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Bottled water production using the condensed water from a concentrated orange juice plant

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ABSTRACT

Evaporated water is produced during the juice concentration process by separation of the condensed water in the evaporator. This evaporated water is fully, but poorly used, for example, washing fruits. Considering it as a fraction of the fruit itself, this study proposed its use as "fruit water" bottled for human consumption. Evaporated water samples were characterized according to the following parameters established by the Brazilian technical regulations for bottled water: inorganic, organic substances, pesticides, microorganisms, and physical properties. The results show that the only parameters that exceed the maximum permitted levels were apparent color and turbidity. Then, tests were conducted in laboratory and in pilot scale for evaluation of technology of the membrane separation process (MSP) for the purpose of reducing the apparent color of the evaporated water of citric juice to less than or equal to 5 units of Pt/Co and simultaneously the turbidity to less than or equal to 1 NTU. The ultrafiltration in cellulose membrane of 30 kDa at 1 bar pressure was effective in reducing the apparent color and turbidity of the recovered water from concentrate orange juice, with values below the maximum allowed by law, demonstrating that this water meets Brazilian quality requirements for human consumption.

1. Introduction

The concentrate orange juice industries use advanced technology and are one of the successful examples of the full use of an agricultural product. In addition to the frozen concentrated orange juice, they produce various byproducts including: pulp-wash (secondary juice), D-limonene, citrus pulp bran (pet food), so that practically any part of the fruit is discarded and become into byproducts of commercial value.

In this context, the evaporated water can be considered an exception because it is the largest volume generated in the processes, representing about 40% of the fruit in the industry. According to Yamanaka [24], it is fully used by the industries in many ways, including fruit cleaning/washing, extraction/recovery of solids residual of the pulp, water replacement in the process of peel essential oil recovery, calories recovery for use in boilers, floor, equipment and restrooms cleaning.

Whereas the concentrated orange juice is about six times in terms of soluble solids it can be estimated that for each kilogram of frozen juice concentrate are produced, theoretically, about 4.9 kg of evaporated water.

Evaporated water is produced during the concentration process. According to Tocchini et al. [21], the juice is concentrated in vacuum evaporator, with up to 8 stages and 6 effects, enabling energy savings. Water in vapor form is separated from the orange juice and serves as a heater for stages working at lower temperatures (higher vacuum), where it condenses.

Few articles were found in the literature on the recovery of evaporated water originating from juice concentrates, with the objective of identifying a use for this industrial byproduct.

DeStefano [9] registered his invention as a method for water recovery from fruit. By the patented method, fruit juice is extracted, and then the product is concentrated by removing water as vapor. According to the author, the water is recovered from the condensation of water vapor and can be bottled and sold.

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The author states that the method can be applied to various fruits such as orange, grapefruit, apple, cherry, grape etc. Then, he reports a characterization example of the obtained product by analyzing a sample, but without informing the fruit from which the water was recovered.

Moussi et al. [15] describes a water recovery method from the concentrated juice of fruits and/or vegetables. The author reports drinking water recovery from concentrated grape juice, characterizing this material before and after the sequence of treatment operations. Results of some chemical and sensory analyzes that prove the potability of the product are presented.

The objective of the present study was the chemical, microbiological and physical properties characterization of evaporated water from concentrated orange juice, produced by an industrial plant located in the state of São Paulo, to prospect the possibility of its use for human consumption through industrialization. For this, it was adopted as reference the Brazilian bottled-water regulations [3,4].

The experimental tests were conducted in laboratory and pilot scale, to evaluate the performance of membrane separation process technology using microfiltration and ultrafiltration for reducing apparent color of evaporated water to value equal or less than 5 units of Pt/Co and simultaneously the turbidity to value equal or less than 1 NTU.

In the following step of the study, evaporated water was processed and bottled in glass bottles. The final product was evaluated by physical, chemical, microbiological, sensory analysis, and the economic viability for an industrial plant (1000 L/h production capacity) was also estimated. These results will be reported in a following article in the writing phase.

2. Methodology

2.1. Raw material

The evaporated water, provided by Louis Dreyfus Commodities, a unit located in Engenheiro Coelho – SP, originated from the process of obtaining frozen concentrate orange juice (*Citrus sinensis*).

Ten samples were collected from a single point of industry line previously defined, which gathers the evaporated water after the last evaporation effect, in ten different dates of juice production along the *Citrus sinensis* orange crop, varieties: "Pera Rio", "Natal" and "Charmute" with "Natal" (blend) between September and October 2010, as shown in Table 1.

Table 1
Varieties of *Citrus sinensis* oranges processed on each of the ten dates of samples collection of evaporated water, in 2010.

Sample	1	2	3	4	5	6	7	8	9	10
Data ofcollection Varieties	September, 1st "Pera Rio"	September, 15th "Natal"	September, 22th "Natal"	September, 27th "Natal"	September, 29th "Natal"	October 6th "Pera Rio"	October 14th "Natal"	October 19th "Charmute" and "Natal"	October 21st "Natal"	October 27th "Natal"

2.2. Raw material characterization

The raw material was characterized by some analysis provided by the technical regulations for bottled water and ice described by ANVISA resolutions No. 274 [3] and No. 275 [4] pertaining to raw material under study, and other determinations as follows:

2.2.1. Inorganic substances

Antimony, arsenic, barium, boron, cadmium, chromium, copper, cyanide, lead, manganese, mercury, nickel, and selenium were quantified on an emission spectrometer with power inductively coupled plasma (ICP OES model VISTA MPX, Varian), while nitrate and nitrite by Standard Method 21,using the methodologies described by the American Public Health Association (APHA), in Eaton et al. [11].

2.2.2. Organic substances

1,1-Dichloroethene; 1,2,3-trichlorobenzene; 1,2,4-trichlorobenzene; 1,2-dichloroethane, benzene, vinyl chloride, dichloromethane, styrene, carbon tetrachloride, tetrachloroethene,

trichlorobenzenes, trichlorethylene were analyzed using methodologies described by EPA 524.2 [12]; acrylamide was analyzed using the methodology described by EPA 8032 [12]A, while the benzopyrene was analyzed using the methodology described by EPA 525.2 [12].

