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Original Research

Study of the Mechanism of Response of Thin Layer Polyaniline to the Aroma of Ginger and Clove Essential Oils

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Abstract: Polyaniline (PANI) is widely used as a gas-sensing Material because it has an active group that can interact with various chemical compounds. This study examined the responses of PANI thin layers to the aromas of ginger oil and clove oil to understand how the chemical properties of the target molecules Influence sensor performance. The test results showed that the sensor had high stability and repeatability in both samples. In ginger oil, the interaction between gingerol–zingiberene with the amine and amine groups of PANI resulted in an average response time of 34.33 ± 0.47 s and a recovery time of 35.33 ± 0.47 s (at a volume of 1 ml). Meanwhile, clove oil showed a faster response time (30.67 ± 0.47 s) but stronger interactions during the recovery phase. A Relative Standard Deviation (RSD) below 2% throughout the test cycle indicates that the adsorption-desorption mechanism is reversible and consistent. These results confirm that the chemical properties of the target molecule determine the response's kinetic profile, while the PANI microstructure provides crucial stability in the design of reliable scent sensors.

Keywords: polyaniline, sensor, aroma, ginger, cloves

1. Introduction

Conductive polymer-based sensors have become a major focus in the development of volatile compound detection technologies because they offer high sensitivity and can operate at room temperature [1]. The working principle is based on changes in the electrical properties of polymers, such as voltage or resistance, due to the interaction between the active group on the sensor surface and the target molecule, so that methods such as the Four Points Probe (FPP) remain relevant to use even if the measured parameter is in the form of voltage. Among various conductive polymers, polyaniline (PANI) stands out for its environmental stability, ease of synthesis, and adaptable conductivity properties through doping processes [2,3]. This flexibility allows PANI to be used across a wide range of sensor platforms, including thin-film-based sensors on conductive substrates, which are an important basis for detecting odours and gases in the air [4].

Various studies have shown that PANI is effective at detecting volatile organic compounds (VOCs), including ammonia, ethanol, acetone, and aromatic compounds, at room temperature [5]. Several factors, including the layer morphology, the structure of the supporting nanomaterials, the level of doping, and the type of interaction between the target molecule and the sensor surface, determine the performance of a PANI sensor. Nonetheless, one of the main challenges is the decreased sensitivity and stability of the sensor due to repeated exposure to volatile molecules, which can alter the coating's structure or conductivity [6]. Therefore, more systematic studies are needed to evaluate how exposure conditions affect sensor response over time.

The essential oils of ginger (*Zingiber officinale*) and cloves (*Syzygium aromaticum*) contain major volatile compounds such as gingerol, zingiberene, and eugenol [7]. Thin layers of polyaniline (PANI) have active amine (–NH–) and imine (=N–) groups, so that specific interactions with the functional groups of these volatile compounds can occur through hydrogen bonds, π–π interactions, and electrostatic tensile forces. These interaction mechanisms can affect the sensor's performance characteristics, including response time, recovery time, and sensitivity. The use of PANI as an active ingredient for the detection of spice aromas not only supports the development of environmentally friendly sensor technology but is also relevant for food safety applications and for monitoring the quality of herbal ingredients [6,8].

PANI has been extensively tested for detecting volatile organic compounds, including ammonia, ethanol, and acetone. Studies comparing its response to essential oil molecules are still limited. Compounds such as gingerol–zingiberene in ginger and eugenol in cloves differ in molecular size, polarity, and functional groups, which may affect hydrogen bonding, π–π interactions, and electrostatic interactions with the amine and imine groups of PANI. These differences can significantly affect sensitivity, response time, and short-term repeatability across repeated exposures with varying analytical volumes.

