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Trimethylamine sensing properties of nano-SnO_2 prepared using microwave heating method

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Abstract

The precursor was obtained through the reaction between $SnCl_4 \cdot 5H_2O$ and NaOH in the presence of PEG400 (polyethylene glycol, $M = 400$). Tin oxide (SnO_2) nano-powders were prepared by heating the precursor with microwave method. SnO_2 thick film sensors were fabricated using SnO_2 nano-materials as sensing materials. The phase composition and morphology of the material particles were characterized through X-ray diffraction (XRD) and transmission electron microscopy (TEM), respectively. The average particle sizes of the samples obtained with 616 W microwave heating and 800 W microwave heating (20 min) are about 5 and 15 nm, respectively. The influence of the heating duration and heating power on the gas-sensing properties of sensors based on SnO_2 nano-materials were investigated. The sensitivities of the sensors based on SnO_2 nano-materials heated with 616 and 800 W for 20 min were higher than those of the sensors based on SnO_2 nano-materials heated with 136, 264 and 440 W for 20 min. When operating at 200–310 °C, the sensor based on SnO_2 heated with 616 W for 20 min exhibits highest sensitivities in all sensors based on SnO_2 heated with 616 W for different duration. The sensitivity to a few kinds of organic gases, such as $(CH_3)_3N$ and $(CH_3)_2CO$ were studied. It was found that the sensor based on SnO_2 nano-materials (with 616 W microwave heating for 20 min) exhibited good performance characterized by high sensitivity and short response time to dilute trimethylamine when operated at 255 °C. The sensitivity to 0.001 ppm $(CH_3)_3N$ at 255 °C was 3. The response time and recovery time were about 30 and 100 s, respectively.

Keywords: Microwave; SnO_2; Trimethylamine; Sensor

1. Introduction

An inspection of food freshness is very important in the field of food industries. Fish freshness has been evaluated chemically and expressed as *K*-value which is a useful index of raw-fish grade. However, the method needs complicated sensor system with several kinds of biochemical substances, reagents and biochemical processes [1]. Hence, a new method is needed at fish markets and restaurant kitchens. The concentration of trimethylamine (TMA) increases significantly during the deterioration process and TMA is an essential objective parameter for fish freshness. Although several methods to detect TMA have been developed [2–5], these approaches need complex and expensive instruments, making them neither portable nor field applicable.

Metal oxide semiconductor sensors are the most promising devices among the solid state chemical sensors, because they have many advantages such as small dimensions, low cost, low power consumption, on-line operation, and high compatibility with microelectronic processing. Several groups have reported TMA sensors based on metal-oxide array [6], TiO_2–Fe_2O_3 thin films [7], Th-doped SnO_2 thin film [8,9], Th-doped SnO_2 pellet [10], SnO_2–ZnO nano-composite [11]. Thorium dopant enhanced the sensitivity towards TMA, the tin oxide pellet containing 3 mass% of thoria exhibits a sensitivity of 1500 to 800 ppm of TMA compared to pure tin oxide whose sensitivity is only 18 at an operating temperature of 225 °C [10]. But the sensors based on Th-doped SnO_2 thin film [8,9], Th-doped SnO_2 pellet [10], and SnO_2–ZnO nano-composite [11] could not detect lower concentration TMA than 1 ppm.

The state and the amount of oxygen on the surface of materials are strongly dependent on the microstructure of the materials, namely, specific area, particle size, as well as the thickness of the sensing film. The gas-sensing properties were

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related to the synthesis method. Compared with the conventional heating, microwave heating has unique effects such as rapid heating, high reaction rate, short reaction time, enhanced reaction selectivity and energy saving [12–15]. Srivastava et al. [12] prepared nano-crystalline SnO_2 in solvent media using a microwave assisted technique and investigated the gas-sensing properties, the results showed that the new aqueous route was better in terms of sensitivity as well as response/recovery time. Liu et al. [13] prepared ZnO nanorods in the presence of PEG400 (polyethylene glycol, $M = 400$) by a microwave-assisted decomposing route and found that ultraviolet–vis absorption peak of ZnO nanorods showed a distinct blue shift from that of the bulk materials. In this paper, we have investigated the TMA-sensing properties of SnO_2 nanomaterials prepared through heating the precursor with microwave method, the results revealed that the sensor based on SnO_2 (heating with 616 W for 20 min) prepared by solid-state reaction and microwave method exhibited high sensitivity to dilute TMA gas. The sensitivity to 0.001 ppm TMA reached 3 when operated at 255 °C.

