Odor Sensor System Using Chemosensitive Resistor Array and Machine Learning

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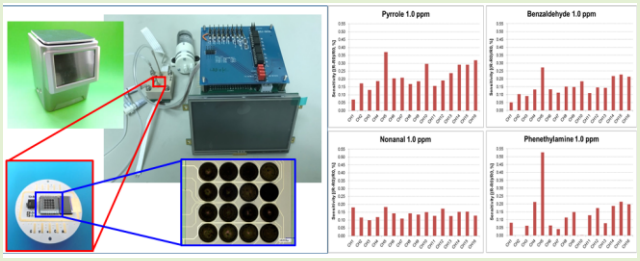
***Abstract*—In this study, we developed an odor sensor system using chemosensitive resistors, which outputted multichannel data. Mixtures of gas chromatography stationary materials (GC materials) and carbon black were used as the chemosensitive resistors. The interaction between the chemosensitive resistors and gas species shifted the electrical resistance of the resistors. Sixteen different chemosensitive resistors were fabricated on an odor sensor chip. In addition, a compact measurement instrument was fabricated. Sixteen channel data were obtained from the measurements of gas species using the instrument. The data were analyzed using machine learning algorithms available on Weka software. As a result, the sensor system successfully identified alcoholic beverages. Finally, we demonstrated the classification of restroom odor in a field test. The classification was successful with an accuracy of 97.9%.**



***Index Terms*—GC materials, carbon black, odor sensor, artificial olfaction, chemical sensor, sensor array, odor discrimination, Weka.**

I. Introduction

SENSORS artificially reproduce the senses of humans or animals. Among them, sensors corresponding to smell and taste are chemical sensors. In particular, animals use smell to monitor their surroundings and to recognize each other because volatile organic compounds (VOCs) in the air can indicate the signature of creatures. Animals realize monitoring and recognition using several hundred types of receptors. Therefore, materials with wide diversity are needed as receptors to recognize odors.

Many chemical sensors have been reported as electric nose (e-Nose) systems. These use various transducers such as metal oxide semiconductor field-effect transistors (MOSFETs), metal oxide semiconductors (MOSs), surface acoustic wave (SAW) devices, quartz crystal microbalance (QCM), conducting polymers, calorimetric, optical, carbon black composites, and carbon nanotubes [1]–[8].

We had previously given attention to an odor sensor system with 576 channel sensor [9], [14], [15]. Twenty four different composites of polymers and carbon black (CB) were used for the sensor system. The interaction with gas species and each composite changed the electrical resistance of the composite. 576-channel data of resistance changes were obtained using the sensor system when a gas was allowed to flow. The discrimination of three different gas species was realized by the principal component analysis (PCA) of the multichannel data. The gas species were 200 ppm of ethyl acetate, 500 ppm of acetone, and 100 ppm of ethanol.

In this study, an odor sensor system was fabricated using multichannel chemosensitive resistors and machine learning.

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TABLE I
GC MATERIALS AND THEIR PROVIDERS

CH	GC materials	Provider
1	Tetrahydrohyethylenediamine (THEED)	GL Science
2	N,N- Bis (2-cyanoethyl) formamide (BCEF)	TCI
3	LAC-3R-728	GL Science
4	Diethylene Glycol Succinate (DEGS)	Sigma Aldrich
5	Poly(ethylene succinate) (PES)	Sigma Aldrich
6	UCON 75-HB-90000	SCI
7	1,2,3- Tris (2-cyanoethoxy) propane (TCEP)	Sigma Aldrich
8	SP-2330	Sigma Aldrich
9	SP-2340	Sigma Aldrich
10	Diglycerol	TCI
11	Reoplex 400	GL Science
12	Poly [di (ethylene glycol) adipate] (PDEGA)	Sigma Aldrich
13	Poly (ethylene glycol) 4000 (PEG4000)	Sigma Aldrich
14	Poly (ethylene glycol) 20K (PEG20K)	USP REFERENCE
15	Poly (ethylene glycol) 20M (PEG20M)	SCI
16	Free Fatty Acid Phase (FFAP)	Sigma Aldrich

TCI: Tokyo Chemical Industry co., Ltd.; SCI: Sinwa Chemical Industries Ltd.

