

*Original Research*

# Tailored Nitrogen Sites on Biomass-Derived Activated Carbon for Enhanced Formaldehyde Detection at Room Temperature

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## ABSTRACT

This research introduces nitrogen atom into biomass-derived activated (AC) to enhance their sensitivity to formaldehyde vapor at room temperature. Nitrogen-doped activated carbon (N-AC) derived from coconut shells was successfully prepared by heat treatment with ammonia (NH<sub>3</sub>) at 800°C. The atomic ratio of nitrogen to carbon in N-AC is approximately 4.1 at%. AC and N-AC were separately mixed with ethanol and then dropped onto printed circuit board with Au/Cu electrodes, serving as the sensor substrate. As-fabricated AC and N-AC sensors were exposed to formaldehyde concentrations ranging from 1 to 100 parts per million (ppm) at room temperature. N-AC sensor demonstrated a sensor response approximately 5- to 6-fold higher than that of the AC sensor. The enhancement of formaldehyde sensitivity is attributed to the increased surface area, porosity and nitrogen doping into AC. The sensing mechanism can be explained by charge transfer from formaldehyde molecules to sensing materials. Nitrogen-doped sites play a crucial role in improving formaldehyde adsorption due to their high binding energy and significant charge transfer, resulting in an enhanced sensor response. This work proposes an effective method involving NH<sub>3</sub> heat treatment for biomass-derived AC to improve surface area, porosity, and nitrogen doping of AC to enhance the sensitivity of formaldehyde detection at room temperature.

## INTRODUCTION

Air pollution caused by volatile organic compounds (VOCs), is a serious concern due to its harmful effects on human health and the environment. VOCs can lead to health problems such as lung inflammation, respiratory damage, cancer, and death at high levels (Ogbodo et al. 2022, Shen et al. 2024). The control of air quality is a necessary and urgent issue. High-performance liquid chromatography and gas chromatography-mass spectrometry are excellent conventional methods for analyzing VOCs, with high sensitivity, selectivity, and reliability (Reinnig et al. 2008, Delgado-Rodríguez et al. 2012, Xu et al. 2023). Nevertheless, the drawbacks of high-performance liquid chromatography and gas chromatography-mass spectrometry include high cost, complex procedures, time consumption, lack of real-time monitoring, and requiring expert technicians to operate equipment. Recently, gas sensor technology has played a crucial role in monitoring air pollution. The advantages of gas sensors is a simple operation, low cost, fast response time, portability and real-time monitoring. Activated carbon (AC) is a porous material with high specific surface area, porosity, well-defined pore size distribution and chemical stability (Xue et al. 2019), which is suitable for application as sensing materials in gas sensor technology. To achieve the sustainable development goals (SDGs), the production of AC from agricultural waste has attracted considerable attention (Chew et al. 2023, Neme et al. 2022, Ukanwa et al. 2019). Using agricultural waste to produce AC supports waste reduction and resource efficiency, which promotes responsible consumption and production (SDG 12). A gas sensor based on biomass-derived AC for detecting VOCs such as acetone, ethanol, methanol vapors, and ammonia gas was proposed (Ranasinghe et al. 2024). Sensor-based on AC shows the response to ammonia gas and VOCs, particularly methanol vapor. To enhance the sensing performance, biomass-derived AC has been combined with metal oxides and polymers for the detection of ammonia (NH<sub>3</sub>) (Travlou et al. 2017), hydrogen sulfide (Bibi et al. 2023, Luo et al. 2023, Rodiawan et al. 2024), acetone, and ethanol (Chao et al. 2019, Jena et al. 2022). Although AC combined with metal oxides or polymers significantly improves sensing performance, AC-metal oxide-based gas sensors require high-temperature operation and consume high power to achieve high performances, whereas AC-polymer-based sensors suffer from poor thermal stability, limited selectivity, and degradation. Heteroatom doping, such as nitrogen atom, into the surface of biomass-derived AC, is another approach to enhance the adsorption of NH<sub>3</sub> molecules (Travlou et al. 2015, Travlou et al. 2016, Kyokunzire et al. 2024). Nitrogen atoms were incorporated into biomass-derived AC through nitric acid soaking and melamine heat treatment. It can be found that nitrogen sites play an important role in NH<sub>3</sub> adsorption. So far, biomass-derived AC, combined with metal oxides, polymers, and heteroatom doping have been widely used for the detection of a few gases and vapors, such as NH<sub>3</sub>, hydrogen sulfide, acetone, ethanol, and methanol.

