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Odorant analysis of sake using a palm sized ball SAW gas chromatograph

手のひらサイズのボール SAW ガスクロマトグラフによる日本酒の香気成分分析

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1. Introduction

On-site analysis of many kinds of volatile organic compounds (VOC) has various needs. For example, monitoring of the work environment by combining robotics and sensors and quality control by analyzing odorants in food production and distribution.

A gas chromatograph (GC) is effective for analyzing many kinds of gases, but GC is large and difficult to apply in the field. In contrast, we have developed a portable ball surface acoustic waves (SAW) GC using a ball SAW sensor [1], which uses SAWs orbiting the surface of a sphere in multiple turns [2-4].

In this study, we report the development of a palm sized ball SAW GC and the analysis of odorants of sake.

2. Palm sized Ball SAW GC

A schematic diagram of the ball SAW GC is shown in **Fig. 1**. The sample gas is drawn by a pump and collected in the preconcentrator. Then, by switching the valve to rapidly increase the temperature of the pre-concentrator tube by resistive heater, the collected components are instantly heated and desorbed, and the hydrogen carrier gas flows introduced into the separation column in a backflush. Each component is separated in time by the difference in adsorption on the stationary phase coated on the inner surface of the column, and adsorbed on the sensitive film deposited on the ball SAW sensor, which is detected as SAW delay time change and amplitude change.

The palm-sized ball SAW GC fabricated in this study is shown in **Fig. 2**. This system is equipped with a hydrogen canister for gas supply, a pressure regulator, the preconcentrator, the column, the ball SAW sensor, the valve manifold, the ball SAW drive circuit, and a valve drive and heater circuit, and can be connected to a PC via USB for control and analysis.

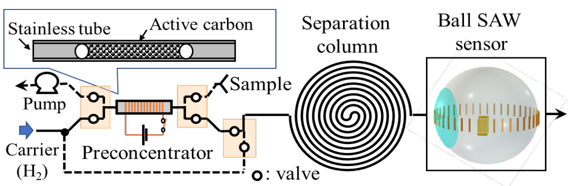

Fig. 1 Schematic diagram of a ball SAW GC with pre concentrator.

The preconcentrator was a stainless-steel pipe with an outer diameter of 1.61 mm and a wall thickness of 0.18 mm, packed with approximately 2 mg of Tenax TA as the adsorbent, and wrapped around the periphery with nichrome wire for resistive heating. The column was a metal capillary, 30 m long Ultra-Alloy®, coated with polyethylene glycol (PEG) as the stationary phase, and wound in a solenoid shape.

Fig. 2 A palm sized ball SAW gas chromatograph

3. Experiment

We used the palm-sized ball SAW GC to analyze the headspace gas of sake. The samples of sake were placed in a vial and the headspace gas was collected in the preconcentrator at 16 ml/min for 2 min. The temperature profile of the Wax column was maintained at 40 °C for 5 min and then the temperature was raised to 140 °C at 10 °C/min using a sheet heater in the column unit. Samples were prepared from different brands of Honjozo-shu A, B, C and Ginjo-shu D, E, F.

4. Result and discussion

Figure 3 shows the chromatograms obtained from the analysis of Honjozo-shu A and Ginjo-shu D. The components of each peak were identified as 1: ethyl acetate, 2: ethanol, 3: water, 6: isoamyl alcohol, 8: ethyl caproate, and 9: ethyl caprylate. As for the peaks 4 and

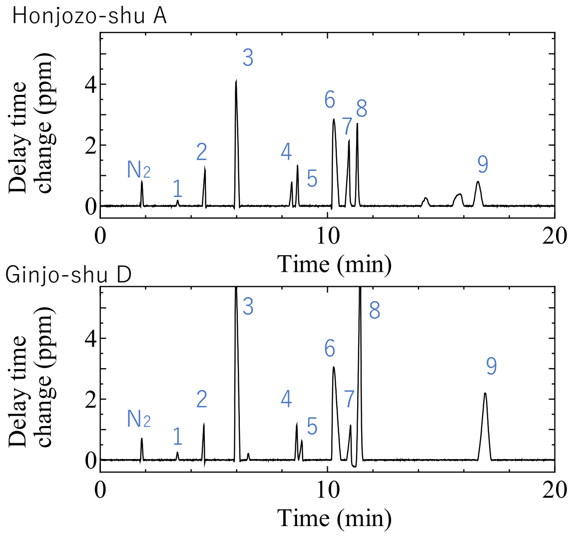

Fig. 3 Chromatograms obtained by analysis of Honjozo-shu A and Ginjo-shu D.

