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

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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

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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/

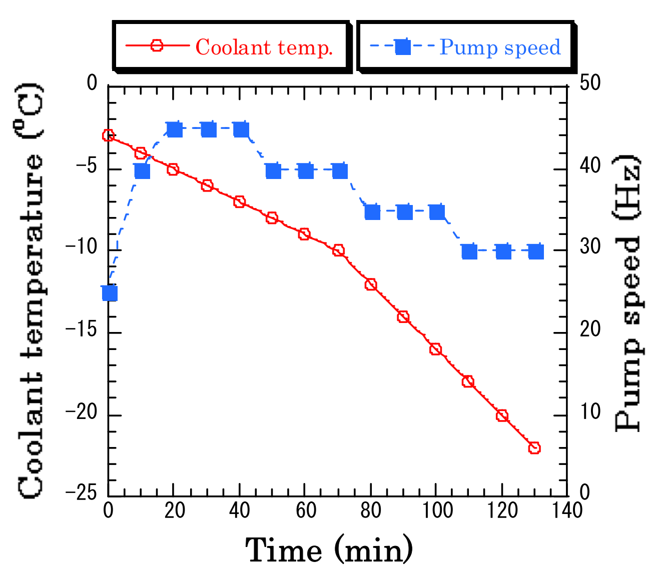

Fig. 1. A program of coolant temperature and circulation pump speed to concentrate apple juice by tubular system for progressive freeze-concentration.