Gas chromatography stationary materials (GC materials) and CB were mixed to make the chemosensitive resistors. The GC materials are appropriate for gas sensing because they have excellent gas sorption ability and wide diversity (246 species are known). Sixteen materials were selected for aldehyde gas sensing on the basis of their McReynolds constant [10]. The reason why aldehydes were chosen was that they are often contained in human-derived VOCs [16]. One of our goals is to use our sensor system used for diagnostics using the smell of humans. The 16 materials used are shown in Table I. A complex of a GC material and CB interacts with gas species allowed to flow over the complex. The interaction can change the electrical resistance of the complex. CB particles have a string like structure and exist in GC materials, and they are entangled with each other to form a conductive path. When the GC materials interact with the gas, they expand and the CB conductive path changes. This is the mechanism by which the electrical resistance changes [11]. Sixteen different chemosensitive resistors were fabricated on a silicon chip with electrodes. Moreover, the multichannel data obtained from the 16 chemosensitive resistors were analyzed using machine learning. The classifier models evaluated in this study were obtained as Weka free software (Waikato Environment for Knowledge Analysis), which is data mining software with many machine learning algorithms. We successfully identified alcoholic beverages by sensing their odor using the sensor system. Finally, we demonstrated the classification of restroom odor in a field test.

II. MATERIALS AND METHODS

Table I [12] shows the names and providers of the GC materials. CB was obtained from Sigma Aldrich (St. Louis, MO, USA) and TOKAI CARBON (Tokyo, Japan). 2-phenethylamine (PHE), pyrrole (PYR), benzaldehyde (BZAL), and nonanal (NAL) were purchased from Wako Pure Chemical Industries (Osaka, Japan) as model gases.

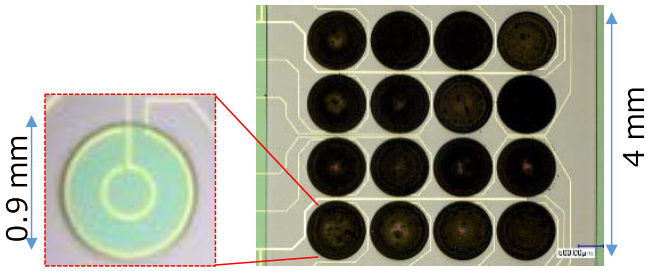


Fig. 1. Odor sensor chip of 16 channels and design of electrodes.

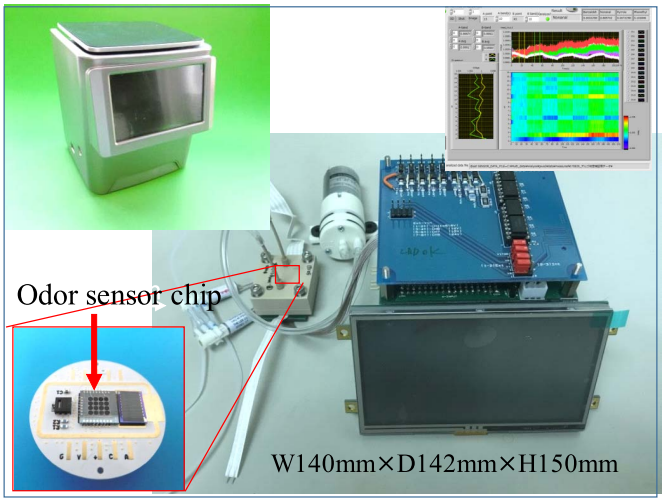


Fig. 2. Compact measurement instrument.

The fabrication process of the chemosensitive resistors was as follows. First, the GC materials and CB were separately dissolved in dimethylformamide. Next, solutions of one of GC materials and CB were mixed with a ratio of 1:1. After that, each mixed solution was added dropwise to a silicon chip with separated electrodes. The silicon chip was fabricated using a photolithography process and had 16 pairs of electrodes that were separated electrically. The 16 kinds of mixed solution were added dropwise to the 16 pairs of electrodes using an automatic spotting machine (customized machine, Musashi Engineering, Inc., Tokyo, Japan) with a needle syringe. After drying the solvent, an odor sensor chip with 16 channels was obtained (Fig. 1).