2. Experimental

All of the chemical reagents used in the experiment were of analytical grade. Microwave reaction was performed in a domestic microwave oven (Model-PJ23C-SC1, frequency 2.45 GHz, maximum power 800 W, multimode oven, made by Meidi Company). The samples were heated with five grades of heating power: 136, 264, 440, 616 and 800 W. The detailed synthesizing process was as follows: 0.003 mol $SnCl_4 \cdot 5H_2O$ was ground in a mortar for 5 min before it was mixed with 2.0 ml of PEG400 (polyethylene glycol, $M = 400$) by grinding 5 min, and then 0.012 mol NaOH was added. After 30 min of grinding, the mixture was washed with distilled water and alcohol to remove any by-product (NaCl and PEG400). After filtration, it was dried in air and the precursor was obtained. The alumina crucible (the caliber and the height of the crucible are 2 and 3 cm, respectively.) containing 0.5 g precursor were placed on the central section of the microwave oven plate, heated for different heating duration with different heating power. To avoid very high temperature, the samples were irradiated for 2 min with intervals of 1 min, namely, the samples were heated for 2 min and were heated again after 1 min interval. The phase composition of the particles was characterized by X-ray diffraction (XRD) with a Bruker D8 diffractometer using monochromatic CuKα radiation operated at 40 kV and 30 mA. TEM images were collected on a JEOL-2100 transmission electron microscope at 200 kV.

The paste prepared from a mixture of SnO_2 with a PVA (polyvinyl alcohol) solution was coated on an Al_2O_3 tube on which two gold leads had been installed at each end. The Al_2O_3 tube was about 8 mm in length, 2 mm in external diameter and 1.6 mm in internal diameter. A heater of Ni–Cr wire was inserted into the Al_2O_3 tube to heat the sensor to the operating temperature which could be controlled in the range of 80–500 °C. The electrical resistance of the sensor was measured in air and in test gases. The voltage on the sensor was measured by a high precision voltmeter and the data were recorded by a computer. The relative humidity in air and test gases was about 55%. The sensitivity (S) was defined as the ratio of the electrical resistance in air (Ra) to that in a test gas (Rg).

3. Results and discussion

Fig. 1 shows the XRD profiles of samples obtained by heating for 20 min with heating power 0, 616 and 800 W. The diffraction peaks are quite similar to those of bulk SnO_2, which can be indexed as the tetragonal rutile structure of SnO_2. This is in good agreement with the JCPDS file of SnO_2 (JCPDS 41-

