CARBON NANOTUBE-CHITOSAN THIN FILM ON QCM (QUARTZ CRYSTAL MICROBALANCE) FOR DETECTION OF IPA (ISOPROPYL ALCOHOL)

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ABSTRACT: This study presents the development of a quartz crystal microbalance (QCM) sensor coated with a multi-walled carbon nanotube–chitosan (MWCNT-COOH/CS) composite for the detection of isopropyl (IPA) vapor, a common volatile organic compound (VOC). The composite was synthesized via glutaraldehyde crosslinking to enhance bonding between carboxyl-functionalized MWCNTs and chitosan, followed by sonication and stirring to ensure uniform dispersion. Material characterization was carried out using Raman spectroscopy, Fourier-transform infrared spectroscopy (FTIR), and field-emission scanning electron microscopy (FESEM), confirming successful integration and interaction between MWCNTs and CS. Static QCM analysis showed that the MWCNT-COOH/CS composite achieved a balanced frequency shift of approximately 106 Hz with a response time of ~40 seconds, outperforming standalone CS and MWCNT layers in terms of response speed and signal stability. Dynamic measurements across IPA concentrations from 300 to 700 ppm revealed a linear frequency shift trend with a correlation coefficient (R^2) of 0.9713. Compared with similar sensors reported in the literature, the developed composite exhibits promising sensitivity, faster response time, and ease of fabrication, suggesting strong potential for real-time VOC monitoring applications.

KEY WORDS: *QCM, MWCNT, Chitosan, Nanocomposite sensor, and IPA.*

1. INTRODUCTION

Volatile organic compounds (VOCs) are organic chemicals characterized by high vapor pressure and low boiling points, allowing them to evaporate easily at ambient temperatures. Examples include benzene, toluene, isopropyl (IPA), and acetone [1]. VOCs are known to pose significant short- and long-term health risks, including respiratory irritation, neurological effects, and in severe cases, carcinogenicity [2]. Additionally, they contribute to environmental pollution and photochemical smog formation. Given their adverse effects, the real-time monitoring and detection of VOCs is critical across various sectors, including environmental monitoring, industrial safety, and healthcare [3]. Among various sensing technologies, the quartz crystal microbalance (QCM) stands out due to its high sensitivity to mass changes at the nanogram scale [4]. However, bare QCM crystals lack chemical specificity and are therefore inadequate for detecting VOCs without a suitable functional coating [5]. To enhance sensitivity and selectivity, QCM sensors are typically modified with functional sensing layers.

Carbon nanotubes (CNTs), particularly multi-walled carbon nanotubes (MWCNTs), have attracted significant attention in gas sensing due to their unique structural, electrical, and surface properties. MWCNTs offer high surface area, porosity, and active sites (e.g., defects, functional groups) that enhance the adsorption of gas molecules [6]. Their multi-layered structure and intrinsic hollowness make them superior to single-walled counterparts in specific applications [7]. Nevertheless, pristine CNTs often exhibit limited selectivity due to their non-polar nature and tendency to agglomerate, which hinders effective dispersion [8]. Functionalization techniques, including oxidation or surface grafting, are thus employed to improve their solubility, dispersibility, and chemical reactivity [9].

Chitosan (CS), a natural biopolymer derived from chitin, possesses amino and hydroxyl functional groups that can interact with polar VOCs through hydrogen bonding and electrostatic attraction [10]. It exhibits desirable characteristics such as hydrophilicity, film-forming ability, and biocompatibility [11]. However, its low porosity and moderate mechanical strength can limit its standalone performance in gas sensing applications [11]. To overcome these limitations, researchers have developed CS-based composites by incorporating nanomaterials such as metal oxides or CNTs to improve sensitivity, selectivity, and film stability [11]. Additionally, CS exhibits piezoelectric behavior, which further supports its integration into QCM-based sensor systems [12].

Several studies have explored the synthesis and application of MWCNT/CS composites. For instance, Hassan and Karam [8] prepared oxidized MWCNT/CS for liquid-phase pollutant adsorption. Zaman et al. [13] examined CNT/CS composites under gamma irradiation, and Siregar et al. [14] synthesized MWCNT/CS from bio-derived sources, including oil palm shell-derived activated carbon and chitosan from horseshoe crab shells.

To address these challenges, functionalization techniques have been explored to improve CNT stability in composites and introduce specific interaction sites, thereby potentially enhancing selectivity and sensitivity for certain analytes [14]. The degree of enhancement, however, depends on the chemical compatibility between the functional groups and the target VOC. Carboxyl-functionalized MWCNTs (MWCNT-COOH) offer better dispersion in polar matrices such as chitosan and can form hydrogen bonds or electrostatic interactions with polar analytes. This functionalization may enhance adsorption and sensor stability, though the improvement is strongly influenced by the analyte's polarity, molecular size, and the composite's microstructure [6, 11, 13]. Fig. 1 shows the structural formula for CS and MWCNT-COOH.