Finally, the odor sensor chip was embedded in a laboratory-built compact measurement instrument (Fig. 2). The measurement instrument consisted of a pump, a flow cell, A/D converters, a touch panel display, valves, and a computer unit. The sampling rate of the A/D converter was 1 kHz. The gas exposure profile was N_2:sample gas:N_2 =15 s:15 s:15 s. The following experiments were carried out using this setup and method.

III. RESULTS AND DISCUSSION

A. Evaluation of Machine-Learning Algorithms

First, we measured the responses to the four model gases of PHE, PYR, BZAL, and NAL using the sensor system. The results are shown in Fig. 3. The values in Fig. 3 were

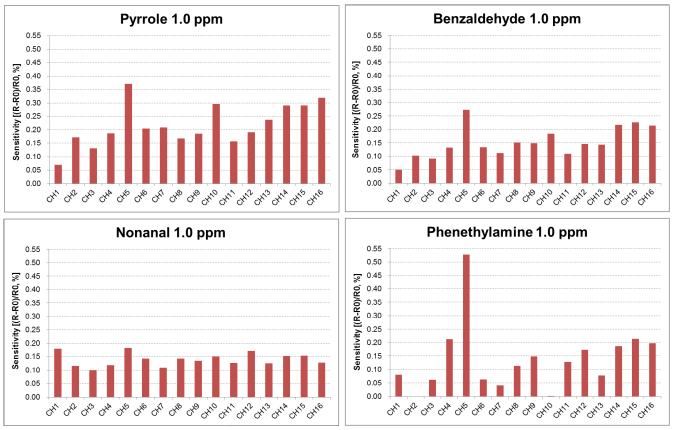


Fig. 3. Sensor responses to the four model gases.

TABLE II
RANKING OF F-MEASURE OF CLASSIFIERS

Rank	Classifier	TPRate	FPRate	Precision	Recall	FMeasure	ROCArea
1	meta.RotationForest	0.9250	0.0250	0.9432	0.9250	0.9258	0.9933
2	lazy.IB1	0.9000	0.0333	0.9061	0.9000	0.8985	0.9333
2	lazy.IBk	0.9000	0.0333	0.9061	0.9000	0.8985	0.9333
4	lazy.KStar	0.8917	0.0361	0.9075	0.8917	0.8918	0.9856
5	meta.ClassificationViaRegression	0.8750	0.0417	0.8863	0.8750	0.8734	0.9686
6	trees.FT	0.8750	0.0417	0.8765	0.8750	0.8712	0.9639
7	functions.Logistic	0.8667	0.0444	0.9077	0.8667	0.8648	0.9800
8	functions.MultilayerPerceptron	0.8417	0.0528	0.9067	0.8417	0.8384	0.9819
9	functions.SimpleLogistic	0.8333	0.0556	0.8470	0.8333	0.8350	0.9764
10	trees.LMT	0.8333	0.0556	0.8470	0.8333	0.8350	0.9764
11	meta.LogitBoost	0.8333	0.0556	0.8392	0.8333	0.8333	0.9583
12	trees.RandomForest	0.8333	0.0556	0.8480	0.8333	0.8311	0.9713
13	meta.MultiClassClassifier	0.8250	0.0583	0.8754	0.8250	0.8257	0.9622
14	meta.Decorate	0.8167	0.0611	0.8454	0.8167	0.8173	0.9583
15	meta.RandomCommittee	0.8083	0.0639	0.8439	0.8083	0.8102	0.9425
16	functions.RBFNetwork	0.8000	0.0667	0.8379	0.8000	0.7999	0.9294
17	trees.J48graft	0.7583	0.0806	0.7745	0.7583	0.7567	0.8357
18	meta.RandomSubSpace	0.7583	0.0806	0.7896	0.7583	0.7561	0.9535
19	meta.END	0.7500	0.0833	0.7764	0.7500	0.7494	0.9257
20	trees.J48	0.7500	0.0833	0.7644	0.7500	0.7492	0.8343

calculated using

$$\Delta R = \frac{R - R_0}{R_0} \tag{1}$$

where R is the resistance when the sensor material reacts with the sample gas and R_0 is the resistance when the material reacts with 100% nitrogen gas. The concentration of the sample gas was 1 ppm. A different pattern of the response to each model gas was obtained using the sensor system. We carried out three experiments for each concentration of 1000, 750, 500, and 250 ppb. We thus acquired a dataset of {4 gas species $\times$ 4 concentrations X 3 trials = 48} for machine learning.

