Enhanced Room-Temperature Ethanol Detection by Quasi 2D Nanosheets of an Exfoliated Polymeric Graphitized Carbon Nitride Composite-Based Patterned Sensor

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ABSTRACT: A novel room-temperature gas sensor composed of polymeric graphitic carbon nitride composite was fabricated and used for the detection of ethanol vapor under ambient conditions. Polymeric carbon nitride (PCN) microstructures composed of fluffy nanosheets were synthesized via a thermal polycondensation mechanism using melamine as the precursor, followed by vigorous chemical exfoliation. These sheet-like microstructures were employed as active materials in the form of composites, along with carbon paste consisting of graphite nanoplatelets and carbon black. The active sensing layer was fabricated on a PET sheet and assembled on an interdigitated gold electrode. The as-fabricated sensor exhibited excellent sensing efficiency (>100% response at 10 ppm) along with high selectivity and stability. In particular, for ultralow concentrations such as 1 ppm (>10% response), this resistive-based sensor exhibited a swift response time provided under ambient conditions. The exfoliated PCN composite sensor was found to be working with appreciable efficiency at moderate relative humidity (%) with the least fluctuation in response signals also demonstrating long-term stability for 30 days with consistent response signals.

1. INTRODUCTION
Ethanol (or ethyl alcohol) is a commonly used alcohol as an organic solvent in chemical laboratories, pharmaceuticals, and cosmetics. Ethanol is one of the most volatile organic compounds (VOCs) and heavy exposure of ethanol can be dangerous to human health and other organisms in the environment. Direct uptake of ethanol above permissible limit can cause dizziness, nausea, and vomiting. Higher uptake by humans in vivo (1.25 mg/L) can cause haziness and loss of consciousness. Moreover, the excessive leakage of ethanol can cause explosion due to its high flammability. Hence, there is an urgent need for a swift and precise sensing device for ethanol vapor detection under ambient conditions. Resistive gas sensors have been found to be excellent for the accurate and precise determination of VOCs compared to heavy and nonportable analytical equipment such as gas chromatography—mass spectrometry, liquid chromatography—mass spectrometry, etc. Resistive sensors utilize the fluctuation in electronic accumulation at the surface of the sensing material, resulting in a change in conductivity and resistance of the electronic circuitry. These resistive sensors can be employed to observe any type of leakage at a commercial unit. Generally, metal oxide/sulfide semiconductor-based sensors have been developed for ethanol sensing. However, most of the conventional sensors work at high temperature conditions, which require inevitable use of micro-heaters in the sensing units. Both n- and p-type semiconductors have been successfully applied for the detection of VOCs. In general, common metal oxides (e.g., ZnO, TiO$_2$) and metal sulfides (e.g., PbS, CdS, SnS$_2$) have been used for the detection of VOCs. Since these sensing materials operate at elevated temperatures, recent efforts have been directed toward the development of sensing materials that can be operated at room temperature. Recently, hybrid materials such as CuO-ZnO, Gd$_{50}$-BaSnO$_{39}$, CNT@ZnSnO$_3$, Zn$_{0.89}$Fe$_{0.11}$O$_{1.8}$, ZnFe$_2$O$_4$NS, and PdOx/Co$_3$O$_4$ were reported to be ethanol sensitive at elevated temperatures. On the other hand, hybrid sensors consisting of PbS:SnS$_2$ oxidized single-walled carbon nanohorn-SnO$_2$-polyvinylpyrrolidone, hematite (α-Fe$_2$O$_3$)/silver nanoparticles, ZIF-8&Ag@SiNWs, and MoO$_2$-Ni-graphene ternary composite have been found to be effective for ethanol sensing at room temperature. However, these materials are metal-based and expensive. Therefore, economically viable resistive sensors are extremely in demand at this time. Recently, cost-effective carbon-based nanomaterials such as graphitic carbon nitride, graphene/graphene...
derivatives, and carbon quantum dots have been found catalytically efficient for solar energy conversion and energy storage applications.\textsuperscript{13,21–23} Considering the potential application in heterogeneous catalysis for solar energy conversion, electrocatalysis, and energy storage devices, we attempted to use graphite-like polymeric carbon nitride (PCN) to devise the two-dimensional (2D) sensing platform for the detection of VOCs.

