Journal of Food Engineering 171 (2016) 153–158

Contents lists available at ScienceDirect

Journal of Food Engineering

journal homepage: www.elsevier.com/locate/jfoodeng

Progressive freeze-concentration of apple juice and its application to produce a new type apple wine

Osato Miyawaki [a, *], Mihiri Gunathilake [b], Chiaki Omote [a], Takashi Koyanagi [a], Tetsuya Sasaki [c], Harumi Take [c], Akira Matsuda [c], Kana Ishisaki [d], Syouji Miwa [d], Shigeru Kitano [e]

[a] *Department of Food Science, Ishikawa Prefectural University, 1-308 Suematsu, Nonoichi, Ishikawa 921-8836, Japan*
[b] *Department of Food Science and Technology, University of Sri Jayewardenapura, Nugegoda, Sri Lanka*
[c] *Department of Chemistry and Food, Industrial Research Institute of Ishikawa, 2-1 Kuratsuki, Kanazawa, Ishikawa 920-8203, Japan*
[d] *Ishikawa Agriculture and Forestry Research Center, 295-1 Saida, Kanazawa, Ishikawa 920-3198, Japan*
[e] *Maywa Co., Ltd., 3-8-1 Minato, Kanazawa, Ishikawa 920-0052, Japan*

ARTICLE INFO

Article history:
Received 24 August 2015
Received in revised form
15 October 2015
Accepted 17 October 2015
Available online 20 October 2015

Keywords:
Progressive freeze-concentration
Apple juice
Organic acid distribution
Flavor profile
Apple wine
Chaptalization

ABSTRACT

Progressive freeze-concentration (PFC) by a tubular ice system was successfully applied to concentrate apple juice from 13.7 to 25.5 °Brix under a program controlled operation for the coolant temperature and the circulation pumping speed. The organic acid distribution and the flavor profile analysis revealed that no substantial differences were observed for the juice before and after concentration both in organic acids and flavor components showing the high quality concentration by PFC. This was also confirmed by electronic taste and flavor analyzers. The PFC-concentrated apple juice was fermented to obtain a new type apple wine with alcohol content as high as 13.7 vol-% without chaptalization. The organic acid distribution was slightly changed before and after fermentation while the flavor profile changed drastically. The present technique will be applicable to produce new type of wine from many other fruits.

1. Introduction

To produce a wine with enough alcohol content, the sugar content of the original juice is crucial because the alcohol content of a wine theoretically is about one half of the sugar content of the original juice before fermentation. For a juice with the lower sugar content, chaptalization (addition of sugar) or application of concentration technique is necessary to increase the sugar content (Versari et al., 2003, 2004).

To concentrate fruits juice, various membrane techniques are available (Jiao et al., 2004). Apple juice (8.7 °Brix) has been concentrated up to 28.1 °Brix by reverse osmosis and to 51.2 °Brix by osmotic evaporation (Aguiar et al., 2012). Apple juice (11.2 wt-%), grape juice (15.9 wt-%), and roselle extract (9.1 wt-%) have been concentrated up to 57.0, 66.8, and 54.9 wt-%, respectively, by osmotic evaporation (Cisse et al., 2011). In these membrane technique, however, some aroma compounds, especially those with the lower molecular weight are substantially reduced to change the flavor balance although the process is athermic as compared with evaporation.

Cider is a traditional apple wine, which has an alcohol content as low as 5% because the sugar content of the original juice is around 10% (Rita et al., 2011). Therefore, chaptalization (Satora et al., 2008) or membrane concentration technique have also been applied to increase the alcohol content after fermentation. Another option to concentrate apple juice is freeze concentration. This method has been well-known as the concentration method with the best quality (Deshpande et al., 1982). However, the presently available method is known as suspension crystallization. This method requires a complicated system including surface-scraper heat-exchanger for seed ice, recrystallization vessel for the ice crystal growth by Ostwald ripening mechanism, and wash column to separate ice crystals from concentrated mother solution (Huige and Thijssen, 1972). Complexity of this system along with the high capital cost resulted in the limited practical use of this method for

* Corresponding author.
E-mail address: osato@ishikawa-pu.ac.jp (O. Miyawaki).

http://dx.doi.org/10.1016/j.jfoodeng.2015.10.022
0260-8774/

concentration of liquid food (Miyawaki, 2001).

