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Sensors and Actuators B 126 (2007) 387–393
SENSORS AND ACTUATORS B CHEMICAL
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New sensing technology for detection of the common inhalational anesthetic agent sevoflurane using conducting polypyrrole films

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Received 27 August 2006; received in revised form 17 March 2007; accepted 19 March 2007
Available online 27 March 2007

Abstract

A new type of gas sensor to measure resistance was developed to detect sevoflurane concentrations at room temperature. The measured FTIR spectrum of sevoflurane vapor was given. The sensing material polypyrrole was synthesized in situ by UV-photopolymerization. The relative resistance variation was 3.9 for a sevoflurane concentration of 1.2%. The sensor response time (60 s) and recovery time (20 s) were very short. Molecular dynamic calculations made for sevoflurane adsorption on polypyrrole revealed formation of a new bond, N–H–F. The bond length was predicted to be in the range of 0.2–0.4 nm. The bond energy was about 7.2 kcal/mol. Sensor responses to the concentrations 0.1–10% were 1.57–5.9, respectively.

Keywords: Sevoflurane; Polypyrrole; Conducting polymer; UV-photopolymerization; VOC sensor

1. Introduction

Among numerous inhalational anesthetic agents in current use are potent volatile liquids of halogenated derivatives of alkanes, particularly halothane ($CF_3CHClBr$; 1956 as fluorothane), the halogenated derivatives of ethers, especially enflurane ($CHClFCF_2OCHF_2$; 1972 as ethrane), the malodorous isoflurane ($CF_3CHClOCHF_2$; 1981 as forane), the more volatile but equally malodorous desflurane ($CF_3CHFOCHF_2$; 1992 as suprane), and lastly the non-irritating, sweet to taste, highly insoluble sevoflurane ($C_4H_3F_7O$; 1990 as ultane). All play vital roles in the pain management of patients undergoing surgery [1–4].

The most commonly used inhalational anesthetic agent, sevoflurane, is a fluorinated derivative of methyl isopropyl ether (International Union and Applied Chemistry, IUPAC nomenclature; fluoromethyl 2,2,2-trifluoro-1-(trifluoromethyl) ethyl ether). Its structure is shown in Fig. 1. Owing to its many medical applications, sevoflurane has rapidly replaced halothane as an inhaled anesthetic agent. Its benefits include a quick induction and emergence from anesthesia, a non-pungent odor, allowing for mask induction, and decreased airway irritation. On the downside are several side effects, including seizures during induction and maintenance, elevation in plasma, and an increased incidence of emergence delirium over halothane. Monitoring the concentration of sevoflurane, particularly in the surgical wards where support staff may be exposed, is essential. In recent times, measuring the concentration of sevoflurane in blood and in clinical inhalation has become important [5,6]. Exposure to waste anesthetic gases can strongly impact workers in operating rooms, dental offices, and veterinary offices. It has also been reported that some of the highest levels of waste anesthetic gases have been found in post-operative recovery rooms [1(b)].

Several instrumental techniques, such as simple gas chromatography and hyphenated/coupled methods like gas chromatography-mass spectrometry (GC–MS), are applied to the detection of concentrations of sevoflurane in blood [5–7]. Ho

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doi:10.1016/j.snb.2007.03.026

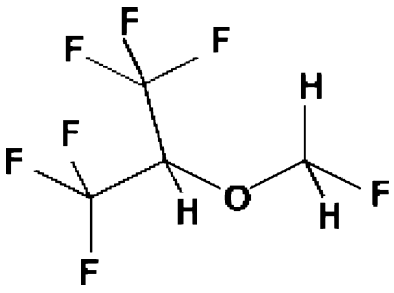

Fig. 1. Structure of sevoflurane.