Based on this background, the novelty of this study lies in the use of two aromas of spice essential oils, namely ginger (*Zingiber officinale*) and cloves (*Syzygium aromaticum*), as volatile analyzers that have dominant components and different chemical characters, as well as in the analysis of the response of PANI thin layers in repeated exposure with variations in exposure volume. The study not only compares the magnitude of the response but also emphasizes differences in sensitivity, response times, and recovery times between the two scents, thereby providing additional insights into the role of target-molecule selection in designing PANI-based scent sensors. In line with this novelty, this study aims to compare the electrical response of PANI thin layers to the aroma of ginger and clove essential oils across varying exposure volumes (0.5, 1.0, 1.5 mL) and repeated exposure (three adsorption–desorption cycles). The parameters analyzed included sensitivity, response time, and recovery time to evaluate the differences in the response patterns of the two scents under the same test conditions.

2. Method

A thin layer of polyaniline (PANI) was synthesised via electro-polymerisation using cyclic voltammetry (CV), as applied in this study [9,10]. The electrolyte solution consists of aniline monomer 0.25 M (≥ 99%, Sigma-Aldrich, USA), HCl 0.5 M (≥ 37%, CIMS), and aqueduct (pH 8.6 laboratory). Deposition is performed at a potential range of –0.4 V to +1.0 V (100 mV/s scan rate, 10 cycles) using a three-electrode cell with ITO glasswork electrodes (8-12 Ωsq, Sigma-Aldrich, USA), Ag/AgCl reference, and a Pt. The electrical bias during the test was supplied by a DC power supply (Texio Technology Corp., Japan). At the same time, the voltage was monitored with a Sanwa (Sanwa Electric Instrument Co., Ltd., Japan) digital multimeter. Test samples of ginger and clove essential oils were obtained from local suppliers in Surabaya, Indonesia.

Chemical characterization was performed using Fourier Transform Infrared Spectroscopy (FTIR) ATR mode to identify the main functional groups of ginger oil (*Zingiber officinale*) and clove oil (*Syzygium aromaticum*) with reference [11,12,13] for ginger, and [14,15] for cloves. The potential interaction was analyzed by matching the functional groups in the essential oil FTIR results with the typical imine (=N–) and secondary amine (–NH–) groups reported in the literature [16,17]. The surface morphology of PANI after sensing was observed using Scanning Electron Microscopy (SEM) to evaluate particle size distribution and structural changes resulting from repeated aroma exposure, as reported in [18,19], which noted that PANI morphology affects gas diffusion and the active surface area for target molecular interactions.

The sensing test was carried out in a closed chamber using the Four-Point Probe (FPP) method, with the configuration shown in Figure 1 of the sensor measurement section. Electrical property measurements were carried out using the Four-Point Probe (FPP) method with a constant current of 0.7 mA to monitor changes in the PANI layer's electrical properties upon exposure to volatile compounds. A fan is used to prevent airflow from directly hitting the sensor surface, minimizing mechanical interference with the probe contacts. Two outer probes inject a constant current into the sample, with the measurement current held constant throughout the test. In comparison, two inner probes measure the potential difference (ΔV) across the mm probes on the coating. Voltage changes during exposure are used as an indicator of coating conductivity. Since the current is kept constant, the change is directly proportional to the change in the layer's effective electrical resistance. So, a decrease in voltage during exposure is interpreted as a decrease in resistance (an increase in conductivity) under the same measurement conditions.

Before the analyte's exposure begins, the signal is recorded under clean-air conditions until the baseline is relatively stable; then, recording is performed continuously during the response (exposure) and recovery (discontinued exposure) stages. To minimize noise in the measurements, the probe contact position is kept fixed during the test, the electrical connection is maintained, and all data are processed uniformly across all curves. Analyte exposure is defined to begin at time t_0, which is when the chamber is tightly closed after the sensor and essential oils are placed in the chamber. Response time is defined as the interval from t_0 to the time when the signal meets the set response criteria. In contrast, recovery time is defined as the interval from the time exposure is stopped (the chamber is opened and the analyte is ejected) until the signal returns to near baseline.

