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Highly sensitive and selective sensors to volatile organic compounds using MWCNTs/SnO_2

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ABSTRACT

Multiwall carbon nanotubes (MWCNTs)/SnO_2 gas sensors were fabricated using ultrasonic-assisted deposition–precipitation method and used for detection of ethanol, acetaldehyde, acetone, toluene and trichloroethylene (TCE) VOCs at various temperatures. The MWCNTs/SnO_2 samples were characterized by BET specific surface area measurement, FE-SEM and XRD. The sensors responses to 300 ppm of VOCs were measured in a flow system at various temperatures in the range of 100–350 °C. MWCNTs are coated with SnO_2 nanoparticles and embedded into the SnO_2 matrix. The sensors' responses dramatically increase by adding 0.05 and 0.10 wt% MWCNTs to SnO_2. Response enhancements up to 2.4, 4.4, 5.3, and 3.6 times are observed for ethanol, acetaldehyde, acetone and toluene, respectively. The 0.05 wt% MWCNTs/SnO_2 sensor shows significant responses to sub-ppm levels of acetaldehyde and acetone, as the diabetes diagnostic gas in breath. Moreover, significant enhancement of the sensors selectivity to ethanol, acetaldehyde and acetone with respect to TCE and toluene are observed by addition of the MWCNTs.

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1. Introduction

Various pure volatile organic compounds (VOCs), due to their contribution to global warming, stratospheric ozone depletion and environmental health concerns, are the most common hazardous pollutants in outdoors [1,2]. The main sources of VOCs in outdoors are combustion processes, traffic vehicles and fuel evaporation. Nowadays, people generally spend more than 80% of their time in indoor environments such as homes, offices, cars and shopping centers. Indoor concentrations of VOCs are 2–5 times higher than their corresponding outdoor concentrations [3,4]. Among the VOCs, acetone, ethanol, acetaldehyde, toluene, and TCE are commonly found in indoors. Indoor contaminants are emitted by human breath; building materials such as paints and coatings and furnitures [3]. Since, the probability of over exposure to these contaminants is very high, the progress of monitoring systems for their early detection is necessary. VOCs detection may also be applied in breathalyzers and medical processes such as disease diagnoses and controlling. As an example, the maximum concentration of acetone in a healthy individual should be below 0.9 ppm and concentration above this value warns that the individual has diabetes [5].

Several methods have been used for detection and measuring indoor VOCs, e.g. gas chromatography/mass spectrometry [6], calorimetric method [7], optical method [8] and sensors based on catalytic combustion [9]. Semiconductor metal oxide sensors are the most promising due to their simple structure and low fabrication cost. Among the various metal oxides used for developing gas sensor systems, SnO_2 is a well-known substance for detection of different pollutants and combustible gases. SnO_2 is a wide band gap semiconductor with high chemical stability and excellent electrical properties [10]. The major drawbacks of the SnO_2-based gas sensors are that they are not selective in gas mixture and show excellent responses only at high temperatures [11,12]. In order to improve sensitivity and selectivity of SnO_2-based sensors and also reduce their operating temperature, dopants such as Pt [11], Au [13] and carbon nanotubes (CNTs) [14] have been introduced to the SnO_2 substrate.

Addition of functionalized CNTs to a SnO_2 matrix increases its specific surface area and enhances its conductivity. Several studies have reported the excellent sensing properties of CNTs/SnO_2 sensor for detection of CO [14], NO_2 [15] and NH_3 [16] at low temperatures. However, there are few reports of CNTs/SnO_2 gas sensors for VOCs monitoring.

In this study, MWCNTs/SnO_2 sensor was developed by adding functionalized MWCNTs into a SnO_2 substrate by sonochemical deposition–precipitation. Gas sensing properties of the fabricated sensors for detection of ethanol, acetaldehyde, acetone, toluene and TCE VOCs were measured.

