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A rapid discreteness correction scheme for reproducibility enhancement among a batch of MOS gas sensors

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ABSTRACT

Metal oxide semiconductor (MOS) gas sensors have been widely reported in machine olfaction system (i.e. electronic nose/tongue) for rapid detection of gas mixture components due to their positive characteristics of cross-sensitivity, broad spectrum response and low-cost. However, the discreteness of MOS gas sensors caused by inherent sensor variability during the manufacturing process results in the failure of the batch-oriented applications of MOS gas sensors due to their weak reproducibility. Certainly, it will also cause negative influence to the development of electronic nose/tongue based on MOS gas sensors (e.g. accuracy and consistency during electronic nose/tongue detections). Therefore, the contribution of this paper is to solve the discreteness and improve the reproducibility of sensors by designing an effective and easily realized scheme for large-scale calibration. Experimental results demonstrate that the proposed scheme can effectively and rapidly realize the calibration of the sensors' discreteness in batch of electronic noses production and the proposed scheme have also been used in industry. Besides, this paper also proves that one sensor's discreteness is constant and keeps unchanged when the sensor is exposed to different kinds of gas components.

1. Introduction

Electronic nose (E-nose), as an artificial olfaction system, is an instrument comprised of a chemical sensor array with partial specificity and an appropriate pattern recognition algorithm [1,2]. Metal oxide semiconductor (MOS) gas sensors have been widely reported in E-nose for detection of chemicals [3–12]. MOS sensors have also been used in odor-discrimination system for fruit detection [13]. Recently, a variety of algorithms have been proposed for dealing with the sensor drift problem [14–18]. Fonollosa et al. also studied the sensor failures in discrimination of chemical substances [19]. However, most of the research in E-nose based on MOS gas sensor array focus on the pattern recognition analysis using one fixed sensor array, and without considering the problem of sensor's reproducibility that will result in the difference of electrical signal between two sensor arrays of the same type [9]. In other words, other sensor arrays with completely the same type may not be appropriate with the learned pattern recognition model (i.e. artificial neural network) due to the weak reproducibility [20]. MOS gas sensors are operated with the principle that volatile odor components can produce a reaction inside the sensor in contact with a catalytic metal, changing the electrical resistance of the sensor device and producing some voltage signal [21]. Generally, the sensing material is metal oxide, most typically SnO_2 [1]. The principles can be described as follows.

When the metal oxide crystal is heated at a certain high temperature in air, oxygen is adsorbed on the crystal surface with a negative charge. In the presence of a deoxidizing gas, the surface density of the negatively charged oxygen will decrease so that the barrier height is reduced which will decrease the sensor resistance. The detection principle of MOS gas sensors is based on the chemical adsorption and desorption of chemicals on the sensor's surface. Besides, the ambient temperature and humidity will also affect the sensitivity characteristics of sensor by changing the rate of chemical reaction [22]. Therefore, from the complex sensing principle and the various factors related in sensing, the reproducibility of MOS gas sensors should be taken into consideration in the industrial production of MOS gas sensors based instruments.

In batch of E-nose production, the homogeneity of multiple electronic noses' predictions when exposed to the same gas component is very important and the week reproducibility would

Abbreviations: MOS, metal oxide semiconductor; FPGA, field programmable gate array; DC, Direct current; RH, Relative humidity; JTAG, joint test action group; CPU, central processing unit; SDRAM, synchronous dynamic random access memory; LCD, liquid crystal display; CO, carbon monoxide; NH_3, ammonia; NO_2, nitrogen dioxide.

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seriously degrade the homogeneity [23]. The homogeneity completely depends on the MOS gas sensor array embedded in E-nose, because the pattern recognition module used internally is identical among E-noses and different inputs from sensors would lead to different predictions. Unfortunately, the MOS gas sensor array of identical type reflects diverse responses to the same chemical in the same experimental condition due to the inherent sensor variability and discreteness during the manufacturing process [9]. The kind of sensor discreteness must cause the reduction of E-nose prediction accuracy and reproducibility. To solve the problem of sensor discreteness, specific correction methods have been studied in previous publications [23,24]. Comparatively, the GAT–RWLS method proposed in [23], for its simplification of algorithm, is easier to implement for calibration in real-time E-nose detection. However, in large-scale sensors application (i.e. production of E-nose instruments), a rapid discreteness correction scheme is very necessary to reduce the complexity and also promise the accuracy, especially for regular calibration. The problems of sensor's discreteness and reproducibility in large-scale application of MOS gas sensors have been mentioned and fully solved in this work.

