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High sensitivity in Al-doped ZnO nanoparticles for detection of acetaldehyde

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ABSTRACT

The gas sensing properties of Al-doped ZnO nanoparticles (NPs), which were synthesized via a hydrothermal method, have been described for the detection of volatile organic compounds (VOCs) such as acetaldehyde, toluene, and benzene. The maximum sensing response (R = 2250) of these Al-doped ZnO NPs was observed upon exposure to 10 ppm acetaldehyde at an optimal operating temperature of 500 °C, which was almost 173 and 125 times higher than that observed for toluene and benzene, respectively. The highest dipole moment of acetaldehyde among the three VOCs is responsible for its highest sensitivity. The Al-doped ZnO NPs also show a higher selectivity toward acetaldehyde than that of different interfering gases and their response to acetaldehyde was superior compared with the previously reported responses of other materials. The enhanced sensing performance of Al-doped ZnO NPs to acetaldehyde compared to undoped ones may be explained by an increase in specific surface area, oxygen vacancies, and conductivity after Al doping.

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1. Introduction

Each day in modern life, large quantities of volatile organic compounds (VOCs) are released into the atmosphere from both anthropogenic and natural sources [1,2]. The presence of these VOCs in the atmosphere is concerning as they are environmental pollutants and their inhalation poses severe health risks [2,3]. Thus, the detection of VOCs while monitoring the environmental pollution and identification of various diseases caused by these pollutants is of utmost importance. Therefore, it is necessary to develop highly efficient gas sensors for accurate detection and classification of VOCs. Different technologies such as gas-chromatography (GC) and mass spectrometry (MS) have been adapted for detecting VOCs owing to their high selectivity; however, these techniques involve complicated sampling and analysis procedures, and suffer from low sensitivities and long detection times [4,5].

In order to address these issues, gas sensors based on metal oxide semiconductors have been investigated for the detection of VOCs because of their high sensitivity, easy fabrication, and high chemical stability. A number of metal oxide semiconducting materials, including WO_3 [6], SnO_3 [7], In_2O_3 [8], TiO_2 [9], and ZnO [10], show good results as gas sensors. Among these, ZnO is well known for its wide band gap of 3.37 eV and has been extensively used as a gas sensing material owing to its high mobility of conduction electrons and excellent chemical and thermal stability under the operating conditions of sensors [11,12]. In addition, it has been reported that ZnO is highly sensitive toward the detection of flammable or toxic gases [13,14]. In order to improve the performance of ZnO-based gas sensors, two strategies have been adopted. One is fabricating sensors by controlling their structures and morphologies, such as in nanoparticles (NPs) [15], nanowires [16], nanorods [17], and nanosheets [18]. Nanostructured metal oxide semiconductors are promising candidate materials in VOC sensors because these structures have large surface-to-volume ratios. Furthermore, they possess several advantageous attributes such as improved gas diffusion and electrical properties. The second strategy involves the use of metal dopants such as Al [19], Cd [20], Co [21], Cu [22], and Ag [23]. The sensing properties of doped ZnO can be modulated by changing the particle morphology, crystal structure, energy band structure, and number of surface sites available for gas interaction [24]. The sensing properties of Al-doped ZnO NPs have been recently investigated; these materials show a high response and sensitivity for the detection of dimethyl methylphosphonate (DMMP) [25] and 2-chloroethyl ethyl sulfide (2-CEES) [26] as simulants of chemical warfare agents. Thus, in this work, the sensing performance of Al-doped ZnO NPs was investigated for the detection of VOCs such as acetaldehyde, toluene, and benzene. These three VOCs are the most frequently encountered compounds

