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Short communication

Flexible polyaniline/carbon nanotube nanocomposite film-based electronic gas sensors

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ABSTRACT

Real-time ammonia (NH_3) detection in anaerobic digestion is highly desirable, due to the ammonia inhibition on methane production. Here, the interconnected nanocomposite network of polyaniline (PANI) nanoparticle-coated carbon nanotube (CNT) and PANI nanofiber is fabricated by adding ammonium persulfate into CNT-containing aniline solution for PANI polymerization and film deposition. The film is assembled as high-sensitive ammonia sensors, exhibiting highly sensitive NH_3 sensing from 200 ppb to 50 ppm, fast response/recovery time, room-temperature operation without external aid, reliable flexibility and excellent selectivity to NH_3 compared to other volatile organic compounds.

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1. Introduction

The rapidly growing global concerns over the structure of energy demand and the environment pollution have stimulated efforts to develop renewable energy [1,2]. As a method to produce renewable energy, anaerobic digestion has attracted a lot of attention in the world [3]. Anaerobic digestion technology is an important means to dispose organic solid waste, including manure, industrial waste and food waste, for preventing corruption and stink, and to produce methane by fermentation as valuable renewable energy resources for employed in vehicles after purification [4]. Organic wastes containing nitrogen, such as protein, urea and amino acid, are converted to ammonia (NH_3) by anaerobic digestion. Thus, the concentration of ammonia nitrogen will accumulate in a long term. However, ammonia inhibition of anaerobic digestion caused by ammonia accumulation is one of the main reasons to lead the low efficiency and poor stability for anaerobic digestion [5]. High concentration of ammonia has inhibitory and toxic effects on anaerobic digestion operation for degrading organic matters and methane production [6,7]. Thus, it is highly expected that ammonia inhibition phenomenon can be discovered earlier and timely for real-time adjustment of reaction conditions to eliminate the inhibitory effect on anaerobic digestion for both environmental and economic concern. Therefore, real-time ammonia detection in anaerobic digestion by high-sensitive room-temperature electronic gas sensors, is highly desirable.

Some ammonia detection instruments have actually been available, such as gas chromatograph and the traditional ceramics tube or inter-finger probe based gas sensing devices. However, they are lack of relative portability, real-time sensing, low cost, transparency and flexibility [8,9]. Therefore, they cannot meet the growing future application call for flexible gas sensors.

Recently, flexible conductive film-based gas sensors have attracted widely scientific attention due to the wearability, flexibility, transparency and compatibility over curved substrates of integrated electronics [10–14]. Among the various nanomaterials-based flexible electronic gas sensors, CNT film-based sensors demonstrated stable room-temperature NH_3 sensing performance [15–18]. But they displayed relatively low sensing performance. Meanwhile, polyaniline (PANI) based sensors have been well established due to the facile synthesis, simple acid/base doping/dedoping chemistry and high NH_3 sensing response [19–21]. A dramatic conductance decrease can be observed for PANI gas detection by transforming emeraldine salt form of PANI into its emeraldine base form exposed to the strong electron donating

