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## Qualitative characteristics and dead-end ultrafiltration of chicory juice obtained from pulsed electric field treated chicories

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#### a r t i c l e i n f o

Article history: Received 8 October 2012 Received in revised form 7 December 2012 Accepted 10 January 2013

Keywords: Pulsed electric field (PEF) Extraction Chicory juice Inulin Ultrafiltration

#### A B S T R A C T

Qualitative characteristics and dead-end ultrafiltration behavior of chicory juice obtained at different temperatures from untreated and pulsed electric field (PEF) treated chicory roots are investigated. The chicory juices obtained at lower temperatures (30 and 60 ℃) from PEF treated chicories are less colorized, have lower turbidity, lower protein content, and higher purity than conventional juice obtained at 80 ◦C.

The ultrafiltration behavior of the untreated and PEF treated chicory juice was studied in the deadend filter cell under different pressures (1, 2 and 4 bars) and with stirring at 500 rpm. Three types of polyethersulfone membranes with MWCO of 150, 50 and 5 kDa were used. The membrane with MWCO of 50 kDa produced the most purified filtrate, while the membrane with lower MWCO (5 kDa) produced less purified filtrate probably due to the partial retention of inulin molecules. The increase of TMP from 1 to 2 bars increased the filtration flux and the juice purity. The ultrafiltration behavior of obtained chicory juices was successfully described by the filtration model with intermediate blocking mechanism.

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#### **1. Introduction**

Inulin is a kind of non digestible carbohydrate and can be used as a fat replacer in food fabrication [\(Vendrell-Pascuas](#page--1-0) et [al.,](#page--1-0) [2000\).](#page--1-0) It belongs to fructan group, consisting of 2–70 repeating units of fructose [\(Franck](#page--1-0) [and](#page--1-0) [De](#page--1-0) [Leenheer,](#page--1-0) [2005\).](#page--1-0) Inulin molecules obtained from different plants differ in their degree of polymerization (DP) and molecular weights [\(Lopez-Molina](#page--1-0) et [al.,](#page--1-0) [2005\).](#page--1-0)

Chicory roots are widely used for commercial production of inulin [\(Toneli](#page--1-0) et [al.,](#page--1-0) [2008;](#page--1-0) [Baert,](#page--1-0) [1997;](#page--1-0) [Baert](#page--1-0) [and](#page--1-0) [Bockstaele,](#page--1-0) [1992\),](#page--1-0) as well as for herbicidal product extraction ([Wang](#page--1-0) et [al.,](#page--1-0) [2011\).](#page--1-0) Conventionally, harvested chicory roots are washed under highpressure water jet, sliced and then transported into extractor for the aqueous diffusion of inulin at 70–80  $\degree$ C during 1–2 h [\(Loginova](#page--1-0) et [al.,](#page--1-0) [2011a\).](#page--1-0) The thermal denaturation of chicory tissue in a hot water leads to intensive inulin transfer into the juice. Additionally, other cell components, for instance proteins, pectins, colloids and colorants, pass into the juice decreasing its purity ([Zhu](#page--1-0) et [al.,](#page--1-0) [2012\).](#page--1-0)

Pulsed electrical field (PEF) has been used to realize more selective non-thermal or moderate thermal extraction of active components from various plants. For instance, [Loginova](#page--1-0) et [al.](#page--1-0) [\(2011b\)](#page--1-0) reported higher purity of juice obtained by cold diffusion  $(30\degree C)$  from the PEF treated sugar beet. [Zhu](#page--1-0) et [al.](#page--1-0) [\(2012\)](#page--1-0) reported

the similar or better quality of juice obtained by moderate thermal diffusion (50–60 $\degree$ C) from the PEF treated chicory in compare to the juice obtained by conventional diffusion (70–80 $\degree$ C) from the untreated chicory.

Industrially, one possible purification process of chicory juice consists of pre-liming, liming, first carbonation, first filtration, second carbonation and second filtration [\(Franck](#page--1-0) [and](#page--1-0) [De](#page--1-0) [Leenheer,](#page--1-0) [2005\).](#page--1-0) This multistage process is similar to the sugar beet juice purification [\(Loginova](#page--1-0) et [al.,](#page--1-0) [2012;](#page--1-0) [Asadi,](#page--1-0) [2007\).](#page--1-0) After such multistage purification, most juice impurities can be removed. The quantity of lime used for purification depends on the initial juice composition and purity. It may be speculated that more pure juices, obtained with PEF treatment, are easier to purify than the juices obtained by conventional diffusion at 70–80 $°C$ . This is the way to decrease the lime consumption or even to purify the PEF juice without lime, using only membrane ultrafiltration.

