Desalination 250 (2010) 1101–1104

Contents lists available at ScienceDirect

Desalination

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

Physico-chemical parameters of cactus pear (*Opuntia ficus-indica*) juice clarified by microfiltration and ultrafiltration processes☆

Alfredo Cassano *, Carmela Conidi, Enrico Drioli

Institute on Membrane Technology, ITM-CNR, c/o University of Calabria, via P. Bucci, 17/C, I-87030 Rende (Cosenza), Italy

ARTICLE INFO

Available online 13 October 2009

Keywords:
Cactus pear juice
Microfiltration
Ultrafiltration
Nutraceutical foods

ABSTRACT

The health-promoting capacity of cactus pear fruit is highly attractive for the development of nutraceutical foods. The increasing market demand towards this fruit and products, which combine added value with a fresher taste, has challenged researchers to develop procedures to lengthen storage life. In addition, the possibility to obtain natural colorants from the cactus pear fruit rather than synthetic colorants for drinks and dairy products represents another interesting perspective.

In this study the effect of microfiltration (MF) and ultrafiltration (UF) processes on the physico-chemical composition of the cactus pear juice produced from fruits of Italian (Sicily) origin was investigated in order to evaluate the influence of the clarification treatment on the content of main parameters characterising the nutritional and functional properties of the fruit. Effects of operating parameters on the performance of both processes in terms of permeate fluxes were also evaluated.

1. Introduction

Cactus pear (*Opuntia ficus-indica* [L.] Miller) fruit can be considered a food of nutraceutical and functional importance due to its high content of chemical constituents characterised by nutritional and health-improving properties [1]. It is known in the traditional medicine for its hypoglycemic and hypolipidemic actions [2]. Scientific evidence has been provided about the benefits from fruit ingestion *in vivo* (decrease of body oxidative stress and cardiovascular protective effects in humans, antiulcer and hepatoprotective effects in rats) with special consideration for non-nutritive components as potentially active antioxidant phytochemicals [3]. A marked antioxidant activity was recognised by Butera et al. [4] in aqueous extracts from red, yellow and white fruits from Sicilian cultivars. Protective effects and activity of fruit extracts in animals and biological models have been also investigated [3].

The nutritional importance of cactus pear fruit is mainly due to the content of ascorbic acid, fibres and free amino acids (particularly proline, glutamine and taurine) [5]. Other components such as lipids, proteins, organic acids and minerals do not differ significantly from other tropical fruits. The fruit is also characterised by a high content of betalain, a widely used natural colorant in the food industry.

In some countries cactus pear juice is consumed at home, in vegetarian restaurants or in local health-food stores. Since technological problems are associated with its production, no commercial products are produced at industrial level. The high pH value of the pulp (5.3–7.1) combined to its low acidity (0.05–0.18% in citric acid) strongly affects its storage life in the fresh state and the processing operations [6]. In addition, highly reactive molecules such as free radical-scavengers and antioxidants may be damaged during conventional operations, such as thermal sterilisation or evaporation, for juice preparation.

Different studies were done in order to reduce the pH value of the juice [7,8]. Another possibility related to cactus pear juices was concentrated juice production. Concentrated juices can be obtained with 63–67°Brix by centrifuge vacuum evaporator at approximately 40 °C. However the thermal treatment determines damage to the colour and herbaceous aroma appears after the concentration process [9].Colour changes were also observed during thermal treatment in pasteurised and concentrated juices of green cactus pear [10]. Researches were also made in order to obtain clarified juices. Using a NOVO prepared with a mix of pectolitic enzymes and a high activity of arabanase, Sáenz et al. clarified cactus pear juice with success [11].

The multiple-ingredient characteristics of cactus pear should encourage research to obtain different products with emerging technologies. In particular, the health-promoting capacity of cactus pear fruit is highly attractive for the development of nutraceutical foods. In addition, the possibility to obtain natural colorants from the cactus pear fruit rather than synthetic colorants for drinks and dairy products represents another interesting perspective.

