

Miniaturized Electronic Nose System Based on a Personal Digital Assistant

Yong Shin Kim, Yoon Seok Yang, Seung-Chul Ha, Hyeon-Bong Pyo, and Chang Auck Choi

A small electronic nose (E-Nose) system has been developed using an 8-channel vapor detection array and personal digital assistant (PDA). The sensor array chip, integrated on a single microheater-embedded polyimide substrate, was made of carbon black-polymer composites with different kinds of polymers and plasticizers. We have successfully classified various volatile organic compounds such as methanol, ethanol, *i*-propanol, benzene, toluene, *n*-hexane, *n*-heptane, and *c*-hexane with the aid of the sensor array chip, and have evaluated the resolution factors among them, quantitatively. To achieve a PDA-based E-Nose system, we have also elaborated small sensor-interrogating circuits, simple vapor delivery components, and data acquisition and processing programs. As preliminary results show, the miniaturized E-Nose system has demonstrated the identification of essential oils extracted from mint, lavender, and eucalyptus plants.

Keywords: Electronic nose, PDA, sensor array, carbon black-polymer composite, gas sensor.

I. Introduction

Conventionally, most chemical sensors have worked on the basis of highly selective physicochemical interactions between a designed receptor and an analyte of interest, called a lock-and-key mechanism. This approach is appropriate when target analytes have specific interactions in the presence of controlled backgrounds and interferences. However, it is not particularly useful for analyzing, classifying, or identifying complex chemical mixtures such as perfumes, foods, and oils. For the analysis of complex real samples, an intelligent arrayed gas sensor system, that is, an electronic nose (E-Nose), was first realized more than 20 years ago by Persaud and Dodd [1]. This E-Nose system, conceptually similar to the mammalian sense of olfaction, is principally composed of an array of vapor detection sensors with partial specificity and an appropriate pattern recognition algorithm.

E-Nose systems have attracted much attention due to their potential applications in food quality control, medical diagnosis, and environmental monitoring of pollutants [2]-[5]. At present, there are continuous efforts to improve E-Nose systems even though several desk-top or hand-held products are commercially available. In particular, a miniaturized and intelligent E-Nose module could become a promising digital component, possibly integrating into the personal mobile phone as digital cameras have done. To fulfill this purpose, a small E-Nose must satisfy additional requirements such as low-power consumption and mass productivity through batch fabrication, together with sufficient sensing abilities in terms of sensitivity and selectivity.

Array-based gas sensors for an E-Nose system have been fabricated by using various different kinds of sensing materials: metal-oxide semiconductors [6], conducting polymers [7], and

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carbon black-polymer composites [8] for chemoresistive sensors; sorbent polymers [9] for quartz crystal microbalance and surface acoustic wave devices; and so on. Among them, arrays of carbon black-polymer composite sensors have recently received considerable interest due to their excellent capability in the classification, identification, and quantification of volatile-organic-compound (VOC) vapors [10]-[13]. These sensors are broadly responsive to various analytes unlike the traditional lock-and-key mechanism. Their sensing properties can be easily modulated by choosing organic polymers and optional additive plasticizers, and further by regulating relative quantities among them, which gives a great deal of flexibility in the sensor array design. In addition, they also provide an opportunity to fabricate a small-size, light-weight sensor array chip with low-power consumption because of their inherent ambient-temperature operation. A previous report has shown that the size of these sensors could be minimized up to $0.1 \text{ mm} \times 0.1 \text{ mm}$ by using the conventional microelectromechanical system (MEMS) technologies without any significant deterioration in sensing capability due to the reduction of the detection area [14].

Here, we report on the development of a miniaturized E-Nose module small enough to be directly attached to and operated by a personal digital assistant (PDA). Within our best knowledge, this system is the first PDA-based E-Nose system integrating all vital elements into a PDA-compatible size, although there have been some relevant investigations [15]-[18]. This system is composed of four major parts: mechanical components for sampling; a single 8-channel carbon black-polymer composite array chip fabricated on flexible polyimide substrate; sensor-interrogating and digital interfacing printed-circuit-boards (PCBs); and a real-time data acquisition program executed in a PDA. Actual performances of our array chip and E-Nose system have been evaluated for the classification of eight different VOCs and three natural oils, respectively. These results are described and discussed below in addition to the embodiment processes of the PDA-base E-Nose system.

II. Fabrication of PDA-Based Electronic Nose System

1. Single Flexible Sensor Array Chip

A sensor array substrate was prepared by modifying the fabrication methods used in a flexible-printed-circuit-board (FPCB). The fabrication processes were previously reported in detail [19] and are possible to produce in mass quantity due to their compatibility to the roll-to-roll process of the FPCB industry. Figure 1 displays two optical images for the front and back sides of a fabricated sensor array chip. The array chip

with a total size of $18 \text{ mm} \times 18 \text{ mm}$, except for the pad area for electrical connection located below, consists of eight gas sensors with an active diameter area of 2 mm. Each sensing element has a pair of interdigitated detection electrodes on the front side of the base polyimide film, while a single microheater is positioned on the back side. Both sides are protected by using two additional adhesive and polyimide sheets machined to have open windows only for the sensing and pad areas. The schematic diagram in Fig. 2 shows the cross-sectional layer structure of one sensing element.