There are various structural parameters that govern the sensing mechanism, such as the thickness of the sensing layer and the shape and structure of the sensing active species. 2D carbon-based nanomaterials are in trend for energy and environmental applications and the exfoliation of the layered structure into nanosheets allows for large surface area, plenty of catalytic active centers, and unique physicochemical/electronic properties. In this context, 2D nanosheet-based sensors have been reported for low-level detection of VOCs.\textsuperscript{22,23}

Here, we present a novel PCN composite consisting of exfoliated PCN with graphite nanoplatelets (GNPs) as active material for ethanol sensing under ambient conditions. PCN was synthesized by a simple thermal polycondensation process using an inexpensive compound, melamine (C\textsubscript{3}H\textsubscript{6}N\textsubscript{6}, Sigma-Aldrich; 99\%) at 550 °C for 1 h at 10 °C/min in a muffle furnace.\textsuperscript{24} Further, PCN monolayer nanosheets were produced through chemical exfoliation. The as-synthesized PCN (1 g) was added to 10 mL of H\textsubscript{2}SO\textsubscript{4} (>95 wt %, Daejung) in a 50 mL flask and stirred for 8 h under ambient conditions.\textsuperscript{25} This chemical exfoliation process changed the yellow color of the bulk CN to light yellow of CN monolayers. Subsequently, the reaction mixture was gradually transferred to 100 mL of deionized water and sonicated for exfoliation. This caused an increase in the temperature of the resultant acid solution, and the color changed from yellow to light yellow. The final acid suspension was centrifuged at 3000 rpm for 10 min. The recovered exfoliated PCNs were dried overnight at 70 °C in an oven. Structural defects were eliminated through an additional treatment of the as-prepared exfoliated PCN with 150 mL of methanol under reflux on a hot mantle at 65 °C for 6 h. Finally, the exfoliated PCN nanosheets were obtained after centrifugation and drying at 70 °C overnight.

2. EXPERIMENTAL SECTION

2.1. Materials Synthesis. PCN was synthesized via a simple thermal polycondensation method using melamine (C\textsubscript{3}H\textsubscript{6}N\textsubscript{6}, Sigma-Aldrich; 99\%) at 550 °C for 1 h at 10 °C/min in a muffle furnace.\textsuperscript{24} Further, PCN monolayer nanosheets were produced through chemical exfoliation. The as-synthesized PCN (1 g) was added to 10 mL of H\textsubscript{2}SO\textsubscript{4} (>95 wt %, Daejung) in a 50 mL flask and stirred for 8 h under ambient conditions.\textsuperscript{25} This chemical exfoliation process changed the yellow color of the bulk CN to light yellow of CN monolayers. Subsequently, the reaction mixture was gradually transferred to 100 mL of deionized water and sonicated for exfoliation. This caused an increase in the temperature of the resultant acid solution, and the color changed from yellow to light yellow. The final acid suspension was centrifuged at 3000 rpm for 10 min. The recovered exfoliated PCNs were dried overnight at 70 °C in an oven. Structural defects were eliminated through an additional treatment of the as-prepared exfoliated PCN with 150 mL of methanol under reflux on a hot mantle at 65 °C for 6 h. Finally, the exfoliated PCN nanosheets were obtained after centrifugation and drying at 70 °C overnight.