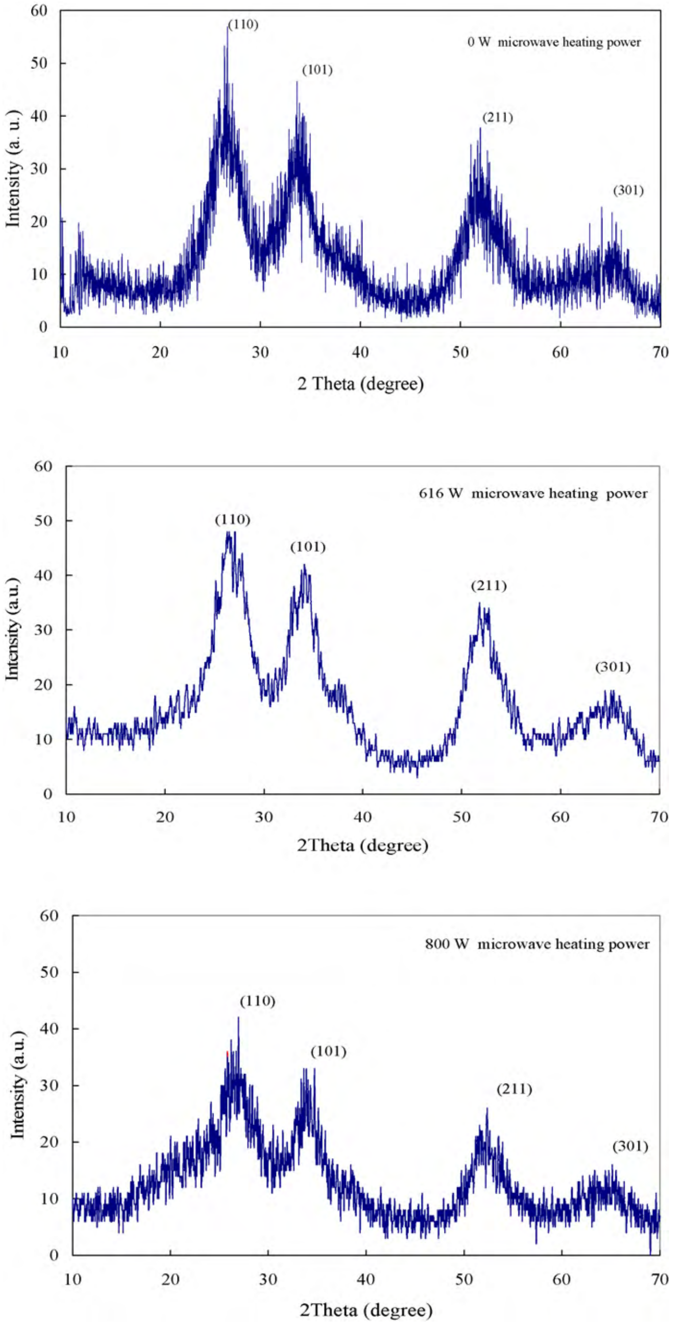
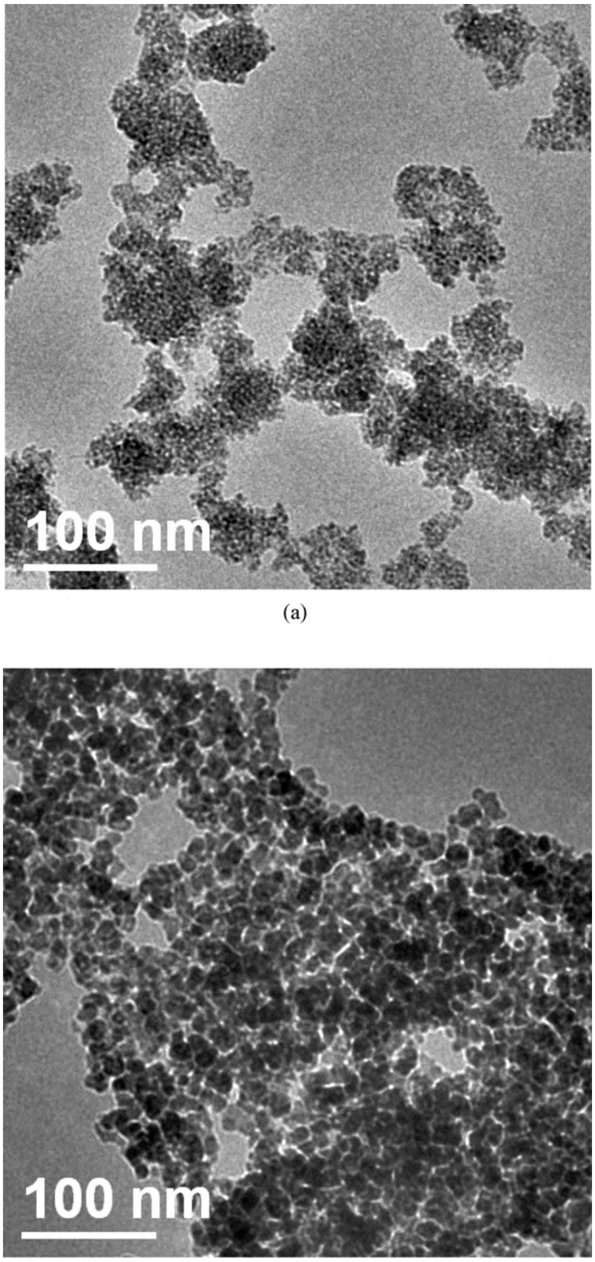


Fig. 1. The XRD profiles of samples obtained by microwave heating for 20 min with 0, 616 heating power and 800 W heating power.



(a)

(b)

Fig. 2. The transmission electron microscopy micrographs of SnO_2 samples heated by microwave for 20 min with (a) 616 W heating power and (b) 800 W heating power.