Therefore, the contribution of this paper is to present an effective implementation scheme of sensors' discreteness correction coupled with GAT–RWLS method for batch-oriented instruments production. In addition, this paper also reveals that the sensors' discreteness has little relation with the type of measured gas.

2. Materials and method

2.1. Sensors' discreteness and gases experiments

MOS gas sensors' discreteness will cause large difficulties in batch-oriented instruments development. Especially that the discreteness extremely lowers the accuracy of electronic nose instruments. The discreteness can be illustrated in two facets:

(1) Baseline difference: the sensitive resistance Ro of identical sensors in the standard environment (clean air) with temperature 20 °C and relative humidity (RH) 60% is variable which results in that the baseline of sensor with identical type is different in the same environment.
(2) Sensitivity difference: when exposed to some kind of pollutant gas, the MOS sensors with identical type also have different sensitivity which can be denoted as Rs/Ro (Rs is the sensitive resistance in the pollutant gas and Ro is the sensitive resistance in clean air). This will result in that the sensor responses with identical type are also different when exposed to the same type of gas with the same concentration in the same environment. That is, the same two sensors in the same environment have different outputs.

Therefore, it is not difficult to infer that the discreteness of MOS sensors can largely influence the accuracy of detective instruments, and rapid correction of the discreteness without changing the sensor circuits is very significant in improving the sensors' reproducibility and the performance of instruments, especially in batch-oriented application.

For studying of the sensor discreteness and its rapid correction in batch-oriented application, we have employed multiple kinds of gases experiments using 6 electronic nose systems embedded with identical sensor array. The electronic nose system based on Field Programmable Gate Array (FPGA) processor has been introduced in our previous publication [5]. For visualization, the picture of our E-nose has been illustrated in Fig. 1 (the left part in bottom). Considering the characteristics of broad spectrum and low-cost of metal oxide semiconductor gas sensors, four metal oxide semiconductor gas sensors from Figaro Inc. including TGS2602, TGS2620, TGS2201A and TGS2201B are used in the sensor array. The heating voltage of TGS2620 and TGS2602 is 4 V (Volt), and the heating voltage of TGS2201A/B is 7 V. The supplied power voltage of system is DC12 V. The experiments of electronic noses were employed in the climate chamber (LRH-150S). The experimental process including gas preparation, climate chamber, E-nose system and data collection is illustrated in Fig. 1. The typical response of an array of four gas sensors with four phases in the sampling process (1. baseline, 2. transient response, 3. steady state response, 4. recover process) can be observed in Fig. 1 (the top part). Totally, 126 formaldehyde samples, 72 benzene samples, 66 toluene samples, 58 carbon monoxide samples, 27 ammonia samples and 30 nitrogen dioxide samples were obtained. The experimental conditions and concentrations are different from each other. The discreteness of TGS2620, TGS2602, TGS2201A and TGS2201B sensors when exposed to the same concentration of formaldehyde gas has been illustrated in Fig. 2. Note that TGS2620 (1–6), TGS2602 (1–6), TGS2201A (1–6) and TGS2201B (1–6) represent six sensors with completely the same type, respectively. We can see from Fig. 2 that the discreteness of TGS2620 is weaker than other three MOS gas sensors. In other words, the reproducibility of TGS2620 is comparatively better. This phenomenon results from several facets. Though their sensing principles in detection are similar, their manufacturing process, electrical characteristics, and sensitivity will also influence the reproducibility. For instance, the sensor resistance (Rs) of TGS2620 is 1–5 kΩ, while 10k–100 kΩ is for TGS2602, 250 kΩ and 25 kΩ are for TGS2201A and TGS2201B, respectively. In our experiments, TGS2620 shows the best reproducibility and stability, while TGS2602 performs the worst.

2.2. Review of the previous GAT–RWLS method

GAT–RWLS method using reference formaldehyde gas for discreteness correction was proposed in our previous publication [23]. Through a large number of electronic nose experiments, we found that there exists a good linear relation between two sensors with the same type when exposed to the same environment and conditions. That is, the discreteness can be easily corrected in a linear way. For simplification, the calibration transfer model is shown by

$$y_{i,n} = a_i \cdot x_{i,n} + b_i, \quad i = 1, \ldots, k; \qquad n = 1, \ldots, N \tag{1}$$

where k denotes the number of sensors being calibrated, N denotes the number of calibration samples, x denotes the response of slave, y denotes the estimation of the master, a_i and b_i represent the calibration coefficients of the ith sensor obtained using reference gas (formaldehyde). In this model, a master should be determined as the standard electronic nose in advance, take other electronic nose as slaves, and calibrate the slaves to the master.