2.2.3. Pesticides

2.4-D, Alachlor, atrazine, bentazone, hexachlorobenzene, metolachlor, molinate, pendimethalin, pentachlorophenol, permethrin, propanil, simazine and trifluralin were analyzed using methods described by EPA 525.2 [12]; the substances aldrin and dieldrin, chlordane, DDT (isomers), Endosulfan, endrin, heptachlor and heptachlor epoxide, lindane (g BHC) methoxychlor were analyzed using methods described by EPA 508.1 [12], while the glyphosate analysis used the methodology described by EPA 547 [12].

2.2.4. Microorganisms

Total coliforms in 100 mL; *Escherichia coli,* in 100 mL; enterococci in 100 mL; total count of aerobic mesophilic; count of yeasts and molds; *Alicyclobacillus* and *Alicyclobacillus acidoterrestris* were analyzed using methods described by APHA, in Eaton et al. [11] and Downes and Ito [10].

2.2.5. Physical properties

Analyses of: pH (pH meter Digimed DM20); apparent color (colorimeter HACH DR 2010); turbidity (turbidimeter HACH 2100 NA); electrical conductivity (conductivimeter Digimed DM31) and hardness (titration with EDTA 0.01N) were conducted according to the methods described by APHA [11].

2.2.6. Volatile profile

The volatile substances were qualitatively analyzed according to the methodology provided by NBR 13058 [1], Winne and Dirinck [23] and Padula and Borghetti [16], using a gas chromatograph model Agilent Technologies HP 6890 coupled to a mass spectrometer detector model HP 5973 operating with a HP-5MS capillary column (5% phenyl methyl siloxane) (30 m × 0.25 mm × 2.5 μm).

2.3. Testing ultrafiltration to reduce apparent color and turbidity

2.3.1. Tests on laboratory scale

The experiments occurred between January and May 2011, coinciding with orange off season. Thereby, it was used, from January to April, evaporated water of lime juice, for its similarity with the evaporated water of orange juice with respect to parameters of apparent color and turbidity. Thus, there was a rationalization of the study time, and it allowed to continue the research in the following orange harvest. The evaporated water during the concentration of lime juice was collected at the same point of orange evaporated water.

In preliminary tests (data not shown), the performance was evaluated with respect to apparent color and turbidity of two microfiltration membranes (MF): polyethersulfone, 0.05 μm (PES 0.05) and polyvinylidene fluoride, 0.2 μm (PVDF 0.2); five ultrafiltration membranes (UF): polyethersulfone, 30 kDa (PES 30), 50 kDa (PES 50) and 100 kDa (PES 100), cellulose, 30 kDa (CEL 30) and 100 kDa (CEL 100), testing pressures between 0.5 and 2.0 bar. All tests were conducted at 60 °C, which is the average temperature of the water flow of the industrial evaporators' condenser. PES and CEL 30 membranes were selected to carry on with the study.

In the laboratory unit, six tests were conducted with polyethersulfone membrane 0.05 μm (PES) under pressure at 0.5 bar and in triplicate, for the cellulose membrane of 30 kDa (CEL 30), filtration pressure at 1 and 2 bar. All experiments regarding lime water had about 800 mL, temperature at 60 °C and rotation (magnetic stirrer) of 500 rpm.

After this, in the same laboratory unit, under the same conditions, it was tested for orange evaporated water the procedure of ultrafiltration membranes at 1 bar. At the permeate flow, it was placed a beaker with 1000 mL on the electronic balance (±0.01 g, Marte AS 2000) for obtaining the permeate mass as a function of time. The feeding was concentrated until the concentration factor (CF) reached 5.

2.3.2. Tests on pilot scale

From the results obtained in the laboratory unit, the experiments in Pilot Unit were conducted, using the CEL 30 membrane at pressure at 1 bar for both lime and orange evaporated water. All tests were carried out with feeding volumes of about 30 L at 60 °C and a flow rate of 0.86 m^3/h corresponding to 6 m/s speed.

The pilot plant consists of a stainless steel tank jacketed and insulated (triple wall) with a 30 L capacity, a centrifugal pump (brand ALFA LAVAL, model ALC 1/140 S 4.0 kW), a flow magnetic meter (0–6 m^3/h) (Conault), two manometers (0–9.8 bar), one was positioned at the entrance and the other at the exit of the membrane, a butterfly valve at the exit of the tank and the other below the module with the membrane, and a needle valve in the exit of the membrane. The membrane module consists of stainless steel AISI 304 with 7.5 cm wide and 52 cm long, with a cross-sectional area of 40 mm × 1 mm and permeation area calculated considering the dimensions: 400 × 40 mm (0016 m^2). Pipes, valves and bends with 1″ diameter are sanitary works. In this equipment, the adjustment of the operating conditions (pressure and flow) is done by simultaneously pump rotation control through a frequency inverter and needle valve, while the temperature is controlled by water circulation at the appropriate temperature to the process by the jacketed tank.

A container with a 20-L capacity was placed on an electronic balance (±5 g, Marte, model LC 20) at the permeate exit to collect and record the permeate mass in function of time. Permeate mass data were collected to predetermined time intervals up to CF equal to 5, in all experiments.

To calculate the flow, it was used flow area (F_a) or cross-sectional area equal to 40 × 1 mm and tangential velocity at 6 m/s, according to the following equation:

$$V = v \times F_a,$$

where V = flow rate (m^3/h); v = tangential velocity (m/s) and F_a = cross flow area (m^2).

2.3.3. Evaluation of ultrafiltration processes

For each test in laboratory and pilot scale, supply, permeate and retentate samples were collected and analyzes performed in triplicate, apparent color and turbidity using colorimeter HACH turbidimeter HACH DR 2010 and 2100 NA, respectively.

