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OPTIMIZING FORMALIN DETECTION IN FISH USING QCM SENSORS WITH TOMAC MEMBRANE COATINGS FOR PRODUCT QUALITY MONITORING

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ABSTRACT

Detection of formalin in fishery products is a significant concern in the food industry to ensure consumer safety. This study compared the performance of Quartz Crystal Microbalance (QCM) sensors without a membrane and with a Trioctyl methyl ammonium Chloride (TOMAC) membrane coating in detecting formalin in fish samples. The research findings indicate that QCM without a membrane for formalin samples has a lower detection limit of 150 ppm and an upper detection limit of 350 ppm with a sensitivity of 2194.171 Hz/ppm. On the other hand, QCM with a TOMAC membrane coating has a lower detection limit of 400 ppm and an upper detection limit of 550 ppm with a sensitivity of 842.7551 Hz/ppm. Meanwhile, QCM without a membrane for formalin in fish samples has a lower detection limit of 450 ppm and an upper detection limit of 650 ppm with a sensitivity of 15386.38 Hz/ppm. At the same time, QCM with a TOMAC membrane coating for formalin in fish samples has a lower detection limit of 350 ppm and an upper detection limit of 500 ppm with a sensitivity of 23108.9 Hz/ppm. Response time analysis shows that both sensors reach a steady state condition after 12 seconds. This study highlights the importance of selecting appropriate sensors for detecting formalin in fishery products, considering detection limits, sensitivity, and response time as crucial criteria. Thus, these findings can guide the fisheries industry in choosing effective and accurate formalin detection technology.

Keywords: Detection limit; Lipid; Quartz Crystal Microbalance; Response time; Sensitivity

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INTRODUCTION

The increasing public interest in fish as a healthy source of animal protein has driven the growth of the fisheries industry [1-2]. Fish are not only known for their high-quality protein content but also for being rich in omega-3 fatty acids, vitamins, and minerals essential for human health [3-6]. Additionally, with the abundance of fishery resources in certain regions, fishermen often face challenges preserving fish to maintain their quality during transportation and storage. One common preservative is formalin, which exhibits effective antimicrobial properties [7-8]. However, using formalin in fish preservation has raised serious concerns regarding consumer health, mainly due to its proven toxic effects. Therefore, an effective and reliable formalin detection method is needed to ensure the safety of fish consumption, thus assuring the health of the consuming public.

Several methods have been employed to detect formalin, including spectrophotometry[7] and gas chromatography[8]. While these techniques demonstrate good sensitivity, they often require expensive equipment, complicated sample preparation, and lengthy processing times. One promising method for detecting formalin is through the use of QCM sensors. This technology has advantages in detecting very small mass changes [9-11], thus capturing even low concentrations of formalin in fish samples. However, despite its great potential, QCM sensors still require improvement in sensitivity and detection specificity [12-14], especially when applied to complex matrices such as fish. Further development is needed to address these challenges, focusing on improving membrane coating systems on QCM electrodes. These membrane coatings play a crucial role in enhancing the interaction between the sensor and the analyte [11][15-17], thereby enhancing the sensor's ability to accurately and sensitively detect formalin. Therefore, this research will focus on characterizing QCM sensors with TOMAC membrane coatings on gold electrodes to optimize sensor response to formalin in fish samples. It is hoped that with further development of this sensor technology, a more reliable formalin detection method can be created to ensure the safety of fish consumption.

Several previous studies have investigated using various types of membranes on QCM electrodes to enhance the detection of chemicals [18-20]. Among the various membranes tested, lipid membranes stand out as promising [17]. TOMAC possesses several advantages that make it an attractive candidate for use in formalin detection in fish. One of the critical advantages of TOMAC is its ability to enhance sensor response to formalin [18]. With TOMAC coating on the QCM electrode, the interaction between formalin and the sensor can significantly increase, thereby enhancing detection sensitivity and specificity. Additionally, TOMAC exhibits chemical properties suitable for formalin detection applications, including good solubility in various solvents commonly used in chemical analysis [22-23]. Another advantage of TOMAC is its high stability, allowing the sensor to perform well over extended periods without degradation or significant changes in response.