Hence, this study proposes the use of an MWCNT-COOH/CS composite as a sensing layer for QCM-based VOC detection. Isopropyl alcohol (IPA) was selected as the model analyte to evaluate the sensor's static and dynamic performance.

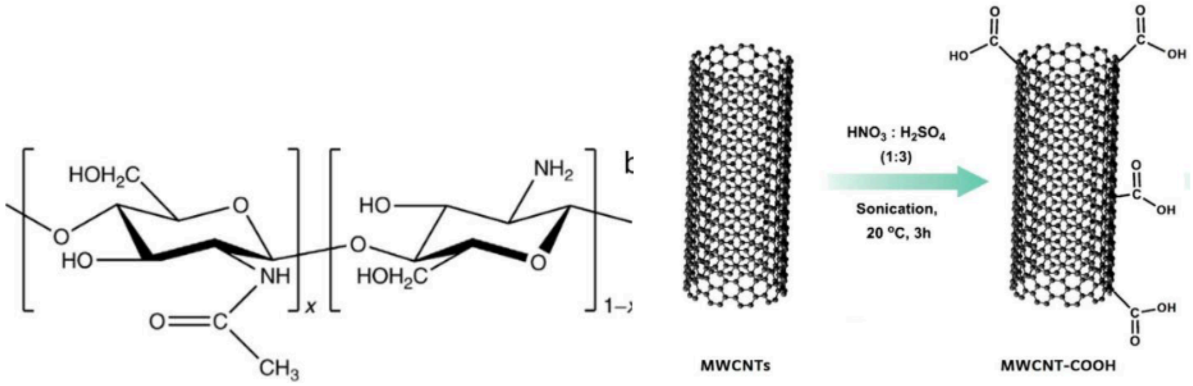

Fig. 1. Structural formula for CS [15], and MWCNT-COOH [16].

2. EXPERIMENTAL WORK

2.1 Materials and Apparatus

MWCNT powder with a purity of 98%, was purchased from Chengdu Zhongke Times Nano Energy Tech Co., Ltd. Chitosan (CS) powder (80–95%) was obtained from Friendemann Schmidt Chemical. Additional reagents including glacial acetic acid (molecular weight 60.05 g/mol; HmbG Chemicals), sulfuric acid (98.08 g/mol; 99.999% purity), nitric acid (63.01 g/mol; 70%), Isopropyl (IPA, 60.10 g/mol, purity 99.98%) and glutaraldehyde (Grade II, 25% in water, 100.12 g/mol) was also used. A 10 MHz quartz crystal microbalance (QCM) was supplied by Novatech SRI. Prior to use, the QCM crystals were cleaned with acetone and subsequently dried in an oven maintained at 50 °C.

2.2 Preparation and Characterization of MWCNT-COOH/CS Composite on QCM

The preparation of the acetic acid solution was based on the method proposed by Lukman Hekiem et al. [17], with quantities adapted from Qi et al. [18]. Specifically, 0.05g of chitosan (CS) powder was dissolved in 0.05L of 2.0 wt% acetic acid. The solution was heated and magnetically stirred at 60 °C and 1200 rpm for 15 minutes, then allowed to cool to ambient temperature. The dispersion of MWCNT-COOH was carried out using our previous research [19].

To synthesize the MWCNT-COOH/CS composite, the method was adapted from the procedures outlined by Siregar & Agusnar [14]. A total of 0.05 g of MWCNTs was introduced into the 50 mL CS solution, followed by 30 minutes of ultrasonication to enhance dispersion. The resulting mixture was then stirred continuously for 2 hours at 60 °C and 1200 rpm. Following this, diluted ammonium hydroxide was gradually added dropwise until the solution reached a pH of 10. The mixture was then transferred into a conical flask and incubated in a water bath at 60 °C. To initiate crosslinking, 3.5 mL of 2.5% glutaraldehyde was added, and the solution was allowed to react for 2 hours. Afterward, the mixture was centrifuged at 9000 rpm for 15 minutes. The supernatant was discarded, and the resulting pellet was dried in an oven at 40 °C for 12 hours until a constant mass was achieved. A total of 0.113 g of dry MWCNT-COOH/CS composite was obtained.

The synthesized powders of CS, MWCNT-COOH, and MWCNT-COOH/CS were characterized using FTIR and FE-SEM. FTIR analysis was conducted using a NICOLET iS50 instrument over the range of 1200–4000 cm^{-1} with a resolution of 4 cm^{-1}. Surface morphology was examined using a ZEISS MERLIN FE-SEM operating at 15 kV with a resolution of 0.8 nm.