2.2. Characterization of Sensing Materials. The morphologies of samples were characterized by scanning electron microscopy (SEM; FEI Quanta 450 FEG) and transmission electron microscopy (TEM; Titan G2 60-300, FEI, USA). The X-ray diffraction (XRD) pattern was acquired by PANalytical X'Pert Pro MPD using Cu K\alpha radiation. The functional groups present in PCN were investigated by Fourier transform infrared (FTIR) spectroscopy; the spectra were obtained over the range of 400–4000 cm\textsuperscript{-1}. Specific surface area (SSA) and total pore volume were obtained using nitrogen (N\textsubscript{2}) adsorption and desorption at 77 K performed on a gas adsorption/desorption analyzer (BELSORP-max, Microtrac BEL Corp., Japan). Prior to the N\textsubscript{2} adsorption/desorption measurements, the samples were degassed at 120 °C under vacuum for 12 h. The SSA of the samples was calculated using the Brunauer–Emmett–Teller (BET) method.
from the data with the relative pressure \( \frac{P}{P_0} \) in the range of 0.1–0.3.

### 2.3. Sensor Fabrication and Test.

The sensing layer was composed of an active sensing material consisting of PCN composite with a carbon paste consisting of GNP and carbon black in a ratio of 1:10. This dispersion ratio was optimized for good dispersion of the active exfoliated PCN. Ethyl cellulose and solution of a polyester derivative (TEGO 670) are used as the binder and dispersing agent, respectively. Subsequently, all individual components of the sensing material were mixed well in a centrifugal mixer at 2200 rpm for 15 min. The sensing layer was deposited on the PET film using a Meyer rod and then dried in a convection oven. Subsequently, the specific dimensions of the sensing layer were fabricated on interdigitated Au with a masking tape, maintaining a uniform layer across the electrode panel. The electrode ends were connected using Ag paste, followed by drying at 100 °C for 30 min. The ethanol vapor was injected into the gas sensor test chamber with Ar carrier gas. The concentrations of ethanol vapor were varied in the range of 1–10 ppm using a mass flow meter. A schematic of the overall ethanol sensing process of ethanol sensing using a resistive sensor electrode is shown in Figure 1.

The exact concentration of the ethanol vapors was determined using the following equation:

\[
C_{\text{ppm}} = \left[ \frac{p_{\text{Sat}}}{V_{\text{Sat}}} \times 10^6 \right] \times \frac{V_1}{V_2}
\]

where \( V_1 \) is the volume of ethanol required (mL) and \( V_2 \) is the fixed volume of the cylindrical gas chamber with a height of 25 mm and a diameter of 130 mm \( (V_2 = 331830.655 \text{ mm}^3) \). The entire set of experiments was conducted at 60 ± 5% relative humidity.

After reaching a steady resistance response, the chamber was purged with air, and the experiment was repeated to detect different concentrations of ethanol. The sensor response was calculated in terms of the sensitivity \( (S) = R_{\text{gas}}/R_{\text{air}} \) and response (%) = \( (R_{\text{gas}} - R_{\text{air}}) \times 100/R_{\text{air}} \), where \( R_{\text{gas}} \) and \( R_{\text{air}} \) are the resistances of the electrode under exposure to air and ethanol, respectively.\(^{1,8,9,11-13} \) All sensing experiments were performed at 20 ± 2 °C in an environment containing ethanol vapor and ambient air.

This work presents the fabrication of gas sensor materials at ambient conditions. So, we used nanocarbon materials as a platform with binding materials like ethyl cellulose that are...
soluble only in an organic solvent. This water-resistant binder protects the carbon material platform from moisture. Therefore, moisture cannot penetrate gas sensing materials. As a result, it does not get affected by moisture content in its surroundings.

3. RESULTS AND DISCUSSION

3.1. Physicochemical Properties. Figure 2 consists of SEM and TEM images demonstrating the surface morphologies of PCN and exfoliated PCN. Figure 2a shows the stacked sheet-like structures of the PCN. After vigorous chemical exfoliation using concentrated sulfuric acid, the stacking loosened, opening the sheets (Figure 2b). The TEM image in Figure 2c shows the PCN sheet within the nano-regime.
The dimensions of the PCN sheets were found to be less than 100 nm and are referred to as nanosheets. It has been reported that even after the ideal separation of the PCN monolayer structures, the formation of agglomerates and crumples of the monolayer is unavoidable, arising from van der Waals interlayer interactions. This fact was further substantiated using XRD. Figure 2d–f shows the elemental mapping of the exfoliated PCN, confirming the spread of the CN units. The elemental analysis of the exfoliated PCN in Figure S1 shows equally distributed percentages of carbon and nitrogen.

