

PEN-BASED WRITING OF FUNCTIONALIZED MWCNT-PEDOT:PSS INK ON FLEXIBLE SUBSTRATE FOR APPLICATION IN AMMONIA GAS SENSOR

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Abstract

Functionalized multi-walled carbon nanotubes dispersed in a polymer mixture of poly (3,4-ethylenedioxythiophene): poly (styrenesulfonate) (*f*-MWCNT-PEDOT:PSS) have been presented as a functional ink for fabrication of room-temperature gas sensor. The ink was deposited onto the flexible substrates with silver interdigitated electrodes by a handwriting method. The sensor shows the sensitivity and selectivity to NH₃ at room-temperature. The high performance of sensor has attributed to the functionalized effect in the increment of extra active area for gas adsorption. The sensor can be worked without noticeable reduction in gas response although it is bent to the heavy disorder. The sensing mechanisms of sensor have proposed based on the reaction between NH₃ molecules and oxygen species on the surface of *f*-MWCNTs. The direct charge-transfers and the film swelling of polymer have been also proposed as possible NH₃-sensing mechanisms. From these findings, the direct ink writing is an effective method for preparing the sensing film of gas sensor. Furthermore, the *f*-MWCNT-PEDOT: PSS ink has the high potential to be selected as a good material for flexible NH₃-sensing applications.

Keywords: Ink, Carbon Nanotube, PEDOT: PSS, Gas Sensor, Pen-Based Writing

Introduction

Ammonia (NH₃) has been known as a technologically important gas for use in various industries associated with pharmaceutical production, medical treatment, environmental applications and food processing. However, NH₃ is toxic, harmful and flammable. Therefore, the gas sensing study has been focused on the NH₃ applications to monitor the leakage of NH₃. Poly (3,4-ethylenedioxythiophene): poly (styrenesulfonate) (PEDOT:PSS), Multiwalled carbon nanotubes (MWCNTs) and their composites based gas sensors have attracted attention according

to their performances in real-time monitoring, fast response and recovery for NH₃ and other hazardous gas detections (Isa *et al.*, 2017; Chiou *et al.*, 2019; Janudin *et al.*, 2018; Lv *et al.*, 2019; Fan *et al.*, 2019). Many researchers have concentrated on the enhancement of NH₃ sensitivity at room-temperature by using the functionalization of some organic compounds and the decoration of metal nanoparticles on the MWCNT sidewalls (Wongchoosuk *et al.*, 2010; Alvi *et al.*, 2014; Han *et al.*, 2014; Alshammari *et al.*, 2017; Casanova-

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Cháfer *et al.*, 2019). Moreover, there have been many techniques reporting the developments of deposited sensing films in terms of different printing methods such as inkjet printing (Pakdee *et al.*, 2019, 2020), screen printing (Pugh *et al.*, 2015), three-dimensional (3D) printing (Wu *et al.*, 2018), transfer printing (Sosada-Ludwikowska *et al.*, 2019), roll-to-roll (R2R) printing (Lin *et al.*, 2015) and pen-based direct ink writing (Loh *et al.*, 2019). Among these processes, the direct ink writing is outstanding because it is being a fast method, having low cost and being user-friendly process to deposit the sensing films onto the substrates. However, the low solubility of MWCNTs in various solutions is still an important problem for the preparation of functional ink.

In this study, the room-temperature NH_3 gas sensor based on MWCNT-PEDOT: PSS composite ink was fabricated using a direct-handwriting method. An ordinary office marker pen was modified for the deposition of sensing ink onto the flexible and transparent substrates with silver interdigitated electrodes. The low solubility of MWCNTs in solution was managed together with an enhancement of NH_3 gas-sensing properties by using the functionalization of carboxylic groups (COOH) onto the MWCNT sidewalls. Furthermore, the NH_3 sensing behavior of fabricated sensor was evaluated under the curvature-disorder conditions. The sensing mechanism of *f*-MWCNT-PEDOT: PSS towards NH_3 was also discussed based on chemisorbed oxygen reaction, direct charge-transfers and polymer swelling.

Materials and Methods

Growth of MWCNTs on Stainless-Steel Foils

The stainless-steel (SS) foils (Type 304) with a thickness of $50\mu\text{m}$ were used as catalytic substrates. The MWCNTs were grown on the 304SS foils by thermal chemical vapor deposition (CVD) using acetylene (C_2H_2) gas as a carbon source. The foils were placed in a home-built thermal CVD system. The system has been explained by the previous work of the first author (Pakdee *et al.*, 2017). The MWCNTs were grown under atmospheric pressure with the temperature of 700°C while the argon (Ar), hydrogen (H_2) and acetylene (C_2H_2) gases were fed into the reactor chamber with the flow rate of 50, 200 and 160 sccm , respectively.

