Highly sensitive carbon nanotubes–SnO₂ nanocomposite sensor for acetone detection in diabetes mellitus breath

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A B S T R A C T
Multiwall carbon nanotubes (MWCNTs)/SnO₂ nanocomposites were synthesized by ultrasonic-assisted deposition–precipitation method and used for detection of acetone in the same range of concentrations as in diabetes mellitus breath. The nanocomposite samples were characterized by BET, FE-SEM, XRD and TEM. MWCNTs with an average size of about 60 nm are coated with and uniformly dispersed in 5–10 nm SnO₂ nanoparticles matrix. To diagnose diabetes, the sensors responses to ppm concentrations of acetone were measured in a flow system at various temperatures and 85% relative humidity. Addition of MWCNTs dramatically enhances the sensors response to acetone. Moreover, the sensors response to ethanol, as an interfering species with acetone in human breath, was also measured. Breath humidity increases the conductivity of the sensing materials and therefore, lowers the sensors response to acetone.

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1. Introduction

The major part of breath contains nitrogen, oxygen, carbon dioxide, water and inert gases. About 1000 trace volatile organic compounds (VOCs) form the remaining small fraction. The VOCs concentrations vary from several ppb to ppm. Regarding to the types and quantities of VOCs, each person’s breath is unique. However, there are few common VOCs such as isoprene, acetone, ethane and ethanol in the human breath. VOCs are usually divided in two groups: endogenous, produced by metabolic process, and exogenous, originating from exposure to environmental contaminants. Modern breath analyses started in the 1970s, when the presence of several hundreds of VOCs in human breath were detected by gas chromatography [1].

Endogenous VOCs elucidate some practical information of possible disease state. Elevated levels of some common metabolites are known to be indicative of disease conditions. For instance, renal impairment increases breath isoprene and/or ammonia, dimethyl- and trimethyl-amine are found in the breath of uremic patients, and acetone is closely related to diabetes mellitus [2].

Diabetes in industrial countries is the third largest cause of death. Lack of ability of producing or consuming insulin properly in a body is diabetes. Insulin is a hormone required for converting nutrition into energy [2,3]. A complex disturbance of metabolism results from insufficient effect of insulin. Too much insulin relative to blood glucose level and oppositely lack of insulin followed by intensified lipolysis, are two dominant metabolic changes in diabetes mellitus. Amplified lipolysis producing acetone and ketogenic flux in the liver instigate a rise in circulating ketone bodies: acetoacetate, beta-hydroxybutyrate and acetone. In a normal person, ketone bodies are known to circulate stress hormone or to protect response to fasting, as insulin level falls [3]. Metabolic products of diabetes can be revealed by acetone concentration in the breath, since the gas phase acetone in the blood equilibrates with alveolar air through alveoli [1].

The mean acetone concentration in a healthy human breath is 0.35–0.85 ppm and in diabetic people is more than 2–2.5 ppm [4]. Turner et al. [5] have shown that the mean exhaled ethanol level in healthy human breath samples over 6 months was 196 ppb. Very low concentrations of ethanol are found in the breath, due to the fermentation of glucose by intestinal bacterial flora in response to glucose ingestion or changes in systemic glycemia [6]. Galassi et al. [7] discovered that the breath ethanol and acetone are highly correlated with the corresponding blood glucose values during an oral glucose tolerance test, according to hyperglycemia conditions. Some studies have shown that oral glucose tolerance test will increase the concentration of ethanol up to 1 ppm in the
breath [5,8]. Alcohols in diabetes may be produced by gut bacteria in response to glucose ingestion [9]. The number of VOCs with similar molecular structure and high water content in breath are the problems which impede the widespread use of breath analysis.

Due to the low cost and simple fabrication process, semiconductor metal oxides are promising materials for breath analysis applications. Among the semiconductors, SnO₂ has received the most extensive attention, owing to its high sensitivity, chemical stability, and rapid response and recovery times. SnO₂ is an n-type semiconductor, resistance of which decreases in the presence of a reducing gas. The poor selectivity and high optimum temperatures are the limitations of SnO₂-based sensor for medical applications. To overcome the shortcomings, different dopants like Pt, Au, CNTs are usually added to SnO₂ [10].

