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Measurement of Basic Taste Substances by a Fiber Optic Taste Sensor Using Evanescent Field Absorption

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A multichannel taste sensor using evanescent field absorption in fiber optics was developed, and its characteristics were evaluated for several substances with basic and mixed tastes. Optical response patterns were obtained from two kinds of sensing membranes; one is a dye/silicon polymer membrane, and the other is a dye/lipid/PVC-PVAc-PVA copolymer membrane. The sensor output exhibits different patterns for chemical substances that are responsible for tastes such as salty, sour, bitter, sweet, and umami. This optical fiber taste system was successfully applied in testing several commercial soybean sauces and ionic drinks. Principal component analysis (PCA) was utilized to express these taste qualities quantitatively on the basis of the output patterns of the taste sensor. These subtle differences are difficult to differentiate with the human tongue, but our experimental results demonstrated that it is possible to discriminate between tastes by the difference in their output patterns.

1. Introduction

Taste is classified into five basic qualities elicited by many substances (i.e., sweetness, saltiness, bitterness, sourness, and umami). In addition, the mixture of basic tastes brings

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out tastes of spiciness, astringency, palatability, acridity, metalicness, colloid and so on. The sense of human taste depends on complex physical and mental conditions, in addition to the appearance, including color, flavor, aroma, texture, nutritional attributes and microbial contents. The reason for this complexity in taste recognition is that the sense of taste develops more slowly than other human senses such as touch, vision, hearing and smell.

Thus far, the analysis of taste has mainly depended on methods of sensory evaluation by humans, but this method is easily affected by the health or mental condition of the testers. In addition, the human tongue is easily dulled by repetitive testing and needs to be refreshed with water or a slice of bread. These problems are related to the difficulty of quantitative analysis, the complexity of the analytical process and the long time involved, and other issues. Therefore, it is necessary to develop novel methods for measuring tastes objectively, qualitatively and quantitatively. During the last ten years, many electronic taste sensors[1–4] based on potentiometric measurements have been developed to detect basic tastes and to analyze beverages such as coffee, mineral water, wine, soy sauce, fruit juice, and tomato juice. These electronic tongues use various lipid membranes as sensing materials and show specific responses to taste substances or metal ions. However, the lipids are easily soluble in water, which results in poor reproducibility. If the polymer membrane including a specific dye sensitive to taste substances is applied to an optical taste sensor system, the system is stable in water and has better reproducibility than a lipid membrane system. Also, if the membrane is thicker than the penetration depth of the evanescent field, the sensor responds independently of the taste substance's color, viscosity and various other characteristics. Therefore, in this study, an optical taste sensor based on evanescent field absorption[5–6] in an optical fiber was developed. Its characteristics were evaluated for two kinds of sensing membranes (dye/silicone polymer and dye/lipid/PVC-PVAc-PVA copolymer membrane), and output patterns were obtained for substances with five basic and mixed tastes. This sensor was developed specifically to classify and identify five basic tastes corresponding to chemical stimuli. It has a wide dynamic range and high sensitivity to low concentrations. It is possible to make measurements in weak light-absorbing media as well as in strong absorbing ones. The sensor system is very simple, easy to handle and is inexpensive. Moreover, the results data from it can be expressed in a two-dimensional plane by statistical analysis (i.e., principal component analysis, PCA). The characteristics of multidimensional sensor signals obtained from the six sensors were analyzed using the PCA technique. We can analyze the possibility of classifying a species using PCA. The PCA technique is well known as a method of data analysis for data reduction and visualization of complex nonlinear data with multiple dimensions. This sensor can be applied to on-line monitoring of fermentation or maturity, detection of toxicity, quality evaluation of foods and so on in the food industry, in place of sensory evaluation.