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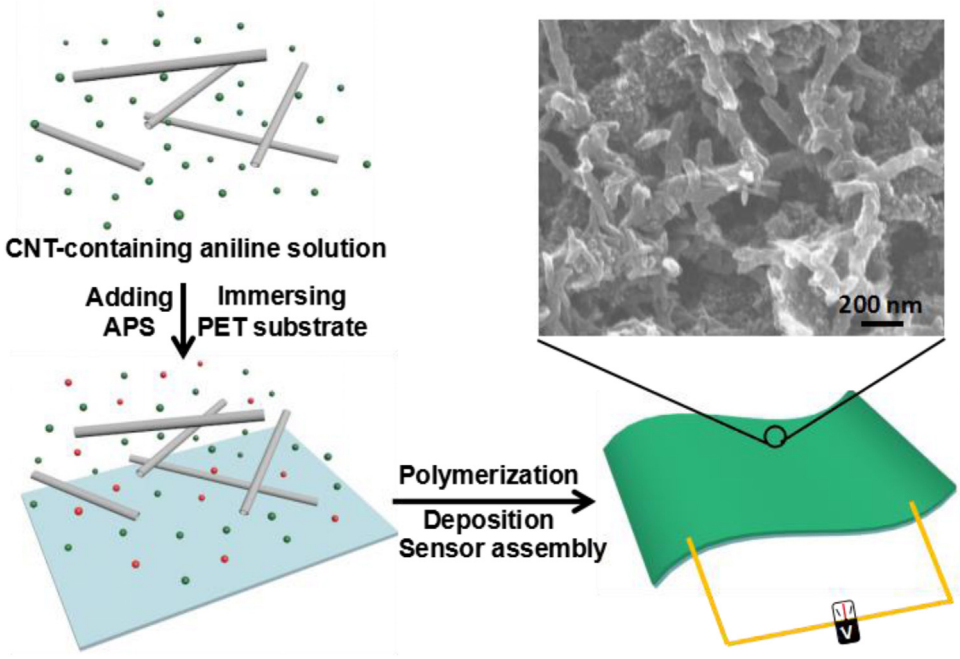


Fig. 1. Schematic illustration for the fabrication of the interconnected nanocomposite networks of PANI nanoparticle-coated CNT and PANI nanofiber, and the assembled flexible gas sensor.

NH_3. However, they are lack of long-term environmental stability. Conductive polymer nanocomposites based gas sensors have been well reported. J. F. Feller and co-workers fabricated polycarbonate-CNTs transducers with hierarchical structure prepared by spray layer by layer for vapour sensing [22]. S. Abdulla and coworkers demonstrated a room temperature gas sensor based on PANI/CNTs nanocomposite for ammonia detection [23]. However, compared to these sensors, highly sensitive room-temperature electronic gas sensors are greatly desirable. Thus, the combination of these materials into nanocomposite network films with intrinsic mobility, chemical stability, and high-sensitive NH_3 performance for NH_3 sensors remains to be addressed.

Herein, we fabricated a hierarchical polyaniline/carbon nanotube (PANI/CNT) nanocomposite network films to be assembled