Formaldehyde is one of the toxic VOCs that is released from various sources such as building materials, furniture, tobacco smoke, and combustion processes. Formaldehyde can irritate of skin, eyes, nose, and throat, inflammation bronchi and lungs, nasal cavity cancer, and leukemia (Golden 2011, Kim et al. 2011). The National Institute for Occupational Safety and Health (NIOSH) defines the immediately dangerous to life or health

(IDLH) concentration for formaldehyde as 20 parts per million (ppm), and the permissible short-term exposure limit (STEL) as 2 ppm in air over 15 min (Huang et al. 2024). Therefore, monitor and early warning for formaldehyde exposure are essential to ensure the occupational safety. In this work, formaldehyde sensors were fabricated using AC and nitrogen-doped AC (N-AC) as the sensing materials. Coconut shells were used as the biomass source for producing AC. N-AC was prepared by heat treatment under an  $\text{NH}_3$  atmosphere. N-AC sensor exhibited remarkably improved formaldehyde detection at room temperature compared to the undoped AC.

## 2. MATERIALS AND METHODS

### 2.1. Preparation of Nitrogen-Doped Activated Carbon

Coconut shell-derived AC (VPAC-P4/1050)-supported FILTER MATCH Co., Ltd. (Thailand) was placed into a horizontal tube furnace under argon gas with a flow rate of 500 mL/min. The temperature was raised from room temperature to 800°C at a rate of 5°C/min. AC was treated at 800°C under a mixture of argon (100 mL/min) and ammonia (50 mL/min) gases for 60 min. Finally, as-doped samples were cooled to room temperature under argon gas (500 mL/min). Both AC and N-AC were ground into a fine powder.

### 2.2. Characterization Techniques

The morphologies were characterized by scanning electron microscopy (SEM, JEOL, JSM-7800F). The crystallinity was analyzed by Raman spectroscopy (Horiba LabRam HR) using an Ar laser light source with a wavelength of 532 nm. X-ray photoelectron spectroscopy (XPS, AXIS UI-traDLD, Kratos Analytical) was utilized to analyze the surface elements and chemical states. Surface area and porosity were characterized by the Brunauer-Emmett-Teller (BET, Micromeritics, TriStarII 3020) from the nitrogen adsorption-desorption isotherms at 77 K.

### 2.3. Gas Sensor Fabrication and Measurement

10 mg of AC and N-AC were separately dispersed in 10  $\mu\text{L}$  of ethanol using ultrasonication for 60 min. A 50  $\mu\text{L}$  of the mixed sample-ethanol solution was dropped onto the printed circuit board in 30 drops while maintaining the sensor substrate at 80°C during the drop-casting process. To evaporate the residual solvent in the sensing materials, as-fabricated AC and N-AC sensors were kept in an oven at 80°C for 60 min. The percent of the sensor response (SR%) was recorded as the change in the electrical resistance under air and formaldehyde atmospheres using a multimeter (UNI-T; UT71D). The SR% can be calculated using Equation (1), whereas  $R_{\text{HCHO}}$  and  $R_{\text{air}}$  are the electrical resistance of the sensor under formaldehyde vapor and air atmosphere, respectively. As-fabricated AC and N-AC sensors were separately placed in a stainless-steel chamber and kept under an air atmosphere for 10 min to provide the baseline signal of the sensors. The formaldehyde was injected into the detection system and kept in the closed system for 10 min to observe the change in electrical resistance