2. Measuring Principle

The fundamentals of optical waveguide theory have been presented by Kang *et al.*[5] In evanescent wave measurements with polymer-coated plastic fibers, the silicone cladding leads to total reflection of the measuring light at the core/cladding interface of the fiber. The angle of incidence is greater than the critical angle α_c (where $\alpha_c = \sin^{-1}(n_2/n_1)$), which is dependent on the refractive indices ($n_1 > n_2$) of the core and cladding materials. Due to the interaction of incident and reflected light, a standing wave exists close to the interface which penetrates a small distance into the cladding as the so-called evanescent wave. The electric field of an evanescent wave generated at the interface decays exponentially with distance from the interface. The depth of penetration (d_p),[6] defined as the distance required for the electric field amplitude to fall to 1/e of its value at the interface, is given by

$$d_p = \frac{\lambda_0}{\left\{2\pi n_2\left[\sin^2\alpha - (n_2/n_1)^2\right]\right\}^{1/2}}, \quad (1)$$

where λ_0 (400 nm ~ 700 nm for white light) is the free space wavelength of the incident beam n_1 (1.457) and n_2 (1.40) are the refractive indices of media core and cladding, respectively and α (90°) is the incident beam angle with respect to the interfacial normal. The theoretically determined penetration depth in this sensor is below 0.4 μm. Incidentally, the thickness of the sensing membrane is 4 μm and the evanescent field absorption was interpreted based on aspects of the taste solutions such as color, viscosity, and other physical characteristics. Figure 1 is a block diagram of a sensor and shows the generation of an evanescent field.

3. Materials and Methods

3.1 *Preparation of substances with representative tastes*

Five basic substances — glucose (sweetness), HCl (sourness), quinine-HCl (bitterness), monosodium glutamate (MSG ; umami), and NaCl (saltiness) — were dissolved in distilled water at concentrations ranging from 1×10^{-7} M to 1×10^{-2} M. The detection limit over the range of threshold taste values (TTVs) is sensitive for humans. Furthermore, to measure the interaction or influences between the basic tastes, each mixture (for example, glucose and HCl, quinine-HCl and glucose, NaCl and HCl) was increased in concentration

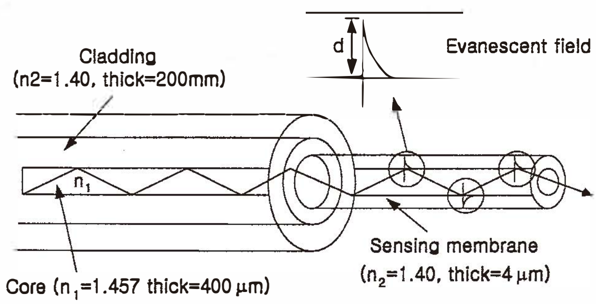

Fig. 1. Schematic diagram of sensor.

for additional taste. Distilled water was used for all samples and reference solutions. The test taste solutions were not controlled in terms of pH or ionic strength. In conventional chemical sensors, these control processes must be preceded and are an important factor. However, the taste sensor differs to the conventional chemical sensor, in that the pH and ionic strength of factors affects the final taste in each test solutions. Therefore, in taste sensors, these effects need not control or determine, only need to detect a "final taste" and the patterns for different tastes.

3.2 *Preparation of the sensing membrane*

There are two kinds of membranes: one is the dye/silicone membrane; the other is the dye/lipid/PVC-PVAc-PVA copolymer membrane. The former membrane is composed of 1×10^{-3} M dye, a silicone polymer, and the latter is composed of 1×10^{-3} M dye, 70 wt.% lipid, and 30 wt.% PVC-PVAc-PVA. Six kinds of potentially sensitive dyes were used for each sensing membrane (Rhodamine B (Sigma-Aldrich Co.,), NK1939,[7] NK2272,[7] NK2606[7] (Nippon Kankoh-Shikisho Co.), TMX (tetramethyl murexide), and 6TF (DOJINDO Co.)) .[8] These dyes showed specific characteristics for the basic taste substances of glucose (sweetness), HCl (sourness), quinine-HCl (bitterness), MSG (umami) and NaCl (saltiness), respectively. The silicone polymer was selected as a matrix for sensing membrane 1 because it had the same refractive index as optical fiber cladding. Figure 2 shows the molecular structure of the dyes. Membrane 2 was composed of two kinds of lipids, (decyl alcohol and bis (2-ethylheyl) hydrogen phosphate). The 12 sensing membranes used in the multichannel taste sensor are listed in Table 1. Each prepared material was dissolved in toluene and coated on the surface of the fiber core stripped

