A chemosensitive based ammonia gas sensor with PANI/PEO- ZnO nanofiber composites sensing layer

Gözde Konuk Ege
Department of Mechatronics, Gedik Vocational School, Istanbul Gedik University, Istanbul, Turkey, and
Özge Akay and Hüseyin Yüce
Department of Mechatronics Engineering, Faculty of Technology, Marmara University, Istanbul, Turkey

Abstract

Purpose – The purpose of this study is to investigate the ammonia sensing performance of polyaniline/polyethylene oxide (PANI/PEO) and polyaniline/polyethylene oxide/zinc oxide (PANI/PEO-ZnO) composite nanofibers at room temperature.

Design/methodology/approach – Gas sensor structures were fabricated using micro-fabrication techniques. First, onto the SiO_2 wafer, gold electrodes were fabricated via thermal evaporation. PANI/PEO nanofibers were produced by the electrospinning method and the ZnO layer was deposited by RF magnetron sputtering on the electrospun nanofibers as a sensing layer. Fourier transform infrared spectroscopy (FTIR) and scanning electron microscopy (SEM) were performed for characterization analysis of nanofibers. After all, gas sensing analysis of PANI/PEO and PANI/PEO/ZnO nanofibers was performed using an experimental setup at room temperature conditions.

Findings – FTIR analysis confirms the presence of functional groups of PANI, PEO and ZnO in nanofiber structure. SEM images demonstrate beads-free, thinner and smooth nanofibers with ZnO contribution to electrospun PANI/PEO nanofibers. Moreover, according to the gas sensing results, the PANI/PEO nanofibers exhibit 115 and 457 s response time and recovery time, respectively. However, the PANI/PEO/ZnO nanofibers exhibit 245 and 153 s response time and recovery time, respectively.

Originality/value – In this study, ZnO was deposited via RF magnetron sputtering techniques on PANI/PEO nanofibers as a different approach instead of *in situ* polymerization, to investigate and enhance the sensor response and recovery time of the PANI/PEO/ZnO and PANI/PEO composite nanofibers to ammonia. These results indicated that ZnO can enhance the sensing properties of conductive polymer based resistive sensors.

Keywords Composites, PANI, Nanofibers, Gas sensor, Polymer

Paper type Research paper

1. Introduction

In recent years, the rapid increase in air pollution has increased the need for gas sensor devices (Kim *et al.*, 2022). Especially MOx and conducting polymer based gas sensor devices are heavily studied. Conducting polymers such as polyaniline (PANI), poly(3,4-ethylenedioxythiophene) (PEDOT) and polypyrrole (PPy) are used as a sensing mechanism of gas sensor devices because of their flexible and simple composition along with basic layout, also metal oxide (MOx) (ZnO, SnO, TiO_2, etc.) semiconductor compounds are very promising in terms of sensing performance (Ramakrishnaiah *et al.*, 2022). Although metal oxide gas sensors show high gas detection capability thanks to their wide bandgap, good conductivity properties and variable oxygen stoichiometry, these sensors have a high operating temperature (300°C–400°C) (Kunzo *et al.*, 2012). On the other hand, conductive polymers sometimes exhibit unstable behavior and relatively low sensitivity owing to their high sensitivity to ambient moisture. With respect to these reasons, nanocomposite structures of conducting polymers and MOx materials attract attention of researchers because of their improved specific properties such as room temperature operation and highly effective gas detection and they are widely studied in gas sensor applications (Ali *et al.*, 2020; Li *et al.*, 2018a, 2018b; Lobotka *et al.*, 2011; Bhanvase *et al.*, 2015; Trajcheva *et al.*, 2021; Tai *et al.*, 2008).