and co-workers [5] further modified the previously developed headspace (HS) GC–MS method to analyze and distinguish simultaneously the five common clinical inhalation anesthetics, which includes sevoflurane. An infrared (IR) method was used to measure the concentration of sevoflurane in the inhalation and expiration of a patient's breath [8]. The lack of identification in each IR absorption peak of sevoflurane was studied. Hahn and co-workers [9] used an electrochemical reduction method to detect sevoflurane both at low concentrations (<0.2%, v/v) and high concentrations (0.5–2%, v/v). Reverse-phase high performance liquid chromatography (HPLC) was applied for indirect determination of sevoflurane by measuring the urinary metabolite, hexafluoroisopropanol (HFIP) [10]. The detection limit of HFIP was close to 1 μg/L. High resolution two-dimensional (2D) fluorine-19 NMR was used to investigate the sevoflurane [11].

Polypyrrole (Ppy) is a characteristic-conducting polymer and promising sensing material employed in various chemical sensors [12–18]. The bicarbonate-doped Ppy was coated as the material of pH-indicator electrode to probe carbon dioxide [12]. Pyrrole was polymerized with the matrix of cross-linking polyvinyl alcohol (PVA) to form a composite sensing material and that could detect NH_3 [13]. A Ppy filament sensor was sensitive to ethanol and ozone [14]. By using a resistance change method [15], various anion-doped Ppy films could detect methanol gas. Ppy and copper-doped Ppy films revealed an increasing in resistance upon exposure to reducing gases such NH_3, H_2 and CO [16]. Blending Ppy with either one of poly(caprolactone), poly(ethylene oxide), poly(methylmethacrylate), poly(vinyl alcohol) or poly(vinyl-acetate) exhibited a promoted sensitivity to methanol, ethanol, carbon tetrachloride, and benzene [17]. Ppy-based electronic noses [18] are applied to the measurement of ammonia, nitrogen oxides, carbon monoxide, sulphur dioxide, hydrogen sulphide, methane, oxygen, hydrogen, alcohol, phenol, benzene, and water vapor.

The purpose of this study was to investigate the applicability of the newly developed in situ UV photopolymerized sensing material Ppy for sensing the inhalational anesthetic agent, sevoflurane. This anesthetic gas sensor was based on variation in resistance and is suitable for detecting sevoflurane at room temperature.

2. Experimental

All the chemicals used were analytical reagent (AR) grade (purity > 99%), purchased from Sigma–Aldrich Co., Inc., USA, unless otherwise mentioned. All the chemicals were used as received. Water was distilled and de-ionized (DI) using a Milli-Q water purification system (Millipore Corp.).

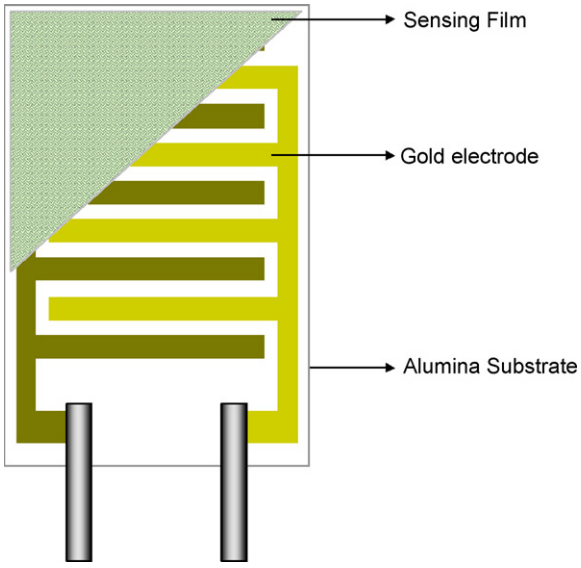

Fig. 2. Sensing film coated on an alumina substrate.