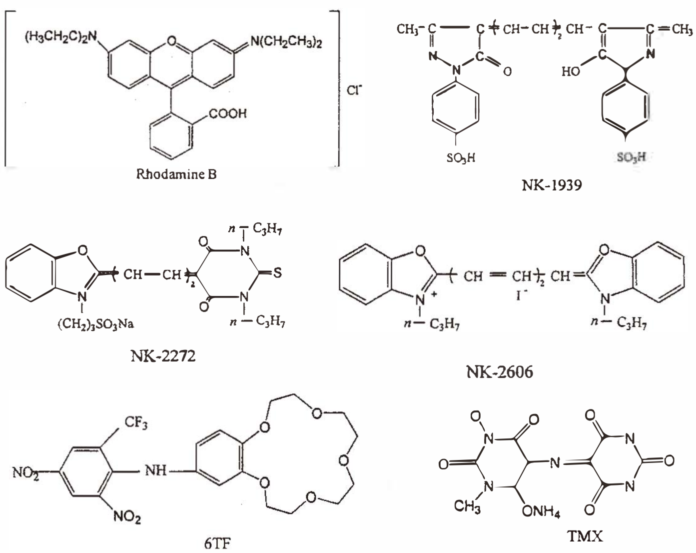

Fig. 2. Molecular structures of the six dyes.

Table 1.
The composition of each sensing membrane.

Mem. No.	Channel No.	Matrix	Dye
Mem. 1	1	Silicone polymer	Rhodamine B
	2	Silicone polymer	NK1939
	3	Silicone polymer	NK2272
	4	Silicone polymer	NK2606
	5	Silicone polymer	Tetramethyl murexide
	6	Silicone polymer	6TF
Mem. 2	1	DA*/PVC-PVAc-PVA	Rhodamine B
	2	BEHP*/PVC-PVAc-PVA	NK1939
	3	DA:BEHP*(1:1)/PVC-PVAc-PVA	NK2272
	4	DA/ PVC-PVAc-PVA	NK2606
	5	BEHP/ PVC-PVAc-PVA	Tetramethyl murexide
	6	DA:BEHP(1:1)/PVC-PVAc-PVA	6TF

* DA is notation of decyl alcohol, and BETH is bis (2-ethylheyl) hydrogen phosphate.[4]

cladding. It was then coated by dipping and drying at room temperature for 5 h. Otherwise, the optical fiber stripped cladding was cleaned in acetone and both fiber end-faces were polished with fiber polisher (Lake Bluff, IL. USA) to enhance the in-coupling of optical light. The thickness of the membranes was about 4 μm as measured from an SEM photograph. The dried membrane was equilibrated in distilled water and then measured in order to establish a reference value.

3.3 *Experimental setup*

We fabricated a multichannel optical taste sensing system based on evanescent field absorption in an optical fiber. The optode was made of a plastic-clad-silica (3M Co., FP-400-UHT) fiber 10 cm long with a 400 μm diameter core. The cladding of the fiber was removed for three centimeters at the middle portion and then the sensing membrane was coated on the surface of the core. Its thickness was about 4 μm. The sensor system was composed of a white LED light source and a silicon photodiode detector (detection wavelength: 400 nm ~ 1100 nm). The output signal was converted into a voltage by a current-voltage (I-V) converter, amplified and then fed into a computer. Distilled water was used as the reference to evaluate the output from taste substances and then the output voltage, Vo. After the sample solution was pumped by the motor, the output voltage was then V. In this way, a sensor response, namely, the relative intensity, $RI = V/Vo$, was obtained. Figure 3 shows a schematic diagram of the experimental setup of the taste sensor.

4. Results

4.1 *Response patterns of the sensor to basic taste substances*

The selected dyes show different characteristics for reactions with the five basic taste substances (glucose (sweetness), HCl (sourness), quinine-HCl (bitterness), MSG (umami),

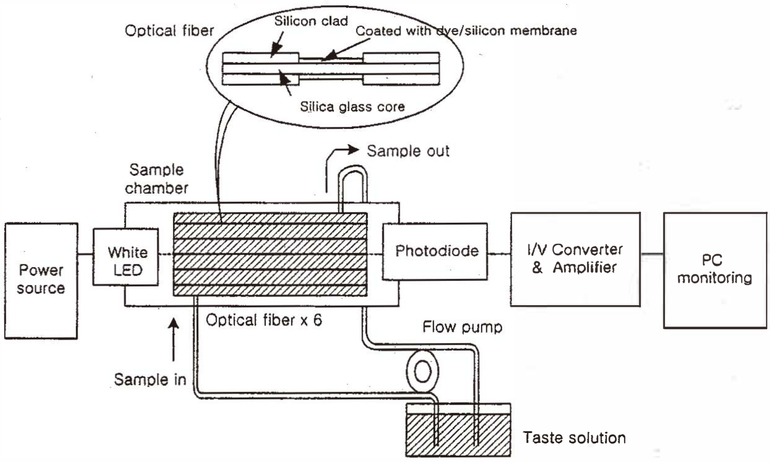

Fig. 3. Schematic diagram of taste sensor system.