The PANI layer on the ITO is connected to a signal recording device to monitor voltage changes due to exposure to volatile compounds. The chamber's internal volume is 4.485 L (23 cm × 15 cm × 13 cm) (Figure 1). The test procedure was performed separately for ginger oil and clove oil, each with three volume variations (0.5 mL, 1.0 mL, and 1.5 mL) and three adsorption–desorption cycles. These values do not represent the calibrated vapour concentration (ppm or mg/m^3) on the sensor surface because the headspace concentration is affected by evaporation and adsorption conditions and was not directly measured in this study. One cycle consists of a response stage (a decrease in voltage due to the interaction of the target molecule with the imine and amine groups in PANI) and a recovery stage (an increase in voltage towards the initial state after exposure is stopped). The recorded voltage changes are used to calculate the sensitivity, response time, and sensor recovery time.

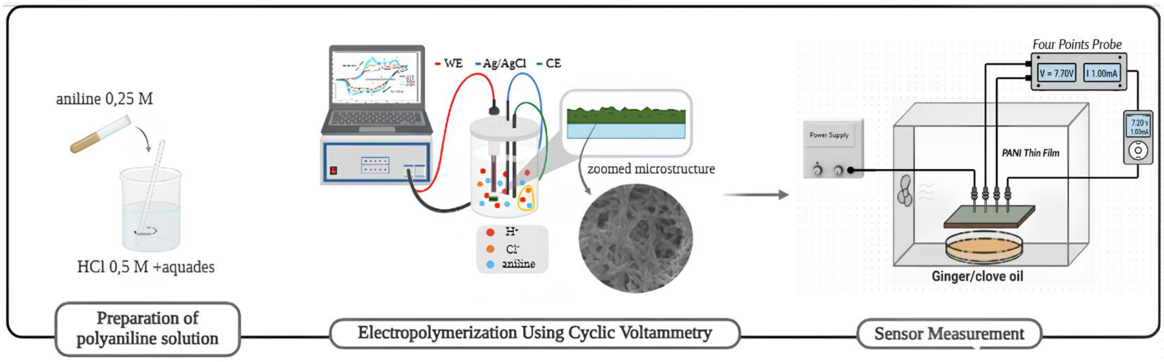


Figure 1: Polyaniline thin-layer synthesis scheme and sensitivity measurement.

3. Results and discussion

3.1. Characterization of PANI thin layer

The Cyclic Voltammetry (CV) curve in Figure 2 shows a peak of oxidation at +0.82 V and a reduction peak at +0.21 V, indicating a reversible process between the forms of leukoemeraldine, emeraldine, and pernigraniline in polyaniline. This scheme is consistent with the research [9,10] on the stages of aniline polymerization in acidic media. During the initial cycle, the coating undergoes a gradual colour change from blue (pernigraniline) to the typical green of emeraldine salt. This colour change is caused by the gradual protonation of the imine and secondary amine groups, which increases conductivity and yields a stable green colour in the final cycle.

