



A comparative study of black mulberry juice concentrates by thermal evaporation and osmotic distillation as influenced by storage

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ABSTRACT

In the present study, black mulberry juice was concentrated using thermal evaporation and osmotic distillation after clarification and pasteurization. The main quality parameters of the concentrates were comparatively investigated after reconstitution with different storage times and temperatures. The parameters investigated were: color, turbidity, pH, titratable acidity, anthocyanin content, percent polymeric color, antioxidant activity, 5-hydroxymethylfurfural (HMF) content and volatile profile. The anthocyanin content, volatile content and turbidity values of osmotically distilled samples were found to be higher than those of the thermal concentrates. Neither HMF nor furfural was detected in the samples soon after the processing; however, levels of process contaminants were found to increase gradually throughout the storage period in both concentrates. Thermal evaporation resulted in markedly higher HMF and furfural formation in comparison to the osmotic distillation process. The anthocyanin content and antioxidant activity of the concentrates decreased, whereas polymeric color ratio and turbidity values increased within longer storage time and higher storage temperature.

Industrial relevance: Concentration is one of the key steps affects the chemical and physical properties of black mulberry juice since black mulberry phytochemicals, mainly anthocyanins, are labile to heat treatment and storage. Thermal evaporation has some drawbacks such as loss of fresh juice flavors, color degradation, reduction of nutritional value and formation of mutagenic compounds such as HMF and furfural. These drawbacks require alternative methods that involve minimal processing. Osmotic distillation can be recommended for black mulberry juice concentration in order to preserve its heat-sensitive components and to produce high-quality product.

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1. Introduction

The mulberry belongs to genus *Morus* of the family Moraceae. Three species (white (*Morus alba*), red (*Morus rubra*) and black (*Morus nigra*) mulberry) are widely grown in Turkey. The mulberry has been cultivated in Turkey for >400 years for traditional products such as “mulberry pekmez”, “mulberry pestil” and “mulberry kome” (Ercisli & Orhan, 2007, 2008). The black mulberry in particular has attracted attention in fruit markets and the food industry due to its phytochemicals and unique flavor (Jiao, Cassano, & Drioli, 2004; Özgen, Güneş, et al., 2009). Black mulberries can be consumed fresh or processed into marmalade, pekmez, natural dye, liquor and juice (Ercisli & Orhan, 2007; Fazaeli, Yousefi, & Emam-Djomeh, 2013; Pawlowska, Oleszek, & Braca, 2008).

Black mulberry juice is a potential source of anthocyanins that have high antioxidant activity and thus many health benefits (Kong, Chia, Goh, Chia, & Brouillard, 2003; Özgen, Serçe, & Kaya, 2009). However,

black mulberry phytochemicals, mainly anthocyanins, are labile to heat treatment and storage depending on temperature, light and pH (Fazaeli et al., 2013; Özgen, Serçe, et al., 2009; Tiwari, O'Donnell, & Cullen, 2009).

In order to reduce transport, storage, and packaging costs, fruit juice is concentrated. In addition, concentrates are more stable, presenting higher resistance to microbial activity than juices. The fruit juices are usually concentrated by thermal evaporation at high temperatures. This process results in loss of fresh juice flavors, color degradation, reduction of nutritional value and a cooked flavor formation. An additional drawback of thermal evaporation is the high energy cost despite the use of energy saving systems (Jiao et al., 2004; Petrotos & Lazarides, 2001). These disadvantages require alternative methods that involve minimal processing. Osmotic distillation is one of the promising non-thermal juice concentration methods to ensure safety and improve product quality. It is known to be successful in concentrating many liquid foods product such as milk, fruit and vegetable juice, beer, coffee, tea and herbal tea extracts (Bánvölgyi, Horváth, Stefanovits-Bányai, Békássy-Molnár, & Vatai, 2009; Jiao et al., 2004; Onsekizoglu, Bahceci, & Acar, 2010; Torun et al., 2014). This concentration technique can be

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used to extract selectively the water from aqueous solutions at atmospheric pressure and ambient temperatures, thus avoiding thermal degradation of the solutions. Moreover, volatile compounds have a lower mass diffusivity in liquid and gas phases than water. Thus, transfer of aroma compounds is not significant, compared with the transfer of water. Osmotic distillation is, therefore, adapted to the concentration of fruit juices that are rich in heat-sensitive and volatile components. However, due to some limitations of membrane modules, juices must be clarified before the concentration (Aguiar et al., 2012).

