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Effect of the operating variables on the extraction and recovery of aroma compounds in an osmotic distillation process coupled to a vacuum membrane distillation system

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ABSTRACT

The coupled operation of osmotic distillation (OD) and vacuum membrane distillation (VMD) for concentration of fruit juices and simultaneous recovery of their aroma compounds was studied. The simulated aqueous fruit juices containing four common aroma compounds were concentrated using osmotic distillation where the feed solution was in contact with a brine solution of $CaCl_2$, through a hydrophobic macroporous membrane contactor. Aroma compounds absorbed in the extraction brine were extracted using a membrane evaporator under vacuum and collected into a cold trap. This way, both concentration and aroma recovery of fruit juices were achieved simultaneously using two hollow fiber membrane modules. The transfer of the aroma compounds was evaluated by using different operating variables such as hydrodynamic conditions, brine concentration and vacuum pressure. The experiments show that the loss of aroma compounds during the concentration processes can be avoided by means of extraction of the aroma compounds from the brine separately, resulting in an average of 75% recovery in aroma compounds. In general, the process of aroma removal and recovery is faster than the concentration process of the fruit juices by osmotic distillation at a technical and commercial level (higher than 45°Brix). Thus, the simultaneous operation of these two membrane processes can be used to decrease the energy requirements for a given production capacity.

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1. Introduction

Conventional thermal concentration of liquid foods present major drawbacks such as heat induced deterioration of perception and the loss of the nutritional value of the concentrated product. It is well known that most of the aroma compounds in the raw juice are lost in the first few minutes of evaporative concentration and the aroma profile degrades irreversibly causing a reduction in its quality (Petretos and Lazarides, 2001). An attractive alternative to conventional processes for fruit juice concentration is to use membrane separation techniques. The application of membrane techniques in fruit juices has been under investigation for a long time including osmotic distillation (Hogan et al., 1998; Valdés et al., 2009; Cassano et al., 2003, 2004, 2006, 2007; Cassano and Drioli, 2007; Vaillant et al., 2005; Koroknai et al., 2006; Rektor et al., 2006; Alves and Coelhoso, 2006; Belafi and Koroknai, 2006), ultrafiltration (Cassano et al., 2003, 2004, 2006, 2007; Cassano and Drioli, 2007), reverse osmosis (Cassano et al., 2003), pervaporation (Cassano et al., 2006), microfiltration (Vaillant et al., 2005), and membrane distillation (Koroknai et al., 2006; Rektor et al., 2006; Alves and Coelhoso, 2006; Belafi and Koroknai, 2006; Gunko et al., 2006). Membrane processes have promising possibilities for the fruit juice industry to concentrate diluted solutions containing heat-sensible compounds as they operate in an environmental-friendly and cost-efficient way at ambient temperatures and mild conditions. Both osmotic and vacuum membrane distillations are applied in this kind of operations effectively.

The content and the composition of the aroma compounds in a fruit juice concentrate define the quality of the product since these volatile compounds play an essential role in the sensory perception of the product. Osmotic distillation is an emerging membrane technique for fruit juice concentration because it does not generate thermal degradations. However, a loss of aroma compounds occurs during the operation. During the osmotic distillation some loss of the aroma compounds were reported in several studies (Ali et al., 2003; Torres et al., 2007; Talens et al., 2003; Shawet al., 2001; Diban et al., 2008; Varavuth et al., 2009). Ali et al. (2003) carried out a study about transfer of several aroma compounds which are produced from fruit juices during osmotic distillation opera-

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Nomenclature

$C_{f,t=0}$	initial concentration of a specific aroma compound in the feed solution (kg m^{-3})
$C_{f,t}$	concentration of a specific aroma compound in the feed solution at time t (kg m^{-3})
P_1, P_2	partial pressure of the component at the interfaces (Pa)
X_1^F	molar fraction of the component at the feed side interface (–)
X_2^B	molar fraction of the component at the brine side interface (–)
X_b^B, X_b^F	molar fraction of the component at feed boundary layer and brine boundary layer, respectively (–)

tions and reported that experimental loss ranged from 37% to 73% for hexyl acetate, from 43% to 77% for ethyl butyrate, from 36% to 52% for benzaldehyde and from 25% to 38% for hexanol. Shaw and coworkers (2001) studied the loss of 35 volatile compounds in orange juice and 22 volatile compounds in passion fruits after osmotic distillation process and reported that the average retention of individual volatile compounds was 66–69% for the orange juice while the average percent retention for the volatile component was 61% for the passion fruit. Diban et al. (2008) studied the loss of aroma compounds in wine after dealcoholization process using osmotic distillation. According to the experiments with different conditions carried out by Dibat et al., the percentages of loss of ethyl acetate varies between 37.4% and 65.2%, ethyl hexanoate varies between 29.3% and 65.5%, isoamyl acetate varies between 31.3% and 51.8%, ethyl octanoate varies between 57.5% and 98.1%.