Although previous studies have discussed the use of QCM for formalin detection [21], there still needs to be a significant knowledge gap regarding the characteristics of QCM sensors on gold electrodes for formalin detection. This is because no specific research has focused on using TOMAC layers in the context of formalin detection in fish. This gap is primarily related to the need for more adequate data regarding the characterization of QCM sensors and testing in realistic fish matrices. This data is crucial for evaluating sensor performance more comprehensively, including detection limits, response times, and sensor sensitivity to formalin under conditions approaching those in the fisheries industry [25-27]. Accurate detection limit measurements are vital to determining the extent to which sensors can detect deficient concentrations of formalin, which is an essential requirement in efforts to ensure food safety. Meanwhile, sensor response time reflects how quickly sensors can respond to the presence of formalin in fish samples, which is highly relevant in situations where rapid detection is needed to prevent excessive exposure to formalin [28-29]. Additionally, optimal sensor sensitivity will enable more accurate and sensitive detection of formalin [13], thus providing better protection for consumer health.

The main objective of this research is to characterize QCM sensors with TOMAC membrane coating on gold electrodes for formalin detection in fish. This study aims to address this knowledge gap and provide new contributions to formalin detection in fish. The scope of the research includes evaluating sensor detection limits, response times, and sensor sensitivity to formalin in realistic fish matrices. Therefore, this research will provide a deeper understanding of the performance of QCM sensors with TOMAC coating in formalin detection in fish. Thus,

this research is expected to contribute to developing sensor technology to ensure the safety of fisheries products and protect consumers from health risks associated with formalin exposure.

METHOD

TOMAC Coating on QCM

The electrode coating was performed using the spin coating method with the following steps: Firstly, the membrane was diluted with THF in a 1:1 ratio in another bottle and allowed to stand for 10 minutes until homogeneous. Next, the spin coater machine was prepared by turning on the power button and vacuum pump and then vacuuming the machine for approximately 10 seconds. The QCM sensor was then mounted on the spin coater, and the rotary spin coater chamber was closed before running the machine. Subsequently, the run button on the machine was pressed to operate the device, where the machine would rotate at an initial speed of 2000 rpm for 3 minutes. While the machine was spinning, approximately 150 microliters of membrane solution were taken and injected into the QCM sensor. Afterwards, the QCM sensor was gently lifted using tweezers. Finally, the QCM sensor was dried for 24 hours and ready for sample testing.

Sample Preparation

The first sample preparation began with a 40% formalin solution, which was diluted using distilled water. This dilution process resulted in a range of formalin concentrations from 100 to 750 ppm, with increments of 50 ppm. The small concentration intervals were carefully chosen to detect even the smallest changes in frequency during the detection process. For the second sample, fresh fish was used, with its mass predetermined for consistency. The fish was ground to produce an extract, which was then mixed with the previously prepared diluted formalin solution. The concentration of formalin in the fish extract was adjusted to match the predetermined concentration range, ensuring the sample was consistent with the study's parameters. Both samples were then ready for use in the detection system to measure the sensor's response to formalin in different forms.

System Measurement

The system measurement process began with the use of the open QCM instrument, controlled by the Open QCM 1.2 reader software. Initially, the QCM sensor was carefully mounted onto the prepared holder. Each sample, prepared in varying concentrations, was gradually dropped onto the sensor. Once the sample was applied, the Open QCM 1.2 software was activated, and the system was allowed to run for five minutes to stabilize the sensor's response. After testing each concentration, the QCM sensor was thoroughly cleaned with distilled water to remove any residual material and prevent cross-contamination between samples. This cleaning ensured accuracy in detecting new concentrations. Once all concentration variations had been measured, the software was turned off, and the recorded sensor data were saved in Microsoft Excel format for further analysis. This methodical approach allowed for systematic recording and evaluation of the sensor's response to each formalin concentration.