Fabrication of Gas Sensor

After the MWCNT growth, the MWCNT powder was scraped off by a plastic rod. The powder was then placed in a quartz tube which inserted in a tubular furnace for removing the amorphous

regions on the MWCNT sidewalls. The powder was kept under N_2 ambient at 1000°C for 30 minutes. Then, the 0.5 g powder was treated ultrasonically in 80 ml mixture of sulfuric acid and nitric acid (3:1 $\text{H}_2\text{SO}_4/\text{HNO}_3$) for 2 h. After purification process, the powder was rinsed with distilled water, and dried at 80°C in an oven for overnight. For preparing the functional ink, the dry powder was dispersed in 80 ml of deionized water (DI water) under continuous sonication for 45 minutes. The 5.0 vol% of MWCNT solution was ultrasonically dissolved in 85.8 vol% of PEDOT: PSS, 5.4 vol% of dimethyl sulfoxide (DMSO), 3.6 vol% of ethylene glycol (EG) and 0.2 vol% of triton x-100 for 2 h. It should be noted that the MWCNT solution has been limited to 5.0 vol% due to the fiber-nib clogging problem. The roles of MWCNTs and PEDOT: PSS are the sensing materials. For the DMSO, it was used as a solvent with low baseline resistance, while EG was used to improve of the viscosity to prevent of fast drying in the fiber-nib of marker pen. For the triton x-100, it is a nonionic surfactant. It is one of the most widely used to improve the surface tension of the electronic ink (Seekaew *et al.*, 2014). The given concentrations of each chemicals were selected as the optimum concentrations for avoiding the inhomogeneous sensing-film problem. The final solution in form of functional ink was fully stirred for 24 h at room-temperature. The ink was carefully refilled into the empty reservoir of a marker pen. The substrates were prepared by screen-printing of silver paste as interdigitated electrodes onto the polyethylene terephthalate (PET) sheets. Direct ink writing of sensor pattern was conducted onto the substrates using a handwriting method. The size of pattern was approximately $0.3\times 0.8\text{ cm}^2$. The all fabricated sensors were tested under ammonia (NH_3) and other target gases using a gas-testing system.

Gas-Sensing Measurement

The fabricated sensor was inserted into the Swagelok 4-way cross fitting as a test chamber of the gas testing system. The details for the system are shown in Figure 1. The dry-air from the air pump operated with a filter and NH_3 solution were used as the carrier gas and test gas, respectively. The two gases were controlled their flow-rates by two standard flow meters (Cole-Parmer; TW03227-12) and two ball-valve switches (Swagelok; B42S4). The system was connected to a designed voltage divider circuit. A LabVIEW software and a NI USB DAQ 6008 device operated with a laptop computer were used to monitor the gas-sensing signals by measuring the sensor resistance in every second. After testing the gas-sensing measurement, the all

gases were drained out to the outlet by using an exhaust fan.

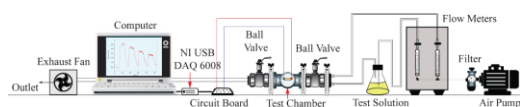


Figure 1. Schematic diagram of gas-testing system in this study.

Results and Discussion

For preparing the gas sensor, the functionalized multiwalled carbon nanotubes (*f*-MWCNTs) were proven possible to attach the COOH groups on MWCNT sidewalls by using acid treatment procedures (3:1 H₂SO₄/HNO₃). Figure 2(a) shows the photograph of *p*-MWCNTs and *f*-MWCNTs immersed in DI water. It is seen that the *f*-MWCNTs are in well-dispersed condition for 30 days. The spectra of Fourier transform infrared spectroscopy (FTIR) for the PEDOT: PSS, *f*-MWCNTs and their composites are shown in Figure 2(b). In the FTIR spectrum of *f*-MWCNTs, the peaks at 1095, 1338, 1412, and 1713 cm⁻¹ are assigned to C-O bonding, C-C bonding, O-H deformation and C = O in vibration modes, respectively. It can be confirmed that the *f*-MWCNTs contain the COOH groups onto the MWCNT sidewalls after purification process. In cases of the FTIR spectra for the PEDOT: PSS and *f*-MWCNT-PEDOT: PSS, the C = C and C-O peaks are slightly shifted from 1521 and 1126 cm⁻¹ to 1518 and 1120 cm⁻¹, respectively. This is due to the delocalization of electrons from C-O to the π -bonds of MWCNTs when the MWCNTs are embedded in PEDOT: PSS matrix.