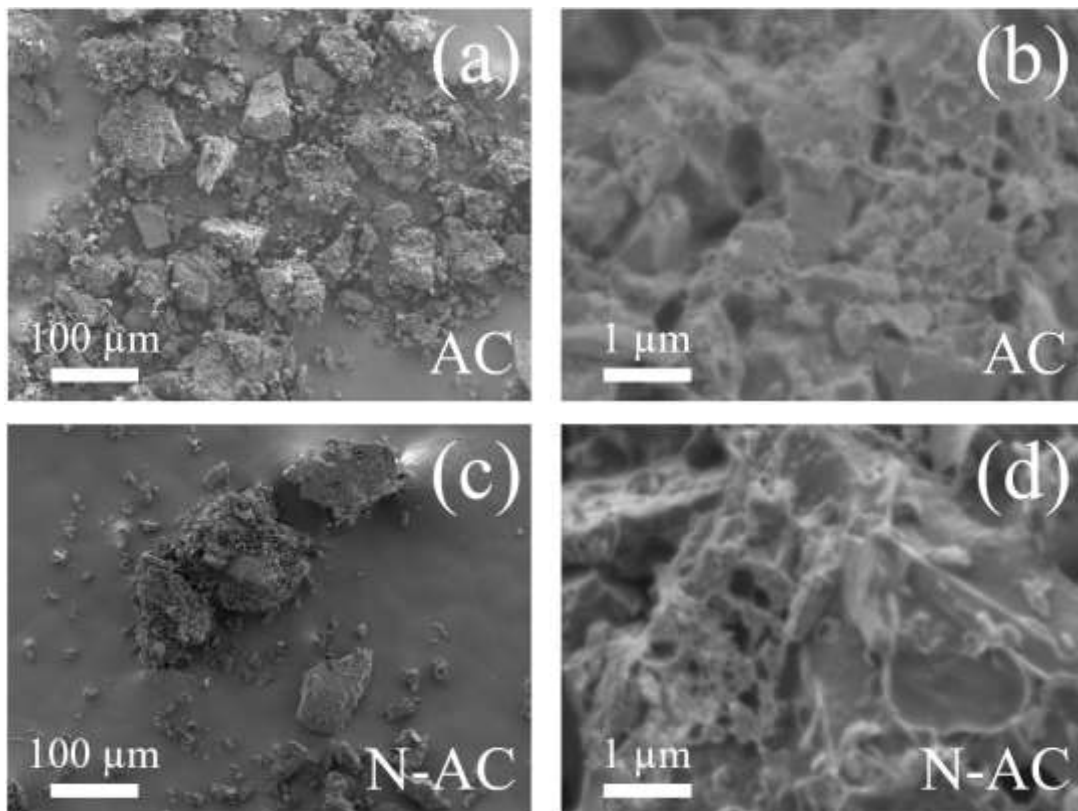
under formaldehyde exposure. Then, all sensors were recovered in an air atmosphere for 10 min. The concentration of formaldehyde varied in the range of 1–100 ppm.