and NaCl (saltiness)). The UV-visible spectrophotometer provided estimated values in the solutions. Sensing membrane 1 based on these results was fabricated and attached to the optical fiber sensor system and its responses to the basic taste substances are displayed in Fig. 4. The figure shows the relative absorbance of each channel according to the concentrations of the basic taste substances from 1×10^{-7} M to 1×10^{-2} M. The representative sweet taste substance, glucose, shows a large negative tendency in channels 1 and 4, NaCl shows a positive tendency in channels 2, 3, and 5 but a negative tendency in channel 1. The solution of HCl shows no tendency in channels 3 and 6, but shows a larger variance in the other channels than the other tastes. MSG shows a positive tendency in channel 1, but shows the most negative tendency in channel 3, while quinine-HCl shows a negligible tendency in every channel. However, it does show a positive tendency in channel 4. The figure shows a linear and specific response for each of the taste substances. These results show that six dye systems can classify the basic tastes in the form of different patterns for each taste substance.

Figure 4 can be converted using a hexagonal pattern as shown in Fig. 5. Each hexagon represents specific characteristics for each result of maximum concentration among the responses to various concentrations. As shown in Fig. 5, the hexagon is the optical response pattern of the dye/silicone membrane for the five basic taste substances. A different response pattern to each taste substance was obtained from the sensor output of a six-channel fiber optic sensor to a taste solution at a concentration of 1×10^{-2} M. The tastes could thus be analyzed using these patterns. The taste recognition response is produced by the interaction between the molecules of the taste substances and the sensing membranes. The five taste qualities reflect differences in the degrees of interaction with dyes in the membrane.

Using the same method, Fig. 6 displays results obtained with membrane 2. Figures 5 and 6 show different patterns because the output signals depend not only on the reaction of