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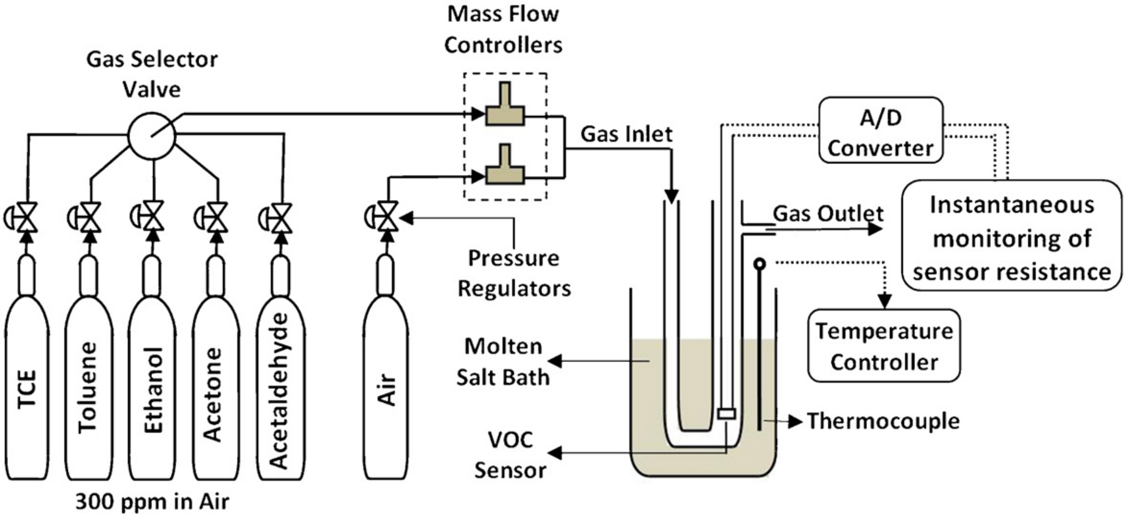

Fig. 1. Schematic of the experimental set-up used for measuring the performance of the VOC sensors.

2. Experimental

2.1. Sensing material preparation

MWCNTs were prepared by chemical vapor deposition floating catalyst method [17]. After removing amorphous carbon and metal catalyst by controlled oxidation in air and acid washing, the purified MWCNTs were acid functionalized in a concentrated nitric acid under reflux for 4 h [17]. The 0.05 and 0.10 wt% MWCNTs/SnO_2 samples were prepared by a sonication-assisted precipitation method [10]. Using an ultrasound homogenizer (model-250 UL, Helchier, 250 W), proper amounts of MWCNTs was dispersed in 0.15 M $SnCl_4$ (Merck) aqueous solution, and then, $SnCl_4$ was deposited on MWCNTs by ammonia, while sonicating at 70 °C and pH 9.0 for 2 h. The precipitate was centrifuged, washed with deionized water several times and dried. The obtained powder was calcined at 400 °C for 3 h.

A paste of each sample powder was screen-printed on the surface of an alumina substrate which was already covered by gold electrodes with 1 mm spacing. The fabricated samples were dried at 100 °C for 1 h and annealed at 400 °C for 1.5 h.

2.2. Sensing material characterization

Microstructure of the samples were characterized using a field emission scanning electron microscopy (FE-SEM) operating at 5.0 kV (JSM 6700F; JEOL, Tokyo, Japan). Phase structure of the prepared samples was examined by X-ray powder diffraction (XRD), using Cu Kα radiation. The specific surface areas of the samples were measured by Brunauer–Emmet–Teller (BET) method, using a Quanta chrome CHEMBET-3000 apparatus.

2.3. Gas-sensing measurement

Continuous monitoring of the sensors' electrical resistance was performed in an experimental set-up (Fig. 1). Each VOC (300 ppm in air) stream was selected by the gas selector valve and further diluted with an air stream, flows of which were controlled by two mass-flow-controllers. The diluted VOC enters a Pyrex glass chamber containing the sensor. The chamber is immersed in a molten salt bath with uniform temperature controlled by a PID temperature controller. The sensor response toward various VOCs is defined as the ratio of the sensor resistance in air to that in presence of the target VOCs ($R_{\mathrm{air}}/R_{\mathrm{gas}}$).