$$SR\% = ((R_{HCHO} - R_{air})/R_{air}) \times 100 \quad \dots(1)$$

### 3. RESULTS OR RESULTS AND DISCUSSIONS

#### 3.1. Characterization Results

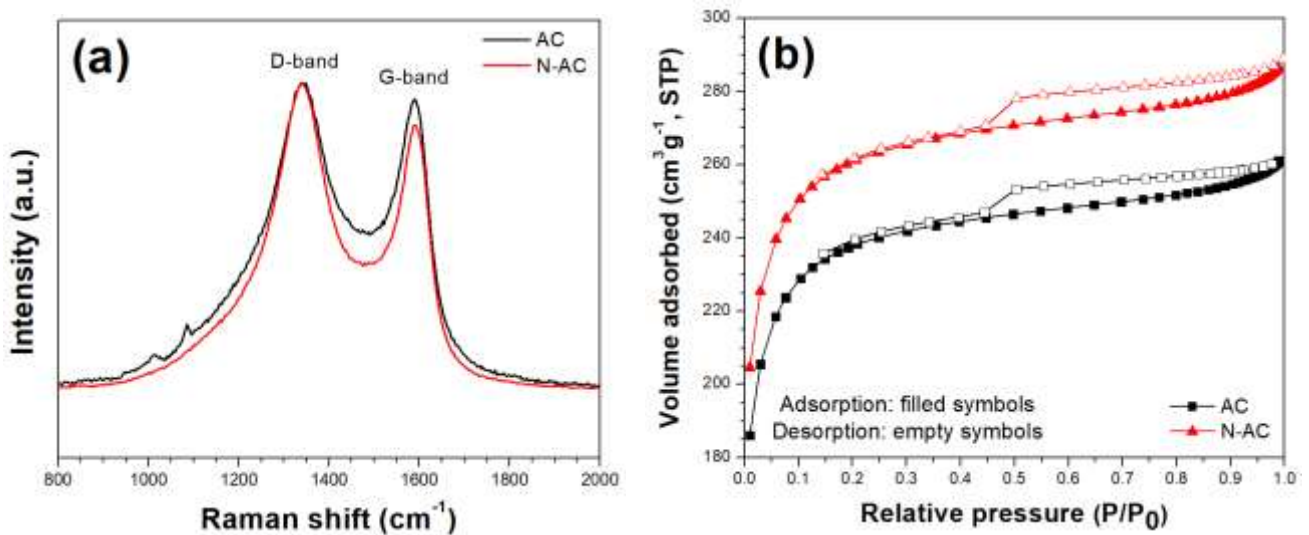
The surface morphologies AC and N-AC were characterized by SEM. SEM images revealed that both AC and N-AC consist of irregularly shaped micron-sized carbon particles, as shown in Fig. 1(a,c). At high magnification SEM images showed that AC and N-AC exhibit porous morphologies, as shown in Fig. 1(b,d). Furthermore, the pore structure in N-AC increased after  $NH_3$  heat treatment. To confirm the effect of  $NH_3$  heat treatment on the increase in surface area and porosity, AC and N-AC were evaluated using BET analysis, as discussed later.



**Fig. 1:** Low- and high-magnification SEM images of (a,b) AC and (c,d) N-AC.

Raman spectra of AC and N-AC are shown in Fig. 2(a). Both AC and N-AC exhibit two peaks at  $\sim 1340$  and  $\sim 1590$   $cm^{-1}$ , corresponding to the disordered graphitic (D-band) and crystalline graphitic (G-band) structures, respectively (Liu et al. 2017, Shimodaira et al. 2002). The ratio value of the D-band to the G-band ( $I_D/I_G$ ) can be used to estimate the degree of crystallinity. Namely, a high  $I_D/I_G$  value indicates low crystallinity (Liu

et al. 2017, Shimodaira et al. 2002). The  $I_D/I_G$  ratios of AC and N-AC were  $1.06 \pm 0.04$  and  $1.17 \pm 0.02$ , respectively. The increase in the  $I_D/I_G$  ratio implied a decrease in crystallinity. The decrease in crystallinity is attributed to the incorporation of nitrogen atoms into graphitic structure (Qian et al. 2019). Fig. 2(b) presents the  $N_2$  adsorption–desorption isotherms at 77 K for AC and N-AC, obtained from BET analysis. The  $N_2$  adsorption isotherms exhibit characteristics of both type I and IV isotherms, according to the International Union of Pure and Applied Chemistry classification. The isotherms are typical of microporous materials (Kumar et al. 2016, Zhang et al. 2018). After  $NH_3$  heat treatment, the total specific surface area, total pore volume, micropore volume, and pore diameter increased, as shown in Table 1. The total surface areas of AC and N-AC were  $749.19$  and  $822.11 \text{ m}^2 \cdot \text{g}^{-1}$ , respectively, while the total pore volumes were  $0.3213$  and  $0.3515 \text{ cm}^3 \cdot \text{g}^{-1}$ , respectively. The micropore volumes of AC and N-AC were  $0.2903$  and  $0.3158 \text{ cm}^3 \cdot \text{g}^{-1}$ , respectively, while their pore diameters were  $1.5404$  and  $1.5442 \text{ nm}$ , respectively.  $NH_3$  heat treatment not only induces the incorporation of nitrogen atoms into the AC structure but also increases the surface area and porosity due to the etching effect of  $NH_3$  (Katsura et al. 1998, Mochizuki et al. 2022).