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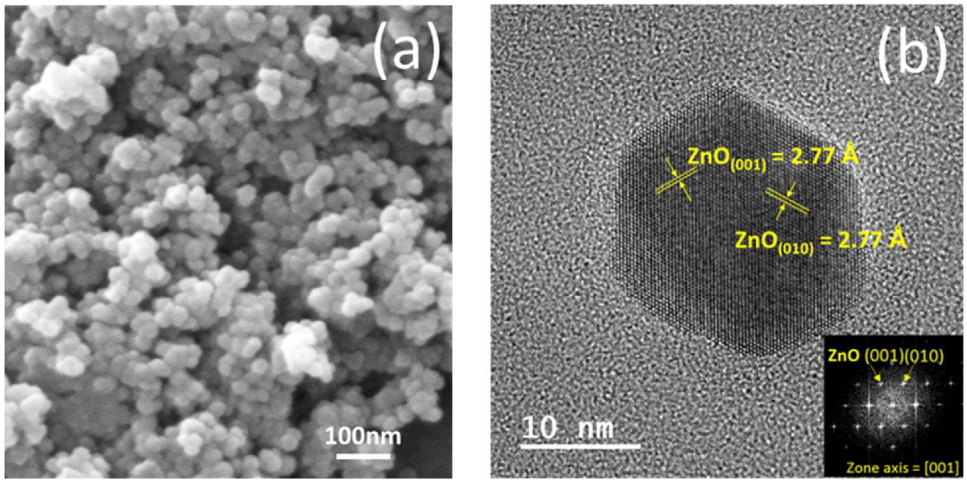

Fig. 1. (a) SEM image and (b) high-resolution TEM image with the electron diffraction pattern of Al-doped ZnO NPs.

in human life that cause the sick house syndrome and health problems such as allergies or cancers.

The Al-doped ZnO NPs are more sensitive to acetaldehyde than to benzene, toluene, and other interfering gases such as NH_3, CO, NO, NO_2, and C_3H_9N (trimethylamine). The sensing mechanism of Al-doped ZnO NPs has been correlated with the dipole moment of each VOC.

2. Experimental

Al-doped ZnO NPs were synthesized by a hydrothermal method. Aluminum acetate was used as the Al source to add to the zinc precursor solution under stirring. The doping concentration of Al was controlled to 1 at% (0.065 g of aluminum acetate for 1 at% of Al). The synthetic procedure has been previously described in detail [25,26]. In order to fabricate a sensor device based on the Al-doped ZnO NPs, the annealed product was mixed with α-terpineol paste and dispersed onto the interdigitated Pt electrodes by using a dropping method. Subsequently, to remove the α-terpineol paste and enhance the stability of the NPs, the NPs was dried at 300 °C for 1 h and annealed at 600 °C for another hour.

The shape and morphology of the as-synthesized Al-doped NPs were probed by a field-emission scanning electron microscopy (FE-SEM; JEOL 7001F). Its microstructural properties were characterized by transmission electron microscopy (TEM, JEOL JEM ARM 200F). The gas sensing measurements were conducted by using the gas detecting system, as described in a previous work [25–28]. The target gases including acetaldehyde, toluene, and benzene were used as synthetic air. A gas concentration of 0.1–10 ppm was achieved by controlling the partial pressure by means of mass flow controllers. All gas-sensing measurements were carried out at an operating temperature of 300–550 °C. The sensing response for the VOCs is defined as $(R_a - R_g)/R_g$, where R_a and R_g are the electrical resistances of the undoped and Al-doped ZnO NPs in air and in the gas mixture of pure air and target gases, respectively. The response time is defined as the time required to attain 90% of the total electrical resistance change upon exposure to the test gas.

3. Results and discussion

The SEM image of the Al-doped ZnO NPs after annealing on the Pt electrodes (Fig. 1(a)) shows a uniform distribution of the spherical particles on the electrode surface. Fig. 1(b) shows the high-resolution TEM image of a single Al-doped ZnO NP with a diameter of ~20 nm. The lattice spacing of ~0.28 nm can be indexed to the (001) and (010) planes of the hexagonal wurtzite structure of ZnO. The FFT (fast Fourier transformation) pattern along the [001] zone axis in the inset of Fig. 1(b) represents the single crystallinity of the Al-doped ZnO NPs. The doping of Al into the ZnO NPs was confirmed by X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) studies (data not shown here). Further characterization details are described in a previous work [26].