Collins et al. [11] have demonstrated that carbon nanotubes (CNTs) exhibit very good adsorption properties, since they have high specific surface areas, nanoscale structures, and large number of sites, where gases can interact. CNTs electric properties are altered by adsorption of gaseous molecules onto the surface and become enabled to act as gas sensors [12]. Improvement of sensing properties by adding functionalized CNTs to SnO₂ have been reported in various studies [12–15].

Choi et al. [13] have used 1.0 wt% MWCNTs/SnO₂ fabricated on micro-platforms, in order to detect low ppm concentrations of NO₂. Espinosa et al. [14] have developed hybrid metal oxide and MWCNTs gas sensors, in order to detect low concentration of NO₂ and CO at low operating temperatures. 0.25–5.0 wt% MWCNTs incorporated into tin oxide thin films have increased the specific surface areas of the samples and improved sensors responses to ethanol [15]. Carbon nanotubes added to alpha-Fe₂O₃ gas sensors have shown enhanced selectivity and gas sensing properties [16].

In the present study, we have used low amounts of 0.05 and 0.10 wt% MWCNTs/SnO₂ nanocomposite sensors to detect low acetone concentrations in the range of 0.5–5 ppm in dry and humid air and determined the sensor selectivity to acetone in the presence of ethanol, for diabetes diagnosis applications. The added MWCNTs significantly enhance the sensors responses and lower the detection temperature.

2. Experimental

The MWCNTs/SnO₂ nanocomposite samples were synthesized by the same method as Ahmadnia-Feyzabad et al. [17]. In summary, MWCNTs were prepared by a chemical vapor deposition (CVD) method and after purifying by controlled air oxidation and acid washing, were functionalized by nitric acid. A sonication-assisted precipitation method was employed to coat MWCNTs and disperse them in the SnO₂ matrix. The precipitate was collected after dispersing appropriate amounts of MWCNTs in SnCl₄ aqueous solution and raising the pH to 9 by ammonia. The precipitate was washed with the deionized water several times to remove chloride ions, and then dried and calcined at 400 °C for 3 h, to prepare the nanocomposite powder. A thick paste of each sample was screen printed on 0.5 mm × 6 mm trench between two gold electrodes on an alumina flat substrate.

A schematic of the experimental setup used for measuring the response of the sensors under various conditions is presented in Fig. 1. The experimental setup comprised of a gas manifold to prepare a flow of air containing 0.5–5 ppm acetone or 1.0 or 2.5 ppm ethanol, a bubbler in a water bath to humidify the air, and a glass tube as the sensor chamber. The sensor was placed in the chamber, which was located in an electric furnace equipped with a PID temperature controller, and connected to an electrical circuit via platinum wires. By using electrical measurement devices interfaced to a computer, the sensor resistance was continuously monitored. Since the exhaled breath contains about 85% relative humidity at 37 °C (0.035 g H₂O/g dry air) the following system was employed to prepare the humid air. A measured flow of 99.995% pure air was passed through a water bubbler placed in a water bath, temperature of which was controlled by a PID temperature controller. The temperature of the tubes connecting the bubbler outlet to the entrance of the glass chamber was also heated to the bubbler temperature, in order to prevent condensation of the water vapor in the transferring lines. The humid air was mixed with zero dry air containing acetone or ethanol. This way, the stream passed over the sensor contained acetone or ethanol and water vapor, the amount of which corresponded to 85 ± 5% relative humidity at 37 °C, i.e. the body temperature.

The response is defined as the ratio of sensor resistance in dry air (Rₓ) to that in the target gas with or without humidity (Rᵧ). The sensors responses were measured at various temperatures in the presence of 2.5 ppm acetone and various concentrations of acetone in the presence of 1.0 ppm ethanol at an optimum temperature. The sensors responses were then measured in the humid air containing acetone at similar conditions.