Progressive freeze-concentration (PFC) is an alternative freeze concentration method where only a single ice crystal is formed on a cooling plate with a moving ice front (Matthews and Coggeshall, 1959; Miyawaki et al., 1998). Due to the ease of separation of concentrated solution, this method requires a simple and less expensive system as compared with the suspension crystallization. A falling film reactor has been developed for the scaling up of PFC (Flesland, 1995) and has been applied for concentration of a variety of liquid food (Hernandez et al., 2009, 2010; Sanchez et al., 2010, Sanchez et al., 2011). In this system, the ice crystal grows on a vertically placed cooling plate on which the solution to be concentrated flows as a falling film. This reactor has an open air surface which could lead to the loss of volatile flavor components. Retention of flavors is a major advantage in PFC (Ramos et al., 2005).

As for PFC system with a different design, we proposed a closed tubular ice system with a circulating flow (Miyawaki et al., 2005). This provides a good mass transfer at the ice–liquid interface and a controlled heat transfer in a closed system. This system is expected to give a high separation efficiency and a high-quality for the concentrated product especially in the retention of volatile flavors (Gunathilake et al., 2014a). In the present paper, we applied PFC to apple juice to obtain a high-quality concentrate with high sugar content. The concentrate was fermented to produce apple wine with alcohol content higher than 10%.

2. Experimental method

2.1. Materials

Apple fruits (mainly, *Malus domestica* Borkh. "Senshu") were grown and harvested in Ishikawa Agriculture and Forestry Research Center. After removing the core, the fruits were crushed with addition af ascorbic acid at 0.2%, strained by Pulper Finisher (Sun Food Machinery, Tokyo), treated with 0.4% pectin-degrading enzyme (Sucrase N, Mitsubishi Chemical Foods, Tokyo) for 2 h at 40 °C, and filtrated by a filter press (M200, Makino, Aichi). After the filtration, the apple juice was packed and heated for 10 min in boiling water for inactivation of the added enzyme and sterilization.

2.2. Progressive freeze-concentration

A vertically placed tubular ice system (Miyawaki et al., 2005) with circulating flow (MFC-10, Mayekawa, Tokyo) was used to concentrate apple juice. This system consists of jacketed cylindrical tube (59.5 mm in diameter, 1800 mm × 2 in length), circulation pump (EZ2E008BSCD, Toyo Stainless Steel Industries, Okayama), and feed tank. A coolant, the temperature of which was controlled by a controller and a refrigerator (ERA-RP22A, Mitsubishi Electric, Tokyo), was supplied to the jacket side of the tube to cool down the tube to form ice layer inside. Concentration process was controlled by a program for the coolant temperature and the speed of the circulation pump. Sample temperature was monitored by a thermocouple placed inside the tube.

For the PFC-concentration of apple juice, which has a relatively high osmotic pressure, the seed ice lining step was necessary to prevent the initial supercooling for obtaining higher yield (Miyawaki et al., 2005). For this purpose, pure water was firstly introduced into the tubular system to form a small amount of seed ice on the cooling plate. Then the water was discharged and the precooled apple juice sample was introduced into the system to start PFC-concentration. After the concentration process, the concentrated solution was removed from the system to recover the concentrate. Then, the coolant temperature was raised up to 20 °C to melt the ice surface formed in the system. The ice slid out gravimetrically from the bottom of the vertically placed tube. Thus the ice phase was recovered without washing.

2.3. Fermentation of concentrated apple juice

The PFC-concentrated apple juice was pasteurized at 63 °C for 30 min and 100 ppm sodium sulfite was added. Yeast, *Saccharomyces cerevisiae* OC2, was preincubated in 20 ml YPD medium at 30 °C for 24 h. The preincubated yeast cells were centrifuged at 5000 rpm for 3 min and washed with 5 ml of PFC-concentrated apple juice. The cells were redistributed in 5 mL of PFC-concentrated apple juice again, and then inoculated into 900 mL PFC-concentrated apple juice to start fermentation at 25 °C for 13 days without shaking.

2.4. Analytical method

The concentration in °Brix was analyzed for the original juice, the concentrate after PFC, and the melted ice by a refractometer (APAL-1, As One, Osaka). Organic acids were analyzed by Organic Acid Analyzer (ICS 1500, Thermo Fisher Scientific, Yokohama) with Dionex IonPac ICE-AS6 column (250 mm × 9 mm id) and electro-conductivity detector. The column was equilibrated and eluted with 0.4 mM heptafluorobutyric acid (Wako Chemical, Osaka) in ultra-pure water at a flow rate of 1 mL/min. Alcohol was analyzed by Alcomate (AL-2, Riken Keiki, Tokyo).