PANI is a p-type conducting polymer which is an easy to preparation and protonation; furthermore, PANI can be easily converted to conductive (emeraldine salt) and insulating (emeraldine base) form by electrochemical redox, reversible acid/base and doping methods (Vhanakhande *et al.*, 2008; Bavatharani *et al.*, 2021). Moreover, simple and flexible production processes are the advantages of PANI (Alhartomy, 2015). Zinc oxide (ZnO) is an n-type MOx semiconductor and it provides perfect selectivity to selected gases (Bhati *et al.*, 2020; Yang *et al.*, 2021). The heterostructure of PANI/ZnO

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nanocomposite exhibits p-n heterojunction that plays an important role in the sensing mechanism due to the presence of the depletion layer (Kunzo *et al.*, 2013). The sensing layer acts as an exclusive receptor through p-n heterojunctions and absorbs gas analytes, as a consequence of this absorption a significant increase is observed in resistance (Tai *et al.*, 2008; Yang *et al.*, 2021; Mahajan and Jagtap, 2020). Betty *et al.* (2015) prepared SnO_2-PANI heterostructure film to investigate toxic gas response at room temperature and they attained specific responses for NO_2 and SO_2. Li *et al.* (2018a) prepared ZnO/PANI nanocomposite to investigate NH_3 sensing properties. The nanocomposite structure exhibited perfect selectivity thanks to p-n heterojunction. Nimkar *et al.* (2015) developed PANI/TiO_2 nanocomposite and they observed high sensitivity for CO_2 gas.

One-dimensional nanostructures allow flexible bandgap control and also offer high sensitivity in sensor applications with their high surface area/volume ratio (Huang *et al.*, 2003). Electrospinning is the most used technique to produce nanofibers. This method ensures porosity adjustments thanks to nanofiber diameter control; moreover, different fiber morphologies are observed such as core–shell and randomly-blended (Izwan *et al.*, 2015). Li *et al.* (2021) developed moisture sensors based on polymer composite nanofiber and thin film structures and compared the sensing properties of both. They achieved the best recovery and response time with the humidity sensor based on composite nanofiber. Li *et al.* (2011) produced PANI/TiO_2 composite nanofibers to investigate sensing properties for NH_3 gas. They obtained better performance with PANI/TiO_2 composite nanofiber sensor than pure PANI sensor.

In this study, a gas sensor sensitive to NH_3 gas was fabricated. PANI/polyethylene oxide (PEO)/MOx composite nanofibers ensure stable and high sensitivity to NH_3 gas owing to high surface:volume ratio. Gold interdigital transducers (IDTs) were deposited on the substrate. PANI/PEO nanofibers were produced by electrospinning method and ZnO semiconductor was produced onto nanofibers by using RF sputter technique. PANI/nanofibers and PANI/PEO/ZnO composite nanofibers were used as a sensing layer. A schematic representation of gas sensor production is shown in Figure 1. We investigated sensing properties of both PANI nanofibers and PANI/ZnO composite nanofibers. The real-time monitoring and recording of NH_3 gas at room temperature were achieved by the resistance change method.

2. Experimental section

2.1 Materials

PANI emeraldine base (Mw = 50,000), camphor sulfonic acid (CSA), PEO (Mw = 900,000), sodium dodecyl sulfate (SDS) and gold (Au, 99.99%) were purchased from Sigma-Aldrich. Dimethylformamide (DMF) and chloroform that were used for preparation of PANI/PEO solution were purchased from Merck. Other commercial chemicals, which were guaranteed-grade reagents, were used without additional purification.