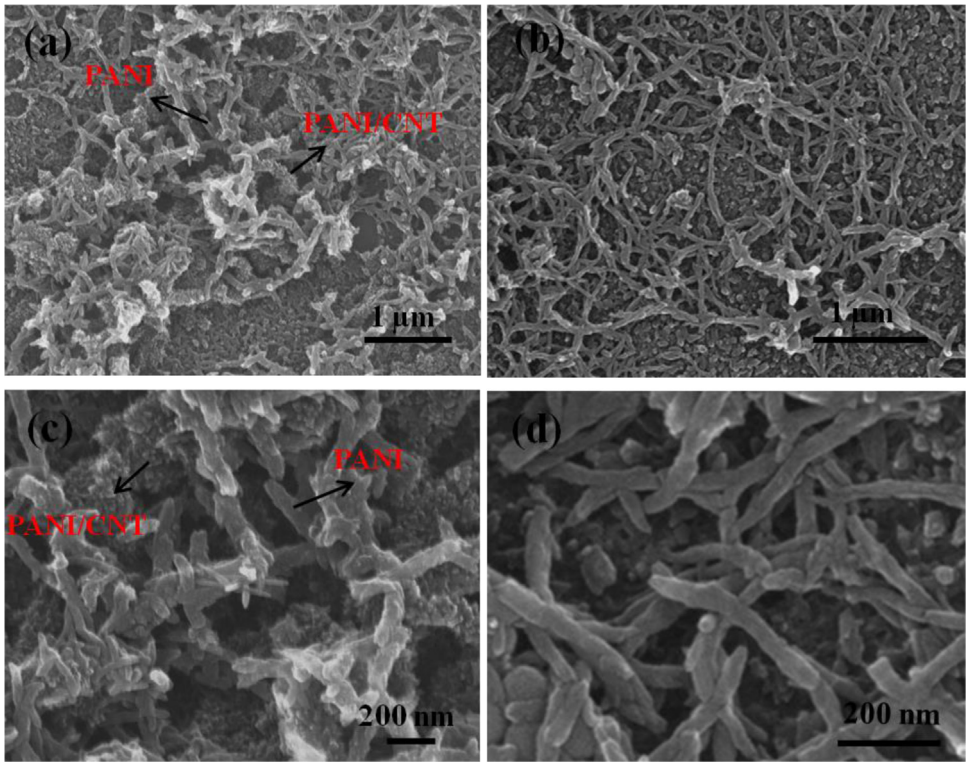


Fig. 2. SEM images of (a) PANI/CNT network (scale bar: 1 μm) and (b) PANI fiber (scale bar: 1 μm). Enlarged SEM images of (c) PANI/CNT network (scale bar: 200 nm) and (d) PANI fiber (scale bar: 200 nm).

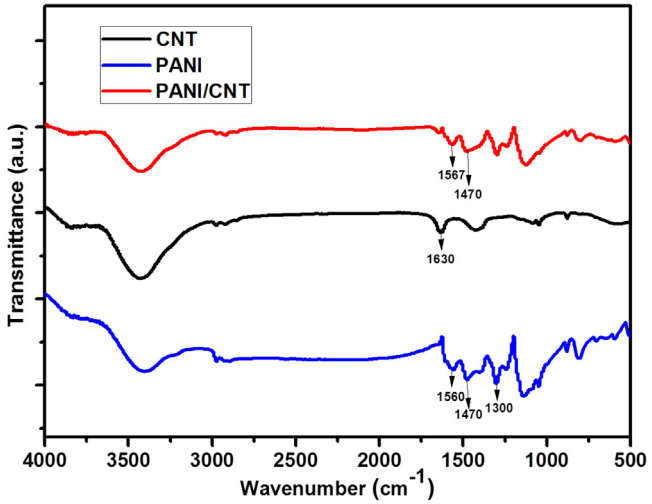


Fig. 3. FTIR spectra of the hierarchical PANI/CNT nanocomposite, PANI fiber and CNT.

as gas sensors for high-sensitive NH_3 detection. The hierarchical nanocomposite films are obtained by adding ammonium persulfate (APS) into CNT-containing aniline solution for in-situ PANI polymerization and film deposition on PET substrate. The PANI nanoparticle coated CNT and PANI nanofiber were successfully interconnected to form the hierarchical nanocomposite network. The assembled gas sensor exibits highly sensitive NH_3 sensing from 200 ppb to 50 ppm, fast response/recovery time, room-temperature operation without external aid, and reliable flexibility (Fig. 1). The hierarchical nanocomposite film sensor demonstrated a better sensing response and cycle stability than CNT and PANI based films, which thus holds a great promise for real-world sensor applications.

2. Experimental details

2.1. Materials

Ammonium persulfate (APS), concentrated HCl, concentrated H_2SO_4, concentrated HNO_3 and ethanol, were purchased from Beijing Chemical Reagent Factory. Aniline (distilled before use) was