2.1. Preparation of sensing material

0.07 g of monomer pyrrole was added to 100 ml of ethyl alcohol to form a solution. An approximate quantity of $AgNO_3$ was added to the solution and stirred thoroughly, making it equally distributed throughout the pyrrole alcohol matrix and available for initiating photopolymerization. Subsequently, a chip was dip coated with the pyrrole material. The chip was made of an alumina substrate with dimensions of 10 mm × 5 mm, on which a pair of comb-like interdigitated gold electrodes were screen printed (see Fig. 2), followed by UV treatment for 5.5 h to photopolymerize at room temperature. The dipping and withdrawing speed of the substrate were about 3 and 5 cm/s, respectively.

2.2. Sensing system

We employed a dynamic flow system for the sevoflurane-sensing chamber, as shown in Fig. 3. The sevoflurane (ultane) used was obtained from Abbott Laboratories, UK (http://www.abbott.com/). Several concentrations of sevoflurane were prepared using a divided flow method, and two mass flow controllers were used to tune the dilution factor. Within the test system, the concentration was varied from 0.1% to 10%. The flow rate of the test gas was fixed at 1000 cm^3/min during measurements. A simple voltage circuit was designed as follows in order to facilitate measurements. The sensor resistance and response (S) can be obtained from Eqs. (1)–(5).

$$I = \frac{V_s}{R_s + R_r} = \frac{V_m}{R_r} \quad (1)$$

$$R_s = R_r \frac{V_s - V_m}{V_m} \quad (2)$$

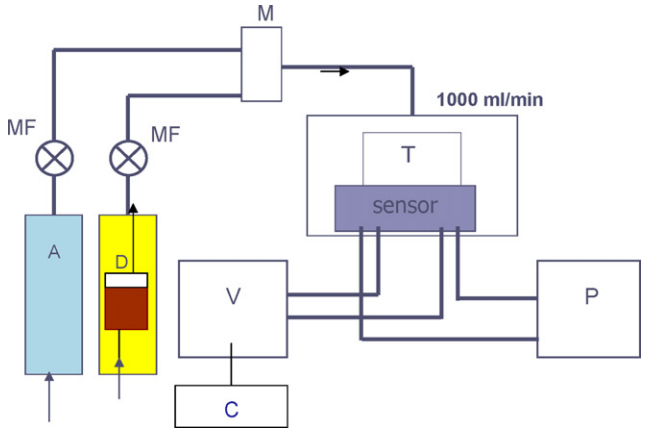

Fig. 3. Dynamic divided flow sevoflurane sensing system—MF: mass flow controller; A: air pump; D: sevoflurane (or ethanol, acetone) bottle; M: mixer; T: test chamber; C: computer; V: voltage transfer circuit and voltage meter; P: power supply.

$$R_{air} = R_r \frac{V_s - V_{air}}{V_{air}} \quad (3)$$

$$R_{gas} = R_r \frac{V_s - V_{gas}}{V_{gas}} \quad (4)$$

$$S = \frac{R_{gas}}{R_{air}} = \frac{V_{air}(V_s - V_{gas})}{V_{gas}(V_s - V_{air})} \quad (5)$$

where V_s is the input voltage ($V_s = 2.0$ V), R_s the cascade sensor head resistance, and R_r is the reference resistance ($R_r = 18.2$ MΩ). The input data consists of the voltage difference (V_m) across the reference resistance. All the sensing experiments were done at 23.0 ± 0.5 °C, and humidity was controlled at $63 \pm 10\%$ RH.

2.3. UV–vis testing and SEM

The obtained Ppy was investigated as evidence of completion of polymerization using UV–vis spectroscopy. The UV–vis spectrometer used here was SINCO, SUV-2100 series (http://www.scinco.com; Korea). Light sources for the UV–vis were deuterium and tungsten-halogen lamps, respectively, and the wavelengths scanned ranged from 200 to 700 nm. The morphology and thickness of the samples were obtained with a JEOL scanning electron microscope (SEM model number: JSM-6500), using an accelerating voltage of 5–15 kV.