Gas chromatography combined with mass spectroscopy (GC/MS) was used with solid phase micro extraction (SPME) for the head space flavor analysis. A 5 ml sample, mixed with 20 ppm cyclohexanol as an internal standard was transferred into a 20 ml screw-cap vial and heated up to 50 °C and held for 10 min then the SPME fiber (50/30um, DVB/CAR/PDMS (Grey), Supelco Analytical, PA, USA) was inserted into the head space of the vial for the extractions and adsorption of flavor components to the SPME fiber for 30 min. Then, the SPME fiber was removed from the vial and was inserted into the injection port of GC/MS (7890A/5975C, Agilent Technologies Inc., Palo Alto, CA) with a DB-Wax fused-silica capillary column (60 m × 0.25 mm id, df = 0.25 μm). The oven temperature, controlled by a program, was started from 40 °C (held for 10 min) and raised up to 230 °C (held for 12 min) at a rate of 4 °C/

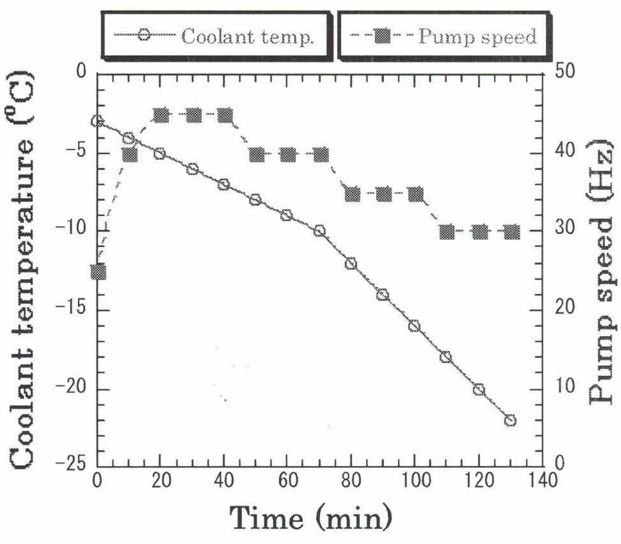

Fig. 1. A program of coolant temperature and circulation pump speed to concentrate apple juice by tubular system for progressive freeze-concentration.

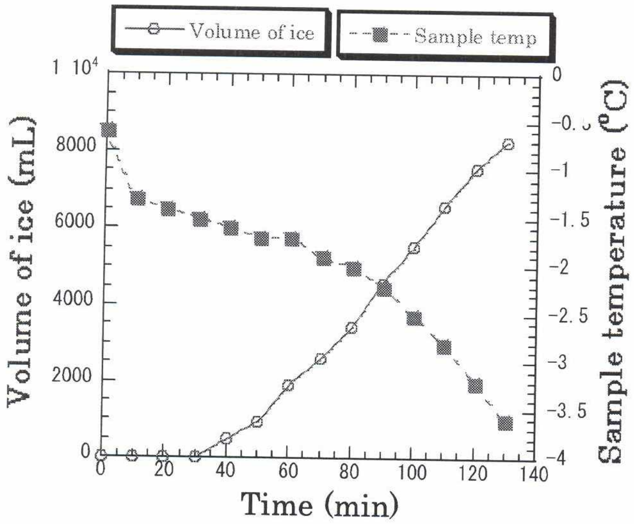

Fig. 2. Change in ice volume formed and sample temperature during the concentration process of apple juice by progressive freeze-concentration.

min. Helium was used as the carrier gas at a flow rate of 1.2 mL/min. The split ratio at the injection port was 1:2. The mass spectra were obtained by electron-impact ionization at an ionization voltage of 70 eV. GC peaks were identified by Wiley and NIST mass spectra database.

2.5. Quality analysis of taste and flavors by electronic sensor

Taste analysis was carried out by Taste Analyzer (SA402, Inteligent Sensor Technology, Kanagawa) with sensors for umami (AAE), saltiness (CT0), sourness (CA0), bitterness (C00), and astringency (AE1). For flavor analysis, Flagrance Analyzer (FF2A, Shimadzu, Kyoto) was used at 20 °C and 40% relative humidity. The result was analyzed by PostPro and Asmell softwares (Shimadzu, Kyoto).

3. Results and discussion

3.1. Progressive freeze-concentration of apple juice

A tubular ice system was used to concentrate apple juice. In this system, a small amount of seed ice layer was formed inside the tubular reactor and the apple juice sample, precooled down to the freezing point, was introduced into the system. The freezing point of the apple juice was −1.37 °C. In the tubular ice system, the ice crystal growth rate and the circulation flow rate in the reactor are the two important operating parameters (Miyawaki et al., 1998, 2005). The former was controlled by the temperature of the coolant in the jacket surrounding the tubular reactor. The latter was controlled by a pump in the circulation loop. These operating parameters were controlled by a computer program. The program was changed depending on the freezing point (concentration) and other physical properties of the sample. In the present case, the program employed was shown in Fig.1, where the coolant temperature was decreased with time to counteract the increase in the heat transfer

Table 1
Organic acid analysis of progressive freeze-concentrated apple juice (g/L).

