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Multiwalled carbon nanotube/chitosan composite on quartz crystal microbalance for formaldehyde detection

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ABSTRACT

This study introduces multi-walled carbon nanotubes (MWCNT)/chitosan (CS) composite as a potential new sensing material for quartz crystal microbalance (QCM) formaldehyde sensors. This sensing material selectively binds target molecules, causing a measurable frequency shift proportional to the added mass. CS, MWCNT, and the MWCNT/Cs composite samples were prepared for comparison via sonification, crosslinking and dispersion methods. The morphology character was studied using Raman spectroscopy, Fourier transform infrared (FTIR) spectroscopy, and field emission scanning electron microscopy (FE-SEM). Next the samples were drop cast on the QCM working electrode. An adsorption test was conducted to study the static and dynamic response for the formaldehyde detection. The frequency shift of the formaldehyde adsorption for the CS, MWCNT-COOH, and MWCNT/CS-based sensors were 114.98 Hz, 108.23 Hz, and 196.63 Hz respectively. The calculated sensitivity of 23.48 Hz/ppm and regression line R2 at 0.95076 were recorded shows that the MWCNT/CS can be a promising sensing layer to detect formaldehyde vapour.

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1. INTRODUCTION

Volatile organic compounds (VOCs) are organic chemicals with high vapour pressure and are easily evaporated at room temperature [1]. Examples include benzene, toluene, formaldehyde, isopropyl alcohol, and acetone. Short-term and long-term adverse effects can be attributed to these compounds on human health and the environment. In addition to contributing to air pollution and smog formation, exposure to VOCs has been associated with respiratory problems, eye and throat irritation, headaches, and, in some cases, even more severe health effects [2]. Due to their potential health risks and environmental impact, monitoring and detecting VOCs is of great importance in various industries and settings [3], [4]. Hence, to solve this problem, various studies on VOC detection have been conducted. One of them is using quartz crystal microbalance (QCM) sensor. QCM is a sensor that can detect changes in nanogram mass [5]. QCM alone is

inadequate for detecting sensing VOCs as it lacks sensitivity and selectivity for a gas sensor [6], [7]. Thus, various sensing layers were suggested to be deposited.

Formaldehyde (CHOH) is a VOC and can be formed through natural sources such as biomass combustion and decomposition, anthropogenic activities such as industrial emissions and fuel usage by vehicles [8]. It is usually used as a base chemical material for manufacturing building and household materials such as furniture, antiseptics, cosmetics, adhesives, and so on. The World Health Organization (WHO) established a guideline in 2010 for formaldehyde, as prolonged exposure can cause respiratory disease and cancer in humans. Thus, the WHO limits the exposure of formaldehyde to 0.08 ppm for 30 minutes only [9].

Carbon nanotubes (CNT) are a well-known material with nano-size, powerful mechanical strength, thermally conductive, and chemically stable [10]. CNT exhibits favourable characteristics for sensing applications due to large surface area, porous structure, and presence of active sites (such as vacancies, impurities, functional groups, topological defects, and deformations). Their classification is related to the graphene layers' number in the cylindrical structure [11]. Multi-walled carbon nanotubes (MWCNT) possesses superior surface area and hollow geometry compared to single-walled CNT (SWCNT). Minor defects in the form of holes in MWCNT allow for enhanced material adsorption performance [12]. Previous research has employed CNTs-based sensors for the detection of various gases, both toxic and non-toxic, such as NH_3, COx, CH4, O_2, and H_2. However, a limitation of using CNT as a sensing material is its lack of selectivity for different gases and its limited sensitivity to analytes that do not interact strongly with CNT. To address these challenges, functionalization techniques have been proposed to enhance the advantages of CNT [13].

Chitosan (CS) is a polysaccharide derived from the deacetylation of chitin, possessing functional groups including amino and hydroxyl groups [14], [15], and is used in various applications such as medicine, biochemistry, and membrane technology. The reason is that CS has remarkable characteristics, including hydrophilicity, excellent mechanical strength, chemical versatility, and film-forming capacity [16]. However, the drawback of CS lies in its low porosity [16]. Consequently, researchers have investigated CS composites to enhance the sensitivity and selectivity by incorporating other materials, such as metal oxide and CNTs, addressing the limitations in capacity and mechanical properties of CS itself [16]. Furthermore, CS exhibits piezoelectric behaviour, rendering its suitability for utilization as a sensing material in QCM applications [17].