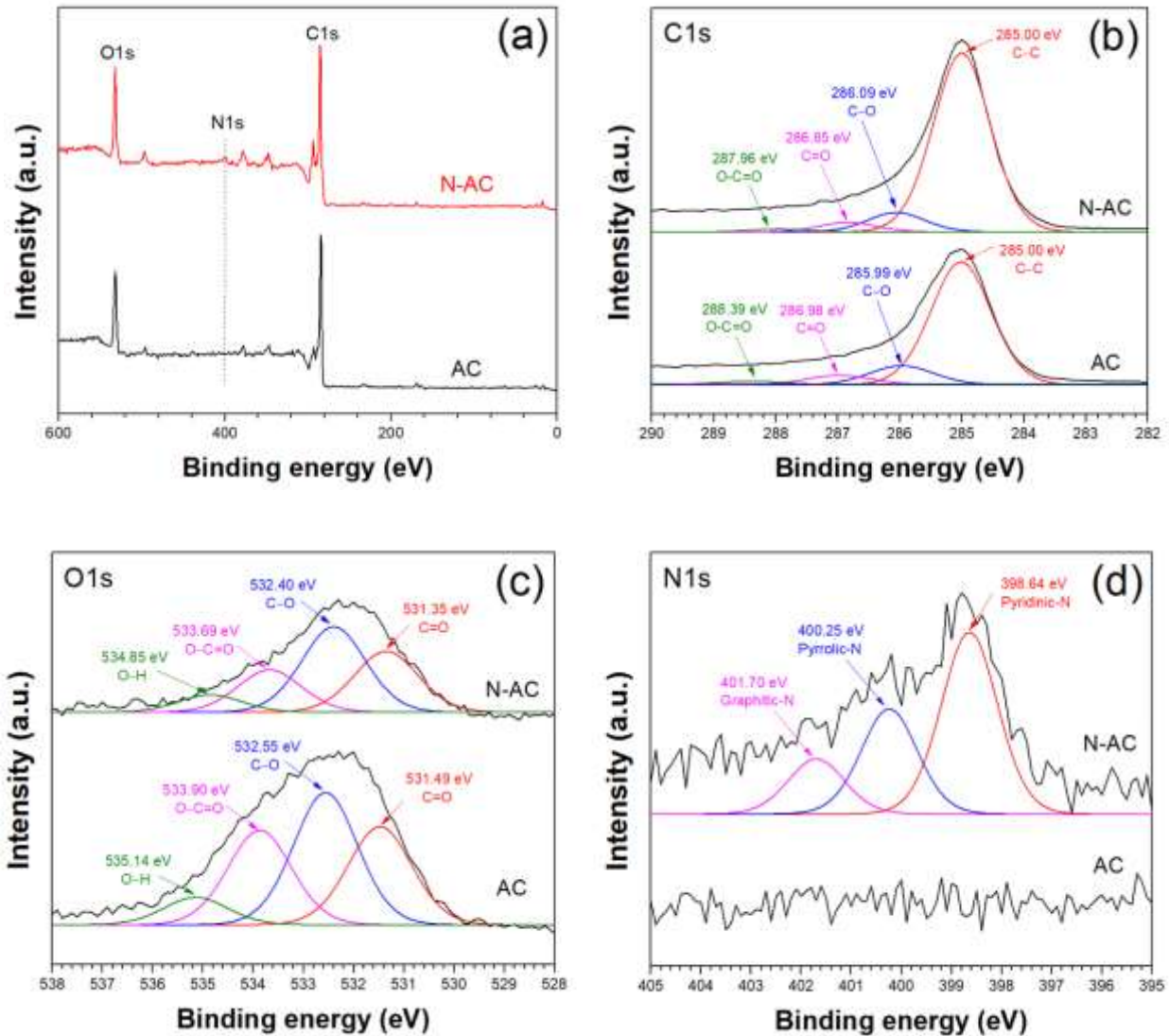
**Fig. 2:** (a) Raman spectra of AC and N-AC. (b) Nitrogen adsorption-desorption isotherms of AC and N-AC.

