Quantitative Analysis of Aroma Components by Gas Chromatography-Mass Spectrometry and its Subjective Evaluation for Olfactory Media Content

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***Abstract*—With the aim of applying to the olfactory media content, this paper investigates effects on a subjective evaluation of lemon aroma of its temperature and quantified density. We first made 12 aromatic gases (4 amounts of lemon essential oil (0.5, 1, 5, and 10 mg in a glass bottle of 1.35 L volume) × 3 temperatures (5, 25, and 60 degrees in Celsius)), and performed a quantitative analysis of limonene, β-pinene, γ-terpinene, and citral in the gases by a gas chromatography-mass spectrometry (GC-MS) measurement. We next asked subjects to evaluate them by using the semantic differential (SD) method with respect to the following five words—clean, thick, refreshing, relaxing, and favorite—and their antonyms. As results, we found that the effect of the temperature of aromatic gas was dominant for the evaluation items; cleanliness, refreshing feeling, relaxing, and preference, and that the lemon aroma less or equal 25 degrees in Celsius was effective for the impressions of cleanliness, refreshing feeling, relaxing, and preference.**

Keywords—Lemon aroma; Subjective evaluation; SD method; Olfactory media content;

I. Introduction

Various studies have been conducted on olfactory media content for enhancing the feeling of presence for an application to multimedia environments [1–5]. There is a need for research from various viewpoints on the psychosomatic effect of aroma. There are many studies on the psychosomatic effect of aroma [6–10]. However, there are few studies on the influence of aroma temperature and density of aroma components on subjective impression.

In this study, we aim to clarify the psychological effects caused by temperature and density of aroma components. For that purpose, we carried out a quantitative analysis of lemon aroma components by gas chromatography-mass spectrometry (GC-MS) and a subjective evaluation experiment using the quantified aroma stimuli.

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II. Proposed Method

A. Samples of Aromatic gases

In this experiment, we used a lemon essential oil (08-449-3430, Tree of Life Co., Ltd.) extracted by using pressed method. We first instilled a lemon oil into a 1.35 L glass bottle with a hermetically sealing lid. The lemon oil was weighed to 0.5, 1, 5, and 10 mg with a ± 5% accuracy using a microbalance (METTLER TOLEDO MT5). We then heated/cooled the bottle using a thermostat (SIB-35CP, Sansyo Co., Ltd.; CN-25C, Mitsubishi Electric Engineering Co., Ltd.) for 3 h. The temperature of thermostat was set at 5, 25, and 60 °C. Consequently, we made 12 aromatic gases and used them as experimental samples.

B. Gas Chromatography-Mass Spectrometry Measurement

To measure the density of aroma components in the aroma samples, we used a Varian Saturn 2200 GC-MS (with CP3800) that was installed with a Varian CP8944 column (0.25 mm × 30.0 m × 0.25 μm). We used helium as the carrier gas at a flow rate of 1.0 mL/min. The injector temperature was set at 200 °C. The oven temperature was initially programmed at 40 °C for 2 min, then increased from 40 °C to 200 °C at 15 °C/min, and held at the temperature for 1 min. We used a split injection method with 1:20 split ratio. We injected 0.5 mL aromatic gas taken from the glass bottle using a gas-tight syringe.

C. Subjects

We recruited 32 subjects (19 male, 13 female; subjects in their 20s) for participation in the experiment. They passed an olfactory test using the standard odor kit (Daiichi Yakuhin Sangyo Ltd.).

D. Procedure

We explained the experimental method to the subject before this experiment. At the same time, we conducted an oral survey on the physical conditions of subject and carried out experiments only for healthy subjects. Sakai and Toda reported that the evaluation result changes due to a cognitive factor of aroma stimulus, so we did not explained information of the aromatic gases [11, 12]. We made the gases under same conditions mentioned in Chapter II. A to minimize the variation in the intensity of each subject. Bottles included the aromatic gases were covered their openings with wrap films in order to prevent a diffusion of the gases. The subject sniffed the aromatic gas in the bottle using a simple respiratory equipment, and evaluated it by using the semantic differential (SD) method with respect to five pairs of words on a scale of 1 (lowest) to 7 (highest). We used the following five words—clean, thick, refreshing, relaxing, and favorite—and their antonyms based on the results of previous research [13] (Fig. 1). After an interval of at least 100 s, the subject evaluated the other sample presented in random order. Each subject evaluated all 12 aromatic gases in a session. We set the temperature of the laboratory at 25 ± 1 °C.