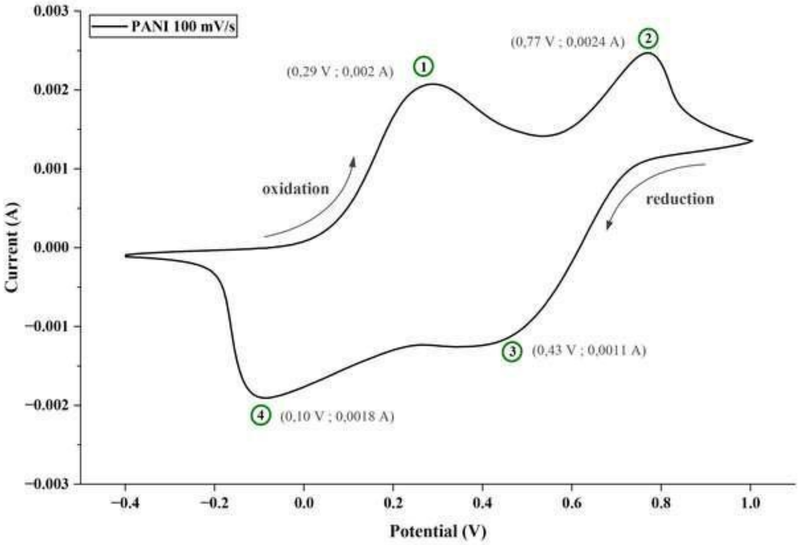


Figure 2: Cyclic Voltammetry (CV) Curve of PANI electro polymerization process on ITO substrate.

The FTIR spectrum of the PANI layer after the sensing process in figure 3 shows a C–N strain band (secondary amine) at 1298 cm^{-1}, an aromatic C=C at 1564 cm^{-1}, an aromatic C–C at 1482 cm^{-1}, as well as a typical band N=Q=N at 1155 cm^{-1} representing the structure of an oxidized quinoid in the PANI conjugation chain. The presence of this quinoid band indicates that the conjugation pathway and conductive properties of PANI are maintained after interaction with volatile molecules [16,17]. Changes in band intensity compared to the literature indicate the involvement of imine (=N–) and secondary amine (–NH–) groups in hydrogen bonding or π–π interactions with the target compound.

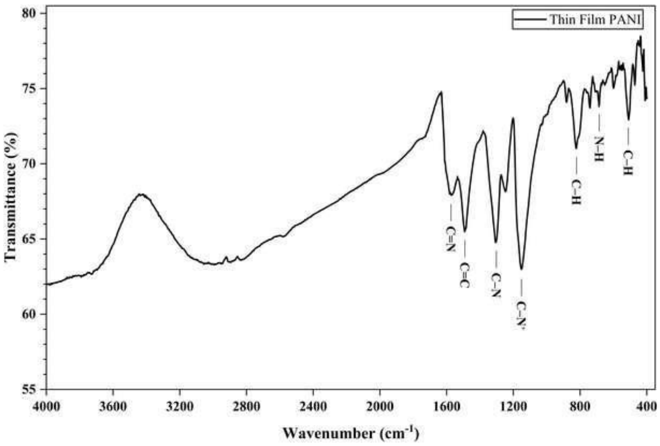
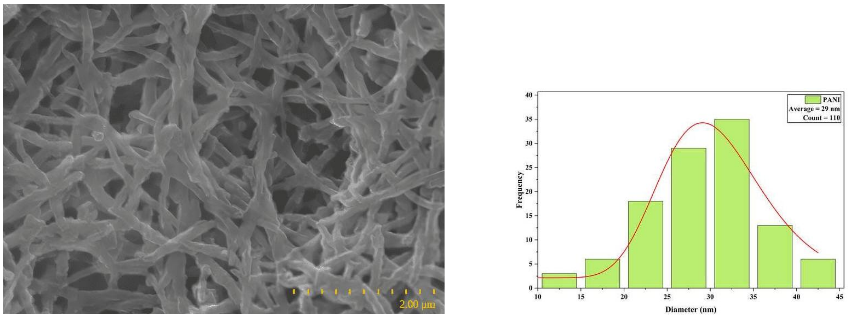


Figure 3: Thin-layer FTIR spectrum PANI/ITO.

The size distribution shown in Figure 4(b), spanning 80-750 nm, provides critical physical evidence supporting the hypothesis that incompletely desorbed analyte molecules partially occupy active sites within the PANI layer. Specifically, the observed buildup of material suspected to be essential oil residues provides experimental evidence of this phenomenon, particularly for clove oil, which exhibits a higher affinity for the polymer matrix. This accumulation acts as a physical barrier, obstructing diffusion pathways and covering active sites, thereby hindering analyte access during subsequent testing cycles. Such conditions are consistent with the nanostructure approach [18, 19], highlighting that any alteration in the nanoscale morphology or the blocking of the high surface area-to-volume ratio can directly diminish the sensor's overall performance [4].