**Table 1:** The total surface area, total pore volume, micropore volume, and pore diameter of AC and N-AC.

Sample	Total surface area ( $\text{m}^2 \cdot \text{g}^{-1}$ )	Total pore volume ( $\text{cm}^3 \cdot \text{g}^{-1}$ )	$V_{\text{micropore}}$ ( $\text{cm}^3 \cdot \text{g}^{-1}$ )	Pore diameter (nm)
AC	749.19	0.3213	0.2903	1.5404
N-AC	822.11	0.3515	0.3158	1.5442

The chemical state and composition were analyzed by XPS. As shown in the survey spectra in Fig. 3(a), AC and N-AC exhibited two prominent peaks at  $\sim 285$  and  $\sim 532 \text{ eV}$ , which correspond to the C1s and O1s peaks, respectively (Lennon et al. 2002, Ojha et al. 2025). Additionally, the N1s peak was observed at  $\sim 400 \text{ eV}$  in the N-AC, confirming the successful incorporation of nitrogen atoms into the carbon structure. The C1s

spectra of AC and N-AC were deconvoluted to the C–C, C–O, C=O, and O–C=O, as shown in Fig. 3(b). Fig. 3(c) presents the O1s spectra of AC and N-AC, which consist of contributions from C=O, C–O, O–C=O, and O–H (Chen et al. 2020, Jerng et al. 2011, Zhang et al. 2018). Fig. 3(d) shows the N1s spectra of AC and N-AC, which exhibit three deconvoluted bonding configurations: pyridinic-N, pyrrolic-N, and graphitic-N (He et al. 2022, Sun et al. 2019). The atomic ratio of nitrogen to carbon in N-AC is 4.81 at.%. The contents of pyridinic-N, pyrrolic-N, and graphitic-N in N-AC were 2.56, 1.48, and 0.77 at.%, respectively.