2.1. Characterization of MWCNTs/SnO₂ samples

The samples structures were identified by X-ray powder diffraction (Philips-Xpert diffractometer), using Cu Kα radiation. To determine the morphology and particles sizes of the nanocomposite samples, a field emission scanning electron microscopy (FE-SEM, JSM 6700F; JEOL, Tokyo, Japan) operating at 5.0 kV and a transmission electron microscopy (TEM, Zeiss EM10C microscope operating at 80 kV) were employed. The specific surface areas of the samples were measured by Brunauer–Emmett–Teller (BET) method, using a Quantachrome CHEMBET-3000 apparatus.

3. Results and discussion

3.1. Characterization

Fig. 2a–c shows the FE-SEM images of SnO₂, pristine MWCNTs, and 0.05 wt% MWCNTs/SnO₂ samples, respectively. Fig. 2a shows a uniform and narrow size distribution of SnO₂ agglomerates in the range of 50–100 nm. Fig. 2b illustrates arrays of multi-wall carbon nanotubes with an average diameter of about 60 nm. Fig. 2c shows the presence of the MWCNTs in the composite with
Fig. 2. SEM micrographs of (a) SnO$_2$, (b) functionalized MWCNTs, (c) 0.05 wt% MWCNTs/SnO$_2$, and (d, e) the TEM micrograph of 0.05 wt% MWCNTs/SnO$_2$ at different magnification.

the SnO$_2$ nanoparticles in the form of agglomerates forming the SnO$_2$ matrix. Fig. 2d and e demonstrates the TEM micrograph of 0.05 wt% MWCNTs/SnO$_2$. The MWCNTs are coated with a layer of SnO$_2$ nanoparticles and dispersed in agglomerates of SnO$_2$ with diameters in the range of 5–10 nm. The coated MWCNTs provide MWCNTs/SnO$_2$ hetero-structures for gas sensing.

MWCNTs and SnO$_2$ have specific surface areas of 248 and 197 m$^2$/g, respectively. When 0.05 and 0.10 wt% MWCNTs are added to SnO$_2$, the surface areas increase slightly to 201 and 207 m$^2$/g, respectively.

Fig. 3 presents XRD patterns of MWCNTs and 0.05 wt% MWCNTs/SnO$_2$. The XRD pattern of MWCNTs shows a sharp peak at 26.23° attributed to graphitic carbon (JCPDS 41-1487). All peaks appeared at 26.56°, 32.28°, 51.49° and 64.96° in the XRD pattern of 0.05 wt% MWCNTs/SnO$_2$ sample are associated with (1 1 0), (1 0 1), (2 1 1) and (1 1 2), respectively, crystal planes of tetragonal rutile structure of SnO$_2$ [2]. Because of the low amount of MWCNTs in the nanocomposite, the characteristic peak of the nanotubes was not detected.

3.2. Gas sensing properties

Fig. 4 presents conductivity of the samples in air, as a function of temperature. The conductivity increases with temperature and MWCNTs contents of the samples. The conductivity of 0.05 wt% MWCNTs/SnO$_2$ is slightly higher than that of SnO$_2$, and as the MWCNTs content of the nanocomposite increases to 0.10 wt%, the conductivity almost doubles at temperatures higher than 150°C. SnO$_2$-coated MWCNTs may be uniformly distributed throughout the SnO$_2$ matrix. The SnO$_2$-coated MWCNTs for the sample
containing 0.10 wt% MWCNTs may have formed a network, resulting in the enhanced conductivity.

The MWCNTs, depending on the temperature, act as a conductor or semiconductor. At temperatures higher than the room temperature, MWCNTs are expected to be a p-type semiconductor with a low band gap of about 0.05 eV [18]. Furthermore, the Schottky barrier between SnO2 and MWCNTs and the difference in their work function is low. Hence, electrons are conducted more easily through MWCNTs matrix and MWCNTs/SnO2 samples show higher conductivities [19].