1445), that manifests pure SnO_2 can be obtained even if the precursor is not heated with microwave. No additional peaks were observed, indicating the high purity of the final products. However, the peaks were relatively broad, corroborating the very small crystal size. There is no obvious difference among the half peak widths in the XRD patterns of the three samples, the phenomenon certifies that the difference among the average crystalline sizes in the three samples is slight. The XRD profiles of the samples heated for 20 min with 136, 264, and 440 W are similar to those shown in Fig. 1 in terms of the peak positions and the half peak widths. According to Scherrer formula, the crystallite sizes of SnO_2 obtained with 0, 136, 264, 440, 616 and 800 W (20 min) are 5, 5, 5, 5, 5, and 5 nm, respectively. The intensities of the peaks in XRD patterns of the samples heated with 0 and 616 W microwave power are higher than those of the sample heated with 800 W microwave power, indicating that microwave heating cannot promote crystallization and high microwave power suppress crystallization. The real reason why high microwave power suppress crystallization is under investigation. Fig. 2 shows the transmission electron microscopy micrographs of SnO_2 heated for 20 min with 616 and 800 W. It could be seen from the micrographs that the average particle sizes of the samples obtained with 616 and 800 W (20 min) are about 5 and 15 nm, respectively. Comparing with the results of XRD profiles, there is almost no agglomerates in the sample obtained with 616 W, the big particles in the sample obtained with 800 W may be composed of smaller particles. These results indicate that the microwave power has little influence on the crystallite sizes of the samples and high power may result in the agglomeration of small crystallites. In the literature [12], SnO_2 samples were prepared in solvent media using a microwave assisted technique, the crystallite sizes were about 10 nm. Krishnakumar et al. [16] prepared SnO_2 precursor in solution and irradiated the precursors in microwave oven, the crystallite sizes also were about 10 nm. The solvent media may result in the growth of crystals of precursors because mass transfer proceeds easily in liquid phase. In our experiment, the precursor is SnO_2 which forms in the mortar according to the reaction: $Sn^{4+} + 4OH^- = SnO_2 + 2H_2O$; the precursor are prepared through the reaction between $SnCl_4{\cdot}5H_2O$ and NaOH in the presence of PEG400; because $SnCl_4{\cdot}5H_2O$ and NaOH cannot dissolve in PEG400, $SnCl_4{\cdot}5H_2O$ and NaOH are surrounded by PEG400 and PEG400 can restrain reaction between $SnCl_4{\cdot}5H_2O$ and NaOH; the precursor can form when $SnCl_4{\cdot}5H_2O$ contact NaOH in the process of grinding, and the precursor particles grow slowly; microwave heating process terminates in a short time, SnO_2 crystallite sizes augment slightly. The solid-state reaction between $SnCl_4{\cdot}5H_2O$ and NaOH, the presence of PEG400 and the microwave heating method may act together toward the small primary particle sizes of SnO_2 in our experiment. The particle sizes of SnO_2 heated for 20 min with 616 W are also smaller than those in SnO_2 gas sensors fabricated by spray pyrolysis [17].

SnO_2 is an n-type semiconductor with excess metal ions or oxygen vacancies acting as electron donors. Molecular oxygen acts as an acceptor in air. They deplete electrons from the conduction band and become chemisorbed oxygen on the surface and on grain boundaries in the form of O_2^-, O^-, or O^{2-} at different temperatures [18]. The equations are as follows:

$$O_2\,(\text{adsorbed}) + e^- = O_2^-\;(<100\,^\circ\text{C}) \tag{1}$$

$$O_2^- + e^- = 2O^-\;(100\text{–}300\,^\circ\text{C}) \tag{2}$$

$$O^- + e^- = O^{2-}\;(>300\,^\circ\text{C}) \tag{3}$$

When the operating temperature increases, O_2 (adsorbed) capture the electrons from the conduction band of the sensor.

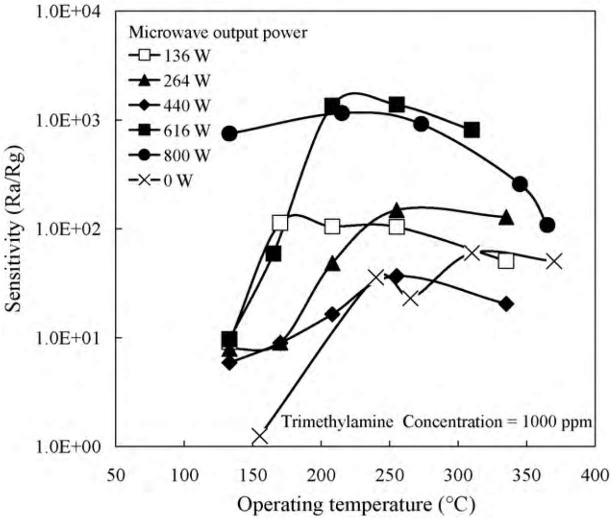


Fig. 3. The effect of microwave heating power (20 min) on the sensitivities of SnO_2 sensors to 1000 ppm trimethylamine gas.