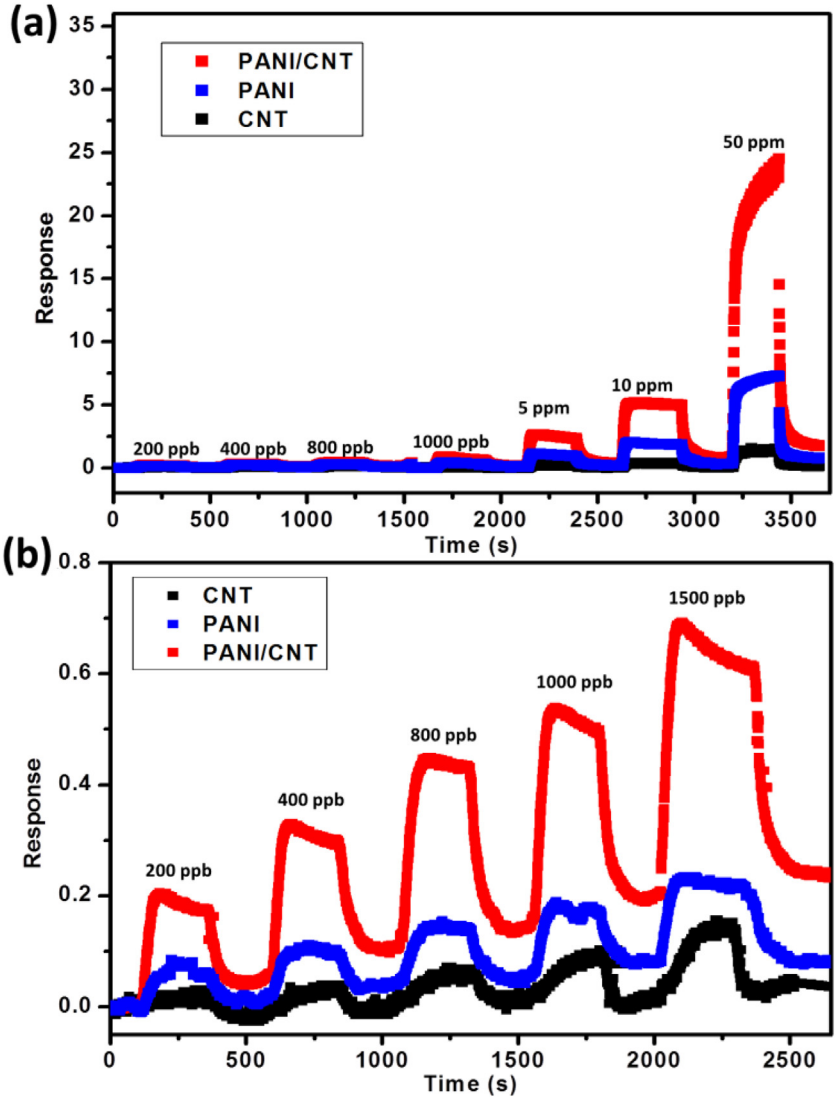


Fig. 4. (a) Gas sensing performance of PANI/CNT nanocomposite, the PANI fiber aggregate and the CNT film devices to different concentrations of NH_3 from 200 ppb to 50 ppm. (b) The detailed gas sensing response of PANI/CNT nanocomposite, the PANI fiber aggregate and the CNT film devices to different concentrations of NH_3 from 200 ppb to 1500 ppb.