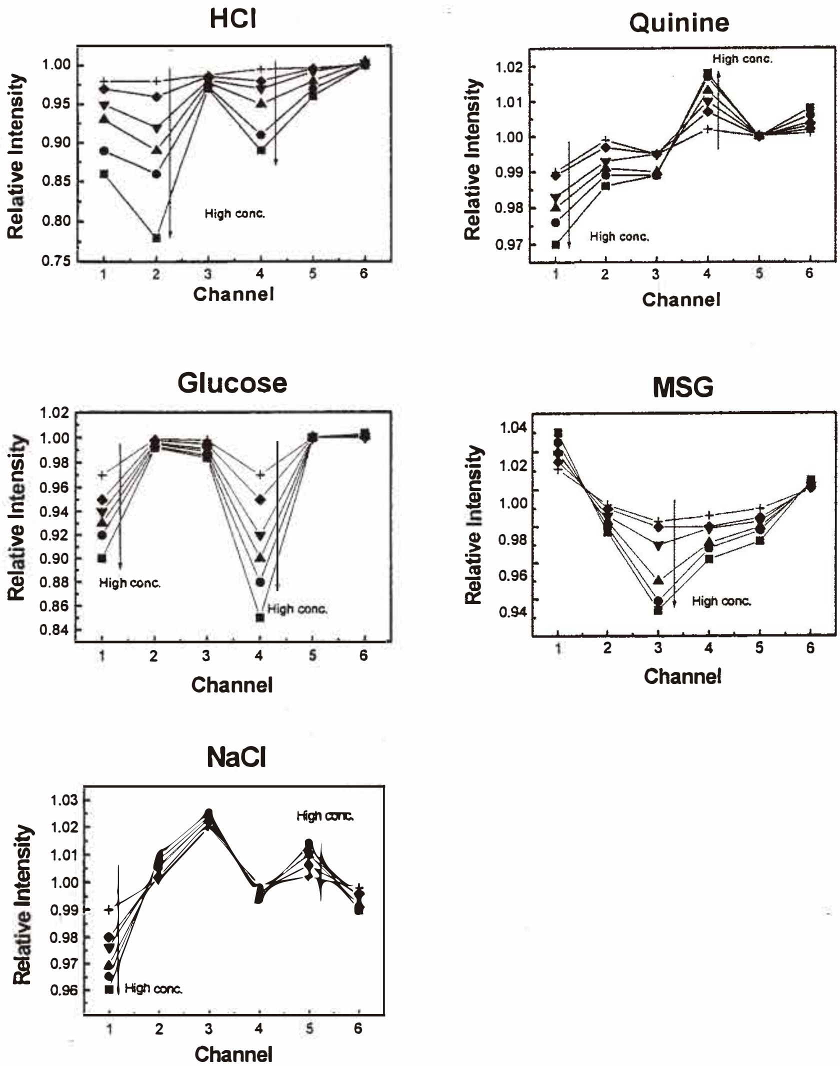

Fig. 4. Response patterns of basic taste substances at various concentrations ranging from 1×10^{-7} M to 1×10^{-2} M.

the dye, but also on the other matrix materials (i.e., lipid, PVC-PVAc-PVA copolymer, and silicone polymer). The response time of membrane 2 is shorter than that of membrane 1, below 10 s, but membrane 2 is more soluble. This disadvantage is evident in its poor reproducibility and reliability. Therefore, membrane 1 is more suitable as a sensing membrane than membrane 2. Based on this result, membrane 1 can be also used for substances containing mixed tastes.

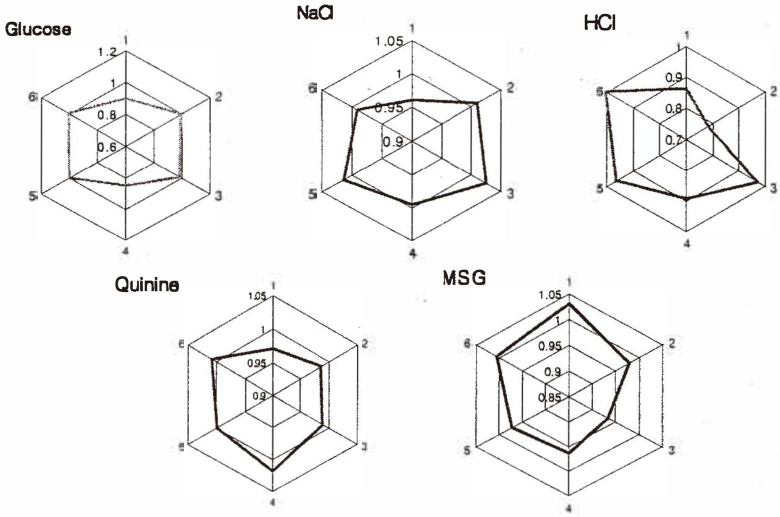

Fig. 5. Optical response patterns for the basic taste substances at maximum concentration with dye/silicone membranes.

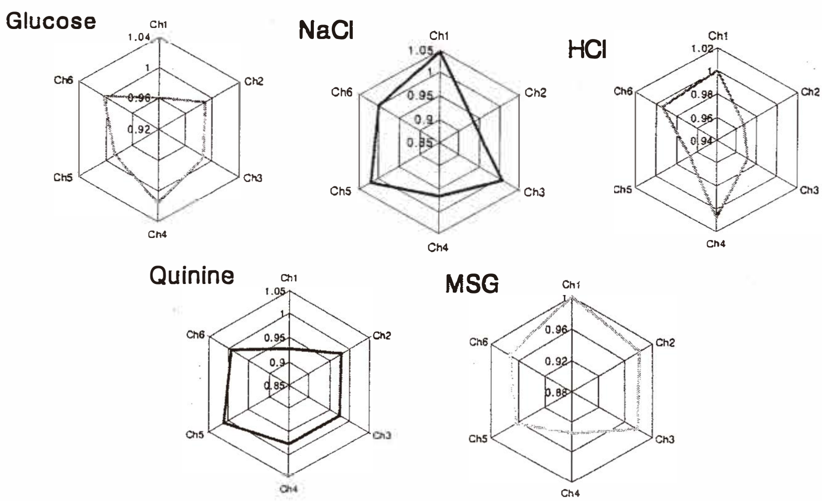

Fig. 6. Optical response patterns for the basic taste substances at maximum concentration with dye/lipid/PVC-PVAc-PVA copolymer membranes.

