Journal of Food Engineering 107 (2011) 366–373

Contents lists available at ScienceDirect

Journal of Food Engineering

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

Clarification and concentration of pomegranate juice (*Punica granatum* L.) using membrane processes

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ARTICLE INFO

Article history:
Received 30 April 2011
Received in revised form 28 June 2011
Accepted 3 July 2011
Available online 13 July 2011

Keywords:
Pomegranate juice
Ultrafiltration
Osmotic distillation
Integrated membrane processes

ABSTRACT

The production of concentrated pomegranate juice was investigated by using a two-step membrane process based on: (1) a clarification step of the fresh non-depectinized juice performed by hollow-fiber ultrafiltration (UF) membranes; (2) a concentration step of the clarified juice by using an osmotic distillation (OD) apparatus. Both processes were performed at ambient temperature (25 ± 2 °C) producing a clear juice and a concentrated juice with a total soluble solids (TSS) content of 162 g kg^{-1} and 520 g kg^{-1}, respectively.

The performance of UF and OD operations was evaluated in terms of productivity (permeate and evaporation fluxes) and quality of the processed juice.

Suspended solids were completely removed in the clarification step, while soluble solids and organic acids were recovered in the permeate stream of the UF process. Rejections of the UF membrane towards polyphenols and anthocyanins were of 16.5% and 11.7%, respectively.

The antioxidant activity of pomegranate aril juice, attributed to a great extent to total phenols and anthocyanins content, was efficiently preserved during the concentration step independently on the achieved level of total soluble solids.

An integrated membrane process scheme for the production of concentrated pomegranate juice with potential applications for food, pharmaceutical and cosmetic sectors was proposed.

1. Introduction

Significant progresses have been made over the past 10 years towards a much more comprehensive understanding of some pharmacologic components of pomegranate fruit (*Punica granatum*). Clinical research studies suggest that some compounds of the pomegranate juice are characterized by anticarcinogenic, antimicrobial, antioxidant and antiviral activities (Malik et al., 2005; Noda et al., 2002; Braga et al., 2005; Vidal et al., 2003). Further, as reported in biological studies, pomegranate juice is enriched in anti-atherosclerotic and anti-atherogenic compounds which have been shown to reduce blood pressure and low density lipoprotein (LDL) oxidation (Aviram and Dornfeld, 2001). These activities have been attributed to its phenolic fraction containing a significantly high level of hydrolyzable tannins, as well as anthocyanins (delphinidin, cyanidin and pelargonidin 3-glucosides and 3,5-diglucosides), which exhibit high antioxidant activity (Lansky and Newmann, 2007; Visioli and Hagen, 2007). Besides, different studies have shown that pomegranate juice contains much more antioxidant compounds than other fruit juices and beverages (Gil et al., 2000).

Due to these characteristics and increasing public awareness about nutritional food, the demand for the pomegranate fruit and its by-products has increased significantly in the last years. Consequently, many industries producing pomegranate juice, as well as pharmaceutical companies extracting health beneficial compounds from the fruit, have been developed (Seeram et al., 2006).

Traditional methods of processing fruits limit the possibility to obtain products able to retain as much as possible the peculiarity of the fresh fruit and its high content in health-beneficial compounds. For instance, conventional juice clarification processes are based on the use of fining agents (gelatine, bentonite, silica sol, diatomaceous earth, etc.) which create serious problems of environmental impact due to their disposal (Vaillant et al., 1999). Moreover, the concentration of fruit juices, aiming at ensuring a longer storage life and an easier transportation, is performed by thermal evaporation methods resulting in color degradation and reduction of most thermally sensitive compounds, with a consequent remarkable qualitative decline (Jiao et al., 2004).

Product quality improvement and energy savings have guided the development of *minimal processing* techniques. Compared with traditional juice processing methods, membrane processes are low-cost and athermal separation techniques which involve no

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doi:10.1016/j.jfoodeng.2011.07.002

phase change or chemical agents. These features are becoming very important factors in the production of new fruit juices with natural fresh tastes and additive-free.

Ultrafiltration (UF) membranes are commonly used to clarify and stabilize fruit juices and commercial processes have already implemented on industrial scale for juices such as apple and orange (Girard and Fukumoto, 2000). They are able to retain large species such as microorganisms, lipids, proteins and colloids while small solutes such as vitamins, salts, and sugars flow together with water. Advantages of the UF over conventional fruit juice processing are in terms of: increased juice yield; possibility of operating in a single step; possibility of avoiding the use of gelatins, adsorbents and other filtration aids; reduction in enzyme utilization; easy cleaning and maintenance of the equipment; elimination of needs for pasteurization; better juice clarity; reduction of filtration times and waste products (Fukumoto et al., 1998).

Concentration processes of the clarified pomegranate juice by using thermal evaporation have already been proposed. In particular, Chen and Song (1994) developed a fresh-preserving concentrated pomegranate juice producing method adopting a low temperature vacuum process for the concentration of the filtered juice. Pang et al. (2008) patented a method in which the clarified juice was concentrated by thermal evaporation and then submitted to pasteurization before the aseptic packaging. The concentrated juice is characterized by a pH value between 2.8 and 3.7, turbidity from 0 to 25 NTU, a transmittance (T_{625}) equal to or greater than 90% and a color ratio (A_{520nm}/A_{430nm}) equal to or greater than 1.0.