The variation of the sensor conductivity with temperature, i.e. its rise to 150 °C, a decrease to 200 °C, and monotonous increment at higher temperatures are explained as follows. Oxygen adsorption plays an important role in the electrical properties of the MWCNTs/SnO2 nanocomposite, depending on the temperature. Oxygen adsorbs on the surface of the sensor and forms O2−, O22− ions by capturing electrons from the conductance band and creates an electron-depleted space-charge layer in the surface region of the sensor nanoparticles. Oxygen chemisorption does not occur effectively at temperatures lower than 150 °C. From 150 to 200 °C, chemisorbed O2− converts into O2−/O22−, and thus more electrons are absorbed from the space-charge layer and the conductivity decreases. At temperatures higher than 200 °C, the conductivity increases, due to the dominance of the electron thermal excitations [20,21].

Responses of the SnO2 and MWCNTs/SnO2 samples upon exposure to 2.5 ppm acetone in air as a function of temperature are shown in Fig. 5. At 200 °C, as 0.05 and 0.10 wt% MWCNTs are added to SnO2, the sensors responses are enhanced by factors of 10 and 9, respectively. Acquiring high sensors responses at the low temperature of 200 °C would increase their long term stability and decrease their power consumption.

Samples responses exhibit volcano-shaped behaviors versus temperature. This can be related to the competition of diffusion and surface reactions rates on the sensing film. The sensing mechanism of the target gas on the semiconductor gas sensor includes diffusion of the target gas, adsorption, surface reaction with chemisorbed oxygen on the semiconductor, and desorption. Kinetics of these processes depend on the sensor morphology and temperature [22]. An increase in the temperature and thus an increase in the rate of surface reaction in the range of 100–200 °C, raise the sensor response. The decrease in the utility factor of the gas sensing body, due to the diffusion limitations, results in a decrease in sensor response at higher temperatures. At the temperature of the maximum response, the target gas molecules have optimal reactivity for diffusion in the sensing part (large utility factor) as well as for exerting sufficiently large interaction with the surface [18]. Addition of 0.05 wt% nanotubes enhances the response of the gas sensor at all temperatures. Also it decreases the optimum temperature at which maximum response to the target gas occurs, from 250 to 200 °C. Nanotubes may enhance gas diffusion through SnO2 structure, by providing more paths in the sensing layer, and consequently the sensor does not require high temperatures for high responses [23]. It has been also reported that the enhancement effect is related to the nano-channels formed by MWCNTs embedded in SnO2. The nano-channels in SnO2 enhance the local electric field at CNT–SnO2 interface by increasing gas molecules diffusion into the SnO2 surface [24].

The gas sensing mechanism of semiconductors generally involves oxygen adsorption, target gas adsorption and surface reaction of adsorbed molecules with oxygen species. The oxygen vacancies in SnO2 nanoparticles act as electron donors providing electrons to the conduction band. As the sensors are exposed to air, the oxygen molecules are adsorbed and capture electrons from the conduction band. This adsorption leads to oxygen ionosorption in molecular (O2−) and atomic (O−, O2−) forms. The dominating species depend on temperature and, probably, on the type of dopants present in the sensing material. Consequently, the depletion layer on the surface is expanded, resulting in an increase in the resistance of the sensor. When the sensor is exposed to acetone, the reducing acetone reacts with the adsorbed O−, as described in Eq. (1), and the trapped electrons are released back to the...
conduction band, leading to an increase in the carrier concentration and a decrease in resistance of the sensor [25].

\[
\text{CH}_3\text{COCH}_3\text{(adsorbed)} + 8\text{O}^-(\text{adsorbed}) \rightarrow 3\text{CO}_2 + 3\text{H}_2\text{O} + 8\text{e}^- \tag{1}
\]

Addition of MWCNTs enhances sensitivity through promoting gas diffusion and surface adsorption in the sensing layer, decreasing resistance, and assembling hetero-structure of n-SnO₂/p-MWCNTs. The most possible mechanism is the last one and can be explained as follows: MWCNTs act as a p-type semiconductor, when exposed to an ambient atmosphere [26]. Regarding the detection mechanism, Wei et al. [12] have suggested that a hetero-structure is formed at the interface between n-type SnO₂ and p-type MWCNTs. This leads to the formation of two depletion layers: one on the surface of SnO₂ particles, and the other at the interface between MWCNTs and SnO₂ (p–n junction). The adsorption of the target gas probably induces a modulation of surface charges that directly affect the electronic transfer in the junction, and also induces a variation in the resistance of the sensing layer [18]. Potential barrier of the interface is greatly affected by adsorption of VOC molecules and it leads to changes of depletion layers thickness at the n/p-junction of MWCNTs and SnO₂. Adsorption of acetone and its interaction with the adsorbed oxygen may change the depletion layer at both the grain surface and n/p-junction of MWCNTs/SnO₂. Therefore, the sensitivity is developed due to the amplification effects of junction structure combined with the surface reactions of the target gas [27,28].