The blackish circle-shape blots at the center region of the left picture in Fig. 1 are sensors made of different carbon black-polymer composites. The individual sensor was formed by the casting of a composite solution prepared by homogeneously mixing carbon black nanoparticles and different organic chemicals as described in [20]. The front cover film with open windows for the sensing areas allows the composite solution to be placed reproducibly in a specific and well-constrained area during the drop-coating process. Figure 3 shows the chemical structures of organic materials used in the 8-channel composite array chip. Two additive plasticizers, di(ethylene glycol) dibenzoate (DGD) and dioctyl phthalate (DOP), were used to attain a fast response and further diversity in the vapor detection characteristics [12].

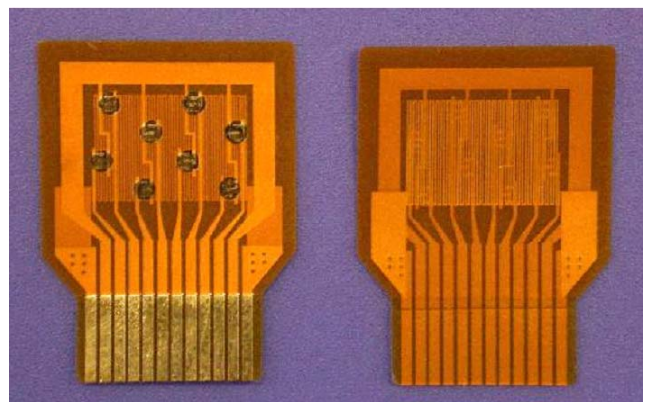


Fig. 1. Optical images for front (left) and back (right) sides of a single 8-channel sensor array chip.

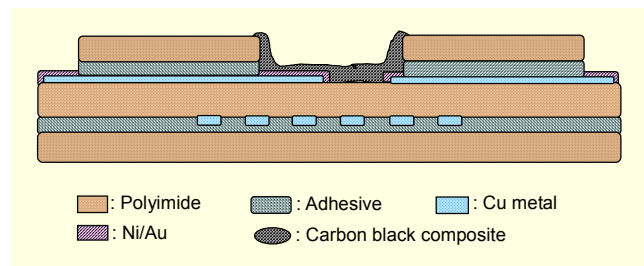


Fig. 2. Schematic cross-sectional structure for one sensing element of the sensor array chip.