2.3 Sensor Static and Dynamic Measurement

For sensor fabrication, 0.015 g of MWCNT–COOH/CS and 0.02 g of MWCNT–COOH powders were dispersed in deionized water (5 mg/mL), stirred for 6 h, and sonicated for 1 h. A 5 µL aliquot of each dispersion (CS, MWCNT–COOH, MWCNT–COOH/CS) was drop-cast onto the center of a QCM electrode (Fig. 2).

The coated QCM was mounted in an OpenQCM Arduino holder connected to a Teensy microcontroller. Baseline frequency was recorded before exposure, then 5 µL of IPA was introduced into the chamber using a microsyringe. Frequency shifts were monitored with OpenQCM software (Fig. 3).

Static measurements were performed at a single analyte concentration, while dynamic tests for IPA were conducted at 300–700 ppm in sequence, after stabilization at each step. All experiments were repeated three times using three independent QCM sensors, and results were reported as mean values.

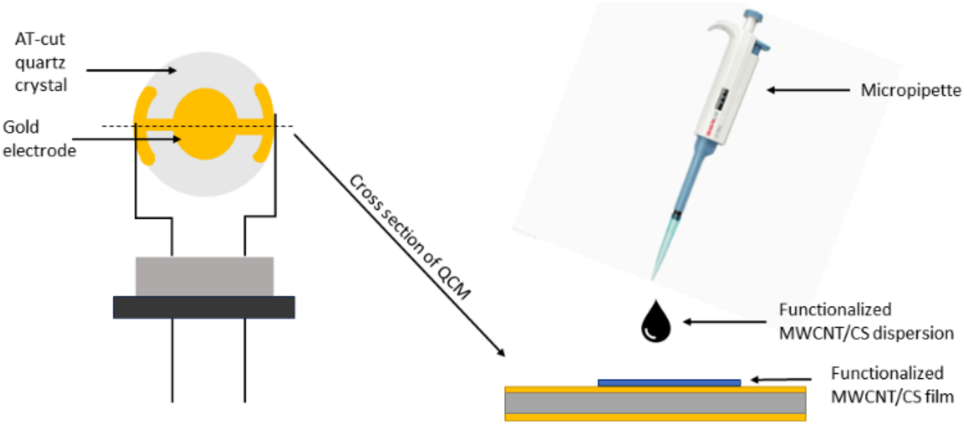

Fig. 2. Schematic illustration of the drop-casting process for depositing the MWCNT-COOH/CS composite sensing layer onto the quartz crystal microbalance (QCM) surface.

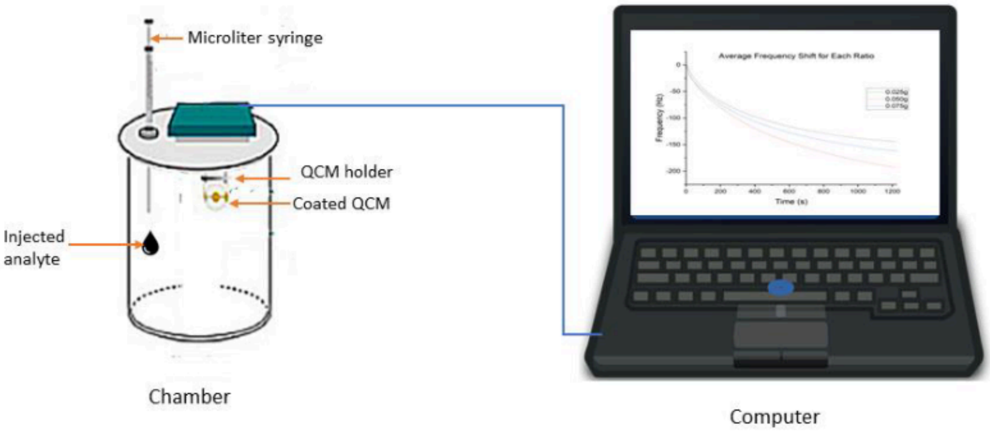

Fig. 3. The experimental setup when the IPA was injected into the chamber and the reading was recorded in the software.

3. RESULTS AND DISCUSSION

In this section, the adsorption characteristics of this composite were evaluated using quartz crystal microbalance (QCM) measurements. The MWCNT-COOH/CS sensing layer demonstrated excellent adsorption capacity and rapid response when exposed to isopropyl (IPA), affirming its potential for volatile organic compound (VOC) detection.

