



Impact of instant controlled pressure drop on phenolic compounds extraction from pomegranate peel



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ARTICLE INFO

Article history:

Received 30 April 2016

Received in revised form 15 July 2016

Accepted 19 August 2016

Available online 24 August 2016

Keywords:

Instant controlled pressure drop

Pomegranate peel

Texturing

RSM

ICPD

ABSTRACT

In this research work, instant controlled pressure drop process (ICPD) was used as a texturing pretreatment to enhance extraction efficiency of phenolic compounds from pomegranate peel. Response surface methodology (RSM) was used to optimize ICPD. Experiments were carried out at steam pressures from 1 to 3 bar, steam exposure time from 10 to 60 s and number of pressure drop cycles from 1 to 5. The textured samples were extracted by 60% (v/v) methanol solution and the best operating conditions for ICPD were determined to be at 3 bar, 60 s and 1 cycle. In comparison with un-textured samples, total phenolic content (TPC) and antioxidant activity as inhibition percent were increased from 38.77 to 46.02 mg GA/g dry basis, and from 62.10 to 74.12%, respectively. Scanning electron microscopy (SEM) of the treated peel illustrated remarkable modification in the texture. Results showed a linear relationship between TPC and antioxidant activity of the textured pomegranate peel.

Industrial relevance: Increasing valuable compounds extraction efficiencies and achieving higher qualities are some of most important goals in food industry. Extraction of total phenols and antioxidants from natural resources and using them in different food products formulations is an interesting research area. This paper shows an efficient way for texturing pomegranate peel to enhance extraction yield of total phenols and antioxidants.

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1. Introduction

Because of the worldwide trend toward the use of natural antioxidants, there is an interest in finding them in plants (Yanishlieva, Marinova, & Pokorny, 2006). In general, herbs are one of the most valuable sources of natural antioxidants, such as phenolic compounds; the secondary metabolites of plants that have one or more aromatic rings and one or more hydroxyl groups (Fresco, Borges, Diniz, & Marques, 2006; Randhir, Lin, & Shetty, 2004). Phenolic compounds have a wide range of properties such as antimicrobial, antiallergenic, anti-inflammatory, antioxidant activity and radical scavenging properties (Benavente-Garcia, Castillo, Marin, Del Rio, & Ortuno, 1997; Cook & Samman, 1996; Nawaz, Shi, Mittal, & Kakuda, 2006). One of the fruits, rich in polyphenols is pomegranate. Pomegranate (*Punica granatum* L.) is native to Mediterranean region, China, Indian subcontinent and Iran (Celik, Temur, & Isik, 2009; Lansky & Newman, 2007; Murthy, Jayaprakasha, & Singh, 2002). Consumption of pomegranate fruit has nutritional and medical benefits including anticancer and antibacterial activities (Negi & Jayaprakasha, 2003). The edible part of the pomegranate fruit (50%) consists of 40% arils and 10% seeds. Arils contain 85%

water (Viuda-Martos et al., 2011). The by-product of pomegranate juicing is normally called pomegranate Marc and contains 78% peel and 22% seeds and are normally disposed in the field (Qu, Pan, & Ma, 2010). Pomegranate peel is also used as a source for producing natural colorant (Adeel et al., 2014; Ajmal et al., 2014; Gulzar et al., 2015; Kulkarni, Gokhale, Bodake, & Pathade, 2011).

Many studies have shown that pomegranate peel is a good source of phenolic compounds (Al-Rawahi et al., 2014; Cam & Hisil, 2010; Fischer, Carle, & Kammerer, 2011; Guo et al., 2003; Negi, Jayaprakasha, & Jena, 2003). Extraction of phenolic compounds from plants can be performed using different kinds of methods. The conventional methods are distillation and solvent extraction (He et al., 2012). Also, some new and advanced methods such as pressurized water extraction (Cam & Hisil, 2010), ultrasound assisted extraction (Pan, Qu, Ma, Atungulu, & McHugh, 2011) and microwave assisted extraction (Garofulic, Dragovic-Uzelac, Jambrak, & Jukic, 2013; Zheng, Liu, Li, & Zhu, 2011) have been applied. Application of new extraction methods increase the efficiency of extraction and on the other hand, decrease the extraction time (Boussetta, Lanoisellé, Bedel-Cloutour, & Vorobiev, 2009). Natural structure of plants resists against solvent diffusion extraction and the process is very slow. To intensify extraction, initial structure can be modified, for instant, by grinding or cutting (Amor & Allaf, 2009). In general, the solid-liquid extraction includes rapid extraction of free accessible solutes from open cells which is mainly occurs at the surface,