5, the retention time at 8.6 min was identified as isoamyl acetate, and the peak at 7 has not been identified yet.

The peak areas of isoamyl acetate, which is characteristic of Honjozo-shu, and ethyl caproate and ethyl caprylate, which are Ginjo-shu incenses, are shown in **Fig. 4**.

In the case of isoamyl acetate, the values from Honjozo-shu A to C were high. And in the case of ethyl caproate, the values of Ginjo-shu D to F were high. For ethyl caprylate, the value of D was higher than that of F in the previous report [5], However, the value of D was the same as that of F in this experiment. It is assumed that this is due to the preservation condition of the samples.

Ginjo-shu D has both the Ginjo incense of ethyl caproate and the Honjozo incense of isoamyl acetate. We confirmed with brewers that the yeast from Ginjo-shu and Honjozo-shu were used together.

The flavor wheel is known as an index that links the odorants obtained from chromatograms to the sensory evaluation. In the case of sake, a system using the analysis results as standard terminology was compiled in 2006[6]. In the flavor wheel, smell and taste are divided into different classes, with class 1 being Ginjo incense, fruity note, aromatic (esters), alcoholic and flower note.

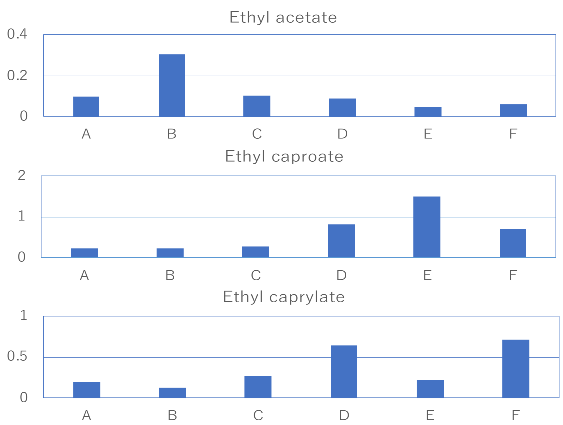

Fig. 4 Peak area of aroma components of each brand

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Figure 5 shows a radar chart of the peak areas of the chromatograms in their odorants. Since human sensory perception is proportional to the logarithm of the concentration of a substance, we plotted the logarithm of the peak area of the chromatogram.

Although the peak area values of Honjozo-shu A, B, and C were different, their shapes of the radar chart were very similar. The radar charts of Ginjo-shu D, E, and F were also similar. Their shapes differed from the radar chart of Honjozo-shu in that they were elongated in the vertical direction.

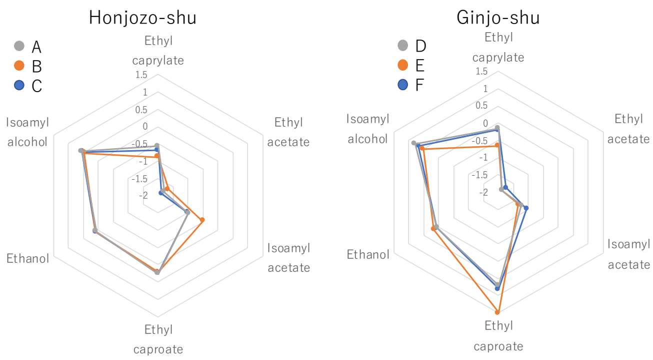

Fig. 5 Radar chart of each brand according to the flavor wheel

5. Conclusion

We developed a palm sized ball SAW GC and used it to analyze the headspace gas of sake. We showed that the ratio of peak areas of odorants differed depending on the sake brand, and found differences in the shapes of Honjozo-shu and Ginjo-shu in the radar chart. The GC of this size is applicable to the quality control of sake brewing process and the on-site analysis of products in yeast breeding and development, and the highly sensitive analysis is expected to be useful for the brewing industry in general.

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