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Establishment of ultrasound-assisted extraction of phenolic compounds from industrial potato by-products using response surface methodology

Ylenia Riciputi[a], Elixabet Diaz-de-Cerio[b], Hazal Akyol[c], Esra Capanoglu[c], Lorenzo Cerretani[d], Maria Fiorenza Caboni[a,e], Vito Verardo[f,g,*]

[a] Inter-departmental Centre for Agri-Food Industrial Research (CIRI Agroalimentare), Alma Mater Studiorum-University of Bologna, via Quinto Bucci 336, 47521 Cesena, FC, Italy
[b] Department of Analytical Chemistry, University of Granada, C/Fuentenueva s/n, 18071 Granada, Spain
[c] Department of Food Engineering, Faculty of Chemical and Metallurgical Engineering, Istanbul Technical University, Ayazağa Campus, 34469 Maslak, Istanbul, Turkey
[d] Pizzoli SpA, via Zenzalino Nord 1, 40054 Budrio, Bologna, Italy
[e] Department of Agriculture and Food Sciences, Alma Mater Studiorum-University of Bologna, Piazza Goidanich 60, 47521 Cesena, FC, Italy
[f] Department of Nutrition and Food Science, University of Granada, Campus of Cartuja, 18071 Granada, Spain
[g] Institute of Nutrition and Food Technology 'José Mataix', Biomedical Research Centre, University of Granada, Avenida del Conocimiento s/n, E-18071 Granada, Spain

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ABSTRACT

Potato processing generates large amounts of by-products, which include potato peels and the outer layers of flesh, which contain phenolic compounds. The purpose of this study was to establish an extraction method for phenolic compounds from industrial potato by-products by using response surface methodology (RSM). Box-Behnken design (BBD) was performed to optimize the extraction conditions of phenolic compounds considering different extraction temperature, ratios of ethanol/water, time of extraction and sample/solvent ratio. The optimum extraction conditions were obtained with ethanol/water 55/45 (v/v) by ultrasound bath during 35 min at 35 °C and 1/10 sample/solvent ratio. The best conditions were applied to determine the phenolic content in five potato by-products. The analyses by HPLC-DAD-ESI-MS showed that chlorogenic acid accounted for a 49.3–61% of the total phenolic compounds.

Positive Pearson correlations between HPLC data and antioxidant activity confirmed that the phenolic compounds had significant antioxidant properties.

1. Introduction

Potato is one of the most consumed foods in the world and it also has a wide place of industry. Although the potato cultivated globally belongs to just one botanical species, *Solanum tuberosum* L., the tubers come from thousands of varieties with great differences in size, shape, colour, texture, flavour and cooking characteristics (FAO, 2008).

The global use of potatoes is moving from fresh potatoes to processed products such as fries, chips, mashed and canned potatoes (Sabeena Farvin, Grejsen, & Jacobsen, 2012; Tierno et al., 2015). After potato processing, a lot of waste is produced as peels, causing handling and storage problems (Singh & Saldaña, 2011).

Potato provides noteworthy portions of phenolic compounds located in the peel and the close tissues. In many studies, it is reported that potato is a good source of phenolics with an antioxidant capacity (Albishi, John, Al-Khalifa, & Shahidi, 2013; Kanatt, Chander, Radhakrishna, & Sharma, 2005; Koduvayur Habeebullah, Nielsen, & Jacobsen, 2010; Mohdaly, Sarhan, Smetanska, & Mahmoud, 2010). Among the reported phenolic components in potato peels, caffeic acid and chlorogenic acid play the main roles in the antioxidant activity (Nara, Miyoshi, Honma, & Koga, 2006; Rodriguez de Sotillo, Hadley, & Holm, 1994; Wu et al., 2012). These components aid to constitute defence mechanisms against plant diseases and protect cells from excessive oxidation and free-radical damage. In nutrition, they have received significant consideration as they contain potentially protective factors because of their potent antioxidant properties (Akyol, Riciputi, Capanoglu, Caboni, & Verardo, 2016; Friedman, 1997; Mäder, Rawel, & Kroh, 2009).