Next, about 40 classifiers that were useable on Weka software were evaluated to identify the gas species. The procedure was as follows. First, three different datasets were acquired through experiments conducted on different day. Two of these datasets were used for the learning process. The other dataset was used to validate the results of machine learning. We carried out tests using three different combinations of the datasets for cross-validation. The classifiers with the top 20 F-measures are shown in Table II, where the F-measure is the harmonic average of the precision and recall. The highest overall rank was obtained by meta.RotationForest.

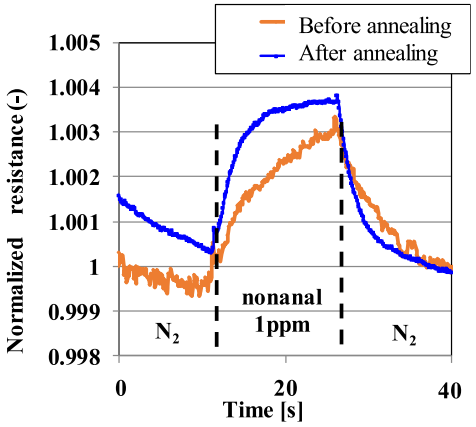


Fig. 4. Waveforms of sensor response before and after annealing process.

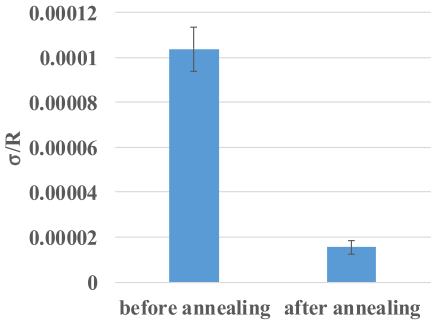


Fig. 5. Effect of annealing on noise magnitude (n=4).

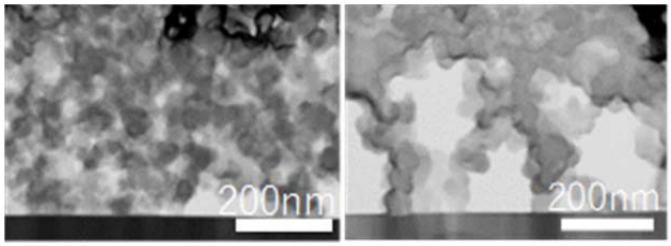
Therefore, meta.RotationForest was selected as the machine learning algorithm with the highest performance.

B. Effect of Annealing Process

We evaluated the annealing process to improve the sensor sensitivity. The sensitivity is determined from the noise level and response magnitude. The sensitivity increases with decreasing noise level and increasing response magnitude. We thought that the noise level would decrease if the CB particles of the sensor materials could be aggregated by an annealing process, because the dispersibility of the CB we used was poor and aggregation may result in a stable conduction path. Therefore, the fabricated sensor chips were annealed in vacuum.

Figs. 4 and 5 respectively show the waveforms and noise magnitudes of the sensor response before and after annealing. The noise magnitude was decreased after annealing. The variability rates were calculated from the mean and standard deviation of the response to nitrogen gas to quantify the noise level. The variability rate was 0.000103 before the process and 0.000015 after the process; thus, the noise level was decreased more than fourfold.

In addition, the response speed increased. Fig. 6 shows transmission electron microscope (TEM) images of a sensor material before and after annealing. The particles of CB after the process were aggregated compared with those before the process. This result indicated the reason for the decreased noise level. In addition, the network of CB particles became porous. This indicated the reason for the increased response



(a) Before annealing (b) After annealing

Fig. 6. TEM images of a sensor material.

