



Osmotic distillation applying potassium pyrophosphate as brine

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ARTICLE INFO

Article history:

Received 22 September 2017

Received in revised form

10 January 2018

Accepted 15 February 2018

Available online 16 February 2018

Keywords:

Fruit juice

Concentration

Membrane

Stripping solution

ABSTRACT

Potassium pyrophosphate was applied as stripping solution in osmotic distillation of fruit juices. The permeate ranged between 0.56 and 1.45 kg/m²h, being more sensitive to the variation of the feed temperature. After pre-saturating the experimental apparatus, no significant effect on the degradation of ascorbic acid was found and the losses of volatile compounds were no larger than 62% while 100% of the aroma compounds were lost during thermal concentration. In addition, orange juice was concentrated from 10 to 27 °Brix and the process did not change the physicochemical characteristics of the final juice. In conclusion, potassium pyrophosphate can advantageously substitute calcium chloride as the stripping solution of osmotic distillation in the concentration of fruit juices, allowing brine reconcentration using direct contact evaporation with flue gas.

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1. Introduction

In liquid foodstuffs, for example fruit juices, the water is the major component. The reduction of water content by concentration brings about several advantages in the production and commercialization of these products (Thanedgunbaworn et al., 2007). The goal of any concentration process is to manufacture a concentrated food product unchanged in quality and nutrition value that can be reconstituted just by adding water. There are many methods to remove water from the liquid foodstuff, including those that promote a phase change to vapor, like ohmic heating (Sabanci and Icier, 2017) and thermal evaporation (Cyklis, 2017), or ice, like freeze concentration (Sánchez et al., 2009), and several membrane processes (Jiao et al., 2004; Bhattacharjee et al., 2017) including reverse osmosis (Wenten and Khoiruddin, 2016), osmotic distillation (Gryta, 2005), direct contact membrane distillation (Ashoor et al., 2016), vacuum membrane distillation (Abu-zeid et al., 2015) and forward osmosis (Sant'anna et al., 2012).

From a technological point of view, the thermal concentration is the most developed and economically favorable technique. However, the operating temperatures are high enough to promote chemical changes like Maillard reactions (Dincer et al., 2016; Perez-cacho and Rousef, 2008); Rattanathanalerk et al. 2005) and the loss

of volatile compounds (Lin et al., 2002; Perez-cacho and Rousef, 2008; Varming et al., 2004), causing undesirable changes in color and flavor.

Since they can be operated at room temperature and atmospheric pressure, osmotic evaporation (OE), direct-contact membrane distillation (DCMD) and osmotic distillation (OD) or osmotic membrane distillation (OMD) are membrane processes separation that are alternatives to concentrate solutions with high content of heat-sensitive and volatile compounds (Yilmaz and Bagci, 2018; Quis-jensen et al., 2016; Bahçeci et al., 2015). In these processes, the solution to be concentrated and the stripping solution are separated by a porous hydrophobic membrane that prevents the intrusion and the passage of the liquid into the pores, as long as an operational limit of transmembrane pressure is respected. However, vapor can be transported through the membrane pores due to the chemical potential difference between the solutions. The main difference between these processes concern in the driving force. In OE the driving force is created using an extracting solution on the permeate side, in DCMD by the temperature difference across the membrane and in OD by adding the effect of temperature and extraction solution (Gryta, 2005). Since the permeate flux reported for OD are higher than for MD and OE, OD is more attractive and recommended to concentrate vegetable juices (Bahçeci et al., 2015; Onsekizoglu, 2013; Bélafi-bakó and Koroknai, 2006).

The osmotic distillation has been studied to concentrate a variety of fruit juices, like orange juice (Alves and Coelho, 2006; Badu et al., 2006; Bahçeci et al., 2015, Cassano et al., 2003, Cissé

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et al., 2005; Destani et al., 2013; Galaverna et al., 2008), grape juice (Bailey et al., 2000; Cissé et al., 2011; Kujawski et al., 2013; Rektor et al., 2006; Thanedgunbaworn et al., 2007), apple juice (Bélafi-Bakó Koroknai, 2006; Cissé et al., 2011; Onsekizoglu et al., 2010a, 2010b), pomegranate juice (Onsekizoglu, 2013), noni juice (Valdés et al., 2009), kiwi juice (Cassano and Drioli, 2007), lime juice (Nagaraj et al., 2006), melon juice (Vaillant et al., 2005), camu-camu juice (Rodrigues et al., 2004; Souza et al., 2013), passion fruit juice (Vaillant et al., 2001), cranberry juice (Zambra et al., 2015) among others.

Solutions of sodium chloride and calcium chloride are the main brine solutions used in osmotic distillation. The first one has the advantage of being cheaper, while the other results in brine solution with higher osmotic pressure and, consequently, higher permeate flux (Ongaratto et al., 2015a). Other osmotic solution, but less usual, are propylene glycol, glycerol and glycerol/sodium chloride (Celere and Gostoli, 2004), magnesium chloride (Gostoli, 1999) and glucose and sucrose solutions (Petrotos et al., 1998).

