Sensor materials

Flexible and Printed Chemiresistive Ammonia Gas Sensors Based on Carbon Nanotube and Conjugated Polymers: A Comparison of Response and Recovery Performance

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Abstract—Carbon nanotube (CNT)-based chemiresistive gas sensors are known to not fully recover to the baseline after gas exposure. This is why often heat is applied to thermally activate sensor and improve the gas desorption process occurring at the surface. In this letter, we combine single-walled CNTs (SWCNTs) with conjugated polymers (CPs), i.e., Poly(3-hexylthiophene)(P3HT), to realize chemiresistive gas sensors with improved recovery. Five configurations were fabricated with diverse positions of the sensing material layers with respect to the interdigitated electrodes (IDEs), namely: SWCNTs/IDEs and P3HT/IDEs as controls for the CNTs and P3HT singular materials: SWCNTs/P3HT/IDEs, SWCNTs/IDEs/P3HT, and P3HT/SWCNTs/IDEs as combined sensing materials layers configurations. The results show that in particular, the P3HT/SWCNTs/IDEs sensors exhibit better performance with respect to the other configurations when exposed to 5, 25, and 50 ppm NH_3. Improvements in terms of response time (103 s faster at 5 ppm), sensor response (+26.83% at 50 ppm), and improved recovery to the baseline (+25.69% and +11.34% at 5 and 50 ppm, respectively) were obtained when evaluated against SWCNTs/IDEs sensors. These findings suggest that addition of CPs (i.e., P3HT) may be a promising approach to enhance the performance and stability of SWCNTs-based chemiresistive gas sensors, providing a new direction for future research in this field. Future research to investigate the effect of others CPs on the CNTs based chemiresistive gas sensors will be needed to realize more efficient and reliable ammonia gas sensors.

Index Terms—Sensor materials, manufacturing processes, ammonia, carbon nanotubes, chemiresistive gas sensor, conjugated polymers, flexible gas sensors, P3HT.

I. INTRODUCTION

Chemiresistive gas sensors are extremely important to detect gases in various fields of applications ranging from industry to healthcare. They typically consist of an active sensing layer and interdigitated electrodes (IDEs). Careful selection of the active materials is essential to achieve an efficient sensor performance, in terms of sensitivity, response, and recovery time, selectivity, linearity, and limit of detection [1]. In particular using flexible substrates [e.g., Polyimide (PI)] provide advantages of being lightweight, cost-effective, and possesses the ability to bend which is crucial for applications in wearables, flexible displays, and healthcare [2], [3]. To date, semiconducting metal oxides (MOX) are the most used sensing materials in chemiresistive gas sensors. MOX-based sensors operating temperatures are in the range of $400\,°C$ to more than $700\,°C$ [4], [5]. Nevertheless MOX-based gas sensors exhibit high sensitivity to several gases, their poor selectivity is one of the principal issues [6]. Carbon-based materials such as carbon nanotubes (CNTs) and graphene are undoubtedly promising alternatives, overcoming current limitations of MOX, principally due

to the fact that their sensing mechanism is effective at room temperature [7]. Thus, single-walled carbon nanotubes (SWCNTs), 1-D carbon allotropes, are especially interesting due to their unique mechanical, electrical, and optical properties, in gas sensing [8]. Due to their outstanding properties in terms of high surface area and sensitivity SWCNTs based gas sensors have been extensively studied for the detection of a variety of gases among which ammonia (NH_3) is one of the most important in medical care [9], [10].

