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Recognition of complex odors with a single generic tin oxide gas sensor

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ABSTRACT

We report, for the first time, discriminating among complex odors using a single generic tin oxide gas sensor. The sensor heater is biased with high magnitude voltage impulses of predetermined thermal impacts adjusted to produce step-like pallet temperature rises of different magnitudes. The sensor pallet is exposed to aromas collected from various herbs and spices at different concentrations. The test period is 4 s only, during which the sensor pallet undergoes four step-like temperature jumps. The discriminative features extracted from the normalized response patterns recorded for six different herbs and spices, each at various concentrations in air, afford their clear segregation in the feature space and recognition. The capacity of the device to discriminate among complex odors and single-component small molecule gases in a unified feature space is demonstrated. The odor discrimination power of the system is almost aging drift-proof; response patterns recorded for a specified odor at independent experiments carried out during the nominal lifetime (six months) of the sensor join the correct cluster in the feature space.

1. Introduction

Discriminating among odors and complex gas mixtures without systematic quantitative analyses, similar to that taking place in the mammalian olfaction systems [1], is valuable for many industrial and domestic applications. The majority of these applications require low cost, compact size, light weight, durable, rugged, and user-friendly gas or odor recognition systems [2,3]. Sensor array-based odor classification systems [4,5] are, in principle, suitable for these purposes, but they suffer from the unpredictable [6] and predictable [7] drifts of the array components [8], which necessitate frequent cumbersome recalibrations and/or costly array replacements.

Almost all generic chemoresistive and diode-type gas sensors [9] operate at elevated temperatures. The responses of an operating temperature-modulated gas sensor provide gas-related information which can be extracted and used for the recognition of the analyte gas [10–13]. The amount of extractable information is enough for discriminating among a few single-component contaminants in air [14–16]. We have recently reported the possibility of providing step-like temperature variations on the pallets of generic metal oxide sensors [17]. This has been realized by applying voltage impulses of predetermined thermal impacts to the microheater of the sensor. It was shown that a thermal shock-induced (TSI) generic gas sensor, compared to any temperature-modulated bulk gas sensor, can generate responses significantly richer in extractable gas-related information which can be used for the classification of the target gas. The amount of information obtained from the responses of a single TSI generic tin oxide-based gas sensor was shown to be enough to facilitate the correct differentiation of the four butanol isomers each in various concentrations in air [17].

Here, as the logical next step to our previous report, we are reporting the successful use of a TSI generic tin oxide gas sensor for discriminating among complex odors each at various concentrations in air. These odors are extracted from different natural herbs and spices and their precise composition and concentration are unavailable. This is the first report of complex odor classification with a single generic gas sensor. Within a few seconds of exposure, the device is able to discriminate among all the complex odors examined. It is shown that the devised gas classification system is resilient against the sensor's aging providing true gas classification results all along the nominal lifetime of the generic sensor utilized.

2. Experimental

2.1. Aroma extraction

The herbs and spices examined are all in the solid form commercially available for culinary use. These are listed along with their major flavor or odorant components of their extracted aromas,

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Table 1
Herbs and spices used in the experimental work along with their main odorant(s).

No.	Herb/spice	Major flavor component	Herb/spice category	Reference
1	Saffron (*Crocus sativus*)	Safranal	Iridaceae	[18]
2	Spearmint (*Mentha spicata*)	R-carvone	Lamiaceae	[19]
3	Thyme (*Thymus mongolicus*)	Thymol/carvacol	Lamiaceae	[20]
4	Cinnamon (*Cinnamonum zeylanicum*)	Cinnamic aldehyde/eugenol	Lauraceae	[20]
5	Cumin seed	Para-cymene	Umbelliferae	[21,22]
6	*Heracleum persicum*	Anethole	Umbelliferae	[23]