Due to the fact that the human breath contains about 85% RH at 37 °C, detection of acetone in the presence of the RH is of great importance. Fig. 6 shows that, when humid air is used, the 0.05 wt% MWCNTs/SnO₂ sensor response to acetone decreases by a factor of up to 4.6 times, however, the sensor response of 120 to 2.5 ppm acetone at 200 °C is still dramatically high. Humidity, depending on temperature, affects the sensor response. It may be due to the competition between acetone and water adsorption. The influence of water vapor on the resistance of SnO₂-based sensors has been investigated by different research groups [29,30].

The most accepted mechanism is that, similar to the reducing gases, water vapor increases the conductance of semiconductor gas sensors. When water molecules are chemisorbed on a tin-oxide surface, the conductivity is influenced in two ways. The first is that the molecules dissociate into hydroxyl species, which act as electron donors. These electrons cause a direct increase in the sensor conductivity [31]. The second mechanism is that hydrogen atoms produced from the water molecule dissociation may react with lattice oxygen by creating vacancies. These vacancies then diffuse to the bulk, where they act as electron donors [32]. Hydroxyl groups, but not the molecular H₂O, result in an increase in the conductivity. The form of adsorbed species is highly dependent on the temperature. It is proposed that, at temperatures higher than 200 °C, no molecular species are observed but the hydroxyl groups [33]. As the temperature decreases from 400 °C to 200 °C, more H₂O is adsorbed on 0.05 wt% MWCNTs/SnO₂ and blocks active sites for the acetone adsorption, resulting in the 4.6 times decline in the sensors response.

Fig. 7 reveals the sensors responses to various concentrations of acetone in the range of 0.5–5 ppm in dry and humid air at 200 °C, as the optimum operating temperature. Humidity decreases the 0.05 wt% MWCNTs/SnO₂ sensor responses at all concentrations. Fig. 8 shows the response and recovery times required to reach 90% of the full responses and recoveries of 0.05 wt% MWCNTs/SnO₂, exposed to 2.5 ppm acetone in dry air. Response times of 67, 56, 42 s and recovery times of 32, 10, 5 min are observed at 200, 300, and 400 °C, respectively. 0.05 wt% MWCNT/SnO₂ at 200 °C is not completely recovered, thus, the temperature was increased in order for the sensor to be recovered completely. Recovery times significantly decrease as the temperature increases.

In breathe analysis, the presence of other VOCs like alcohols interfere with acetone detection. Ethanol in ppb levels always present in human breath. Fig. 9 presents the responses of 0.05 wt% MWCNTs/SnO₂ sensor exposed to 1.0 ppm ethanol in dry and humid air as a function of temperature. Below 300 °C the responses...
to ethanol dramatically decrease in humid air. However, at higher temperatures responses to ethanol in dry air are less than those in humid air. At the optimum sensing temperature of 200 °C, the 0.05 wt% MWCNTs/SnO2 sensor response to 2.5 ppm acetone in the humid air is about 10 times higher than its response to 1.0 ppm ethanol. At the sensor operating temperature of 200 °C and in the presence of 85% humid air, the 0.05 wt% MWCNTs/SnO2 sensor response to 2.5 ppm acetone is about 100 (Fig. 6), while that to 1.0 ppm ethanol is about 10 (Fig. 9). According to several studies selective detection of acetone and ethanol is quite challenging because of their close responses on various sensors [29,34–37]. Similar chemical nature of CH3CH2O chemisorption could explain the close responses. The sensitivities to ethanol and acetone become more similar at higher temperature of 300–400 °C, due to the rapid kinetics of ethanol and acetone oxidation at elevated temperatures [38]. However, 10 times larger sensitivity of 0.05 wt% MWCNTs/SnO2 sensor to acetone than to ethanol at 200 °C and 85% humid air in this study may indicate minor interfering effect of ethanol.