Compared with traditional juice processing methods, membrane processes are low-cost and athermal separation techniques which involve no phase change or chemical agents. These features are becoming very important factors in the production of new fruit juices with natural fresh taste and additive-free [12]. The application of membrane processes for fruit juices has been extensively studied and some industrial processes are already consolidated for juices like

☆ Presented at the 12th Aachener Membrane Kolloquium, Aachen, Germany, 29-30 October, 2008.

* Corresponding author. Tel.: +39 0984 492067; fax: +39 0984 402103.
E-mail address: a.cassano@itm.cnr.it (A. Cassano).

0011-9164/$ – see front matter © 2009 Elsevier B.V. All rights reserved.
doi:10.1016/j.desal.2009.09.117

apple and orange [13–15]. However few works were developed for cactus pear juice [16,17].

In this study the effect of microfiltration (MF) and ultrafiltration (UF) processes on the physico-chemical composition of the cactus pear juice produced from fruits of Italian (Sicily) origin was investigated in order to evaluate the effect of the clarification treatment on the content of main parameters which characterise the nutritional and functional properties of the fruit. Permeate and retentate samples were analysed in order to evaluate their potential interest for the production of nutriaceutical foods, beverages and colouring foodstuffs.

2. Materials and methods

2.1. Cactus pear juice

Cactus pear juice was supplied by Citrech Snc (Messina, Italy). It was obtained from fresh *O. ficus-indica* [L.] Mill. fruits of the yellow cultivar purchased from Sicily/Italy. Fruits were washed in water and then manually peeled. Seeds and mesocarp fibres were removed with a squeezer and then washed with water. The raw puree and the cleaning solution were mixed and then treated with a pectinase from *Aspergillus aculeatus* (Pectinex Ultra SP-L, Novo Nordisk A/S, Novo Allè, Bagsuaerd, Denmark) in concentration of 1%, for 4 h at room temperature. The depectinised juice was filtered with a nylon cloth and stored at −17 °C. It was defrosted to room temperature before use.

2.2. Experimental set-up and procedure

Micro- and ultrafiltration experiments were performed by using a laboratory pilot unit manufactured by Permeare Srl (Milan, Italy). The equipment consists of a feed tank with a capacity of 10 L in stainless steel 316 L, a volumetric pump for high pressure operation (up to 75 bar), a cooling device, a thermometer, a feed flow meter, two pressure cells for flat sheet membranes, two manometers to measure the inlet pressure of each cell, a manometer to measure the outlet pressure of both cells and an electric board.

Polyvinylidenfluoride (PVDF) flat sheet membranes were supplied by Microdyn-Nadir GmbH (Wiesbaden, Germany). The MF membrane (MV020) was characterised by a pore size of 0.20 μm; the nominal molecular weight cut-off of the UF membrane (FMU6R2) was 200 kDa. The effective filtration area of each membrane was 11.33 cm^2.

The juice was clarified according to a batch concentration procedure (the permeate was collected separately and retentate was recycled to the feed tank) in selected operating conditions (transmembrane pressure 2.2 bar, axial feed flow rate 500 L/h, temperature 25 ± 2 °C).

2.3. Physico-chemical parameters

Samples of fresh, clarified and concentrated juice coming from MF and UF experiments were collected and stored at −20 °C for further analyses.

The suspended solid content (SS) was determined in relation to total juice (w/w%) by centrifuging, at 2000 rpm for 20 min, 45 mL of a pre-weighted sample; the weight of settled solids was determined after removing the supernatant. Total soluble solids (TSS) were determined by an Abbe refractometer Bellingham + Stanley 60/DR (Bellingham + Stanley Ltd., Kent, UK) at 20 °C. pH was measured by an Orion Expandable ion analyzer EA 920 pH meter (Allometrics, Inc. LA, USA) with automatic temperature compensation. Proteins were determined by the Bradford method. The procedure is based on the observation that the absorbance maximum for an acidic solution of Coomassie Brilliant Blue G-250 shifts from 465 nm to 595 nm when binding with protein occurs [18]. BSA (bovine serum albumin) was used as standard protein. Total phenolics were estimated colorimetrically using the Folin–Ciocalteau method and the results were expressed as gallic acid equivalents (GAE) in mg/L of cactus juice [19]. Total acidity was determined by titration with 0.1 N NaOH and expressed as percent of citric acid.