2.4. FTIR characterization

FTIR (Fourier transform infrared spectroscopy) was also used to note the spectral signature of the sevoflurane in this study. Sevoflurane characterization was done with ABB-BOMEM FTIR (D8 Series, Canada) equipped with a Mercury–Cadmium–Telluride (MCT) detector. NaCl crystal with a dimension 25 mm × 4 mm (Spectral Systems Inc., #955-3616; USA) was used to collect the spectra. Each spectrum was taken in transmission mode at room temperature under atmospheric pressure, at an average of 64 scans with a 2 cm^{-1} resolution in 400–5000 cm^{-1}. A background spectrum was recorded accordingly.

2.5. Theoretical analysis

A series of molecular dynamics/simulations calculated the bonding energy for sevoflurane molecule adsorption on to the Ppy surface using Materials Studio®, Version 3.2 (Accelrys Software Inc., http://www.accelrys.com/products/mstudio/). By applying the discovery method of the software, the parameters were chosen to minimize the system energy, and the Compass force field was used.

At first, the homopolymer was built and the parameters of polymerization of polypyrrole were optimized. Related conditioned parameters chosen were rings library, pyrrole repeated unit, isotactic tacticity, three chain length, and six chain number. The lattice parameters of polypyrrole were set at 3D triclinic without constraint. From the initial to final potential energy change during the process of calculation, the final potential energy was chosen. This term refers to the most stable structure of the adsorbed molecule and Ppy surface system. According to the simulation of the adsorption on the Ppy surface, bond length and energy of the new formation can be determined.

3. Results and discussion

3.1. FTIR characterization of sevoflurane

Fig. 4 depicts the FTIR spectrum of sevoflurane. Its interpretation is being performed for the first time by our research group. The obtained IR spectrum has several prominent bands in the region between 1033 and 1378 cm^{-1} and a few less significant bands in the region between 2100 and 3000 cm^{-1}, which mostly comprise aliphatic stretching frequencies of –CH, $–CH_2$ and $–CH_3$. The spectrum has all the expected characteristic peaks like stretching frequencies from –CF. ν_{CF} mostly forms between 1100 and 1400 cm^{-1}. Below 1000 cm^{-1} there are three prominent peaks at 692, 746 and 897 cm^{-1}. The peaks at 746 and 897 cm^{-1} are assigned to a symmetrical skeletal vibration of (CF_3), (CF_3)–C–O– and a CH_2 rocking vibration, respectively. The IR band at 1033 cm^{-1} is due to the C–F stretch, and the –C–O stretch is seen at 1123 cm^{-1}. The 1230 cm^{-1} peak

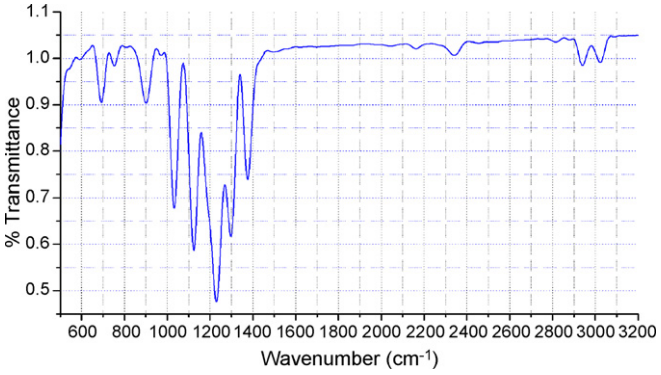

Fig. 4. FTIR spectrum of sevoflurane.