2.2 Fabrication of transducer layer

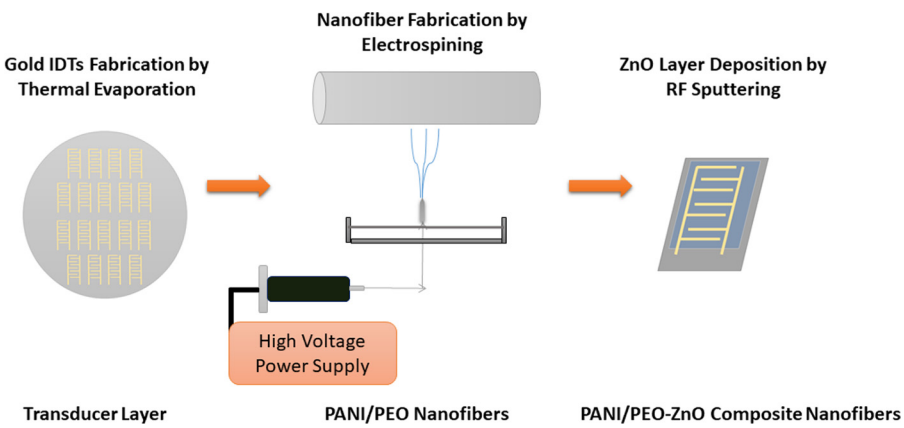
The gas sensor was fabricated on the silicon substrate. IDTs that were used as a transducer layer were defined by UV photolithography on the substrate and they were deposited (Ti/Au-20 nm/100 nm) by NANOVAK NVTS-400 magnetron sputtering system with DC sputter (Ti) and thermal evaporation (Au) technique. Width of the IDT fingers and the gap between the fingers were designed as 1.0 mm and 0.5 mm, respectively.

2.3 Preparation and fabrication of PANI/PEO nanofibers and PANI/PEO-ZnO composite nanofibers

2% w/v PANI (emeraldine base) doped with CSA (PANI:CSA-1:1.5) was kept in DMF:chloroform (1:1) suspension for 24 h to transform PANI emeraldine base to emeraldine salt (conducting) form. Accordingly, 2% w/v PEO was added in the PANI solution and mixed for 24 h. At last, 0.5 w/v SDS was mixed with the PANI/PEO suspension and stirred during the 24 h as we mentioned in our previously article (Konuk Ege *et al.*, 2021).

PANI/PEO nanofibers were produced on the transducer layer with electrospinning method as a sensing layer. Inovenso NE300 Nano Spinner device was used during the electrospinning process. During the electrospining process, 25 kV and 18-gauge nozzle and 10 mL syringe were used. The feed rate of the syringe pump and collector rotation speed were adjusted to 1.3 mL/h and 500 rpm, respectively. The distance between the collector and nozzle was 20 cm. Subsequently,

Figure 1 Schematic representation of gas sensor production



ZnO layer was deposited on the PANI/PEO nanofibers by the NANOVAK NVTS-400 magnetron sputtering system with RF sputtering with a thickness of 0.5 nm.

2.4 Characterization

Scanning electron microscopy (SEM) images of products were performed on a Zeiss-Evo j MA10. Fourier transform infrared (FTIR) spectra of the products to exhibit functional groups were recorded on a PerkinElmer Spectrum 100 series.

2.5 Measurements

The resistance change method was used in the gas detection processes of the sensors that were produced PANI/PEO and PANI/PEO-ZnO sensing layers. The gas sensing measurements were accomplished in the 1 L volume gas chamber at room temperature and humidity (25°C, 40%RH). 3.367 μL (250 ppm) ammonia was injected into the gas chamber with a micropipette. The resistance changes of the sensors were measured as a function of time with a Fluke 189 True RMS multimeter. The sensitivity response (S) of gas sensors was calculated via equation (1) (Kulkarni *et al.*, 2019):

$$S(\%) = \left(\frac{R_{gas} - R_{air}}{R_{air}}\right) x100 \quad (1)$$

2.6 Sensing mechanism

Regarding the sensing mechanism as represented in Figure 2, the change of resistance of sensing layer was detected with/without ammonium gas and analyzed with respect to time. The ammonia gas has a reducing character which means when reacts with the PANI, it accepts the proton and form NH_4^+. Along with that, the resistance of PANI increases due to the loss of protons, which leads to a decrease in conductivity. This reaction is also possible vice versa. In the air, the NH_4^+ gives a proton to PANI and became ammonia molecule. Then, the density of electrons in PANI decreases and its resistance decrease to initial value (Kulkarni *et al.*, 2019).