The surface roughness of the exfoliated PCN was analyzed by AFM measurement, as presented in Figure 2g–i. The

Figure 5. Dynamic response for ethanol vapor detection at ambient conditions over the resistive sensor: (a) resistance vs time at different concentrations of ethanol; (b) response \( \frac{R_{\text{gas}}}{R_{\text{air}}} \) vs time at different concentrations of ethanol; (c) response \( \left( \frac{R_{\text{gas}} - R_{\text{air}}}{R_{\text{air}}} \right) \times 100 \) vs time at different concentrations of ethanol over the patterned exfoliated PCN composite sensor; (d) response \( \frac{R_{\text{gas}}}{R_{\text{air}}} \) vs time at different concentrations of ethanol over the nonpatterned sensor; (e) response \( \frac{R_{\text{gas}}}{R_{\text{air}}} \) vs time at different concentrations of ethanol over the patterned bulk PCN composite sensor; (f) repeatability curve (resistance vs time) for 1 ppm ethanol; (g) repeatability curve (response \( \frac{R_{\text{gas}}}{R_{\text{air}}} \) vs time) for 1 ppm ethanol; and (h) response \( \left( \frac{R_{\text{gas}} - R_{\text{air}}}{R_{\text{air}}} \right) \times 100 \) vs time at different concentrations of various VOCs over the patterned exfoliated PCN composite sensor.
theoretical thickness of the PCN monolayer is reported to be 0.4 nm in the literature. The height profile of the exfoliated PCN suggested that there are five to six multilayers formed due to exfoliation substantiating XRD and topology analysis by SEM and TEM.

Elemental states in bulk PCN and exfoliated PCN were analyzed by XPS, as illustrated in Figure 3. The wide scan in Figure 3a,d depicts the elemental composition of bulk PCN and exfoliated PCN, confirming the presence of carbon and nitrogen. Figure 3b represents the C1s XPS spectra of bulk PCN. This carbon spectrum has two peaks placed at 284.6 and 288.0 eV. The peak at 284.6 eV is associated to the sp²-hybridized C bonded to another sp²-hybridized C (C=C). The peak at 288.0 eV is further deconvoluted into two Gaussian-Lorentzian peaks positioned at 287.9 and 288.6 eV. The former peak corresponds to the sp²-hybridized carbon bonded to N within triazine rings, whereas the latter one belongs to sp²-hybridized carbon in triazine rings bonded to NH₂. On the contrary, C1s peaks at 287.9 and 288.6 eV corresponding to exfoliated PCN have been shifted toward higher binding energy as in Figure 3e. This is attributable to the strengthening of −C=N− bonds as a result of electronic localization after de-stacking of bulk PCN during the exfoliation process as well as defect generation during methanol treatment. Figure 3c,f demonstrates the N1s XPS spectra exhibiting two peaks with maxima at 398.6 and 404.2 eV. This single peak is deconvoluted into two Gaussian-Lorentzian peaks. The prominent peak observed at 398.7 eV is attributed to sp²-hybridized N inside triazine rings, whereas the peak at 399.7 eV appears due to the bridging N atoms as N−(C) linked to triazine rings, while the low-intensity peak at 404.2 eV is due to N−H bonds in amino functional groups in heptazine rings.

Figure 4a shows the XRD patterns of PCN and exfoliated PCN. It consists of two characteristic peaks at 13.1 and 27.4° associated with the (100) plane (i.e., interlayer reflection of the stacking structures) and the (002) plane (reflection from the in-plane repeating motif of tri-s-triazine units), respectively, which correspond to graphitic PCN as existed in JCPDS no. 87-1526. The right shift in the position of the characteristic peak at 27.4° was attributed to a decrease in the gallery distance, owing...
to the formation of PCN multilayered nanosheets instead of stacking with the same interlayer spacing.