3. Results and discussion

3.1. Characterization

Fig. 2 shows the SEM micrographs of SnO_2, acid functionalized MWCNTs and 0.05 wt% MWCNTs/SnO_2. The nanotubes (Fig. 2b) are entangled with an average diameter of about 80 nm. The MWCNTs are embedded in and covered by the SnO_2 nanoparticles. Compared with MWCNTs, covered nanotubes have larger diameter and exhibit cracky morphology.

Acid functionalized MWCNTs contain carboxyl groups on their surface and open ends [17]. These functional groups may act as links for electrostatic attachment of tin ions. These links form a crystal nucleus and then they are hydrolyzed to SnO_2 nanocrystals. Afterwards the ions are adsorbed on the as-created SnO_2 layer and crystallized to SnO_2 until surface energy would be minimized. The formation of SnO_2 may be presented as follows [18].

$$Sn^{4+} + 4H_2O \rightarrow Sn(OH)_4 + 4H^+ \qquad (1)$$

$$Sn(OH)_4 \rightarrow SnO_2 + 2H_2O \qquad (2)$$

Acid functionalized MWCNTs have high specific surface area of about 248 m^2/g. Their addition in low concentration, i.e. 0.05 wt%, caused the surface area of the SnO_2 to change from 197 to 201 m^2/g. Compared with other reports [11,19], our synthesized SnO_2 has a higher surface area.

Fig. 3 presents XRD patterns of MWCNTs and 0.05 wt% MWCNTs/SnO_2. The XRD pattern of MWCNT (Fig. 3a) shows a sharp peak at 26.23°, which is assigned to graphitic carbon (JCPDS 41-1487). The peaks at 26.56°, 32.28°, 51.49° and 64.96° (Fig. 3b) can be attributed to (1 1 0), (1 0 1), (2 1 1) and (1 1 2) crystal planes of tetragonal rutile structure of the SnO_2, respectively [20]. No diffraction peaks from other impurities such as Sn and SnO were observed. Since the amount of MWCNTs is very small, the characteristic peak of nanotubes could not be observed in the hybrid pattern.

3.2. Sensing properties

Electrical resistance of the blank SnO_2 and MWCNTs/SnO_2 samples in air as a function of temperature is presented in Fig. 4. Addition of nanotubes decreased SnO_2 electrical resistance and also its dependency to temperature. This may represent a uniform distribution of nanotubes in SnO_2 substrate.