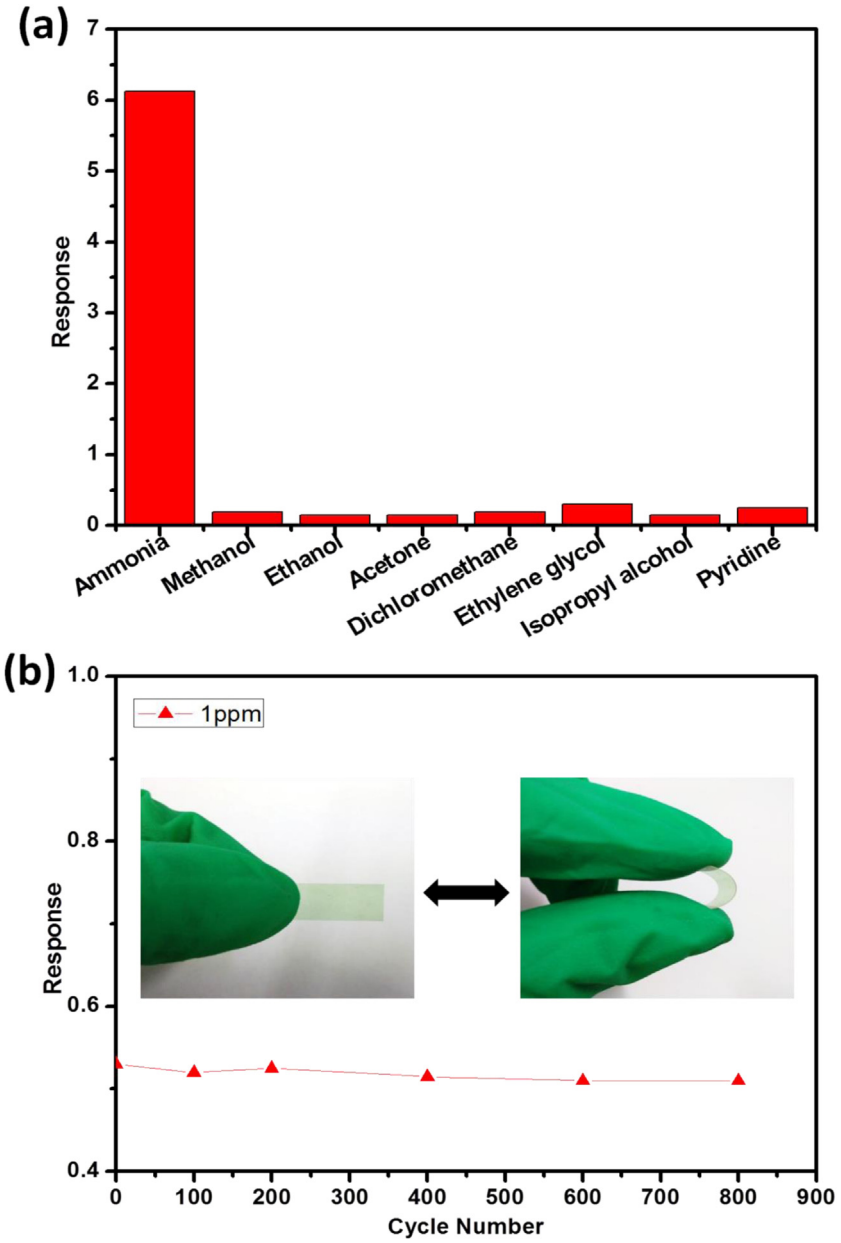


Fig. 5. (a) The selectively sensing response to 10 ppm NH_3 and other volatile organic compounds for the PANI/CNT nanocomposite network film. (b) Gas sensing response of the PANI/CNT nanocomposite network film in bending and extending states to 1 ppm NH_3.

purchased from J&K Chemical. Polyethylene terephthalate (PET, 125 μm) film was obtained from Hui Zhixing Company (Hangzhou, China). Multi-walled carbon nanotubes are commercially obtained from Cnano (FloTubeTM 9000).

2.2. Preparation of PANI/CNT nanocomposite films

The 25 mg multi-walled carbon nanotubes are added into the mixture of 45 mL H_2SO_4 and 15 mL HNO_3 acid, and sonicated for 8 h, then diluted to neutral state [24]. The functionalized CNTs are dispersed in ethanol solution to prepare 0.5 mg/ml CNT solution.

200 μL functionalized CNTs ethanol solution was added to 15 mL of 1 M HCl and sonicated for 15 min. Then 82 μL aniline was added. The mixture was refrigerated in the refrigerator for 15 min at 4 °C. Then, 15 mL of 0.06 M APS in 1 M HCl was dropped into the above solution. After that, the PET substrate treated by O_2 plasma was dipped into the above solution to react for 0.5 h at 4 °C. Then the films were taken out and washed with ethanol and dried at room temperature for further characterization. The PANI fiber films were prepared by the same methods without adding CNTs. And CNT film was prepared by dropping 200 μL CNT ethanol solution onto PET substrate.