The accuracy of each data point measurement was obtained by averaging five measurements for each sample concentration variation. Each measurement produced 60 data points collected over five minutes after the sensor output stabilized. By using the average of five repetitions, the measurement results are reliable and provide a clear representation of the sensor's response to each tested concentration variation.

RESULTS AND DISCUSSION

Figures 1 to 4 illustrate the performance of the Quartz Crystal Microbalance (QCM) sensor in detecting formalin, both without and with the TOMAC membrane coating. Figures 1 and 3 show the performance of the QCM sensor without the membrane, while Figures 2 and 4 demonstrate the sensor's performance with the TOMAC membrane. The QCM measurement results are presented in Figures 1a, 2a, 3a, and 4a, where the dashed lines indicate the upper and lower detection limits. The area below the detection limit signifies a dead zone, and the area above the upper detection limit indicates a saturation region. This highlights the importance of selecting the appropriate detection method to achieve accurate results. Figures 1b, 2b, 3b, and 4b present equations that describe the relationship between sample concentration and QCM frequency. Linear regression analysis reveals a strong relationship between formalin concentration and the sensor's Δf oscillation, with high R-squared values serving as the best indicator of model fit. Based on this analysis, the highest R-squared value of 0.95 was recorded for the QCM sensor without the membrane, indicating optimal performance in detecting formalin. Table 1 summarizes the performance of each sensor, showing that the largest working range is associated with the QCM sensor without the TOMAC membrane. Furthermore, the sensor sensitivity indicates that higher sensitivity values correlate with better performance; the highest sensitivity was observed in the QCM sensor coated with TOMAC for detecting formalin in fish samples. It is important to note that the use of the TOMAC membrane may offer certain advantages, although it may compromise some performance aspects at lower concentrations. This study emphasizes the need for a comprehensive evaluation of various sensor configurations to achieve optimal detection results. These findings can serve as a crucial reference in the development of QCM sensors for applications in food safety and quality control.

