



Sensing methamphetamine with chemiresistive sensors based on polythiophene-blended single-walled carbon nanotubes

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ABSTRACT

A highly sensitive and selective carbon nanotube-based vapor sensor was developed toward the detection of methamphetamine vapor, one of the most widespread, harmful and addictive illegal drugs in the world. Poly[3-(6-carboxyhexyl)thiophene-2,5-diyl] (P3CT) was chosen to non-covalently functionalize carbon nanotubes, which can facilitate the suspension of carbon nanotubes in the solvents as well as introduce a carboxylic acid functional group. The carboxylic acid group in the polymer acts as a binder of organic amines via acid-base interaction. The P3CT-functionalized carbon nanotube sensors show sensitivity to *n*-methylphenethylamine (NMPEA), a detection simulant of methamphetamine, as low as 4 ppb. The sensors were able to distinguish NMPEA from two other amine compounds, various volatile chemical compounds (VOCs), and water vapor by observing the recoverability of the sensor's signal after exposure. The sensor showed higher sensitivity to amine vapors even when the vapor concentrations of amines were several orders of magnitude lower than those of the VOCs. Our research gives a simple but effective method for detecting trace amounts of methamphetamine in ambient air. Meanwhile, the sensor developed in this study can be used as one component in a next generation portable, intelligent, and low-cost chemical sensor array system for more complex real-life applications.

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1. Introduction

N-methamphetamine (NMPA) is a highly addictive central nervous system stimulant and an illegal drug that is a worldwide health problem. [1] The negative consequences of taking NMPA affect not only an individual's health but also his/her family and friends. The synthesis of NMPA is relatively simple and there are clandestine labs producing NMPA illegally worldwide. [2] Meanwhile, these clandestine labs usually lack appropriate protection methods during the synthesis and often cause severe contamination of their local environment. Thus, a sensitive, portable, and reliable NMPA vapor sensor may be used to detect clandestine labs and drugs in transit.

Previous methods developed for detecting NMPA include gas chromatography (GC), [3] mass spectrometry (MS), [4] GC/MS, [5] electrochemiluminescence (ECL), [6] Raman spectroscopy, [7] liq-

uid chromatography, [8] surface ionization [9] and fluorescence sensors. [10–13] Those sensing methods, especially those related to analytical instruments, have the advantage of accurate detection of trace amounts of NMPA in complex samples such as human urine and blood. They are used for screening for drug abuse. However, they have drawbacks of extensive sample preparation, expensive and cumbersome instruments, and complicated instrumental operation and not suitable for the applications of real-time detection and monitoring of NMPA in law enforcement scenarios. Thus, a sensor for NMPA that is simple and affordable is still highly demanded.

Electronic sensors such as chemiresistive sensors [14–17] are promising to fulfill this demand, as they are usually simple and inexpensive to make. [17] In a chemiresistive sensor, only source electrode and drain electrodes are needed for measurements of the signal and only small amount of the sensor materials (in the range of micro-grams) are needed for making a device. Chemiresistive sensors integrate with circuit boards easily and can be small in size; thus, a hidden detector that communicates wirelessly to a computer or a small cell phone accessory for NMPA detection is easy to achieve. Carbon nanotubes (CNTs) are promising materials to use because of their high property sensitivity

