



Sensing of gaseous malodors characteristic of landfills and waste treatment plants

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Abstract. We approached the problem of sensing gaseous pollutants and malodors originating as a result of decomposition of organic compounds via chemoresistive sensors. A set of four screen-printed films based on two types of mixed tin and titanium oxides, mixed tungsten and tin oxides, and zinc oxide has been tested vs. the main gaseous components of malodors. N-butanol was also considered because of its importance as a reference gas in the odorimetric intensity scale. We found that, under proper working conditions, the films can sensitively detect such gases either in dry or in wet environments, within the range of concentrations of interest for their monitoring. We also demonstrated that the array is robust under solicitation by harmful interference gases such as CO, C₆H₆, NO₂ and NO.

1 Introduction

For certain contexts, there is a need for control of the concentration of gases that result from decomposition of organic compounds. Pollutants and malodors often accompany decomposition processes, giving rise to serious health risks and/or discomfort to the human agglomerates neighboring the plants where such gases are generated. Landfills and waste treatment plants are typical cases in which several types of emission gases or vapors must be carefully taken into account. In particular, landfill emissions are characterized by very complex composition, while plants such as incinerators are potential sources of some kinds of pollutants, deriving from waste combustion such as carbon monoxide, benzene and nitrogen oxides. Among the wide variety of decomposition gases we targeted ammonia, ethyl mercaptan and hydrogen sulfide, because they are tracers of decomposition with stinging odor to our noses even down to very low concentrations in the order of ppb. Concerning the gases deriving from waste combustion, we addressed the interference of carbon monoxide, benzene, nitrogen dioxide and oxide. They can be present if the landfill or the waste treatment plant is close to engines that burn fossil fuels or incinerators or be-

cause of circulation of lorries for garbage collection, depositing their loads in the landfills. Since the odorimetric aspect of the application is highly relevant, we also took into account the responses of all the selected sensors to n-butanol, because the latter is the reference gas for the odorimetric intensity scale, useful for fixing the threshold for the human perception of an odor (Van Harreveld and Heeres, 1995; Capelli et al., 2010). The study of the decomposition and waste combustion gases is of interest in several applications, such as monitoring in green-agriculture fertilization, livestock holdings with the decomposition process of manure, landfills, food quality, and chemical industries. Chemoresistive gas sensors based on metal oxides are extremely sensitive devices, appreciated for their low cost, compactness and full compatibility with standard electronics (Capone et al., 2003; Wang et al., 2010; Fine et al., 2010). A preliminary study to monitor landfill gas emissions was done in Guidi et al. (2012), Comini et al. (2004, 2005), Micone and Guy (2007), Romain et al. (2008), and Wetchakun et al. (2011). However, lack of selectivity is an issue and for that special care is needed while selecting the sensing units. In this work, we addressed the problem of detecting typical gaseous products of waste storage, by means of gas sensors based on thick films of metal

oxides and mixed solid solutions of them, also taking into account the possible interference of typical combustion products. The response at various temperatures to identify the best detecting temperature for each type of sensing material tested by the different gases will be shown. Afterwards the results of an increasing concentration in the range of interest using the same gases will be discussed. After the tests on the typical gases responsible for malodors in landfills, an analysis of possible interfering gases such as CO, C₆H₆, NO₂ and NO was performed to determine the robustness of the array of sensors for in-field applications.

2 Experimental

2.1 Selection of the sensors and preliminary operations

A set of four metal oxide semiconducting films has been selected for the purpose. The materials chosen are ZnO, two solid solutions of SnO₂ and TiO₂, in proportions of 30–70 % (named ST30) and 90–10 %, respectively (named ST90), and a solid solution of WO₃ and SnO₂ in proportions of 30–70 % (named WS30).