(a) (b)

Figure 4: Morphology of PANI thin layer (a) using SEM at 20,000× magnification, (b) Particle size distribution curve.

3.2 Sensor response results

The FTIR spectrum of ginger oil in Figure 5 shows a peak of phenolic –OH at 3365 cm⁻¹, aliphatic C–H at 2924 cm⁻¹, C=O carbonyl at 1738 cm⁻¹, and aromatic C=C at 1622 cm⁻¹. This pattern aligns with the characteristics of gingerol and zingiberene, commonly found in ginger oil (21,13). Meanwhile, clove oil FTIR spectra from the literature show peaks at 3350 cm⁻¹ (phenolic –OH), 1264 cm⁻¹ (C–O–C ether), and 1032 cm⁻¹ (phenolic C–O), confirming eugenol's dominance (22, 14).

In this study, the mechanism was interpreted based on changes in the electrical properties of the ITO/PANI layer during analyte exposure and upon its cessation. When exposure is stopped, and the test chamber is flowing clean air, the sensor signal gradually returns to near its initial value. This suggests that signal changes mainly occur during exposure and recovery after exposure is stopped, which can generally be attributed to adsorption–desorption or changes in the level of doping in PANI [23]. Based on differences in the main functional groups of volatile compounds and the chemical properties of PANI (imine and amine groups), hydrogen bonds, dipole–dipole interactions, and π–π interactions are hypothesised to contribute to the different responses of ginger and clove essential oils. However, this study presents these as hypotheses, as it has not provided direct experimental evidence or quantitative theoretical support.

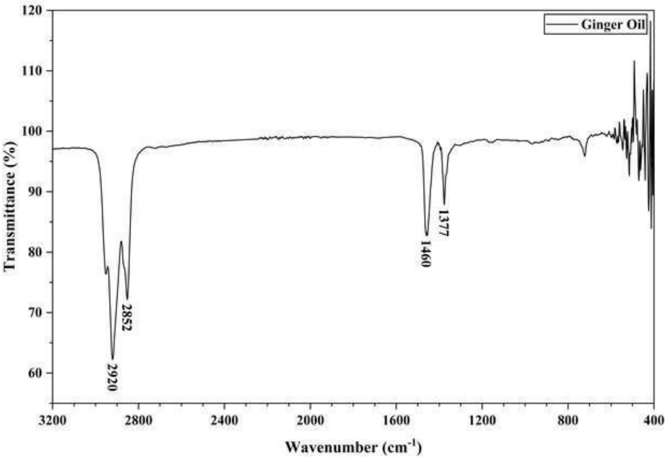
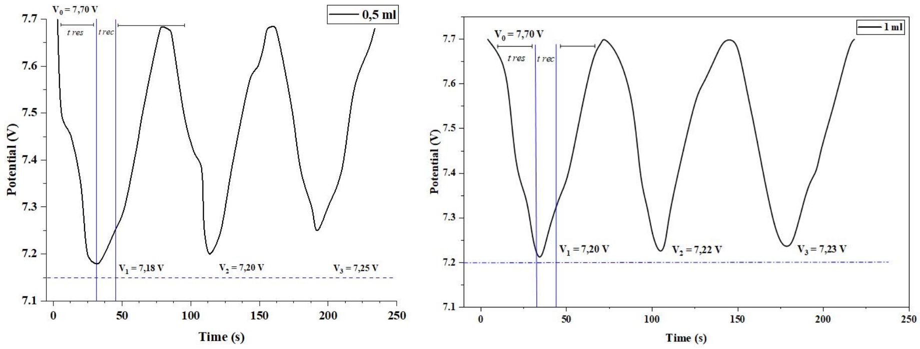
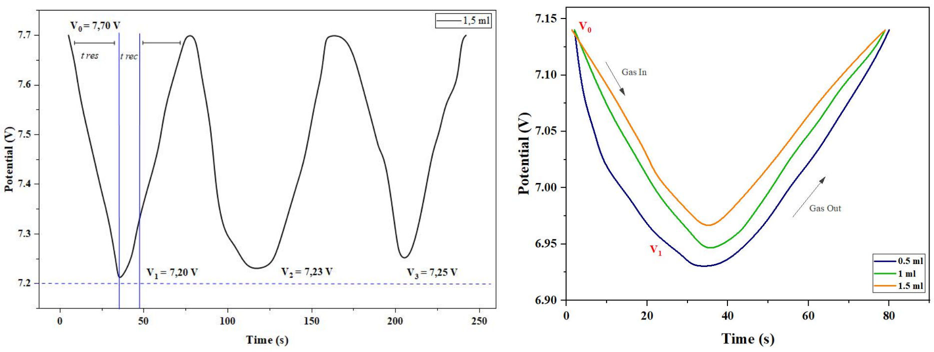