The proposal of model (1) is based on a global affine transform (GAT) with scaling and translation in a special way that the sensors in a sensor array influence each other in calibration. That is, the calibration is independent for each sensor. Also, considering the experimental error of a number of samples which will result in the inaccuracy of the calibration model, a robust weighted least square (RWLS) method was used for regression (1) and obtaining the parameters a and b. Experimental results of the formaldehyde samples correction demonstrate that the proposed method was very effective and easy to realize. We refer readers [23] for the details of the GAT–RWLS method.

In this paper, the calibration parameters obtained using formaldehyde as reference gas would also be validated for correction of the sensors' discreteness when exposed to other five kinds of gases in different conditions and concentrations.

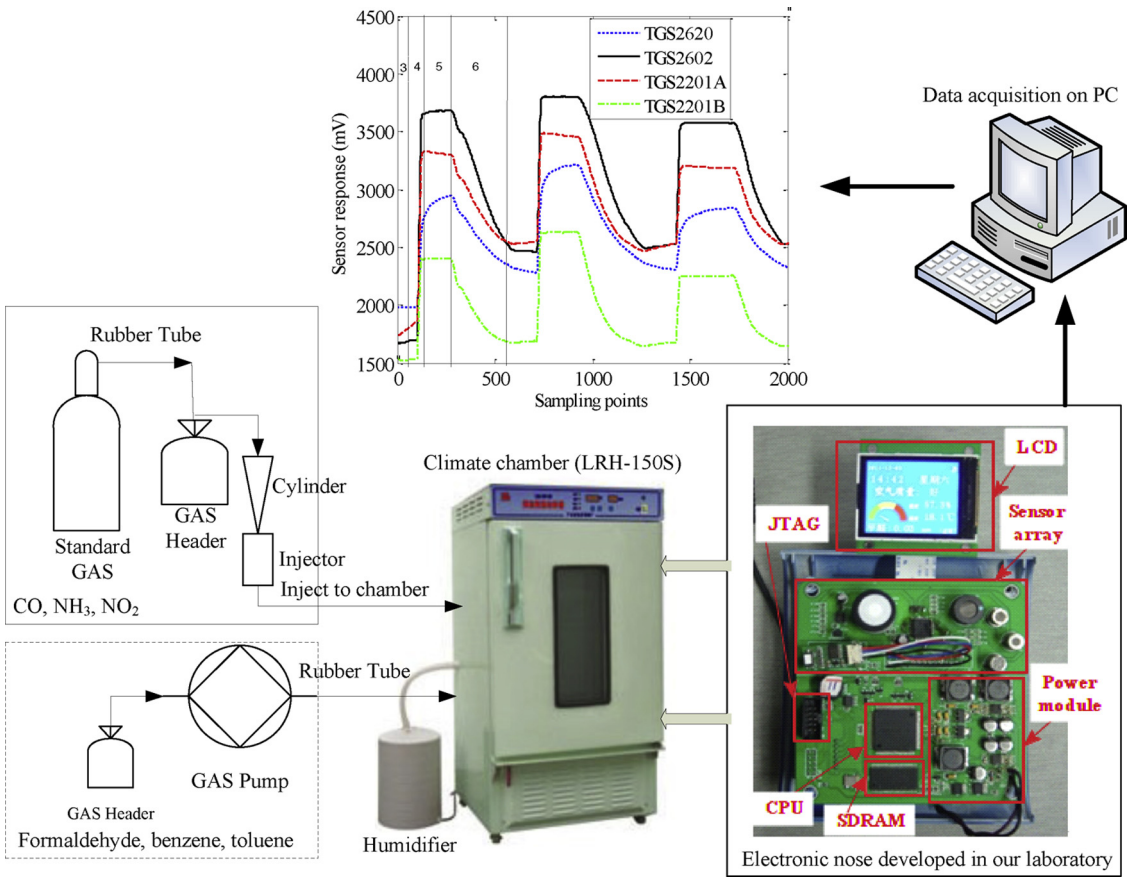

Fig. 1. Typical experimental platform of electronic nose; the left part in bottom is the gas collection being injected into the chamber; the right part in bottom is the electronic nose system placed in the chamber; the top part is the data collection.