In order to assess the sensing properties of the Al-doped ZnO NPs, tests for 10 ppm of acetaldehyde, toluene, and benzene were performed at different operating temperatures. Fig. 2 shows the variation in the sensing responses of the Al-doped ZnO NPs in the temperature range of 300–500 °C. The sensing responses for each gas are strongly dependent on the operating temperature. At 500 °C, the sensing response of the Al-doped ZnO NPs was 13 and 18 for toluene and benzene, respectively, while that for acetaldehyde significantly increased to a value of 2250. This enhancement is almost 173 and 125 times compared to that of the toluene and benzene, respectively, and is the highest response to acetaldehyde ever reported [18,31–36]. After the reaction of VOCs with oxygen species on the surface of Al-doped ZnO NPs, the number of electrons released from benzene or toluene is more than that obtained from acetaldehyde, according to Eqs. (1)–(3). However, the Al-doped ZnO NPs are very sensitive to acetaldehyde compared to benzene and toluene. This suggests that the increased sensitivity may be related to the dipole moment of these molecules in their gaseous state; the dipole moments of acetaldehyde, toluene, and benzene are 2.7, 0.36, and 0 D, respectively. Therefore, the number of molecules of acetaldehyde that adsorb on Al-doped ZnO NPs is more than that of benzene or toluene and consequently, the response of this Al-doped ZnO NPs to acetaldehyde is the highest.

$$2CH_3CHO_{(gas)} + 3O^{2-} \xrightarrow{ZnO} 2CO_2 + 4H_2O + 3e^- \quad (1)$$

$$2C_6H_{6(gas)} + 15O^{2-} \xrightarrow{ZnO} 12CO_2 + 6H_2O + 15e^- \quad (2)$$

$$C_6H_5CH_{3(gas)} + 9O^{2-} \xrightarrow{ZnO} 7CO_2 + 4H_2O + 9e^- \quad (3)$$

Fig. 3 shows the responses and the response times of the Al-doped ZnO NPs to acetaldehyde, toluene, and benzene at their different concentrations in the operating temperature range of 300–550 °C. Interestingly, the sensing responses for acetaldehyde, toluene, and benzene increased gradually with increasing operating temperature, as shown in Fig. 3(a). The sensing response of the Al-doped ZnO NPs to acetaldehyde decreased significantly at 550 °C after the highest response of 2250 was achieved at the optimal temperature of 500 °C (Fig. 3(a)). The SEM images exhibited no

Table 1
Summary of sensing properties of various metal oxide-based sensors used for the detection of acetaldehyde.

Material	ZnO nanosheet [18]	TiO_2 thin film [31]	Sm_2O_3-doped SnO_2 nanoparticles [32]	ZnO tetrapod [33]	Lamella-like ZnO [34]	Co doped ZnO thin film [35]	Hexagonal grid-like ZnO lamella structure [36]	Al-doped ZnO nanoparticles (this work)
Response	0.75 (0.1 ppm)	3129 (100 ppm)	1333 (1000 ppm)	50 (50 ppm)	114 (100 ppm)	16.4 (75 ppm)	800 (10 ppm)	2250 (10 ppm)
Response Temperature	220 °C	Room temperature	250 °C	400 °C	100 °C	Room temperature	Room temperature	500 °C
Sensitivity	7.5/ppm	3.1/ppm	2.5/ppm	1/ppm	1.1/ppm	0.22/ppm	8/ppm	225/ppm
Response time	8 s	50 s	–	100 s	12 s	4 s	280 s	2 s
Lowest detection limit	0.05 ppm	5 ppm	50 ppm	0.05 ppm	1 ppm	10 ppm	4 ppm	0.1 ppm