3. Results and discussion

The functional groups of the PANI/PEO nanofibers and PANI/PEO/ZnO composite nanofibers were identified by FTIR spectroscopies. Figure 3 compares the functional groups of PANI nanofibers with PANI/ZnO composite nanofibers. The appearance of the broad peak at around 3500 cm^{-1} region indicates the presence of O—H groups. The characteristic peak of the PANI-emeraldine salt was observed at 1254 cm^{-1} region.

The presence of carrier polymer-PEO appeared in PANI-CSA/PEO blend at around 2850 cm^{-1} and this peak is related to C—H aliphatic bending. 1582 and 1400 cm^{-1} peak represent C=N and C—N, respectively. 893 cm^{-1} refers to

Figure 2 Schematic illustration of sensing mechanism PANI/PEO gas sensor

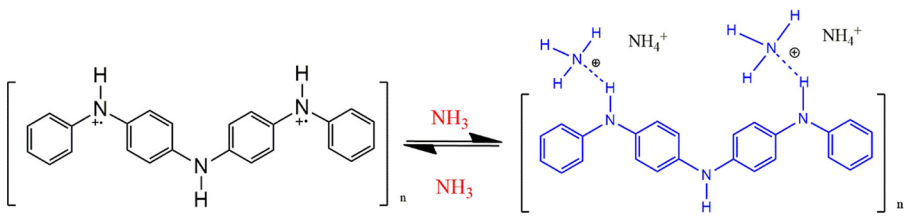


Figure 3 FTIR spectroscopies of PANI/PEO and PANI/PEO/ZnO nanofibers

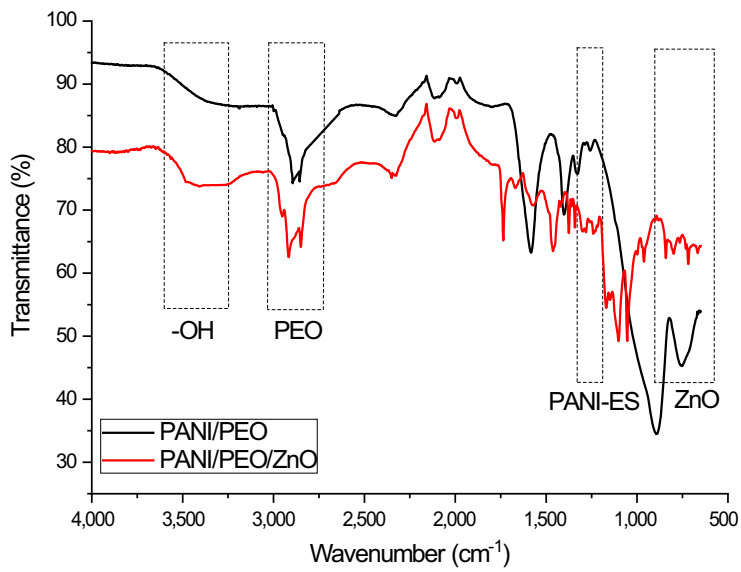


Figure 4 SEM images of (a) PANI/PEO and (b) PANI/PEO/ZnO nanofibers

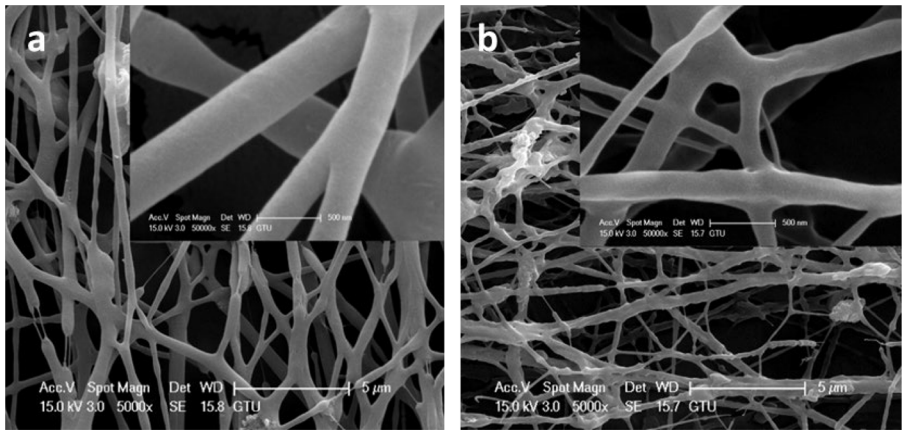


Table 1 Sensing response (%), response time and recovery time of PANI/PEO and PANI/PEO/ZnO nanofiber composites toward NH_3

Sensing analyzes	PANI/PEO	PANI/PEO-ZnO
Response time (s)	115	245
Recovery time (s)	457	153
Sensitivity response (%)	60	44

