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Vacuum-assisted block freeze concentration applied to wine

Guillermo Petzold *, Patricio Orellana, Jorge Moreno, Elisabeth Cerda, Paula Parra

Group of Emergent Technology and Bioactive Components of Food, Department of Food Engineering, Universidad del Bío-Bío, Casilla 447, Chillán, Chile

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ABSTRACT

Vacuum is an assisted technique to improve the efficiency of freeze concentration process, and wine is an interesting raw material to be concentrated. In this study reported the vacuum-assisted block freeze concentration to force the separation of solutes from a frozen wine sample. By applying vacuum (40 kPa), the solids content in the concentrated fraction increased significantly compared to the initial value (8°Brix), reaching a value near of 21°Brix after 5 min, although decreasing over time, indicated that the concentrated extract was collected mostly in the first fractions. The efficiency achieved high values near of 90% in a short time (15 min, 45 ml of initial sample), with high values of ice purity over time of suction. The pH, alcohol, acidity and total polyphenol content increased significantly in the concentrated sample (50% of concentrate) compared the fresh wine. The color evaluation indicating that cryoconcentrate (50% of concentrate) was darker than the initial sample, with a ΔE^* value >40 CIELAB units. The vacuum-assisted block freeze concentration applied to wine is an effective technique to obtain a concentrated sample with attractive properties for possible gastronomic use.

Industrial relevance: The vacuum assisted block freeze concentration allows producing wine concentrates with attractive properties, such as a high solid content, darker color and higher alcohol content than the initial sample. In this work has applied vacuum (40 kPa) as an assisted technique to improve the efficiency of block freeze concentration applied to wine, obtaining promissory results.

1. Introduction

The concentration of liquid foods is an important unit operation, because concentrated products occupy less space and weightless, and food manufacturers can potentially save on transportation costs, warehousing costs, as well as handling costs for materials required for its products (Ramaswamy & Marcotte, 2006). This unit operation could be made primarily through three mechanisms: high temperature (evaporation), freeze concentration and membrane technology. Freeze concentration or cryoconcentration is a method for recovering a food solute from a solution based on the separation of pure ice crystals from a freeze-concentrated aqueous phase. As compared to evaporation and membrane technology, freeze concentration has some significant potential advantages for producing a concentrate with high quality because the process occurs at low temperatures where no vapor/liquid interface exists resulting in minimal loss of volatiles (Morison & Hartel, 2007).

The innovations of freeze concentration has been associated more with developments in the configuration of one-step systems (block freeze concentration or progressive freeze concentration) than conventional freeze concentration systems (suspension crystallization), because of the simpler separation step (Petzold & Aguilera, 2009; Sánchez, Ruiz, Auleda, Hernández, & Raventós, 2009; Sánchez, Ruiz, Raventós, Auleda, & Hernández, 2010; Miyawaki, Kato, & Watabe, 2012). Another advantage of these one-step systems is their simplicity in terms of both the construction and operation of the equipment (Sánchez et al., 2009).

The basis of block freeze concentration is as follow: a food liquid solution is completely frozen, the whole frozen solution is thawed and then the concentrated fraction is separated from the ice fraction by gravitational thawing assisted or not by other techniques to enhance the separation efficiency (Aider & de Halleux, 2008a, 2008b). Under these conditions, the ice block acts as a solid carcase through which the concentrated fraction passes, and it is possible to reach efficiency higher than 90%, meaning that the amount of the entrapped solute in the ice crystal is reduced to a minimal level (Aider & de Halleux, 2009).