2.3. Proposed correction scheme in batch of portable electronic noses

An easily realized and fast correction scheme is very necessary in batch of more than 50 electronic noses production. Therefore, this paper aims to propose an effective scheme for sensors' discreteness based on the GAT–RWLS calibration method.

The sensors' discreteness correction should be completed through experiments in this paper. Considering that the uniformity of reference gas in the chamber in experiment is very necessary, the volume of the chamber should not be too large. Note that the uniformity denotes that all electronic noses placed into the climate chamber can be exposed to the same concentration of reference gas. Then 11 electronic nose including the master and 10 slaves were employed in each batch of experiments, and the time consumption is approximately 1 h. Therefore, totally 5 batches of calibration experiments would be employed to complete all calibrations of 50 slaves to the master, and only about five hours are needed. Considering the environmental robustness of calibration, three experimental conditions 15 °C/60%RH, 25 °C/60%RH, and

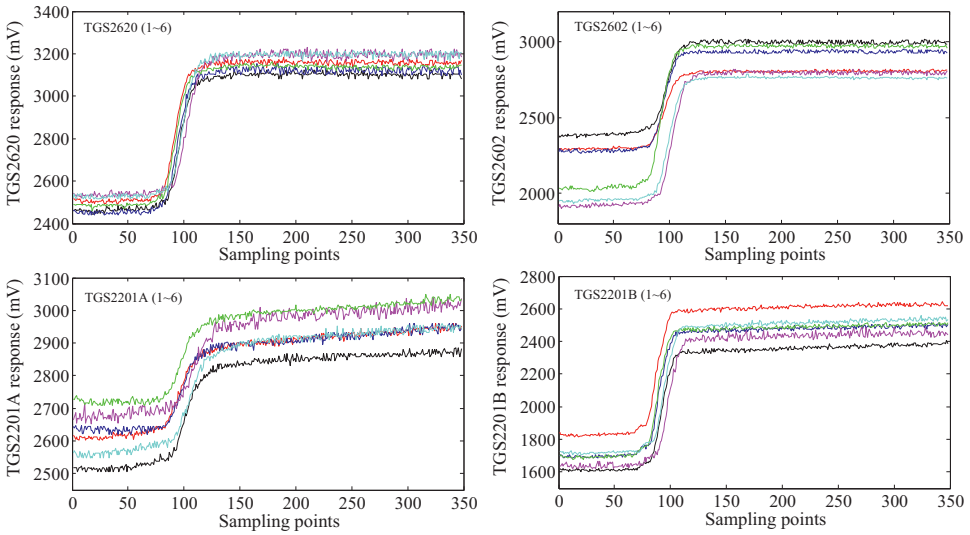

Fig. 2. Responses of six sensor arrays of the same type with TGS2620, TGS2602, TGS2201A and TGS2201B when exposed to the same concentration of formaldehyde.

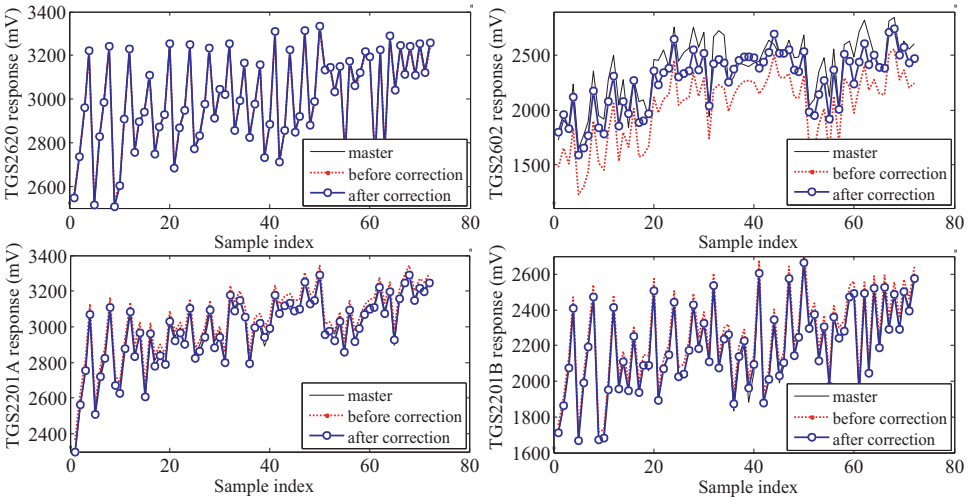

Fig. 3. Correction of 72 benzene samples using the calibration coefficients obtained with reference gas (formaldehyde).