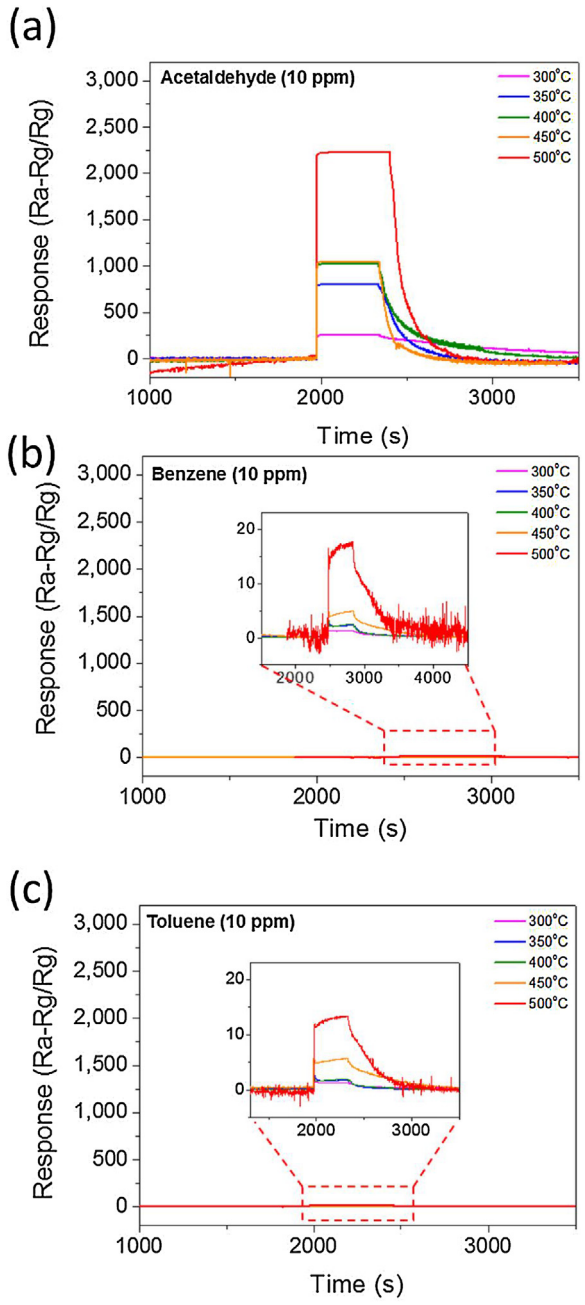

Fig. 2. Change in the sensing response of the Al-doped ZnO NPs in the range of 300–500 °C upon exposure to 10 ppm of (a) acetaldehyde, (b) benzene (c) toluene.

morphological difference in the Al-doped ZnO NPs after the measurement at temperatures up to 550 °C. The decrease in the sensing response at 550 °C may be attributed to the decreased electrical resistance of the 1.0 at% Al-doped ZnO NPs in air with increasing temperature (from 300 to 550 °C, as shown in Fig. 3(b)). The decrease in electrical resistance is a result of two factors, namely, the intrinsic properties of the semiconducting metal oxide and the amount of oxygen adsorption on the surface of the Al-doped ZnO NPs. The initial increase in the response of the Al-doped ZnO NPs at the temperature up to 500 °C can be explained by the increased conductivity and oxygen vacancies of ZnO NPs. However, as the temperature increased further (to 550 °C), the competing desorption of the chemisorbed oxygen on the surface of Al-doped ZnO NPs also needs to be considered [29]. That is, the temperature dependence of the sensing response results from the differing stabilities

