LWT - Food Science and Technology 93 (2018) 511–516

Contents lists available at ScienceDirect

LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

Selectivity of ultrasound-assisted aqueous extraction of valuable compounds from flesh and peel of apple tissues

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ARTICLE INFO

Keywords:
Apple flesh and peel tissues
Ultrasound-assisted extraction
Soluble matter
Catechin
TPC

ABSTRACT

Ultrasound-assisted aqueous extraction (UAE) of soluble matter (°Brix), catechin and total phenolic contents (TPC) from flesh and peel of apple tissues were studied. The commercial green (Granny Smith) and red (Red delicious) apples were used in the investigation. All extractions were done at fixed temperature, $T = 50$ °C, and protocols with different specific energy inputs, S_1 ($W = 0$ kJ/kg), S_2($W \approx 6.5$ kJ/kg), S_3($W \approx 10.6$ kJ/kg), S_4($W \approx 21.3$ kJ/kg), and S_5($W \approx 26.8$ kJ/kg) during the initial period of extraction (first 300–350 s). The total aqueous extraction time was up to 3 h. The kinetics of extractions, and correlations °Brix and concentration of catechin, C, were compared for flesh and peel tissues. The increase of energy resulted in increase of °Brix and C values. For both green and red apples, the values of °Brix and C were noticeably higher for peel as compared to flesh. The distinct correlations between saturation levels of C_m and TPC_m at the long extraction time (≈ 3 h) were observed. However, the relative content of catechin in TPC (i.e., ratio C_m/TPC_m) evidenced the presence of selectivity in extraction of catechin. This selectivity depends from the type of tissue (flesh or skin), apple variety (green or red) and applied treatment protocols.

1. Introduction

Apples contain various nutrients beneficial to human health with strong anti-inflammatory effects and high ability to prevent chronic diseases (González-Gallego, García-Mediavilla, Sánchez-Campos, & Tuñón, 2010). They include vitamin C, soluble fibre, and different dietary polyphenols (flavanols, flavonols, phloridzin, procyanidin, chlorogenic acid, anthocyanin) (Boyer & Liu, 2004; Weichselbaum, Wyness, & Stanner, 2010). The content of most abundant polyphenols in apple ranges between 19.6 and 55.8 mg for flavanols (e.g., (+)-catechin and (−)-epicatechin), 17.7–33.1 mg for flavonols (e.g., quercetin), and 10.6–80.3 mg for phenolic acids (e.g., chlorogenic acid) (McGhie, Hunt, & Barnett, 2005). Nowadays the extraction of phenolic compounds from apple pomace (peels, seeds, core, stem and calyx) attracts a great attention (Ćetković et al., 2008). Apple pomace is the solid waste product resulting from industrial processing of apple juice or cider production and it can be considered as a potential source of food antioxidants (Lu & Foo, 2000).

The different methods of polyphenols extraction from plant products include conventional solvent extraction (CE), extraction assisted by enzymatic, ultrasound, microwave, pulsed electric fields, and other treatments (Acosta-Estrada, Gutiérrez-Uribe, & Serna-Saldívar, 2014; Ameer, Shahbaz, & Kwon, 2017; Barba et al., 2015; Caballero-Valdés, Olivares-Miralles, Soto-Maldonado, & Zúñiga-Hansen, 2016; Donsì, Ferrari, & Pataro, 2010).

Ultrasound has been previously used for polyphenols extraction from apples (Pingret, Fabiano-Tixier, Le Bourvellec, Renard, & Chemat, 2012; Virot, Tomao, Le Bourvellec, Renard, & Chemat, 2010; Yue, Shao, Yuan, Wang, & Qiang, 2012). Being environmentally friendly the ultrasound-assisted extraction (UAE) technique is widely recognized as “green and innovative”, which typically involves reduced operating and maintenance costs, moderate energy consumption and small processing time, low quantity of water and solvents (Chemat et al., 2017a; Chemat et al., 2017b). Application of the UAE technique also allows reduction of wastes and elimination of generation of hazardous substances.