ZnO powders were prepared by dissolving a proper amount of Zn(NO₃)₂·4H₂O in doubly distilled water (Carotta et al., 2009a). The reaction mixture was stirred for 1 h and kept at room temperature for 24 h; then the product was washed, filtered and dried at 80 °C, and finally calcined at 450 °C for 2 h. WS30 is a solid solution of W and Sn oxides (with Sn : W = 30 : 70) produced via hydrolysis of a WCl₆ and Sn(II)ethyl hexanoate solution prior to calcination at 550 °C for 2 h under airflow. Preparation and characterization of nanostructured powders are described in Chiorino et al. (2001) and Shouli et al. (2010). Nanostructured powders of the solid solutions of Sn and Ti mixed oxide were produced via symplectic gel coprecipitation of stoichiometric Sn(4+) and Ti(4+) hydroalcoholic solutions, after calcination of the resulting xerogel at 550 °C for 2 h under airflow. The solid solutions of Ti_xSn_{1-x}O₂ at two values of x ($x = 0.3, 0.9$) will be hereinafter labeled as ST30 and ST90 (Carotta et al., 2008a, b, 2009b). The crystalline phase of the powders was investigated by X-ray diffraction (XRD) (Philips PW 1820/00 Cu K radiation with $\lambda = 1.54 \text{ \AA}$) performed at room temperature.

The powders were used to screen-print the sensing layers onto miniaturized alumina substrates. The layers were fired for 1 h at a selected temperature from 650 to 850 °C. Measurements were performed with the flow-through technique in a sealed test chamber. Air and target gases were supplied by certified bottles; humidity was provided by means of a bubbler filled with distilled water. The gases chosen for gaseous malodor application were ethyl mercaptan, ammonia, hydrogen sulfide and n-butanol, while CO, C₆H₆ and NO₂ were probed as interferents. The first three gases are primarily responsible for malodors in decomposition products, the fourth is a reference gas for the odorimetric inten-

sity scale (Van Harreveld et al., 1995; Capelli et al., 2010; Lee et al., 2013) and the last two are carcinogen combustion products. After the best operational temperature was determined, a dedicated experiment was performed to assess the effect of CO, C₆H₆, NO₂ and NO on the array of sensors. Humidity was monitored through a HIH-3610 Series Honeywell sensor. We investigated the responses of the sensors under dry (RH < 2 % at 35 °C) and wet (RH = 23 % at 35 °C) conditions. For a reducing gas the response R is defined as $(G_{\text{gas}} - G_{\text{air}})/G_{\text{air}}$, where G_{gas} and G_{air} are the conductance values in gas and in air, respectively, while for an oxidizing gas the response is defined as $\Delta R/R$.

2.2 N-butanol: reference gas for the odorimetric intensity scale

Generally, odoring compounds can be classified according to their structure, dimensions, compound family, and functional group, and they can be characterized on the basis of their concentration, intensity and hedonic tone. The majority of odoring molecules exhibit a low olfactory threshold; then odor is well detected even if the gas concentration in air is relatively low. In the literature, it is well known that the odor intensity is not proportional to the gas concentration (ASTM International E679-04, 2005; UNI EN 13725, 2004; Segura and Feddes, 2005; Brattoli et al., 2011; Lee et al., 2013; Lewis et al., 2005). In fact, due to the synergic and/or inhibiting effects between odorants, the relationships between the odor concentration and the chemical concentrations of the compounds are difficult to realize and reproduce (Micone and Guy, 2007). The odor concentration measurements can be carried out by three methods: analytical determination through mass spectroscopy or marker substance individualizations, electronic nose and olfactometry (Capelli et al., 2008; Muñoz et al., 2010). The first is not a completely efficient method for the scope of this work because it does not give information about human olfactometric threshold and it is too expensive for the purpose of designing a portable device (Davoli et al., 2003; Dincer et al., 2006; Fang et al., 2012; Zarra et al., 2009). The electronic nose consists of a network of non-specific chemical sensors, the sensor array, twinned with a data-processing treatment unit in order to recognize or measure the concentration of a gas or an odor. This device allows one to get rid, firstly, of the day-to-day human subjectivity of the olfactometry, secondly, of its expensive cost and, thirdly, of elaborate data analysis used by other analytical instruments (Micone and Guy, 2007). Electronic noses are able to classify and to measure odor intensity but not the concentration of gases dangerous for health (Gardner and Bartlett, 1994; Dentoni et al., 2012; Giuliani, 2010). Lastly, olfactometry is the technique currently used worldwide in odor quantification because it gives more useful information than the other two methods (UNI EN 13725, 2004; Segura and Feddes, 2005). A test panel formed by selected and practiced persons sniffs odorous gas samples