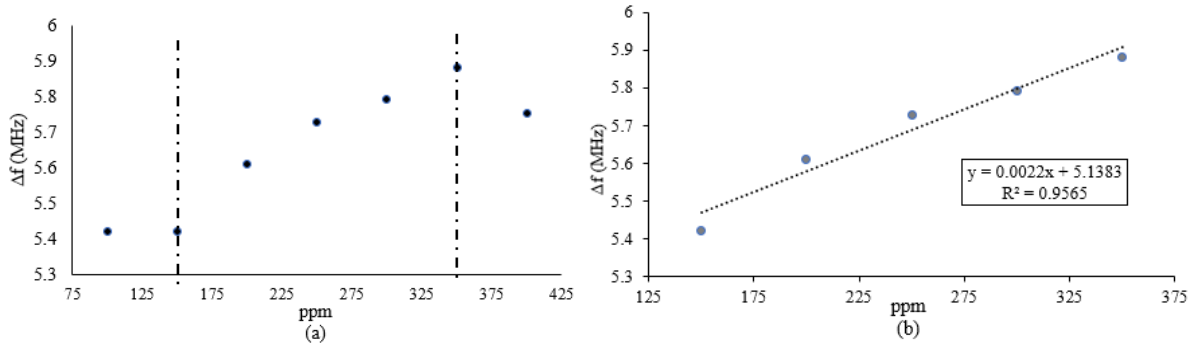

Figure 1. Performance of the QCM without membrane in detecting formalin

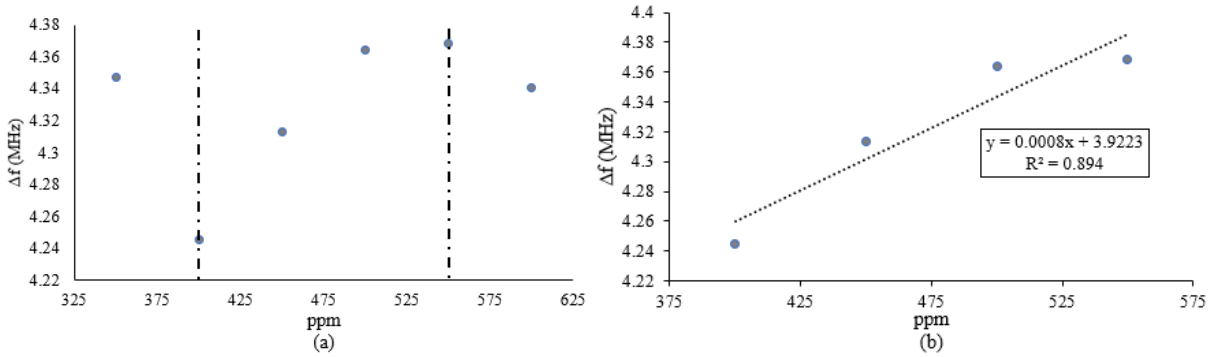

Figure 2. Performance of the QCM with TOMAC membrane in detecting formalin

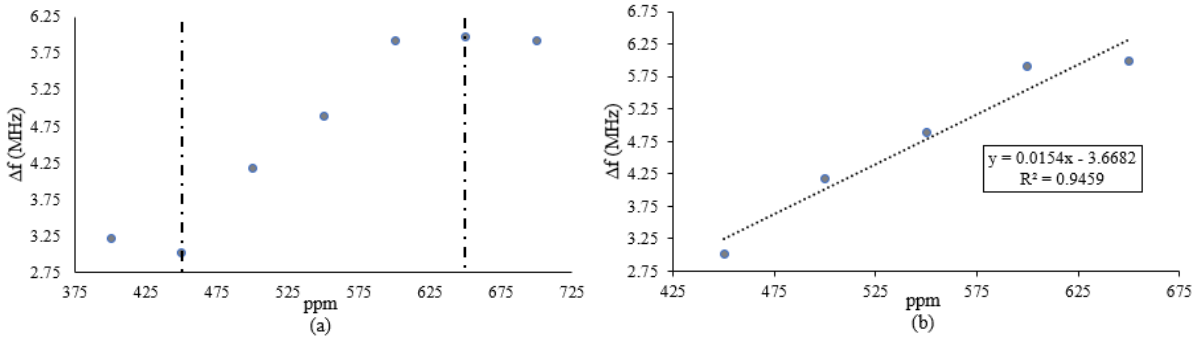

Figure 3. Performance of the QCM sensor without membrane in detecting formalin in fish

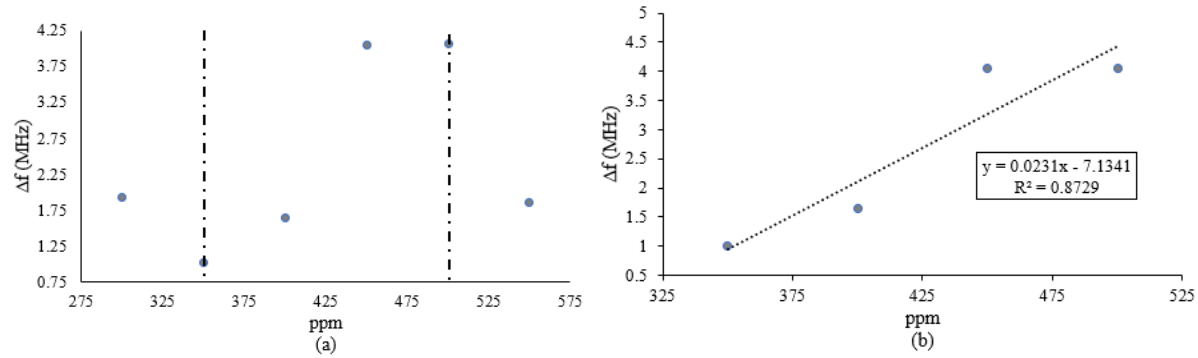

Figure 4. Performance of the QCM sensor with TOMAC membrane in detecting formalin in fish