3.1. Functional Group Analysis of the Pure Samples and the Composite

Fourier-transform infrared (FTIR) spectroscopy was performed to investigate the functional groups present in MWCNT, chitosan (CS), and the MWCNT-COOH/CS composite. The top spectrum in Fig. 4, corresponding to chitosan, displays prominent features associated with its polysaccharide structure. While multiple absorption bands are visible in the chitosan spectrum (Fig. 4, top), only the dominant peaks at 3359, 3275, 2870, and 1594 cm^{-1} were emphasized here, as they correspond to the functional groups most directly involved in VOC interactions. Other smaller bands, such as C–O stretching (1020–1150 cm^{-1}) and amide II (~1420 cm^{-1}), are also present but were not the primary focus of this analysis. Broad and intense bands at 3359 cm^{-1} and 3275 cm^{-1} are attributed to overlapping O–H and N–H stretching vibrations, typical of hydrogen-bonded hydroxyl and amine groups [11]. The peak at 2870 cm^{-1} corresponds to C–H asymmetric stretching, while the band at 1594 cm^{-1} represents N–H bending from primary amine groups. These features confirm the presence of abundant polar functionalities capable of forming hydrogen bonds and electrostatic interactions—important for both dispersion and sensing. As shown in the middle panel of Fig. 4, the FTIR spectrum of MWCNT exhibits a minimal number of characteristic peaks, reflecting its chemically inert nature [12]. A weak absorption band is observed at approximately 881 cm^{-1}**,** which corresponds to C–C stretching vibrations within the hexagonal carbon framework—characteristic of graphitic structures [20]. The flat baseline and absence of polar functional group peaks support the hydrophobic and non-reactive nature of the raw MWCNT surface. In the composite spectrum (bottom panel, Fig. 4), several key characteristics were observed. The bands of CS at 3359, 3275, 2870, and 1594 cm^{-1} remain present in the composite spectrum, indicating that the chitosan backbone is retained. The broadening and slight shift of the O–H/N–H stretching band region (~3300 cm^{-1}) suggest hydrogen bonding interactions between –OH/–NH$_2$ groups of CS and –COOH groups introduced onto the MWCNT surface during acid functionalization. The shared peaks between the composite and its individual components confirm that both materials are present in the final product, as summarized in Table 1.

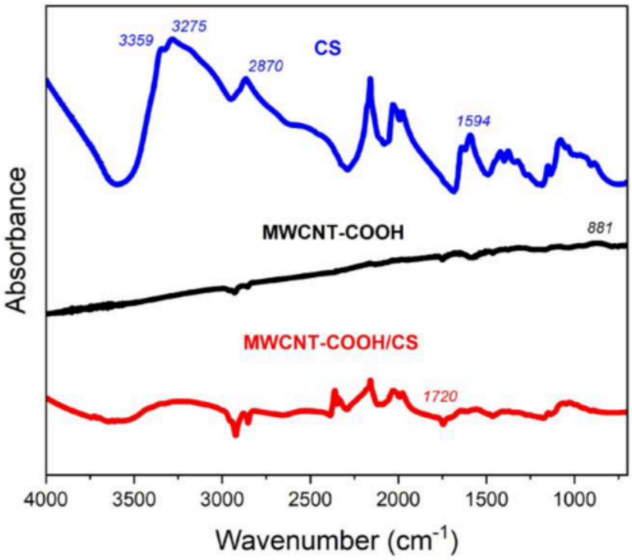

Fig. 4. FTIR spectra of pristine MWCNT, chitosan (CS), and MWCNT-COOH/CS composite, indicating key functional group regions and evidence of intermolecular interactions.

Table 1: Functional groups of MWCNT-COOH/CS based on Fig. 4

Wavenumber (cm^{-1})	**CS**	**MWCNT-COOH**	**MWCNT-COOH/CS**
3359-3275	Broad and intense bands Overlapping O-H and N-H stretching	-	Broad and intense bands Overlapping O-H and N-H stretching (A little bit shifted)
2870	C-H asymmetric stretching	-	C-H asymmetric stretching
1594	N-H bending from primary amine groups	-	N-H bending from primary amine groups
881	-	Weak absorption band C–C stretching vibrations (graphitic structures)	Weak absorption band C–C stretching vibrations (graphitic structures)
1720	-	-	Absent (Most carboxyl groups are engaged in bonding interactions and do not exist in a free form)