2.4. Result analysis

The results were statistically analyzed by using ANOVA; Tukey test was used to compare treatment means, setting $p < 0.05$ as significance level. The means and standard deviations were calculated from tests performed in triplicate for physical properties, inorganic, organic substances and pesticides. The data were then compared using the software GENES – quantitative genetics and experimental statistics – VS 2009.7.0 [7].

The results obtained of the tests of apparent color and turbidity were also evaluated by outlier test, for the application of Q test, described by Brendolan [5].

3. Results and discussion

3.1. Physical properties

The orange varieties studied ranged between dates of collection. The mixture of different varieties of oranges throughout the production period is a common practice in industrial plants, and aims to produce juice concentrates that meet the specifications defined by each contract in terms of relationship between Brix/total titratable acidity (ratio) and product color. Thus, the study involved samples representing the routine production of an industrial plant, which in turn, represented the dynamic performance of this sector.

The physical properties values of ten samples of evaporated water are shown in Table 2, while the result of microbiological analyzes and inorganic substances can be seen in Tables 3 and 4, respectively.

The pH variation was significant between the 10 samples, as shown in Table 2. These results are expected, in function on the naturally observed variation in fruit throughout the harvest period and also for different varieties and growing regions.

Although significant differences were observed in the hardness, the values obtained were low and are in agreement with the analysis results of inorganic substances (Table 4), where low levels of salts that affect the hardness was observed.

The water electrical conductivity presented variation, however, this is not a limiting factor for the quality of drinking water standards according to RDC No. 274 [3].

The parameters of apparent color and turbidity are above standard for bottled water according to the same RDC No. 274. These values varied significantly from sample to sample, reaching a peak of 364 units of Pt/Co and 39.37 NTU. The analysis of these parameters of the sixth sample by the *Q* test identified this sample as an outlier, that is, as not belonging to the average population. This peak can be explained by the low efficiency of the aroma recovery on the sixth sample collection date, on October 6th, which contributed to the retention of aromatic compounds in the evaporated water.

There were no references for comparison of the results of apparent color and turbidity. These may be associated with the presence of essential oils, aromatic compounds and occasionally to the level of nitrite found. DeStefano [9] reports turbidity values of 0.1 units and 0 units APHA to color of only one sample, without describing the fruit from which the water was recovered.

3.2. Microbiological analyses

RDC 275 provides, among other microorganisms, determination of total coliforms and enterococci, as these are indicators of hygienic conditions in general, not necessarily of fecal origin. The total coliform group includes about 20 species, among which are both bacteria from the gastrointestinal tract of humans and other warm-blooded animals, as well as many genera and species of non-enteric bacteria, such as *Serratia* and *Aeromonas*, for example. For this reason, their enumeration in food and water is less representative as an indication of fecal contamination than the enumeration of *E. coli* [20].

The total coliforms presence was detected in two samples and enterococci in one sample, but the absence of *Escherichia coli* indicates that these results are not representative of fecal origin contamination. These microorganisms are easily eliminated in the usual stages of water treatment.

The determination of *Alicyclobacillus acidoterrestris* was performed to verify the occurrence of spores of this bacterium in evaporated water because it is a source of concern for the environment in the industrial production sector of concentrated citric juice, due to its potential to drop the product quality. Although the presence of this bacterium has been detected in three samples, the

Table 2
Physical properties of evaporated water in the ten sampling dates.

Sample	1	2	3	4	5	6	7	8	9	10	MSD
pH	4.15 ± 0.02a	4.00 ± 0.14a	4.12 ± 0.03a	3.75 ± 0.00b	3.65 ± 0.02b	4.04 ± 0.08a	3.98 ± 0.05a	4.08 ± 0.01a	3.44 ± 0.10c	4.02 ± 0.01a	0.19
Conductivity (mS/cm)	47.23 ± 0.15de	62.00 ± 7.55b	43.67 ± 0.32e	57.67 ± 1.11bc	95.00 ± 0.43a	28.47 ± 0.58f	52.17 ± 0.42cd	30.07 ± 0.15f	53.50 ± 0.10cd	47.20 ± 0.30de	7.12
Color (un. Pt/Co)	78.67 ± 3.21b	81.00 ± 3.00b	23.33 ± 4.72d	8.00 ± 1.00e	24.67 ± 0.58d	364.00 ± 1.73a	40.67 ± 4.04c	39.00 ± 1.00c	28.00 ± 1.00d	24.33 ± 2.08d	7.67
Turbidity (NTU)	11.80 ± 0.30b	11.40 ± 0.35b	4.12 ± 0.03fg	1.94 ± 0.05h	3.50 ± 0.00g	39.37 ± 0.61a	6.06 ± 0.14c	5.71 ± 0.01cd	4.67 ± 0.04ef	5.01 ± 0.02de	0.72
Hardness(mg $CaCO_3$/L)	3.24 ± 1.01a	2.35 ± 0.51ab	2.35 ± 1.02ab	1.18 ± 0.51b	2.65 ± 0.88ab	1.18 ± 0.51b	1.47 ± 0.51ab	1.47 ± 0.51ab	1.77 ± 0.00ab	1.47 ± 0.51ab	1.94

Samples (mean ± standard deviation) followed by the same letter in the same row do not differ significantly at 5%. MSD = minimum significant difference.

Table 3
Microbiological determination of evaporated water in ten sampling dates.

Sample	1	2	3	4	5	6	7	8	9	10
Total count (CFU/mL)[a]	<1	10	<1	<1	10	<1	<1	10	<1	<1
Yeast and molds (CFU/mL)	<10	<10	<10	<10	<10	<10	<10	<10	<10	<10
Alicyclobacillus (CFU/mL)	<10	<10	<10	<10	<10	<10	<10	20	<10	<10
A. acidoterrestris (in 10 mL)[b]	A	P	A	A	P	A	A	P	A	A
Total coliforms (CFU/mL)	<1	<1	<1	<1	<1	<1	<1	2	1	<1
Escherichia coli (in 100 mL)[b]	A	A	A	A	A	A	A	A	A	A
Enterococos (CFU/100 mL)	<1	<1	<1	<1	<1	<1	<1	<1	<1	54

[a] Colony forming units.
[b] A = Absent; P = present.