An ordinary office marker pen becomes ready to write by filling the *f*-MWCNT-PEDOT: PSS ink into the empty reservoir. The pen is composed of fiber nib, cover, foam, compression spring, plunger, polyester reservoir and plug as shown in Figure 3(a). The ink can be flowed from ink reservoir into the fiber nib by capillary action using nib pressing. The prepared ink can move through tiny spaces or channels in a solid foam. The flow-rate of the ink can be controlled by using a plunger and compression spring. The photographs of a marker pen before and after refilling the *f*-MWCNT-PEDOT: PSS ink can be seen in Figure 3(b) and Figure 3(c), respectively.

The 3.0×8.0 mm² ink can be written on a PET substrate with fabricated silver interdigitated electrode. The schematic illustration and photograph of gas sensor prepared by a handwriting method are

shown in Figure 4(a) and Figure 4(b), respectively. The initial resistance of fabricated sensor was approximately 1 k Ω after drying in air. Figure 5(a) shows the morphology of 10.0×16.0 mm² interdigitated silver electrode (Ag-IDE). It is seen that the width and the interdigit spacing of the electrode are found to be ~1.0 mm. Figure 5(b) shows the SEM images of 3.0×8.0 mm² *f*-MWCNT-PEDOT: PSS sensing film on PET substrate with Ag-IDE. The inset in the Figure 5(b) shows the

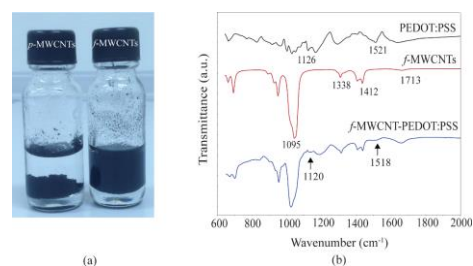


Figure 2. (a) Photograph of *p*-MWCNTs and *f*-MWCNTs immersed in DI water after room-temperature storage in 30 days. (b) FTIR spectra of PEDOT:PSS, *f*-MWCNTs and their composites.

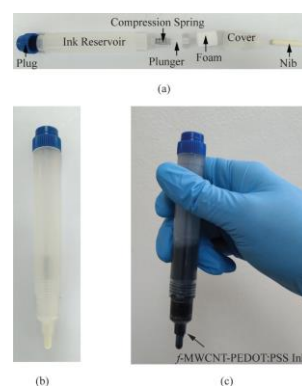


Figure 3. (a) Photograph of a marker pen and its parts. Photographs of a marker pen (b) before and (c) after refilling the *f*-MWCNT-PEDOT:PSS ink.

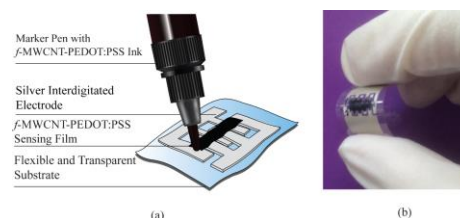


Figure 4. (a) Schematic illustration and (b) photograph of gas sensor prepared by handwriting method using a marker pen with *f*-MWCNT-PEDOT:PSS ink.

dense-packed *f*-MWCNT-PEDOT: PSS sensing films on the substrate. As can be seen that the films tend to form in a high compress and well coverage on the substrate surface. Then, it was inserted into the test chamber of the gas-testing system. The fabricated sensor was investigated for its response to NH_3 and other target gases.

Figure 6(a) shows the resistance changes of *f*-MWCNT-PEDOT:PSS gas sensors prepared by drop-casting and pen-based writing methods exposed to four pulses of 80 ppm NH_3 . It can be observed that the both sensors show the increment

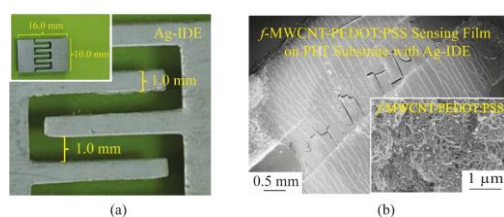


Figure 5. (a) Photograph of Ag-IDE electrode and (b) SEM image of *f*-MWCNT-PEDOT: PSS sensing film on PET substrate with Ag-IDE prepared by handwriting method (Inset shows the high magnification SEM image of *f*-MWCNT-PEDOT: PSS sensing film).