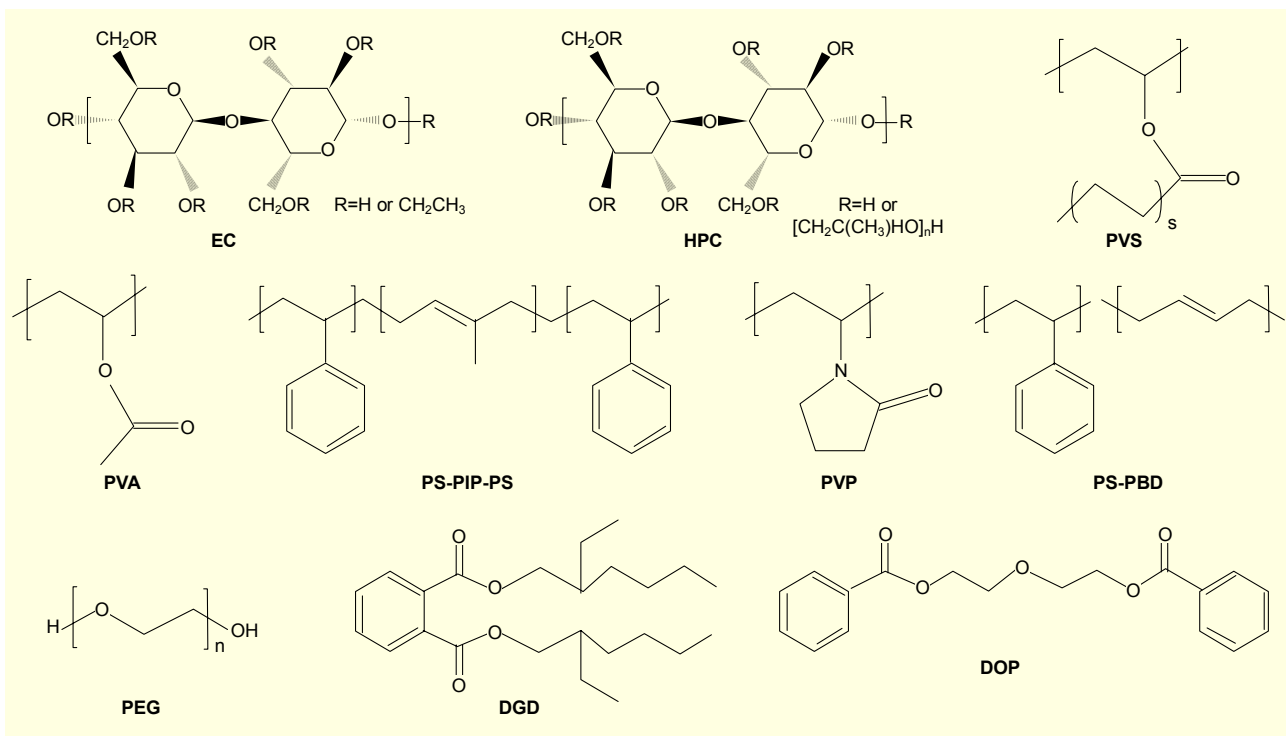


Fig. 3. Chemical structures of the polymers and two plasticizers used in this work listed with sensor channel number and chemical name [abbreviation]: 1, ethyl cellulose [EC]; 2, hydroxypropyl cellulose [HPC]; 3, poly(vinyl stearate) [PVS]; 4, poly(vinyl acetate) [PVA]; 5, polystyrene-black-polyisoprene-black-polystyrene [PS-PIP-PS]; 6, poly(vinyl pyrrolidone) [PVP]; 7, poly(styrene-co-butadiene) [PS-PBD]; 8, poly(ethylene glycol) [PEG]. Sensors 2 and 4 were plasticized with 50% DGD and the plasticizer of DOP was used for sensor 6.

In the carbon black-polymer composite sensors, the carbon black endows electrical conductivity to the films, whereas various polymers are the source of detection diversity. Swelling of the composite sensing layer upon exposure to vapor analytes generally leads to the increase in sensor resistance, thereby providing an extraordinary simple means for monitoring the presence of chemicals [10], [11].

2. Sensor Signal Processing Circuit Boards

In order to obtain sensor responses for analyte exposure, sensor-interrogating circuits were made on a small PCB with the size of 32 mm × 35 mm as shown in Fig. 4. These circuits simultaneously measure the resistance changes of all 8 sensors in the sensor array chip. They are composed of a voltage divider circuit, which is a standard method for measuring large resistance change, and a voltage follower circuit to avoid electrical interference between the resistance-measuring and post-processing circuits. The right picture of Fig. 4 shows the real arrangement of the discrete electric devices such as ICs, resistors, and diodes, while the left picture displays the reference resistors serially-connected to the sensors in the voltage divide circuit and the sensor array chip connected by

the surface-mounted-type FPCB connector (FH10A-12S-1SHB, Horose). This connector permits repetitive replacement of the array chip through movable locking flaps and high density in the electrical connections due to a small pitch dimension of 1 mm. The lower electrical pads in Fig. 4 are directly connected to the electrical socket of a digital interface card (DAQ 6062E, NI) inserted into the slot of a PDA (iPAQ 5550, HP). Measured multivariate analog signals are digitized and manipulated in the DAQ card, and then delivered to the memory of the PDA.

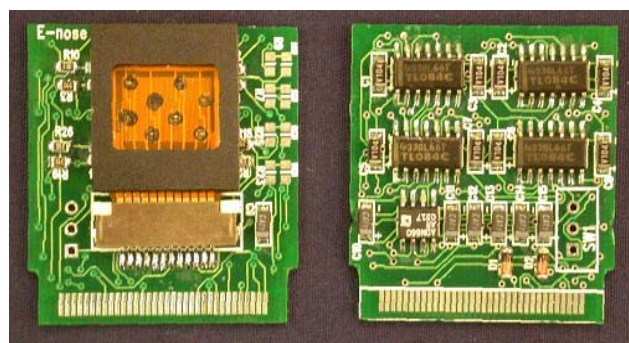


Fig. 4. Two pictures for front (left) and back (right) sides of the sensor interface PCB.

3. Real-Time Data Acquisition and Pattern Recognition

A data acquisition program is developed using a LabVIEW PDA module (NI), which compiles LabVIEW programs to run on PDA targets and download the completed application into the PDA. It samples and stores the multivariate data with a rate of about 2 Hz in real-time. The obtained data can be transferred to a desktop computer by Microsoft ActiveSync programs.

The classification work in an E-Nose system is carried out by running pattern recognition programs with the aid of feature parameters. In this work, the feature parameter is a relative magnitude in sensor resistance change, that is, the percentage ratio of maximum resistance change with respect to the initial resistance, which has been generally used in the case of a chemoresistive sensor system. The detection response, R_{det} , defined as a representative feature, is expressed by

$$R_{det} = 100 \times \frac{(R_{max} - R_0)}{R_0}, \quad (1)$$

where R_{max} is the maximum resistance of the sensor and R_0 is the stabilized initial resistance. Actually, the multivariate data were pre-smoothed with their neighboring points to suppress unwanted high-frequency noise. The pattern recognition was performed by using the principal component analysis (PCA) algorithm provided in the MatLab (MathWorks) software package.

A statistical approach was also utilized to define a quantitative metric for evaluating the resolving power of the sensor array chip in various VOC detection tasks, as previously reported [13]. The statistically defined metric is especially informative when the detection response is a linear function of analyte concentration because in such a case the concentration-normalized response patterns do not change with respect to the analyte concentration. To evaluate the magnitude of this metric, the points in d -dimensional space are projected orthogonally onto a line. In our sensor array chip, the classification problem in eight dimensions is transformed to one-dimensional work. When the data are projected into one dimension, it is desirable to maximize the distance between the means of the two classes being separated, while minimizing their within-class variation. Such a ratio can be expressed as a resolution factor RF by

$$RF = \frac{\delta}{\sqrt{\sigma_1^2 + \sigma_2^2}}, \quad (2)$$

where δ is the distance between the two class means, and σ_1 and σ_2 are the standard deviations of the two classes.