Alicyclobacillus sp. was observed in quantitative level in only one in ten samples, with low number of 20 CFU/mL (colony forming units). Since evaporated water has features near to distilled water, with a shortage of nutrients, it is unlikely that spores are able to develop and multiply, becoming a source of spread of this bacteria by the industrial environment. Thus, its presence in the evaporated water should not be considered as a critical factor for consumption, since it is not pathogenic bacterium [25].

3.3. Inorganic substances analyses

The best known function of selenium is to present antioxidant property, carried out by the association of this element with the enzyme glutathione peroxidase [6,14,17,19]. In the late 90s, it was found that selenium is a constituent of 5′-iodinase, an enzyme active in the metabolism of the thyroid hormones, and that syndromes of iodine deficiency are more severe when there is simultaneous selenium deficiency [22].

The third sample showed selenium levels above 0.01 mg/L, the maximum value allowed by RDC No. 274. Numerous surveys show that the selenium concentration in food can have wide variation, depending on the contents in the soil. According to the study presented by Ferreira et al. [13], the presence of selenium in certain formulations of fertilizers, animal food and the content in the soil may be the explanation for the wide variation of this component between samples of the same type of food.

The nitrite level was above the allowed level (0.02 mg/L) by RDC No. 274 [3] in 3 samples: 2, 3 and 7, but less than the recommended level by the ordinance 2.914 [2]. Rezende [18] related water contamination by nitrite with runoff of chemically fertilized land, sewage and erosion of natural deposits.

There is no maximum limit according to RDC No. 274 for the presence of iron in bottled water for human consumption. All other substances showed levels below the maximum allowed in 10 samples of evaporated water.

Analyses of all organic substances and pesticides resulted in no detection or values below quantifiable limit, through the used methods.

The results of the volatile profile characterization of evaporated water are shown in Table 5. The principal volatiles detected were limonene (RT = 8.30 min), ethyl 3 hydroxy hexanoate (RT = 9.88), valencene (RT = 14.38 min) and nootkatone (RT = 16.17 min). All volatiles tentatively identified are naturally present in the orange or in its shell.

Samples 2–-4, 5, 7, 9, and 10 were obtained from the evaporated water from the variety of “Natal” juice while the samples 1 and 6 were obtained from “Pera Rio”, as shown in Table 1. The sample 8 was obtained from a mixture of “Charmute” and “Natal” varieties. Although they had different varieties, the major tentatively volatiles identified in the samples were the same.

The sample 6, collected in October 6th, showed in addition to volatile described in Table 5, other components tentatively iden-

Table 4
Inorganic substances, in μg/L, in ten sampling dates.

Substance	Sample										
	1	2	3	4	5	6	7	8	9	10	MSD
Calcium	nd < 3e	230 ± 10a	120 ± 10b	nd < 5e	63 ± 3c	nd < 0.01e	nd < 0.01e	21 ± 5 d	nd < 3e	nd < 3e	14.1
Aluminium	nd < 3d	nd < 3d	8 ± 2b	nd < 5d	12 ± 0a	nd < 5d	nd < 5d	nd < 5d	nd < 5d	6 ± 0c	1.8
Antimony	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Arsenic	nd < 5a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Mercury	nd < 0.25a	nd < 0.3a	nd < 0.3a	nd < 0.3a	nd < 0.3a	nd < 0.3a	nd < 0.3a	nd < 0.3a	nd < 0.3a	nd < 0.3a	2.9
Boron	nd < 5a	nd < 5a	nd < 5a	nd < 5a	nd < 5a	nd < 5a	nd < 5a	nd < 5a	nd < 5a	nd < 5a	3.0
Copper	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Iron	6 ± 2b	16 ± 1a	5 ± 0b	nd < 3c	15 ± 4a	4 ± 0bc	4 ± 0bc	3 ± 0bc	nd < 3c	nd < 3c	4.2
Phosphorus	305 ± 3b	353 ± 1a	87 ± 11e	23 ± 1f	131 ± 2 d	17 ± 2f	105 ± 2e	156 ± 14c	139 ± 8cd	132 ± 2d	18.7
Potassium	2.160 ± 80b	4.240 ± 180a	1.020 ± 20ef	149 ± 3g	1.520 ± 90cd	118 ± 1g	1.704 ± 207c	1.653 ± 32c	1.245 ± 52de	855 ± 2f	283.6
Sodium	305 ± 3a	14 ± 1c	nd < 5d	nd < 5d	34 ± 3b	nd < 3d	nd < 3d	nd < 3d	nd < 10d	nd < 3d	4.0
Magnesium	11 ± 0h	193 ± 2a	44 ± 1g	6 ± 1i	88 ± 1c	10 ± 0h	74 ± 1d	104 ± 2b	70 ± 1e	58 ± 1f	3.5
Barium	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Cadmium	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Selenium	8 ± 3 b	nd < 3c	15 ± 2a	9 ± 2b	nd < 3c	nd < 3c	nd < 3c	nd < 3c	nd < 3c	nd < 3c	3.8
Nickel	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Lead	nd < 3b	6 ± 2a	nd < 3b	nd < 3b	nd < 3b	nd < 3b	nd < 3b	nd < 3b	nd < 3b	nd < 3b	1.8
Manganese	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Chromium	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Zinc	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	nd < 3a	2.9
Cyanide	nd < 5d	8 ± 0c	19 ± 1b	9 ± 1bc	11 ± 1bc	nd < 5d	nd < 5d	nd < 5d	42 ± 3a	nd < 5d	3.2
Nitrate	890 ± 27.6b	1.330 ± 41.3a	890 ± 27.6b	440 ± 13.6c	440 ± 13.6c	nd < 440d	440 ± 13.6c	890 ± 27.6b	440 ± 13.6c	1.330 ± 41.3a	74.3
Nitrite	nd < 10f	33 ± 0.3a	30 ± 0.3b	nd < 7f	13 ± 0.1d	nd < 7f	27 ± 0.3c	12 ± 0.1e	nd < 7f	nd < 7f	0.5

Samples (mean ± standard deviation) followed by the same letter in the same row do not differ significantly at 5%, nd = not detected <quantitation limit. MSD = minimum significant difference.