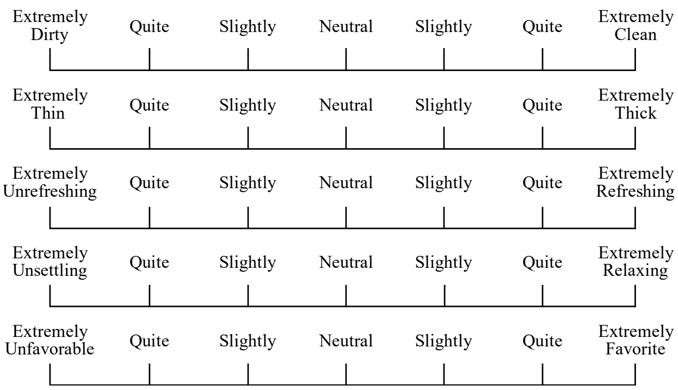

Fig. 1. Evaluation sheet with antonyms on both ends of lines.

III. Results and Discussion

A. Results of Quantitative Analysis of Aroma Components

To calculate the densities of aroma components, the calibration curves of aroma components were constructed by GC-MS measurement. In this study, we determined the following four components as major aromatic components of lemon oil; limonene, β-pinene, γ-terpinene, and citral. We first prepared a standard solution by mixing (+)-Limonene (L0047), (−)-β-pinene (P0441), γ-terpinene (M0318), and Citral (cis- and trans-mixture, D0762) (Tokyo Chemical Industry Co., Ltd.) in same amounts. We next instilled the six standard solutions in the range of 0.2 mg to 30 mg into six glass bottles, which were mentioned in Chapter II. A. After heating/cooling the bottles at the temperature of 60 °C for 3 h, we performed a quantitative analysis under the same conditions as in Chapter II. B, and constructed the calibration curves.

We obtained the mass of each aroma component contained in the experimental samples by using the calibration curve. We finally calculated the densities of the major aromatic components in 12 aromatic gases based on the mass by applying the ideal gas law (Table 1).

TABLE I. Density of Aroma Components for Temperature of Lemon Aroma and Amount of Lemon Oil.

Temperature of lemon aroma (°C)	Amount of lemon oil (mg)	Density of aroma components (ppm)			
		Limonene	β-pinene	γ-terpinene	Citral
5	0.5	31.06	6.89	4.36	2.03
	1	40.84	10.45	4.63	1.12
	5	95.81	42.60	7.96	1.17
	10	96.50	47.08	7.84	1.11
25	0.5	41.66	7.77	4.91	1.09
	1	69.70	16.08	8.06	1.89
	5	336.18	82.61	43.48	4.37
	10	353.17	121.52	38.38	2.84
60	0.5	56.62	7.09	7.94	1.72
	1	120.54	20.46	15.52	11.35
	5	487.92	109.03	69.85	22.19
	10	724.87	183.94	112.58	40.04

B. Results of Subjective Evaluation on Temperature Variation

Fig. 2 shows the mean evaluation scores calculated for the aroma temperatures. As shown in Fig. 2, the scores of “clean,” “refreshing,” “relaxing,” and “favorite” tend to decrease as the temperature become higher. On the other hand, the score of “thick” tends to increase as the temperature become higher. The Wilcoxon rank sum test revealed significant differences in the following results ($p < .05$). There were significant differences among all temperatures for “relaxing.” There were significant differences between 5 °C and 60 °C, 25 °C and 60 °C for the others. As results, subjects rate the evaluation of “relaxing” higher as the aroma temperature become lower, and the evaluations of “clean,” “refreshing,” and “favorite” higher when the aroma temperature is less or equal 25 °C. In other words, the relaxation effect of lemon aroma is enhanced as the aroma temperature becomes lower. The lemon aroma less or equal 25 °C is effective for the impressions of cleanliness, refreshing feeling, relaxing, and preference.