3.3. Surface Morphology Analysis of the Samples

The surface morphology of chitosan (CS), functionalized multi-walled carbon nanotubes (MWCNT-COOH), and the MWCNT-COOH/CS composite were investigated using scanning electron microscopy (SEM) and field emission scanning electron microscopy (FESEM), as shown in Fig. 6. Fig. 6(a) presents the SEM image of pure CS at 1000× magnification. The surface appears dense, irregular, and compact, with noticeable folded and uneven morphology [14]. This morphology is typical of chitosan films, which are known for their poor porosity and limited surface area in the absence of additional structuring agents. Fig. 6(b) shows the FESEM image of MWCNT-COOH at 50,000× magnification. The image reveals a web-like, entangled network of nanotubes resembling rope-shaped bundles. This morphology is characteristic of acid-treated MWCNTs, where the individual nanotubes remain loosely aggregated due to van der Waals forces despite partial surface functionalization [14]. The introduction of –COOH groups via acid treatment improves hydrophilicity and reactivity, but cannot entirely eliminate the natural tendency of MWCNTs to agglomerate [21]. In contrast, Fig. 6(c) illustrates the surface morphology of the MWCNT-COOH/CS composite at the same magnification (50,000×). The image displays a hybrid structure in which chitosan appears to coat or interpenetrate the MWCNT network. This dispersion suggests successful interfacial interaction between the two components, likely through hydrogen bonding and electrostatic interactions between the –COOH groups on the MWCNT and –OH or $–NH_2$ groups of CS [22]. However, some uneven distribution of CS is observed, potentially due to residual MWCNT agglomeration that limits uniform coating or matrix penetration. Such morphological features are consistent with partial phase separation and the formation of microvoids, which are advantageous for vapor adsorption due to the increased surface area and porosity [21].

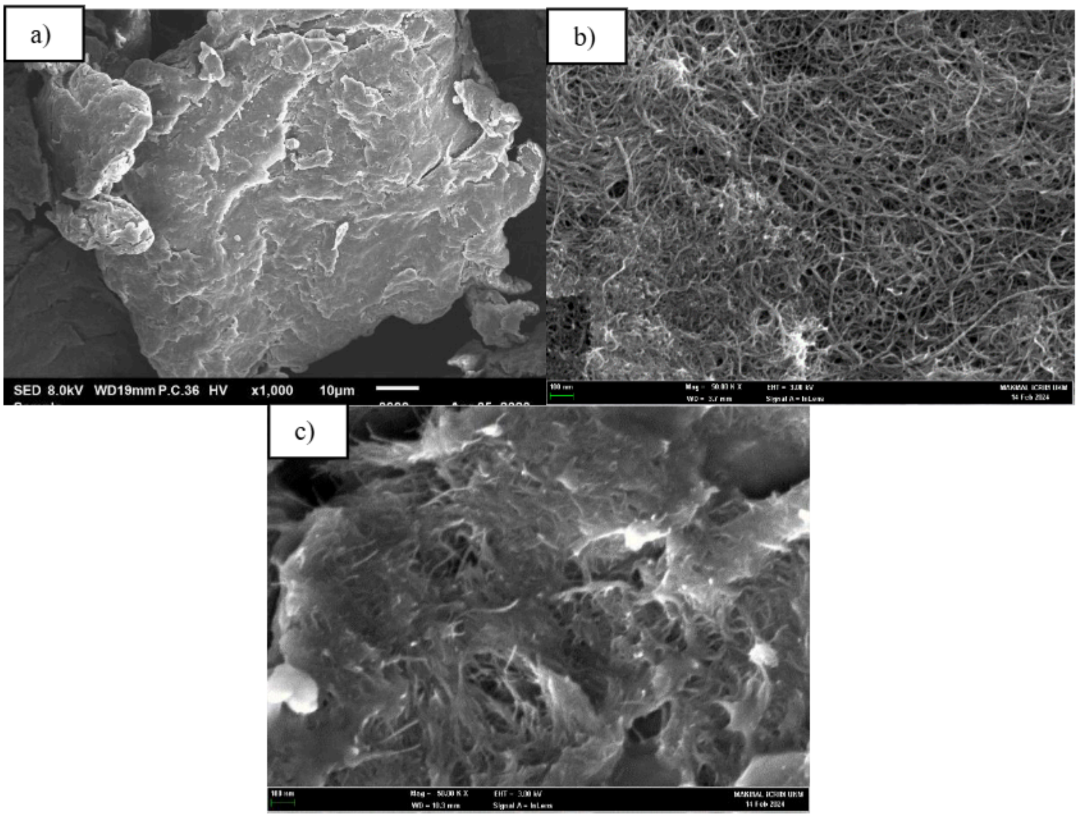

Fig. 5. Surface morphology of (a) SEM for CS magnification at x1,000 (b) FESEM for MWCNT-COOH magnification at x50,000 and (c) FESEM for MWCNT-COOH/CS composite magnification at x50,000.

3.4. Response of Sensors Towards IPA

3.4.1. Static Response of Sensors

The static adsorption response of quartz crystal microbalance (QCM) sensors coated with chitosan (CS), carboxyl-functionalized multi-walled carbon nanotubes (MWCNT–COOH), and their composite (MWCNT–COOH/CS) was evaluated upon exposure to isopropyl alcohol (IPA) vapor (Fig. 6). Hypothetically, the MWCNT–COOH/CS composite was expected to yield a lower frequency shift than pure CS because the –COOH groups on MWCNT can interact with $–OH/–NH_2$ groups in CS via hydrogen bonding, partially occupying active sites and reducing available adsorption sites for IPA. Furthermore, the incorporation of MWCNT reduces the overall hydrophilicity of the sensing layer, which may limit IPA uptake despite offering faster diffusion pathways.