Table 5
Volatile profile of evaporated water in ten sampling dates.

R.T.(min)	No. CAS	Tentatively volatile identified	Sample									
			1	2	3	4	5	6	7	8	9	10
1.46	64-17-5	Ethanol	X	X	X	X	X	X	X	X	X	X
3.01	513-86-0	3-hydroxy-2-butanone (acetoin)	ND	X	X	X	NI	NI	NI	NI	ND	
7.64	123-35-3	Beta myrcene	NI	ND	X	ND	ND	X	X	X	X	ND
8.30	138-86-3	Limonene	X	X	X	X	X	X	X	X	X	X
9.50	124-19-6	Nonanal	X	X	X	X	X	X	NI	X	ND	ND
9.88	2305-25-1	3-Hydroxy-ethyl hexanoic acid ester (ethyl-3-hydroxy hexanoate)	X	X	X	X	X	X	X	X	X	X
10.90	144-39-8	linalyl propanoate	X	X	X	X	X	NI	X	X	X	X
11.08	112-31-2	Decanal	X	X	NI	NI	NI	X	NI	NI	ND	ND
12.93		NI	ND	ND	ND	X	X	X	ND	ND	X	X
14.38	10219-75-7	1,2,3,5,6,7,8,8 a-octahydro-1, 8a-dimethyl-7- (1-methylethenyl)-[1R-(1alfa, 7beta, 8aalfa)] naphthalene (valencene)	X	X	X	X	X	X	X	X	X	X
14.55	483-76-1	1,2,3,5,6,8 a-hexahydro-4,7-dimethyl-1-(1-methylethyl)- (1S-*cis*)-naphthalene	X	X	X	X	X	X	X	X	X	NI
16.17	4674-50-4	4,4 a,6,7,8-hexahydro-4-4a-dimethyl6-(1-methylethenyl)-, [4alfa, 4aalfa, 6beta)] 2 (3H) naphthalenone (Nootkatone)	X	X	X	X	X	X	X	X	X	X
17.69	484-12-8	7-Methoxy-8-(3-methyl-2-butenyl) coumarin(Ostole)	NI	NI	X	X	ND	ND	ND	ND	NI	ND

ND = Not detected.
NI = Detected but identification not confirmed.

tified as: ethyl butanoate, α-pinene, -pinene, sabinene, β-pinene, octanal, 3-carene, α-terpinene, β-ocimene, γ-terpinene, 1-octanol, terpinolene, Linalool, 4-terpineol, α and β-citral, nerol acetate, caryophyllene, -selinene among others, which could not be identified under the analysis conditions. These results are in accordance with the industry production register where the samples were collected, which report low aroma recovery efficiency of that data collection.

3.4. Membranes performance

3.4.1. Microfiltration of lime recovered water

The six microfiltration tests of lime recovered water in membrane PES at pressure at 0.5 bar are shown in Fig. 1. The six flow curves obtained for the membranes PES at 0.05–0.5 bar are presented different, but are similar to the typical curves of micro and ultrafiltration, although they have not reached the steady flow stage due to process interruption. Even with all the constant operating parameters there was no good repeatability. This can be attributed to the raw material used, which showed different values of apparent color and turbidity. For all runs, there were high flow values pointing to an attractive scenario for the application of this process, even for the minimum value of 700 kg/h m^2 for laboratory unit.

The results obtained in the evaluation of apparent color and turbidity of the feeding, permeate and retentate samples in each of the six tests are shown in Table 6.

By observing the apparent color and turbidity parameters of the raw material (feeding), there is a range of variation for apparent color between 15, 27 and 42 and for turbidity, between 1.26, 2.7 and 6.1. This demonstrates the existence of three groups of similar raw materials for these experiments. From the point of view regarding the final product, the permeate met the desired specifications in five different runs, except run 6, as can be seen in Table 6. But the difference marked between these three groups contributes to explain the different curves with marked permeate flow values different between them. There is an approximate correlation between runs 1 and 2; 3 and 6; 4 and 5 with the permeation curve values and apparent color for feeding. The data between runs 3 and 6, despite the apparent color and turbidity of the feeding are quite different, the run 6, with much higher permeation curves, the results were similar. Thereby combinations 1 and 2 and 4 and 5 maintain similar values of apparent color and turbidity and flow results with similarities between the curves.

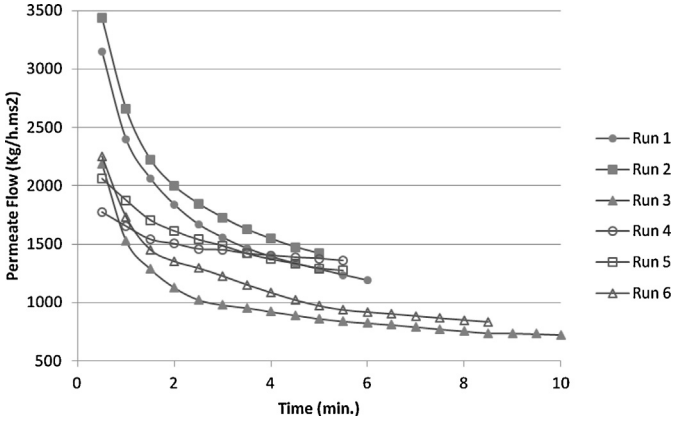

Fig. 1. Permeate flow obtained in 6 tests of recovered water microfiltration of concentrated lime juice into membrane PES 0.05 μm at 1 bar.

