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Direct contact membrane distillation for the concentration of clarified orange juice

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ABSTRACT

Multi-stage vacuum evaporation is a conventional unit operation in the industrial production of concentrated fruit juices. Membrane processes, such as direct contact membrane distillation (DCMD) and osmotic distillation (OD), offer several advantages over the traditional thermal evaporation, since they operate at a lower temperature thus preserving the nutritional and organoleptic properties of the fresh juice. These processes involve transport of water vapor through the pores of hydrophobic membranes due to a vapor pressure driving force provided by temperature and/or solute concentration differences across the membrane. In DCMD water is used on the permeate side instead of a stripping solution, thus increasing the sustainability of the process.

The aim of this study was to evaluate the potential of DCMD for concentrating clarified orange juice on laboratory scale. The raw juice was previously clarified by ultrafiltration (UF) in order to remove suspended solids and juice turbidity; the clarified juice, with an initial total soluble solids (TSS) content of about 9.5 °Brix, was pre-concentrated up to 24 °Brix and then concentrated up to 65 °Brix through a two-step DCMD process. The performance of both UF and DCMD processes was evaluated in terms of productivity and juice quality. Samples from both processes were analyzed in terms of total suspended solids, TSS, total antioxidant activity (TAA) and phenolic compounds. Concentrated samples were also analyzed in order to evaluate the presence of crystals.

Experimental results indicated that, at high concentration, the transmembrane flux decay can be mainly attributed to the increasing of the juice viscosity. Moreover, analytical measurements proved that all the antioxidant compounds of the clarified orange juice are well preserved through the DCMD process. Therefore, the UF/DCMD integrated process may be used to obtain high quality concentrated juices, as in the final product the organoleptic, nutritional and antioxidant properties of the fresh juice are efficiently preserved.

1. Introduction

Nowadays fruit juices reconstituted from concentrate (RFC) are the main products available on the market. Indeed, the concentration procedure reduces packaging, storage, transportation and distribution operations with great economic benefits. In addition, the removal of water from fruit juices through concentration guarantees their conservation due to the water activity reduction. Commercial concentration processes usually involve the use of multi-stage vacuum evaporation. This process leads to a thermal degradation of sensorial and nutritional characteristics with partial loss of aroma and nutrients, induction of cooked taste due to furfural formation and browning due to Maillard-reactions (Arena et al., 2001). Mild technologies, such as freeze-concentration, preserve the juice quality but they are more expensive and limited in

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the degree of achievable concentration.

Membrane processes represent relevant alternatives to traditional unit operations in food productions thanks to their capacity to operate at moderate temperatures avoiding phase changes and the use of chemical additives (Daufin et al., 2001). The use of reverse osmosis (RO) to concentrate clarified fruit juices has been largely investigated in the past: this process is characterized by a lower power consumption when compared to thermal evaporation, a high rate of retention of compounds of nutritional and sensorial interest and preservation of thermosensitive compounds (Jiao et al., 2004). However, the efficiency of the process is strongly limited by the high pressure build-up for concentration which increases the operation costs; therefore, lower concentration levels can be achieved in comparison with thermal evaporation. For this purpose RO is generally used as a pre-concentration step of clarified fruit juices in order to reach concentration levels up to 25–30 °Brix (Jesus et al., 2007).

Membrane distillation (MD) and osmotic distillation (OD) are non-pressure driven membrane processes which have been introduced as new membrane techniques to concentrate heat-sensitive fruit juices. In these processes the driving force for mass transfer is a vapor pressure difference across the membrane generated by either a temperature gradient (in MD) or a water activity difference (in OD). Their main advantages over traditional pressure-driven membrane processes are: low fouling, possibility of treatment of highly viscous solutions, high retention of species, low energy consumption. Furthermore, these processes are not limited by high osmotic pressures and allow to reach concentration levels similar to those obtained in thermal evaporation (Belafi-Bako and Koroknai, 2006).

A full exploitation of the potential of these techniques may be achieved through their combination with microfiltration (MF) and ultrafiltration (UF) processes that remove suspended solids and pectins from juices, decreasing the juice viscosity and improving MD and OD fluxes with minimal losses of nutrients and flavours. Integrated membrane processes based on a preliminary clarification of juices by MF or UF, followed by an OD concentration step have been investigated and designed for different fruit and vegetable juices such as apple (Aguiar et al., 2012; Onsekizoglu et al., 2010), blackcurrant (Kozák et al., 2008; Sotoft et al., 2012), blood (Galaverna et al., 2008) and yellow orange (Cisse et al., 2005), acerola (Pagani et al., 2011), bergamot (Cassano et al., 2013), camucamu (Souza et al., 2013), carrot (Cassano et al., 2003), grape (Rektor et al., 2006), passionfruit (Shaw et al., 2001), red radish (Patil and Raghavarao, 2007), cactus pear (Cassano et al., 2007), melon (Vaillant et al., 2005), kiwifruit (Cassano et al., 2004), pineapple (Hongvaleerat et al., 2008) and pomegranate juices (Cassano et al., 2011).