MWCNT has the advantage of a large surface area, which enhances the adsorption capability of gases. CS is a natural biopolymer with a high affinity with VOCs, as it is very hydrophilic. A few studies on MWCNT/CS have been conducted. For example, Hassan and Karam (2021) [18] synthesized MWCNT using the chemical fragments flame deposition method (FFD) and grafting MWCNT and CS to prepare oxidized MWCNT/CS. Their aim for this composite is to create adsorbents that remove pollutants in liquid. Zaman *et al.* [13] used the CNT/CS composite to determine gamma radiation's effect. Siregar *et al.* [19] prepared the composite from the natural material, such as converting oil palm shells into activated carbon, then growing the CNT by pyrolysis carbon vapour deposition (CVD), and processing horseshoe crab shells into CS.

CNTs are well-known for their superior mechanical and electrical properties. However, a significant limitation that stops the real applications is their lack of selectivity and sensitivity, especially VOCs. In the case of CS, a biopolymer, it has various functional groups, leading to high selectivity to analytes. However, it has weak mechanical properties and conductivity. A possible method to solve this problem is to functionalize them with biomaterials specific to the target VOCs. Hence, this paper introduces a multi-walled carbon nanotubes/chitosan (MWCNT/CS) hybrid as a potential material, harnessing the advantages of both.

2. RESEARCH METHOD

2.1. Material and apparatus

MWCNTs powder with a purity of 98%, outer diameter (OD) of 4-6 nm, and length of 10-20 µm were purchased from Chengdu Zhongke Times Nano Energy Tech Co., Ltd. CS powder (80-95% deacetylation, Friendemann Schmidt Chemical), acetic acid glacial (MW=60.05 g/mol, HmbG Chemicals), sulfuric acid (MW=98.08 g/mol, purity of 99.999%, Sigma Aldrich), nitric acid (MW=63.01 g/mol, 70%, Sigma Aldrich), isopropyl alcohol, formaldehyde solution min 37% (MW=30.03 g/mol, Friendemann Schmidt Chemical) and glutaraldehyde (Grade II, 25% in H2O, MW=100.12 g/mol) were used as received. The 10 MHz AT-cut QCM with Ti/Au electrodes with a frequency of 10.0 MHz was purchased from Novatech Sri. The QCM was rinsed in acetone and then dried in the oven at 50 ℃.

2.2. Preparation and characterization of multi-walled carbon nanotubes/chitosan composite on quartz crystal microbalance

The procedure for preparing acetic acid was adapted from Hekiem *et al.* [20], with the amount determined according to the proposal by Qi *et al.* [10]. 50 mg of CS powder was added to a 50 ml solution of 2.0 wt% acetic acid. The solution was heated and stirred using a magnetic stirrer at 60 ℃ and 1,200 rpm for 15 minutes. Finally, the solution was allowed to cool down to room temperature. MWCNT solution was prepared and modified using the method from Jang *et al.* [21]. The MWCNT powder was mixed with IPA solvent to become a 0.1 wt% MWCNT solution.

To prepare the MWCNT/CS composite, the procedure described by Qi *et al.* [10], Siregar *et al.* [19] were followed. Initially, 0.05 g of MWCNTs was added to the 50 ml CS solution and sonicated for 30 minutes to become MWCNT/CS solution. The solution was then stirred on a magnetic stirrer for an additional 2 hours at a temperature of 60 ℃ and a speed of 1,200 rpm. Diluted ammonium hydroxide solution was gradually added drop by drop into the solution until the pH reached 10. Next, the solution was transferred to a conical flask and placed into a water bath at 60 ℃. Next, 3.5 ml of 2.5% glutaraldehyde was introduced into the conical flask as a crosslinking agent and was left for 2 hours. The resulting mixture was then transferred to a centrifuge tube and centrifuged at 9,000 rpm for 15 minutes. The supernatant of the product was discarded, and the pellet was further dried in a drying oven for 12 hours at 40 ℃ until a constant weight was achieved. 0.113 g of MWCNT/CS powder was obtained. The summary of the methodology is shown in Figure 1.

