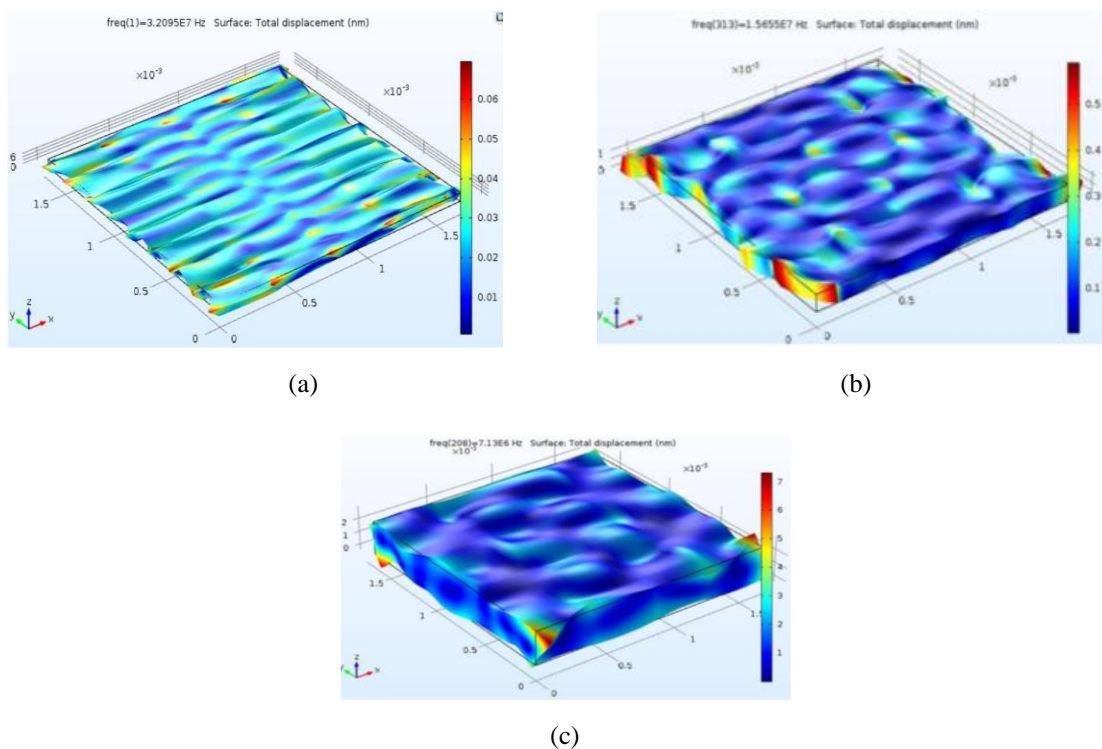


Figure 3. Contour plot for displacement for with different quartz thickness, d , (a) $d=50 \mu\text{m}$, (b) $d=100 \mu\text{m}$, (c) $d=250 \mu\text{m}$

The results from this simulation shows graph of displacement in frequency domain analysis. Displacement graph is a crucial part to obtain resonant frequency value. This resonant frequency varied along the piezoelectric thickness. It can be presumed that when the thickness of quartz is enlarged, the value for resonant frequency will be reduced. Figure 4 demonstrates the graph of resonant frequency versus thickness of quartz. From the graph, it can be concluded that as the thickness of resonant frequency increase, the resonant frequency will decrease. The highest value of resonant frequency is 32.475 MHz at 50 μm while the lowest is 3.560 at 400 μm . Thus, the thickness of quartz is inversely proportional with resonant frequency. The highest resonant frequency achieved is 32.475 MHz for piezoelectric thickness of 50 μm .