Despite these advantages, also SWCNTs gas sensors come with their own set of challenges mostly related to recovery time, limited gas detection, poor selectivity to different gasses and inability to work in humidity conditions [11]. Hoa et al. [12] showed SWCNTs sensor with sensor response of 17% when exposed to 40 ppm of NH_3. In fact, it was observed that the recovery to the initial baseline resistance was incomplete after the gas exposure was stopped. Hannon et al. [13] functionalized SWCNTs (f-SWCNTs) with carboxylic acid and found an incomplete recovery for both pristine SWCNTs (20–30%) and f-SWCNTs (60–70%) along with a drift in the baseline. The incomplete recovery and drift, due to the gas chemical adsorption, can lead to false readings, needs for recalibration, and decreased accuracy over time [14]. UV irradiation, and heating are some of the methods more often used to improve the gas desorption behavior [15]. One original and interesting method to overcome SWCNTs based gas
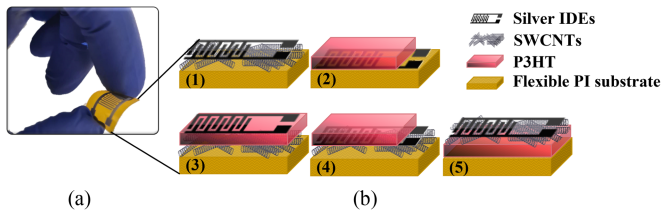
Fig. 1. (a) Final fabricated flexible chemiresistive sensor with schematization of the diverse configurations studied in this work (b): (1) control SWCNTs (SWCNTs/IDEs), (2) control P3HT (IDEs/P3HT), (3) SWCNTs/P3HT/IDEs, (4) SWCNTs/IDEs/P3HT, (5) P3HT/SWCNTs/IDEs.

sensors drawbacks in terms of recovery time and sensor response is to combine SWCNTs with conjugated polymers (CPs) such as polythiophene (PTh) (e.g., poly(3,4-ethylenedioxythiophene) (PEDOT) and poly (3-hexylthiophene-2,5-diyl) (P3HT)), polyaniline (PANi), or polypyrrole (PPy). This approach was already employed with multiwalled carbon nanotubes (MWCNTs) based gas sensors. Husain et al. [16] demonstrated that a PTh/MWCNTs based sensor was ultrasensitive to ammonia with a detection limit of 0.1 ppm. Boonthum et al. [17] found an incomplete recovery of chemiresistive gas sensors with MWCNTs fabricated via thermal chemical vapor deposition (CVD) technique after exposure toward 80 ppm NH_3. Interestingly, MWCNTs based gas sensors with the addition of CP composed of poly(3,4-ethylenedioxythiophene) polystyrene sulfonate (PEDOT:PSS) provided a better recovery to the baseline. Nevertheless, under 80 ppm NH_3, the response value of MWCNTs based sensor reduced from 67.5% to either 66.3%, 44.6%, or 32.5% when adding PEDOT:PSS in concentrations of 87.8 v/v, 88.8 v/v, or 90.3 v/v, respectively.

Despite the great promises, to date there are only very few research works combining SWCNTs with CPs for gas sensing. To the best of our knowledge, here we report for the first time a flexible chemiresistive gas sensor based on the combination of SWCNTs and CP, namely P3HT, for the sensing of NH_3 gases. In particular, we present a comparative study between different sensor configurations dependent on the position of the sensing materials (SWCNTs, P3HT, and their combination) with interdigitated electrodes (IDEs) printed on flexible PI substrates. The best obtained sensor based on SWCNTs combined with P3HT exhibited improvements in terms of response time (103 s faster at 5 ppm NH_3), sensor response (+26.83% at 50 ppm NH_3), and improved recovery to the baseline (+25.69% and +11.34% at 5 and 50 ppm NH_3 respectively) respect to SWCNTs based sensors.

II. MATERIALS AND METHODS

A. Sensor Fabrication

The presented chemiresistive NH_3 gas sensors consist of three main elements: i) a PI substrate, precleaned and treated in oxygen plasma (30%, 1 min); ii) the sensing material, SWCNTs, and P3HT layers deposited by spray- and spin-coating, respectively; iii) screen-printed silver (Ag) IDEs. Five configurations were fabricated with diverse positions of the sensing material layers with respect to the IDEs (see Fig. 1), namely: control SWCNTs (SWCNTs/IDEs), control P3HT (IDEs/P3HT), SWCNTs/P3HT/IDEs, SWCNTs/IDEs/P3HT, and P3HT/SWCNTs/IDEs. For the sensor fabrication, carboxymethyl cellulose (CMC) was used as a surfactant to disperse the SWCNTs (P3-SWNT (CNTs) (Carbon Solutions, Inc., Riverside, CA, USA) in water and untangle their bundles in a concentration of 0.5%wt. following [18], [19]. The already made CMC stock solution was