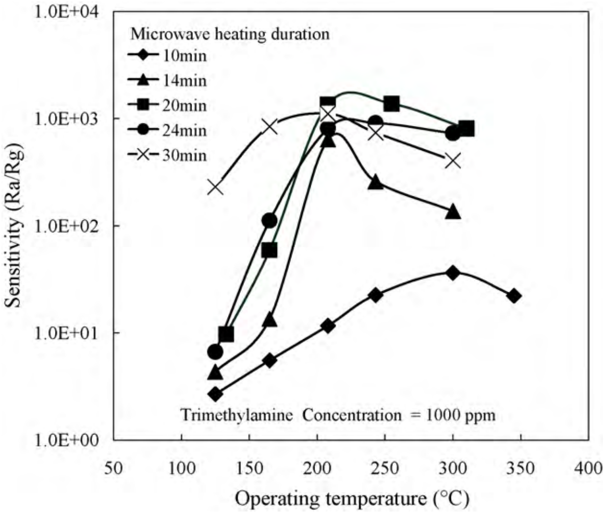


Fig. 4. The effect of microwave heating duration (616 W heating power) on the sensitivities of SnO_2 sensors.

The reaction of the reducing gas with oxygen on the surface of SnO_2 makes the electrons trapped by the adsorbed oxygen go back to the conduction band of SnO_2, leading to a decrease in sensor resistance. The possible reaction between trimethylamine and O_2^- is:

$$4N(CH_3)_3 + 21O_2^- = 2N_2 + 18H_2O + 12CO_2 + 21e^- \qquad (4)$$

Fig. 3 manifests the effect of heating power (20 min) on the sensitivities of SnO_2 sensors to 1000 ppm trimethylamine gas in the operating temperature range of 130–370 °C. The sensor based on SnO_2 that is not heated with microwave shows low sensitivity to trimethylamine gas, the maximum sensitivity to 1000 ppm trimethylamine gas is 60; Although the maximum sensitivities of SnO_2 sensors obtained with different heating power appear at different operating temperatures, it is obvious that the sensors based on SnO_2 heated 136, 264 and 440 W exhibit low sensitivities to trimethylamine. For example, the maximum sensitivities of the three sensors are lower than 150 when operating in the temperature range of 130–370 °C. The sensors based on SnO_2 heated with 616 and 800 W exhibit high sensitivities to trimethylamine. The maximum sensitivity of the sensor based on SnO_2 heated with 616 W appears when operating at 255 °C and reaches 1374. The maximum sensitivity of the sensor based on SnO_2 heated with 800 W emerges when operating at 215 °C and attains 1155. Fig. 4 shows the effect of heating duration (616 W) on the sensitivities of SnO_2 sensors. The heating duration has a great influence on the sensitivities of sensors. When operating at 120–200 °C, the sensitivities to trimethylamine increase with increasing in the heating duration. But when operating at 200–310 °C, the sensor based on SnO_2 heated for 20 min exhibits highest sensitivities, the sensitivities are higher than 1354 when operating at 208 and 255 °C. In general, the particle sizes increase with increase of the heating temperature and the heating duration, but the sensitivities change in reverse order [19,20]. In this study, the crystallite sizes of SnO_2 samples heated for 20 min with 136, 264, 440, 616 and 800 W are 3, 3, 5, 5 and 5 nm, respectively. There are obvious sensitivity differences even if the average crystallite sizes of samples are almost same, indicating that the sensitivity differences are not mainly conduced by the crystallite size disparity. The active sites on the surface of a gas-sensing material are very important for the same material, the active sites can adsorb test gas and oxygen. The more the number of the active sites on the surface of gas-sensing materials, the higher the sensitivity is. We cannot ascertain the exact reason why the sensor based on SnO_2 heated for 20 min with 616 W shows high sensitivity at present, appropriate heating power and proper heating duration may increase the active sites on the surface of particles that could adsorb the reducing gas and oxygen.