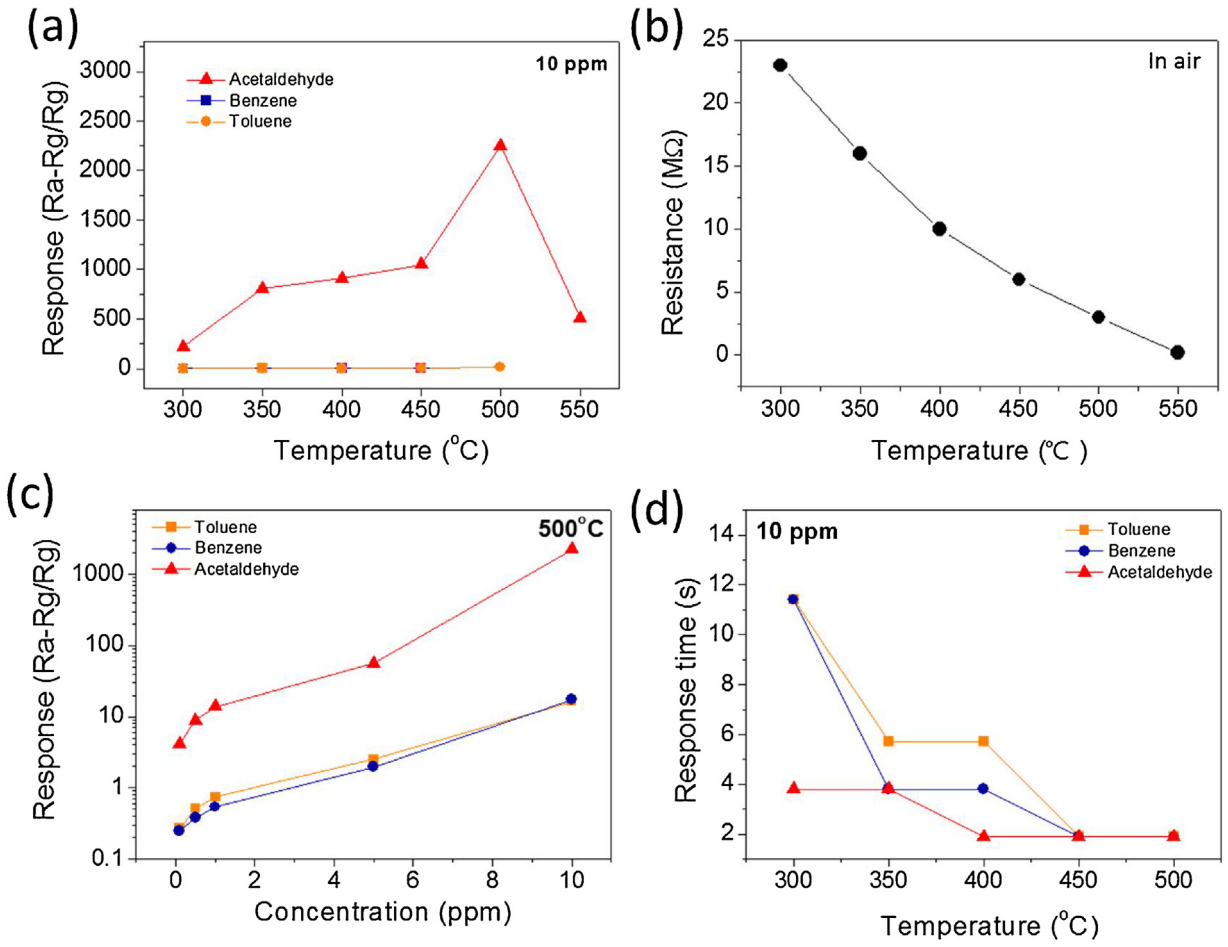

Fig. 3. (a) Sensing response of the Al-doped ZnO NPs upon exposure to 10 ppm of acetaldehyde, toluene, and benzene. (b) Resistance of the Al-doped ZnO NPs in air in the temperature range of 300–550 °C. (c) Sensing response of Al-doped ZnO NPs to VOCs concentrations in the range of 0.1–10 ppm. (d) Response time of Al-doped ZnO NPs toward 10 ppm of different VOCs (acetaldehyde, toluene, and benzene) at 500 °C.