then combined with SWCNTs at a concentration of 0.05% wt. The dispersion was homogenized for 25 min, alternating 50% and 30% of the power in 5 min cycles, using a horn sonicator probe (Fisherbrand FB-505). The resultant dispersion was centrifuged for 100 min at 13 000 rpm using a Thermo Scientific SL 16 centrifuge (Waltham, MA, USA). Before the spray deposition, the CNTs main dispersion was diluted in 1.3 mM CMC using different dilution ratios, for the preparation of a 1:15 dilution. 20 layers of SWCNTs were spray-coated on PI substrate from the obtained SWCNTs solution (Spraymatic with an industrial air atomizing spray valve Nordson EFD, USA), with 1 min of drying at 70 °C in between two 10 layers spray cycles. The sensors with sprayed SWCNTs with the respective configurations underwent post acid treatment by being submerged in nitric acid (HNO_3) solution (HNO_3 65%, 1/5 dilution) for 60 min, followed by H_2O for 10 min. Samples were then dried for 60 min at 100 °C on a hotplate. For the IDEs, commercial Ag paste (EC-1010 from LOCTITE) was screen-printed using a semi-automatic screen-printer (C290, from Aurel automation S.P.A.) and consequently cured at 120 °C for 15 min. Poly (3-hexylthiophene-2,5-diyl) (P3HT, 93,6% Regio-regular, MW 24.480 from Ossila) was dissolved in chlorobenzene (anhydrous 99.8%, from Merck) in concentrations of 2 mg/ml and stirred overnight at 60 °C. 60 μl P3HT:CB solution was spin-coated at 1000 r/min for 60 s on the substrates. After spin-coating, samples were annealed at 100 °C for 10 min. For each sensor configuration, the sensor response of three sensors was evaluated. The HNO_3 treatment used to remove the CMC and to increase SWCNTs conductivity was found to dissolve and damage the silver ink electrodes. It was therefore decided to use only configurations where the SWCNTs is deposited below the IDEs. Furthermore, for the P3HT control, it was decided to present the IDEs/P3HT configuration as with P3HT/IDEs, where the electrodes were easily detached when contacted. By comparing the different configurations, we investigate the effect that the CPs have on the SWCNTs-based gas sensor.

B. Sensor Characterization

The electrical characterization of the obtained sensors was carried out using a custom-made setup consisting of a sealed gas chamber, mass flow controllers (MFCs), and a dedicated data acquisition system, including a high-performance digital multimeter (Keithley Model 2000 with a 6 1/2-digit), a measure unit (Keithley Series 2600B), and a developed LabView program. The sensors were exposed to a continuous flow of synthetic air (20% O_2 and 80% N_2) for 60 min, then the target gas (i.e., NH_3) was injected in the chamber for 20 min. Different concentrations of NH_3 were considered in this work, i.e., 5, 25, and 50 ppm. All tests were performed at room temperature (25.23 °C $\pm$ 0.36) and in dry air with a relative humidity (RH) of 1.75% $\pm$ 1.40. The temperature and the RH were monitored using a PT100 and a Honeywell HIH-4000-001 sensor. All data are presented as the mean on three samples ($n = 3$) and elaborated with OriginPro (2021b). Results are given as means $\pm$ standard deviation (SD). The sensor response was calculated using the following:

$$\text{Response}(\%) = (R_g - R_a)/R_a \times 100 \qquad (1)$$

where R_g is the resistance of the sensor in presence of the analyte gas, NH_3, while R_a is the resistance measured in the presence of air. Here, the response time was calculated as the amount of time it takes for the sensor to respond to a target gas to the extent of 90% of its complete response. Recovery time is the amount of time required for a sensor to retrieve 90% of its initial baseline signal after the gas is switched off [20]. The recovery (%) was calculated as the resistance value measured 60 min after the gas injection stopped, in % with respect to the sensor response (%) of the complete response (100%).