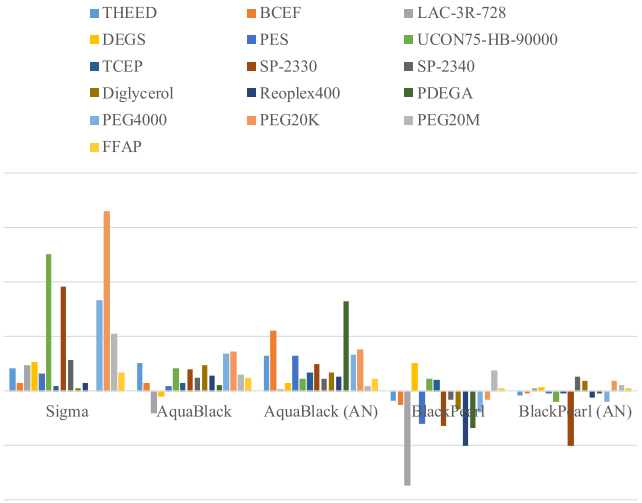


Fig. 7. Sensor responses to benzaldehyde of 600 ppb when the type of CB was changed.

speed, i.e., gas molecules were able to reach the sensor materials in a shorter time owing to the larger surface area. In conclusion, the annealing process reduced the noise level of the sensor and increased the response speed.

C. Change in Response Characteristics With the Type of Carbon Black

The GC materials were evaluated by a QCM sensor before forming the chemosensitive resistors in our previous study [12]. In the study, we choose 21 types of GC materials on the basis of the McReynolds constant [10]. Then, 16 of the GC materials were determined from the results of experiments using a QCM sensor. However, for some GC materials, the responses of the QCM sensor did not match those of the chemosensitive resistors. Therefore, we investigated the change in the response characteristics with the type of CB.

We prepared three types of CB: Sigma, Aqua Black, and Black Pearl. For Aqua Black and Black Pearl, we evaluated the annealing effect. Fig. 7 shows the results. The graph shows the response to 600 ppb of benzaldehyde. Even with the same GC material, the response characteristics changed when the type of CB was changed. This means that not only the type of GC material but also the type of CB will change the response characteristics of chemosensitive resistors to gases. This is highly advantageous for a gas sensor for identifying gases. This is because the recognition ability is improved since there are many sensors having various response characteristics to a certain gas. However, the mechanism behind the change in the characteristics of chemosensitive resistors with the type of CB is not clear, but we hope to clarify in our future work.

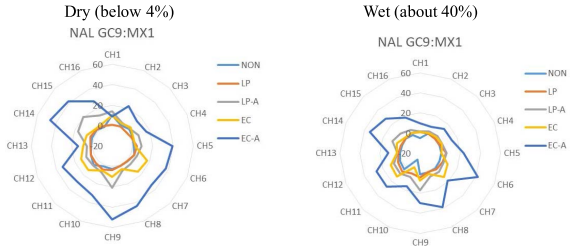


Fig. 8. Results of the effect of humidity on the sensor response. The humidity of the dry condition was below 4%. That of the wet condition was about 40%. “LP” means DSPE-PEG. “EC” means ETHOCEL. “NON” means that LP/EC was not added. “-A” means that the mixed materials were annealed.

D. Effects of Sensor Response on Humidity and Mixing of Hydrophilic and Hydrophobic Polymers

Many chemical sensors are known to be affected by humidity. Many experiments are generally performed in a laboratory with low humidity, in practice, sensors are used indoors and outdoors, where the humidity may be high.

Therefore, the effect of humidity on the sensor response was examined. In addition, to lessen this effect, a hydrophilic polymer and a hydrophobic polymer were mixed with the GC materials, and experiments were performed to determine which was more advantageous. 1,2-distearoyl-sn-glycero-3-phosphoethanolamine-N-[amino(polyethylene glycol)-2000] (DSPE-PEG) was used as the hydrophilic polymer, and ETHOCEL (ethyl cellulose) was used as the hydrophobic polymer. The ratio of the polymer and the GC materials was 1:9. Experiments were carried out using the four model gases, and the results for the NAL gas are shown in Fig. 8.

First, the responses of almost all sensors were reduced at the higher humidity. Second, the LP-mixed GC materials showed the same responses as nonmixed GC materials. Finally, although the EC-mixed GC materials showed the same responses before annealing, the responses greatly increased after the annealing. In conclusion, the highest responses were obtained under humidity of about 40% using GC materials mixed with the hydrophobic polymer after the annealing process.