Component	Concentration (g/L)				
	Original	Concentrate	Ice	Reconstituted	Fermented (312h)
Pyruvic acid	ND	ND	ND	ND	0.08
Citric acid	0.03	0.06	0.01	0.04	0.01
Malic acid	3.66	7.24	1.55	4.10	6.96
Acetic acid	tr[a]	tr	tr	tr	0.66
Succinic acid	0.04	0.07	0.01	0.04	0.67
Total	3.73	7.37	1.57	4.18	8.38

[a] trace.

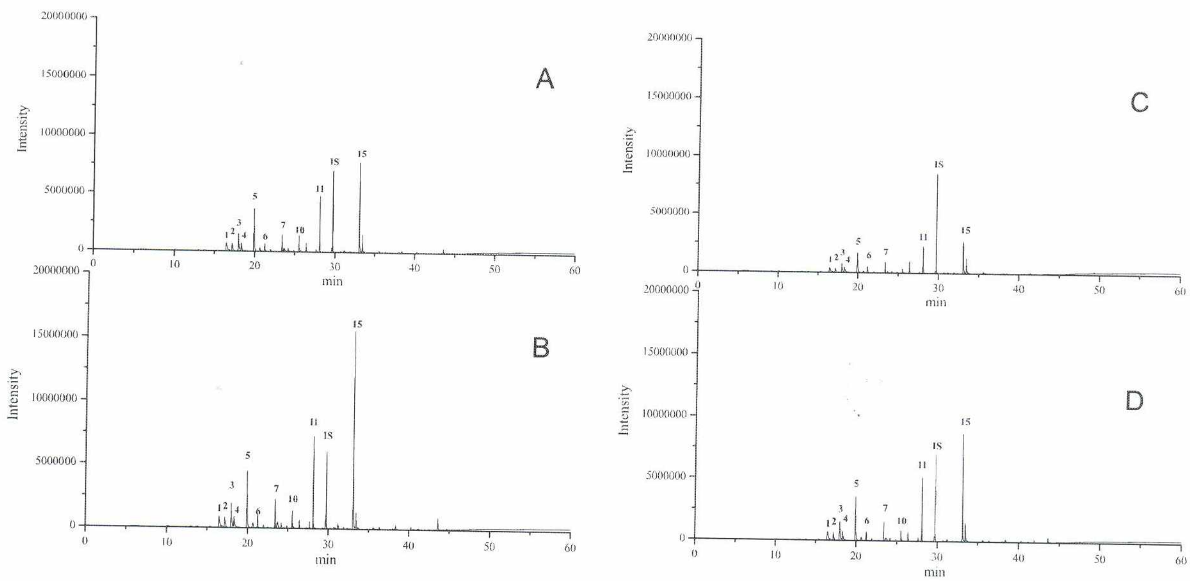

Fig. 3. GC/MS analysis of head space flavor profile of original apple juice (A), its concentrate (B), ice phase (C), and reconstituted juice (D) after progressive freeze-concentration.

Table 2
Flavor analysis of progressive freeze-concentrated apple juice.

No.	Component	RT[a] (min)	Relative peak area[b]			
			Original	Concentrate	Ice	Reconstituted
1	Ethyl butyrate	16.48	0.28	0.41	0.12	0.28
2	Ethyl 2-methylbutyrate	17.20	0.21	0.28	0.07	0.18
3	Butyl acetate	17.98	0.47	0.70	0.20	0.48
4	Hexanal	18.34	0.23	0.33	0.11	0.24
5	2-Methylbutyl acetate	19.92	1.00	1.45	0.37	0.98
6	Butanol	21.28	0.14	0.23	0.08	0.15
7	2-Methyl-1-butanol	23.42	0.24	0.44	0.12	0.25
10	Hexyl acetate	25.53	0.22	0.25	0.03	0.14
11	Hexanol	28.11	0.68	1.27	0.28	0.73
IS[c]	Cyclohexanol	29.47	1.00	1.00	1.00	1.00
15	Benzaldehyde	33.07	1.50	3.35	0.47	1.66

[a] Retention time.
[b] Relative peak area to internal standard (cyclohexanol).
[c] Internal standard.

resistance because of the increase in ice thickness. The pumping speed was also decreased with time because of the decrease in the inner diameter of circulation flow because of ice crystal growth.

Under the program described above, the ice volume increased in the reactor and the temperature of the sample decreased, without supercooling, as shown in Fig.2. The sample temperature corresponds to the freezing point, which decreased along with the progress in the concentration process. By this system, 12.18 L of the apple juice was concentrated, in about 2 h's operation time, to 3.50 L with concentration increased from 13.7 to 25.5 °Brix with a yield of 63.8%. In this case, the yield may not be satisfactory but this could be improved to the necessary level by applying the partial ice-melting technique (Miyawaki et al., 2012; Gunathilake et al., 2014b). We are now trying to set-up an integrated system of PFC with the partial ice melting apparatus for more universal industrial applications of PFC.