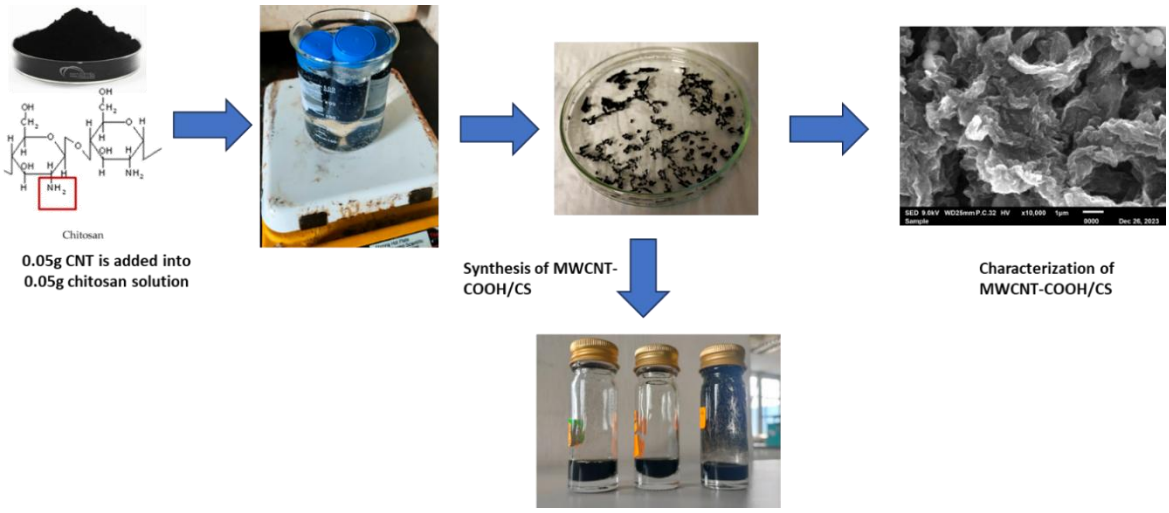


Figure 1. The schematic illustration of MWCNT/Cs composite preparation

A small amount of the powders (MWCNT, CS, and MWCNT/CS) were characterized using a Raman spectrometer, Fourier transform infrared (FTIR) spectrometer, and field emission scanning electron microscopy (FE-SEM). Raman spectra were obtained using RENISHAW in Via Raman Microscope with laser wavelength of 785 nm, exposure time of 240 s, and Raman shift range between 100 to 3200 cm^{-1}. The functional groups of the sample were taken using NICOLET iS50 FT-IR, with the spectral range within 1200 and 4000 cm^{-1}, and a resolution of 4 cm^{-1}. The FE-SEM images were obtained through the ZEISS MERLIN electron microscope with a resolution of 0.8 nm and 15 kV.

0.015 g of the MWCNT/CS powder was added into 3 mL of deionized water to produce 5 mg/mL of MWCNT/CS dispersion solution. Similarly, 0.02 g of functionalized MWNCTs were added into 4 ml of deionized water to produce 5 mg/ml of dispersion solution. The solution was mixed for 6 hours and sonicated for 1 hour. 5 µL of each of the three solutions was deposited at the center of the QCM electrode using a micropipette, as shown in Figure 2. Subsequently, 1 µL of formaldehyde was inserted into the QCM using a 10 µL micro syringe. The frequency measurements of the QCM were conducted using the OpenQCM Software before and after the adsorption of VOCs. Figure 3 shows the experimental setup of the QCM sensor for formaldehyde detection.