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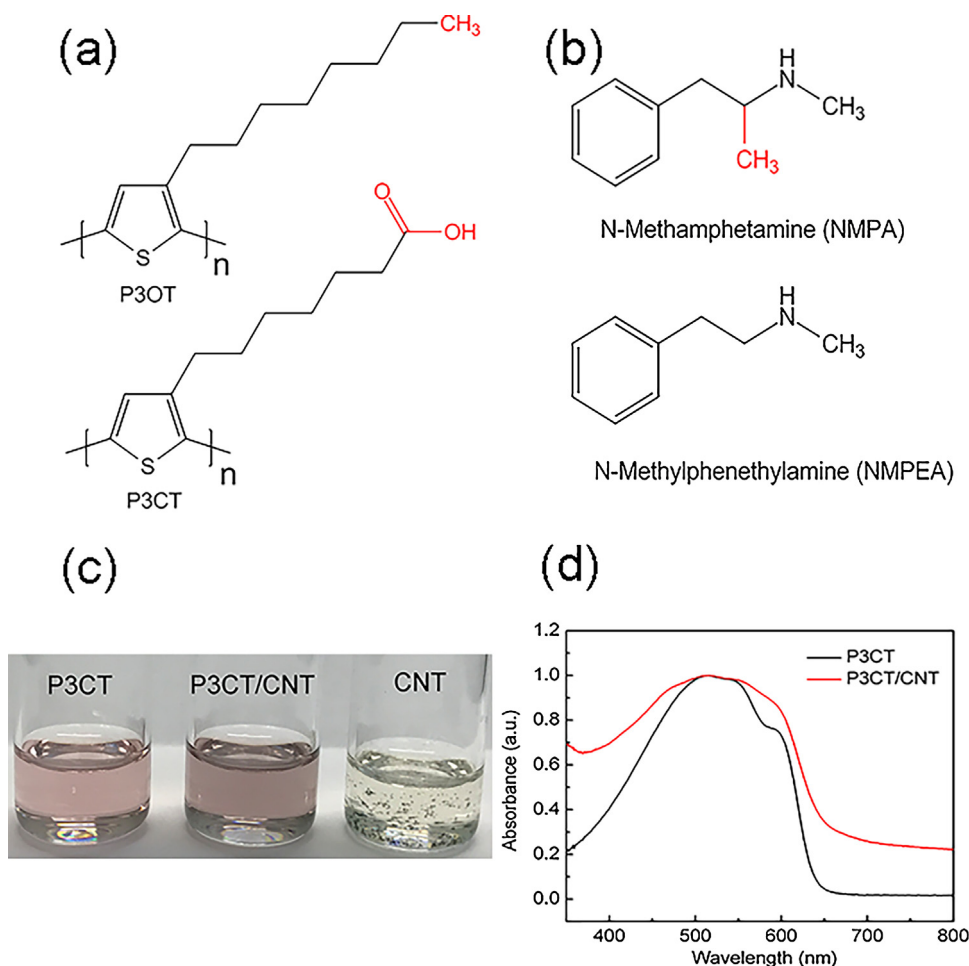


Fig. 1. (a) Molecular structures of two polythiophene derivatives (P3OT and P3CT) for noncovalent functionalization of CNTs. (b) Molecular structure of NMPA and its analog NMPEA which was used in the vapor sensing tests. (c) Photos of P3CT (7.5 $\mu\text{g}/\text{mL}$), P3CT/CNT and CNT in DMSO. (d) UV-vis spectra of P3CT and P3CT/CNT in DMSO.

to environmental changes, [18] good conductivity, and availability for surface functionalization. [19–21] Previous studies have shown numerous examples of CNT-based chemiresistive sensors for monitoring trace amounts of dangerous gases and chemical vapors such as carbon monoxide, [22] hydrogen sulfide, [23] and diethylchlorophosphate [24] in the environment. However, little to no work has been published on using carbon nanotubes as NMPA vapor sensors. Herein, we designed a chemiresistive sensor based on single-walled CNTs functionalized with a polythiophene derivative for the detection of trace NMPA vapor. Polythiophene has been demonstrated as an effective materials to noncovalently functionalize CNTs and disperse them in solvents. [25] We chose poly[3-(6-carboxyhexylthiophene-2,5-diyl)] (P3CT) (Fig. 1a) because of the hypothesis that the carboxylic acid group in the polymer will facilitate the sensing of NMPA because the carboxylic acid tends to react with the amine group in the NMPA through acid-base interaction. We have demonstrated our hypothesis by directly comparing sensors functionalized with a very similar polymer without carboxylic acid functional group, namely, poly(3-octylthiophene-2,5-diyl) (P3OT) (Fig. 1a).

2. Results and discussion

UV-vis absorption spectra (Fig. 1d) were obtained from the P3CT/CNT suspension and the P3CT solution using an Agilent Cary 100 UV-vis spectrophotometer. The UV-vis absorption spectra of P3OT/CNT suspension and P3OT solutions were reported in our

previous study. [26] The spectrum of the P3CT shows a broad absorption band with maximum absorption wavelength at around 515 nm and a weak shoulder around 600 nm, which is very similar to the previously reported UV-vis absorption spectra of poly(3-hexylthiophene) [27]. Upon mixing with CNTs, the overall baseline of the absorption spectrum of P3CT/CNT is increased in comparison to the P3CT solution due to the absorption of CNTs in longer wavelength.