Some epidemiological investigations showed a negative correlation between the intake of dietary antioxidant components and diseases, such as atherosclerosis, cancers, faster aging of the body, and heart attacks (Lindberg Madsen, Andersen, Jorgensen, & Skibsted, 2000; Lotito & Frei, 2004; Othman, Ismail, Abdul Ghani, & Adenan, 2007).

In this two-stage study, first optimum extraction conditions were

* Corresponding author at: Department of Nutrition and Food Science, University of Granada, Campus of Cartuja, 18071 Granada, Spain.
E-mail address: vitoverardo@ugr.es (V. Verardo).

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determined for the *Fontane* cultivar. Afterwards, antioxidant properties and total phenolic contents of potato peels were evaluated in *Fontane* and four other cultivars (*Bintje, Challenger, Daisy and Innovator*) using the optimum extraction conditions. HPLC-DAD-MS (high performance liquid chromatography-diode array detector-mass spectrometry) was used to identify and quantify phenolic compounds. The activity of antioxidants present in the potato peels were also evaluated using in vitro antioxidant activity assays namely, 1,1′-diphenylpicrylhydrazyl (DPPH), and 2,2′-azinobis(3-ethylbenzothiazolin)-6-sulphonate (ABTS).

2. Materials and methods

2.1. Chemicals and reagents

All the reagents were purchased from Merck (Darmstadt, Germany). All the analytical standards were supplied by Sigma-Aldrich (Saint Louis, MO, USA).

2.2. Samples

The samples were supplied by Pizzoli SpA in January 2016. All samples were harvested in Corticella (BO, Italy, 44°32′55.42″N 11°21′17.54″E) in the same field. Potato by-products were obtained from 5 potato varieties (*Bintje, Challenger, Daisy, Innovator and Fontane*) during French-fries production process. Briefly, after washing and removing the peels with steam, potato peels were collected by brushing before the blanching step. Then they were frozen at −23 °C before freeze-dried (Thermo HETO, powerdry LYOLAB 3000; Waltham, USA) and grounded to a fine powder in a blender mixer (Ika-Werke M20; Staufen, Germany).

2.3. Experimental design to set up the phenolic extraction

Box-Behnken design (BBD) was chosen to determine the extraction parameters for phenolic compounds from potato peels due to its advantages (Ferreira et al., 2007). The current design comprised 27 experimental runs with three levels (−1, 0, 1) for each factor, in order to normalize parameters, and was used to evaluate the effect on the phenolic compounds quantified via HPLC-DAD-ESI-MS. The independent factors selected for the optimization of ultrasound assisted extraction (UAE) were: ethanol/water ratio (% (v/v)) (X_1), temperature of the bath (°C) (X_2), time (min) (X_3), and solid-to-solvent (s-s) ratio (% (w/v)) (X_4). Table 1 shows the experimental design (coded and natural values of the factors) for each run.

The evaluation of the predicted model, by response surface methodology (RSM), was done adjusting the response variable to a second-order polynomial model equation (Eq. (1)) using Statistica 7.0 (2002, StatSoft, Tulsa, OK):

$$Y = \beta_0 + \sum_{i=0}^{4} \beta_i \chi_i + \sum_{i=0}^{4} \beta_{ii} \chi_{ii}^2 + \sum_{i=0}^{4} \sum_{j=0}^{4} \beta_{ii} \chi_i \chi_j . \quad (1)$$

where Y represents the response variable, sum of phenolic compounds (SPC) via HPLC-DAD-ESI-MS, X_i and X_j are the independent factors affecting the response, and β_0, β_i, β_{ii}, and β_{ij} are the regression coefficients of the model (intercept, linear, quadratic and interaction term).

To test the predicted model on the response variable, an analysis of variance (ANOVA) with 95% confidence level was performed to evaluate the effect of each factor. Besides, the regression coefficient (R^2), the *p*-value of the regression model and the *p*-value of the lack of fit (LOF) were employed to evaluate the fitness of the regression model. Optimal conditions were chosen considering the response surfaces (3D plots).

Table 1
Coded and natural values of BBD for the extraction of phenolic compounds from potato by-products.