C—H aromatic and 755 cm^{-1} corresponds to SO_3 group (Alwan *et al.*, 2018; Konuk Ege *et al.*, 2021). The tetrahedral coordination of Zn is observed at 840 cm^{-1} region as an absorption peak. The peaks observed at 718 and 666 cm^{-1} indicate stretching vibrations of sputtered ZnO nanoparticles (Jayarambabu *et al.*, 2015; Ur Rehman *et al.*, 2019).

Figure 4 exhibits the SEM images of PANI/PEO [Figure 4(a)] and PANI/PEO/ZnO [Figure 4(b)] nanofiber composites (Table 1). For both SEM images, beads-free fibers are attained in two cases. The SEM image of PANI/PEO with ZnO composite nanofibers [Figure 4(b)] contains more branch fibers that resulted in thinner nanofibers. These thinner nanofibers increase the porosity of the sensing layer, facilitating the diffusion of gas to the surface. In addition to the increased diffusion effect, the reduction of nanofiber diameters can also increase the conductivity of the sensing layer (Zhang *et al.*, 2018). Hence, more powerful sensing signals against the gas can be obtained. Furthermore, the smoothness of nanofibers was decreased with sputtering the ZnO on PANI/PEO nanofiber composites as represented in Figure 4(b) which may affect the response and recovery time of gas sensors (Pandey, 2016).

Figure 5 Repeatability of PANI/PEO nanofiber gas sensor toward 250 ppm NH_3 at room temperature