TABLE 1. Values of the Performance of the Five Sensor Configurations When Exposed to 5, 25, and 50 Ppm NH₃ Including Sensor Responses, Response and Recovery Times With Standard Deviation (SD) Representing 3 Sensors. *2 of the 3 Control P3HT (IDEs/P3HT) Sensors Reached Cut-Off Values (>50 MΩ), and Therefore, No Values Could Be Provided. **upward Drift With Increased Resistance

Configuration	NH₃ (ppm)	Injection 1			Injection 2		
		Response% ± SD	Response time (sec) ± SD	Recovery% ± SD	Response% ± SD	Response time (sec) ± SD	Recovery% ± SD
Control P3HT*	5	x	x	x	x	x	x
	25	18.72	867	-28**	12.91	x	x
	50	76.96	839	-10**	33.57	545	-19**
Control SWCNTs	5	7.79 ± 0.88	740 ± 156	71.40 ± 2.26	6.11 ± 0.21	762 ± 181	85.07 ± 2.22
	25	14.49 ± 2.60	796 ± 59	76.53 ± 2.61	12.35 ± 1.55	706 ± 62	84.07 ± 4.17
	50	30.99 ± 3.98	844 ± 20	49.33 ± 1.46	19.20 ± 1.90	743 ± 70	68.17 ± 1.33
SWCNTs/P3HT/IDEs	5	3.43 ± 0.44	1054 ± 129	38.03 ± 17.99	2.51 ± 0.24	820 ± 125	59.67 ± 11.06
	25	12.14 ± 1.65	984 ± 39	16.77 ± 8.35	8.50 ± 0.08	887 ± 89	32.83 ± 9.83
	50	45.27 ± 8.26	966 ± 16	28.00 ± 3.05	25.31 ± 4.19	924 ± 42	53.43 ± 4.46
SWCNTs/IDEs/P3HT	5	3.62 ± 0.40	803 ± 62	62.47 ± 4.45	3.48 ± 0.13	983 ± 11	74.80 ± 1.76
	25	14.33 ± 0.72	983 ± 6	38.83 ± 7.09	11.66 ± 0.58	949 ± 82	49.40 ± 1.65
	50	46.62 ± 1.83	935 ± 33	39.43 ± 3.31	26.36 ± 0.87	912 ± 39	67.60 ± 2.07
P3HT/SWCNTs/IDEs	5	11.22 ± 0.46	637 ± 57	97.09 ± 2.67	10.38 ± 0.32	728 ± 98	99.37 ± 2.97
	25	33.07 ± 0.50	779 ± 77	75.42 ± 0.26	24.98 ± 0.16	693 ± 32	89.16 ± 0.21
	50	57.82 ± 0.96	739 ± 39	60.67 ± 3.20	28.54 ± 0.56	627 ± 74	93.97 ± 4.13