Quantification of betalains was carried out in triplicate in deionised water without pH adaptation applying the molar extinction coefficients of betanin ($\varepsilon = 60{,}000$ L/mol cm in H_2O; $\lambda = 538$ nm; MW = 550 g/mol) and indicaxanthin ($\varepsilon = 48{,}000$ L/mol cm in H_2O; $\lambda = 480$ nm; MW = 308 g/mol). The juice was diluted with deionised water to obtain absorption values of $0.8 \le A \le 1.0$. The betalain content (BC), expressed as mg/L, was calculated by using the following equation:

$$BC = \frac{A \times DF \times MW \times 1000}{\varepsilon \times L} \quad (2.3.1)$$

where A is the absorption at 538 and 480 nm for betacyanins and betaxanthins, respectively; DF is the dilution factor and L the pathlength of the 1-cm cuvette. For MW and ε, the molecular weights and extinction coefficients of the representative compounds betanin and indicaxanthin have to be considered, respectively [20].

Total antioxidant activity (TAA) was determined by an improved version of the 2,2′-azino-bis-(3-ethylbenzothiazoline-6-sulfonic acid) (ABTS) free radical decolourisation assay [21] in which the ABTS radical cation is generated by reaction with potassium persulphate before the addition of the antioxidant [22]. The decolourisation of the ABTS is measured as the percentage inhibition of absorbance at 734 nm. The concentration of antioxidant giving the same percentage inhibition of absorbance of the radical cation at 734 nm as 1 mM 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (Trolox) was calculated in terms of Trolox Equivalent Antioxidant Capacity (TEAC) at 5 min contact.

ABTS, potassium persulphate and Trolox were obtained from Sigma-Aldrich (Milan, Italy).

Spectrophotometric measurements were performed by a UV-160A UV–Visible Recording spectrophotometer (Shimadzu Scientific Instruments, Inc., Japan) at 30 °C.

The quantification of ascorbic acid was performed with an HPLC system (Agilent 1100 Series, USA) equipped with a pump, an UV–Vis detector and a data acquisition system. Chromatographic separation was performed by using a Luna C18(2) column (250 × 4.6 mm, 5 μm) (Phenomenex, Torrance, CA, USA); samples were eluted in isocratic mode by using a mixture of 20 mM KH_2PO_4 solution and acetonitrile at 95:5. Operating conditions were as follows: flow rate 0.8 mL/min, operating temperature 25 °C, pressure 90 bar. A sample volume of 20 μL was used. Analyses were monitored at 245 nm. Prior to HPLC analysis, all samples were filtered by using acetate cellulose filters with 0.45 μm pore size and diluted with pure water. The concentration of ascorbic acid was determined from experimental peak area by analytical interpolation in a standard calibration curve and was expressed as mg/L of juice.

3. Results and discussion

Fig. 1 shows the time course of the permeate flux obtained in the MF and UF treatment of the depectinised cactus pear juice in selected operating conditions (TMP = 2.2 bar; feed flow rate = 500 L/h; temperature = 25 °C). The permeate flux decreased gradually with the operating times due to the concentration polarization and gel formation. The J_p vs. time curve could be divided in three periods: an initial period in which a rapid decrease of permeate flux occurs; a second period characterised by a smaller decrease of the permeate flux; a third period in which the permeate flux reaches a steady-state value (48.8 L/m^2h in the MF process and 41.8 L/m^2h in the UF process, respectively).

Tables 1 and 2 show the results of physico-chemical determinations performed on samples coming from the MF and UF treatments. The original juice is characterised by a high pH value (5.5–5.7) and a very low acidity (0.03% in citric acid). These values are in agreement with those reported in literature for the yellow Italian cultivar [23]. Total soluble solids, pH and acidity remained unchanged in the clarified juice of both processes. On the contrary, suspended solids were completely removed by MF and UF membranes. Total soluble solids appear to be higher in the retentate: this behaviour is probably