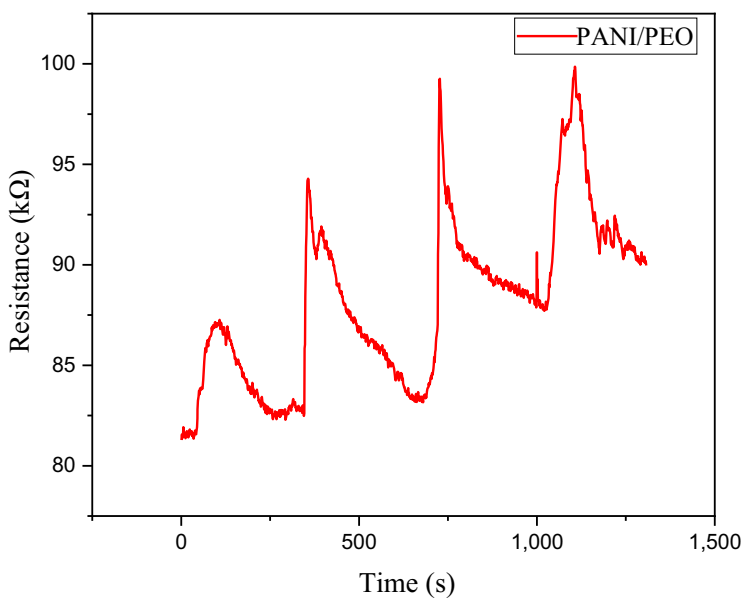
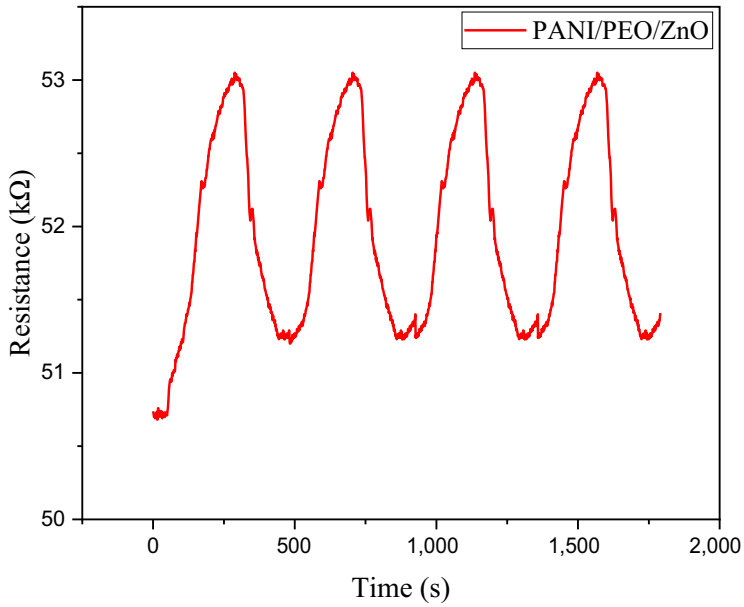


Figure 6 Repeatability of PANI/PEO/ZnO nanofiber gas sensor toward 250 ppm NH_3 at room temperature



Regarding sensing mechanism, the response of gas sensors contains two different sensing layers were analyzed by measuring the resistance of the sensing layer as a function of time when exposed to the 250 ppm ammonia gas.

The sensitivity response (%), response time and recovery time of PANI/PEO nanofibers and PANI/PEO/ZnO composite were investigated in detail. The quantitative results are shown in Table 1. According to the results obtained from the time-dependent resistance values of the gas sensor containing the PANI/PEO sensing layer, the sensor shows a different response value for each cycle as indicated in Figure 5. These unstable responses mean that the sensor possesses low repeatability which is one of the most undesirable results for sensor applications. On the contrary, via sputtering ZnO on the PANI/PEO composite nanofibers, the more stable results were attained as shown in Figure 6.

According to response and recovery time of gas sensors in two cases, although response time of PANI/PEO was low about 115 s when compared to the response time of PANI/PEO/ZnO (245 s), it has more recovery time (457 s) than PANI/PEO/ZnO sensing layer (153 s). Hence, the recovery time fell by a third by adding ZnO on conductive polymer PANI/PEO.

4. Conclusion

In this study, PANI/PEO nanofibers and PANI/PEO/ZnO composite nanofibers based on NH_3 gas sensor were developed. ZnO was deposited via RF magnetron sputtering techniques on the PANI/PEO nanofibers to enhance the sensing properties as per PANI/PEO nanofibers to ammonia. FTIR analysis confirms the presence of functional groups of PANI, PEO and ZnO in nanofiber structure. Gas sensing measurements were performed at room temperature and investigated response times, recovery times and sensitivity responses. Although PANI/PEO nanofibers demonstrate better sensitivity response (60%), their response was unstable and they have lower repeatability compared to PANI/PEO/ZnO composite nanofibers. PANI/PEO/ZnO composite nanofibers exhibit better NH_3 sensing behavior as regards fast recovery time (153 s), stable response time and high repeatability than PANI/PEO nanofibers. All results stated that ZnO can enhance the sensing properties of conductive polymer based resistive sensors and stable, high-reproducibility, room-temperature sensors can be developed with the ZnO composite structures of PANI nanofibers.

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Corresponding author

Özge Akay can be contacted at: ozge.akay@marmara.edu.tr