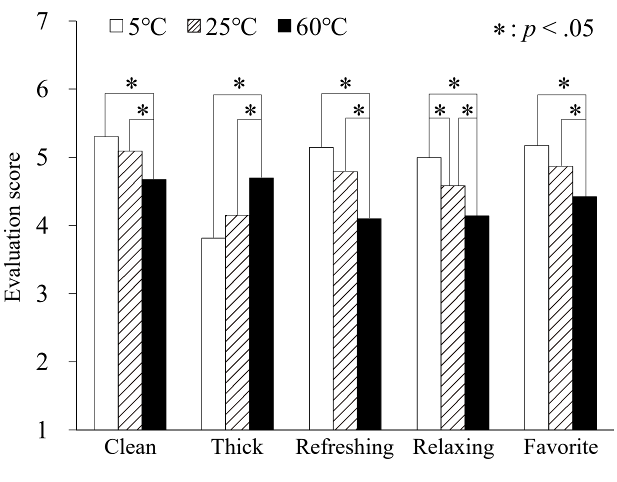

Fig. 2. Evaluation scores of each evaluation term for temperatures.

C. Results of Subjective Evaluation on Temperature and Density Variations

Figs. 3 to 7 show the evaluation scores and densities of aroma components for evaluation terms. In these figures, the bar graphs show evaluation scores for each temperature and each amount of lemon oil. The line graphs show densities of aroma components. The bar graphs are arranged from left in ascending order.

As shown in Figs. 6 and 7, the scores of “relaxing” and “favorite” become higher as the temperature decreases regardless of the densities of aroma components. The scores of “clean” and “refreshing” show the similar tendency (Figs. 3 and 5). We then carried out an analysis of variance, the aroma temperature and the density as factors. As results, the main effect of temperature was confirmed for all evaluation terms; “clean,” “thick,” “refreshing,” “relaxing,” and “favorite” ($p < .001$). On the other hand, the main effect of density was confirmed only evaluation terms of “thick” ($p < .001$). These results indicate that the influence of the aroma temperature is dominant than the density of aroma components for the evaluation terms; “clean,” “refreshing,” “relaxing,” and “favorite.”

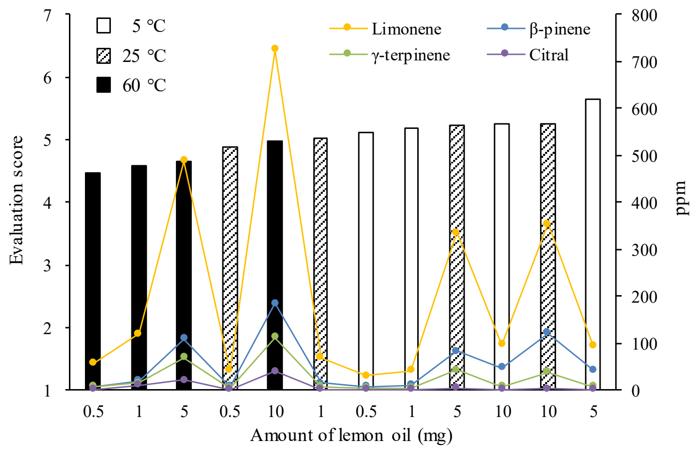

Fig. 3. Evaluation scores and densities of aroma components for evaluation term “clean.”

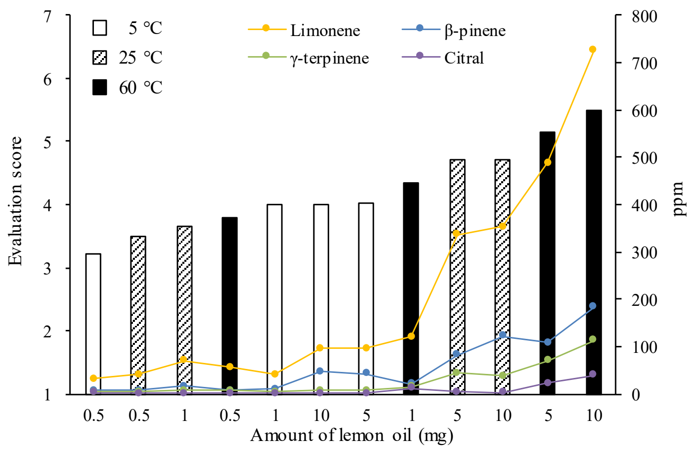

Fig. 4. Evaluation scores and densities of aroma components for evaluation term “thick.”