3.2. *Quality analysis of apple juice concentrate*

In Table 1, the organic acid distribution is compared for the apple juice before and after PFC-concentration. The major acid in the apple juice was malic acid which was concentrated by about two times proportional to the concentration in °Brix. Citric acid and succinic acid, as minor components, were also concentrated in the similar way. After the reconstitution by the dilution based on °Brix, almost the similar organic acid distribution was recovered with the original juice.

Fig. 3 compares the flavor profiles in the head space among the original juice (A), PFC-concentrated juice (B), ice phase (C), and the reconstituted juice (D) based on °Brix. Table 2 shows the relative peak area compared with the internal standard (IS). The major flavor components were butyl acetate, 2-methylbutyl acetate, hexanol, and benzaldehyde, all of which were concentrated by PFC along with the other components. The reconstituted juice shows almost similar flavor profile with the original juice.

The taste and flavor profiles measured by electronic sensors are shown in Figs. 4 and 5. The excellent similarities were again observed between the reconstituted juice and the original. These results on the organic acid distribution, the flavor profile, and the electronic sensor analysis clearly show that the original qualities are kept unchanged in PFC at a high quality. This is the strongest point of PFC compared with the other methods of concentration such as evaporation and reverse osmosis (Gunathilake, 2014a).

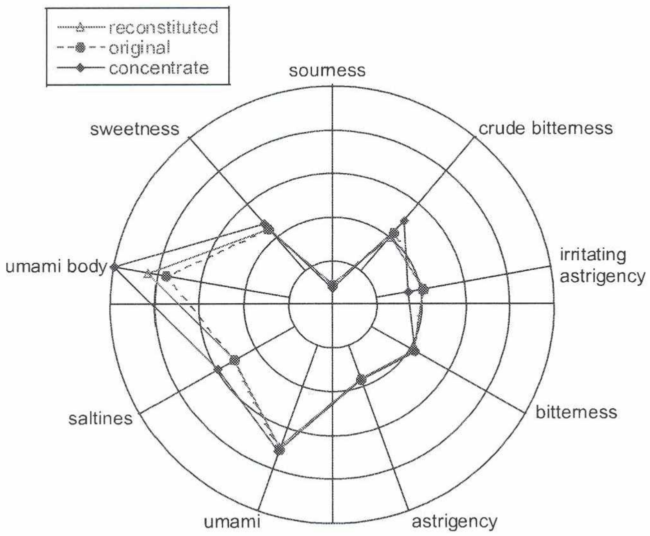

Fig. 4. Comparison of taste profile among original apple juice, its concentrate, and reconstituted juice after progressive freeze-concentration measured by an electronic taste sensor.

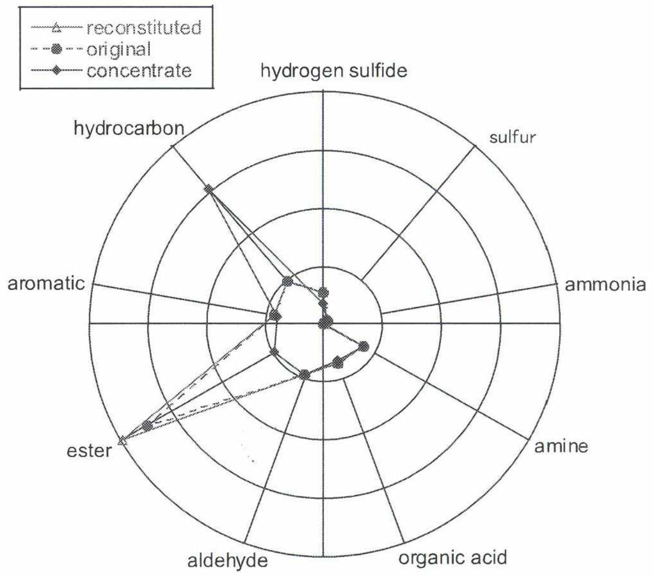

Fig. 5. Comparison of flavor profile among original apple juice, its concentrate, and reconstituted juice after progressive freeze-concentration measured by an electronic flavor sensor.