Figure 5: FTIR spectrum of ginger oil (Zingiber officinale) with the marking of the main peak of volatile compounds.

Tests using *the Four Points Probe* (FPP) method showed a clear difference in stress response between ginger oil and clove oil across three exposure volumes (0.5, 1.0, and 1.5 mL) during the three exposure–release cycles. In the ginger oil shown in Figure 6(d), an increase in volume is typically accompanied by increased sensitivity and shorter response times, indicating that the number of volatile molecules reaching and interacting with the PANI active site increases. In contrast, in clove oil, the highest sensitivity is obtained at the lowest volume (0.5 mL) and decreases at larger volumes, as shown in Figure 6(a). This indicates that the sensor's active site is saturated or blocked at high exposure levels.



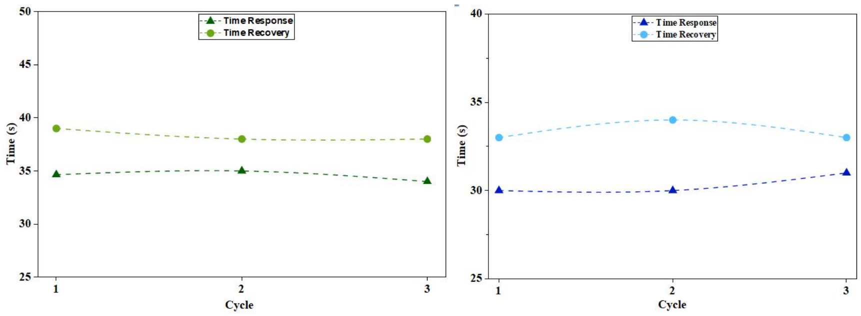
(a) *(b)*



(c) *(d)*

Figure 6: PANI sensor response to exposure to volatile aromas: (a) 0.5 mL (3 cycles) clove oil, (b) 1.0 mL clove oil (3 cycles), (c) 1.5 mL (3 cycles) clove oil; 1,0; and 1.5 mL (1 cycle).

The stability and repeatability test was carried out by repeatedly exposing the scent to three cycles for each essential oil sample. The results of visual observation of the sensor's response profile to the aroma of ginger oil and clove oil are presented in Figure 7(a) and Figure 7(b), respectively. Based on these data, ginger oil has a very stable response time of 34.65 s to 34 s, with a consistent recovery time of 39 s to 38 s.



(a) *(b)*

Figure 7: PANI sensor response time and recovery time: (a) ginger oil at three exposure cycles, (b) clove oil at three exposure cycles.