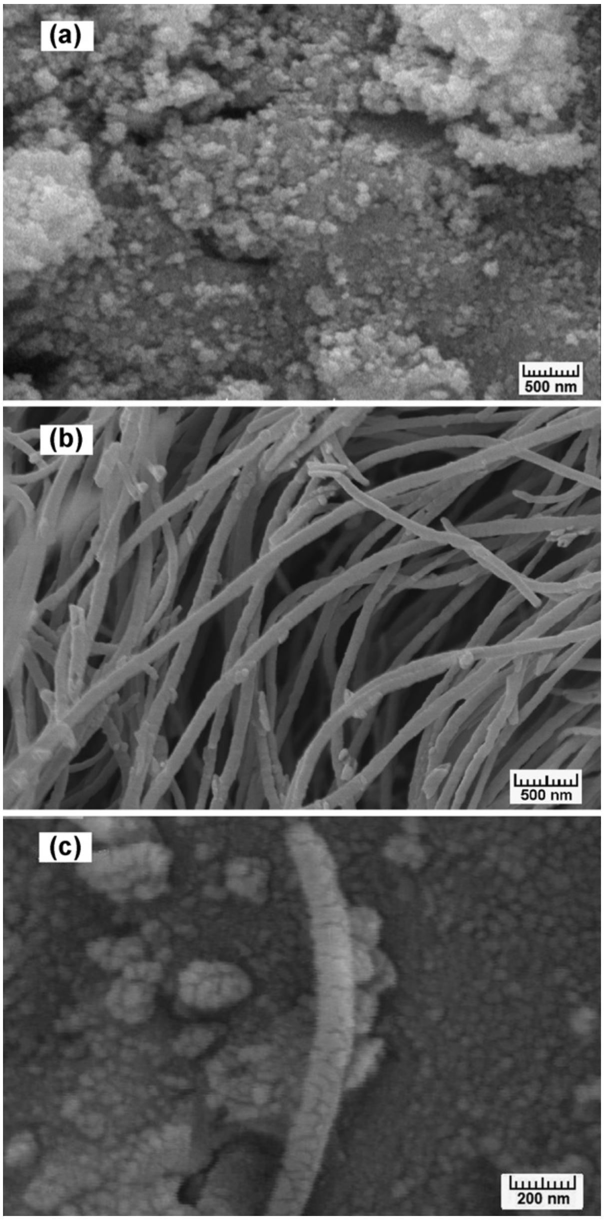

Fig. 2. SEM micrographs of (a) SnO_2, (b) functionalized MWCNTs and (c) 0.05 wt% MWCNTs/SnO_2.

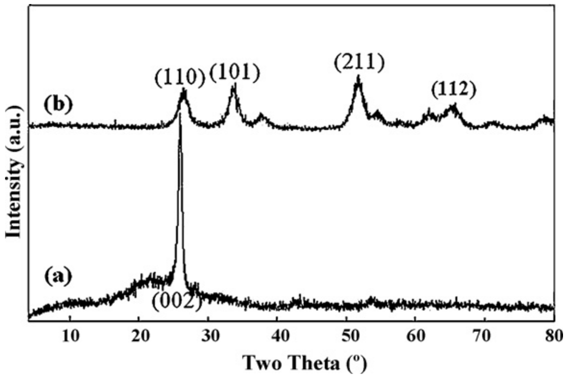

Fig. 3. XRD patterns of (a) MWCNTs and (b) 0.05 wt% MWCNTs/SnO_2.

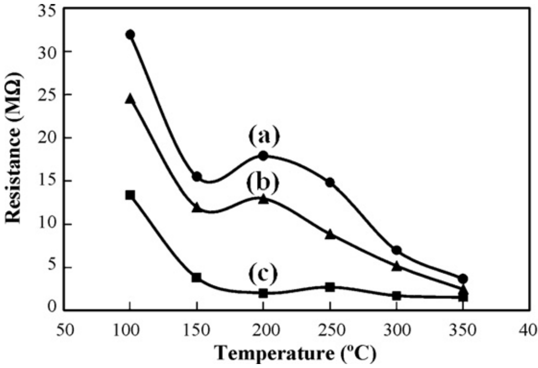

Fig. 4. Electrical resistance of (a) SnO_2, (b) 0.05 wt% and (c) 0.10 wt% MWCNTs/SnO_2 samples in air at different temperatures.

MWCNTs typically show metallic behavior at room temperature and are expected to be semiconductor (with low band gap of about 0.05 eV) at higher temperatures [21]. Moreover, the work function of MWCNTs is around that of SnO_2 and the Schottky barrier between them is low. Therefore, MWCNTs/SnO_2 shows a lower resistance which in turn causes the electrons to be conducted more easily through SnO_2 matrix [22].

On the other word, nanotubes create new passages in SnO_2 matrix [27] and improve gas diffusion through its structure. Diffusion enhancement may remove high temperature requirements for larger sensor responses. Table 1 illustrates the effect of nanotubes on operating temperatures for maximum responses to various VOCs. Addition of MWCNTs reduces the optimum operating temperatures for the all sensors.

Response of the blank and MWCNTs/SnO_2 samples upon exposure to 300 ppm ethanol, acetaldehyde, acetone, toluene and TCE VOCs in air is shown as a function of temperature in Fig. 5.

The response variation with temperature for the all sensors exhibit volcano-shaped curves. This correlation has been attributed to the competition of surface reaction with diffusion and consequently utility factor of sub-layers in the sensing film [23]. Addition of 0.05 wt% nanotubes, excluding TCE, caused an enhancement in the response of all gas sensors at their optimum temperatures, at which maximum response to the VOC occur. Responses increased at these temperatures for ethanol, acetaldehyde, acetone and toluene, by a factor of 2.4, 4.4, 5.3, and 3.6, respectively. The sensor comprising 0.10 wt% MWCNTs shows sensitivity increase for toluene detection by a factor of 3.4 at 200 °C.