Previous study shows that the sugar beet juice obtained from PEF assisted diffusion needs less lime consumption [\(Loginova](#page--1-0) et [al.,](#page--1-0) [2012\)](#page--1-0) or alternatively can be purified by ultrafiltration ([Loginov](#page--1-0) et [al.,](#page--1-0) [2011\).](#page--1-0)

Membrane filtration of different bio-suspensions and juices has been intensively studied ([De](#page--1-0) [Bruijn](#page--1-0) et [al.,](#page--1-0) [2002;](#page--1-0) [Cassano](#page--1-0) et [al.,](#page--1-0) [2008;](#page--1-0) [Rai](#page--1-0) et [al.,](#page--1-0) [2006;](#page--1-0) [Yazdanshenas](#page--1-0) et [al.,](#page--1-0) [2010\).](#page--1-0) Ultrafiltration was reported to be a possible technique for purification of raw and partially clarified sugar beet and sugar cane extracts [\(Ghosh](#page--1-0) et [al.,](#page--1-0) [2000;](#page--1-0) [Kishihara](#page--1-0) et [al.,](#page--1-0) [1989;](#page--1-0) [Hamachi](#page--1-0) et [al.,](#page--1-0) [2003;](#page--1-0) [Dornier](#page--1-0) et [al.,](#page--1-0) [1995\).](#page--1-0) It was shown that ultrafiltration may decrease concentration

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of impurities (pectins, proteins and colloids) in the juice and increase juice purity ([Shahidi](#page--1-0) [and](#page--1-0) [Razavi,](#page--1-0) [2006;](#page--1-0) [Hakimzadeh](#page--1-0) et [al.,](#page--1-0) [2006;](#page--1-0) [Vern](#page--1-0) et [al.,](#page--1-0) [1995;](#page--1-0) [Gyura](#page--1-0) et [al.,](#page--1-0) [2002\).](#page--1-0) However, the membrane filtration of conventional sugar beet and chicory juice is not yet industrially implemented due to the low filterability of these juices. The common phenomenon in pressure-driven membrane filtration is the membrane fouling, which can result in permeate flux declining with time. This phenomenon is resulted by accumulation of feed components in the membrane pores as well as on the membrane surface ([Gokmen](#page--1-0) [and](#page--1-0) [Cetinkaya,](#page--1-0) [2007\).](#page--1-0) Several efforts have been devoted to minimize membrane fouling, such as pre-treatment of feed juice and more suitable membrane selection ([Saha](#page--1-0) et [al.,](#page--1-0) [2009\).](#page--1-0)

The juices extracted alternatively, with PEF treatment, contain less impurity than conventional juice obtained at the elevated temperatures. Therefore, it can be supposed that the PEF juices are better adapted for the membrane filtration. However, the membrane filtration of chicory juices obtained with PEF treatment was not yet studied.

In this study, ultrafiltration was used to purify chicory juice obtained with PEF treatment on the pilot scale extractor. The qualitative juice characteristics (color, proteins content, purity) were determined for different extraction temperatures (30, 60 and  $80^{\circ}$ C). The effects of molecular weight cut-off (MWCO) of membrane, transmembrane pressure (TMP) and juice extraction conditions (PEF treatment and diffusion temperature) on the filtrate purity and membrane fouling were investigated.

#### **2. Materials and methods**

#### 2.1. Preparation of chicory juice for ultrafiltration

The fresh chicory roots provided by COSUCRA, Belgium were sliced and then electrically treated using the stainless steel electrodes and a pilot PEF generator 5 kV–1000A (Hazemeyer, France). The monopolar pulses with electric field intensity of  $E = 600$  V/cm, pulse duration  $t_i$  = 100  $\mu$ s and pulse repetition time  $\Delta t$  = 5 ms were applied. The number of pulses  $n$  was fixed at 500. The total PEF treatment time  $t_{PEF}$  was  $n \cdot t_i$  = 0.05 s and the electric energy consumption was 25 kJ/kg of slices.