Table 6
Analyzes results of turbidity and apparent color in feeding, permeate and retentate samples collected on tests for microfiltration of concentrated lime juice recovered water in membrane PES 0.05 μm at 1 bar.

Test	Sample	Turbidity (NTU)	Apparent color (unit Pt/Co)
Run 1	Feeding	2.73 ± 0.05b	15.00 ± 0.00b
	Permeate	0.21 ± 0.00bc	2.33 ± 0.58b
	Retentate	4.38 ± 0.07d	25.00 ± 0.00d
Run 2	Feeding	2.73 ± 0.05b	15.00 ± 0.00b
	Permeate	0.25 ± 0.02b	2.33 ± 0.58b
	Retentate	7.72 ± 0.03c	43.00 ± 0.00c
Run 3	Feeding	2.75 ± 0.02b	12.00 ± 0.00c
	Permeate	0.21 ± 0.01bc	2.00 ± 0.58b
	Retentate	10.43 ± 0.07b	54.33 ± 0.58b
Run 4	Feeding	1.26 ± 0.02c	7.33 ± 0.58d
	Permeate	0.17 ± 0.01c	1.00 ± 0.00bc
	Retentate	3.60 ± 0.06e	22.67 ± 0.58e
Run 5	Feeding	1.26 ± 0.02c	7.33 ± 0.58d
	Permeate	0.17 ± 0.01c	0.00 ± 0.00c
	Retentate	3.40 ± 0.05f	22.00 ± 0.00e
Run 6	Feeding	6.08 ± 0.06a	42.33 ± 1.53a
	Permeate	0.47 ± 0.06a	5.33 ± 1.15a
	Retentate	21.83 ± 0.06a	151.00 ± 1.00a
MSD	Feeding	0.11	2.01
	Permeate	0.08	1.77
	Retentate	0.16	1.5

Samples (mean ± standard deviation) followed by the same letter in the same row (feeding; permeated; retentate) do not differ significantly at 5%. MSD = minimum significant difference.

3.4.2. Ultrafiltration of lime recovered water

The ultrafiltration experiments in triplicate (A–C) of lime evaporated water at pressure at 1 bar and 2 bar conducted on a laboratory scale, using cellulose membrane 30 kDa (CEL 30) are shown in Fig. 2.

It can be observed by Fig. 2 the permeate flows in membrane CEL 30 at pressure at 1 bar did not show typical exponential curve. The flow stabilization occurred in the range 800–900 kg/h m^2, and these values were considered high for laboratory scale experiments. Furthermore, permeate levels of apparent color and turbidity of the permeate were achieved in all tests at laboratory scale, with adequate margin of safety, as can be seen in Table 7.

Fig. 2 also allows the conclusion that the increased pressure in the membrane CEL 30 from 1 to 2 bar nearly doubles the permeate flow, thereby reducing the time to achieve the established concentration factor. The increased pressure did not compromise the results regarding permeate apparent color and turbidity, producing satisfactory results as can be seen in Table 7. In UF process, polarized layer formation prevents, after a certain value, that the increase in pressure corresponds to an increase in flow, due to its greater compression, resulting in higher transport resistance of solvent (Habert et al., 2006) [27]. Furthermore, the physical tests results support the conclusion that there was no further gain reduction in apparent color and turbidity when the operating pressure was increased from 1 to 2 bar. Thus, the use of lower pressure, at 1 bar, was selected for further investigation, for demonstrating better stability of flow over time; the sharpest drop in flow in the experiment at a pressure at 2 bar would lead to more frequent interruptions to cleaning during industrial processes.

The results obtained in the evaluation of apparent color and turbidity of the samples of feeding, permeate and retentate in each of the tests are shown in Table 7.

In this ultrafiltration process, the raw material had turbidity values of 2.75 and 4.09, whereas the apparent color, 12.00 and 27.67. The obtained permeates resulted in similar values of both apparent color and turbidity, in all runs and met the Brazilian regulations regarding these quality parameters. In the ultrafiltration, the pore diameter is smaller compared to the microfiltration and even though the raw material presents variation, as in this case, the formation of polarized layer acts as a true and effective filter, on the membrane surface, resulting similar permeate in relation to apparent color and turbidity and with flow curves whose behavior demonstrates good repeatability.

Tests using lime recovered water membrane in CEL 30 were, then, reproduced with orange evaporated water.

3.4.3. Ultrafiltration of orange recovered water

The flow curves and the results of apparent color and turbidity for tests with recovered water from concentrate orange juice, in duplicate, runs 1 and 2, using membrane CEL 30 at pressure at 1 bar, on a laboratory scale are shown in Fig. 3 and Table 8, respectively.

There are, in this case, flows in the order 800–900 kg/hm^2 whose stabilization happened almost from start. These values are lower than those obtained with recovered water of lime juice, which in general for other membranes reached higher values, despite continued decline over time. This result is very attractive and interesting by stabilizing the flow that allows, at the beginning, greater confidence in the performance of the membrane. It should be noted that this experiment has a short time, of only 5 min, and in a pilot or industrial process this behavior can be different.

It can be seen that the raw material has now apparent color and turbidity very close and that both permeation curves are quite

Table 7
Analyzes results of turbidity and apparent color in samples of feeding, permeate and retentate collected on tests for ultrafiltration of recovered water of concentrated lime juice in CEL 30 membrane at pressures at 1 and 2 bar.