215

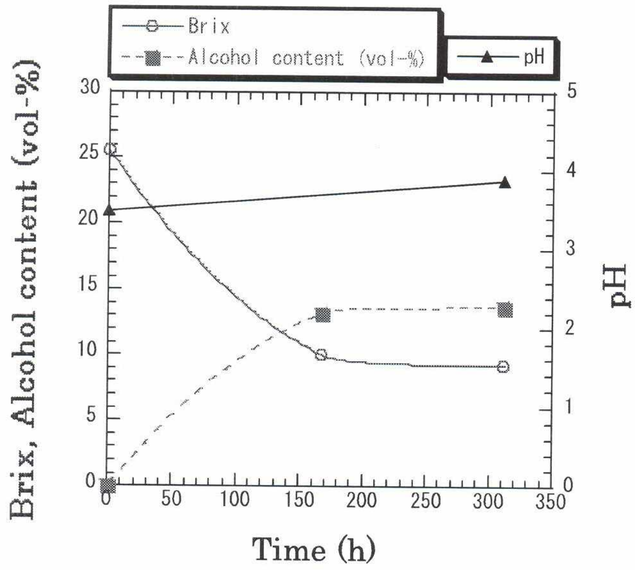

Fig. 6. Time course in fermentation of PFC-concentrated apple juice at 25 °C.

Moreover, the organic acid distribution and the flavor profile in the ice phase, though diluted, are not much different from those in the original. This reflects the nonspecific separation nature in the PFC (Gunathilake, 2014b) because the mechanism for solute incorporation into the ice phase is nonspecific mechanical involvement (Watanabe et al., 2013). This is another strong point of PFC compared with evaporation and reverse osmosis. In the evaporation, components are separated depending on the vapor pressure in the concentration process. In the reverse osmosis, components are separated basically depending on their molecular weight. Therefore, the relative component-distribution cannot be kept in these methods after concentration. On the contrary, the relative component distribution is kept unchanged in PFC even in the ice phase as well as the concentrate.

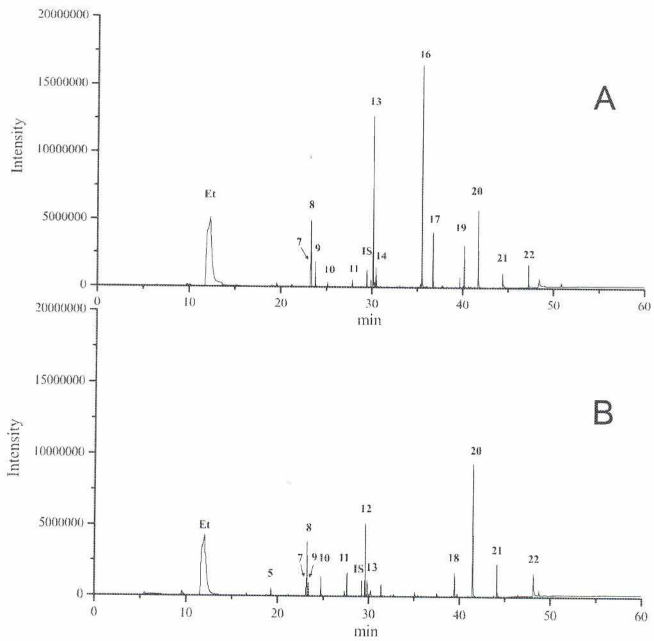

Fig. 7. GC/MS analysis of head-space flavor profile of the wine from PFC-concentrated apple juice (A) and cider obtained from market (B).

3.3. *Fermentation of apple juice concentrate to produce apple wine*

Fig. 6 shows the time course of the fermentation of the PFC-concentrated apple juice. During the fermentation process in 13 days, the alcohol concentration increased up to 13.7 vol-%, which is much higher than that of cider (5.5 vol-%) obtained in market. During this period, the concentration in °Brix changed from 25.5 to 9.2 °Brix and pH changed slightly from 3.48 to 3.88. In the literature, Rita et al. (2011) fermented apple juice with total sugar of 9.6–10.4% to obtain apple wines with alcohol content of 5.1–5.3 vol-%. Satora et al. (2008) added sucrose to apple juice with sugar content of 9.37–10.44% to obtain wines with alcohol content of 8.21–8.84 vol-%. In the present case, alcohol content of apple wine was much higher than 10% because of the preconcentration of apple juice by PFC.

In Table 1, the organic acid distribution was compared before and after fermentation. After the fermentation of PFC-concentrated apple wine, acetic acid and succinic acid had much increased, pyruvic acid slightly increased, and citric acid slightly decreased. As a whole, the total acid had slightly increased.