The sensitivities to a few kinds of gases of sensor based on SnO_2 (616 W, 20 min) are shown in Fig. 5. The sensitivities to 1000 ppm benzene and 1000 ppm toluene are all lower than 10

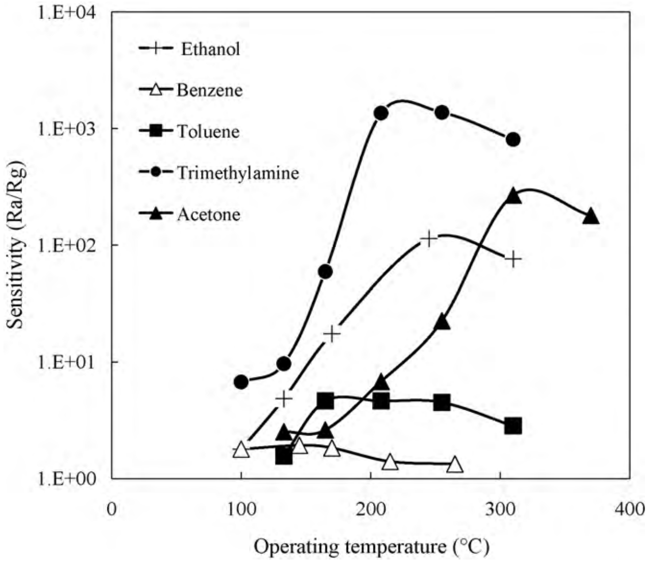


Fig. 5. The sensitivities to a few kinds of gases of sensor based on SnO_2 (616 W, 20 min).

when operating in the temperature range of 100–310 °C. The maximum sensitivity (113) to 1000 ppm ethanol is observed while operating at 245 °C. The maximum sensitivity (269) to 1000 ppm acetone is observed while operating at 310 °C. But the sensitivities to 1000 ppm trimethylamine are very high while operating in the temperature range of 100–310 °C, the maximum sensitivity appears at 255 °C, the sensitivities at 208 and 255 °C are 1354 and 1374, respectively. Hence, the sensor is established to have good selectivity to trimethylamine. The sensitivity is also related to the reducing ability and the adsorbing ability of detected gas on surface of the material. CH_3-is electron-donating group [21], the electron cloud density around N atom in $(CH_3)_3N$ is higher than those of O atom in ethanol and acetone, the attractive force between N atom in $(CH_3)_3N$ and Sn^{4+} on the surface of SnO_2 can facilitate the adsorption of $(CH_3)_3N$ on the surface of material. The bond energy has great influence on the stability of compound, the lower the bond energy, the easier the bond breaks. The bond strengths of C–H, C–C, C–N, C=O, O–H are 411, 345, 307, 748.2, 462 kJ/mol [22], the bond energy of C–N in $(CH_3)_3N$ is low, $(CH_3)_3N$ is unstable in terms of bond energy. The reducing ability of $(CH_3)_3N$ is higher than those of C_2H_5OH, CH_3COCH_3, benzene and toluene, leading to the highest sensitivity to $(CH_3)_3N$ in the all gases detected. The benzene ring in benzene and toluene is stable, resulting in the low sensitivities of the sensor to benzene and toluene. When operating at 208–255 °C, the sensitivity to $(CH_3)_3N$ is greatly higher than those to other gases, the sensor based on SnO_2 (616 W, 20 min) exhibits high selectivity to $(CH_3)_3N$. The selectivity $S_{1000\ \mathrm{ppm}\ (CH3)3N}/S_{1000\ \mathrm{ppm\ ethanol}}$ at 255 °C is about 12. When the sensor is utilized to detect the freshness of fish, there is almost no alcohol or acetone gas to disturb the measurement. Hence, the sensor based on SnO_2 (616 W, 20 min) may be used to measure $(CH_3)_3N$ emitted from the fish tissue.

Fig. 6 shows the correlation between the trimethylamine concentration and the sensitivity of the sensor based on SnO_2 (616 W, 20 min) operating at 255 °C. Although the sensitivity decreases with decrease of the trimethylamine concentration, the curve of sensitivity versus gas concentration was not linear. The sensitivity to 0.001 ppm trimethylamine was 3, while the sensitivities to 0.1, 1 and 10 ppm were only 12, 13 and 14, respectively. The curve of sensitivity vs. concentration is almost flat in the range of 0.1–10 ppm. Dopant should be added in the material to enhance the sensitivities to 1 and 10 ppm trimethylamine.