The osmotic distillation process produces large amounts of a dilute brine solution that must be concentrated to be recycled (Rosa and Giroux, 2001). An economically effective solution is to thermally concentrate it with energy integration, using a direct contact evaporation operating with flue gas (Ongaratto et al., 2015a). However, this precludes the usage of calcium chloride as the osmotic agent due to formation of calcium carbonate due to the presence of carbon dioxide in combustion gases. Potassium pyrophosphate does not precipitate in these same conditions and it generates solutions with similar osmotic potential, being a good substitute for calcium chloride (Michaels and Johnson, 1998; Ongaratto et al., 2015a). Furthermore, the aroma losses can be reduced when potassium pyrophosphate is used as brine. This salt is more soluble than those most used in osmotic distillation (sodium and calcium chloride). Since the activity of any volatile compound decreases when the salt concentration increases in brine, the volatile compound permeation through the membrane decreases too (Alves and Coelho, 2006). Considering that the use of potassium pyrophosphate as draw solution was only reported by Michaels and Johnson (1998) and Ongaratto et al. (2015a), there is very little available information of its usage as brine solution in the osmotic distillation of fruit juices.

This work aims at studying the effect of some operating conditions, such as feed and brine solution velocities and temperature, on the permeate flux and loss of volatile compounds using potassium pyrophosphate as stripping solution during the osmotic distillation of a surrogate solution. In addition, experiments were also carried out using orange juice as feed.

2. Materials and methods

2.1. Experimental setup

The experimental setup was explained in details in a previous work (Ongaratto et al., 2015a) and it is shown in Fig. 1. It simply consisted of a bench-scale membrane module through it the brine and feed solutions are circulated in the shell and tube sides, respectively, at controlled flow rates from two agitated tanks of 1.4 L maintained at chosen temperatures. The membrane module (model MD 020 CP 2N, Microdyn, Germany) had 21 mm of inner diameter and equivalent hydraulic diameter of 0.96 mm and it consists of 40 polypropylene capillaries, each one with 2.8 and 1.8 mm of outer and inner diameters, respectively. Its effective membrane internal area was 0.1 m², having an average pore size of 0.2 μm. The water extracted was continuously measured by weighting the overflow of the brine solution from its tank by a drainer, which allows the determination of the permeate flux by

Eq (1):

$$J = \frac{m}{A \cdot t} \quad (1)$$

where m is the total mass of the drained solution (kg), A is the total area of the membrane (m²) and t is the elapsed time (h).

2.2. Evaluation of operating conditions

The temperature and the flow velocity of feed and brine solutions were varied to find the highest attainable permeate flux. Thus, 11 experiments were performed according to a 2⁴⁻¹ fractional factorial design and the experimental data were processed using Statistica 7.0 software (StatSoft Tulsa, USA). The factor levels are shown in Table 1. The flow rates of the brine solution were lower because of the high viscosity of the solution and the maximum pressure of 0.5 kgf/cm², which did not allow liquid intrusion in the membrane pores.

A potassium pyrophosphate solution with initial concentration of 1291 g/L was used to concentrate a sucrose solution (160 g/L), which is a good surrogate for fruit juice in this membrane process (Ali et al., 2003; Courel et al., 2000). All the experiments were performed for a period of 3 h, so the draw solution was not reconcentrated. It was just replaced after each experimental run.

After each trial, the membrane module was cleaned by a three step cleaning process. The first step consisted in washing the module with deionized water until reaching an electrical conductivity of the wastewater between 5 and 10 μS/cm. In the next step the membrane module was dried with dry compressed air (relative humidity of the dew point at 0 °C). At last, a permeability test to nitrogen at 0.1 kgf/cm² was carried out to verify the drying efficiency, since the permeated gas flux needed to be equal to that found in the previous trial.

2.3. Volatile compounds loss and ascorbic acid stability

In order to evaluate the losses of volatile compounds and the ascorbic acid stability during the concentration process, a surrogate solution was applied. It was comprised of 30 μL/L of ethyl acetate (Vetec, Brazil), 70 μL/L of buthyl acetate (Vetec, Brazil), 30 μL/L of hexanal (Sigma-Aldrich, Germany), 80 μL/L of linalool (Sigma Aldrich, Germany), 260 μL/L of limonene (Sigma Aldrich, Germany), 10 μL/L of mircene (Sigma Aldrich, Germany), 300 mg/L of ascorbic acid (Sigma Aldrich, Germany) and 160 g/L of sucrose (Vetec, Brazil), all of them with purity higher than 98%, dissolved in distilled water.

At this step, it was employed a brine solution of either sodium chloride (Vetec, Brazil) or potassium pyrophosphate (Plury Química, Brazil). The initial concentrations of the brine solutions were 316 g/L and 1291 g/L, respectively, which correspond to the saturation concentration of each salt. The brine and surrogate solutions flow rates were 44.3 and 45.1 L/h, respectively, corresponding to a mean velocity of 12.3 cm/s. However, the potassium pyrophosphate solution had to be circulated at 23.4 L/h, corresponding to a mean velocity of 6.5 cm/s, in order to avoid reaching the maximum allowed pressure of 0.5 kgf/cm². All the experiments were performed for a period of 4 h at the temperature of 25 ± 1 °C and the draw solution was not reconcentrated during the experimental run.

In order to compare the losses of volatile compounds during the concentration by osmotic distillation with the thermal concentration process, it was used a rotary evaporator (model 801, Fisaton, Brazil) at 65 °C and 190 mmHg to concentrate the surrogate solution.

The effect of pre-saturation of the installation was also

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