Although there are reported works about the transfer of aroma compounds in liquid food operations (Ali et al., 2003; Torres et al., 2007; Diban et al., 2008), a procedure was not encountered to recover these aroma compounds during the concentration operations. The literature on the subject tends to indicate that aroma recovery processes using membrane techniques are independent from juice concentration. In this work, a vacuum membrane distillation (VMD) system was coupled to the brine stream of an osmotic distillation (OD) process in order to overcome the aroma compounds loss problem by recovering them from brine. Both osmotic and vacuum membrane distillation use similar membrane materials and module configurations, and the driving force for both membrane processes is the vapor pressure difference between the two sides of the membrane. The brine solution that leaves the osmotic distillation module was pumped through the vacuum membrane distillation and circulated in a close loop while a vacuum is applied on the other side of the membrane evaporator. Thus, the aroma compounds were extracted from the brine, and collected and preserved in a cold trap that was settled on the vacuum line. With the use of OD and VMD coupling, simultaneous concentration and aroma recovery of fruit juices was achieved in terms of an integrated membrane process for the production of a concentrated fruit juice with a high content of aroma compounds. The effect of process parameters on the aroma extraction was evaluated by analytical measurement of the content of the aroma compounds in the cold trap and the feed solution, the sum of these represent the quality of the final product.

1.1. Osmotic distillation (OD) of fruit juices

The applications of osmotic distillation for fruit juice concentration have been under investigation lately (Hogan et al., 1998; Valdés et al., 2009; Cassano et al., 2003, 2004, 2006, 2007; Cassano and Drioli, 2007; Vaillant et al., 2005; Koroknai et al., 2006; Rektor et al., 2006; Alves and Coelhoso, 2006; Belafi and Koroknai, 2006). Osmotic distillation has several advantages over traditional thermal concentration: no thermal degrading of the volatiles, has potential to concentrate liquids to a very high level under mild operating conditions and it presents no product destruction.

Osmotic distillation for fruit juice concentration applications employs a hydrophobic membrane to separate two liquid phases, the feed and a brine solution, that differ greatly in terms of solute concentration. The hydrophobic nature of the membrane prevents penetration of the pores by aqueous solutions, creating air gaps within the membrane. The difference in solute concentration, and consequently in water activity between the two sides of the membrane, induces a vapor pressure difference causing water vapor transfer across the pores from a high-vapor pressure phase to the lower one. Accordingly, the process takes place in three steps, which includes vaporization and condensation on the boundary layers, and water vapor diffusion across the membrane as shown in Fig. 1. The participating solutions cannot come in contact with each other, which in fact is very important in the applications of the food industry.

In the osmotic distillation process the water flux through the membrane is proportional to the bulk water vapor pressure difference between two sides of the membrane. The most important parameters that affect the OD flux are basically feed and brine solution flow rate and concentration of brine. The flow rates directly affect the thickness of the boundary layer that presents a resistance for the mass transfer, while the concentration of brine affects the vapor pressure gradient through the membrane, which is directly related with magnitude of the driving force.

1.2. Vacuum membrane distillation (VMD)

Vacuum membrane distillation process is based upon using a macroporous hydrophobic membrane for the separation of an aqueous feed solution and a downstream gaseous phase kept under vacuum. The process is characterized by evaporation of the liquid mixture at the liquid-membrane interface and by mass transport of vapors through the membrane pores by means of the pressure difference induced by the vacuum on the permeate

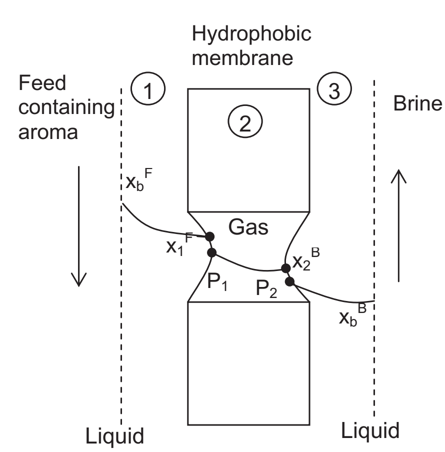

Fig. 1. Concentration profile through the membrane in the OD process.

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