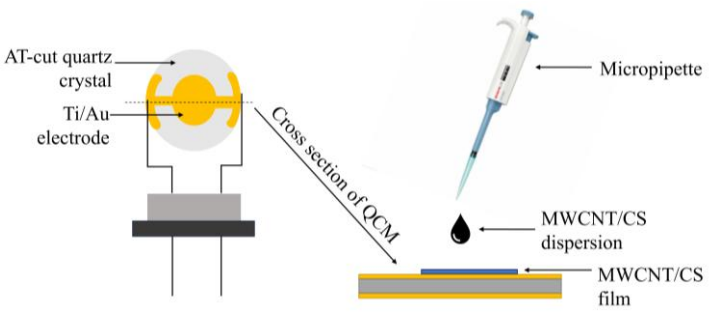


Figure 2. The drop-casting of the MWCNT/CS sensing layer onto the surface of QCM

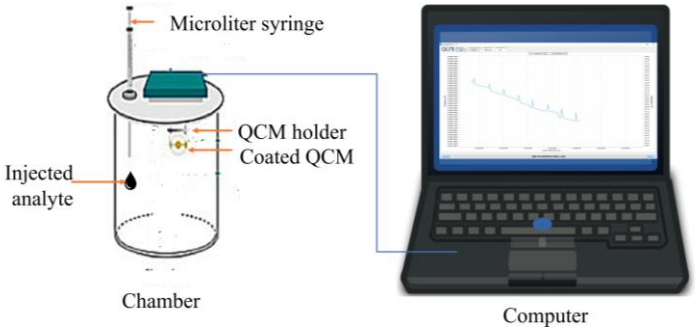


Figure 3. The experimental setup of the QCM sensor for formaldehyde detection

3. RESULTS AND DISCUSSION

3.1. Raman spectroscopy analysis of multi-walled carbon nanotubes

Previous research by Chen *et al.* [22] mentioned that adding different functional groups into MWCNT might cause changes in the morphology of MWCNT's surfaces, therefore, Raman spectrum was investigated. This paper also investigates the changes in the surface of MWCNT using Raman spectroscopy and SEM. Raman spectroscopy is a commonly employed technique for characterizing CNTs because of its excellent spatial resolution, high sensitivity, and minimal requirement for sample preparation [23]. Figure 4 is the Raman spectra for MWCNT, MWCNT-COOH, and MWCNT/CS. Two condensed graphene peaks at 1331 cm^{-1} and 1600 cm^{-1} display the D-band and G-band of the sample [23]. D-band indicates the sp3 of the carbon atom on the CNT and comes from the tangential vibrations from the atom of carbons [22]. G-band shows the sp^2 of CNT, which is a good indicator of the presence of defects on the graphene, proving that the sample is CNT [18], [22]. Figure 4 illustrates no changes in the Raman shift for all three samples. Moreover, the small ratios of D-band and G-band exhibit the high quality of the structure of MWCNTs, as there is a low amount of amorphous carbon [23]. However, this sample has a higher peak for the D-band, indicating a large ratio of the D/G band.

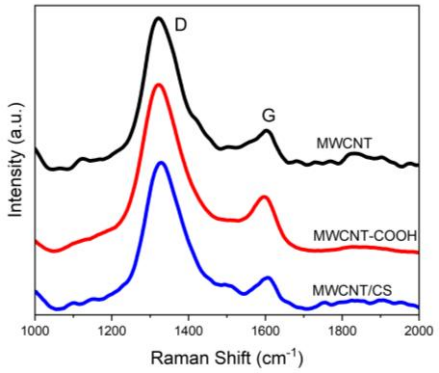


Figure 4. Raman spectra of MWCNT, MWCNT-COOH, and MWCNT/CS composite

3.2. Functional group analysis of the pure samples and the composite

FTIR analysis was conducted to characterize functional groups of the pure samples (MWCNT, CS) and the composite (MWCNT/CS). The chemical bond and functional groups of the sample are usually analyzed by FTIR spectroscopy. The importance of this analysis is to understand the surface chemistry of CNTs and the interaction of functional groups on the CNT's surface [23]. Based on the infrared spectrum of MWCNT, the surface is almost flat because CNTs itself is chemically inert, as there is no functional group [24]. In addition, the presence of C-C at 881.52 cm^{-1} indicates the characteristic of CNT, as shown in Figure 5 [23].

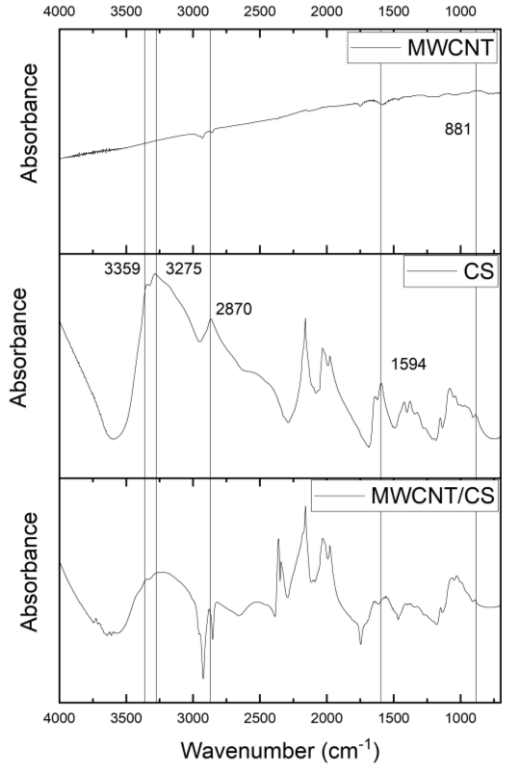
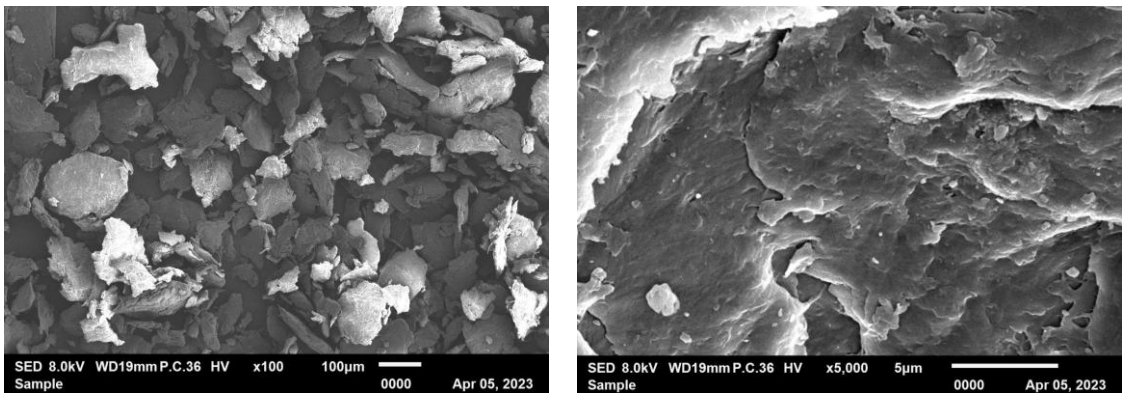