In all cases, exposure to IPA caused a decrease in resonance frequency, indicating mass loading due to analyte adsorption. CS exhibited the highest frequency shift (202.84 Hz), attributed to the abundance of hydroxyl groups that form strong hydrogen bonds with IPA molecules [23]. However, it also had the longest response time (≈674 s), likely due to its dense, low-porosity morphology that hinders analyte diffusion into the bulk of the film.

Fig. 6(b) shows MWCNT–COOH layer reached equilibrium rapidly (≈20 s) but produced a moderate frequency shift of 69.73 Hz. This quick response is attributed to its high surface area and porous, entangled structure, enabling rapid IPA diffusion, while the moderate shift reflects fewer polar adsorption sites [24]. Functionalization with –COOH groups improved compatibility with IPA, consistent with Sun et al. [6], who reported enhanced adsorption for analytes with similar polarity to the dispersing solvent. Although pristine MWCNTs are largely inert, the observed response in this case is enhanced by the presence of carboxyl functional groups and the solvent compatibility with IPA.

The MWCNT–COOH/CS composite showed an intermediate performance, with a frequency shift of ~106 Hz and a response time of ~40 s. This balance reflects the combined advantages of CS's polar functional groups and MWCNT's porous network, along with enhanced dispersion due to hydrogen bonding and electrostatic interactions between the two components. The comparative behavior and mechanism of the composite sensing layers is discussed further in Section 3.5.

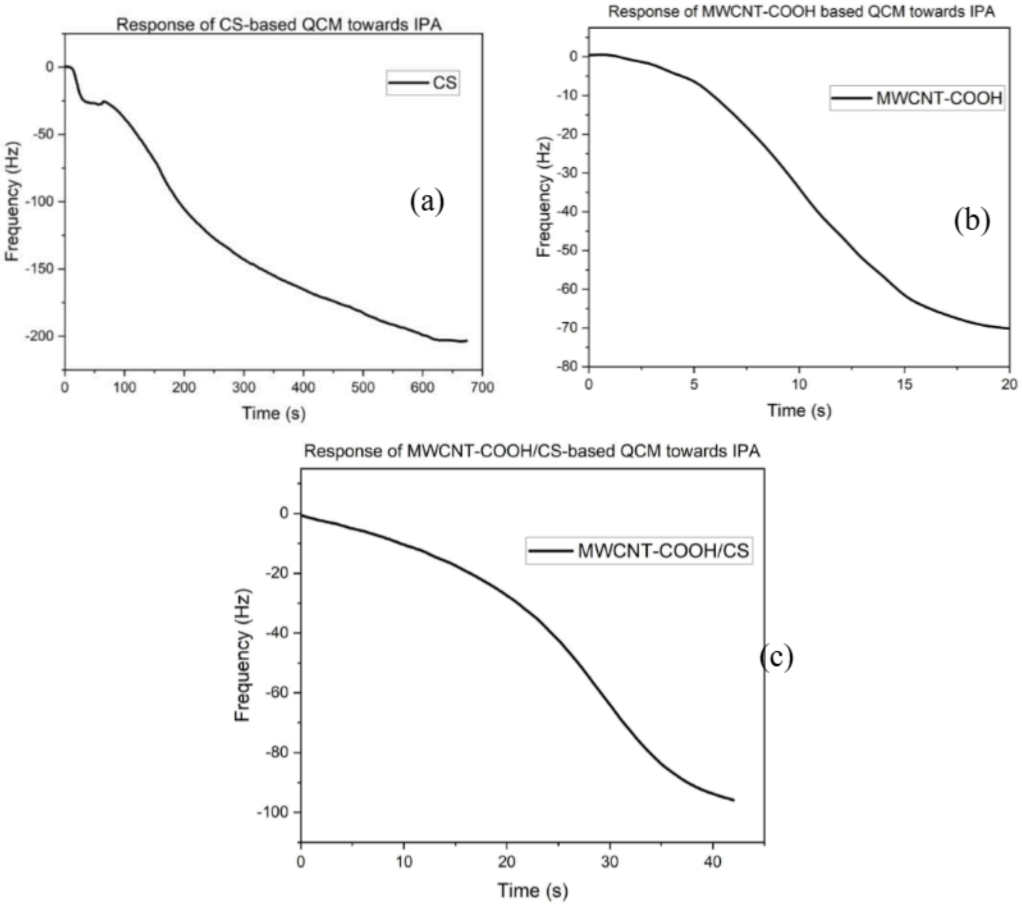

Fig. 6. Frequency shift comparison of static measurements for chitosan (CS), MWCNT-COOH, and MWCNT-COOH/CS composite upon exposure to IPA vapor.