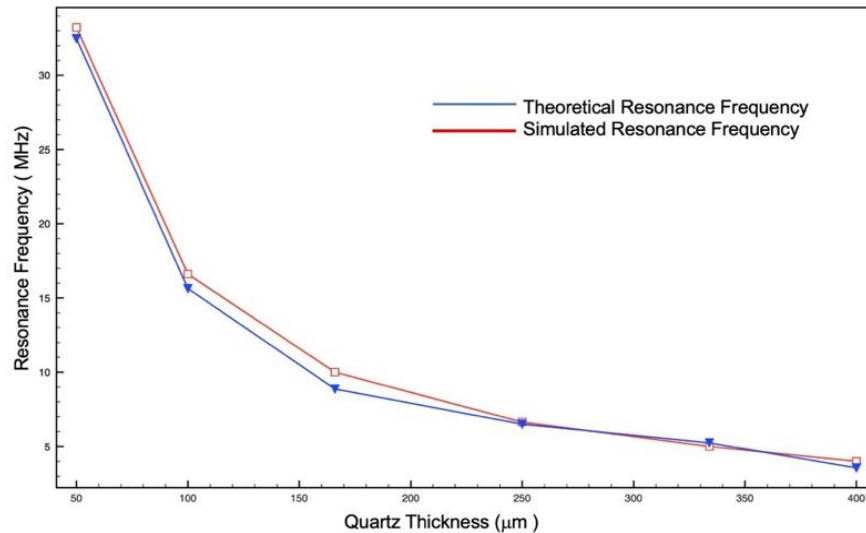
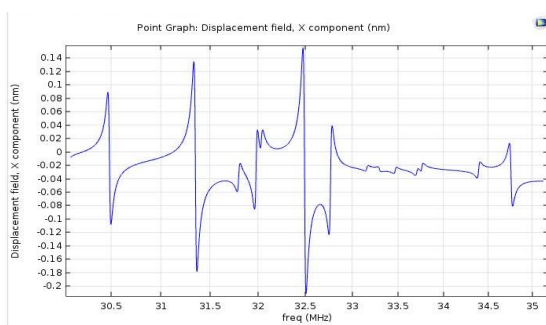
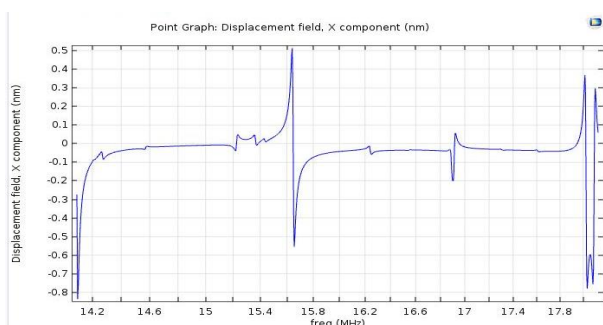


Figure 4. Quartz thickness vs simulated and theoretical resonance frequency before sensing

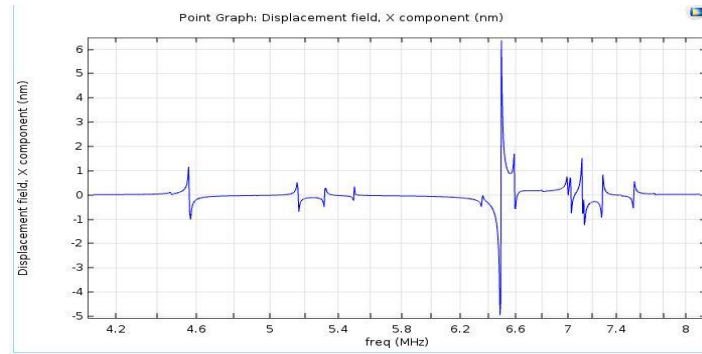
This displacement graph also provides the parallel resonance frequency, f_p and series resonance frequency, f_s as shown in Figure 5. Coupling coefficient is the magnitude value that calculate the effectiveness of piezoelectric to convert from mechanical signal to electrical signal. Next, the formula (3) is used to calculate the value of coupling coefficient, k_2 . The higher the thickness of piezoelectric material, the higher the electromechanical coupling coefficient. The most optimum value of coupling coefficient acquired is 0.00691 with piezoelectric thickness 400 μm as displayed in Figure 6.