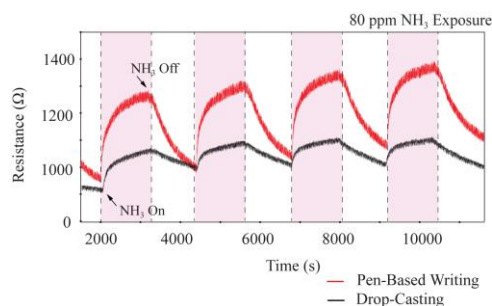


Figure 6. Resistance changes of *f*-MWCNT-PEDOT: PSS gas sensors prepared by drop-casting and pen-based writing methods.

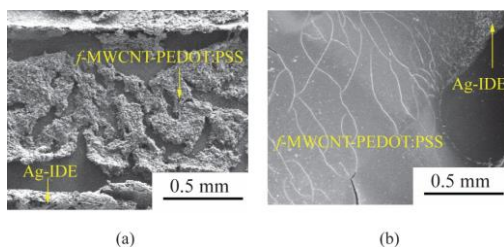


Figure 7. SEM image of *f*-MWCNT-PEDOT: PSS sensing films prepared by (a) drop-casting and (b) pen-based writing methods.

of resistance upon exposure to NH_3 vapors. When the flow of NH_3 was stopped, the resistances of *f*-MWCNT-PEDOT: PSS gas sensors immediately decreased and nearly recovered to their baseline lines. The drifts of baseline resistances for the two sensors after four sensing pulses was also observed. This is due to the incomplete desorption of NH_3 molecules after purging the dry-air on the sensor surface at room-temperature (Isa *et al.*, 2017). The gas sensors were evaluated in their performances using gas response, sensitivity and selectivity properties of the sensor upon exposure to the target gas. For the gas response property, it was defined by an equation as $S (\%) = [(R_{\text{gas}} - R_{\text{air}}) \times 100] / R_{\text{air}}$, where R_{air} and R_{gas} represent the resistances of sensor in dry air and in target gas, respectively. For the *f*-MWCNT-PEDOT: PSS gas sensors prepared by drop-casting and pen-based writing methods, the values of gas response calculated from the first pulse of 80 ppm NH_3 are found to be 5% and 61%, respectively. The time to reach 90% of maximum change for the sensor resistance after the exposure to target gas was defined as the response time. It is observed in Figure 6 that the response time of two sensors are found to be ~ 14 min. For the recovery time, it was also defined by the time of the resistance change for the sensor to get back to the baseline line after the flow of target gas was discontinued. The recovery time of two *f*-MWCNT-PEDOT: PSS gas sensors are found to be almost equal to the response time, i.e., ~ 14 min.

Again, it is found from the resistance changes of two sensors as shown in Figure 6 that the calculated value of gas response for the pen-written gas sensor is higher than the value obtained from the drop-casted gas sensor. The homogeneous gain effect was claimed to explain as an important cause for enhancing the sensing signals of gas sensor (Kim *et al.*, 2011; Benchirouf *et al.*, 2016; Pakdee *et al.*, 2019). Figure 7 shows the SEM images of the *f*-MWCNT-PEDOT: PSS sensing films on the substrate with Ag-IDE prepared by drop-casting and pen-based writing methods. It is seen that the morphologies of two surfaces are obviously different. For the drop-casted condition, the morphology of *f*-MWCNT-PEDOT: PSS is low compress and poor coverage due to the high surface tension of PEDOT:PSS (Pakdee *et al.*, 2019). For the pen-based writing condition, the sensing film reveals the smooth surface and good coverage. Therefore, the good compress and coverage of sensing films onto the electrode obtained from the pen-based writing method may lead to enhance the sensing signals of gas sensor by increment for the conductive pathways in electron transports.