2.3. Gas sensing measurement

The gas sensing measurement was performed in a WS-30A measuring system (Zhengzhou Winsen Electronics Technology, China) as reported previously[9]. The functional films (8 mm by 8 mm) were attached on the probes of voltage-testing devices with silver paint (the operating humidity range at 30–47%, and the operating temperature at 25–35 °C in Beijing), as shown in Fig. S1. By dropping calculated amount of ammonium hydroxide onto a hot plate in the chamber, the corresponding NH_3 concentrations were rapidly

generated in the chamber. The response of NH_3 is defined as the normalized resistance change.

$$R = (R_g - R_0)/R_0 = \Delta R/R_0$$

where R_g is the resistance of the film after exposure to gas analytes and R_0 is the resistance of the film in the air.

2.4. General techniques

SEM images were obtained from a Zeiss Supra 55 instrument at a voltage of 20 kV. UV–vis transmittance spectra were recorded by a Shimadzu UV-3150 UV spectrophotometer. Fourier transform infrared (FTIR) spectra were collected on a Nicolet 6700 FTIR spectrometer. The sheet resistance was measured by four-probe tester (RST-8).

3. Results and discussion

3.1. Morphology and structure

Hierarchical PANI/CNT nanocomposites were obtained, as shown in Fig. 2a and c. After treatment with inorganic acid, CNTs were functionalized with various oxygenated groups (e.g., carbonyl, hydroxyl and epoxy groups) (Fig. S2), providing well dispersibility for CNTs in aqueous solutions [24]. The functionalized groups acted as active sites for attaching anilinium ions via electrostatic attraction for the further PANI polymerization [25,26]. By adding APS into CNT-containing 0.06 M aniline solution (the ratio of [aniline]/[APS] = 1:1) for in-situ PANI polymerization and film depostion, the hierarchical nanocomposite with the interconnection of PANI nanoparticle-coated CNT and PANI nanofiber were obtained and deposited on PET substrate. For comparison, the PANI fiber aggregates were obtained in aniline polymerizing solution without CNTs on PET film, as demonstrated in Fig. 2b and d. In our previous report, after aniline was dropped into the mixed solution of CNT and APS (an aniline concentration at 0.01 M and a ratio of [aniline]/[APS] at 1.75:1), PET was dipped into the solution to react for 6 h, 8 h, 10 h, or 12 h. The hierarchical nanocomposite network of the PANI nanorods-coated FMWCNTs was obtained[17].

FTIR spectra were conducted to confirm the formation of the hierarchical PANI/CNT nanocomposites. As shown in Fig. 3, for CNTs, the main peaks at 1630 cm^{-1} are attributed to the stretching of the C=C in the quinoid ring. For PANI, characteristic bands are observed at 1560 cm^{-1} (C=C stretch of the quinoid ring), 1470 cm^{-1} (benzenoid ring) and 1300 cm^{-1} (C–N stretch of the second aryamine), respectively[25]. For PANI/CNT nanocomposite, the main peaks centred at 1575 cm^{-1} and 1470 cm^{-1} are attributed to the C=C stretching vibration of the quinoid ring and the benzenoid ring in the PANI chains, indicating the presence of the emeraldine salt state of PANI

3.2. Gas-sensing properties of PANI/CNT nanocomposite

NH_3 is a volatile, corrosive and poisonous gas, which can cause inhibitory and toxic effects on anaerobic digestion for methane production[27]. The prepared hierarchical PANI/CNT nanocomposite films were assembled as flexible gas sensors at room temperature for highly sensitive NH_3 sensing to potential real-time discovery of ammonia inhibition phenomenon. Fig. 4 demonstrates the gas sensing response for PANI/CNT nanocomposite, PANI fiber aggregate and the CNT film devices to different concentrations of NH_3 from 200 ppb to 50 ppm at room temperature (the detailed response to NH_3 from 200 ppb to 1500 ppb in Fig. 4b). The response increased gradually with the increased NH_3 concentration. Meanwhile, the response for the PANI/CNT nanocomposite film is better

