



## Evaluation of the quality of coffee extracts concentrated by osmotic evaporation

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### ABSTRACT

Concentration of commercialized beverages is of the utmost importance for the reduction of transportation, storage, packaging and conservation costs. Osmotic evaporation (OE) is a process widely used in beverage concentration avoiding degradation of color and aromas associated with the use of higher temperatures. In this work, the concentration of aqueous coffee extracts was carried out by OE in a hollow fiber membrane contactor. The coffee samples obtained with this process were analyzed over time in terms of their antioxidant activity (using the DPPH and FRAP methods) and the content of antioxidants compounds (by HPLC). The variation of coffee aroma characteristics and pH throughout the concentration process was also studied. The concentration of total soluble solids of the coffee extracts increased from 30.7 to 123.7 g/L in 4 h of operation, while maintaining both the antioxidant activity and the antioxidant compound concentration per mass of total solids. The pH analyses show that the OE process had no effect on the pH of the coffee solution. The effect of the storage temperature of the concentrated solution on the respective pH was also evaluated; pH decreased from 5.6 to 5.2 and 4.9 when stored at 4 °C and ambient temperature, respectively. Finally, the aroma profile of the initial and concentrated coffee solution was characterized by GC/MS. It was observed that during the OE process the loss of aroma components of the coffee solution was not significant. From the 13 components identified and analyzed, corresponding to 64–69% of the total peak area of the volatile composition, only two showed a significant loss during the concentration process.

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### 1. Introduction

The coffee tree is a tropical evergreen shrub of the genus *Coffea*. Two seeds are usually found in each ripe fruit, and the name of these seeds is coffee. The two most common species cultivated are *Coffea canephora* and *Coffea arabica*.

Nowadays, coffee has a great importance in the world economy. Is one of the most valuable commodities, and, for several years, the second most important in international trades for producing countries. Its cultivation, processing, trade and transport promote million jobs around the world. This product is extremely important for the economy and politics of many developing countries, and in some countries, represents about 50% of all earned from exports

(“ICO – International Coffee Organization, 2017 The Story Of Coffee”).

Coffee has a stronger antioxidant activity and is a rich source of phenolic compounds (Higdon and Frei, 2006). Reactive oxygen species - free radicals - are usually produced by metabolism and have the ability to remove electrons from other cellular compounds, which can cause oxidative injuries in several molecules (losing their cellular functions) (Cruzat et al., 2007). Antioxidants and phenolic compounds are substances that inhibit the spread and formation of these free radicals.

Researches indicate that moderate consumption of coffee, about 400 mg of caffeine, the equivalent of five cups of coffee, maximum, per day, is associated with a desirable psychological effect, increasing surveillance, concentration and performance capacities of their consumers (EFSA Panel on Dietetic Products Nutrition and Allergies (NDA), 2011). Furthermore, recent researches indicate a beneficial effect of coffee in certain diseases, such as: diabetes,

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asthma, alcoholic cirrhosis, Alzheimer's and Parkinson's diseases (Riera et al., 2000).

The concentration of a product is aimed to reduce costs of transportation, storage, packaging and conservation (Alves and Coelho, 2007). In addition, this process helps in solving the problem of seasonal crops, allowing economic use of agricultural products (Riera et al., 2000).

For coffee extract concentration, two technologies can be used for this purpose: evaporation and freeze concentration. In the evaporation technique, the quality of the concentrated product may be deteriorated due to heat applied. Therefore, vacuum can be applied in order to reduce the boiling temperature and improve the product quality. However, the associated energy costs make this method very expensive (Gunathilake et al., 2014). Freeze concentration is considered the best method in terms of preservation of the original characteristics of the liquid food, but it is a complex process with high capital costs.

Membrane processes can be used for concentration, in particular, reverse osmosis (RO), membrane distillation (MD) and osmotic evaporation (OE). RO is an attractive option, since it operates at room temperature, causing minimal thermal damage to the product and consuming less energy than the methods previously referred. However, the maximum concentration possible to achieve is around 30% w/w with some fruit juices due to the limitation of applicable pressure. In order to increase the extract concentration, osmotic evaporation and membrane distillation have been proposed. Osmotic evaporation uses an osmotic solution for creating an osmotic pressure gradient across a porous hydrophobic membrane, so that water is removed from the more concentrated solution, migrating to the less concentrated. In membrane distillation both solutions are at different temperatures, increasing the difference of water vapor pressure in the two solutions and promoting the passage of water vapor through the membrane pores. OE has some advantages when compared to the other membrane processes: it operates at ambient temperature and pressure conditions, and does not suffer from osmotic pressure limitations, thus being possible to obtain concentration levels similar to the ones obtained by the conventional evaporation process. When compared to membrane distillation, OE operates at lower temperatures, avoiding the degradation of heat sensitive compounds during concentration. This process has been widely used for the concentration of beverages such as juices (cranberry, orange, camu-camu, apple, cherry) and sage and tea extracts (Belafi-Bako and Boor, 2011; Marques et al., 2017; Sorour et al., 2013; Souza et al., 2013; Torun et al., 2014; Zambra et al., 2014; Zhao et al., 2014).