4. PDA-Based E-Nose System and Sampling Method

A schematic diagram of the PDA-based E-Nose system is

given in Fig. 5. In viewpoint of hardware, it consists of four major parts: sampling components; a sensor array chip and sensor interface PCB (the right-upward dashed box in Fig. 5); NI DAQ card for analog-to-digital conversion and digital interface (the downward dashed region in Fig. 5); and mobile PDA for data acquisition and processing. Figure 6(a) shows a real picture for the E-Nose system in a standby state. The top-extended accessory is a sensor module with the array chip and sensor interface PCB, while the DAQ card inserted into the PDA does not appear in Fig. 6(a).

Three commercially available essential oils extracted from mint, lavender, and eucalyptus plants were used as test samples for the E-Nose system. These samples are shown in Fig. 6(b) together with a dropper and a sampling board with a

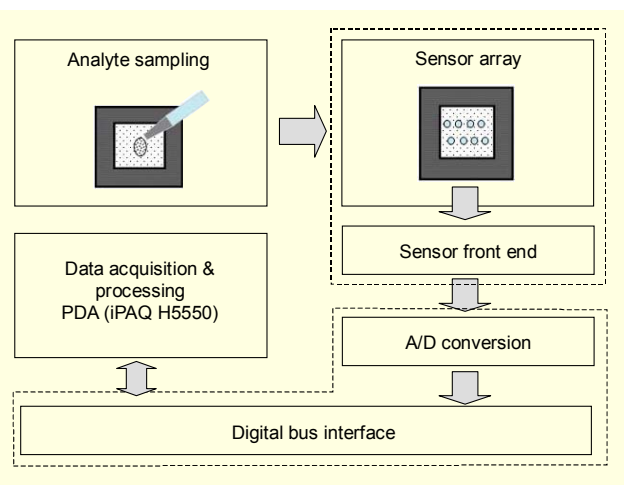


Fig. 5. Basic schematic diagram for the PDA-based E-Nose system.

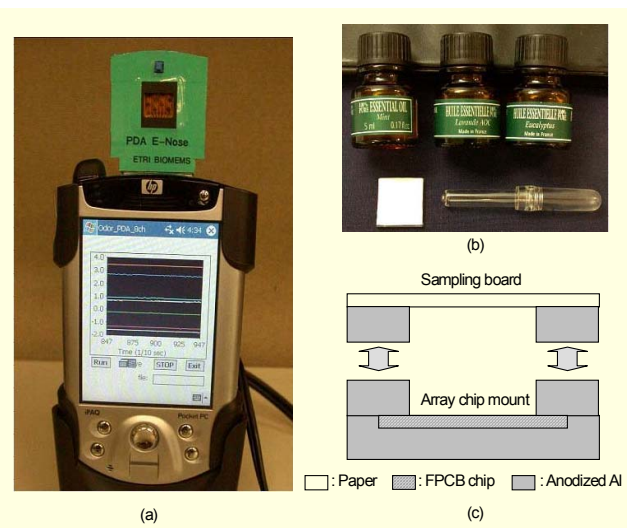


Fig. 6. Pictures for (a) PDA-based E-Nose system and (b) analyzed natural oils and sampling components, and (c) schematic diagram for a semi-headspace chamber with permeable filter paper.

rectangular piece of white filter paper (1004-150, Whatman) at the surface. The sampling method used here is based on the spontaneous evaporation of analyte absorbed by the cellulose paper. The sampling process is as follows: Using a dropper, transfer one drop of natural oil to the paper of the sampling board; wait a few minutes for the sampled liquid oil to permeate into the paper or evaporate to the ambient air; and then post the sampling board on a sensor array chip mount.

Since the chip mount is exposed to the air as demonstrated in Fig. 6(a), the sampling board can be easily attached to or detached from the chip mount by hand through an adhesive layer at the bottom side of the sampling board. Figure 6(c) shows the schematic cross-sectional structures of the sampling board and the chip mount. In an attached state, a small detection chamber with a vacant hexahedron of $10\text{ mm} \times 10\text{ mm} \times 2\text{ mm}$ is formed between the sensor array chip and the analyte-wetted paper. The analyte concentration is determined using the evaporation rate of natural oil from the sampled paper and the diffusion rate from the inside to the outside of the chamber due to the concentration gradient. Judging from the time profiles of the sensor responses in real measurements, the analyte concentration was fairly well saturated into the equilibrium value within a few minutes by the two driving forces, which implies that this method could be utilized as a sampling technique for the PDA-based E-Nose system.