The mechanism of ethanol sensing is related to its oxidation and/or decomposition reactions. As ethanol dissociates, the adsorbed oxygen species form an ionic bond with an unsaturated metal site and a hydrogen atom is bound to a nearby oxygen anion. The residual hydrogen from ethanol adsorption desorbs as H2O or H2. Following the ethoxide formation, oxidation results in electron transfer to a metallic site [39]. It has long been realized that oxidation of ethanol takes place via two routes, the dehydrogenation to acetaldehyde and the dehydration to ethylene, depending on the acid–base properties of the surface. The products formed are then oxidized to CO2, CO and H2O and the first reaction has more sensitivity than the latter [40]. Ethanol is dehydrogenated on SnO2 surface Lewis acid sites by the chemisorbed oxygen and releases free electron [29]. Therefore, the resistance decreases in the presence of ethanol [38,40].

\[
\begin{align*}
\text{CH}_3\text{CH}_2\text{OH} + \text{O}_s & \rightarrow \text{CH}_3\text{CH}_2\text{O} - \text{M}_{\text{ads}} + \text{O} - \text{H}_{\text{ads}} \\
\rightarrow & \text{CH}_3\text{CHO} + \text{H} - \text{M}_{\text{ads}} + \text{O} - \text{H}_{\text{ads}}
\end{align*}
\]

(2)

The effect of the presence of water on ethanol sensitivity depends on temperature and amount of water vapor [41,42]. At high relative humidity, the influence of water molecules on the conductivity of SnO2 film is much larger than that of ethanol, hence the ethanol response decreases. The presence of moisture reveals that two factors may be invoked to clarify the decline of ethanol response in humid ambient. First, hydroxyl groups block sites and prohibit the formation of bridging species. Second, water molecules compete with the adsorbed species and decrease superficial concentrations, in order to prevent catalytic oxidation of ethanol to acetaldehyde [32,43]. However, at higher temperatures, hydroxyl groups resulting from water adsorption contribute to ethanol adsorption and cause more electron transfer to metallic sites. Hence, at higher temperatures, the sensor responses to ethanol in humid air become greater than those in the dry air.

Fig. 10 shows the transient responses and recoveries of 0.05 wt% MWCNTs/SnO2 sensor at 200 °C to 2.5 ppm acetone, at two different times 7 days apart. During this time, the sensor has been under continual operation at different temperatures and gas concentrations. The sensor responses at the 1st and 7th day are 95 and 80, respectively. The response decreases by 16%, while the response and recovery times are varied from 2 to 3 and from 13 to 18 min, respectively. This shows a reasonable durability and stability of the sensor.

4. Conclusion

We have successfully fabricated MWCNTs/SnO2 nanocomposite sensors with dramatically improved responses to 0.5–5 ppm acetone found in diabetic mellitus breath. MWCNTs form heterojunction with SnO2 nanoparticles coated on them, are uniformly dispersed in SnO2 matrix, and significantly lower the resistance of the sensors. Compared to SnO2, the 0.05 wt% MWCNTs/SnO2 sensor shows up to 10 times higher response to 2.5 ppm acetone, at 200 °C. This was anticipated to be due to formation of a heterojunction at the interface between p-type MWCNTs and n-type SnO2 semiconductor along with enhanced adsorption of the target gas at the interface. Water vapor in the humid air has no effect on the 0.05 wt% MWCNTs/SnO2 sensor response to acetone at 300 °C and higher temperatures. However, at lower temperatures, the humidity significantly lowers the response to both acetone and ethanol, possibly due to the competitive adsorption of water and acetone or ethanol on the sensor surface. One order of magnitude higher response of the sensor to acetone than to ethanol indicates minor interfering effect of ethanol at breath conditions.

Acknowledgement

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References


Biographies

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