The sensitivity of the sensor was defined as the slope of linear graph in the relation of gas response versus gas concentration. Figure 8(a) shows the resistance changes of *p*-MWCNT-PEDOT:PSS and *f*-MWCNT-PEDOT:PSS gas sensors prepared by pen-based writing method for different concentration of NH_3 . The calculated values of gas response for the *p*-MWCNT-PEDOT:PSS gas sensor exposed to NH_3 with a concentration of 80, 100, 150 and 200 ppm are found to be 10.3, 13.8, 20.5 and 30.3%, respectively. For the *f*-MWCNT-PEDOT:PSS gas sensor, the calculated values of the gas response under NH_3 concentrations of 80, 100, 150 and 200 ppm are also found to be 57.3, 69.5, 95.1 and 128.2%, respectively. Figure 8(b) shows the gas responses of two sensors exposed to NH_3 in the concentration range of 80 to 200 ppm. The sensitivity of *p*-MWCNT-PEDOT:PSS gas sensor calculated from the slope of linear regression line is found to be 0.144 ppm^{-1} with coefficient of determination $R^2 = 0.999$ and the slope of the trendline for *f*-MWCNT-PEDOT:PSS response is also found to be 0.652 ppm^{-1} with coefficient of determination $R^2 = 0.998$. Thus, the *f*-MWCNT-PEDOT:PSS gas sensor has actually higher sensitivity than the *p*-MWCNT-PEDOT:PSS gas sensor after exposure to NH_3 . This is due to the fact that the N_2 heat treatment has an effect on the removal of non-reacted amorphous regions for the *p*-MWCNTs (Yoon *et al.*, 2015). This treatment increases the number of hybridized sp^2 networks which can be a result of MWCNTs in their high crystalline qualities. Furthermore, the oxygen vacancies on the *f*-MWCNTs obtained from the dispersion of *p*-MWCNTs in acid solution lead to the increase in the sensing signals to the NH_3 (Sharma *et al.*, 2012).

The selectivity property of two sensors was also investigated by testing the sensors under different target gases such as 80 ppm NH_3 , 200 ppm ethanol ($\text{C}_2\text{H}_5\text{OH}$), 200 ppm chloroform (CHCl_3), 200 ppm methanol (CH_3OH), 200 ppm benzene (C_6H_6) and 1000 ppm acetone ($\text{C}_3\text{H}_6\text{O}$). It is observed in Figure 9 that the *p*-MWCNT-PEDOT:PSS and *f*-MWCNT-PEDOT:PSS gas sensors present the highest response to NH_3 at the low concentration of 80 ppm, while their responses to other gases at high concentration of 200 and 1000 ppm are low ($\leq 10\%$).

The behaviors of *f*-MWCNT-PEDOT:PSS gas sensor exposed to 80 ppm NH_3 under disorder curvatures were further evaluated at room-temperature. Figure 10 shows the resistance changes of *f*-MWCNT-PEDOT:PSS gas sensor measured during different bending states such as flat, bend and twist conditions. It is seen that the gas responses of sensor for all bending states did not have an obvious

change. Therefore, the written *f*-MWCNT-PEDOT:PSS gas sensor can be worked without noticeable reduction in gas response although it was bent to the heavy disorder.

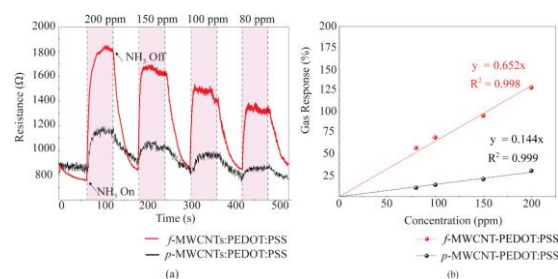


Figure 8. (a) Resistance changes of *f*-MWCNT-PEDOT:PSS and *p*-MWCNT-PEDOT:PSS gas sensors prepared by pen-based writing method. (b) Gas response of *f*-MWCNT-PEDOT:PSS gas sensors as a function of NH_3 concentration.

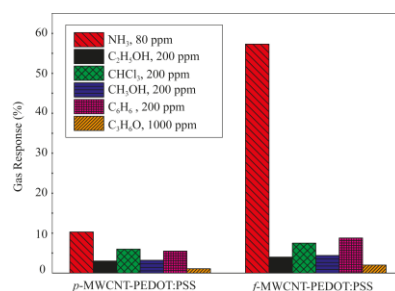


Figure 9. Gas responses of *p*-MWCNT-PEDOT:PSS and *f*-MWCNT-PEDOT:PSS gas sensors exposed to different target gases.