Membrane concentration processes, such as reverse osmosis (RO), membrane distillation (MD) and osmotic distillation (OD), present some attractive options to overcome limitations associated with vacuum evaporation. In particular, OD is a novel athermal membrane-separation process based on the use of a macroporous hydrophobic membrane separating two aqueous solutions having different solute concentration: a dilute solution on one side and a hypertonic salt solution (concentrated brine stripper) on the opposite side. The driving force of the process is given by a water vapor pressure gradient across the membrane generated by the difference in water activity between the two sides of the membrane. The hydrophobic nature of the membrane prevents penetration of the pores by aqueous solutions, creating air gaps within the membrane. The water vapor pressure gradient across the membrane determines a transfer of vapor across the pores from the high-vapor pressure phase to the low one (Hogan et al., 1998; Nagaraj et al., 2006). This migration of water vapor results in the concentration of the feed and dilution of the osmotic agent solution. Since the OD process is operated at room temperature and atmospheric pressure, products can be concentrated without mechanical and thermal damage. Therefore OD is an attractive process for the concentration of solutions containing thermo-sensitive compounds such as fruit juices and pharmaceuticals.

The concentration of clarified fruit juices by OD has been investigated by different authors (Cassano and Drioli, 2007; Cassano et al., 2007; Hongvaleerat et al., 2008; Rodrigues et al., 2004; Shaw et al., 2001; Kozák et al., 2006) and its potential for the better preservation of the quality of the raw material has been clearly confirmed.

The present study was undertaken to investigate the potentiality of an integrated membrane process in the clarification and concentration of pomegranate juice, as an alternative to conventional processing methods. In particular, the fresh juice, with an initial total soluble solids (TSS) content of 162 g kg^{-1}, was preliminarily clarified by ultrafiltration (UF). Then the clarified juice was submitted to a concentration step, at room temperature, by using osmotic distillation (OD) up to a final TSS content of about 520 g kg^{-1}. The performance of UF and OD processes was evaluated in terms of both productivity (permeate and evaporation fluxes) and quality of the processed juice.

2. Materials and methods

2.1. Juice extraction

Pomegranates of Sicilian origin (*Punica granatum* L. cv *Selinunte*) were supplied by Citrech Snc (Messina, Italy). Fruits were washed in cold tap water and drained. They were manually cut-up and the outer leathery skin which encloses hundreds of fleshy arils was removed. Arils were pressed by using an electric tomato crusher: the extracted juice, having a deep-red color, was prefiltered with a stainless steel filter. Then it was stored in a refrigerator cell at −17 °C and defrosted before use.

2.2. UF equipment and procedures

The clarification of the juice was performed by using a laboratory UF bench plant (DSS LabUnit M10) supplied by Danish Separation System AS, Denmark. The equipment consisted of a 5 L feed tank, a cross-flow pump (ECO type GA4-KDT-TTU), two pressure gauges (0–2.5 bar) located at the inlet (P_{in}) and outlet (P_{out}) of the membrane module, a pressure control valve and a multi-tube heat exchanger fed with tap water. The temperature (T) of the feed was controlled by circulating cooling water through the heat exchanger; the axial feed flow-rate (Q_f) and the transmembrane pressure (TMP) were controlled by using a needle concentrate valve and by setting the speed of the pump.

Modified poly(ether ether ketone) hollow fiber (HF) membranes were prepared via the dry-wet spinning technique through the phase inversion process (Tasselli et al., 2007). Their characteristics are reported in Table 1. The UF plant was equipped with a membrane module prepared by embedding five HF membranes inside a 20 cm long glass tube (effective membrane length 18 cm, effective membrane area 46 cm^2) with epoxy resin (Stycast, Emerson and Cuming, Belgium).

The juice was clarified according to the batch concentration mode (recycling the retentate in the feed tank and collecting separately the permeate stream) in the following operating conditions: TMP, 0.96 bar; T, 25 °C; Q_f, 1166 mL min^{-1}.

The hydraulic permeability of the membrane was determined by the slope of the straight lines obtained plotting the water flux values in selected operating conditions (T, 25 °C; Q_f, 900 mL min^{-1}) versus the applied TMP. The value obtained for a new clean membrane is referred to as L_p^0.

The hydraulic permeability measured after the treatment with pomegranate juice was indicated as L_p^1.

After the experiments with pomegranate juice the membrane module was cleaned in two steps. The first cleaning step was performed recirculating distilled water for 20 min through the module at a Q_f of 1500 mL min^{-1} and a TMP of 0.4 bar in order to remove the reversible polarized layer. The hydraulic permeability measured afterwards was L_p^2. In the second step the membrane module was submitted to a cleaning procedure using a NaClO solution at a

Table 1
Characteristics of UF membranes.

Configuration	Hollow-fibers
Material	Poly(ether ether ketone)
Water permeability (L m^{-2} h^{-1} bar^{-1})	655.6
Dextran 68,800 MW rejection (%)	10.0
Thickness (mm)	0.235
Inner diameter (mm)	1.64
Outer diameter (mm)	2.11