3.4.2 Dynamic Response of Sensor

The dynamic response of the QCM sensor coated with the MWCNT-COOH/CS composite film was evaluated under varying concentrations of isopropyl (IPA) vapor, ranging from 300 to 700 ppm. This range was selected to remain below the occupational exposure limit of 400 ppm for IPA (8-hour TWA) while still enabling measurable frequency shifts for sensor performance assessment [25], [26]. The sensor's performance is illustrated in Fig. 7(a), which shows a clear, stepwise frequency shift corresponding to each IPA concentration level. Upon exposure to IPA, the QCM sensor exhibited a progressive decrease in resonance frequency, indicating mass loading due to analyte adsorption. The frequency shift increased with increasing IPA concentration, demonstrating a concentration-dependent response. This behavior confirms the sensitivity of the MWCNT-COOH/CS composite toward polar VOCs like IPA, attributed to the synergistic interactions between the hydroxyl and amine groups in chitosan and the carboxylated surface of the MWCNTs [6].

It is worth noting that the frequency shift between 500 ppm and 600 ppm increased only slightly, suggesting a temporary slowing of adsorption, possibly due to partial occupation of active sites or short-term mass transport limitations. However, the substantial increase observed at 700 ppm indicates that the sensing layer had not reached full saturation and still possessed available sites for IPA adsorption. This may be attributed to multilayer adsorption or enhanced diffusion into the bulk of the composite film at higher analyte concentrations. To evaluate the linearity of the sensor's response, a calibration curve was constructed by plotting the frequency shift against IPA concentration, as shown in Fig. 7(b). The linear regression analysis yielded an R^2 value of 0.9713, with a slope of 0.2669 Hz/ppm, indicating a strong linear correlation between the sensor output and IPA concentration within the tested range. This result aligns well with previous simulation work by Lukman Hekiem et al. [27], and supports the quantitative sensing capability of the developed QCM sensor.

Overall, the MWCNT-COOH/CS composite demonstrates a reliable and linear response to increasing IPA concentrations, making it a promising candidate for real-time VOC detection in environmental or industrial monitoring applications.

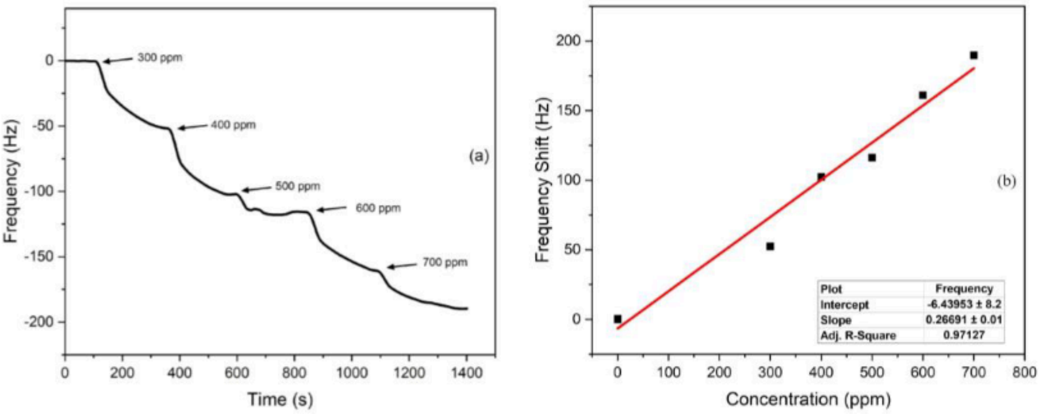

Fig. 7. (a) Dynamic response of MWCNT-COOH/CS film on QCM sensor towards 300 to 700 ppm of IPA (b) The relationship between frequency shift and concentration of IPA in ppm.

When benchmarked against previously reported IPA and alcohol vapor sensors (Table 2), the developed sensor shows a favorable balance between sensitivity and response time. Specifically, it achieved a frequency shift of ~106 Hz and a response time of ~40 seconds within the 300–700 ppm concentration range, which falls within the range of fast-response QCM sensors typically reported (<60 s). While the sensitivity (0.2669 Hz/ppm) is lower than some high-performing alcohol sensors tested at lower concentration ranges, the response time is notably faster than that of pure chitosan films (>600 s) and comparable to CNT-based sensors. This balance highlights the synergistic role of MWCNT-COOH and CS in combining diffusion kinetics with polar functional group interactions.