35 °C/60%RH are employed in each batch of experiments. In each experimental condition, two different concentrations of reference gas are produced for sensitivity correction.

The proposed experimental scheme for discreteness correction of 50 slaves to the master can be shown as follows.

Step 1: For differentiation, we define the standard electronic nose and 50 being calibrated electronic noses as master, slave 1, slave 2, slave 3, slave 4, . . ., and slave 50, respectively; then, divide the 50 slaves into 5 batches (each batch contains 10 slaves).

Step 2: Put the master and the first batch of 10 slaves into the chamber; set the temperature and relative humidity of the chamber as 15 °C and 60%RH, and turn on the humidifier;

Step 3: Wait until the setting temperature and humidity are achieved, and turn off the humidifier. First, the baseline collection of 5 min is sustained. Second, inject reference gas (formaldehyde) into the chamber using a pump for 10 s, and continue the data collection for 5 min. Then, inject reference gas (formaldehyde) into the chamber using a pump for 10 s again, and continue the data collection for 5 min;

Step 4: Set the temperature and relative humidity of the chamber as 25 °C and 60%RH, and turn on the humidifier; then, repeat step 3;

Step 5: Set the temperature and relative humidity of the chamber as 35 °C and 60%RH, and turn on the humidifier; then, repeat step 3;

Step 6: Air exhaust and chamber cleaning. After finish the batch of experiments, air exhaust by a pump is necessary for chamber cleaning to recover the sensor response quickly, and take out the electronic noses in the chamber;

Step 7: Put the master and the other batches of slaves into the chamber, respectively, and repeat step 3∼step 6;

Note that steps 2–6 denote the whole process of calibration experiments in one batch of 11 electronic noses. There are approximately 3000 sampling points collected in the whole process. In correction, we should first determine the positions of three features which correspond to the three experimental conditions 15 °C/60%RH (step 2), 25 °C/60%RH (step 4), and 35 °C/60%RH (step 5) in the steady state response in the master, and obtain the calibration coefficients between each slave and the master using the model denoted by Eq. (1). Then, the obtainedcalibration coefficients would be used to correct the whole curves and verify the performance of the proposed scheme.

3. Results and discussion

For validation of the calibration coefficient obtained from the reference gas (formaldehyde) on other five gases samples, we select one of sensor arrays in electronic noses as slave, and calibrated it to the master. For calibration, we select one point in

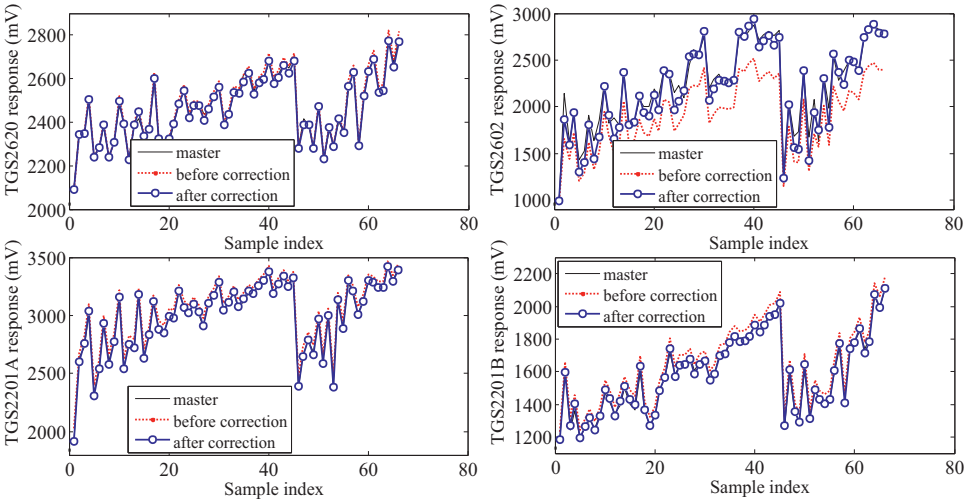

Fig. 4. Correction of 66 toluene samples using the calibration coefficients obtained with reference gas (formaldehyde).