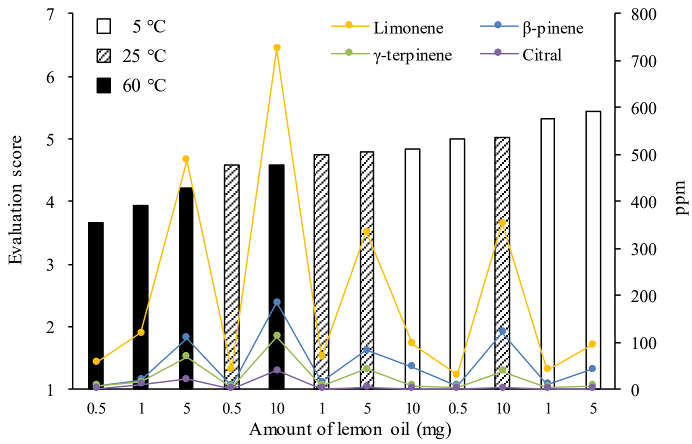

Fig. 5. Evaluation scores and densities of aroma components for evaluation term “refreshing.”

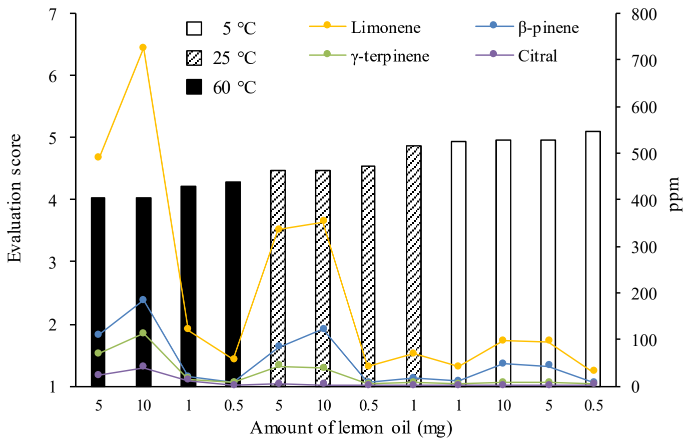

Fig. 6. Evaluation scores and densities of aroma components for evaluation term “relaxing.”

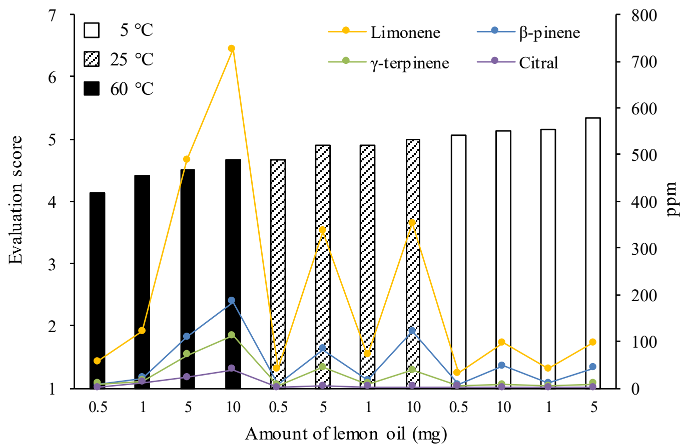

Fig. 7. Evaluation scores and densities of aroma components for evaluation term “favorite.”

IV. CONCLUSION AND RECOMMENDATION

To clarify the psychological effects caused by temperature and density of aroma components, we first performed a quantitative analysis of limonene, β-pinene, γ-terpinene, and citral in lemon aroma by GC-MS. We then conducted a subjective evaluation experiment using 12 aromatic gases as experimental stimuli. As results, we found that the effect of the temperature of aromatic gas was dominant than the density of aroma components for the evaluation items; cleanliness, refreshing feeling, relaxing, and preference. The relaxation effect of lemon aroma was enhanced as the aroma temperature became lower. The lemon aroma less or equal 25 degrees in Celsius was effective for the impressions of cleanliness, refreshing feeling, relaxing, and preference.

It is necessary to examine the contribution of an aroma component—e.g. flavor dilution (FD) factor obtained from aroma extract dilution analysis (AEDA) [14]—to subjects' impression in future study.

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