The process involves the use of a microporous hydrophobic membrane to separate two circulating aqueous solutions at different solute concentrations: a dilute solution and a hypertonic salt solution. The difference between the two solute concentrations, which means difference between the water activities, generates a vapor pressure difference at the vapor–liquid interface causing a vapor transfer from the dilute solution towards the stripping solution. Water transport through the membrane can be summarized in three steps: (i) evaporation of water at the dilute vapor–liquid interface; (ii) diffusional or convective vapor transport through the membrane pores; (iii) condensation of water vapor at the membrane/brine interface (Jiao et al., 2004).

So far, osmotic distillation studies have mainly focused on operation conditions and their affect the concentrates. There are also limited works on the osmotic distillation of juices related to juice quality parameters, especially volatile compounds. However, to our best knowledge there is no work on the osmotic distillation of black mulberry juice. Thus, the aim of the present study was to compare the primary quality parameters of black mulberry juice concentrates produced by osmotic distillation and thermal evaporation, as influenced by storage.

2. Materials and methods

2.1. Sample preparation

Black mulberries (*M. nigra*) were purchased from two different local producers (Eğridir, Isparta, and Tire, İzmir, Turkey) and kept at $-18\text{ }^{\circ}\text{C}$ after being frozen. Then, they were manually pressed into the juice after overnight thawing at $4\text{ }^{\circ}\text{C}$. The juice was depectinized with 2 mL/L Pectinex ultracolor (Novozymes, Denmark) at $50\text{ }^{\circ}\text{C}$ for 90 min. The depectinized juice was clarified with 2.5 g bentonite, 0.1 g gelatin and 0.045 g kieselsol per liter of juice at $50\text{ }^{\circ}\text{C}$ for 2 h. The clarified juice was filtered and stored at $-18\text{ }^{\circ}\text{C}$ until used for treatments and analyses.

2.2. Concentration and storage

The clarified juice was pasteurized as described by Dinçer and Topuz (2015) in a jacketed beaker. Then the juice was concentrated by thermal evaporation and osmotic distillation.

A laboratory-size hollow fiber membrane module (MD 020 CP 2N, Microdyn, Germany) with 40 polypropylene capillaries with 2.8 mm outer and 1.8 mm inner diameter was used for osmotic distillation. The effective internal area of the membrane was 0.1 m^2 , and the average pore size was $0.2\text{ }\mu\text{m}$. The pasteurized black mulberry juice (1100 mL), at $14.5\text{ }^{\circ}\text{Bx}$, was pumped into the tube side. Brine solution (calcium chloride dihydrate at 65% w/w) was pumped into the shell side of the membrane. Both solutions were circulated in countercurrent mode using two peristaltic pumps (Heidolph PD 5006, Germany) (Fig. 1). The recycle flow rate was 20 L/h (flow rate was volumetrically determined) on both sides. The final juice concentration of $65\text{ }^{\circ}\text{Bx}$ was achieved in 810 min. The initial weight of the brine solution was three times higher than that of black mulberry juice in order to prevent significant dilution, which would decrease the driving force during experiments. The temperatures of both, juice and brine were $25 \pm 1\text{ }^{\circ}\text{C}$. After osmotic distillation, the membrane module was cleaned as follows. First, both sides of the membrane were rinsed with deionized water for 5 min. Then NaOH solution (2%) was circulated for 30 min. After rinsing again with deionized water for 5 min, citric acid solution