In the case of TCE, improvement in the response occurred below 150 °C. The response of 0.05 wt% MWCNTs/SnO_2 sensor toward TCE increases by a factor of 97.6 at 100 °C. Response enhancement at this temperature is also occurred for acetaldehyde, using 0.05 wt% MWCNTs/SnO_2, and for toluene, using 0.10 wt% MWCNTs/SnO_2, by a factor of 10.9 and 4.5, respectively. High responses at low temperatures of these sensors increase long-term stability and decreases power consumption of the sensors.

One possible mechanism of sensing behavior can be explained as follows. Acid functionalized CNTs can be considered as p-type semiconductor and its composition with n-type SnO_2 develops a CNTs/SnO_2 hetero-structure [16]. It has been reported that

Table 1
The temperature for maximum-response (°C) of SnO_2-based sensors to 300 ppm of different VOCs.

Samples	Ethanol	Acetaldehyde	Acetone	Toluene	TCE
Blank SnO_2	250	250	300	200	200
0.05 wt% MWCNT/SnO_2	250	200	250	200	200
0.10 wt% MWCNT/SnO_2	200	200	200	150	150

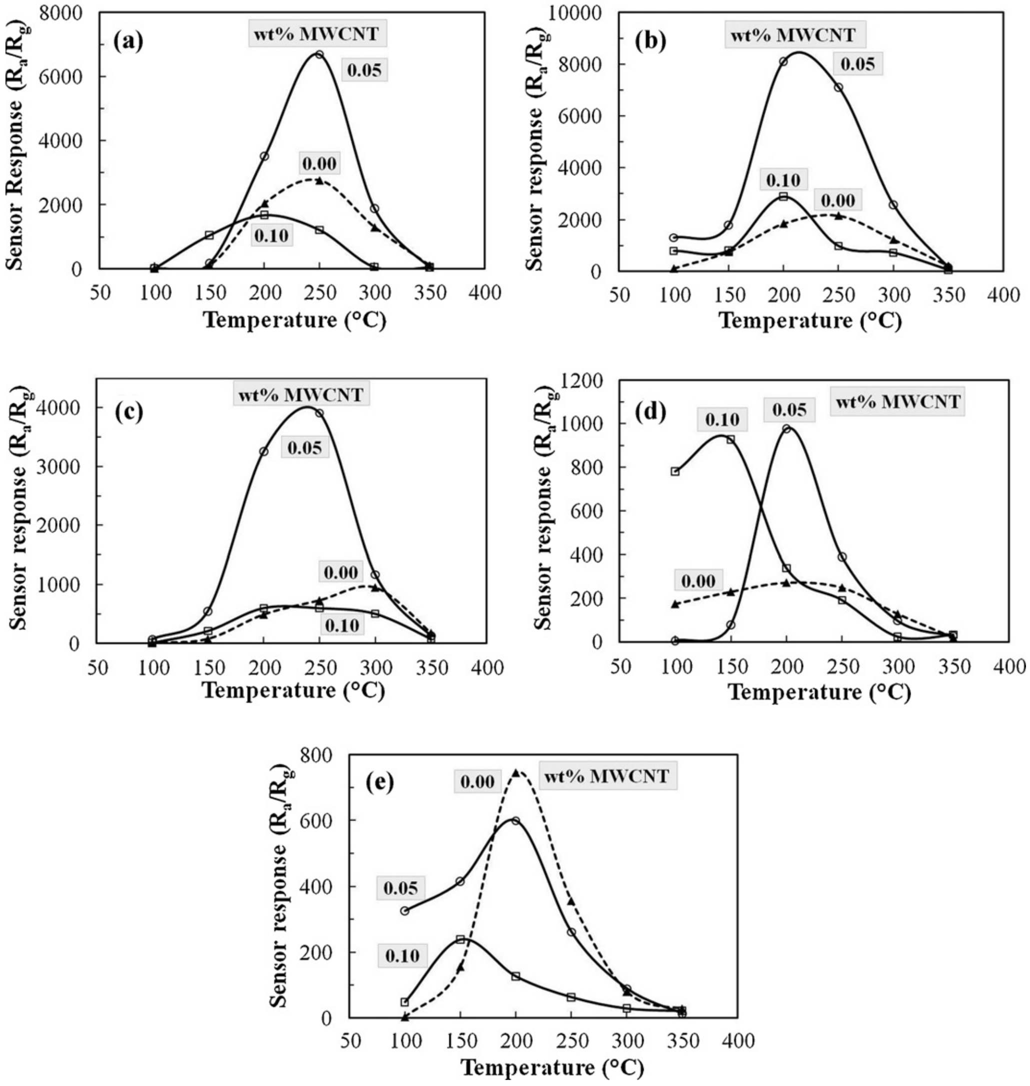

Fig. 5. The sensor response of the SnO_2 and MWCNTs/SnO_2 upon exposure to 300 ppm of (a) ethanol, (b) acetaldehyde, (c) acetone, (d) toluene and (e) TCE in air at different temperatures.

combination of a p-type and n-type structures produce certain conditions under which high sensitivity and good selectivity can be observed [24]. Based on the model suggested by Wei [25], there are two depletion layers in this hetero-structure, one is on the surface of SnO_2 and the other is in the interface of nanotubes and SnO_2.

The adsorption of VOC molecules has a great influence on potential barrier of the interface and leads to changes of depletion layers thickness at the n/p-junction of CNTs and SnO_2 which may improve sensor responses. Sensitivity of the SnO_2-based sensors, in general, is concomitant with the gas adsorption properties of dopants. Therefore, addition of an optimum quantity of CNTs, as a good adsorbent substance, to SnO_2 matrix enhances the sensitivity of the sensors [15].