The aroma and pH have great importance on coffee's quality and acceptance. It is known that the roasting and the preparation of grains process are important in characterizing the coffee flavor and aroma. However, despite the many studies conducted, the flavor characteristics of the coffee are not fully understood yet (Akiyama et al., 2007).

This work presents a study on the concentration of aqueous coffee solutions by osmotic evaporation, evaluating the antioxidant activity and the phenolic compounds content of the concentrated solution with time. Furthermore, the content on volatile organic compounds (VOCs) compounds and pH of the coffee solution in the beginning and end of the process were also determined to assess if any loss of aromas were observed during the concentration process.

## 2. Materials and methods

### 2.1. Coffee and osmotic solutions

Coffee used was roasted and grounded by NovaDelta – Comércio e Indústria de Cafés, S.A. (Campo Maior, Portugal). A

hundred and ten grams of coffee powder were diluted in one liter of boiling water. The final solution was then vacuum filtered with paper filter to remove solids in suspension. Calcium chloride (99%, Panreac) 5 M solution was prepared with deionized water, using an initial volume of four liters of this solution.

### 2.2. Osmotic evaporation

The experimental setup for the osmotic evaporation is shown in Fig. 1. The membrane module consists of a hollow fiber membrane contactor with a surface area of 1.4 m<sup>2</sup>. The characteristics of the contactor and fibers (polypropylene microporous Celgard X50-215) are given in Table 1.

The contactor was placed horizontally so that the deposition of solids at the end of the membrane was minimized, since there is no action of gravity. The coffee solution was pumped through the shell side and the calcium chloride in the fibers. The flow rate of the solutions was in counter-current mode and equal to  $3.5 \times 10^{-6}$  and  $7.5 \times 10^{-6}$  m<sup>3</sup>/s, for the fibers and the shell side, respectively. The concentration process occurred at a temperature of  $26 \text{ }^\circ\text{C} \pm 2 \text{ }^\circ\text{C}$  and atmospheric pressure. The temperatures of both phases were monitored over time.

Two initial and final samples of each experiment were stored, one at 4 °C and the other at room temperature for the evaluation of the pH over time. In addition, coffee samples were taken over time for analysis of their antioxidant activity, phenolic compound content and VOCs characterization.

In order to calculate the water flux, a scale was placed under the coffee reservoir to measure water loss. The permeate flux was calculated according to equation (1):

$$J_w = \frac{\Delta V}{A_s \cdot \Delta t} \quad (1)$$

where  $\Delta V$  is the water volume variation,  $A_s$  is the surface area and  $\Delta t$  the time variation.

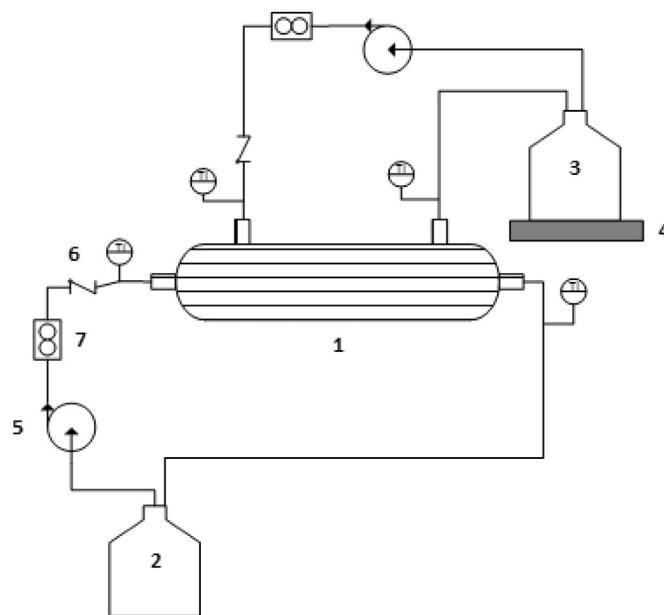


Fig. 1. Experimental setup: (1) membrane contactor; (2) reservoir of dihydrate calcium chloride solution; (3) coffee solution reservoir; (4) balance; (5) pump; (6) pressure valve; (7) flow meter.

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