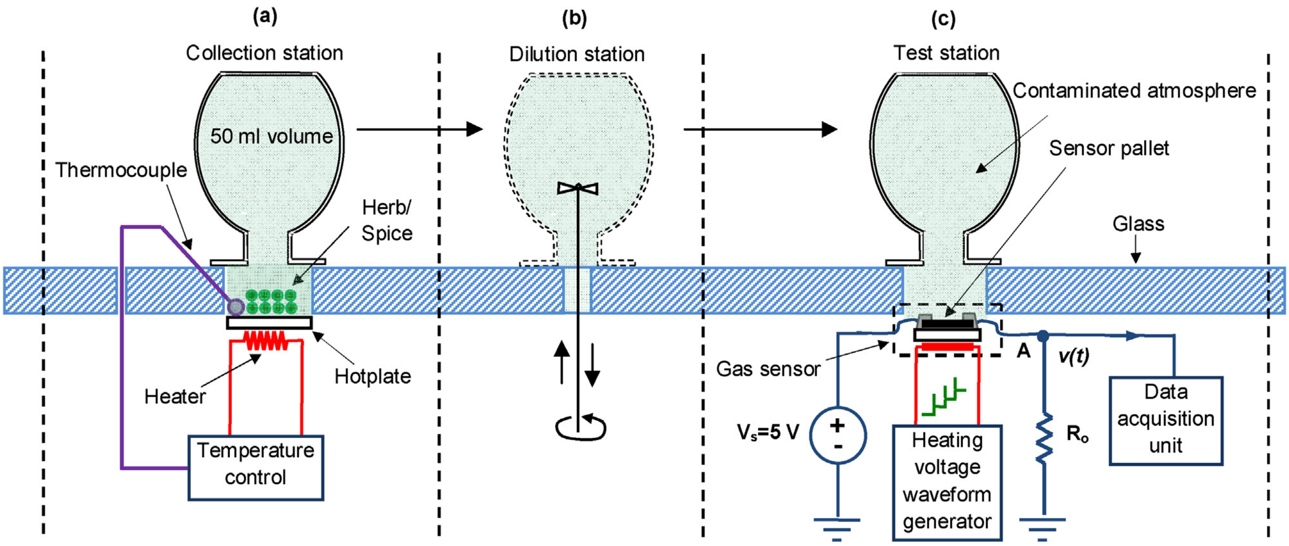

Fig. 1. The schematics of the experimental system used for the response pattern recordings; the system comprises: (a) odor collection, (b) analyte dilution and (c) response recording stations.

according to the background literature, in Table 1. These items are widely available, non-toxic and fairly stable, and are selected to provide a number of specific complex odors for the purpose of the demonstration of the discrimination power of the fabricated sensory system. Accordingly, each material is purchased from a single batch, mixed, powdered and stocked for consumption in all the experiments reported.

A homemade system, schematically presented in Fig. 1, is utilized for aroma collection. The system comprises a temperature controlled mini-hotplate of 15 mm diameter. Accurately weighed amounts of each herb/spice is placed on the hotplate which is preset at $T = 170\,^{\circ}\mathrm{C}$. This temperature was determined by trial and error; lower temperatures produced lower odor levels in the odor collection jar (Fig. 1a) and would result in high noise levels in the normalized response patterns produced (see below), while higher temperatures increased the number of misclassifications (see below), perhaps, due to the significance of the partial oxidations and/or pyrolyses of the odorants. Weighing of the amount of the material placed on the hotplate is for the purpose of repeatability of the experiments. The odor concentration measurement proved unnecessary and was not attempted; eight different concentrations of each analyte were produced by the dilution of the collected odor at the "dilution station" shown in Fig. 1b. In a few seconds of stoppage at this station, the collected odor is diluted with air allowed in to partly replace the balloon atmosphere. During this period, the balloon atmosphere is agitated with a small electric fan (Fig. 1b) to prevent the selective diffusion of the different components of the complex odor. After each stoppage, a lower odor concentration prevails in the balloon. The relative humidity and temperature in the balloon are the same as in the laboratory varying in the respective ranges of 21 to 27% and 20 to 27 °C in all independent experimental sessions.

2.2. Response pattern recording

The experimental set up is schematically presented in Fig. 1c. The sensory system used is similar to that described in Ref. [17]. The sensor used is a low-cost, tin oxide-based, generic gas sensor (SP3-AQ2, FIS Inc., Japan) equipped with a ruthenium oxide thick film microheater. This was decided to be of the same type as the sensor utilized in our previous work [17] as the produced results would demonstrate another facet of the analytical potential of thermal shock induction on the same general sensing element. The nominal heating voltage of the device is 5 V which would produce 330 °C

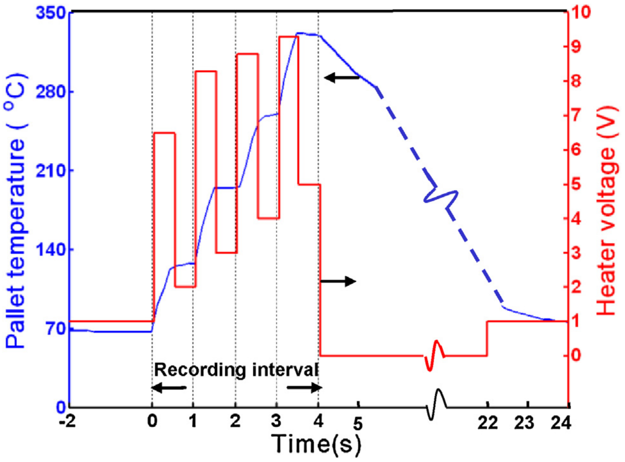

Fig. 2. The heating voltage waveform applied to the sensor before ($t < 0$), during ($0 < t < 4$ s), and after ($t > 4$ s) each response recording. The voltage variations beyond $t = 4$ s prepares the device for the next test.