(a)



(b)



(c)

Figure 5. Frequency vs admittance before sensing with different quartz thickness, d , (a) $d=50 \mu\text{m}$, (b) $d=100 \mu\text{m}$, (c) $d=250 \mu\text{m}$

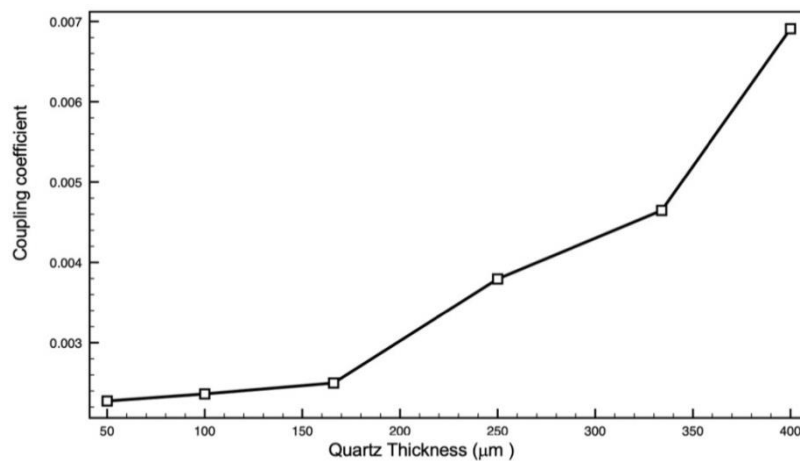


Figure 6. Quartz thickness vs coupling coefficient before sensing

4.1. After sensing

For the sensing gas, this work is focused on the breath analysis and acetone gas was chosen as the sensing gas. This is because acetone gas is the biomarker for diabetes detection through breath. In this section, analysis of simulation results for Quartz sensor with acetone gas will be reviewed. The variation of area of quartz from $435 \mu\text{m}$ to $1435 \mu\text{m}$. Sensitivity can be calculated using (5). As the area get bigger, the value of sensitivity decreasing. It also can be proven by using the Sauerbrey's (2). In conclusion, the relationship between sensitivity and area of quartz is inversely proportional. From Figure 7, the highest sensitivity is when the area of quartz is $435 \mu\text{m}$ which is 16.014 MHz/ng while the lowest sensitivity is at $1435 \mu\text{m}$ area of quartz where the sensitivity value is 8.81 MHz/ng .

In this section the effect of density of acetone gas toward the frequency changes is discussed. The density of acetone gas is diversified from 10 to $50,000 \text{ kg/m}^3$. The frequency before sensing of Quartz sensor is 8.871 MHz and the frequency after sensing is changing with the density of acetone gas due to mass loading. By finding the slope of plotting mass changes versus frequency changes graph, the mass sensitivity of Quartz sensor can be attained. In order to calculate mass changes, Δm , (4) can be used while the frequency change, Δf can be found. Hence, by using the formula below, the mass sensitivity can be acquired. Figure 8 illustrates the relation between frequency changes and mass changes due to the variation in density of acetone gas. From the analysis, it can be observed that the coupling coefficient for optimum design is 0.002499 at $166 \mu\text{m}$ of quartz thickness and the resonant frequency at 8.871 MHz . This result is following with the mass sensitivity for simulation is $0.1672 \text{ MHz.cm}^2/\text{g}$.