The UAE (25 kHz, 150 W) of polyphenols from apple pomace has been tested using the optimal concentration of ethanol/aqueous solution of 50% (v/v), solid/liquid ratio of 15% (w/v), and moderate temperatures $16.0 \leq T$ (°C) ≤ 34.0 (Virot et al., 2010). Application of UAE for 45 min allowed increasing the total phenolics content (TPC) by more than 20% as compared with CE. The UAE for recovery of polyphenols from the unripe apple at different concentrations of ethanol

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https://doi.org/10.1016/j.lwt.2018.04.007
Received 6 December 2017; Received in revised form 25 February 2018; Accepted 3 April 2018
Available online 04 April 2018
0023-6438/

(40–90% v/v), temperature (30–80 °C), time of extraction (10–30 min), and ultrasound power (280–560 W) has been tested. At optimum extraction conditions (ethanol concentration of 50%, temperature 50 °C, time of 30 min, ultrasonic power of ≈520 W), the TPC value of 13.26 ± 0.56 mg GAE/g was found (Yue et al., 2012). Different processing factors can affect the UAE efficiency of polyphenols recovery and their purity (Chemat et al., 2017b; Pingret et al., 2012). However, the impact of UAE protocols on the selectivity of valuable compounds recovery from apple products was not yet elucidated.

This work is focused on the effects of pulsed ultrasonic treatment on the initial period of extraction (300–350 s) of soluble matter (°Brix), catechin and TPC from the different parts (flesh and peel) of green (Granny Smith) and red (Red delicious) apples. The extraction experiments were done at fixed temperature (T = 50 °C). For UAE protocols with different power inputs the correlations between °Brix, TPC and catechin concentration, C, were evaluated.

2. Materials and methods

2.1. Material

Commercial green apples (Granny Smith) and red apples (Red delicious) were selected as the raw material for investigation. The apples with good and uniform quality and near-spherical shape were purchased at the local supermarket (Compiegne, France). In total, 60 apples were taken for experiment analyses. The initial moisture content on wet basis (83.74 g/100 g flesh and 83.07 g/100 g peel for green apples, and 87.44 g/100 g flesh and 85.39 g/100 g peel for red apple) was determined using MA 160 infrared moisture analyzer (Sartorius, Germany).

2.2. Extraction experiments

The flesh tissue (the disks with diameter of 20 mm and thickness of 10 mm) was taken from the central part of the apple. The peel tissue (thin slices with length of 20 mm, width of 10 mm, and thickness of ≈0.1 mm) was removed from the apple with a razor blade.

In extraction experiments the flesh or peel tissue (20 g) were put into a glass beaker filled with preheated (50 °C) distilled water (200 mL), and solid liquid ratio was 1:10. The glass beaker was covered with aluminium foil in order to prevent water evaporation. The total extraction time was up to 3 h. UAE was done directly in the glass beaker using an ultrasonic processor UP 400S (400 W, 24 kHz, Hielscher GmbH, Stuttgart, Germany). The titanium ultrasonic probe (H14, Hielscher GmbH, Stuttgart, Germany) with a tip diameter of 14 mm, and the length of 100 mm was used.

The different extraction protocols presented in Table 1 were performed at fixed temperature (50 °C) of a thermal water bath Polystat 36 (Fisher Scientific, France). The protocol S_1 corresponds to the aqueous CE and protocols S_2-S_5 correspond to the initial ultrasonic treatment with different pulsed modes. The moderate temperature of 50 °C was used to avoid destruction of organic compounds as well as provide an efficient application of ultrasound (Pingret, Fabiano-Tixier, & Chemat, 2013). Prolonged sonication can also cause degradation of targeted compounds. In our experiments the sonication time range chosen (from 50 to 100 s) was relatively short. Note that no specific reaction products after sonication (5–55 min) applied to the isolated phenolic compounds of apple pomace were observed (Pingret et al., 2013).