Figure 5. Raman spectra of MWCNT, MWCNT-COOH, and MWCNT/CS composite

Meanwhile, the strong and broad bands between 3275 and 3359 cm^{-1} for CS are attributed to N-H and O-H stretching with the intermolecular hydrogen bonds [24]. A peak at 2870 cm^{-1} shows the C-H asymmetric stretching of the CS, and 1594 cm^{-1} is the peak for N-H bonding of primary amine [25]. These peaks can also be observed in the MWCNT/CS composite at the same regions, respectively, showing that the functional group in CS and the bond of MWCNT are presented in the composite.

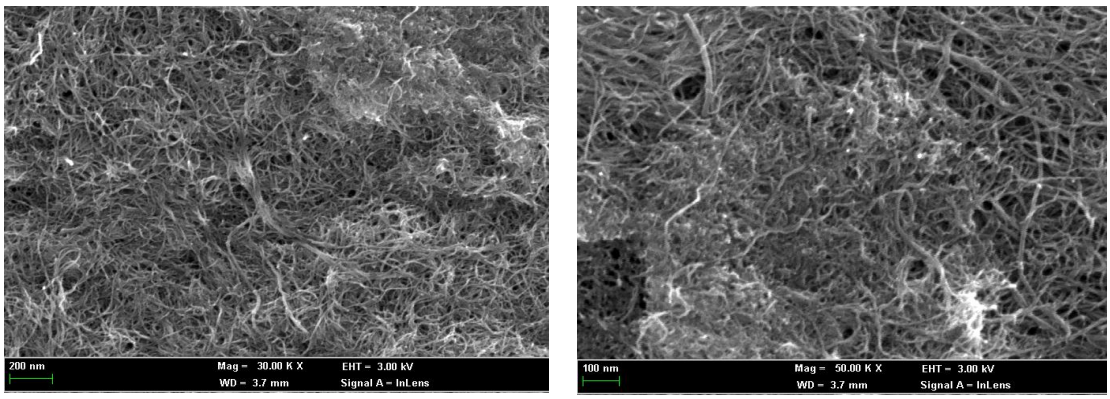
3.3. Morphology analysis of the samples

The surface morphology and homogeneity of MWCNT/CS were analyzed using SEM for CS and FESEM for MWCNT and MWCNT/CS, as illustrated in Figures 6 to 8. Figures 6(a), 7(a), and 8(a) present the overall structures, while Figures 6(b), 7(b), and 8(b) show their corresponding magnified views. From this analysis, CS exhibits a rough, standard surface, as shown in Figures 6(a) and (b) [13]. The functionalized MWCNT forms bundles of entangled rope-shaped tubes, as seen in Figures 7(a) and (b) [13]. Figures 8(a) and (b) depict the hybrid structure of MWCNT and CS, where CS is dispersed among the MWCNT bundles. In Figure 8(b), this dispersion is attributed to the interaction between the -COOH groups on CNT and the -OH groups on CS, enhancing the material's structural integrity [25]. However, the CS might be unevenly scattered due to the agglomeration nature of the MWCNT [26].