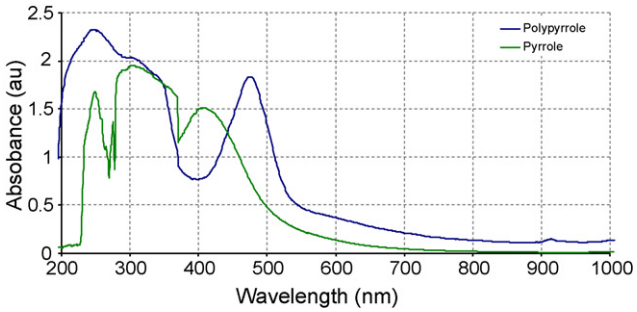

Fig. 5. UV–vis spectra of photopolymerized Ppy.

is due to an asymmetric C–O–C stretching vibration mainly attributed to a CH_2 rocking vibration. The band at 1378 cm^{-1} could also be from CHF_3 stretching. The $–CH_2–O$ stretch is seen at 2819 cm^{-1}. The less conspicuous aliphatic –CH bending vibration is seen between 1410 and 1420 cm^{-1}. Aliphatic –CH stretching, ν_{CH} and $–CH_2$ stretching, ν_{CH_2} are seen at 2878 and 2945 cm^{-1}. The $–CH_2$ stretching is less prominent along with another peak at 3025 cm^{-1}. The reason why aliphatic –CH stretching, ν_{CH} at 3300 cm^{-1} has not been observed may be due to the presence of more bulky atoms around those influencing the $\nu_{C–H}$ stretch.

3.2. UV–vis characteristics

The UV–vis spectrum of pyrrole is depicted in Fig. 5, and the peak of the pyrrole band was at 280 nm after UV treatment at room temperature for 5.5 h. For photopolymerization [19,20] to occur, broad peaks must be seen over a range of 400–520 nm, and the maximum peak height was located at 480 nm, indicating the completion of polymerization, Py → Ppy. This explains that pyrrole monomers were polymerized into polypyrrole. Thus, the obtained prominent absorption peak at 440 nm is due to the electronic transition of π to π^* bonds in Ppy [19,20].

3.3. Thickness related sensor response

Fig. 6(a) shows a side view SEM image of the Ppy film after 5.5 h UV photopolymerization on the alumina substrate. After 1.5, 3.5, 5.5 and 7.5 h, the photopolymerization process was examined to detect the thickness of the polypyrrole film, and the thickness was estimated by SEM to be about 4.4, 5.1, 5.3 and 6.1 μm, respectively. The top side view of the film after 5.5 h UV photopolymerization is revealed in Fig. 6(b), and the smooth surface film morphology is examined. The thickness of the film varied the sensor response. The sensor response to sevoflurane of Ppy was tested to be optimum with the 5.5 h photopolymerization process the thickness was about 5.3 μm. The relative resistance variation of the 5.3 μm thickness was 3.9 for a sevoflurane concentration of 1%.

3.4. Sevoflurane molecule adsorbed on Ppy surface

The local adsorption geometry, sevoflurane molecule adsorption on the Ppy surface, is shown in Fig. 7. It indicates that