Table 1. Table Description is adjusted to the length of the Table

Sensor QCM	Detection Limit (ppm)	Sensitivity (Hz/ppm)
Detection of formalin	150-350	2194.17
With TOMAC detection of formalin	400-550	842.76
Detection of formalin in fish	450-650	15386.38
With TOMAC detection of formalin in fish	350-500	23108.90

Figure 5 illustrates the response time of each sensor. The response time data were taken at different concentrations for each QCM based on its operating area. Based on the provided data, the response time of QCM sensors without membrane and with TOMAC membrane layers to formalin and formalin fish samples is relatively similar. In all cases, sensor response time begins at 3 seconds, with the sensor's Δf continuously increasing until reaching a steady-state condition at the 12th second. However, it is noteworthy that there is a difference in the Δf value at steady-state conditions between the TOMAC membrane and without membrane. When comparing the use of TOMAC membrane on formalin and formalin fish samples, it can be observed that the response time of membrane-less sensors has a higher Δf value at steady state compared to QCM coated with TOMAC. This indicates that the addition of the TOMAC membrane layer to QCM has a significant impact on sensor response time. Specifically, TOMAC membrane layers may slow down the sensor response time to formalin, especially at the onset of exposure. Nevertheless, this does not hinder the sensor's ability to reach a steady-state condition at 12 seconds, indicating that the TOMAC membrane layer still maintains sensitivity and sensor performance in detecting formalin. This is because TOMAC can enhance sensor response to formalin. TOMAC is also chemically stable to ensure the sensor performs well over a long period without degradation. Thus, adding TOMAC membrane layers to QCM can enhance its stability and performance over the long term.

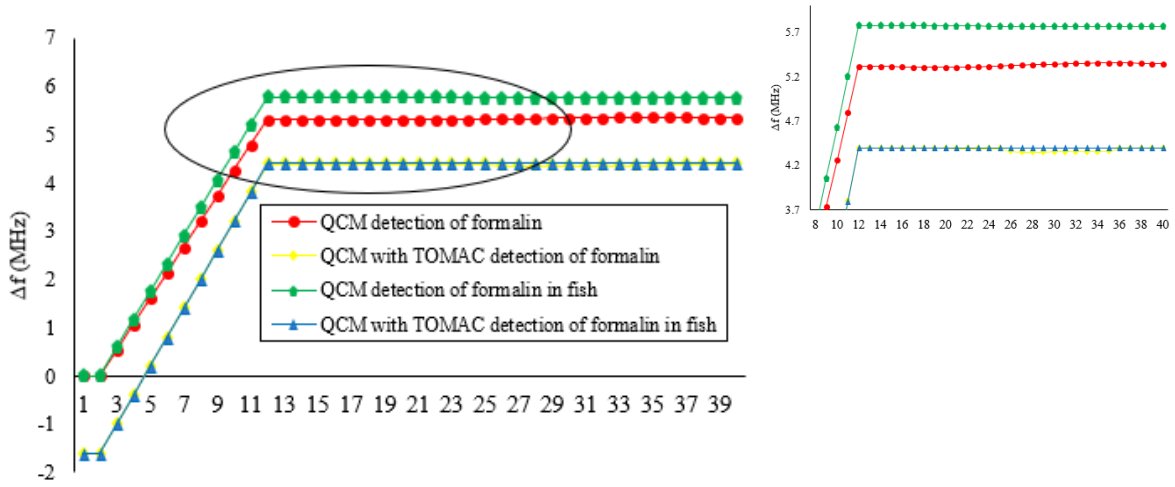

Figure 5. Response time of each sensor

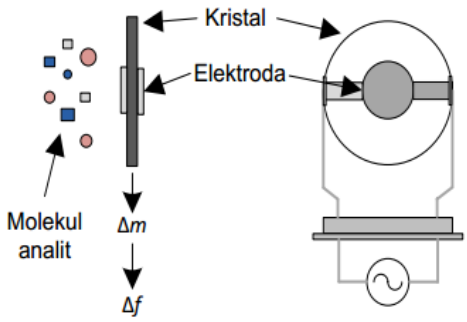

Figure 6. QCM sensor working principle in detecting samples [26]