To test our sensors with NMPA, we used a simulant, *n*-methylphenethylamine (NMPEA), which has a chemical structure very similar with NMPA (Fig. 1b). Fig. 2a shows the response of a representative P3CT/CNT sensor to 4 ppb, 8 ppb, 16 ppb and 32 ppb of NMPEA vapor, which was 0.001%, 0.002%, 0.004% and 0.008% diluted from the saturated vapor of NMPEA (395 ppm under 25 °C). [28] The exposure to NMPEA vapor lasted 20 s and was followed by a recover period of 40 s. Each concentration of NMPEA was introduced to the sensor 3 times to test the reproducibility. Overall, the sensor's response to NMPEA is fast, large, and semi-recoverable (recoverability defined in the Supporting Information, ranging from 10% to 50%). The sensor shows a clear response at concentrations as low as 4 ppb, indicating a limit of detection lower than 4 ppb for NMPEA. Fig. 2b compares the responses of sensors comprised of P3CT/CNT, P3OT/CNT, and nonfunctionalized CNT sensor to different concentrations of NMPEA vapor tested simultaneously. The sensitivity of the P3CT/CNT sensor towards NMPEA vapor is more than an order of magnitude greater than that of the nonfunctionalized CNT sensors and more than 6 times greater than that of

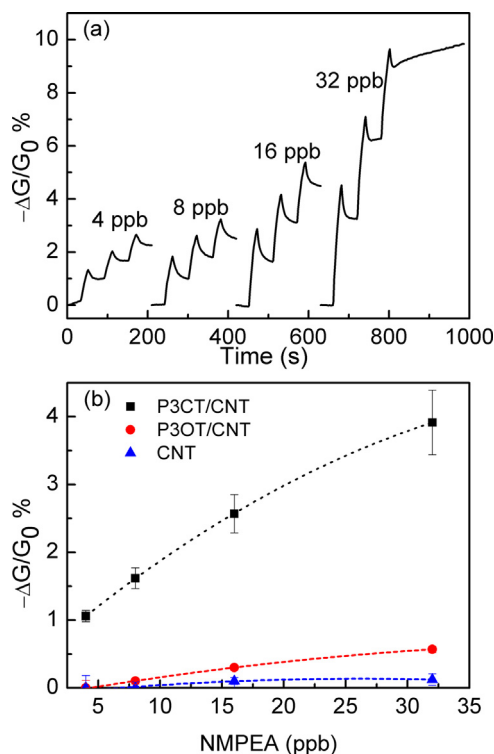


Fig. 2. (a) Real-time sensing responses of a P3CT/CNT sensor towards 4 ppb, 8 ppb, 16 ppb and 32 ppb of NMPEA vapor. The vapor exposure time is 20 s followed by a 40 s recovery time. (b) Responses of a P3CT/CNT sensor, a P3OT/CNT sensor, and a non-functionalized CNT sensor to different concentrations of NMPEA vapor in the same testing environment (dashed lines are the quadratic fitting).

the P3OT/CNT. This demonstrated the improved sensitivity of the P3CT/CNT sensor to NMPEA due to the carboxylic acid group of P3CT that enables strong binding with amines.

Selectivity of the P3CT/CNT sensor against other common chemicals is very important when the sensor is used in real-life applications. Thus, we tested the P3CT/CNT sensor to a number of common volatile organic compounds (VOCs) and water vapor at the concentration of 1% of their saturated vapor pressure, as well as, two amines (aniline and benzylamine) at the concentration of 0.008% of the saturated vapor. The selectivity of the P3CT/CNT sensor draws from two aspects. The first one is the recoverability of the response and the second is the sensitivity. Fig. 3 shows the real-time response profiles of a P3CT/CNT sensor to aniline, benzylamine, toluene and ethyl acetate vapors. The sensor responds to the two amine vapors and after the exposure, the sensor seems to continue to respond but at a slower rate and does not recover. On the contrary, the sensor's responses to toluene and ethyl acetate vapor are fully recoverable (the same as observed for other vapors such as acetone, water and ethanol). This is interesting because we can simply distinguish amines and other common VOCs by observing the recovery behavior of the sensor. Moreover, the sensor also shows significantly higher sensitivity to amines than other VOCs. Fig. 4 shows the responses of the three kinds of sensors to various VOCs at the concentration of 1% of their saturated vapor pressure [29] and also, 32 ppb of NMPEA vapor. Although the vapor concentration of NMPEA is more than four orders of magnitude lower than the VOCs, the P3CT/CNT sensor still shows about one order of magnitude higher response to NMPEA compared to its response to the VOCs. This dramatic difference in response sensitivity helps distinguish NMPEA from the common VOCs.