Run	Independent factors				Response variable
	X_1	X_2	X_3	X_4	SPC (mg/g d.w.)
1	50 (−1)	35 (0)	15 (−1)	55 (0)	5.81
2	100 (1)	35 (0)	15 (−1)	55 (0)	0.21
3	50 (−1)	35 (0)	45 (1)	55 (0)	5.62
4	100 (1)	35 (0)	45 (1)	55 (0)	0.49
5	75 (0)	20 (−1)	30 (0)	20 (−1)	5.26
6	75 (0)	50 (1)	30 (0)	20 (−1)	5.29
7	75 (0)	20 (−1)	30 (0)	90 (1)	4.38
8	75 (0)	50 (1)	30 (0)	90 (1)	5.13
9	75 (0)	35 (0)	30 (0)	55 (0)	5.23
10	50 (−1)	35 (0)	30 (0)	20 (−1)	6.09
11	100 (1)	35 (0)	30 (0)	20 (−1)	0.45
12	50 (−1)	35 (0)	30 (0)	90 (1)	5.68
13	100 (1)	35 (0)	30 (0)	90 (1)	0.50
14	75 (0)	20 (−1)	15 (−1)	55 (0)	4.75
15	75 (0)	20 (−1)	45 (1)	55 (0)	4.90
16	75 (0)	50 (1)	15 (−1)	55 (0)	5.39
17	75 (0)	50 (1)	45 (1)	55 (0)	5.28
18	75 (0)	35 (0)	30 (0)	55 (0)	5.27
19	50 (−1)	20 (−1)	30 (0)	55 (0)	5.86
20	100 (1)	20 (−1)	30 (0)	55 (0)	0.33
21	50 (−1)	50 (1)	30 (0)	55 (0)	5.82
22	100 (1)	50 (1)	30 (0)	55 (0)	0.60
23	75 (0)	35 (0)	15 (−1)	20 (−1)	5.35
24	75 (0)	35 (0)	45 (1)	20 (−1)	5.77
25	75 (0)	35 (0)	15 (−1)	90 (1)	4.96
26	75 (0)	35 (0)	45 (1)	90 (1)	4.94
27	75 (0)	35 (0)	30 (0)	55 (0)	5.35

X_{1-4}: ethanol/water ratio (% (v/v)), temperature of the bath (°C), time (min), and s-s (solid-sample) ratio (% (w/v)). SPC via HPLC-DAD-ESI-MS.

2.4. Determination of phenolic compounds of potato by-products

Powdered potato by-products were extracted according to the optimal data obtained by the experimental design. Briefly, potato peels were extracted using ethanol/water 55/45 (v/v) by ultrasound bath during 35 min at 35 °C and with a solid/solvent ratio of 10% (3 g of samples and 30 ml of ethanol/water 55/45 (v/v)). Ultrasound bath was Starsonic 90 Liarre (Bologna, Italy) equipment with frequency 34 kHz, output power (W) 190RMS, dimensions (H × W × D) 345 × 315 × 246 cm.

HPLC-DAD-ESI-MS was used to determine the phenolic compounds in the extracts according to López-Cobo, Gómez-Caravaca, Cerretani, Segura-Carretero, and Fernández-Gutiérrez (2014).

The analyses were performed on a liquid chromatography apparatus HP 1100 (Agilent Technologies, Palo Alto, CA, USA) including a degasser, a binary pump delivery system and an automatic liquid sampler, and coupled with diode array and mass spectrometer (single quadrupole) detectors. The HPLC column was a Poroshell 120 EC-C18 (100 mm × 3.0 mm, 2.7 µm). Separations were carried out using the gradient elution used by López-Cobo et al. (2014).

The injection volume was 2.5 µL; UV–vis spectra were recorded from 210 to 600 nm, while the chromatograms were registered at 280 and 330 nm. Separation was carried out at 25 °C. The MS analyses were carried out in full scan mode (range *m/z* 50–1000) using an electrospray (ESI) interface in negative mode and the following conditions were applied: drying gas flow, 9.0 L/min; nebulizer pressure, 35 psig; gas drying temperature, 350 °C; capillary voltage, 3000 V; and fragmentor voltage, 80 V.

2.5. Antioxidant capacity evaluation

Antioxidant capacity of the samples and the extracts obtained during the experiments was evaluated by two different antioxidant

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