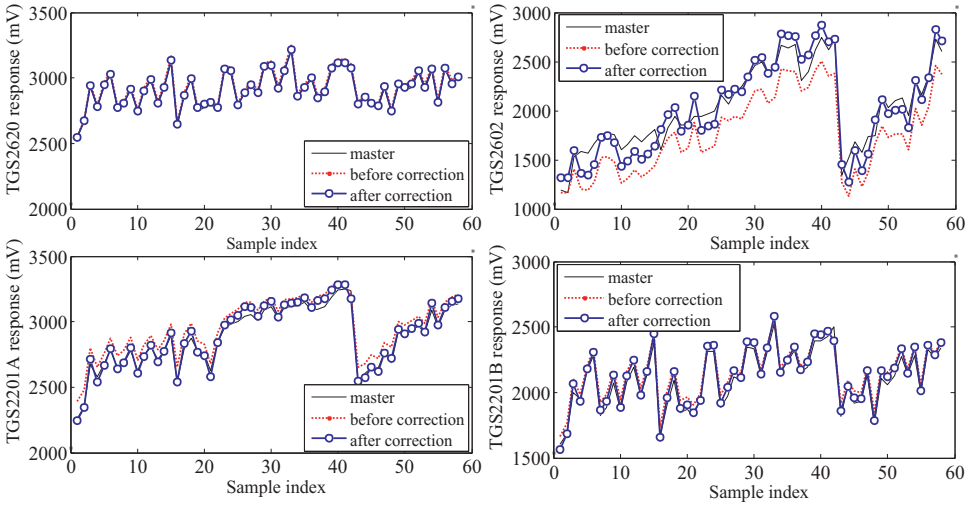

Fig. 5. Correction of 58 carbon monoxide samples using the calibration coefficients obtained with reference gas (formaldehyde).

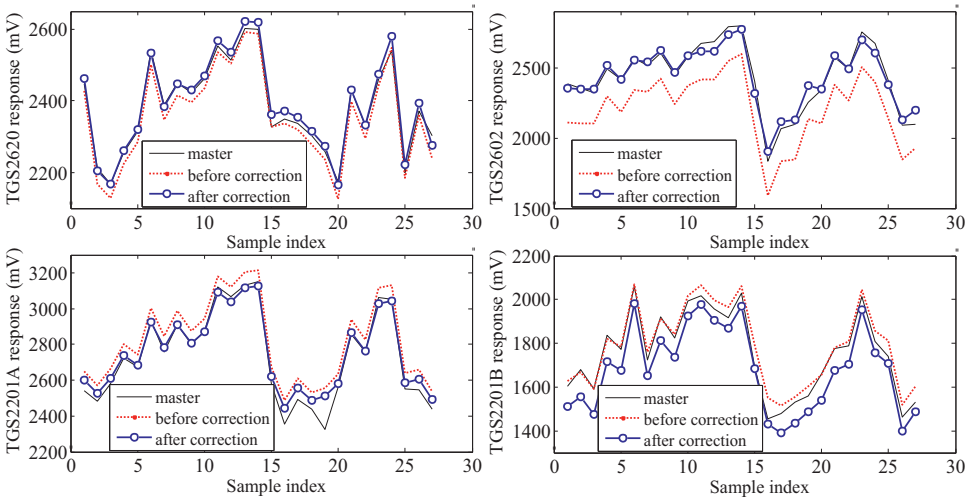

Fig. 6. Correction of 27 ammonia samples using the calibration coefficients obtained with reference gas (formaldehyde).