Based on the quantitative data in Table 2, the sensor shows excellent stability across volume variations. Similarity of responses across multiple cycles showed short-term *repeatability* under the same test conditions, with small variations between cycles, as indicated by SD/RSD. Intersensory-to-sensor reproducibility (n≥3) and interday testing have not been performed, so long-term stability and fabrication variation have not been evaluated in this study. This characteristic indicates the adsorption–desorption efficiency maintained over repeated cycles, which indicates that the diffusion pathways in the PANI microstructure remain open and do not degrade. Similar results were observed with clove oil, which showed a stable response time of 30–31 s and a recovery time of 33–34 s through the third cycle. Overall, the statistical parameters summarised in Table 2 indicate the stability of the interaction between the volatile molecules and the PANI sensor surface, enabling reversible detection with high precision.

Table 1. Statistical analysis of response and recovery characteristics

Sample	Volume (mL)	Cycle	Response time (s)	Recovery time (s)	Standard Deviation Response Time	Standard Deviation Recovery Time	Relative Standard Deviation Response Time (%)	Relative Standard Deviation Recovery Time (%)
Ginger	0.5	1	33	43	0.4714	0.81650	1.44308	1.94404
		2	33	41				
		3	32	42				
	1.0	1	35	36	0.4714	0.47140	1.37302	1.33416
		2	34	35				
		3	34	35				
	1.5	1	34.65	39	0.4143	0.47140	1.19921	1.22975
		2	35	38				
		3	34	38				
Clove	0.5	1	30	33	0.4714	0.47140	1.55408	1.40021
		2	31	34				
		3	30	34				
	1.0	1	30	34	0.4714	0.47140	1.53719	1.35982
		2	31	35				
		3	31	35				
	1.5	1	30	33	0.4714	0.47140	1.55408	1.41421
		2	30	34				
		3	31	33				

The correlation with the sensitivity data contained in Figure 8 strengthens this interpretation. In ginger oil, the smaller, polar gingerol molecule can interact reversibly with the imine (=N–) and secondary amine (–NH–) groups of PANI through hydrogen bonding and adaptive π–π interactions [24]. This mechanism facilitates rapid desorption in subsequent cycles, allowing sensitivity to continue increasing with increasing exposure volume. In contrast, in clove oil, eugenol molecules are larger, contain methoxy groups, and have aromatic rings that tend to form strong hydrogen bonds, dipole–dipole interactions, and π–π stacking interactions. As explained, this kind of interaction can cause saturation and blockage of some active sites at high concentrations, resulting in decreased sensor performance even though the number of exposed molecules is greater [25, 26].

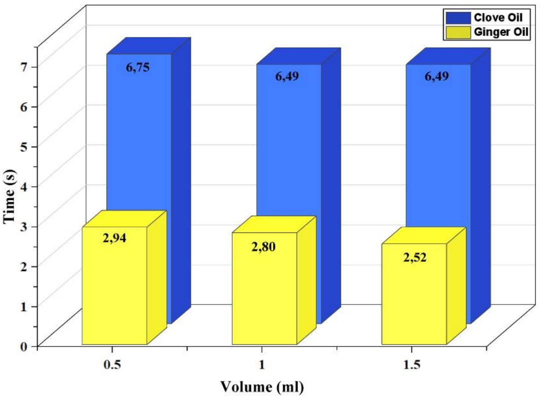
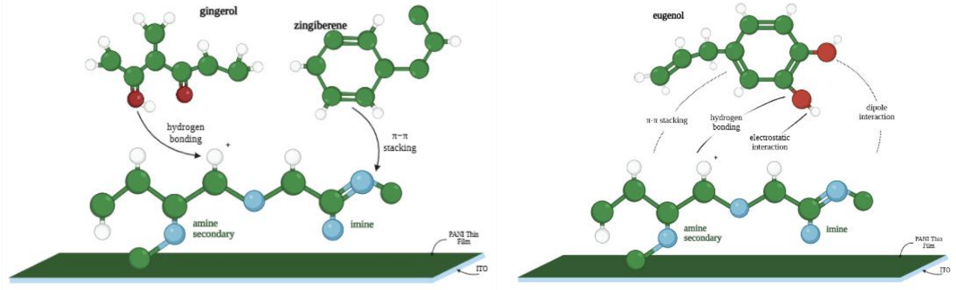


Figure 8: Graph of the sensitivity of the PANI sensor to ginger and clove oil at various exposure volumes.