Response of the sensor to ethanol, acetaldehyde and acetone is much higher than those to toluene and TCE. The reason for this could be found from mechanism of electron transfer reactions. These reactions involve adsorption of gas molecules on the surface and surface reaction of adsorbed molecules with oxygen species. The oxygen species (mainly O^-) are generated by chemisorptions and dissociation of oxygen molecules on the surface of SnO_2. Compared to toluene and TCE, the other VOCs are smaller molecules and more easily diffused. Moreover these gases are nearly similar to each other and have one oxygen atom in their structure. This oxygen atom can facilitate reaction of adsorbed targeted gases with oxygen species. The possible mechanism for these reactions can be expressed as follows [14,19]:

$$CH_3CHO(ads) + 5O^- \rightarrow 2CO_2(g) + 2H_2O(g) + 5e^- \tag{3}$$

$$C_2H_5OH(ads) + 6O^- \rightarrow 2CO_2(g) + 3H_2O(g) + 7e^- \tag{4}$$

$$C_2H_6CO(ads) + 8O^- \rightarrow 3CO_2(g) + 3H_2O(g) + 8e^- \tag{5}$$

Although presence of more O^- on the surface may lead to generation of more electrons, but in the case of inadequate oxygen species on the surface, smaller molecules, i.e., acetaldehyde has more likelihood to completely decompose to CO_2 and H_2O.

Toluene is a benzonoid aromatic hydrocarbon which can react with oxygen species as follows [24].

$$C_6H_5CH_3 + 18O^- \rightarrow 7CO_2(g) + 4H_2O(g) + 18e^- \tag{6}$$

Since toluene is a relatively large molecule, steric hindrance slightly prevents its adsorption on the surface of the SnO_2. Additionally, toluene needs more O^- species to completely decompose to CO_2 and H_2O. TCE has a double bond which is prone to react with oxygen species:

$$C_2HCl_3(ads) + O^- \rightarrow C_2HOCl_3(g) + e^- \tag{7}$$

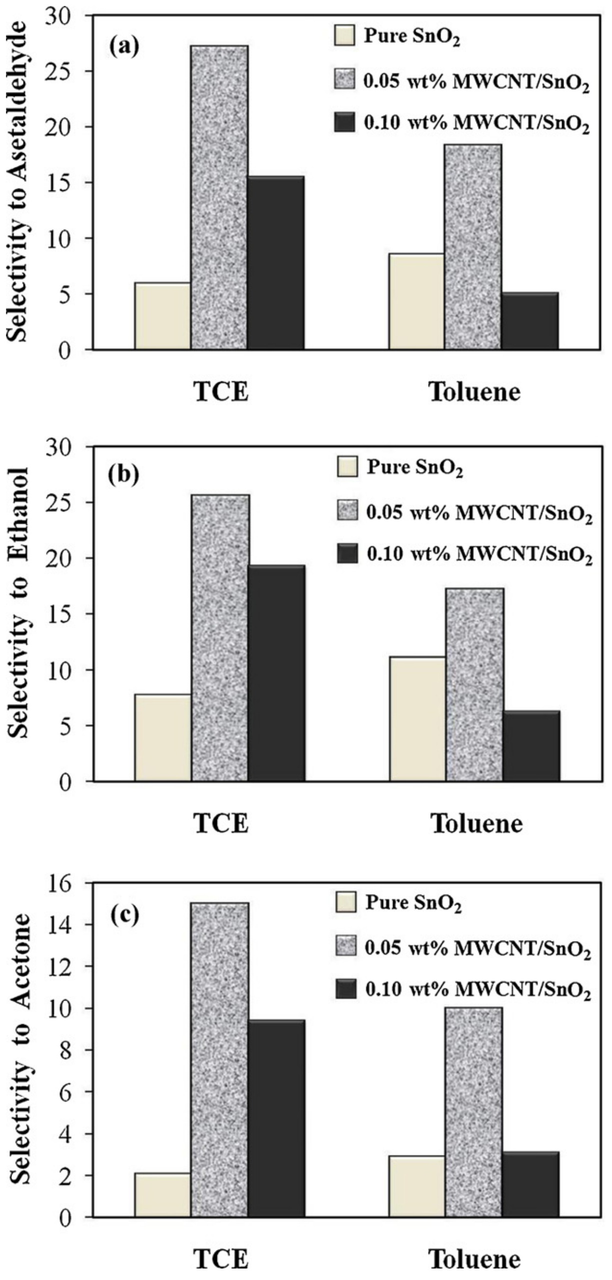

Fig. 6. The selectivity of SnO_2, 0.05 and 0.10 wt% MWCNTs/SnO_2 sample upon exposure to 300 ppm of (a) acetaldehyde, (b) ethanol and (c) acetone in presence of TCE and toluene at 250 °C.

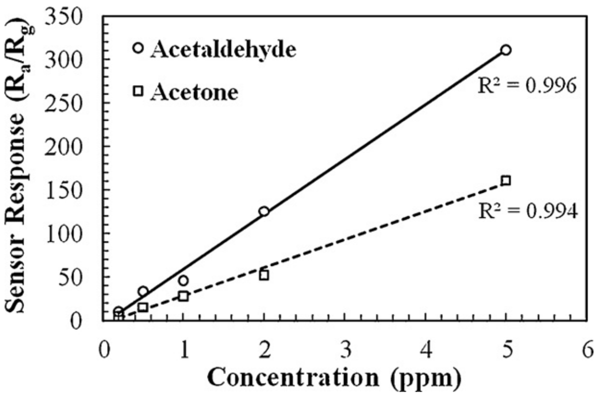

Fig. 7. The 0.05 wt% MWCNTs/SnO_2 sensor response to acetaldehyde at 200 °C and acetone at 250 °C at various low concentrations.

Because of electronegativity of Cl groups, adsorbed TCE transfers only one electron in reaction with O^- and evolved in an epoxy form.

Toluene and TCE are usually present in ambient air and monitoring of other VOCs in the presence of these gases is of great importance [26,27]. Since the response of MWCNTs/SnO_2 samples toward toluene and TCE is low in comparison with acetaldehyde, ethanol and acetone, selectivity of these sensors can be examined. The selectivity is defined as:

$$\varphi_{i/j} = \frac{S_i}{S_j} \tag{8}$$

where $\varphi_{i/j}$ is selectivity to gas i (i.e. acetaldehyde, ethanol and acetone) with respect to gas j (i.e. TCE and toluene) and S_i and S_j are response to gas i and gas j, respectively. Fig. 6 presents the sensor selectivity of blank, 0.05 and 0.10 wt% MWCNTs/SnO_2 samples at 250 °C. All the sensors are selective to acetaldehyde, ethanol and acetone in the presence of TCE and toluene. Addition of nanotubes enhances selectivity of SnO_2-based sensors. For instance, selectivity to acetaldehyde of blank sample in presence of TCE is 6.0 and this value increases to 27.2 for 0.05 wt% sample. Among these sensors, 0.05 wt% MWCNT/SnO_2 shows the highest selectivity.