The Raman spectra in Figure 4b provide the insights about the chemical structure of PCN and exfoliated PCN. There are several characteristic vibrational bands found in the range of 700–2500 cm⁻¹ corresponding to the breathing modes of triazine for the graphitic structure of carbon nitride that are found to be in close agreement with those reported in the literature. The above XRD results. Several Raman bands between 567, 811, 914, 1021, 1124, 1231, 1361, and 1521 cm⁻¹ are due to stretching vibrations of melem like aromatic C–N heterocyclic rings. There is no effect of exfoliation on G-bands at 1400–1600 cm⁻¹, which clearly signifies that the graphitic structure of carbon nitride nanosheets is intact. There is a slight reduction in the intensity of D-vibration bands between 500 and 1300 cm⁻¹, owing to the phonon confinement effect of protonation during exfoliation.

The nitrogen adsorption−desorption isotherms in Figure 4c,d are of type IV with hysteresis loops for both PCN and exfoliated PCN, indicating a mesoporous structure. The BET surface areas of PCN and exfoliated PCN were calculated to be 9.76 and 24.49 m²/g, respectively, with a large portion of mesopores in agreement with the literature for melamine-derived PCN.

The FTIR spectra shown in Figure S2 for PCN and exfoliated PCN are found to be in good agreement with previous reports. The high-intensity peak corresponds to the CN heterocycles of triazine units present in graphitic PCN. The absorption bands in the range of 1207–1633 cm⁻¹ are associated with the stretching vibration modes of the heptazine heterocyclic rings. The broad bands in the range of 3000–3500 cm⁻¹ are due to the stretching modes of the N–H vibration. The intensity of these bands further decreased for the exfoliated PCN.

The optical properties were investigated using UV−vis and photoluminescence (PL) measurements. The UV−vis absorption spectra in Figure 4e reveal that the absorption edge of the exfoliated PCN spectrum undergoes a hypsochromic (blue shift of 14 nm) as well as hypochromic shift compared to PCN, as depicted in Figure 4f, indicating that there is an increase in the band gap as a result of further separation of the valence band and conductive band, causing lower molar absorptivity. This blue shift may result from the decrease in the conjugation length of the heterocyclic ring and the strong quantum confinement effect caused by the single-layer structure of the g-C₃N₄ nanosheets.

### 3.2. Sensing Performance Test

Figure 5 shows the response and recovery curve of ethanol detection at various concentrations ranging from 1 to 10 ppm over the patterned film sensor electrode and nonpatterned sensor electrode. The prepared exfoliated PCN composite with carbon paste was highly responsive to ethanol vapor, with a response of more than 10% at 1 ppm. Moreover, the patterned sensor electrode exhibited a response of greater than 125% at 10 ppm (Rₛ/Rᵝ > 2.2). To our knowledge, this value is the maximum response reported so far for ethanol detection at 10 ppm over nonmetal-based sensor electrodes. The difference in the response offered by the patterned and nonpatterned electrodes can be attributed to the swift movement of the charge carrier across the potential ends. Notably, the linear relationship between the response and the ethanol concentration is highlighted in the response curves in Figure 5a–d. The extrapolation of the response curve in Figure 5c at the sub-ppm level gave an extremely low limit of detection (0.25 ppm) with more than 2.5% response. The response over the patterned exfoliated PCN composite sensor is found appreciably high in comparison to the bulk PCN composite sensor, as shown in Figure 5e. This increment in the response value for the exfoliated PCN composite sensor is attributed to the facile charge transportation through exfoliated multilayered PCN nanosheets. The cycle stability for 1 ppm ethanol vapor exhibited negligible fluctuation or loss in response, even after more than five cycles as in Figure 5f. This enabled the as-prepared sensors to be sustainable and efficient. The as-prepared patterned sensor electrode was also subjected to common VOCs, such as methanol and acetone. However, it selectively detected ethanol with a higher response, as shown in Figure 5h. The higher extent of selectivity toward ethanol can be credited to the reductive characteristic of R−OH. The extrapolated PCN sensor was found to be working with appreciable efficiency at lower relative humidity with least fluctuation in response signals, as demonstrated in Figure 6a,b.