Test	Sample	Turbidity (NTU)	Apparent color (unit Pt/Co)
1 bar – A	Feeding	2.75 ± 0.02a	12.00 ± 0.00a
	Permeate	0.20 ± 0.01b	1.67 ± 0.58a
	Retentate	13.70 ± 0.00a	70.00 ± 1.00a
1 bar – B	Feeding	2.75 ± 0.02a	12.00 ± 0.00a
	Permeate	0.22 ± 0.00b	1.33 ± 0.58a
	Retentate	13.53 ± 0.06b	70.67 ± 0.58a
1 bar – C	Feeding	2.75 ± 0.02a	12.00 ± 0.00a
	Permeate	0.25 ± 0.01a	0.67 ± 0.58a
	Retentate	9.16 ± 0.01c	47.33 ± 0.58b
MSD	Feeding	0.06	2.91
	Permeate	0.02	1.69
	Retentate	0.1	2.17
2 bar – A	Feeding	4.09 ± 0.04a	27.67 ± 0.58a
	Permeate	0.18 ± 0.01c	2.00 ± 0.58a
	Retentate	7.85 ± 0.21c	103.33 ± 4.04b
2 bar – B	Feeding	4.09 ± 0.04a	27.67 ± 0.58a
	Permeate	0.24 ± 0.00b	2.00 ± 0.00a
	Retentate	15.83 ± 0.06b	109.00 ± 2.65a
2 bar – C	Feeding	4.09 ± 0.04a	27.67 ± 0.58a
	Permeate	0.28 ± 0.02a	2.00 ± 1.00a
	Retentate	16.43 ± 0.06a	112.67 ± 0.58a
MSD	Feeding	0.12	1.69
	Permeate	0.04	1.94
	Retentate	0.38	8.17

Samples (mean ± standard deviation) followed by the same letter in the same row (feeding; permeated; retentate) do not differ significantly at 5%. MSD = minimum significant difference.

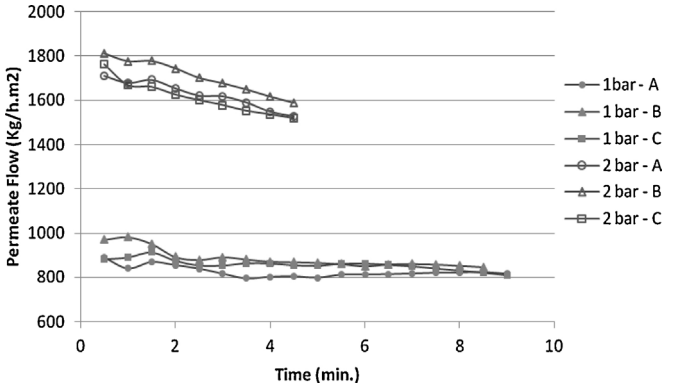

Fig. 2. Permeate flow obtained in 3 tests of ultrafiltration of lime juice recovered water in membrane CEL 30 at 1 bar (A–C) and at 2 bar (A–C).

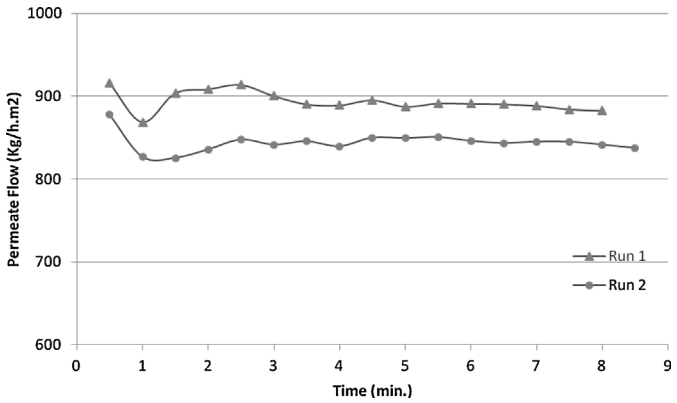

Fig. 3. Permeate flows obtained in two ultrafiltration tests of recovered water of concentrated orange juice in membrane CEL 30 at 1 bar (run 1 and run 2) on a laboratory scale.

Table 8
Analyzes results of turbidity and apparent color in samples of feeding, permeate and retentate collected on tests of ultrafiltration of recovered water of concentrate orange juice in CEL 30 membrane at pressure at 1 bar, laboratory scale.

Test	Sample	Turbidity (NTU)	Apparent color (unit Pt/Co)
Run 1	Feeding	2.15 ± 0.02a	11.33 ± 0.58a
	Permeate	0.29 ± 0.01a	1.33 ± 0.58a
	Retentate	7.33 ± 0.08b	47.00 ± 1.73b
Run 2	Feeding	2.15 ± 0.02a	11.33 ± 0.58a
	Permeate	0.18 ± 0.02b	0.33 ± 0.58a
	Retentate	10.00 ± 0.21a	64.33 ± 0.58a
MSD	Feeding	0.07	2.04
	Permeate	0.06	2.04
	Retentate	0.56	4.56

Samples (mean ± standard deviation) followed by the same letter in the same row (feeding; permeated; retentate) do not differ significantly at 5%. MSD = minimum significant difference.

similar in form and absolute flow values that are quite high and were maintained stabilized for nearly the entire experiment.

3.4.4. Ultrafiltration of recovered water of orange juice and lime juice on Pilot Unit

The experiments conducted in pilot scale with recovered water of concentrated lime juice (LIME 1, LIME 2 and LIME 3) and concentrated orange juice water (orange), using membrane CEL 30 at pressure at 1 bar are shown in Fig. 4 and Table 9.