Table 2: Comparison of QCM-based IPA sensors in literature and present study

Sensing Material	**Sensor**	**Target Analyte**	**Concentration Range (ppm)**	**Frequency Shift (Hz)**	**Response Time (s)**	**Reference**
MWCNT-COOH/CS Composite	QCM	IPA	300–700	~106	~40	This work
Chitosan (CS)	QCM	IPA	300–700	202.84	674	This work
MWCNT-COOH	QCM	IPA	300–700	69.73	20	This work
Electrospun PBINF Nanofibers	QCM	IPA	10–400	~25 at 10 ppm	-	[28]
CS (2% Acetic Acid)	QCM	IPA	Not specified	99.3 (IPA)	~150	[17]
Graphene Coating	QCM	IPA	60–240	8-34.5 Hz	41 s (60 ppm), 55 s (120 ppm)	[29]
ZnO Colloid Spheres	QCM	IPA	15–300	~5.5k	-	[30]
Chitosan-coated QCM	QCM	IPA	20-120	50-450	-	[23]

3.5 Proposed Sensing Mechanism of MWCNT-COOH/CS composite QCM sensor for IPA detection

To further interpret the static and dynamic response behaviors observed in the previous sections, the sensing mechanism of the MWCNT-COOH/CS composite toward IPA vapor is discussed here. The detection mechanism for IPA, as shown in Fig. 8, is primarily dominated by physical adsorption. Carboxyl groups (-COOH) on MWCNT-COOH interact with the hydroxyl groups of IPA via hydrogen bonding. In addition, the hydroxyl (-OH) and amino (-NH_2) groups in IPA interact with the polar functional groups on the sensing layer. These synergistic interactions enhance analyte adsorption, leading to measurable frequency shifts and improved response characteristics. The distinct behaviors of the individual sensing layers also support this mechanism Chitosan exhibited the most significant frequency shift (202.84 Hz) due to its high density of –OH and –NH_2 groups, but the non-porous structure resulted in a slow response time (674 s). In contrast, MWCNT-COOH achieved a very fast response (20 s) owing to its high surface area and porous network, though with a lower frequency shift (69.73 Hz). The composite material integrates these advantages, enabling efficient adsorption sites from CS while benefiting from the rapid diffusion pathways provided by MWCNTs, thereby producing a balanced sensing performance.

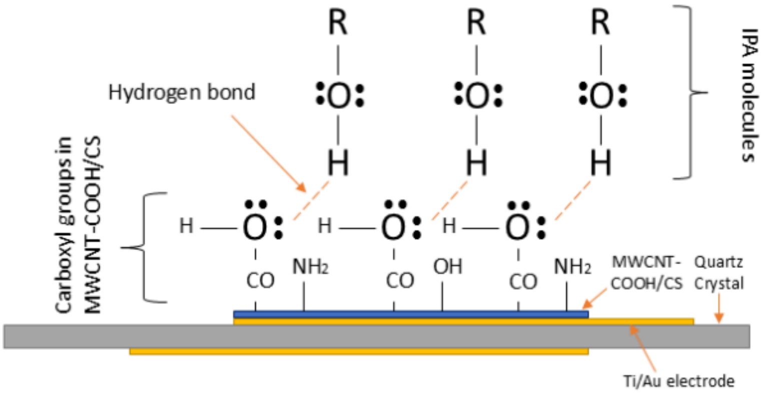

Fig. 8. Schematic of the interaction between MWCNT-COOH/CS and IPA.

A MWCNT-COOH/CS composite was successfully synthesized and applied as a QCM sensing layer for IPA detection. FTIR and FESEM confirmed functional integration and favorable porous morphology. The sensor demonstrated a balanced static response (~106 Hz, ~40 s) and a linear dynamic response ($R^2 = 0.9713$) across 300–700 ppm IPA. When compared to previous work using the identical composite for formaldehyde detection (frequency shift ~196.6 Hz, sensitivity ~23.48 Hz/ppm) [19], the current IPA sensor displays complementary performance characteristics, with faster response time but slightly lower sensitivity. This underscores the versatility of the MWCNT-COOH/CS platform. Future work will extend its application to other VOCs (e.g., acetone and ethanol) and explore array-based selectivity for real-world monitoring.

4. CONCLUSION

A MWCNT-COOH/CS composite was successfully synthesized and applied as a QCM sensing layer for IPA detection. FTIR and FESEM confirmed functional integration and favorable porous morphology. The sensor demonstrated a balanced static response (~106 Hz, ~40 s) and a linear dynamic response ($R^2 = 0.9713$) across 300–700 ppm IPA. When benchmarked against previous reports, the composite exhibited moderate sensitivity (0.2669 Hz/ppm) compared to higher-frequency-shift sensors such as ZnO colloid spheres. Still, it achieved a faster response time than polymer-based systems operating within similar concentration ranges. This indicates that the developed sensor offers a practical trade-off between sensitivity and speed, with the added advantage of simple fabrication. These findings highlight its potential for real-time IPA monitoring applications. Further studies are needed to investigate internal and external factors influencing sensing layer performance.

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