The mechanism for the high sensitivity of the P3CT/CNT sensor to amines is probably a combination of the swelling effect of the conductive CNT network in a non-conductive polymer matrix and

the intrinsic sensing response of bare CNTs to amines, considering the fact that there is always some surface area of CNT free from polymer coverage. The intrinsic response comes from the interfacial electron transfer from the electron-donating amines to the bare surface of CNT where is not covered by the polymer, which results in a decrease of the conductivity of the *p*-type CNTs. [18] Meanwhile, the swelling process also gives a decrease of conductivity when the sensor is exposed to the analytes. The carboxylic acid group in the P3CT acts as a receptor of amine compounds [24] [30,31] and could enhance the interaction between the amine compounds and the sensor materials.

The non-recoverable response of the sensors after exposure to amine vapors may come from the intrinsic properties of the vapor itself, since they tend to be bound to the surface of CNT through charge transfer interaction. Additionally, the carboxylic acid in the P3CT may also decrease the recoverability of the sensor after exposure to amine compounds because of the acid-base reaction. The semi-recoverability of the P3CT/CNT sensor to NMPEA, which is different from the non-recoverability of other amine compounds, may be due to the increased steric hindrance of NMPEA (a secondary amine), which may decrease the surface binding strength.

3. Conclusion

In conclusion, efficient sensors of NMPEA were developed by using CNTs functionalized with P3CT as the chemiresistive sensing materials. The sensors were made by simply drop-casting the P3CT/CNT suspension on the interdigitated electrodes (IDEs). NMPEA was detected at concentrations as low as 4 ppb. The acid-base interaction between the amine compounds and the carboxylic acid groups in the polymer may be the reason of the extraordinary sensitivity of the P3CT/CNT sensor to amine compounds. The sensor was able to distinguish NMPEA from two other amines, various common VOCs. The selectivity of our sensor to NMPEA comes from both the recoverability of the sensor after exposure to the analytes and the much enhanced sensitivity to amine compounds.

4. Experimental section

4.1. Materials

Regioregular P3CT and regioregular P3OT were purchased from Reike metals. Carbon nanotubes (SG65i, single walled, >95% semiconducting species) were purchased from SouthWest NanoTechnologies. All the other chemicals were purchased from Sigma Aldrich or Fisher and used as received. Amine compounds used in this study for the vapor testing were 99% or higher in their purity.

The method of making the P3CT/CNT in dimethyl sulfoxide (DMSO) and P3OT/CNT in 1,2-dichlorobenzene (*o*-DCB) is very similar to the method developed previously in our lab.²⁶ [32] Briefly, CNTs were suspended in *o*-DCB (for P3CT/CNT suspension) or DMSO (for P3OT/CNT suspension) with a concentration of 1 mg/mL, followed by 2 h of sonication. P3CT was dissolved in *o*-DCB and P3OT was dissolved in DMSO with a concentration of 5 mg/mL. Then, the polymer solution and the CNT suspension in the corresponding solvent were mixed with a polymer:CNT weight ratio of 3:1. The mixtures were sonicated for 2 h and then diluted into corresponding solvents to achieve a concentration of CNT of 2.5 μg/mL (estimated) and the diluted suspensions were sonicated again for 2 h. The final suspension of P3CT/CNT was purple to pink in color, and was darker than the solution with only P3CT in it. The suspension was stable without forming aggregates after 3 months while the CNT in DMSO formed aggregations after several days (see Fig. 1c). Overall, the process of making CNT suspensions with P3CT and P3OT is simple, reproducible, and cost-effective.