Due to the importance of trace detection of acetaldehyde and acetone in disease diagnosis, the response of 0.05 wt% MWCNTs/SnO_2 sensor toward sub-ppm level of these gases are studied. Fig. 7 shows sensor response for acetaldehyde at 200 °C and acetone at 250 °C in the concentration range of 0.2–5 ppm. The sensor response is quite high (9.8 for acetaldehyde and 4.7 for acetone) even at low concentration of 200 ppb. Moreover, the sensitivity increased almost linearly with the acetaldehyde and acetone concentrations.

Fig. 8 shows the response and recovery time (i.e. time required to reach 90% of the full response and recovery) of 0.05 wt% MWCNTs/SnO_2, exposed to different concentrations of ethanol in

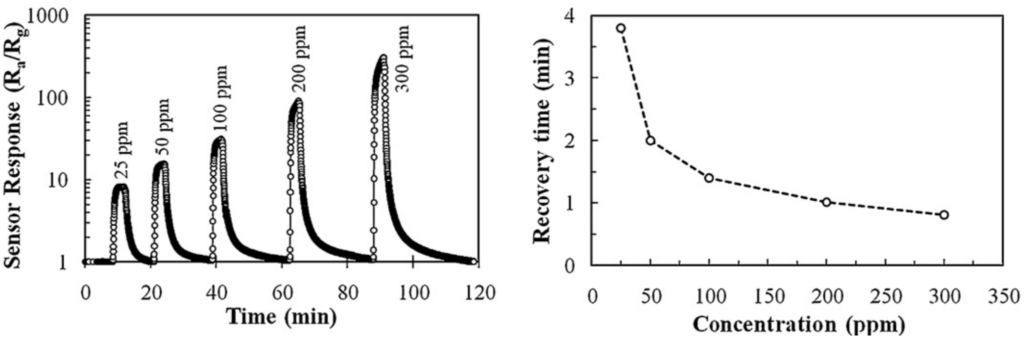

Fig. 8. Transient response and recovery time of 0.05 wt% MWCNTs/SnO_2 sensors exposed to different concentrations of ethanol in air at 350 °C.

air at 350 °C. The recovery times decreased with increasing concentration of ethanol. The average response and recovery time of 0.05 wt% MWCNTs/SnO_2 in various concentrations of ethanol are 150 ± 10 and 108 ± 10 s, respectively.

4. Conclusion

MWCNTs/SnO_2 sensors were fabricated using ultrasonic-assisted precipitation method and their response to ethanol, acetaldehyde, acetone, toluene and trichloroethylene (TCE) VOCs were measured at various temperatures. Layers of SnO_2 nanoparticles are coated on the surface of the added MWCNTs and embedded in SnO_2 matrix. The MWCNTs seems to create easy pathways for electron transfer through SnO_2 matrix. This leads to significantly lower resistances of the sensors.

The added MWCNTs dramatically enhance the responses of SnO_2-based sensors to the VOCs and improve the selectivity of acetaldehyde, ethanol and acetone with respect to TCE and toluene. Up to 2.4, 4.4, 5.3, and 3.6 times enhances in responses to ethanol, acetaldehyde, acetone and toluene, are observed for MWCNTs/SnO_2 sensors, respectively. The 0.05 wt% MWCNTs/sensor shows significant responses to sub-ppm levels of acetaldehyde and acetone, as the diabetes diagnostic gas in breath. Hetrostructure interface between p-type MWCNTs and n-type SnO_2 semiconductors along with enhanced adsorption of the VOCs at the interface seem to be the main causes of the enhanced responses and selectivities.

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