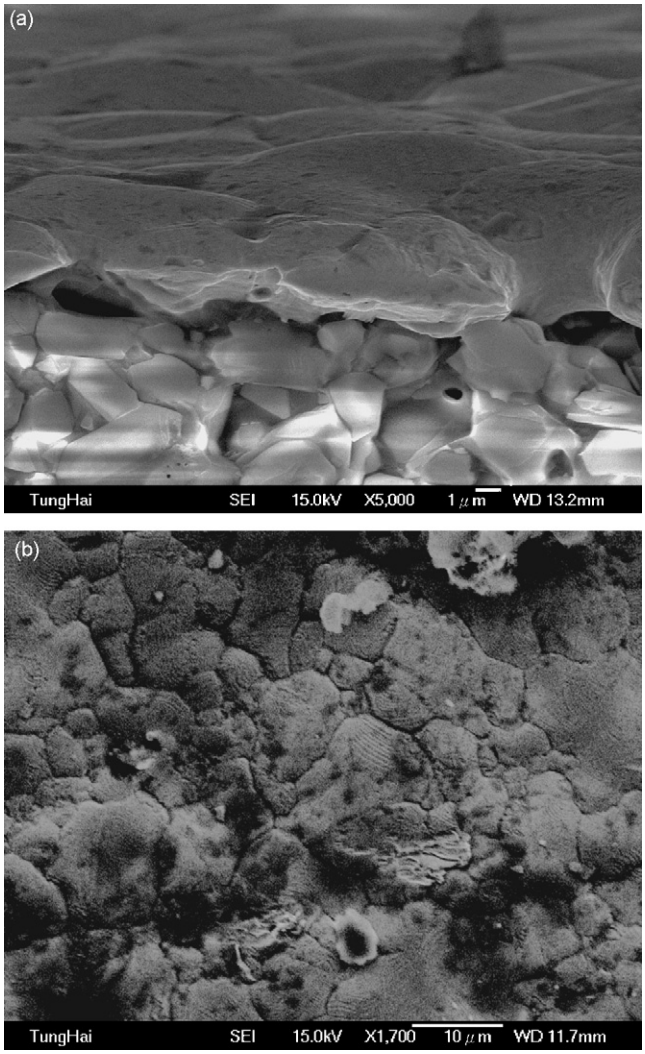

Fig. 6. SEM images of a Ppy sensing film on an alumina substrate: (a) side view and (b) top view.

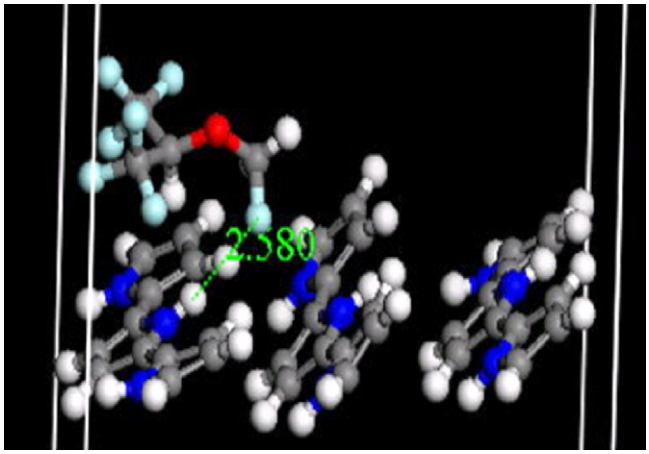

Fig. 7. Simulation of sevoflurane adsorbed on the Ppy surface. White, deep blue, light blue and red ball represent hydrogen, nitrogen, fluorine and oxygen atoms, respectively. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of the article.)

the fluorine atom of sevoflurane was bonded to the H atom of Ppy. According to the calculation of sevoflurane adsorbed on polypyrrole, the new bond N–H–F could have been formed. The bond length was predicted as in a range from 0.2 to 0.4 nm in various dynamic situations. The average bond length was estimated about 0.258 nm. Sevoflurane adsorption on polypyrrole was calculated as an exothermic process.

The bond energy was calculated as −7.2 kcal/mol, and the interaction of sevoflurane and Ppy was interpreted to take the form of a hydrogen bond on the surface. Generally speaking, the hydrogen bond energy and bond length are estimated below 10 kcal/mol and 0.3 nm, respectively. With the hydrogen bond optimized, the physisorption and desorption of sevoflurane molecules on polypyrrole is reversible. Therefore, the response and recovery times to sevoflurane are short.

3.5. Sensing curve of sevoflurane

Sevoflurane was adsorbed on the surface of Ppy. This was given as a sensing characteristic curve in Fig. 8. While sevoflurane was adsorbed on the Ppy, the fluorine atom of sevoflurane bonded to the hydrogen atom of pyrrole. The fluoride atom possesses high electronegativity and can withdraw the electron from the hydrogen atom in Ppy, decreasing the electron density of Ppy and increasing the resistance on the surface. This phenomenon resembles the electrochemical growth method that occurs in an ozone sensor [14]. While the ozone was being dosed on the sensor surface, the resistance of Ppy increased by about 20 Ω [14]. Other volatile liquids like methanol and ethanol were also exposed to the Ppy thin film as gas vapors, and an increase in the resistance of Ppy was observed [21]. The above results occurred because atoms with higher electronegativity than the N atom (like F and O atoms) can withdraw the electron from the hydrogen atom in Ppy, and the resistance of the Ppy surface increases.