Hot water extraction of untreated and PEF treated slices were carried out in a temperature controlled counter current pilot-scale extractor. Construction and operating principles of the extractor are reported in a previous work ([Zhu](#page--1-0) et [al.,](#page--1-0) [2012\).](#page--1-0) Diffusion experiments were carried out at  $T = 30^{\circ}$ C,  $80^{\circ}$ C (for untreated slices), and at  $T = 30 \degree C$ ,  $60 \degree C$  (for PEF treated slices). Diffusion duration was 90 min. Therefore, four different juices were obtained after extraction. They will be referred below as "control juice at 30 ℃", "control juice at 80 ◦C" for the diffusion without PEF treatment, and "PEF juice at 30 $\,^{\circ}$ C", "PEF juice at 60 $\,^{\circ}$ C" for the diffusion with PEF treatment, corresponding to the inulin diffusion temperatures.

#### 2.2. Ultrafiltration of inulin juice

Ultrafiltration set-up is presented in Fig. 1. Dead-end ultrafiltration was performed in a stirred cell Amicon 8200 (effective membrane area  $3.17 \times 10^{-3}$  m<sup>2</sup> and maximal volume 180 mL) (Millipore, Billaica, USA). For each experiment, 100 mL of feed juice was used, and 70 mL of filtrate was obtained. Three types of hydrophilic polyethersulfone ultrafiltration membranes (Microdyn-Nadir GmbH, Germany) with molecular weight cut-off (MWCO) of 5, 50 and 150 kDa were used to purify feed juice. New membrane was used for each set of experiments. The stirring was done by means of magnetic stirrer fixed over the membrane surface and rotating at the constant rate of  $\omega$  = 500 rpm. Ultrafiltration



**Fig. 1.** Schematic diagram of the filtration set-up.

experiments were performed at room temperature applying transmembrane pressure (TMP) of 1, 2, and 4 bars. The mass of filtrate during filtration was recorded by a computer.

#### 2.3. Analysis of juice and filtrate

The concentration of soluble solids (Brix, %) in juice was measured using a digital refractometer PR-32a (ATAGO Co., LTD, Japan). Each 3 g of filtrate were sampled for Brix measurement.

The concentration of proteins was determined by means of Bradford method ([Bradford,](#page--1-0) [1976\).](#page--1-0) The details of analysis are presented in Technical Bulletin for Bradford Reagent (B 6916, Sigma–Aldrich).

The coloration and turbidity of juices were measured and calculated according to recommendations of international commission for uniform methods of sugar analysis (ICUMSA). The juice turbidity was expressed as the difference between the absorbance measured at 720 nm before and after the filtration of the sample through the  $0.45 \,\mu$ m membrane. The coloration of juices (pre-filtered through the 0.45  $\mu$ m membrane) was calculated as:

#### Coloration = (aborbance at  $420 \text{ nm} \times 10^5$ )/Brix (1)

The volume-based function of particle size distribution SDF (%) of extracted juice was measured using an analytical photocentrifuge LUMiSizer 610.0–135 (L.U.M. Gmbh, Germany). Rectangular plastic optical cells, supplied by the photocentrifuge manufacturer, with the optical path length of 10 mm and cross-sectional area of  $7 \times 10^{-5}$  m<sup>2</sup>, were used. SDF was calculated using the original software SEPView 5.1 (L.U.M. Gmbh, Germany) for granulometric analysis.

Inulin content was analyzed using a high performance liquid chromatography (HPLC) [\(Monti](#page--1-0) et [al.,](#page--1-0) [2005\).](#page--1-0) The samples were adjusted with HCl (1 mol/L) solution to assure the pH in the range of 3.9–4.5, then hydrolyzed by novozyme (NOVO inulinase Fructozyme® (previously called SP230)), incubating at  $60-62$  °C for 2 h. After filtered by 0.45  $\mu$ m membrane, 20  $\mu$ L of the filtrates were injected in the HPLC column (Rezex RKP-potassium K+ 8%,  $300 \times 7.8$  mm), column temperature was 85 °C. Water with a flow rate of 0.5 mL min−<sup>1</sup> was used as the mobile phase. The retention time was 20 min. The areas of the identified HPLC peaks were calculated and were used to confirm the inulin concentration by comparison with standards.

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