Table 1
Parameters of applied extraction protocols, S_1-S_5. Here, Δt_u and Δt_w are the duration of pulsed sonication and cooling of the sample during one pulse, respectively, n is the number of pulses, $t_t = n(\Delta t_u + \Delta t_w)$ is the total duration of pulsed sonication, W is specific energy and P is the actual power of ultrasonic treatment. After UAE the CE continued up to 3 h.

Protocol #	Δt_u,s	Δt_w,s	n	t_t, s	W, kJ/kg	P,W
S_1	–	–	0	–	–	–
S_2	10	50	5	300	6.4	28
S_3	10	50	5	300	10.7	47
S_4	20	50	5	350	21.4	47
S_5	10	50	5	300	26.8	118

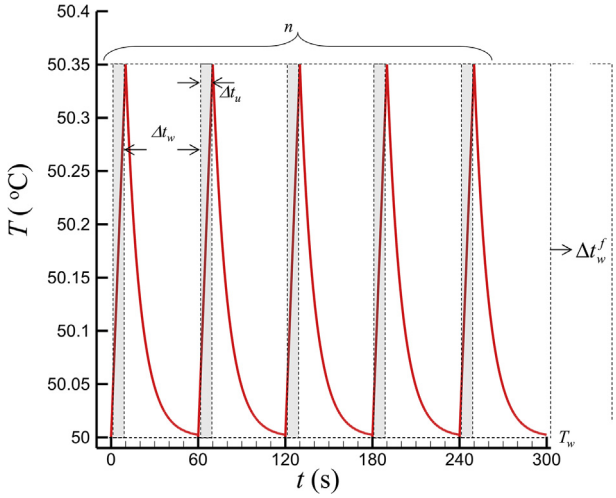

Fig. 1. Scheme of pulsed UAE protocol S_2 (Table 1). Here, the temperature evolution during application of pulses a series of n = 5 pulses with duration of Δt_u = 10 s is presented. During the pause (Δt_w = 50 s), the temperature relaxes to the initial level of T_w = 50 °C by cooling in cold water. After the initial UAE the CE was continued up to Δt_w^f = 3 h.

The actual ultrasonic power introduced to the system was estimated from the temperature elevation ΔT in sample using following equation

$$P = mCp\Delta T/\Delta t_u \quad (1)$$

where m and C_p (≈4.18 kJ/kg K) are the mass and specific heat capacity of sample, respectively, Δt_u is the duration of sonication.

The scheme of applied pulsed sonication treatment for protocol S_2 is illustrated in Fig. 1. During the application of the ultrasonic pulse with duration of Δt_u = 10 s the insignificant temperature elevation ($\Delta T \approx 0.35$ °C) was observed. During the pause with duration of Δt_w = 50 s, the temperature relaxes by cooling in cold water up to the initial value of 50 °C. After UAE with application of n = 5 sequential pulses, the CE was continued up to 3 h. The specific energy input for this protocol was $W \approx 6.5$ kJ/kg. The total time of UAE was 300–350 s and the values of W were increased in the raw S_1-S_4. The applied protocols allows testing the contribution of ultrasound power to the extraction yield during the first part of extraction (Chemat et al., 2017b; Pingret et al., 2012).

2.3. Analysis

The obtained extracts were analyzed for °Brix, catechin concentration and TPC. The concentration of soluble matter °Brix (g of DM/100 g solution) was measured using the refractometer (Atago, USA) at room temperature.

The concentration of catechin, C, was estimated by fluorescence technique using the instrument Cary Eclipse Fluorescence Spectrofluorometer and 10 mm fused-silica cuvette (Agilent Technologies, USA). The fluorescence spectra were obtained using the excitation at λ_e = 280 nm and registration of emission λ = 312 nm (Arts, Van de Putte, & Hollman, 2000). Standard of catechin hydrate (≥0.98%, Sigma-Aldrich) was used for the calibration curve.

The total polyphenols content was determined using the Folin–Ciocalteu method based on a colorimetric oxidation/reduction reaction of phenols (Singleton, Orthofer, & Lamuela-Raventós, 1999). 0.2 mL of diluted extract and 1 mL of Folin–Ciocalteu reagent (Merck,