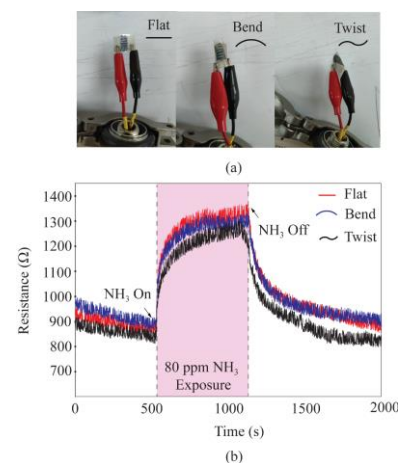
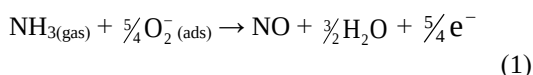


Figure 10. (a) Photograph of *f*-MWCNT-PEDOT:PSS gas sensor under different curvatures. (b) Resistance changes of gas sensor measured during different bending states such as flat, bend and twist conditions under NH_3 vapors.

For the sensing mechanism, it has been discussed based on a result of combination for the sensing-properties of *f*-MWCNTs and PEDOT:PSS. The reaction between NH₃ molecules and oxygen species (O₂⁻) on the surface of *f*-MWCNTs by chemisorption has been proposed. The reducing NH₃ gas will bond with O₂⁻ groups as hydrogen bonds according to the reaction (1):



This reaction results in the discharge of 5/4 electrons into the surface of *f*-MWCNTs as shown in Figure 11, leading to the reduction of hole concentration in the p-type semiconductor material. This leads to an increase of sensor resistance when the NH₃ molecules were adsorbed onto the sensor surface. In addition, the direct charge transfers between NH₃ molecules and *f*-MWCNT-PEDOT:PSS structures has been also proposed. When NH₃ molecules are adsorbed on the surface of *f*-MWCNT-PEDOT:PSS by physisorption, the free electrons in NH₃ molecules will donate to the conduction band of oxide groups in the *f*-MWCNT-PEDOT:PSS. This cause leads to a decrease in the hole concentration for the p-type semiconductor nature of *f*-MWCNT-PEDOT:PSS. Therefore, the resistance of sensor will increase when it is exposed to NH₃ vapors.

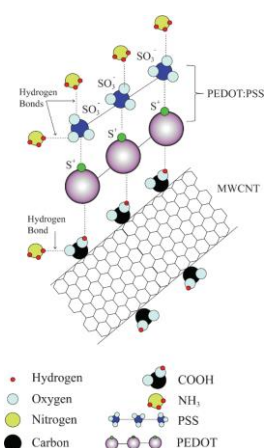


Figure 11. Schematic illustration of proposed NH₃-sensing mechanism for *f*-MWCNT-PEDOT:PSS gas sensor.

Another possible NH₃-sensing mechanism of *f*-MWCNT-PEDOT:PSS gas sensor is the film swelling as also shown in Figure 11. In air flow, the electron hopping between the PEDOT chains can be presented since the distances of their chains are very

short. When the PEDOT:PSS reacts with electron-donating NH₃ molecules by hydrogen bonds, the interchain distance between PEDOT and PSS chains will disrupt due to the film swelling. The electron hopping between the PEDOT chains may be more difficult, which results in an increase of sensor resistance upon exposure to the NH₃ vapors. Moreover, the enhancement of recovery property of *f*-MWCNT-PEDOT:PSS gas sensor can be mentioned to the weak interaction between the polymer and NH₃ molecules. However, the bit upward shift of baseline resistance for the sensor has been explained that the chemisorbed molecules cannot be desorbed completely on the sensing surface at room-temperature. The stability of gas sensors was further investigated. The gas measurements were repeated after room-temperature storage in 30 days. It was found that the sensors present the good stability with only 5% of letdown from its initial response.

Conclusions

The NH₃ gas sensor has been successfully fabricated by direct- handwriting method with *f*-MWCNT-PEDOT:PSS ink. For improving the solubility property of MWCNTs, the purified MWCNTs were functionalized by acid treatment to introduce the carboxylic groups (COOH) onto the MWCNT surfaces. After writing the sensing ink, the sensor was investigated for its response to ammonia (NH₃) and other gases at room-temperature. This investigation indicates that the gas sensor obtained from *f*-MWCNT-PEDOT:PSS has shown a high performance to NH₃ since it was bent to the different disorder- curvatures. The sensing mechanism of fabricated NH₃ gas sensor has been discussed based on a result of combination for the sensing-properties of *f*-MWCNTs and PEDOT:PSS.

Acknowledgments

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