The alternatives of assisted techniques applied to block freeze concentration are external forces such as centrifugation or vacuum. In this way, centrifugation has been proposed by Bonilla-Zavaleta, Vernon-Carter, and Beristain (2006) in frozen pineapple juice, while Luo, Chen, and Han (2010) obtained ice crystals of high purity during the freezing concentration of brackish water, Virgen-Ortíz et al. (2012, 2013) proposed centrifugal cryoconcentration methods to concentrate dilute protein solutions, and Petzold and Aguilera (2013) present a centrifugal freeze concentration method with sucrose solution, and recently Petzold, Moreno, Lastra, Rojas, and Orellana (2015) proposed this technique applied to blueberry and pineapple juices. Vacuum

* Corresponding author.
E-mail address: gpetzold@ubiobio.cl (G. Petzold).

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(suction by a pump) has been proposed by Hsieh (2008) to get drinkable water from sea water to separate salt, converting the ice of sea water into fresh water. On the other hand, Petzold, Niranjan, and Aguilera (2013) applying a vacuum (80 kPa) improved the efficiency over atmospheric conditions in freeze concentration of sucrose solutions, and Moreno, Robles, Sarmiento, Ruiz, and Pardo (2013) concluded that the highest concentration factor was obtained for treatments with vacuum separation (27 kPa), due to the positive effect of pressure difference on the movement of the concentrated liquid fraction in block freeze-concentration of coffee brews. Recently, Pardo and Sánchez (2015) used a vacuum (57 kPa) to the intensification of block freeze concentration applied of sucrose solutions, observed that all treatments that used vacuum as separation method showed a higher efficiency than those in similar processing conditions that used gravity as the separation method.

In this condition, the vacuum as an assisted technique applied to freeze concentration is similar to the principle used by children to suck the sugar solution containing colorants from popsicles, and takes advantage of the hydraulic system existing in the frozen matrix formed by veins (or channels) between the ice crystals containing the concentrated solution (Martel, 2000). In nature, this matrix in a frozen solution is responsible for differences in the concentration of impurities in ancient polar ice (Rempel, Waddington, Wettaufer, & Worster, 2001).

Since it is expected that a cryoconcentrated extract will have different organoleptic properties that their evaporated counterparts, unique applications may be found in high-quality industrial products as well as in gastronomy (Petzold, 2013). In this way, wine is a perfect raw material to be cryoconcentrated for possible gastronomic use; because wine is widespread use in culinary activities (48% of French classical sauces contain wine) and the chefs normally using the "reduce wine" technique when making a sauce by evaporating primarily water, but lose many odorant compounds as well, because of steam evaporation (This, 2013).

The aim of this study was to study the use of vacuum as an assisted technique in block freeze concentration applied to wine.

2. Materials and methods

2.1. Materials

We used red wine variety Cabernet Sauvignon, in a tetra pack format of 1 L, from the vineyard Santa Rita (Central Valley, Chile) acquired in a supermarket in the city of Chillan, Chile. At the time of purchase, it was verified that the date of production (year 2015) and batch number coincide to maintain uniformity of the samples. Wine boxes were kept closed and under refrigeration (5 °C) until processing.

2.2. Experimental procedure

2.2.1. Freezing procedure

Wine samples (45 ml) contained in plastic centrifugal tubes (internal diameter D = 22 mm) were frozen in a static freezer at −30 °C for 12 h. This condition allows a moderate freezing rate, avoiding a fast freezing may occlude solutes (Nakagawa, Maebashi, & Maeda, 2010a; Moreno, Raventós, Hernández, & Ruiz, 2014a). As shown in Fig. 1, the external surface of the plastic tubes was covered with thermal insulation made of foamed polystyrene (8 mm thickness, thermal conductivity K = 0.035 W m^{-1} K^{-1}) so that heat transfer during freezing mainly occurred unidirectionally. During freezing, the temperature in the samples was measured using needle-type copper-constantan thermocouples (Ellab A/S, Rodovre, Denmark) at the geometric center of samples. Thermocouples were connected to a data acquisition system model CTF84-S8 (Ellab A/S, Copenhagen, Denmark) and registered continuously. The freezing rate (mm min^{-1} or μm s^{-1}) was calculated as the thickness divided by the freezing time (assuming that freezing occurs from one side) (Ramaswamy & Marcotte, 2006).