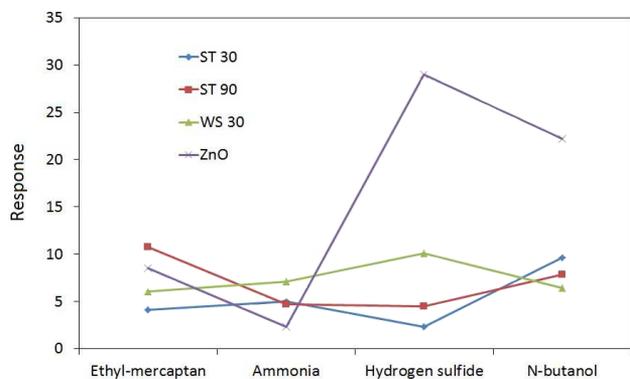


Figure 1. Best responses for each sensor at own working temperature both to gases of interest and to reference gas n-butanol, under dry conditions.

conveniently diluted by odorless air on the basis of determined relations so that each sample is presented to the panel following a decreasing dilution series. In this way the panel identifies the “odor threshold” and it is possible to digitize a sensation in order to create an odor intensity scale (odor units) (Nicolas et al., 2006; Sironi et al., 2010; Sarkara and Hobbs, 2002). Relying essentially on human expertise, it is considered a time-consuming and expensive method when used frequently (Micone and Guy, 2007). In this work we needed a reference gas for the odorimetric intensity scale in order to compare the responses obtained with gaseous malodors. Therefore, we chose n-butanol and tested the sensor array with a concentration of 5 ppm, which corresponds to a weak odor (about 12 OU – odor units) (Van Harreveld and Heeres, 1995; Capelli et al., 2010; Lee et al., 2013).

2.3 Response to decomposition gases and n-butanol

The response of the set of sensors was investigated vs. working temperature in order to determine the optimal detecting condition for ethyl mercaptan, ammonia, hydrogen sulfide and n-butanol (Fig. 1). The concentrations of the target gases were chosen in order to be comparable with the recommended exposure limits (REL) by NIOSH (United States National Institute for Occupational Safety and Health). Indeed, for ethyl mercaptan we chose the REL concentration of 0.5 ppm, 10 ppm for ammonia (REL = 25 ppm), 2 ppm for hydrogen sulfide (REL = 10 ppm) and 5 ppm for n-butanol (REL = 50 ppm).

The experimental results show diversification in the sensor responses to the first three gases and the values obtained are significant. Instead, for n-butanol the temperature test has shown homogeneous behavior and the response at best working temperature was significant for all sensing films. This is an indication of the suitability of this set of sensors to both the selective detection of the three target gases and to generic odor applications.

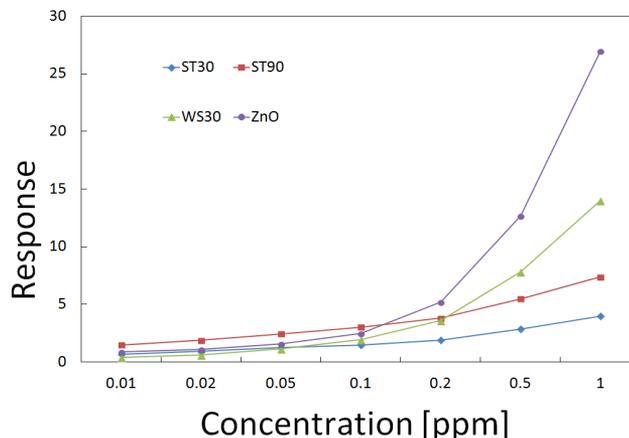


Figure 2. Responses vs. ethyl mercaptan from 0.01 to 1 ppm dispersed in a dry carrier.

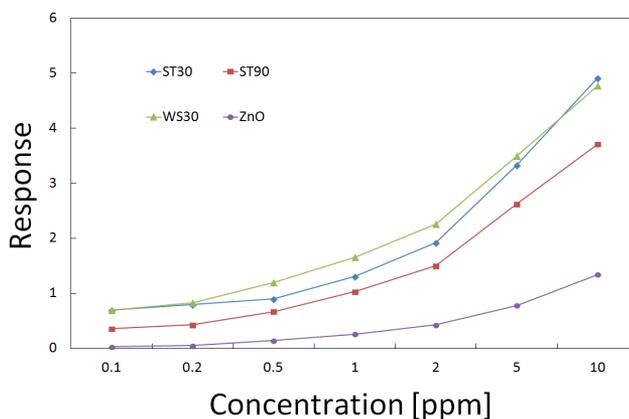


Figure 3. Responses vs. ammonia from 0.01 to 1 ppm dispersed in a dry carrier.