But the response gets decreased by >10% at higher 75 RH% as in Figure 6c. The study on the effect of relative humidity (RH%) shows that the sensor is appreciably efficient at lower RH%, which is similar to ambient conditions. Figure 6d presents the long-term stability study and infers that the composite sensor works efficiently up to 4 weeks as response signals were quite consistent up to 30 days. Later, the sensor
starts losing its efficiency, owing to irreversible adsorption of impurities and moisture.\textsuperscript{16,19,41}

A comparison of this study with previous reports is presented in Table 1. This suggests that the present nonmetallic carbon nitride-based sensor is economically viable and sustainable for ethanol detection under ambient conditions.

3.3. Sensing Mechanism. PCN contains a large amount of pyridine N, as mentioned above in XPS, with a strong affinity to oxygen as reported in the oxygen reduction reaction (ORR).\textsuperscript{48} This affinity to O\textsubscript{2} resulted in a high concentration of O\textsuperscript{−} or O\textsubscript{2−}, which ultimately became the active center for the charge transfer reaction between O\textsubscript{2} and the exfoliated PCN composite. In the absence of ethanol vapor, oxygen molecules get accumulated not only on the composite surface but also to the interior of the structure. The majority of carrier electrons from GNP get transferred to PCN, which results in the formation of the hole accumulation layer. Upon exposure to ethanol vapors, the resistance increased abruptly, owing to the consumption of O\textsubscript{2} by ethanol and the shading of the electron back to the sensing material, where this electron recombines with a major charge carrier, i.e., hole in p-type exfoliated PCN composite, leading to hole depletion,\textsuperscript{19} as depicted in Figure 7. It is evident from the sensing mechanism that the rate of charge transfer is crucially important for the electrical response in ethanol sensing. The porous multilayered nanosheets allow the better percolation of charges across the bulk PCN and result in the high rate of charge transfer via interconnected heterocyclic heptazine units, and so the swift response with higher sensitivity is recorded upon ethanol exposure in comparison to bulk PCN.

4. CONCLUSIONS

Composites composed of exfoliated PCN nanosheets and carbon-based materials were developed and explored as resistive sensing materials for the room-temperature detection of ethanol vapor under ambient conditions. First, bulk PCN microstructures composed of thin nanosheets were synthesized through a thermal polycondensation process using melamine as the precursor and then mechanically exfoliated into individually separated nanosheets. The active sensing materials were deposited on a PET sheet and fabricated on an interdigitated Au electrode. The as-fabricated ethanol vapor sensor exhibited excellent sensing efficiency (>100% response at 10 ppm) along with high selectivity and stability. Even at a very low ethanol vapor concentration of 1 ppm, this sensor showed a response of more than 10% with a swift response time under ambient conditions. The selectivity tests confirmed its exclusive affinity for ethanol vapors in comparison with other common VOCs, such as methanol and acetone. This study lays the foundation for the further functionalization of PCN in sensing applications, particularly for ethanol vapor detection under ambient conditions.
**ASSOCIATED CONTENT**

**Data Availability Statement**
The authors confirm that the data supporting the findings of this study are available within the article and/or its supplementary materials.

**Supporting Information**
The Supporting Information is available free of charge at https://pubs.acs.org/doi/10.1021/acsomega.2c02962.

Band gap calculation (S1); EDS spectrum of exfoliated PCN showing the overlapping of carbon and nitrogen (Figure S1); FTIR spectra of PCN and exfoliated PCN (Figure S2); and References S1, S2, S3, and S4 (PDF)

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**Notes**
The authors declare no competing financial interest.

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