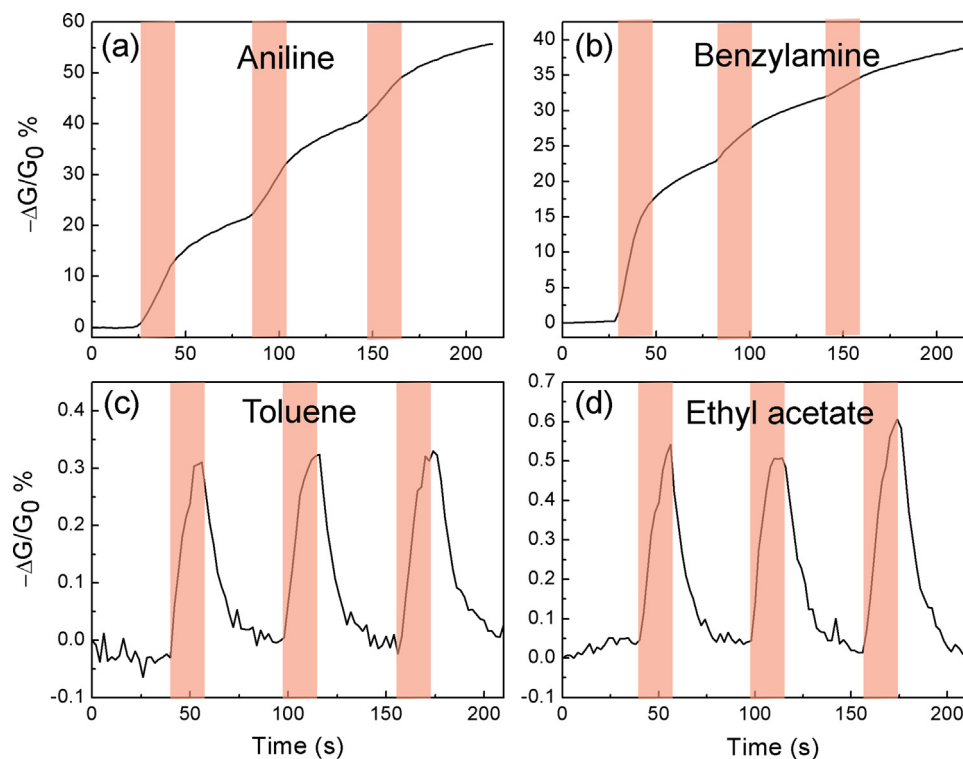


Fig. 3. Real-time sensor's responses to vapors of aniline (64 ppb), benzylamine (74 ppb), toluene (375 ppm), and ethyl acetate (1244 ppm) from a P3CT/CNT sensor. The colored bars represent the time when analyte vapors expose to the sensor.

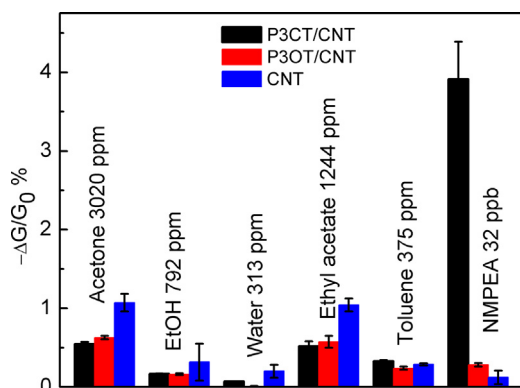


Fig. 4. Responses of the P3CT/CNT sensor, the P3OT/CNT sensor, and the non-functionalized CNT sensor to 20 s vapor exposures of various compounds (1% of saturated vapor) and 32 ppb of NMPEA. Note: the vapor concentration of NMPEA is more than four orders of magnitude lower than the reference VOCs.

The process of fabricating sensor devices is the same as we used in our previous study.^{26, [32]} Sensors were fabricated by drop-casting the P3CT/CNT suspension or the P3OT/CNT suspension onto a pre-patterned interdigitated electrodes (IDEs) (the gap between fingers is 80 μm , see supporting information for details of fabrication with standard photolithography process in a clean room). The IDE chip was then heated to 120 $^{\circ}\text{C}$ for 10 min in an ambient environment to remove the remaining solvent. After that, the electrical resistance of the IDE was tested. Drop-casting the suspension and drying was repeated until the resistance of the device reached 20–200 $\text{k}\Omega$, forming a thin film of polymer/CNT on the IDEs.

The sensors were tested using a homemade chemical vapor generation and testing system. Saturated vapor of analytes other than amine-related analytes (including NMPA, aniline and benzylamine) were loaded into a glass syringe in a programmed syringe pump (NE-4000 New Era Pump System, Inc.). Amine related ana-

lytes were diluted 1000 times by volume before loading to the glass syringe. The analyte vapors in the syringe pump were pumped into a 100 sccm carrier gas (dry air) at rates of 1.01 sccm, 2.04 sccm, 4.17 sccm and 8.7 sccm to create analyte concentrations of 1%, 2%, 4% and 8% of each analyte's concentration in the glass syringe. The syringe pump was programmed to infuse 20 s of the saturated analyte vapor into the carrier gas with a duty cycle of 60 s in order to achieve different diluted vapor concentrations of the analyte. The process was repeated three times to examine the reproducibility of sensor response. The diluted vapors were delivered to a PTFE chamber with sensors inside. The sensors were wire-bonded to a ceramic chip carrier and connected to an Agilent 4156C Precision Semiconductor Parameter Analyzer. The sensors were operated at a constant DC bias of 0.1 V and the electrical currents were monitored by the semiconductor analyzer. Matlab and Microsoft Excel were used to perform the baseline correction and the data analysis.

Notes

The authors declare no competing financial interest.

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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.2017.08.201>.

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