4.2 *Characteristics of responses to the mixed taste substances*

The response patterns for the mixed taste solutions, for example, glucose/NaCl, glucose/quinine-HCl, glucose/HCl, NaCl/HCl, NaCl/quinine-HCl, and HCl/quinine-HCl, are shown in Fig. 7. Each sample was prepared with 1×10^{-3} M of the former target taste

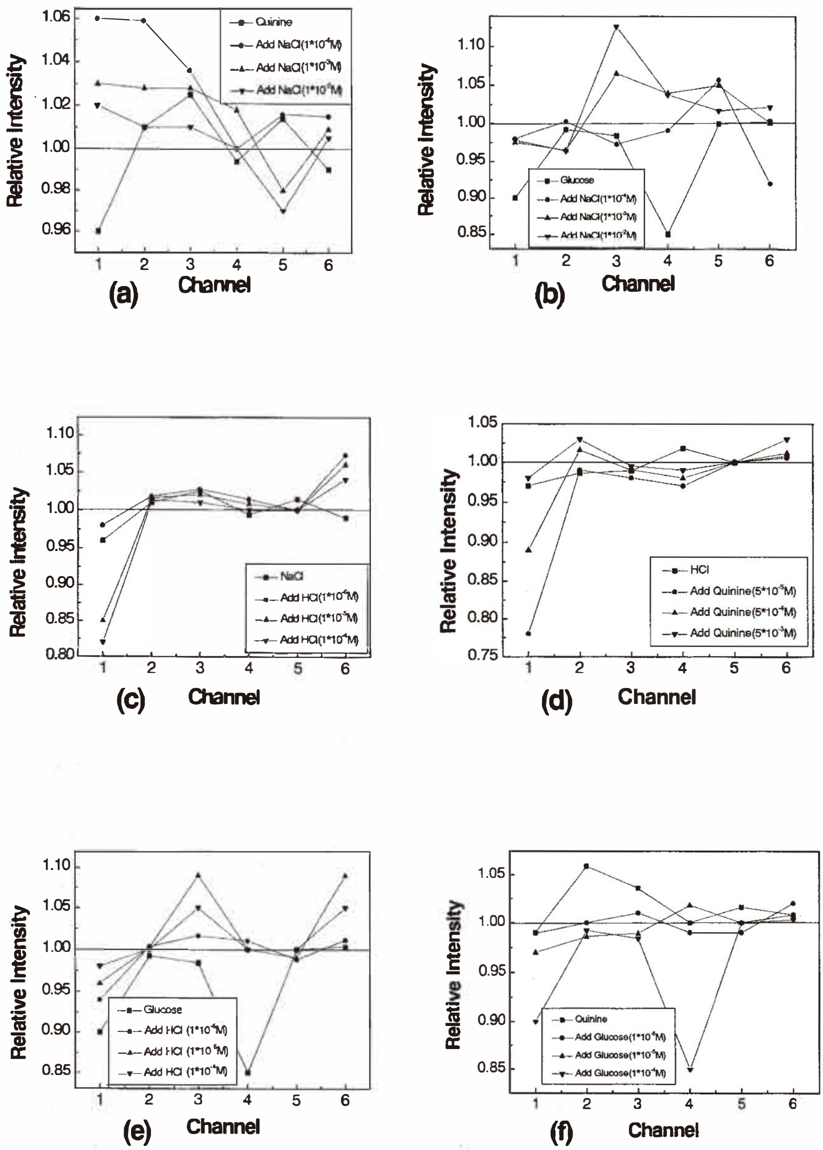

Fig. 7. Optical response patterns for the mixed taste substances; (a) quinine HCI+NaCl, (b) glucose+NaCl, (c) NaCl+HCl, (d) HCl+quinine HCl, (e) glucose+HCl, and (f) glucose+quinine HCl.

molecule mixed with various concentrations of the latter. The mixed tastes had various interactions, such as a synergistic or depressive effect. In general, a synergistic effect is the addition of a sour taste to a salty taste for a sweet taste; the depressive effect is recognized when soy sauce or toenjang (fermented soy sauce used in traditional Korean food) includes salt, but a strongly salty taste is not present. If salt, sugar, quinine, and HCl are mixed in modest concentrations, each basic taste vanishes and a novel harmonious taste appears instead. This is called the offset effect of taste. In addition, there are many complex and subtle effects which remain unknown for various taste mixtures. Figure 7 shows an explanation for these effects of taste mixtures . Besides these effects, various subtle effects can arise between taste substances. A synergistic effect among these is shown in Fig.7(a) and 7(f). Furthermore, basic tastes can be strengthened by a taste added later.