In fact, we initially tried to test a closed detection chamber and the injection of analyte vapor by means of a gas-tight syringe instead of the semi-closed chamber previously described. However, we experienced some difficulties in achieving perfect gas-tight components and fast, easy gas exchange. In addition, the pumping-only sampling scheme, used previously in a portable E-Nose system based on a laptop personal computer [16], was also given up due to the bulk dimension and relatively high power consumption of the mini-diaphragm pump. Consequently, the PDA-based E-Nose system has adopted the manual semi-headspace sampling method with a permeable cellulose paper in order to fulfill the requirements of small size, simple manipulation, and low-power consumption.

III. Results and Discussion

1. Evaluation on the Flexible Sensor Array Chip

The fabricated sensor array chip was evaluated in terms of the detection response for various VOC vapor exposures in controlled conditions using the sampling method of flow injection. Gas-sensing measurements were carried out by placing the array chip in a small chamber with electrical feedthroughs, and by blowing air-diluted analyte vapor over it with a flow rate of 500 mL/min while simultaneously

monitoring resistance changes for the eight composite sensors in Fig. 3.

Figure 7 shows the typical response time-profiles of four sensors with channel numbers from 1 to 4 upon exposure to toluene vapor. The toluene vapor diluted in synthetic dry air was injected into the detection chamber for 2 minutes, which was followed by a resistance recovery interval of 5 minutes. The toluene concentration was regulated to increase from 40 to 2000 ppm for the successive measurements. The measurements show a gradual rise in sensor resistance with an increase of the toluene concentration. The baseline resistances of all used sensors are in the range of 2 to 200 $\text{k}\Omega$, and the maximum sensor resistances increase within 3 % of their initial stabilized values when exposed to 1000 ppm toluene. For example, sensor 2 with the largest sensitivity has an initial resistance of $46.76\text{ k}\Omega$ and a maximum value of $48.15\text{ k}\Omega$ for 1000 ppm toluene exposure, as shown in Fig. 7, which corresponds to a detection response of 3.0. Other important sensing characteristics include sensing-time behaviors, that is, the time to reach a saturated state for the duration of analyte exposure and to recover sensor resistance into an initial position. Even though these sensing times strongly depended on the sensing materials and analyte vapors used, they were found to be fast: the response and decay times were generally

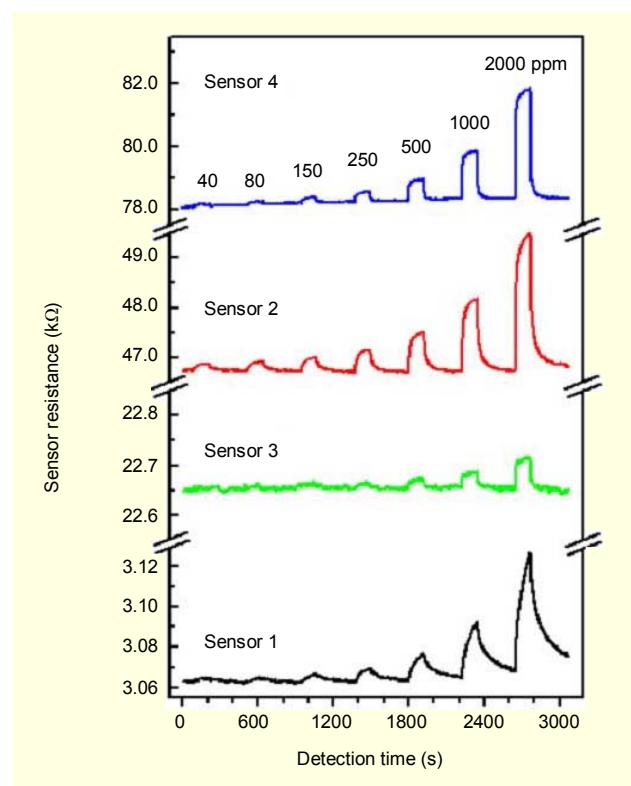


Fig. 7. Sensor response profiles of the four composite sensors from 1 to 4 for successive toluene vapor exposures, with the increase of concentration shown in ppm.

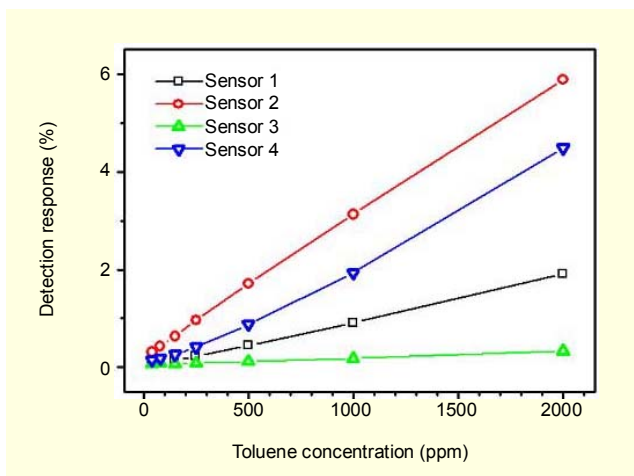


Fig. 8. Plots of detection response vs. toluene concentration obtained from the four sensors in Fig. 7.