Fig. 8 also demonstrates that the resistance increased with the change in sevoflurane concentration in the chamber. The response time T90 (the time required to achieve a signal that is 90% of the equilibrium signal) of 1% sevoflurane sensing curve is less than 60 s, and the recovery time is less than 20 s. The physisorption and desorption of sevoflurane molecules on

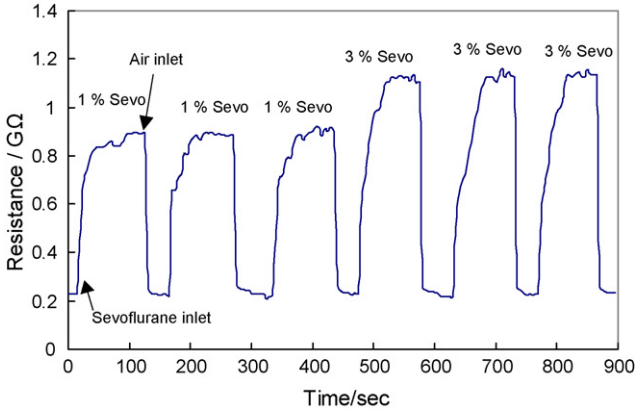

Fig. 8. Sensing curve to 1% and 3% sevoflurane of Ppy.

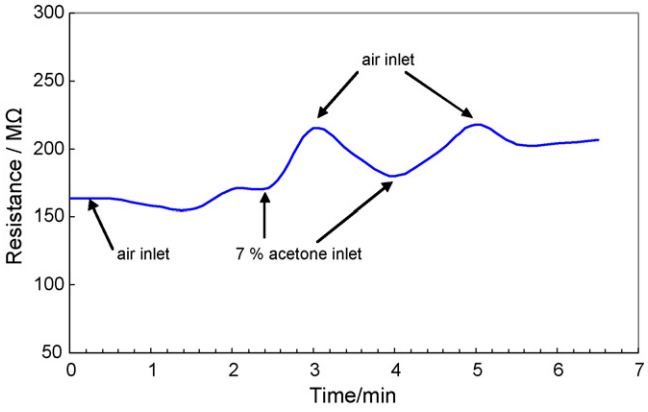

Fig. 9. Sensing curve to 7% acetone of Ppy.

polypyrrole is reversible. Thus, the response and recovery time of sevoflurane is fast. Sensor response ($S = R_{sevoflurane}/R_{air}$) is about 3.95 under a 1% sevoflurane concentration.

Continuous sensing curves to the sevoflurane concentrations of 1% and 3% are shown in Fig. 8. The sensor response was tested thrice at each concentration, and the relative standard deviations were calculated as 5.4% and 6.1%.

3.6. Interference effect with other gases

The sensing curve to 7% acetone/air of Ppy is shown in Fig. 9. The baseline is not stable and increases with time. The sensor response was 1.26 at this concentration. Three different concentrations of acetone and ethanol were tested thrice and the results are illustrated in Fig. 10. Table 1 reveals that the cross-selectivity for sevoflurane against ethanol was 4.04 and that for sevoflurane against acetone was 3.86.