Fig. 7 compares the head-space flavor profiles between the PFC-concentrated apple wine and a cider obtained from market. In Table 3, these are quantitatively compared relative to the internal standard (cyclohexanol). In this table, the relative peak area for ethanol (Et in Fig.7) was not shown because it was too large and quantitatively meaningless. Interestingly, most flavor components in the juice before fermentation were lost after fermentation except 2-methyl-1-butanol, hexyl acetate, and hexanol. Instead, fermentation-specific flavors such as isoamyl alcohol and phenetyl alcohol had newly appeared. Although similar flavor profiles were observed between the PFC-concentrated apple wine and a cider as shown in Fig.7, the individual peak pattern is a little different between the two probably reflecting the difference in varieties of the

Table 3
Comparison of head-space flavors among PFC-concentrated apple juice, apple wine from the concentrate, and a cider obtained from market.

No.	Component	RT[a] (min)	Relative peak area[b]		
			PFC-concentrate	Apple wine	Cider
1	Ethyl butyrate	16.48	0.41	ND[c]	ND
2	Ethyl 2-methylbutyrate	17.20	0.28	ND	0.30
3	Butyl acetate	17.98	0.70	ND	ND
4	Hexanal	18.34	0.33	ND	ND
5	2-Methylbutyl acetate	19.92	1.45	ND	0.61
6	Butanol	21.28	0.23	ND	ND
7	2-Methyl-1-butanol	23.21	0.44	1.19	1.14
8	Isoamyl alcohol	23.32	ND	4.36	4.00
9	Ethyl hexanoate	23.62	ND	ND	1.03
10	Hexyl acetate	25.53	0.25	0.29	1.46
11	Hexanol	28.11	1.27	0.38	1.44
IS[d]	Cyclohexanol	29.47	1.00	1.00	1.00
12	Unknown	29.63	ND	ND	5.39
13	Ethyl octanoate	30.18	ND	10.3	1.90
14	Acetic acid	30.44	ND	1.47	ND
15	Benzaldehyde	33.07	3.35	ND	ND
16	Ethyl decanoate	35.52	ND	15.82	0.14
17	Ethyl 9-decenoate	36.73	ND	3.39	ND
18	Phenetyl acetate	39.45	ND	ND	2.40
19	Ethyl dodecanoate	40.18	ND	2.57	ND
20	Phenetyl alcohol	41.73	ND	4.80	10.6
21	Octanoic acid	44.42	ND	1.43	2.40
22	Decanoic acid	47.25	ND	1.35	2.40

[a] Retention time.
[b] Relative peak area to internal standard (cyclohexanol).
[c] Not detected.
[d] Internal standard.

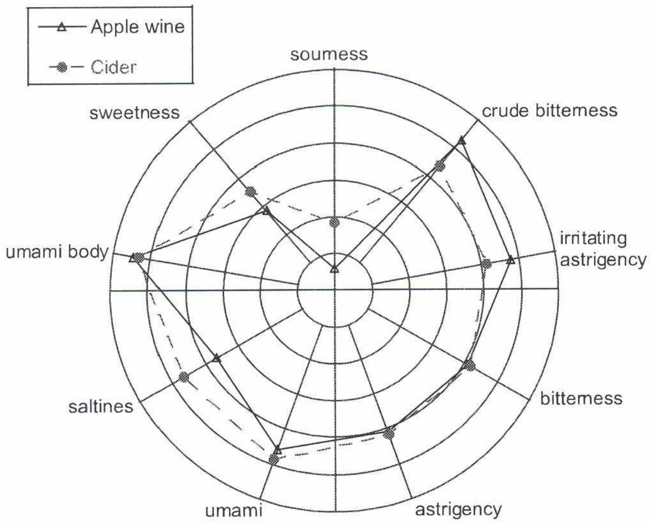

Fig. 8. Comparison of taste profile between the wine from PFC-concentrated apple juice and cider obtained from market measured by an electronic taste sensor.

original fruits and the yeast used in the fermentation.

In Fig. 8, taste profiles are compared between the PFC-concentrated apple wine and the cider measured by an electronic sensor. Although there are some differences in details, the taste profiles are not much different between the two showing that these are in the same category as long as the taste analyzer used here concerns.

4. Conclusion

Progressive freeze-concentration was successfully applied to concentrate apple juice from 13.7 to 25.5 ^{o}Brix at a high quality without changing the organic acid balance and the flavor profile. The PFC-concentrated apple juice was fermented to produce a new-type apple wine with alcohol content as high as 13.7 vol-%. Before and after fermentation, the organic acid distribution slightly changed while the flavor profile changed drastically. The present technique is expected to be applicable to many other fruit juices to produce new type fruit wines.

Acknowledgment

This work was partly supported by Ministry of Economy, Trade and Industry (24142321096), Japan and also by Japan Science and Technology Agency (AS2321218E).

References

Aguiar, I., Miranda, N., Gomes, F., Santos, M., Freitas, D., Tonon, R., Cabral, L., 2012. Physicochemical and sensory properties of apple juice concentrated by reverse osmosis and osmotic evaporation. Innov. Food Sci. Emerg. Technol. 16, 137–142.