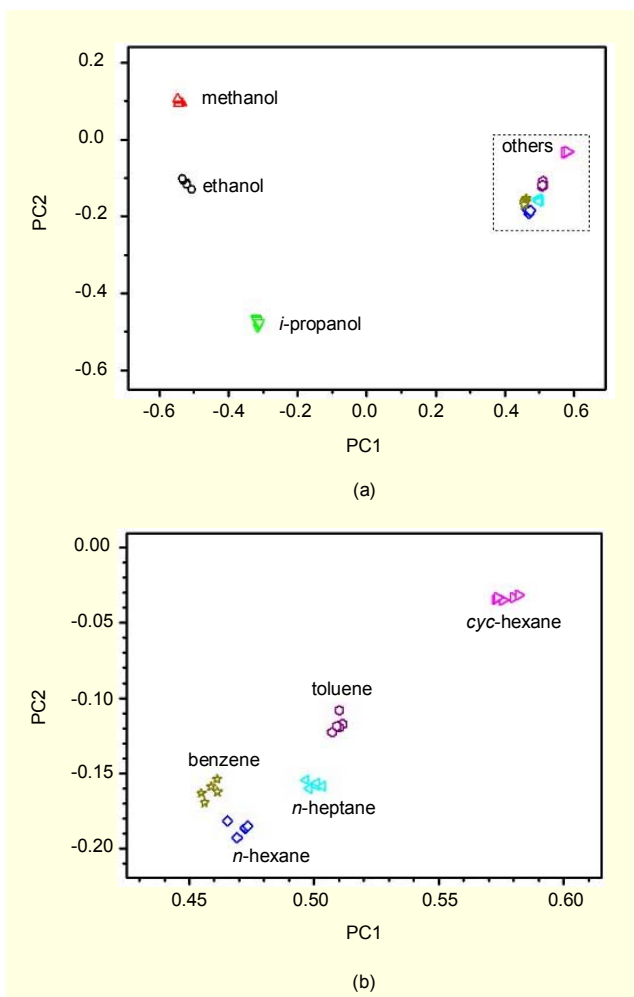


Fig. 9. (a) PCA plot calculated from the detection response patterns of the 8 different VOCs, as presented by the first two principal axes of PC1 and PC2, and (b) enlarged PCA plot of the dotted box in (a) for showing the visual distinguishability among analytes.

less than 1 minute and 5 minutes, respectively. These short times allow our sensor array chip to be utilized in a sensing system for point-of-care applications.

Figure 8 shows the dependences of detection responses on toluene concentration for the four sensors in Fig. 7. They follow a linear relationship between the toluene concentration and the resulting response magnitude within a measured concentration range of less than 2000 ppm. This behavior is consistent with the previous results obtained in the carbon black-polymer composite system [10], [11].

E-Nose systems that rely on array-based sensing require some type of training set and data-processing algorithm in order to classify an analyte upon presentation to the sensor array. In this respect, the performance and range of applicability of such sensor arrays are intimately coupled to the data reduction algorithms and the computational capabilities required to achieve the sensing task of concern. The linear concentration dependence of the detection response is ideal for the minimum possible training set and the minimum requirements on computational capabilities to classify a particular analyte. The PDA-based systems have more serious limitations in computational resources, so that the necessity for the linear response function becomes more important.

Additional flow-injection-based gas-sensing measurements were also performed to confirm the classification capability of the sensor array chip among various kinds of VOCs. These analytes include three alcohol compounds, methanol, ethanol, and *i*-propanol; three hydrocarbons, *n*-hexane, *n*-heptane, and *cyc*-hexane; and two benzene-like chemicals, benzene and toluene. Except for the 2000 ppm toluene, the concentration of these compounds was arbitrarily chosen to be 5000 ppm. The measurements were carried out five times for each analyte under the same experimental conditions.

The feature parameters were calculated using (1) from the measured sensing profiles for the eight VOCs. The multivariate data were further analyzed using the PCA algorithm, which is one of most extensively used statistical pattern recognition methods. This simple method can provide quantitative, visual results for the classification among different VOCs by expressing the response vectors in terms of linear combinations of orthogonal vectors, that is, principal components, along a new set of coordinate axes. These components account for a certain amount of variance in the data and are ordered so that the first principal component, *PC1*, has the largest amount of variance, followed by the next greatest component, *PC2*, and so on. Figures 9(a) and 9(b) show the obtained PCA results presented by the two most important principal coordinates, *PC1* and *PC2*, which account for the variances of 84.7 and 8.8 %, respectively. The three alcohol compounds appear to be

Table 1. Resolution factors quantifying the classification capability of the 8-element carbon black-composite sensor array.

	methanol	<i>i</i> -propanol	hexane	heptane	<i>cyc</i> -hexane	toluene	benzene
ethanol	12.1	22.3	53.3	61.7	65.7	60.2	24.8
methanol		48.0	80.8	110.0	113.9	100.6	27.5
<i>i</i> -propanol			58.5	76.3	85.4	73.5	22.5
hexane				3.6	16.5	24.8	7.6
heptane					20.0	34.0	8.0
<i>cyc</i> -hexane						27.8	7.0
toluene							0.9

well separated from other analyte groups, while the three hydrocarbons and two benzene-like groups are located nearby, as shown in Fig. 9(a). This implies that the alcohol groups are better recognized than those of hydrocarbon and benzene-like compounds in the used sensor array chip. Furthermore, the enlarged plot of Fig. 9(b) clearly demonstrates the capability of classifying or identifying the three hydrocarbons and the two benzene-like compounds.