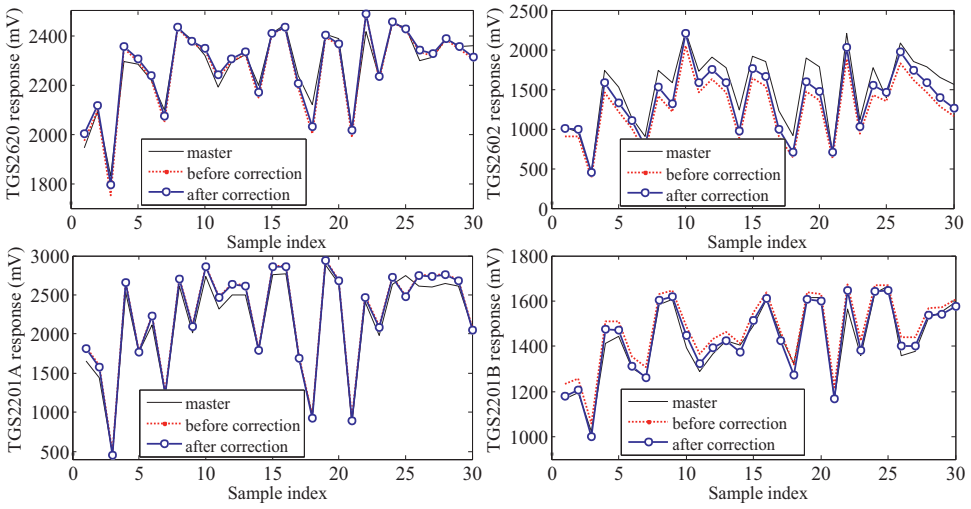

Fig. 7. Correction of 30 nitrogen dioxide samples using the calibration coefficients obtained with reference gas (formaldehyde).

the steady state response as the feature in each sample. The calibration coefficients are calculated by operating the GAT–RWLS method on the 126 formaldehyde samples between the slave and the master. The obtained calibration coefficients in Eq. (1) $a=[1.001,0.916,0.971,1.009]^T$, $b=[-0.01,0.092,0.003,-0.02]^T$ for TGS2620, TGS2602, TGS2201A and TGS2201B between the slave and the master, respectively.

Based on the obtained calibration coefficients of the four gas sensors, Figs. 3–7 illustrate the calibration results of benzene, toluene, carbon monoxide, ammonia, and nitrogen dioxide tested on the same slave electronic nose, respectively. We can see that the sensor response in slave can be calibrated to the master very well. Therefore, we can say that the calibration coefficients obtained by using reference gas (formaldehyde) are also effective in correction of other gases samples.

The results from Figs. 3–7 demonstrate that one sensor's discreteness is almost constant in a certain experimental condition and it keeps changeless when the sensor is exposed to different kinds of gas components. It also demonstrates that the discreteness of sensors has little relation with the types of gases and the discreteness correction model can be determined using any kind of reference gas. The finding is optimistic that the discreteness problem of sensors can be solved and the reproducibility can be improved in batch-oriented applications of sensors by designing an appropriate scheme coupled with some mathematical method.

Then, we have presented the calibration results of the four sensors in one batch of electronic nose instruments. Totally, 11 electronic noses including the *master* were employed in one batch of experiments. Figs. 8(a)–11(a) illustrate the original sensor response curves (approximately 3000 sampling points) of TGS2620, TGS2602, TGS2201A and TGS2201B, respectively. We can find that the 11 sensor curves in Figs. 8(a)–11(a) are not in coincidence which demonstrate that the MOS gas sensor discreteness is very serious and significant in the same environment. Note that there are 11 curves which represent 11 sensors with completely the same type in each figure. To validate the proposed discreteness correction scheme in batch-oriented application, the corrected sensor response curves of TGS2620, TGS2602, TGS2201A and TGS2201B in accordance with Figs. 8(a)–11(a) have been illustrated in Figs. 8(b)–11(b). We can see from Figs. 8(b)–11(b) that the sensor response curves of the 11 sensors with the same type have been coincided together after correction which also demonstrate that the proposed scheme in batch of electronic nose production are very successful and easier to be realized. This shows that the proposed scheme can also be used in industry for large scaled electronic noses manufacture. It can be inferred that the proposed scheme can also be used in the manufacture and production of electronic nose systems based on an array of MOS gas sensors in other applications (i.e. food control, medical diagnosis, etc.).

It is worth noting that some time delays of the sensor response exist among electronic nose systems in Figs. 8–11. In detail, the positions in some inflection points such as 1000, 1750 and 2250

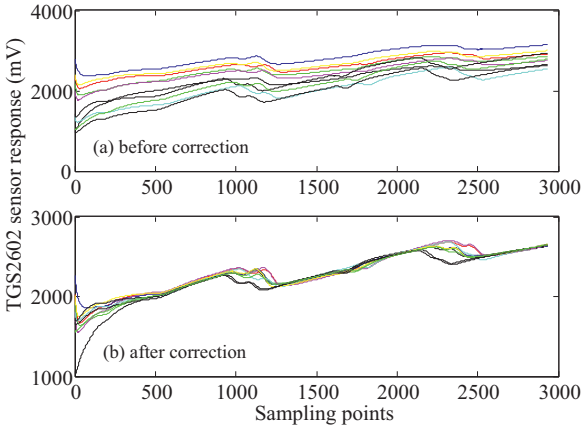

Fig. 9. TGS2602 sensor's discreteness correction of 10 electronic noses using the proposed scheme. (a) sensor responses before correction; (b) sensor responses after correction.