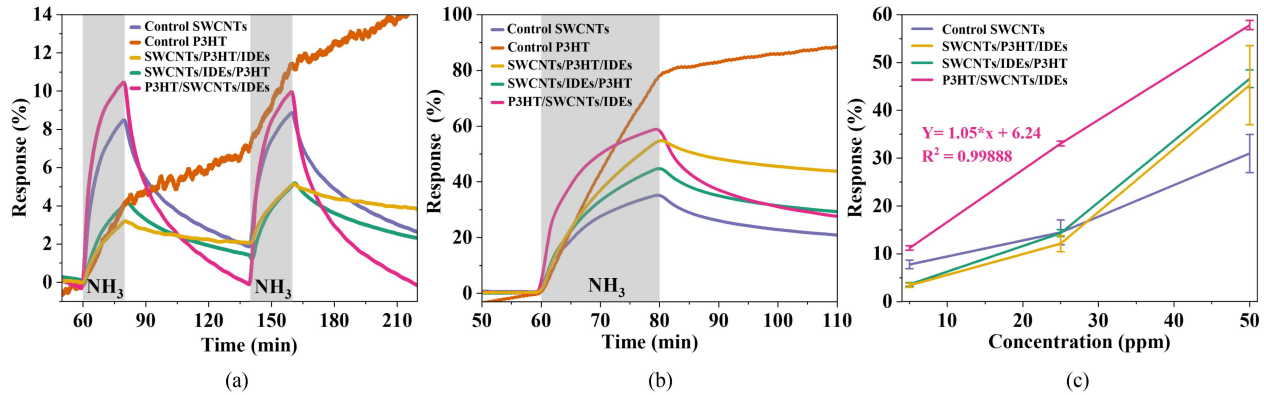Fig. 2. Representative sensor response (of 1 sensor) for each of the five configurations when exposed to (a) 5 ppm of NH₃ for two cycles. (b) 50 ppm of NH₃ (first injection). (c) Calibration curves of control SWCNTs(SWCNTs/IDEs), control P3HT (IDEs/P3HT), SWCNTs/P3HT/IDEs, SWCNTs/IDEs/P3HT, and P3HT/SWCNTs/IDEs for 5, 25, and 50 ppm NH₃ with standard deviations representing 3 sensors for each configuration.

III. RESULTS AND DISCUSSION

A. Sensor Performance

Sensor response values, response time, and percentage of recovery for the compared five configurations are presented in Table 1 and in Fig. 2, where the sensor response plots for 5 ppm NH₃ (a), 50 ppm NH₃ (b), as well as the calibration curves with the response values of 5, 25, and 50 ppm NH₃ (c) are depicted. The control P3HT (IDEs/P3HT) showed very high resistance values close to the cut-off value of 50 MΩ. Additionally, during the 1 h of recovery, an increase in resistance was observed, resulting in an upwards drift of the sensor. Kumpf et al. found similar values for their P3HT-based sensor and further reported that low concentrations of P3HT (<5 mg/ml in toluene) have the tendency to reach cut-off value of 50 MΩ overtime, resulting in a loss of conducting ability [21]. At low concentrations of ammonia (5 ppm), a fluctuation in resistance (noise) made it difficult to accurately calculate the control P3HT sensor performance. Instead, at higher concentrations of NH₃ (25 and 50 ppm), sensor response values of 18.71% and 76.96% were obtained, respectively. These sensor responses are the highest values established for the five configurations. The control SWCNTs (SWCNTs/IDEs) initial resistance was measured to be 4.98 ± 0.93 kΩ. When the SWCNTs were exposed to 5, 25, and 50 ppm of NH₃, sensor response values of 7.79%, 14.49%, and 30.99% were measured, respectively. Dasari et al. exposed SWCNTs, which were functionalized with

carboxyl (–COOH) group using a harsh acid treatment process, toward 50 ppm NH₃ and found sensor responses of 2.9% for pristine SWCNTs and 20.2% for the acid functionalized SWCNTs sensors [22]. This indicates that the SWCNTs/IDEs control has good sensor performance, comparable to the state of the art. When comparing the three combined sensing materials layers configurations SWCNTs/P3HT/IDEs (initial resistance: 78.15 ± 16.67 kΩ), SWCNTs/IDEs/P3HT (initial resistance: 4.38 ± 0.70 kΩ) and P3HT/SWCNTs/IDEs (initial resistance: 3.48 ± 0.74 kΩ), it can be observed that P3HT/SWCNTs/IDEs exhibit the highest response values of 11.22%, 33.07%, and 57.82%, the fastest response times of 637, 779, 739 seconds, as well as the best recovery percentage (%) of 97.09%, 75.42% and 60.67% at the first injection of either 5, 25 and 50 ppm of NH₃ respectively. Since a recovery of more than 90% was obtained for P3HT/SWCNTs/IDEs sensors after 1 h of recovery, it could be determined at which time a 90% recovery was reached. When the first injection of 5 ppm NH₃ was stopped, the sensor reached a 90% recovery after 2342 ± 314 s (±39 min) and the recovery time for the second injection was 2094 ± 339 s (± 35 min). For the second injection of 50 ppm NH₃, it was 2807 ± 342 s (±47 min). SWCNTs/IDEs/P3HT and SWCNTs/P3HT/IDEs sensors did not recover to the baseline within 1 h. Notably SWCNTs/P3HT/IDEs sensors nearly maintained the resistance values of the previously injected NH₃ with an average recovery of less than <30% for the first NH₃ injection (5–50 ppm). It is worth noticing