In order to quantify the classification ability among VOC groups, the resolution factors were calculated in full 8 dimensions by using the equation defined by (2). Table 1 gives the obtained resolution factors among the analyzed eight VOCs. As expected above, the alcoholic VOCs have relatively high resolution factors of larger than 20 except for the classification task between methanol and ethanol with a value of 12.1. The two worst cases are benzene vs. toluene and *n*-hexane vs. *n*-heptane, which have resolution factors of less than 5. These cases can be rationally understood with the fact that they have the closest similarities in their chemical structures and properties.

2. Evaluation on the PDA-Based Electronic Nose System

The operation performance of the PDA-based E-Nose system was evaluated through the classification task among the three essential oils from mint, lavender, and eucalyptus plants. Each measurement for the oils was accomplished five times successively by saving the response time-profile data, which were composed of initial stabilized resistances for 1 minute, resistance variation upon 2-minute exposure to one of the natural oils, and a recovery profile in ambient air. Since the recovery time to restore the sensor resistance into an initial value was found to be larger than 15 minutes, these successive measurements were performed with a time interval of about 25 minutes. The pictures in Figs. 10(a) and 10(b) demonstrate the variation of sensor resistances for the oil vapor exposure and the following recovery process, respectively, during the real-

time operation.

The measured multivariate data proceeded to the same methods described above: extracting the detection response for each sensor as the feature parameter and performing the PCA program with the aid of these parameters. Figure 11 shows 3-dimensional (3D) response bar patterns, that is, a plot of the sensor response vs. the sensor number and measurement time, for the three analyzed oils: Fig. 11(a) for mint, Fig. 11(b) for lavender, and Fig. 11(c) for eucalyptus. These 3D plots provide information on the absolute sensing magnitude of each sensor element and the sensing reproducibility through repetitive measurements. Normalized polar plots are also given at the top-left corner in each figure. The three polar pattern shapes are found to be different according to the oil samples, suggesting the possibility of identifying the oils. Some of the detection responses are observed to be larger than one hundred times those of the previous case of VOC detection. The response variation according to the successive measurements is discussed below in conjunction with the pattern recognition results from the viewpoint of the reliability of the semi-headspace sampling method.

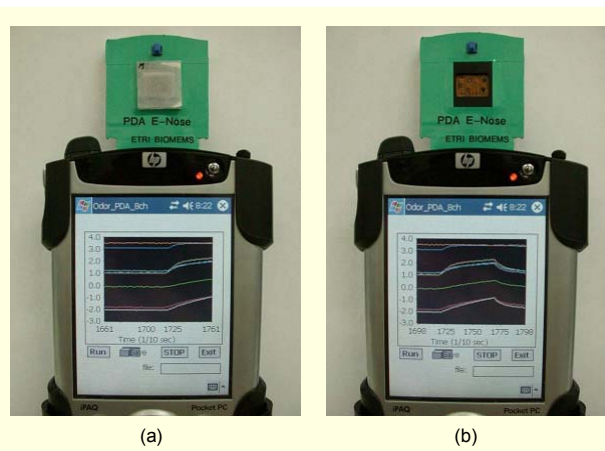


Fig. 10. Photographs taken during (a) the measuring period for the oil samples and (b) the following recovery process.