The optimal working temperature and the relative response for each gas are reported in Table 1.

At each best temperature for each gas, we measured the response to several concentrations of interest, highlighted in Figs. 2–5.

Regarding ethyl mercaptan, ammonia and hydrogen sulfide, the array encompasses sensors that are very unresponsive to at least one gas, while they show significant responses to the others. This feature is a very positive indication of implementation of a high-selectivity array. In particular, ZnO at 450 °C is capable of detecting hydrogen sulfide with little interference by ethyl mercaptan. ST30 at 600 °C can selectively detect ammonia; ST90 at 500 °C is capable of selectively detecting ethyl mercaptan. WS30 is sensitive to ethyl mercaptan and hydrogen sulfide at low temperatures, while it can selectively detect ammonia at high temperatures.

Since the sensors were capable of detecting concentrations of n-butanol even lower than 1 ppm, which corresponds to about 2 OU, one can conclude that sensing units are suitable

Table 1. The optimal working temperatures T_{best} and the corresponding responses R for all analyzed films tested with gases of interest and reference gas n-butanol.

Sensor	ETHYL MERCAPTAN 0.5 ppm REL 0.5 ppm		AMMONIA 10 ppm REL 25 ppm		HYDROGEN SULFIDE 2 ppm REL 10 ppm		N-BUTANOL 5 ppm REL 50 ppm	
	T [°C]	R	T [°C]	R	T [°C]	R	T [°C]	R
ST30	500	4.08	600	5	550	2.33	550	9.65
ST90	500	10.77	550	4.69	550	4.47	450	7.81
WS30	400	6.05	550	7.08	400	10.06	600	6.45
ZnO	400	8.54	400	2.34	400	29	400	22.23

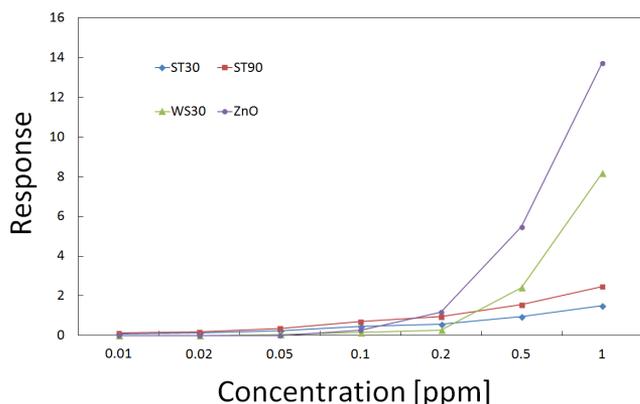


Figure 4. Responses vs. hydrogen sulfide from 0.01 to 1 ppm dispersed in a dry carrier.

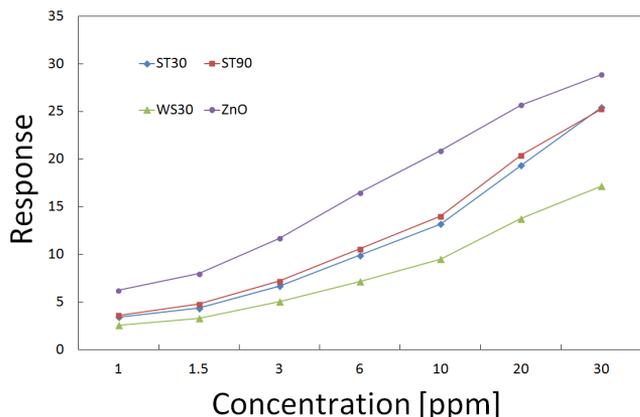


Figure 5. Responses vs. n-butanol from 1 to 30 ppm dispersed in a dry carrier.

not only for environmental monitoring but also for odori-metric applications. About this, since the human perception threshold is about 10 OU, it can be affirmed that the odori-metric performance of the sensors vs. n-butanol appears to be far better than the human olfact.