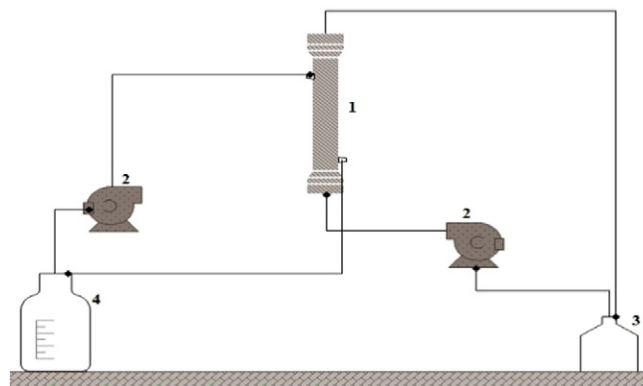


Fig. 1. Osmotic distillation experimental setup: (1) membrane module; (2) peristaltic pump; (3) feed container (4) brine solution container.

(2%) was circulated for 30 min. Finally, the circuit was rinsed with deionized water for 15 min.

Thermal evaporation of the black mulberry juices (500 mL) was performed to $65\text{ }^{\circ}\text{Bx}$ for 120 min by rotary evaporator (IKA RV10, Germany) at 100 rpm rotation speed and pressure of 250 mbar in a water bath maintained at $80\text{ }^{\circ}\text{C}$.

Equal amounts of concentrated samples in screw-cap plastic jars (50 mL) were stored at 4, 25 and $35\text{ }^{\circ}\text{C}$. Duplicate samples were taken at specified time intervals (15th, 30th, 45th, 60th and 90th day for $4\text{ }^{\circ}\text{C}$; 5th, 10th, 20th, 30th and 45th day for $25\text{ }^{\circ}\text{C}$; 2nd, 4th, 8th, 12th, and 16th day for $35\text{ }^{\circ}\text{C}$) from each storage temperature and stored at $-20\text{ }^{\circ}\text{C}$ until analyses. The sampling intervals were selected in regard to previous studies carried out on anthocyanin degradation for different materials (Kirca & Cemeroglu, 2003; Wang & Xu, 2007). All concentrated samples were diluted to the initial concentration of black mulberry juice ($14.5\text{ }^{\circ}\text{Bx}$) with deionized water prior to analyses.

2.3. Analyses

Color analysis was carried out using a tristimulus colorimeter (Konica Minolta Sensing, Inc. Japan) equipped with a CR-400 measuring head. Color values were expressed as L (darkness/whiteness), a (greenness/redness) and, b (blueness/yellowness) (Patras, Brunton, O'Donnell, & Tiwari, 2010). The instrument was standardized against a white ceramic plate where $L = 95.24$, $a = -0.31$ and $b = 3.02$. Turbidity was determined using a turbidimeter (Hach 2100 N Turbidimeter, USA) using sample cells (95 mm high \times 25 mm diameter). The values read from the turbidimeter were expressed as nephelometric turbidity units (NTU) (Tajchakavit, Boye, Belanger, & Couture, 2001). pH was measured with a pH meter (Orion 4-Star pH meter, Thermo Scientific, USA) at $20\text{ }^{\circ}\text{C}$. Titratable acidity (TA) was determined by potentiometric titration with 0.1 N NaOH up to pH 8.1 and expressed as g malic acid/100 mL juice (AOAC, 2002).

Total monomeric anthocyanin levels were measured using a pH-differential method described by Wang and Xu (2007) in combination with a two-buffer system that utilized potassium chloride buffer (0.025 M, pH 1.0) and sodium acetate buffer (0.4 M, pH 4.5). The wavelength of maximum absorption for black mulberry juice anthocyanins was determined as 514 nm. Black mulberry juice was brought to pH 1.0 and 4.5 with the buffer and allowed to equilibrate for 30 min. The absorbance of each equilibrated solution was then measured at 514 nm (λ_{max}) and 700 nm for haze correction, using a spectrophotometer (Shimadzu UV 1800, Japan) at room temperature ($\sim 25\text{ }^{\circ}\text{C}$). All absorbance readings were performed against distilled water as a blank. Total anthocyanins were calculated as cyanidin-3-glucoside according to the following Equation (1):

$$\text{Monomeric anthocyanin (mg/L)} = A \times MW \times DF \times 1000 / (\epsilon \times 1) \quad (1)$$

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