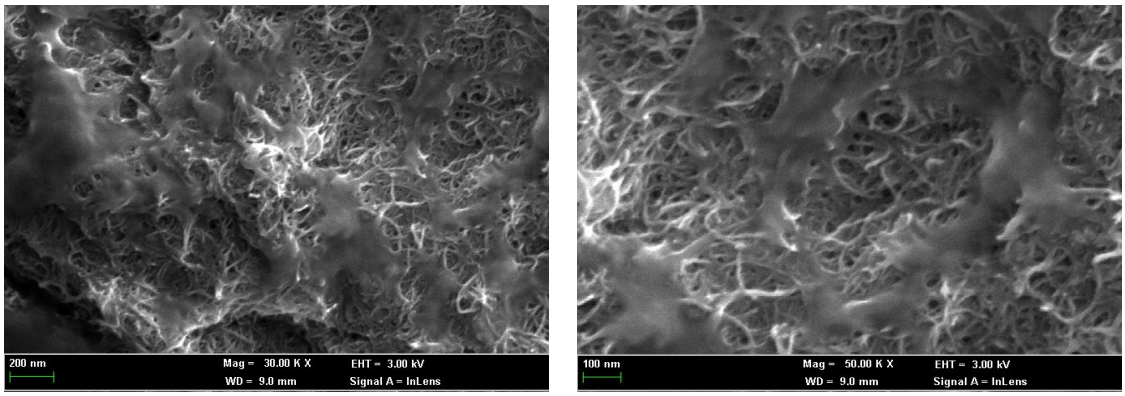
(a) (b)

Figure 6. SEM for CS magnification at; (a) x100 and (b) x5,000



(a) (b)

Figure 7. FESEM for MWCNT magnification at; (a) x30,000 and (b) x50,000



(a) (b)

Figure 8. FESEM for MWCNT/CS composite magnification at; (a) x30,000 and (b) x50,000

3.4. Response of sensors towards formaldehyde

3.4.1. Static response of sensors

The sensor response, consisting of QCM with a sensing layer of CS, MWCNT, and MWCNT/CS composite towards 37% formaldehyde, was conducted. When the formaldehyde was injected into the container, there was a frequency shift due to the adsorption of formaldehyde on the sensing layer. The time taken for the adsorption to reach equilibrium is known as the response time [20]. Figure 9 shows the graphs

for the frequency shift of each sensing layer when the adsorption of formaldehyde occurred until it reached equilibrium before the desorption process.

In Figure 9(a), MWCNT-COOH exhibited the lowest and most unstable frequency shift at 108.23 Hz due to its highly reactive surface chemistry. MWCNT-COOH reacts readily with environmental oxygen and moisture within the chamber, contributing to its instability. This aligns with the findings of Qi *et al.* [10], who highlighted the challenges of using unmodified CNTs in sensing applications due to their poor dispersion and agglomeration tendencies.

Conversely, the CS layer showed a slightly higher frequency shift (114.98 Hz) but demonstrated the slowest adsorption rate, with a longer response time (Figure 9(b)). This slow response is likely due to CS's inherent hydrophilicity and the presence of amine groups, which facilitate hydrogen bonding with formaldehyde molecules. However, the absence of conductive or enhanced reactive sites limits its adsorption efficiency, consistent with prior studies by Qi *et al.* [10] and Wang *et al.* [27], which emphasize the limited performance of CS in solo configurations compared to its composites.

MWCNT-COOH/CS composite outperformed both CS and MWCNT-COOH, exhibiting the highest frequency shift of 196.63 Hz with a faster response time of 1200 s as shown in Figure 9(c). This superior performance is attributed to the combined effects between the carboxyl-functionalized MWCNT and CS. The carboxyl groups introduced during functionalization enhanced the hydrophilicity and dispersion of MWCNT-COOH within the CS matrix [28]. The uniform dispersion and abundant reactive sites facilitated stronger adsorption of formaldehyde molecules, confirming the composite's potential as a robust sensing material. Additionally, the composite's ability to form hydrogen bonds with formaldehyde molecules further amplifies its adsorption capacity, consistent with the mechanisms described by Tai *et al.* (2015) [29] and He *et al.* (2015) [30].