It was observed in the experiments in pilot plant (Fig. 4), a beginning with a very low flow, close to zero, and the occurrence of sudden increase in flow with subsequent stabilization, instead of the characteristic drop in the membrane separation processes. The increased flow phenomenon along time is called "paradoxical flow" and it is characterized by the drag of smaller particles through the membrane by the permeate early in the process. These particles are deposited on the membrane causing the momentary blockage of the pores, and subsequently dragged by the permeate. After drag, there is a majority deposition of macroparticles in the polarized layer, so that the flow tends to increase until the membrane fouling process is started. From this point, a slight decline of permeate flow occurs, until its stabilization or continuous smooth decline. The importance of the paradoxical flow theory is signaling that there is a modification in the composition and arrangement of the feed-membrane interface in the early stages of the filtration process. These changes are responsible for generating changes in permeate flow as was also reported by Silva (2010) [28] and Debien [8]. It is important to highlight that the occurrence of paradoxical flow is directly related to the predominance of tangential flow in the filtration process performed in the pilot plant. In the tests conducted in laboratory it was not observed paradoxical flow, because occurred

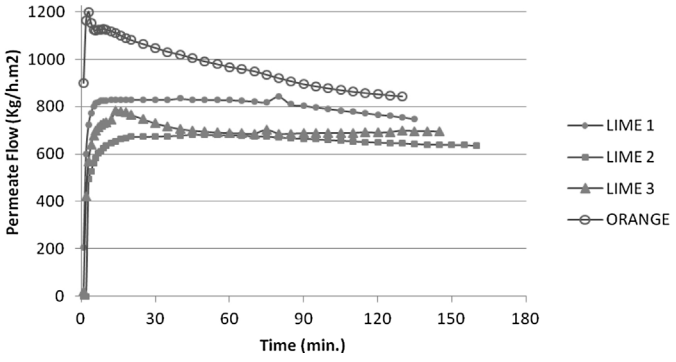

Fig. 4. Permeate flow obtained in 4 ultrafiltration tests of recovered water of concentrated lime juice (LIME 1, LIME 2 and LIME 3) and recovered water of concentrate orange juice (orange) in membrane CEL 30 at 1 bar in Pilot Unit.

Table 9
Analyzes results of turbidity and apparent color in samples of feeding, permeate and retentate collected in the tests of ultrafiltration of recovered water of concentrated lime juice and orange in membrane CEL 30 at pressure at 1 bar, in Pilot Unit.

Test	Sample	Turbidity (NTU)	Apparent color (unit Pt/Co)
LIME 1	Feeding	1.61 ± 0.00c	9.67 ± 0.58c
	Permeate	0.22 ± 0.00c	0.67 ± 0.58a
	Retentate	7.43 ± 0.00d	48.33 ± 1.53c
LIME 2	Feeding	31.27 ± 0.32b	286.00 ± 1.00b
	Permeate	0.27 ± 0.01b	0.67 ± 0.58a
	Retentate	140.67 ± 0.58a	940.00 ± 14.42a
LIME 3	Feeding	63.70 ± 0.44a	471.33 ± 11.72a
	Permeate	0.23 ± 0.01c	0.33 ± 0.58a
	Retentate	76.90 ± 0.26b	497.33 ± 8.33b
Orange	Feeding	1.92 ± 0.03c	11.33 ± 0.58c
	Permeate	0.38 ± 0.02a	0.33 ± 0.58a
	Retentate	8.70 ± 0.02c	46.67 ± 0.58c
MSD	Feeding	0.77	16.68
	Permeate	0.03	1.64
	Retentate	0.9	23.67

Samples (mean ± standard deviation) followed by the same letter in the same row (feeding; permeated; retentate) do not differ significantly at 5%. MSD = minimum significant difference.

predominance of frontal flow, despite the tangential effect caused by the magnetic stirrer rotating horizontally be very expressive.

Fig. 4 still shows that the permeate flow stabilized at around 700 kg/h m^2 for lime recovered water, and around 800 kg/h m^2 for orange recovered water, similar values to those obtained in laboratory scale .

It can be observed in Table 9 that parameter reduction levels of apparent color and turbidity met the objectives proposed in this study, and therefore to the Brazilian regulations for bottled water aiming human consumption. The data in this table also allows us to observe that despite the values of apparent color and turbidity are significantly different for each one of lime feeds, this does not affect the outcome of these parameters for the permeate. It should be noted that the raw material of LIME 1 resembles the orange and the in permeation curves these two raw materials showed similar performance regarding the flow.

Thus, the testing with evaporated water of orange reproduced the results obtained with lime water in both laboratory scale and pilot scale, as it can be seen in Tables 8 and 9 and Figs. 3 and 4. As for the parameter of permeate apparent color the lime recovered water samples did not differ statistically of the orange recovered water sample at 5% probability level.

The literature does not present articles related to the study of evaporated water treatment of orange or lime, applying the micro or ultrafiltration processes. Arnal et al. (2009) [26] argue that ultrafiltration is effective in reducing turbidity of surface water, but the presented results involve the use of membranes of pores more open than those used in this study. In addition, water surface has different characteristics than evaporated water.

Debien [8] used membrane CEL 30 in order to clarify coconut water, reporting a reduction of about 90% in turbidity of the product. This reduction is consistent with the results obtained in this study with lime water, especially in laboratory testing unit, being overcome in the tests performed in pilot plant where the reduction level achieved was higher than 99% in two experiments. In the case of orange recovered water, the reduction of turbidity in pilot scale was about 80%, while the reduction of turbidity in laboratory scale is also in agreement with that reported by Debien [8], of about 90%.

Thus, ultrafiltration of evaporated water of orange juice in membrane CEL 30 at pressure at 1 bar was efficient and, therefore, selected as the most appropriate among those studied for the correction of apparent color and turbidity of orange water to the

recommended levels in accordance with the Brazilian legislation for bottled water.

4. Conclusion

Considering the Brazilian technical regulation for bottled water, the evaporated water, during the concentration of orange juice, is in accordance with the maximum limits established for organic substances, inorganic, pesticides and microbiological requirements. The only parameters that exceeded the limits were apparent color and turbidity. The process of ultrafiltration in cellulose membrane 30 kDa at pressure at 1 bar was effective in reducing the apparent color and turbidity of recovered water of concentrate orange juice, fixing these parameters to appropriate values, even below the maximum limits established by the Brazilian legislation for bottled water. Thus, evaporated water of orange juice concentration can be used for human consumption, after ultrafiltration, with good permeate flow values, in cellulose membrane 30 kDa.

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