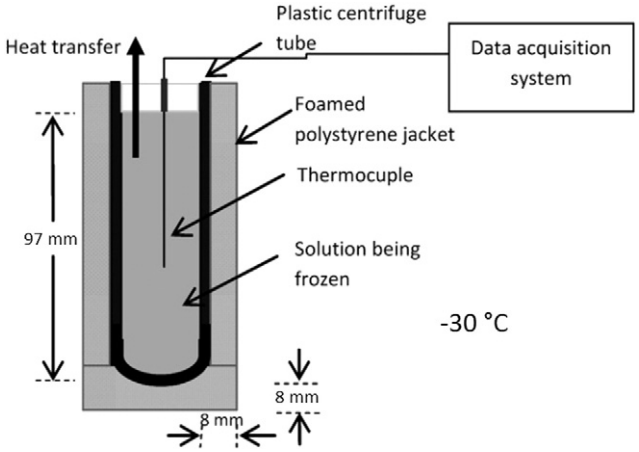

Fig. 1. Freezing condition of wine samples. The samples were frozen in a static freezer at −30 °C for 12 h.

2.2.2. Vacuum suction procedure

The vacuum suction was carried out according to the procedure of Petzold et al. (2013), with slight modifications. The frozen samples were removed from the freezer and rapidly transferred to a suction stage as illustrated in Fig. 2. The suction was generated by connecting a vacuum pump (model medi-pump 1636; Thomas Industries, Sheboygan, WI, USA) to the bottom of the frozen sample at controlled temperature (20 °C ± 1) with a refrigerated incubator (model FOC 215E; Velp Scientific Inc., Milano, Italy) and under vacuum (40 kPa), vacuum that remained constant throughout the test. This vacuum level was lower than previously used in our laboratories because a higher vacuum quickly fractured the frozen sample. Additionally, the vacuum level is in a range mentioned by the literature (27 and 57 kPa) (Moreno et al., 2013; Pardo & Sánchez, 2015). The vacuum was controlled visually with a vacuum manometer of the pump and an external manometer. After batch assays over the time (under vacuum condition), the concentrated solution was collected and the remaining frozen fraction thawed so that the concentration of solids (expressed in °Brix) was determined in both fractions. The concentration of fractions C_f and C_s (solids in the molten frozen phase and solution, respectively) obtained after assays were analyzed at ambient temperature (approx. 22 °C) with an ATAGO refractometer (model PAL-1, Tokyo, Japan) with a precision of ±0.1°Brix. All measurements were made in triplicate and each assay was performed six times with two replicates.

2.3. Physicochemical determinations

The alcoholic strength by volume (% vol.) has been determined by distillation method, according to the OIV method (OIV, 2015a). The total acidity of the samples expressed in grams of tartaric acid per liter was performed according to the OIV method (OIV, 2015b).

The total polyphenol content (TPC) present in the samples expressed in mg GAE/l was determined spectrophotometrically according to the OIV method using the Folin-Ciocalteu reagent (OIV, 2015c). pH of the samples was measured using a pH meter (model 3Star; Thermo Scientific Orion, USA) at ambient temperature (approx. 22 °C).

The color analysis of the samples was performed using a Spectrophotometer (Konica Minolta CM-5, Osaka, Japan) and expressed as CIE Lab values, whose coordinates were obtained using a D65 illuminant and a 2° observer as the reference system. The L* (lightness, black = 0, white = 100), a* (redness > 0, greenness < 0), and b* (yellowness, b* > 0, blue < 0) values were recorded. Ten replicates were performed for each treatment and then mean values were reported. Hue angle (h^*_{ab}, hue angle, red = 0°, yellow = 90°, 180° = green, 270° = blue), chroma (C^*_{ab}, 0 at the center of the color sphere), and total color change