The use of a TOMAC membrane on the QCM sensor can significantly alter the measurement range for formalin detection. Without the membrane, the QCM sensor detects formalin within the concentration range of 150-350 ppm. However, after the sensor is coated with the TOMAC membrane, the QCM's working range shifts, detecting formalin at a higher concentration range of 400-550 ppm. This shift is attributed to the interaction between the TOMAC membrane and formalin, which affects the sensor's sensitivity and selectivity towards formalin. The use of TOMAC membrane on QCM has been shown to enhance sensitivity in detecting formalin samples in fish. TOMAC membrane is an effective membrane layer that can enhance sensor response to the target substance, in this case, formalin [27]. This occurs because the interaction between formalin and TOMAC membrane results in a change in mass on the quartz crystal surface, which can be detected through changes in QCM oscillation frequency[28] as illustrated in Figure 6. The reaction between formalin and TOMAC membrane may involve the formation of hydrogen bonds or ionic interactions between formalin and functional groups on the TOMAC membrane, altering the surface properties of the membrane and eliciting an observable response in the QCM sensor [29]. Additionally, the TOMAC membrane can also act as a filter or selective separator, allowing formalin to pass through while inhibiting unwanted substances [7]. This helps to enhance the sensor's specificity to formalin, contributing to increased sensitivity in detecting formalin in fish samples. With the presence of the TOMAC membrane, the QCM sensor response becomes more specific and responsive to formalin, thereby enhancing the sensor's sensitivity to the substance. Furthermore, using the TOMAC

membrane also enables the detection of formalin at lower concentrations, thereby increasing the sensor's detection limit.

Traditional methods for detecting formalin, such as spectrophotometry and gas chromatography, although offering good sensitivity, often require expensive equipment, complex sample preparation, and lengthy processing times. As an alternative, the QCM method provides a faster, more efficient, and user-friendly approach to formalin detection, particularly in food samples. QCM operates by detecting changes in resonant frequency caused by the interaction of formalin with the sensor, which can be enhanced using a TOMAC membrane layer to improve selectivity. Despite being simpler and quicker, QCM detection results exhibit accuracy and sensitivity comparable to spectrophotometry and gas chromatography, even at low formalin concentrations. The main advantage of QCM lies in its ability to perform real-time detection with equivalent results, making it a superior solution for quality control and food safety monitoring.

These findings affirm that QCM sensors with TOMAC membrane layers have significant potential in detecting formalin at various concentrations. This is consistent with previous research that has explored different types of layers, including formaldehyde, to enhance QCM sensor performance in chemical detection. For example, research using lipid membranes has shown promising results in improving the sensitivity, selectivity, and stability of QCM sensors for formaldehyde detection [18-19]. However, it is essential to note that various factors, including experimental conditions and measurement methods, can influence these sensors' sensitivity and detection limits. Therefore, further adjustment and validation are needed before these sensors can be widely adopted in the fishing industry. Nevertheless, the use of TOMAC membrane on QCM sensors contributes significantly to the development of sensor technology for formalin detection in fish, along with previous efforts to explore various layers to enhance QCM sensor performance in chemical detection [22,20]. Thus, integrating this research with previous studies highlights the importance of ongoing exploration in improving QCM sensors for more effective and reliable chemical detection applications.

CONCLUSION

This study demonstrates that using QCM sensors with and without TOMAC membrane layers holds significant potential in detecting formalin in fish. The research findings indicate that the sensitivity, response time, and detection limit of both sensor types may vary depending on the utilization of the TOMAC membrane. Incorporating TOMAC membrane on QCM sensors enhances stability in response time and sensitivity in formalin fish samples. Overall, this study significantly contributes to developing more effective and reliable formalin detection methods in the fishing industry. These findings serve as a foundation for improving QCM sensors in detecting formalin, which, in turn, can enhance surveillance and quality control in fisheries products, ensuring the safety of products consumed by the public. Therefore, this research has important implications for supporting sustainability and food safety in the fishing industry.

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