From Fig. 10 and Table 1, the interferences with 2% ethanol and 10% acetone were 4.04 and 3.86, respectively. Using the theoretical molecular calculation for sevoflurane, ethanol, and acetone, the molecular adsorption bonding energies were cal-

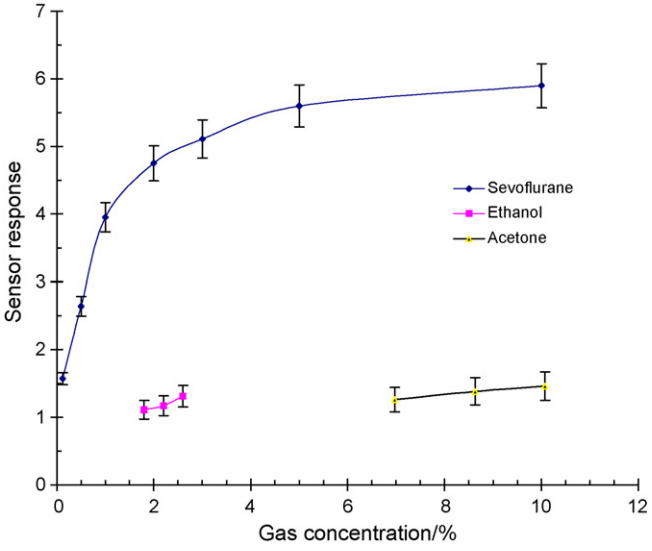

Fig. 10. Sensor response vs. gas concentration.

Table 1
Effect of interferences over sevoflurane

Selectivity (Se)[a]	Ratio of interference (RI)[b]	Gas concentration
4.04	0.256	Sevoflurane = 2% Ethanol = 2.2%
3.86	0.247	Sevoflurane = 10% Acetone = 10.1%

[a] Selectivity, Se = (response to sevoflurane)/(response to interference gas).
[b] Ratio of interference, RI = 1/Se.

Table 2
Parameters of gases adsorbed on the Ppy surface

Adsorbed gases	Calculated adsorbed energy (kcal/mol)	Recovery time (s)
Sevoflurane	−7.2	20
Ethanol	−11.3	45
Acetone	−14.1	60

culated on the Ppy surface. The data in Table 2 indicate that sevoflurane has the lowest bonding energy and the shortest recovery time. The larger the bonding energy, the more recovery time is needed. Larger bonding energy results in more stable adsorbed sevoflurane, which impedes desorption. Desorption is related to the recovery process. In Table 2, the data reveal that the adsorption bond energy of ethanol and acetone is larger than that of sevoflurane. This might arise from the fact that sevoflurane is a bulky molecule with a larger steric effect compare to ethanol and acetone. This effect reduces the adsorption bond energy of sevoflurane.

3.7. Response magnitude versus sevoflurane concentration

The sensor response versus sevoflurane concentration is shown in Fig. 10. Sensor responses were 1.57–5.9 with the concentration between 0.1% and 10%, and the relative standard deviations were calculated as 5.1–7.3%. This response is well suited to the measurement of anesthetic sevoflurane in clinical usage. It was shown the sensor response under high concentrations of sevoflurane (>5%) reached saturation.

4. Conclusion

A new type of resistance gas sensor was developed to detect sevoflurane. Polypyrrole, prepared using an in situ UV photopolymerization method, was used as the sensing material. Molecular dynamic calculations of sevoflurane proved the adsorption on polypyrrole, with the existence of a new hydrogen bond N–H–F. The bond length was also predicted to be in the range from 0.2 to 0.4 nm. The bond energy was calculated to be about 7.2 kcal/mol. The bonding energy was compared with those of various gases, the larger bonding energies needing the higher recovery times. The sensor response was 3.95 at the concentration of 1% while the response and recovery times were as short as 60 and 20 s, respectively. Sensor responses were 1.57–5.9 to the concentration from 0.1% to 10%, and the sensor responses to high concentrations of sevoflurane (>5%) reached saturation. Using the present sensor we have thus far studied volatile sevoflurane concentrations under ambient conditions. In the future, such sensors might be applied to monitoring the presence of anesthetic agents at low levels.

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