Visualization of the interaction mechanism strengthens the correlation between the type of volatile molecule and the sensor's performance. In ginger oil, as shown in Figure 9(a), gingerol is hypothesised to have a hydrogen bond between the –OH or –C=O group and the imine (=N–) and secondary amine (–NH–) groups of PANI, as well as reversible π–π *stacking*. These interactions maintain the availability of active sites, enabling rapid desorption and increasing sensitivity at larger exposure volumes. In contrast, in the clove oil shown in Figure 9(b), eugenol is hypothesised to have a higher affinity to PANI surfaces (e.g., through polar and/or aromatic interactions), which may cause some of the active sites to be more easily covered so that the diffusion of new molecules is inhibited. Sensitivity decreases to repeated exposure, especially at large volumes. This may partially mask the active sites that inhibit the diffusion of new molecules, leading to decreased sensitivity and slower responses to repeated exposures, especially at large volumes.



(a) *(b)*

Figure 9: The interaction mechanism of functional groups on the PANI sensor: (a) gingerol and zingiberene from ginger oil, (b) eugenol from clove oil.

4. Limitations

This study shows that PANI-based sensors exhibit high stability and repeatability in short-term cycling tests. However, there are some limitations to consider. First, the long-term stability and shelf life of the sensors were not evaluated in this study. Secondly, environmental factors such as humidity and ambient temperature, which can affect PANI conductivity, were not specifically controlled during the test. Third, the sensing sensitivity was reported based on injected liquid volume rather than calibrated vapour concentrations (e.g., ppm), which limits direct quantitative comparison with other literature. Fourth, the proposed interaction mechanisms, such as hydrogen bonding and π–π interactions, remain hypothetical, as they have not been supported by direct in situ characterisation or theoretical simulations. Finally, this study focuses on the performance of a single sensor; Therefore, the reproducibility between sensors from different fabrication groups still needs to be investigated further. Future research will focus on addressing these factors to improve sensor reliability in practical applications.

5. Conclusion

Thin-layer polyaniline (PANI) exhibits a different response to volatile compounds from ginger oil and clove oil, which is influenced by differences in the main chemical properties of their components, as reflected in differences in response curves, sensitivity, and response and recovery times under the same test conditions. Gingerol and zingiberene in ginger oil are hypothesised to interact weaklier and more readily, regardless of PANI (amine/imine) active sites, leading to faster recovery and a response that tends to increase with greater exposure volumes. In contrast, the eugenol in clove oil is hypothesised to have a higher affinity for the PANI layer, leading to slower recovery and decreased sensitivity at higher volumes, suggesting potential saturation or blockade of active sites at high exposure levels. However, the specific interaction mechanism in this study is presented as a hypothesis as it has not been supported by direct characterization of bonding/adsorption; thus, the interpretation focuses on the correlation between functional groups, response–recovery patterns, and measured sensitivity changes. Across the two analytes tested (ginger oil and clove oil), these results demonstrate the sensor's ability to differentiate responses under the same test conditions. However, broader selectivity claims require additional interference and analyte tests.

Declarations

Availability of data and material: The datasets used and analyzed during the current study are available from the corresponding author on reasonable request.

Author contributions: Dinda Hana Murty Wardah conceived and designed the research framework; Dinda Hana Murty Wardah carried out the experimental and computational work; Nugrahani Primary Putri provided critical guidance on the analytical methods and interpretation of results; Dina Rizqia Rohmah performed the data analysis; Nur Fitri Indah Lestari prepared the initial draft of the manuscript. All authors have read and approved the final manuscript. All authors contributed to editorial changes in the manuscript. All authors have participated sufficiently in the work and agreed to be accountable for all aspects of the work.

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