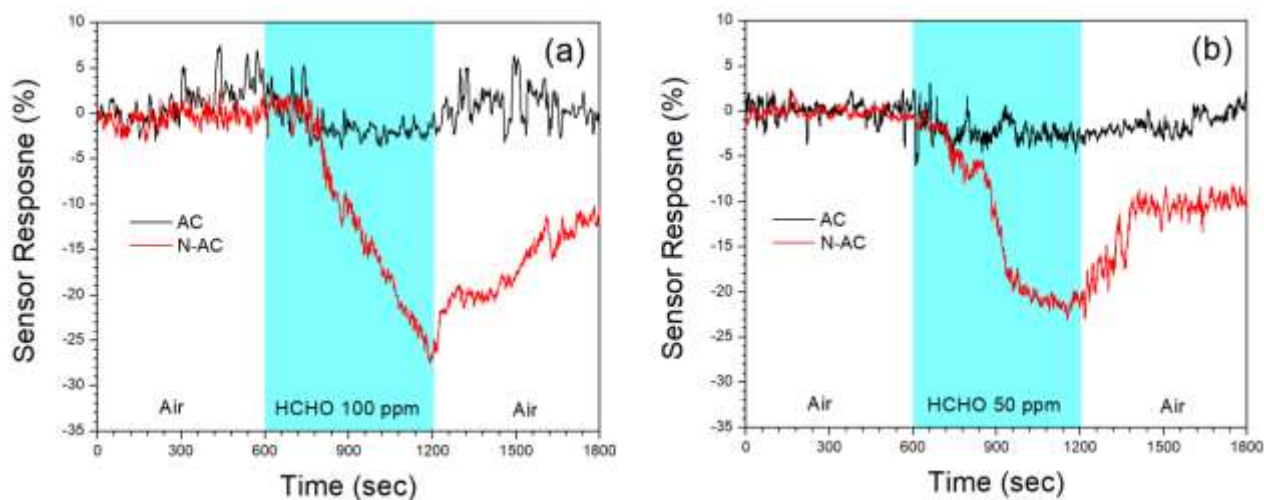


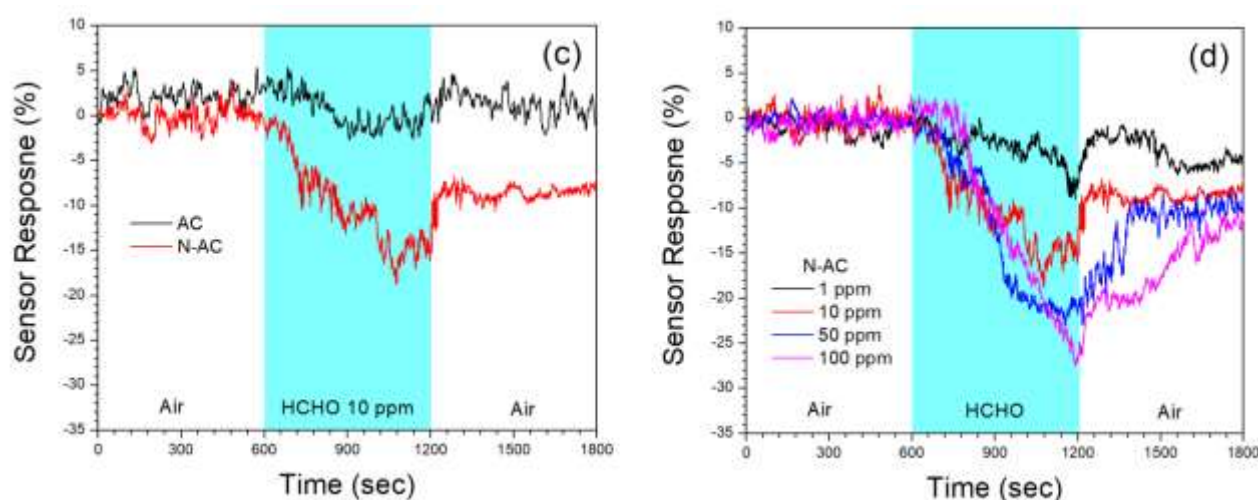
**Fig. 3:** (a) Survey spectra of AC and N-AC. (b) C1s, (c) O1s and (d) N1s spectra of AC and N-AC.

### 3.2. Gas Sensor Results

Fig. 4(a-c) illustrates the time-dependent response of as-fabricated AC and N-AC sensors to formaldehyde in the concentration range of 10-100 ppm at room temperature. The electrical resistance of as-fabricated AC and N-AC sensors increases upon exposure to formaldehyde vapor and recovers to its initial electrical resistance under air exposure. N-AC sensor was unable to recover to its initial electrical resistance under air ambient. The

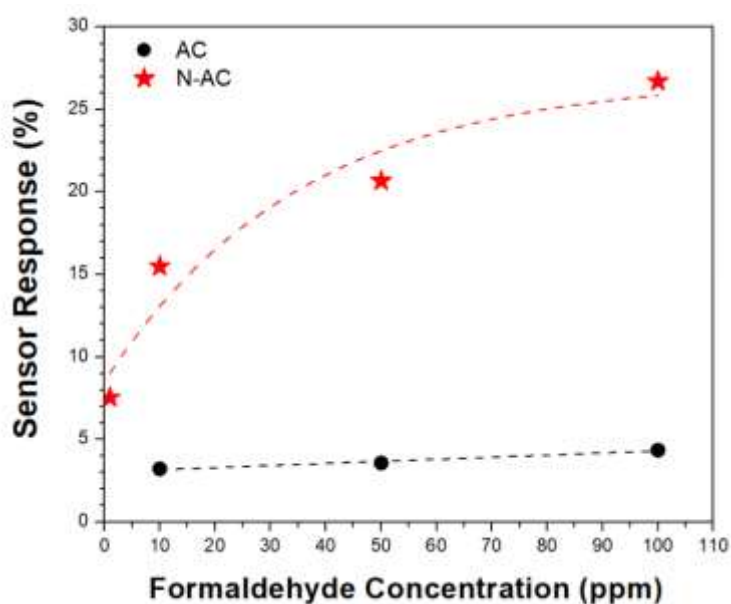
reversibility of the sensor can be improved by UV irradiation and heating. The sensor responses (%) of the as-fabricated AC sensors were 3.22, 3.57, and 4.34% at formaldehyde concentrations of 10, 50, and 100 ppm, respectively, while the responses of the as-fabricated N-AC sensors were 7.54, 15.79, 20.68, and 26.72 at 1, 10, 50, and 100 ppm, respectively. The sensing results demonstrated that the N-AC sensor exhibited 4.90-, 5.79-, and 6.16-fold higher responses than that of the AC sensor at formaldehyde concentrations of 10, 50, and 100 ppm, respectively. The formaldehyde-sensing mechanism is attributed to the charge transfer between formaldehyde molecule and sensing materials. Upon adsorption, formaldehyde molecules donate electron to sensing materials, leading to a decrease in their electrical resistance (Kyokunzire et al. 2024, Ranasinghe et al. 2024, Travlou, et al. 2015). In the case of N-AC, according to theoretical and experimental studies (Chobsilp et al. 2022, Dwivedi et al. 2020, Prakash et al. 2022) have demonstrated that nitrogen sites play a crucial role in enhancing the interaction between N-AC and formaldehyde molecules through chemisorption, owing to their high binding energy, resulting in a significantly enhanced sensor response. The strong interaction hinders the desorption formaldehyde molecules from the N-AC surface, as evidenced by the slow recovery of as-fabricated sensor to their initial electrical resistance. Additionally, in terms of specific surface area and volume of micropore, N-AC exhibits 9.73% and 8.78% higher values, respectively, compared to undoped AC. The enhancement of formaldehyde detection is also attributed the increased surface area. Micropores in porous carbon materials play an important role in gas adsorption (Bai et al. 2016, Do et al. 2015). Thus, a high surface area and micropore volume provide more adsorption sites for formaldehyde molecules. Fig.4(d) presents the time-dependent response of N-AC sensors exposed to formaldehyde at 1-100 ppm. The results indicated that the sensor response increases with higher formaldehyde concentrations due to a large amount of formaldehyde molecules adsorption on the surface of N-AC, resulting in a higher in sensor response.