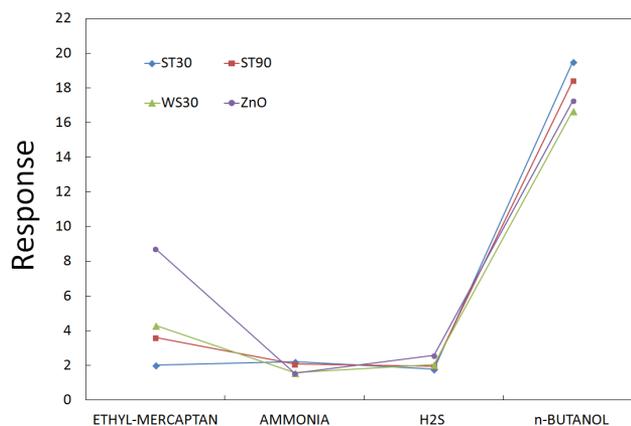


Figure 6. Responses to 0.5 ppm of ethyl mercaptan, 10 ppm of ammonia, 1 ppm of hydrogen sulfide and 5 ppm of n-butanol dispersed in a wet carrier.

In Table 2 the responses at the best working temperature for a concentration of 1 ppm for each gas are presented in order to compare the four sensors more clearly.

Consequently, an array based on ST90 for ethyl mercaptan, ST30 for ammonia and ZnO for hydrogen sulfide can be employed to detect the gases responsible for malodors in waste storage locations. The sensitivities of ZnO and WS30, defined as the derivative of the response vs. gas concentration, strongly increase after 0.2 ppm of H_2S . This is a useful feature since the sensors' lowest detection limit proves to be much lower than the concentration of interest for health risk (10 ppm). For the other sensors vs. the other gases, the response is quite high to concentrations below the exposure limits.

The response of the sensors was also tested vs. the highest concentration of each gas dispersed in a wet carrier ($\text{RH} = 23\%$ at 35°C). Significant decrease in the response was recorded, though it does not invalidate the capability of detection of the target gases (Fig. 6).

Table 2. The optimal working temperatures T_{best} and the corresponding responses for all analyzed films tested with gases of interest and reference gas n-butanol at the same concentration of 1 ppm.

Sensor	ETHYL MERCAPTAN 1 ppm REL 0.5 ppm		AMMONIA 1 ppm REL 25 ppm		HYDROGEN SULFIDE 1 ppm REL 10 ppm		N-BUTANOL 1 ppm REL 50 ppm	
	T [°C]	R	T [°C]	R	T [°C]	R	T [°C]	R
ST30	500	4.0	600	1.31	550	1.6	550	3.42
ST90	500	7.4	550	1.03	550	2.47	450	3.60
WS30	400	14	550	1.66	400	8.18	600	2.55
ZnO	400	27	400	0.26	400	13.72	400	6.22

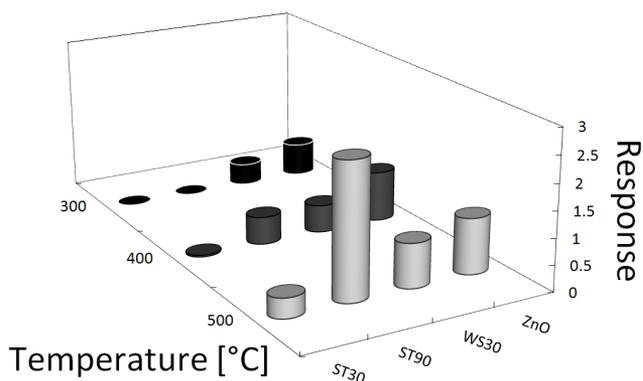


Figure 7. Responses of the sensors to 10 ppm of CO at three different temperatures.

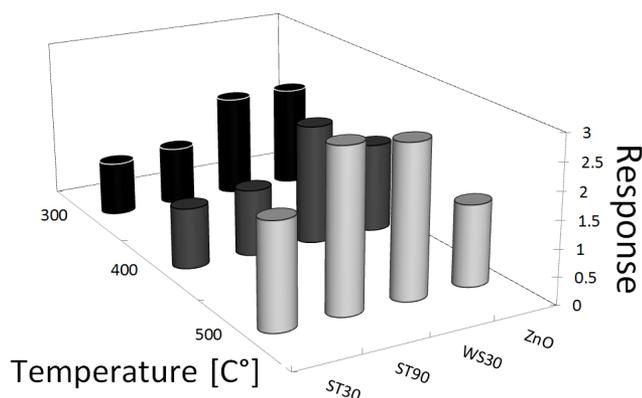


Figure 8. Responses of the sensors to 2 ppm of C_6H_6 at three different temperatures.