that in Fig. 2(b) for P3HT/SWCNTs/IDEs, a bigger transient response slope at the beginning of the measurement is observed which decreases with time to a steady response reaching saturation which is needed to make concentration estimations, while for SWCNTs/P3HT/IDEs the response is very slow to reach a steady state [23]. Fig. 2(c) shows a linear behavior for the P3HT/SWCNTs/IDEs sensors when plotting the response values towards 5, 25, and 50 ppm NH$_3$ while the SWCNTs/IDEs/P3HT and SWCNTs/P3HT/IDEs show higher sensitivity for high NH$_3$ concentrations between 25 and 50 ppm. Furthermore, it could be observed that the standard deviation for the P3HT/SWCNTs/IDEs sensors is very narrow. Fig. 2(a) shows that P3HT/SWCNTs/IDEs also outperforms the control SWCNTs (SWCNTs/IDEs) regarding sensor response, response time, and recovery, especially at low NH$_3$ where a complete recovery% (97.09% and 99.37% for injection 1 and 2 of 5 ppm NH$_3$ respectively) is demonstrated. The P3HT/SWCNTs/IDEs are exposed to the nitric acid (HNO$_3$) used for the treatment of SWCNTs, which makes this sensor different from SWCNTs/IDEs/P3HT and SWCNTs/P3HT/IDEs. Conceivably, the acid (H$^+$) molecules are expected to produce more holes in the P3HT semiconducting layer, affecting it with a doping-like reaction. Thus, NH$_3$, when in contact with these holes, can donate its electron and thereby reduce the hole concentration resulting in an increase of resistance. Further investigations will be performed to understand the sensing mechanisms of the P3HT/SWCNTs/IDEs sensor.

IV. CONCLUSION

In this work, five chemiresistive gas sensors consisting of screen-printed silver IDEs on a flexible PI substrate with diverse positions of sensing materials SWCNTs and P3HT were fabricated and investigated in terms of sensor response, recovery, and response times towards different concentrations of NH$_3$ (5, 25 and 50 ppm). The results demonstrate that the addition of P3HT can significantly enhance the sensing performance of SWCNTs-based gas sensors, especially in the P3HT/SWCNTs/IDEs configuration where P3HT was exposed to nitric acid. SWCNTs when used as individual sensing material led to sensors with response of 7.79%, 14.49%, and 30.99% when exposed to 5 ppm, 25 ppm, and 50 ppm of NH$_3$, respectively, with a recovery of approximately 71.40%. SWCNTs with addition of P3HT (P3HT/SWCNTs/IDEs) yielded sensors with an improved response of 11.22%, 33.07%, and 57.82% to NH$_3$ at the same concentrations, with a significantly increased recovery of 97.09%. Stability tests and investigation of humidity effect on the promising P3HT/SWCNTs/IDEs sensors will be part of future investigations. These results add a novel contribution to the growing body of research on SWCNT-based gas sensors and pave the way for further studies to optimize their performance. Further investigation on the improvement of the CNTs recovery is needed. In the future other CPs/CNTs (both SWCNTs and MWCNTs) combinations should be tested to realize efficient and reliable ammonia gas sensors, also investigating the thermal activation for faster recovery.

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