E. Discrimination of Alcoholic Food Beverages

We evaluated the sensor system using reagents as gas species. However, evaluations using gases containing various volatile compounds are required to demonstrate the use of the system for real applications. For this purpose, we demonstrated the discrimination of different types of alcoholic beverages. A dataset of 40 trials for one beverage was acquired for learning. Eight tests were carried out to evaluate a beverage. The sensor materials used were made from a mixture of CB and GC materials (without the hydrophobic polymer). The materials were annealed. The accuracy was 100%. However, different types of alcoholic beverages have different concentrations of ethanol, the discrimination might have been

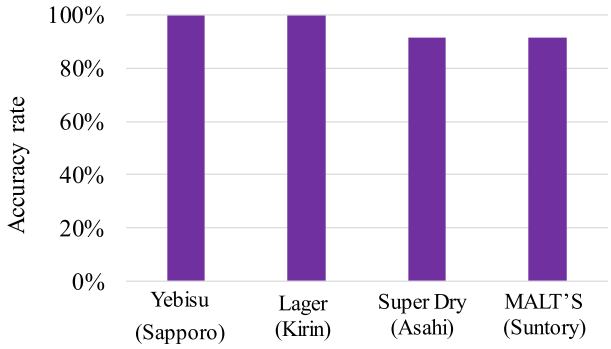


Fig. 9. Results of discrimination of four brands of beer.

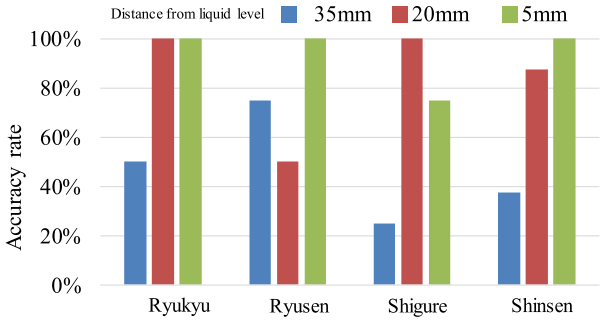


Fig. 10. Results of discrimination of four brands of Awamori.

Fig. 11. Field test in a restroom. The sensor materials used were made from mixture of CB and GC materials (without the hydrophobic polymer). The materials were annealed.

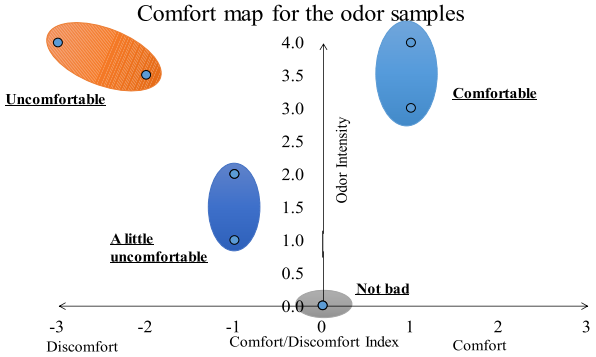


Fig. 12. Comfort map of the four classes.

based solely on the concentration of ethanol vapor. Therefore, we demonstrated the use of the sensor to distinguish alcoholic beverages with the same alcohol percentage. We used two kinds of alcoholic beverage, beer and Awamori, where the latter is a distilled alcoholic beverage mainly produced in the Okinawa region of Japan. The alcohol percentages of beer and Awamori were adjusted to 5% and 30%, respectively.

First, we discriminated four brands of beer; Yebisu (Sapporo), Lager (Kirin), Super Dry (Asahi), and MALT'S (Suntory). Fig. 9 shows the results for beer, where the gases were sampled from the headspace. The accuracy rate was above 90%.

Next, the discrimination of four Awamori brands was carried out under different conditions; the gases were sampled from the headspace of the bottle while changing the distance from the liquid level to. 5, 20, and 35 mm. The results are shown in Fig. 10. The accuracy rate (F- measure) was almost 100% for the distance of 5 mm. However, the accuracy rate decreased with increasing distance.

F. Field Test for Judging Odor of Restroom

In the above experiments, we evaluated our sensor system in a laboratory environment with a low level of disturbance. However, the sensor system must be able to perform robust measurements to enable actual applications. We carried out a field test using the sensor system to evaluate its robustness. As a field test, the sensor system was used to judge the odor in a restroom.