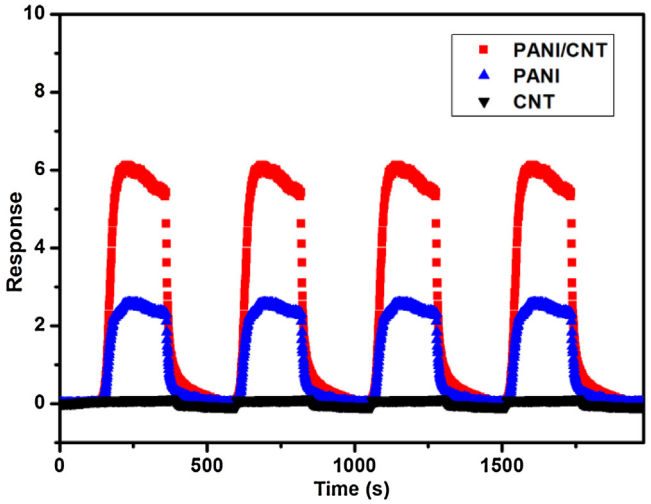


Fig. 6. The sensing repeatability for PANI/CNT nanocomposite, the PANI fiber aggregate and the CNT film sensors to 10 ppm NH_3.

than that for the PANI fiber aggregate and the CNT film devices, displaying the special synergistic effects of PANI/CNT nanocomposite by combined excellent properties of individual materials for high-performance gas sensing. The sensing response of PANI/CNT nanocomposite could still be obtained for NH_3 concentration as low as 200 ppb, implying its high-sensitive NH_3 sensing in chemical detection.

Meanwhile, the selectively sensing response to NH_3 for the PANI/CNT nanocomposite network film was shown in Fig. 5a, compared to other volatile organic compounds with the likely concentrations of interfering gases inside a potential fermentation reactor[1,3,6]. The sensing repeatability for PANI/CNT nanocomposite, the PANI fiber aggregate and the CNT filmsensors was displayed in Fig. 6, showing well NH_3 sensing cycles. The reliable flexibility of the chemical gas sensor device is vitally important for potential integration into wearable electronics. Thus, the flexibility of chemical gas sensing performances was measured. As shown in Fig. 5b, no obvious changes were observed for the sensing response in bent and extending states after 800 bending/extending cycles at room temperature, due to the superior confined nanocomposite network film, revealing the reliable and robust flexibility of the device.

3.3. Sensing mechanisms of PANI/CNT nanocomposite to NH_3

As can be seen from Fig. S3, the response/recovery time is 85 s/20 s, respectively. This high-sensitive sensing performance could be attributed to the synergistic properties of CNT and PANI, the interconnected nanocomposite network with high surface area (Fig. 7) [28]. Firstly, the nanocomposite network with relatively high surface area (54.187 m^2/g), compared to 43.296 m^2/g for PANI, is helpful to the reversible adsorption of more NH_3 for the enhanced sensing response. Secondly, the interconnected network worked as efficiently sensing channels to strengthen the fast charge transport. Thirdly, electrons provided from the adsorbed NH_3 molecules onto PANI, transfer easily from PANI to CNT, because of the lower conductivity of PANI and lower energy barrier between PANI and carbon nanomaterials (~101 meV). Then, the electrons effectively transfer from CNT to the PANI fiber due to lower energy barrier, and finally to the electrodes [19–21,28–31]. Therefore, the sensing response was effectively improved in PANI/CNT nanocomposite network.