In general, the response time and recovery time are defined as the times for a sensor to reach 90% of the final signal. The response transients of the sensor based on SnO_2 (616 W, 20 min) to 1 ppm trimethylamine at 255 °C are shown in Fig. 7. The response time and recovery time were about 30 s and 100 s, respectively. The reproducibility and the stability of the sensors were studied. Three sensors were fabricated from each kind of SnO_2 sample. The three identical devices showed similar sensitivity to the detected gases although the resistance difference was higher than the sensitivity discrepancy, the deviation amplitude of the sensitivity value to the detected gases were smaller than 10%. The long-term stability was not so good, the sensitivities changed little in 4 days and decreased obvious after placing in air for 4 days.

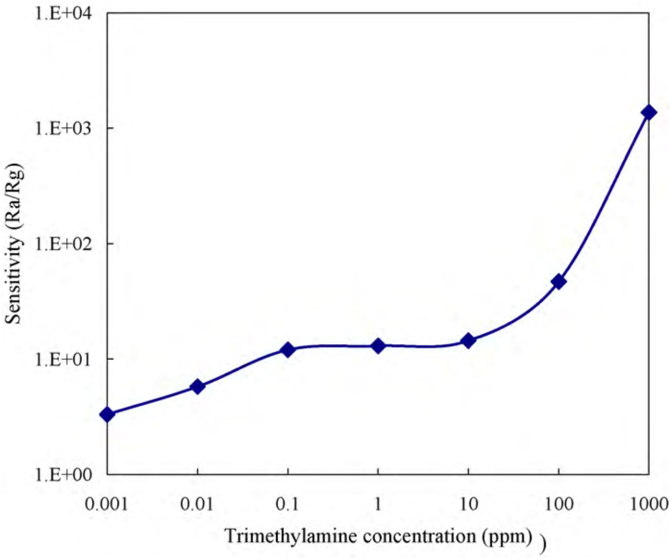


Fig. 6. The correlation between the trimethylamine concentration and the sensitivity of the sensor based on SnO_2 (616 W, 20 min) at 255 °C.

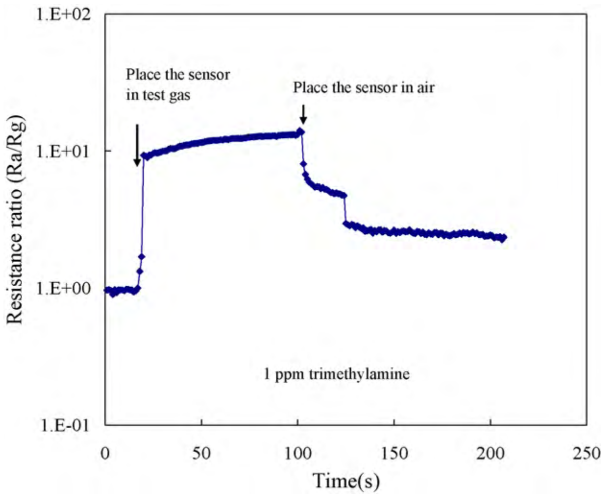


Fig. 7. The response transients of the sensor based on SnO_2 (616 W, 20 min) to 1 ppm trimethylamine at 255 °C.

4. Conclusions

In summary, SnO_2 nano-materials were prepared by heating the precursor with microwave heating method. Although the microwave heating conditions have little effect on the crystallite sizes of the SnO_2, the heating duration and heating power had influence on the gas-sensing properties. The sensitivities of the sensors based on SnO_2 nano-materials heated with 616 and 800 W for 20 min were higher than those of the sensors based on SnO_2 nano-materials heated with 136, 264 and 440 W for 20 min. When operating at 200–310 °C, the sensor based on SnO_2 heated with 616 W for 20 min exhibits highest sensitivities in all sensors based on SnO_2 heated with 616 W for different duration. The sensor based on SnO_2 (616 W, 20 min) exhibited good sensitivity and good selectivity to trimethylamine gas. The sensitivities to low concentrations of trimethylamine were very high, especially the sensitivity to 0.001 ppm trimethylamine was 3 while operating at 255 °C. But the sensitivities to 1 and 10 ppm trimethylamine are not so high, some dopant should be added in the material to improve the sensitivity in the concentration range of 1–10 ppm. SnO_2 (616 W, 20 min) nano-material is a promising candidate for detecting low concentration of trimethylamine gas.

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