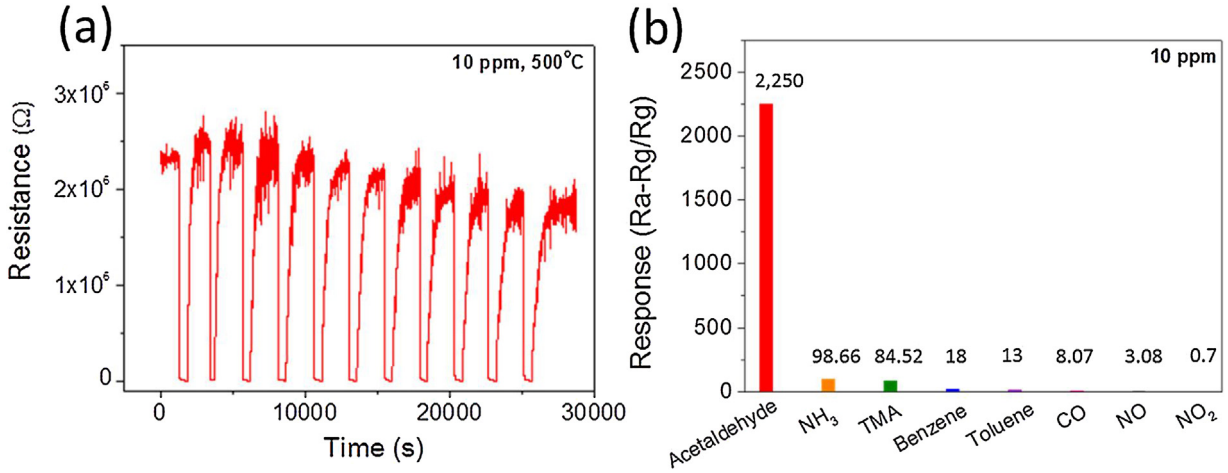

Fig. 4. (a) Reproducibility of the Al-doped ZnO NPs for detection of 10 ppm acetaldehyde at 500 °C for over 10 cycles and (b) selectivity of the Al-doped ZnO NPs for various target gases.

of the surface oxygen species over different temperature ranges despite the increase in conductance with increasing temperature [30]. Thus, the sensing response decreased at temperatures higher than 500 °C because of a reduction in the adsorbed oxygen species available at the sensing sites on the surface for reaction [37].

The response of the Al-doped ZnO NPs to VOC concentrations in the range of 0.1–10 ppm at 500 °C was also recorded (Fig. 3(c)). All the responses were found to increase with increasing VOC concentration. From these results, the dependence of the response time of the Al-doped ZnO NPs on the operating temperature was determined for the concentration of 10 ppm, as summarized in Fig. 4(d). The response time of the Al-doped ZnO NPs to each VOC gas decreased with increasing temperature and was 2 s at 500 °C for all tested gases. In the case of 10 ppm of acetaldehyde, the short