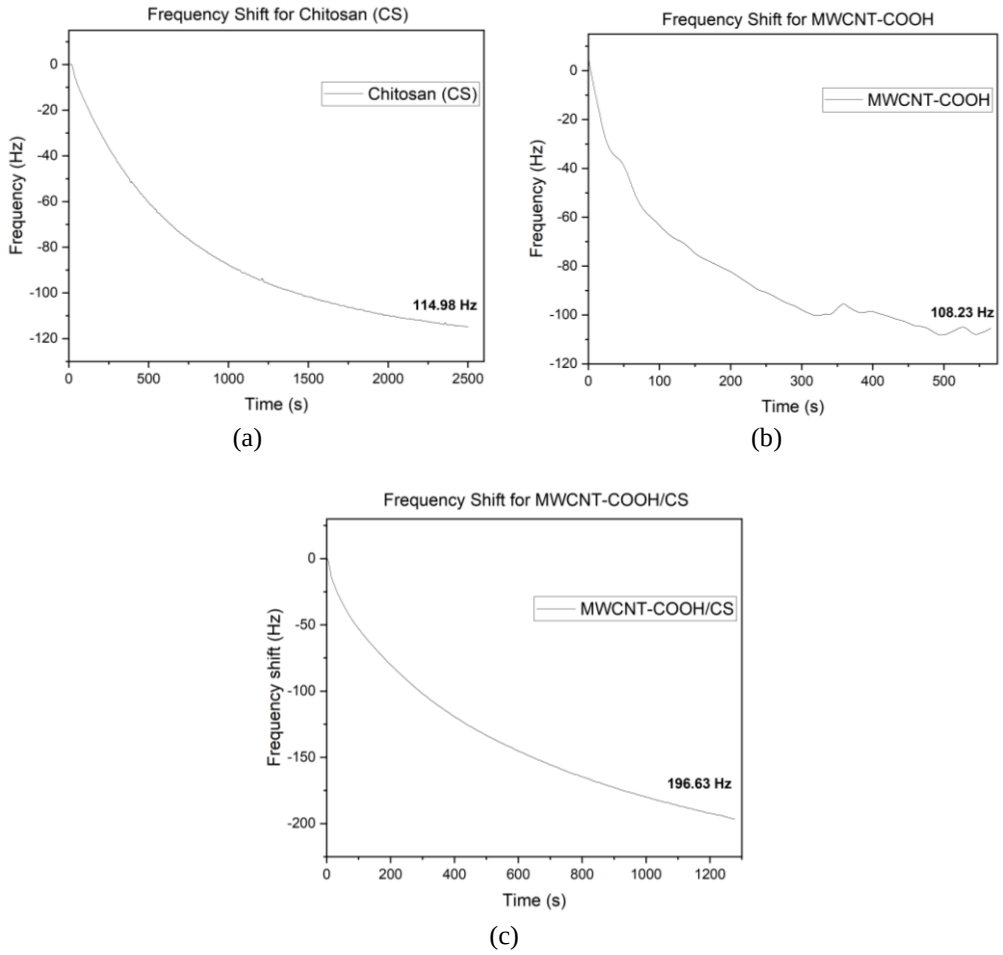


Figure 9. Frequency shift comparison of static measurements for; (a) CS, (b) MWCNT-COOH, and (c) MWCNT-COOH/CS composite upon exposure to 37% formaldehyde vapour

3.4.2. Dynamic response of sensor

The dynamic response of the MWCNT-COOH/CS composite as a sensing layer on a QCM sensor was studied to evaluate the sensor's limit of detection and reliability. The sensor's dynamic response was measured by observing frequency shifts of increasing formaldehyde concentrations under ambient conditions, imitating real-life environmental exposure. The measurement focused on formaldehyde adsorption using varying concentrations ranging from 0.2 ppm to 6.0 ppm. These steps confirm the sensor's stable baseline and its ability to detect formaldehyde even at low concentrations. Each step corresponds to the adsorption of formaldehyde molecules, with frequency shifts stabilizing before the next injection.

Previous study by Tai *et al.* [29] use under 8 ppm of formaldehyde in the experiment. Hence, a sequence of formaldehyde concentrations ranging from 0.2 ppm to 6.0 ppm (0.2 ppm, 0.6 ppm, 1.0 ppm, 2.0 ppm, 4.0 ppm, and 6.0 ppm) were exposed to the MWCNT-COOH/CS based QCM sensor to find the frequency shift as shown in Figure 10. It is noteworthy that the frequency shift of 1.0 ppm, 2.0 ppm, and 4.0 ppm slightly decreases compared to the others. The decreases might happen because the time taken for adsorption to achieve equilibrium is longer. The maximum frequency shift observed was 179.0 Hz at 6.0 ppm, demonstrating the sensor's robust adsorption capacity.