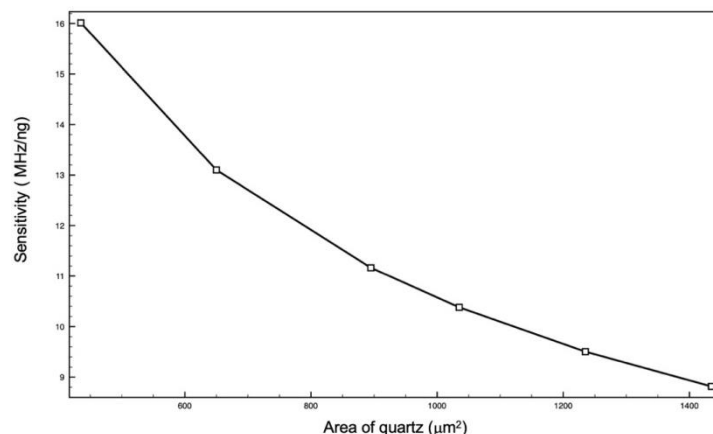


Figure 7. Area of quartz vs mass sensitivity after sensing

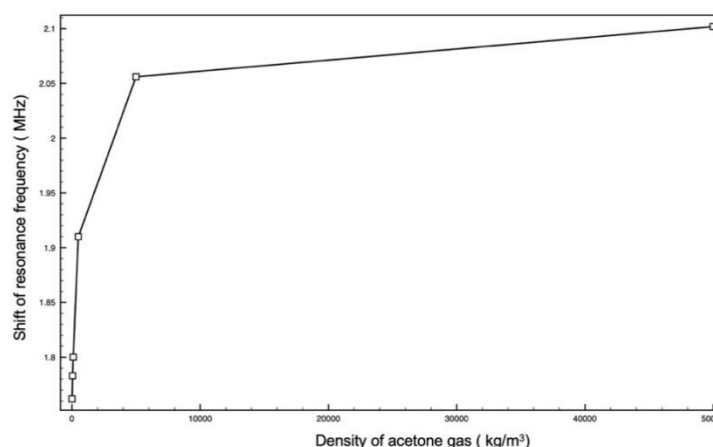


Figure 8. Density of acetone gas vs shift of resonance frequency after sensing

5. CONCLUSION

An investigation has been made on the mass sensitivity of the QCM sensors are studied via finite element simulation. 2D finite element modelling using COMSOL Multiphysics is presented. The simulation result shows that the relationship between sensitivity and area of quartz is inversely proportional. Piezoelectric material has been encountering a worldwide requirement development in the industrial and manufacturing market these days. There are lots of fields showing interest and enthusiasm such as engineering and medical field. Plus, being in the medical field, the utilization of QCM sensor can definitely be evolved. A few suggestions can be used to be implemented on the QCM sensor device. First and foremost, instead of acetone gas, another gas substance can be evaluated for the testing gas on Quartz sensor. This will differentiate between different valued of mass sensitivity of different gas. Therefore, if the breath analysis using an FBAR device, it can identify which type of gases being released by the patient according to the different mass sensitivity value and various types of diseases can be distinguished. It is with high hope that this research can be useful for the humankind in order for us to advance forward.

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Aliza Aini Md Ralib was born in Johor, Malaysia in 1982. She obtained her B. Eng. in Computer and Information Engineering (Electronics) from International Islamic University Malaysia in 2006. Both her MSc and PhD in Electronics Engineering are also from IIUM in 2011 and 2016 respectively. From 2009 to 2016, she was a Research Assistant at Universiti Tenaga Nasional Malaysia and IIUM. Since 2016, she has been Assistant Professor with the Electrical and Computer Engineering Department, International Islamic University Malaysia. Her research interests include Micro Electro-mechanical (MEMS), CMOS-MEMS acoustic wave resonators, electroacoustic sensors, piezoelectric thin film and MEMS piezoelectric energy harvesting. Upon completion of her PhD, she decided to work on developing on surface acoustic wave sensors for

breath analysis as noninvasive solution for early detection of critical disease. Her work has been published both locally and internationally in more than 34 papers in journals and proceedings locally and internationally. She has been awarded IEEE Postgraduate Award of Excellence in Outstanding Project in MEMS and Nanoelectronics organized by IEEE Electron Devices Society Malaysia Chapter in 2016.



Nik Nursyahida Bt Nik Mohd Zamri obtained her B. Eng. in Computer and Information Engineering (Electronics) from International Islamic University Malaysia in 2018. Her final year project title is Design of electric nose based on quartz crystal microbalance sensor. She has been awarded the best final year project for IEEE Electron Devices Society Malaysia Chapter track in 2018.



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Norfarahidah Za'bah currently works as an Assistant Professor at Faculty of Engineering, International Islamic University Malaysia after finishing her PhD in Engineering from Newcastle University in 2012. Back in 1999, she was a former graduate from Universiti Tenaga Nasional in Electrical & Electronic Engineering (Electronics) before continuing her studies at Newcastle University in MSc. Microelectronics in 2004. Her research areas are related to Manufacturing and Production Engineering, Nano-electronics, Applied Sciences and Technologies, Fabrication Technology.



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