2.4 Response to harmful interfering gases

The responses of the sensors were also tested vs. CO, C_6H_6 , NO_2 and NO since they are harmful interfering gases in zones where waste treatment plants are located. The concentrations were chosen as follows: 10 ppm for CO, 2 ppm for C_6H_6 , 4 ppm for NO_2 and 4 ppm for NO, these being concentrations high enough to simulate strong interference from combustion plants near the landfills. The results are summarized in Figs. 7–10, where the responses of the sensors vs. CO, C_6H_6 , NO_2 and NO at several working temperatures are reported. For a reducing gas the response R is defined as $(G_{gas} - G_{air}) / G_{air}$, where G_{gas} and G_{air} are the conductance values in gas and in air, respectively, while for an oxidizing gas the response is defined as $\Delta R / R$.

As can be seen, all sensors show very low responses to these relatively high amounts of interfering gases. In Table 3 the maximum response to interfering gases are presented.

The majority of the responses are lower than 1 and in the worst case the response exceeds 2 in Fig. 7 as well as in Fig. 8, these latter being values low enough to be considered negligible in comparison with the responses to ethyl mercaptan, ammonia and hydrogen sulfide.

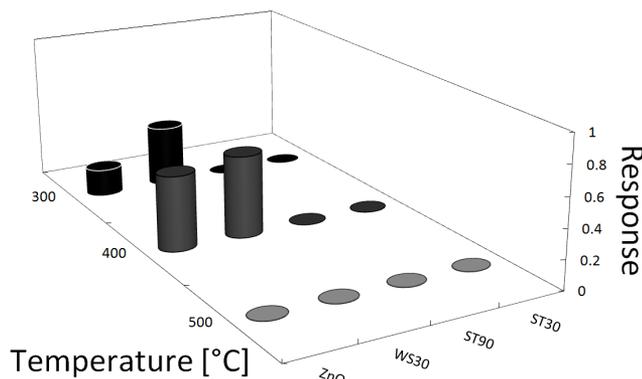
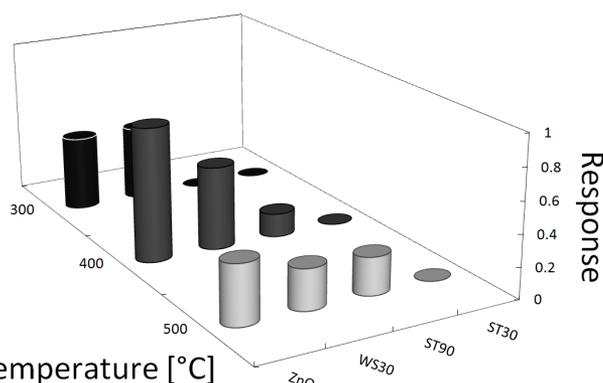


Figure 9. Responses of the sensors to 4 ppm of NO_2 at three different temperatures.

Therefore, the selected array of sensors could be employed in an array capable of selectively detecting the main gaseous malodors in landfills, without the interference of combustion gases that can harmfully interfere.

Table 3. The optimal working temperatures T_{best} and the corresponding responses for all analyzed films tested with interfering gases.

Sensor	CARBON MONOXIDE 10 ppm REL 35 ppm		BENZENE 2 ppm REL 100 ppm		NITROGEN DIOXIDE 4 ppm REL 1 ppm		NITROGEN OXIDE 4 ppm REL 25 ppm	
	T [°C]	R	T [°C]	R	T [°C]	R	T [°C]	R
ST30	500	0.38	500	1.92	300 ÷ 500	0	300 ÷ 500	0
ST90	500	2.58	500	2.94	300 ÷ 500	0	500	0.24
WS30	500	0.85	500	2.79	400	0.56	400	0.53
ZnO	500	1.07	300	1.90	400	0.51	400	0.85

**Figure 10.** Responses of the sensors to 4 ppm of NO at three different temperatures.

3 Conclusions

The problem of sensing harmful gas pollutants and malodors typical of decomposition of organic substances has been addressed via chemoresistive gas sensors. A set of four sensing materials (ZnO, two mixed solutions of Sn and Ti oxides and a mixed solution of W and Sn oxides) has been tested in dry and wet conditions. It resulted that with a proper choice of the working temperatures the array can sensitively detect the target gases and quantify both the health risk and the odor intensity. The interference of typical gaseous species that can be produced in waste treatment plants has been evaluated, and it resulted in a negligible response to CO, C₆H₆, NO₂ and NO by all the sensors.

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