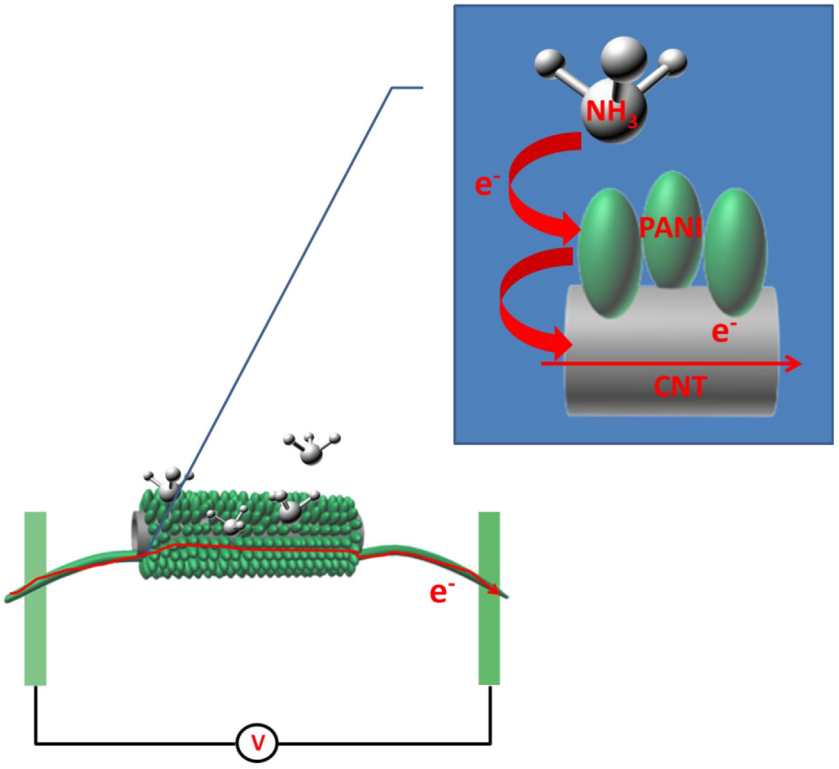


Fig. 7. The proposed NH_3-sensing mechanism of PANI/CNT nanocomposite network.

4. Conclusions

In conclusion, we have fabricated a real-time ammonia (NH_3) detection sensor for potential sensing ammonia in anaerobic digestion for maximum the renewable energy recovery because of the ammonia inhibition of anaerobic digestion for producing renewable energy sources. The sensor was assembled from a hierarchical PANI/CNT nanocomposite network film fabricated by adding APS into CNT-containing aniline solution for PANI polymerization and simultaneous film depostion. It exhibited highly sensitive NH_3 sensing from 200 ppb to 50 ppm, fast response/recovery time (85 s/20 s), room-temperature operation without external aid, reliable flexibility and excellent selectivity to NH_3 compared to other volatile organic compounds. The excellent sensing performance is probably ascribed to the synergetic effects of PANI and CNT, the high surface area (54.187 m^2/g) of nanocomposite films, and effective network sensing channels. The film with simple preparation, high sensitivity, small size and robust flexibility, could be implanted into electronic devices for potentially monitoring NH_3 in anaerobic digestion in real-time for the high efficiency and better stability of anaerobic digestion for renewable energy sources.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.snb.2016.12.064.

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Professor Guangqing Liu serves as the Executive Director of Biomass Energy and Environmental Engineering Research Center at Beijing University of Chemical Technology, also the Executive Secretary-general of China Alliance for Clean Stoves. Dr. Liu has published about 50 academic papers, 5 books and chapters and got 7 China Patents. As the PI, he got more than 20 program grants for China Ministry of Science and Technology, China National Science Founding, World Bank etc. He was invited as the China representatives to give presentation in more than 20 international conferences in USA, Canada, Japan, India, Indonesia, Thailand, Peru, Philippines etc.

Pengbo Wan received his B.S. degree (2006) at Wuhan University. Then he was recommended to join Prof. Xi Zhang's group as a Ph.D. candidate at Tsinghua University and got his Ph.D. degree in 2011. During his Ph.D. study, he worked as an exchange Ph.D. student at University of Leuven, Belgium. After working as a research fellow at Nanyang Technological University in Singapore, he joined Beijing University of Chemical Technology as an Associate Professor in 2013. His research interests include organic/inorganic hybrid nanomaterials for energy conversion and storage, stimuli-responsive biointerface, bioelectrocatalysis, supramolecular assembly, and nanoelectronics.