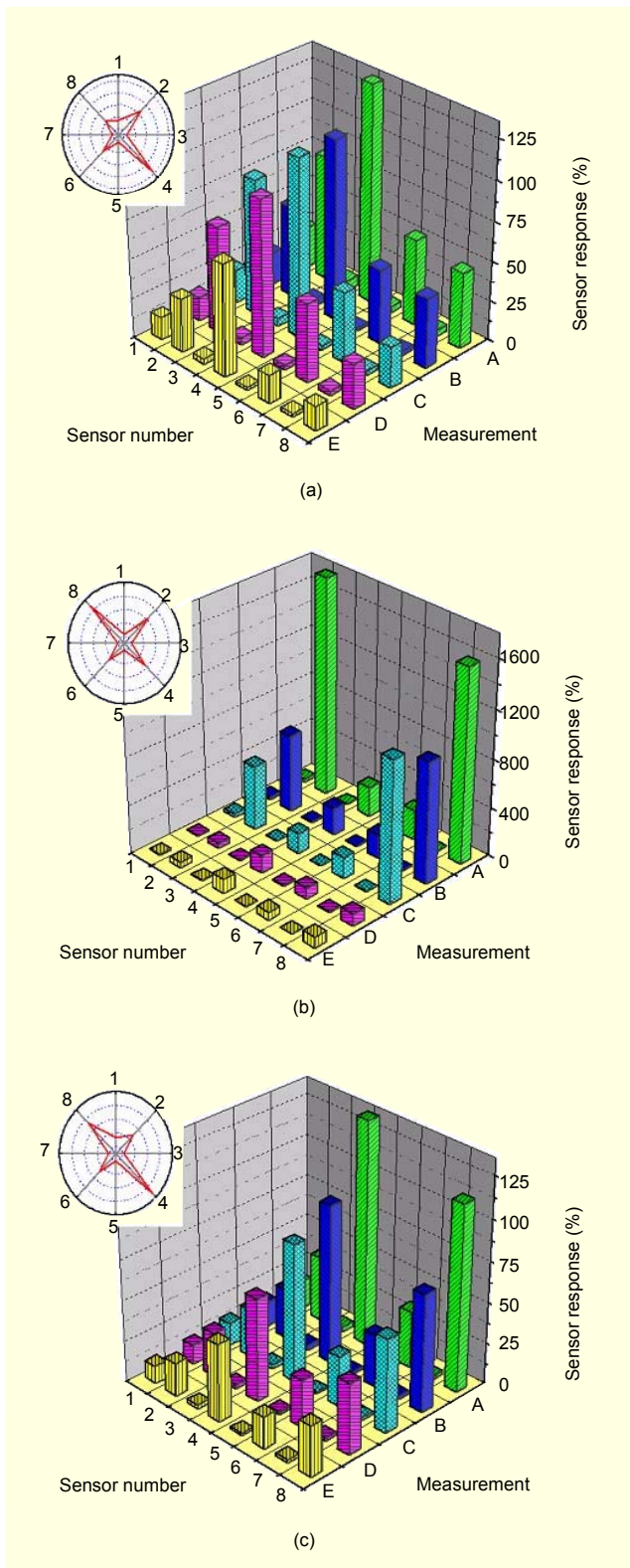


Fig. 11. Bar plots of the detection response magnitudes with respect to the sensor number for the five measurements of (a) mint, (b) lavender, and (c) eucalyptus oils. The inset polar plots are also presented for the normalized average detection responses.

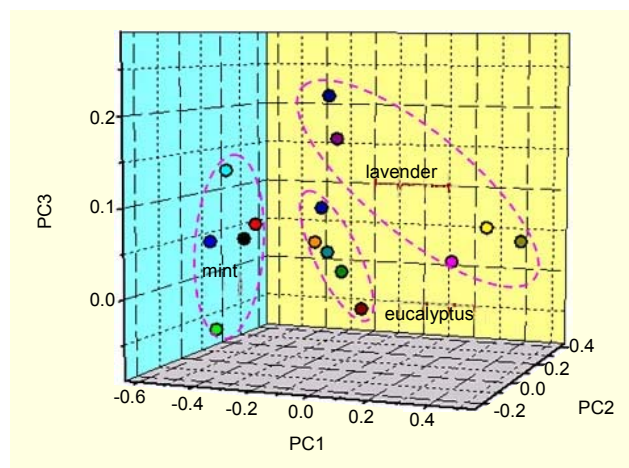


Fig. 12. PCA results obtained from five successive measurements for the essential oils of mint, lavender, and eucalyptus.

As direct evidence for the successful classification among the three oils, the PCA pattern recognition results are presented in Fig. 12. The dashed ellipsoids show the recognition boundaries of the three oils in the three principal axes of $PC1$, $PC2$, and $PC3$ with variances of 74.4, 21.4, and 3.5 %, respectively. Although there are some scatterings among the obtained data, these trained data sets have clearly separated regions defined by the boundaries so that we can recognize the natural oils, unambiguously. Considering that these results have been obtained from five successive measurements after only one sampling process for mint and eucalyptus, they show good grouping behaviors and are not strongly dependent on the elapsed time from the sampling. Since each measurement takes approximately half an hour, the final fifth measurement is performed two hours after the first one. However, this is not true in the measurements for lavender: successive measurements undergo significant reduction in the response magnitude and great movement in the PCA space. These observations could be interpreted with the change in partial vapor pressures of evaporated VOCs from the lavender-sampled board as a function of the elapsed time. Generally, the absolute total amount of evaporated VOCs becomes smaller as the time elapses, and the relative population of the remaining constituents in the sampling board can vary due to the difference in their volatility. If deduced from the PCA results, the relative constituent populations are not varied greatly in the cases of mint and eucalyptus, while the lavender suffers significant variations. In fact, the lavender data in Fig. 12 were obtained with repeated samplings. The three data positioned in the lower-right side were measured immediately after sampling, and the two upper-left circles correspond to the second measurements. These results suggest that the grouping behaviors can be greatly improved under carefully controlled

sampling conditions, which are very important in order to improve the recognition precision of the E-Nose system in practical tasks such as classifying unknown oils.

IV. Conclusion

A small E-Nose system based on a PDA has been developed and has demonstrated the successful classification between three essential oils extracted from mint, lavender, and eucalyptus plants. This system is composed of manually-operated sampling components, a sensor array chip, small sensor-interface PCBs, and data acquisition and processing programs. Among them, the key element of the E-Nose system is the 8-channel sensor array chip fabricated by using carbon black-polymer composites on a flexible polyimide substrate. Next, we will continuously try to miniaturize and improve the E-Nose system by means of the integration of an on-chip circuit and the use of MEMS components, for example, a valve, a pump, and a preconcentrator.

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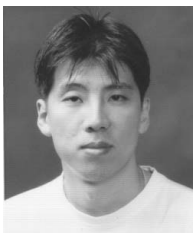
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