Figure 8 shows a taste map obtained experimentally and reconstructed using the statistical method of principal component analysis (PCA).[9] The data points were plotted on the scattering diagram obtained by PCA, which is a multivariate analysis used to reduce the dimensions of data without losing information. In the present case, a six-dimensional space constructed from six-channel outputs was reduced to a two-dimensional plane. PCA enables us to visualize the information produced by a six-channel sensor in a two-dimensional plane. The data points 7 → 8 → 9, 11 →12 → 13, 14 → 15, and 20 → 21 → 22 show that the basic taste is strengthened, and in other cases, the basic taste is converted into another taste according to a high concentration above a given concentration. Some data points also show the "offset taste effect," that is, points 5, 13, 19, and 22 that are gathered near point (0,0). From these results, we can expect to obseve several other effects of taste mixtures.

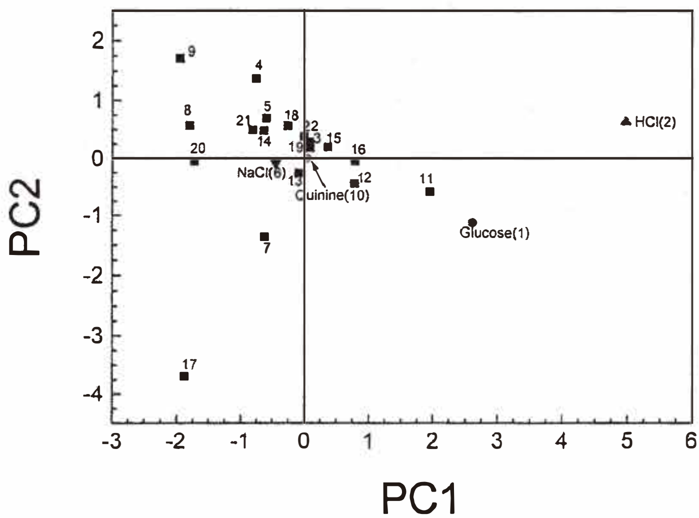

Fig. 8. Results of PCA applied to the data set from taste sensor of mixture taste solutions (3, 4, 5: Glucose+HCl, 7, 8, 9: Glucose+NaCl, 11, 12, 13: Quinine HCl+HCl, 14, 15, 16: NaCl+HCl, 17, 18, 19: Quinine HCl+Glucose, 20, 21, 22: NaCl+Quinine HCl).

5. Conclusions

In this study, an optical multichannel taste sensor using an optical fiber coated dye/polymer membrane was developed and it obtained recognition patterns for five basic taste substances. The sensor could be applied to the taste analysis of commercial foodstuffs such as ionic drinks and soybean sauces. This sensor, based on evanescent field absorption, enables quantitative, objective measurement of taste solutions, with improved sensitivity and lower detection limits, for concentrations ranging from 1×10^{-7} M to 1×10^{-2} M, in comparison to conventional electrode taste sensors or taste threshold values (TTVs). The optical fiber sensor demonstrates that it is possible to differentiate and analyze the five basic tastes and other taste mixtures by generating specified patterns. We evaluated the response characteristics of two membranes, i.e., matrix-based dye/lipid/PVC-PVAc-PVA copolymer and dye/silicone polymer. The membrane based on a polymer matrix is stable in water and has a better reproducibility than the lipid membrane system. With membrane 1 it was possible to repeat the experiment three or four times, and it took about 2–3 min to return the sensor to baseline. As the number of repetitions increased, the sensing membrane including dyes became bleached. However, this sensor could reduce the effect to minimum, because the penetration depth of the detecting ray is only very small part of the entire sensing membrane thickness. This device will be useful for analyzing diverse samples ranging from the food industry to environmental and biomedical monitoring. In particular, these results suggest that this taste sensor will be applicable for quality control in the food industry and to help automate production.

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