There are many buildings in cities in Japan. These buildings may have a huge number of restrooms. Currently, the restrooms are cleaned regularly as part of the maintenance of each building. However, this is a growing problem owing to a shortage of labor in Japan. If the frequency of maintenance can be reduced without sacrificing comfort, it will be possible to solve this problem. If the odor in a restroom is monitored using the sensor system, the restroom needs to be cleaned only when the odor makes users uncomfortable, thus the frequency of maintenance can be reduced. We demonstrated the judgement of odor in a restroom using the sensor system in a field test.

First, odor samples were obtained from four different locations in two different restrooms (the total number of locations was eight). Fig. 11 shows the experimental setup. The air was measured by the sensor system via gas samples obtained using a Tedlar gas bag. The experiments were carried out across multiple days. The number of samples taken at each location was about 70 to 100 and the total number of samples was 580.

Next, the obtained gas samples were divided into four classes by two persons trained as sniffers. The classes were "Uncomfortable", "A little uncomfortable", "Not bad", and "Comfortable". The results are shown in Table III. The data obtained by the sensor system were classified using the results of the discrimination by humans.

The intensity and comfort/discomfort index of each class were evaluated by a six-step odor intensity display method and a nine-step odor discomfort display method [13]. The intensity display method discriminates odors as 0: none odor, 1: threshold odor, 2: identifiable odor, 3: easily identifiable odor, 4: strong odor, and 5: very strong odor. The discomfort display method discriminates odor as -4: extreme discomfort, -3: strong discomfort. -2: discomfort, -1: slight discomfort, 0: normal, +1: slight comfort, +2: comfort, +3: strong comfort, and +4: extreme comfort. However, +3 and +4

TABLE III
NUMBER OF SAMPLES IN EACH ODOR CLASS DISCRIMINATED BY HUMAN SNIFFERS

	Odor class	Number of samples
A	Uncomfortable	154
B	A little uncomfortable	160
C	Not bad	157
D	Comfortable	109
	TOTAL	580

TABLE IV
RESULTS OF THE PREDICTION OF THE CLASSIFICATION USING MACHINE LEARNING

Accuracy rate 97.9%		Prediction by machine learning			
		A	B	C	D
Classification by human sniffers	A	147	0	6	1
	B	1	160	0	0
	C	2	0	143	1
	D	0	0	1	98

are rare. Fig. 12 shows the results. The two data points in each class were obtained by the two different sniffers. An important point is that the "Uncomfortable" class and the "Comfortable" class had similar intensities despite their very different comfort/discomfort indexes.

Finally, the prediction of the classification was performed by machine learning. First, the classified data were divided into 10 datasets with the same proportion of the total number of samples in each class. The datasets were used for the learning process and test process, with the datasets used for the learning process not being used for the test process. The accuracy of machine learning was cross-validated using the datasets. The results are shown in Table IV. The accuracy rate was 97.9%. We thus succeeded in prediction with a high accuracy rate in a real environment using the odor sensor.

IV. CONCLUSION

We developed an odor sensor system with 16 different chemosensitive resistors made from composites of GC materials and CB. The responses of these chemosensitive resistors to each gas species were different each other. The odor sensor system outputted four different datasets of the response of 16 channels when the gases of PHE, PYR, BZAL, and NAL gases with concentration of 1000, 750, 500, and 250 ppb were allowed to flow over the sensor. We evaluated 20 classifiers that were available on Weka software using the obtained data, and the best classifier for our sensor system was found to be meta. RotationForest. The discrimination of alcoholic beverages was successful using our system, and we obtained a high accuracy rate. Finally, a demonstration of classifying the air in a restroom was carried out as a field test, and an accuracy rate of 97.9% was achieved. In our future work, we will increase the number of channels to improve the recognition capability. In addition, we will investigate other applications such as those in healthcare and security.

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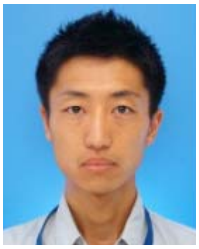

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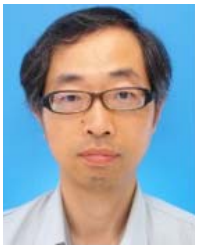

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