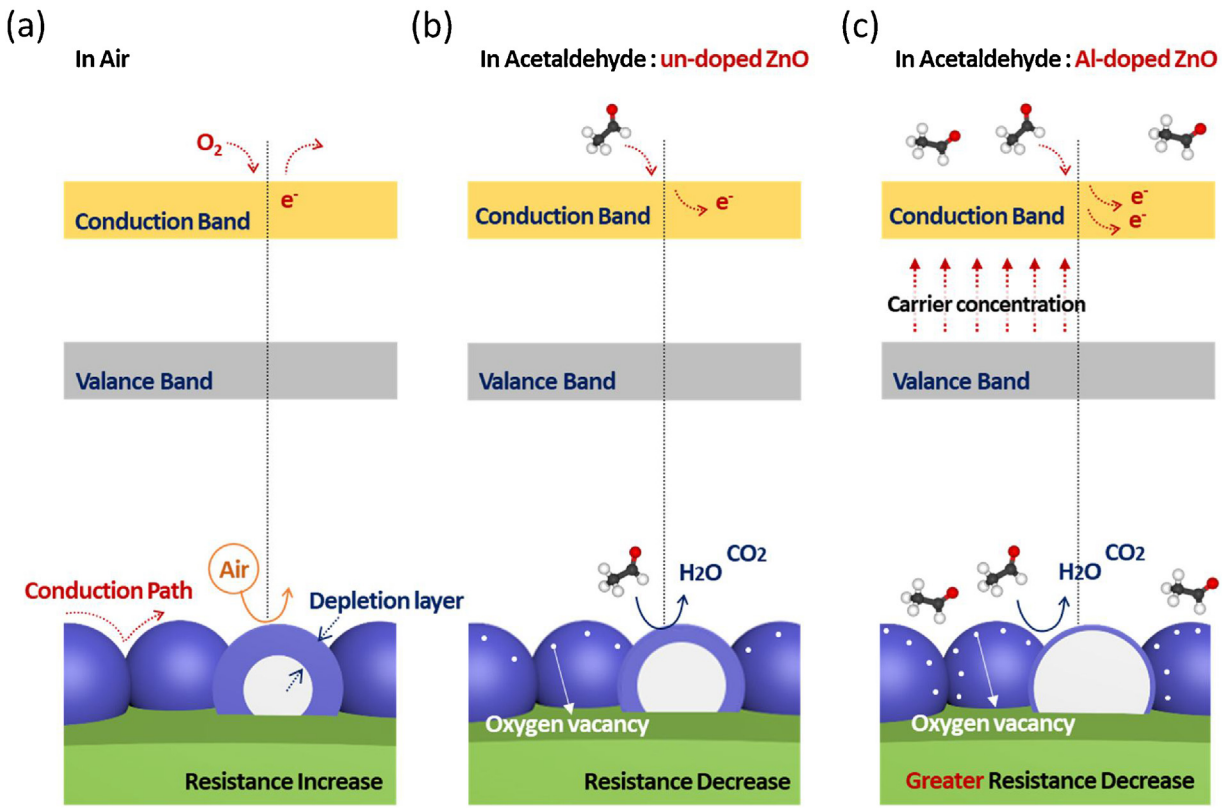

Fig. 5. A schematic of the sensing mechanism of the (a) un-doped or Al-doped ZnO NPs in air, (b) un-doped ZnO NPs in acetaldehyde, and (c) Al-doped ZnO NPs in acetaldehyde.

response time of 2 s appeared to reach saturation between 400 and 500 °C.

Fig. 4(a) and (b) presents the reproducibility of the Al-doped ZnO NPs for sensing 10 ppm of acetaldehyde over ten cycles at 500 °C and the response of the Al-doped ZnO NPs for 10 ppm of various target gases at 500 °C, respectively. As shown in Fig. 4(a), the response was almost constant for ten cycles of gas in/out. This implies a good reproducibility of sensing ability of Al-doped ZnO NPs for 10 ppm of acetaldehyde. It is evident from Fig. 4(b) that the Al-doped ZnO NPs have the highest response and selectivity for acetaldehyde compared with other target gases such as benzene, toluene, NH_3, CO, NO, NO_2, TMA (trimethylamine). This is attributable to significantly larger dipole moment in the molecular structure of acetaldehyde (2.7 D) than that in NH3 (1.47 D), TMA (0.61 D), benzene (0 D), toluene (0.31 D), CO (0.122 D), NO (0.16 D), NO2 (0.32 D). There is a weak attractive force such as Van der Waals force giving rise to interaction between a gas molecule and the Al-doped ZnO NPs. The dipole moment of a gas molecule affects the attractive force between the gas molecule and Al-doped ZnO NPs. Therefore, higher sensing response can be attributed to higher dipole moment of a gas molecule. The sensing performance for the detection of acetaldehyde by Al-doped ZnO NPs has been compared with that of previously reported metal oxide-based gas sensors in Table 1. The sensing response of Al-doped ZnO NPs is much higher than that of the other materials [18,31–36] (see Table 1).