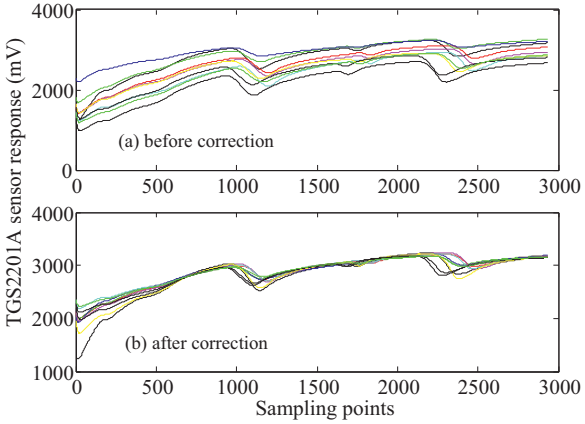

Fig. 10. TGS2201A sensor's discreteness correction of 10 electronic noses using the proposed scheme. (a) sensor responses before correction; (b) sensor responses after correction.

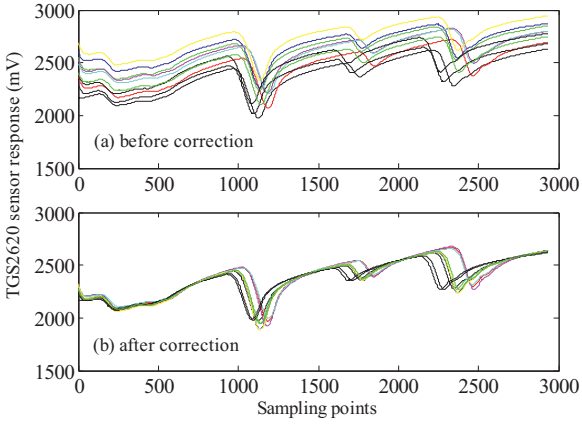

Fig. 8. TGS2620 sensor's discreteness correction of 10 electronic noses using the proposed scheme. (a) sensor responses before correction; (b) sensor responses after correction.

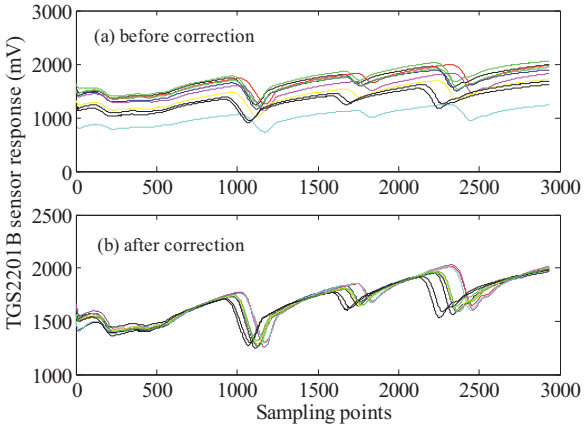

Fig. 11. TGS2201B sensor's discreteness correction of 10 electronic noses using the proposed scheme. (a) sensor responses before correction; (b) sensor responses after correction.

in the sensor curves show the time delays which cannot be coincided after correction without appropriate translation. This is due to the slight differences of sampling frequency which result from the software program running and tiny difference of electronic device in circuits among the electronic systems. Note that the inflection points represent the transient response of sensors. However, it does not influence the calibration of the sensors' discreteness in our work, because the calibration coefficients of each sensor in Eq. (1) are obtained using the selected three appropriate features in the steady state response but not the transient response around the inflection points.

4. Conclusions

This paper proposes an easily realized and rapid scheme for batch-oriented MOS gas sensors' discreteness correction in E-nose instruments production and improve the reproducibility of sensors. The calibration parameters obtained using reference gas (formaldehyde) and the GAT–RWLS method have also been validated on other gases samples (benzene, toluene, carbon monoxide, ammonia and nitrogen dioxide) and demonstrate that the sensor discreteness is independent and has little relation and with the types of gas components. Experimental results also demonstrate that the proposed correction scheme is very effective and can realize rapid sensor discreteness correction in batch of E-noses production.

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Biographies

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