Although both MD and OD processes can be extended in order to reach supersaturation and, therefore, crystals formation, one of the disadvantages of OD is represented by the use of stripping solutions which should be continuously reconcentrated during the process in order to keep high driving forces to sustain the process productivity; additional problems arise from the management and the disposal of brine solutions. On the other hand, in MD distilled water is used on the permeate side instead of a stripping solution thus increasing the sustainability of the process.

This study aimed at evaluating the effect of a combined membrane process of UF and MD on the quality of blood orange juice. The raw juice was previously clarified by UF; then the clarified juice was concentrated by using DCMD in two different steps: a pre-concentration step characterized by an almost constant value of the juice viscosity and transmembrane flux, up to 24 °Brix, and a concentration step up to 65 °Brix, characterized by an increase of the juice viscosity. The performance of both UF and DCMD operations was evaluated in terms of productivity (permeate flux in UF, evaporation flux in DCMD) and quality of processed juice. In addition, at high concentration levels of the juice, concentrated samples were analyzed to check the presence of crystals.

2. Materials and methods

2.1. Juice extraction

Fresh blood oranges of Sicilian origin were purchased from a local market (Rende, Cosenza, Italy). Fruits (129.4 kg) were manually washed in water, cut in two halves and squeezed by a domestic juicer (Aristarco S.r.l., Treviso, Italy). A commercial pectinase from *Aspergillus aculeatus* (Pectinex® Ultra SP-L, Novo Nordisk A/S, Denmark), with activity of 9500 PGU/mL, was used for the enzymatic treatment of the juice. The enzyme is able to hydrolyse both high and low esterified pectins and also partially hydrolyze cellulose, hemicellulose, starch and proteins, thus decreasing the viscosity to a greater extent. The juice was incubated with 20 mg/L of enzyme at room temperature for 4 h and then filtered with a nylon cloth. Finally, 0.1 w/w% sodium benzoate was added in order to prevent the microbial fermentation during the juice treatment. The extracting procedure gave an average juice yield of 46.4% (w/w). The juice was stored at −17 °C and defrosted at room temperature before use.

2.2. Juice clarification

The depectinized juice was clarified by using an ultrafiltration (UF) laboratory unit (Verind SpA, Milan, Italy) equipped with a polysulfone hollow fiber membrane module supplied by China Blue Star Membrane Technologies, Co., Ltd. (Beijing, China) having a nominal molecular weight cut-off (MWCO) of 100 kDa and an effective membrane area of 1.2 m^2.

The UF equipment consists of a 25 L stainless steel feed tank, a feed pressure pump, two manometers (0–400 kPa) and a magnetic flow meter for the measure of the axial feed flow rate. A tube and shell heat exchanger, placed after the feed pump, was used to maintain the temperature of the feed juice constant. The juice was clarified according to a batch concentration procedure (permeate was collected separately and retentate was recycled to the feed tank) at a transmembrane pressure (TMP) of 75 kPa, an axial feed flow rate (Q_f) of 830 L/h and a temperature (T) of 23 ± 2 °C up to a volume reduction factor (VRF) of 6.

At the end of each run, the plant and the membrane module were cleaned first with distilled water and then by recycling 0.1% (w/w) NaOH solution followed by a treatment with an enzymatic detergent (Ultrasil 50, Henkel Chemicals Ltd., Dusseldorf) at a concentration of 1% (w/w). Cleaning solutions were recirculated in the UF plant for 60 min, at a temperature of 40 °C, high flow rates and low TMP in order to avoid pore blocking phenomena. At the end of each cleaning procedure the membrane module was rinsed with distilled water for 20 min.

2.3. Membrane distillation equipment and procedures

The clarified juice, with a total soluble solids (TSS) content of 9.5 °Brix, was processed by using a membrane distillation (MD) laboratory bench plant equipped with two polypropylene hollow fiber membrane modules (Enka Microdyn MD-020- 2N-CP) having a nominal pore size of 0.2 μm and a membrane surface area of 0.1 m^2.

The plant consists of a 10 L stainless steel feed tank and a 5 L plastic permeate tank. It was equipped with all the necessary tools for the circulation of the streams, and for setting and controlling the