**Fig. 4:** (a) Time-dependent sensor response of AC and N-AC sensors exposed to the formaldehyde concentrations of (a) 100, (b) 50, and (c) 10 ppm at room temperature. (d) Time-dependent sensor response of N-AC sensor exposed to formaldehyde at 1-100 ppm at room temperature.

Fig. 5 illustrates the sensor responses of AC and N-AC sensors as a function of formaldehyde concentration. For the AC sensor, the relationship between sensor response and formaldehyde displays a linear response in the range of 10-100 ppm. In the case of the N-AC sensor, the sensor response increases rapidly with increasing formaldehyde concentration from 1 to 50 ppm. The sensor response of the N-AC sensor appears to saturate at formaldehyde concentrations higher than 100 ppm. The N-AC sensor can detect formaldehyde concentrations as low as 1 ppm, which is lower than the standard IDLH level of 20 ppm and the STEL of 1 ppm, as specified by NIOSH. This work demonstrated that the N-AC sensor exhibits high sensitivity for formaldehyde detection at room temperature with a LOD of 1 ppm. To improve the sensor performance, the selectivity of N-AC toward other VOCs and gases and long-term stability will be investigated in the future work.



**Fig. 4:** Sensor responses of AC and N-AC sensors as a function of formaldehyde concentration.

#### 4. CONCLUSIONS

A highly sensitive formaldehyde sensor was successfully developed through the incorporation of nitrogen into the biomass-derived AC structure.  $\text{NH}_3$  heat treatment was employed as an effective method to enhance the surface area, porosity, and nitrogen doping into AC structure. As-fabricated N-AC sensor exhibited a 5- to 6-fold enhancement in the response to formaldehyde concentrations ranging from 10 to 100 ppm at room temperature. The enhanced sensitivity is derived from the combination of high surface area, increased porosity, and the presence of nitrogen-doped sites on the AC. The sensing mechanism is attributed to charge transfer between the sensing material and formaldehyde molecules, facilitated by nitrogen sites on the AC surface, which induce strong interactions with formaldehyde. This work demonstrates that surface and porous structure modification via  $\text{NH}_3$  heat treatment offers a promising approach for developing practical and high-sensitivity formaldehyde sensing materials operating at room temperature. This work supports the SDG related to Industry, Innovation, and Infrastructure (SDG 9) as well as Responsible Consumption and Production (SDG 12).

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**Conflicts of Interest:** The authors declare no conflicts of interest.

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