Cisse, M., Vaillant, F., Bouquet, S., Pallet, D., Lutin, F., Reynes, M., Dornier, M., 2011. Athermal concentration by osmosis evaporation of roselle extract, apple and grape juices and impact on quality. Innov. Food Sci. Emerg. Technol. 12, 352–360.

Deshpande, S.S., Bolin, H.R., Salunkhe, D.K., 1982. Freeze concentration of fruit juices. Food Technol. 36, 68–82.

Flesland, O., 1995. Freeze concentration by layer crystallization. Dry. Technol. 13, 1713–1739.

Gunathilake, M., Simmura, K., Dozen, M., Miyawaki, O., 2014a. Flavor retention in progressive freeze-concentration of coffee extract and pear (La France) juice flavor condensate. Food Sci. Technol. Res. 20, 547–554.

Gunathilake, M., Dozen, M., Simmura, K., Miyawaki, O., 2014b. An apparatus for partial ice-melting to improve yield in progressive freeze-concentration. J. Food Eng. 142, 64–69.

Hernandez, E., Raventos, M., Auleda, J.M., Ibarz, A., 2009. Concentration of apple and pear juices in a multi-plate freeze concentrator. Innov. Food Sci. Emerg. Technol. 10, 348–355.

Hernandez, E., Raventos, M., Auleda, J.M., Ibarz, A., 2010. Freeze concentration of must in a pilot plant falling film cryoconcentrator. Innov. Food Sci. Emerg. Technol. 11, 130–136.

Huige, N.J.J., Thijssen, H.A.C., 1972. Production of large crystals by continuous ripening in a stirrer tank. J. Cryst. Growth 13/14, 483–487.

Jiao, B., Cassano, A., Drioli, E., 2004. Recent advances on membrane processes for the concentration of fruit juices: a review. J. Food Eng. 63, 303–324.

Matthews, J.S., Coggeshall, N.D., 1959. Concentration of impurities from organic compounds by progressive freezing. Anal. Chem. 31, 1124–1125.

Miyawaki, O., Liu, L., Nakamura, K., 1998. Effective partition constant of solute between ice and liquid phases in progressive freeze-concentration. J. Food Sci. 63, 756–758.

Miyawaki, O., 2001. Analysis and control of ice crystal structure in frozen food and their application to food processing. Food Sci. Technol. Res. 7, 1–7.

Miyawaki, O., Liu, L., Shirai, Y., Sakashita, S., Kagitani, K., 2005. Tubular ice system for scale-up of progressive freeze-concentration. J. Food Eng. 69, 107–113.

Miyawaki, O., Kato, S., Watabe, K., 2012. Yield improvement in progressive freeze-concentration by partial melting of ice. J. Food Eng. 108, 377–382.

Ramos, F.A., Delgado, J.L., Bautista, E., Morales, A.L., Duque, C., 2005. Change in volatiles with the application of progressive freeze-concentration to Andes berry (*Rubus glaucus* Benth). J. Food Eng. 69, 291–297.

Rita, R., Zanda, K., Daina, K., Dalija, S., 2011. Composition of aroma compounds in fermented apple juice: effect of apple variety, fermentation temperature and inoculated yeast concentration. Procedia Food Sci. 1, 1709–1716.

Sanchez, J., Ruiz, Y., Raventos, M., Auleda, J.M., Hernandez, E., 2010. Progressive freeze concentration of orange juice in a pilot plant falling film. Innov. Food Sci. Emerg. Technol. 11, 644–651.

Sanchez, J., Hernandez, E., Auleda, J.M., Raventos, M., 2011. Freeze concentration of whey in a falling-film based pilot plant: process and characterization. J. Food Eng. 103, 147–155.

Satora, P., Sroka, P., Duda-Chodak, A., Tarko, T., Tuszynski, T., 2008. The profile of volatile compounds and polyphenols in wines produced from dessert varieties of apples. Food Chem. 111, 513–519.

Versari, A., Ferrarini, R., Parpinello, G.P., Galassi, S., 2003. Concentration of grape must by nanofiltration membranes. Trans. IChemE 81, 275–278.

Versari, A., Ferrarini, R., Tornielli, G.B., Parpinello, G.P., Gostoli, C., Celotti, E., 2004. Treatment of grape juice by osmotic Evaporation. J. Food Sci. 69, 422–427.

Watanabe, A., Miyawaki, O., Watanabe, M., Suzuki, T., 2013. Mechanism of solute incorporation into ice phase in progressive freeze-concentration. Jpn. J. Food Eng. 14, 163–168.