From the above results, a possible mechanism for Al-doped ZnO NPs in the detection of acetaldehyde can be illustrated as shown in Fig. 5. The electrical resistance of the Al-doped ZnO NPs changes when gas molecules adsorb/desorb on the surface of the Al-doped ZnO NPs. When Al-doped ZnO NPs are exposed to air (see Fig. 5(a)), electrons in the conduction band are trapped by the chemisorbed oxygen molecules in air on the surface of the un-doped or Al-doped ZnO NPs, resulting in adsorbed surface oxygen species (O_2^-) (refer to Eq. (4)). This leads to the formation of a thick depletion layer on the surface of ZnO NPs, which increases the electrical resistance in the undoped and Al-doped ZnO NPs. When the undoped ZnO NPs are exposed to acetaldehyde (see Fig. 5(b)), a charge exchange occurs due to the interaction of acetaldehyde and the adsorbed surface oxygen species, giving rise to the oxidation of acetaldehyde and the release of electrons into the conduction band. Accordingly, the electrical resistance of the ZnO NPs decreases with decreasing in the thickness of depletion layer (Eq. (1)) [34].

$$O_{2\,(\text{gas ads})} + e^- \leftrightarrow O^{2-}{}_{(\text{ads})} \quad (4)$$

In order to confirm the sensing mechanism for undoped ZnO NPs in exposure to acetaldehyde, we carried out sensing measurements for undoped ZnO NPs to 10 ppm of acetaldehyde, benzene, and toluene at 500 °C. The sensing response of the undoped ZnO NPs was found to be $R = 135$, $R = 13$, and $R = 2$ for acetaldehyde, benzene, and toluene respectively (not shown), which are much smaller responses than that in the Al-doped ZnO NPs ($R = 2250$, $R = 18$, $R = 13$, see Fig. 2).

We found that the response of the Al-doped ZnO NPs is 16 times higher than that in undoped ZnO NPs in exposure to acetaldehyde. Recently, we investigated the physical and chemical characteristics, such as oxygen vacancies, optical band gap, and specific surface area, of undoped and Al-doped ZnO NPs to demonstrate the significant influence of Al dopant on the enhancement of sensing properties. Firstly, according to x-ray photoelectron spectroscopy (XPS) analysis [26], the amount of oxygen vacancies in the lattice of ZnO was found to increase after Al doping. The oxygen vacancies act as adsorption sites for acetaldehyde on the surface of the ZnO NPs. Secondly, the optical band gap of the pure ZnO NPs is 3.02 eV, whereas the optical band gap of their Al-doped counterparts increased to 3.13 eV. The increase of optical band gap is attributed to the shift in the Fermi level towards the conduc-

tion band, caused by additional carriers from donor Al atoms [40]. Increase of carrier concentration give rise to increase of ionized oxygen ions on the surfaces of ZnO which can react with acetaldehyde. Thirdly, the specific surface areas of the undoped and Al-doped ZnO NPs are ~35 and ~42 m^2/g, respectively. The surface areas of the Al-doped ZnO NPs are larger than those of the undoped ZnO NPs due to volume reduction. Therefore, remarkably enhanced sensing response is attributed to the chemical and electronic synergistic effects originating from oxygen vacancies, optical band gap, and specific surface area, giving rise to a greater number of adsorbed oxygen ions on the surfaces of ZnO that can react with acetaldehyde.

4. Conclusions

In this work, the effect of Al dopant on the gas sensing properties of ZnO NPs was investigated. These NPs were synthesized by a hydrothermal method and used for the detection of VOCs such as acetaldehyde, toluene, and benzene. The Al-doped ZnO NPs showed a maximum sensing response of R = 2250 and a short response time of ~2 s for the detection of 10 ppm acetaldehyde at an optimal operating temperature of 500 °C. The Al-doped ZnO NPs exhibited a high selectivity toward acetaldehyde compared to other VOCs (toluene and benzene) and interfering gases (NH_3, CO, NO, NO_2, and C_3H_9N (trimethylamine)). This is due to the significantly larger dipole moment in the molecular structure of acetaldehyde. Increase in specific surface area, oxygen vacancy, and carrier concentration on the surface of ZnO NPs induced from Al doping has arisen that more oxygen ions can react with acetaldehyde molecules. The Al-doped ZnO NPs show the highest sensing response compared with other works in the literature ever reported. Therefore, the superior sensing performance of the Al-doped ZnO NPs offers opportunities for a wide range of applications, such as in environmental monitoring and industrial systems.

Acknowledgments

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