Figure 11 shows the linear relationship between frequency shift and concentration. The calculated sensitivity of 23.48 Hz/ppm significantly surpasses that reported by Tai *et al.* [29], where a PEI/MWCNT composite yielded a sensitivity of only 0.03102 Hz/ppm. This marked improvement highlights the interaction between MWCNT-COOH and CS, which enhances formaldehyde adsorption due to the increased availability of active sites and improved dispersion of MWCNT-COOH within the CS matrix. Comparing these findings to real-world formaldehyde limits, such as the OSHA permissible exposure limit of 0.75 ppm [31], the sensor's demonstrated limit of detection (LOD) of 0.2 ppm indicates its suitability for real-life applications, including environmental monitoring and workplace safety. This is particularly significant given that prolonged formaldehyde exposure can result in severe eye irritation, breathing difficulties, and burning sensations in the nose and throat [29], [31]. The regression value (R^2=0.95076) further confirms the linearity of the sensor's response, highlighting its reliability, as shown in Figure 11.

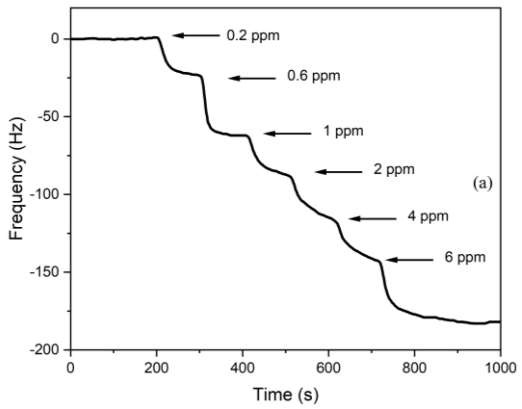


Figure 10. Dynamic response of MWCNT-COOH/CS film on QCM sensor towards 0.2 to 6 ppm of formaldehyde

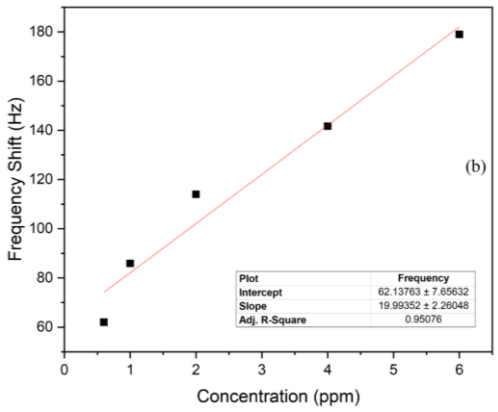


Figure 11. The relationship between frequency shift and concentration of formaldehyde in ppm

4. CONCLUSION

QCM sensors based on CS, MWCNT-COOH and MWCNT/CS composites were obtained. Raman spectroscopy was conducted and confirmed the quality of the MWCNT. Besides, the functional groups of each sample were confirmed through FTIR spectroscopy, where the presence of both MWCNT and CS was found in the MWCNT/CS composite, indicating that the coating of CS to MWCNT is successful. The morphology of the pure and composite samples was checked using SEM and FESEM, respectively, and CS is dispersed among MWCNT in the composite. The samples were further assessed on the QCM as a sensing layer to study its response towards formaldehyde as an analyte. The frequency shift of the formaldehyde adsorption for the CS, MWCNT-COOH and MWCNT/CS-based sensors were 114.98 Hz, 108.23 Hz, and 196.63 Hz respectively. The sensitivity of the composite as a sensing layer was recorded at 23.48 Hz/ppm. However, this research could be further extended to optimize the ratio of MWCNTs and CS in the preparation. The designed sensing layer could be a potential sensing layer in formaldehyde detection.

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AUTHOR CONTRIBUTIONS STATEMENT

This journal uses the Contributor Roles Taxonomy (CRediT) to recognize individual author contributions, reduce authorship disputes, and facilitate collaboration.

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C : **C**onceptualization
M : **M**ethodology
So : **So**ftware
Va : **Va**lidation
Fo : **Fo**rmal analysis
I : **I**nvestigation
R : **R**esources
D : **D**ata Curation
O : Writing - **O**riginal Draft
E : Writing - Review & **E**diting
Vi : **Vi**sualization
Su : **Su**pervision
P : **P**roject administration
Fu : **Fu**nding acquisition

CONFLICT OF INTEREST STATEMENT

Authors state no conflict of interest.

INFORMED CONSENT

We have obtained informed consent from all individuals included in this study.

ETHICAL APPROVAL

Not applicable.

DATA AVAILABILITY

The data that support the findings of this study are available from the corresponding author, Mohd Asyraf, upon reasonable request.

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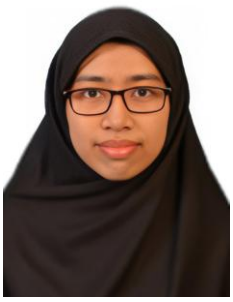

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