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followed by slow extraction by diffusion mechanism from closed and un-ruptured cells which occurs inside the solid particles. From engineering point of view, it is desirable to intensify the last slow step by reducing mass transfer resistance. Some recent studies have shown that instant controlled pressure drop (ICPD) expands and breaks the structure of cells and make the tissue more porous. This technology was defined by Allaf and Vidal (Allaf & Vidal, 1988; Allaf & Allaf, 2014). It helps to achieve more efficient extraction process by changing the cellular structure of the plants. Using ICPD and at the first stage, samples are subjected to an initial vacuum, after that high pressure saturated steam is injected for a short period of time and then, it ended with a fast pressure drop toward vacuum (Rezzoug, Baghdadi, Louka, Boutekedjiret, & Allaf, 1998). ICPD is a thermo-mechanical process. Connecting the extraction vessel to a vacuum tank increases the benefits using higher pressure difference in between the processing vessel and vacuum tank. This generates an auto-vaporization of volatile molecules, accompanying with rapid cooling and expansion of the solid texture. In this manner, solvent extraction can be implemented more efficient in a shorter time and consuming less solvent (Allaf et al., 2014). ICPD has been used in various applications like swell drying of fruits and vegetables with improvement of drying kinetic (Albitar, Mounir, Besombes, & Allaf, 2011; Maritza, Sabah, Anaberta, Montejano-Gaitán, & Allaf, 2012), extraction of essential oils by instant autovaporization with improved availability of some compounds and yield of extraction (Berka-Zougali, Hassani, Besombes, & Allaf, 2010; Besombes, Berka-Zougali, & Allaf, 2010; Rezzoug, Boutekedjiret, & Allaf, 2005) and extraction of non-volatile molecules from plants. Regarding non-volatile compounds, the ICPD texturing process has shown good results in terms of improving the yield of extraction. Amor and Allaf (2009) used this method before solvent extraction of anthocyanin from Malaysian Roselle and their results proved that ICPD could increase both the kinetic and yield. The effective diffusivities were found about $4.62 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ for ICPD treated samples and $4.19 \times 10^{-11} \text{ m}^2 \text{ s}^{-1}$ for untreated materials. ICPD was applied before solvent extraction of total phenols from green coffee beans (Kamal & Allaf, 2013; Kamal, Sobolik, Kristiawan, Mounir, & Allaf, 2008). In this way, the yield increased from 7.8% for raw materials to 15.82% for ICPD treated beans.

The objective of this study was to investigate the effect of texturing by ICPD treatment on total phenolic compounds extraction from pomegranate peel. Response surface methodology (RSM) was used to optimize ICPD parameters.

2. Materials and methods

2.1. Chemicals

Methanol was purchased from Rankem (New Delhi, India). Folin-Ciocalteu reagent, sodium carbonate and gallic acid powder were supplied from Merck (Germany). 2,2-Diphenyl-1-picrylhydrazyl (DPPH) was purchased from Sigma Chemical Co. (USA) and ethanol was from Chem-Lab NV (Belgium).

2.2. Raw materials

Pomegranate (*Punica granatum* L.) was supplied from Yazd (Yazd province, I.R. Iran). The peel were separated manually and cut to about 1 cm^2 parts. To determine moisture content, 3 g of peel was dried in an oven (Teb Azma, Iran) for 8 h at $105 \text{ }^\circ\text{C}$. The measurements were triplicate. The moisture content of the raw material was $35.08 \pm 0.71\%$ (db, dry basis).

2.3. UV analysis

UV analyses for determination of the absorbance of samples were performed using a Perkin-Elmer spectrophotometer (Lambda 25, USA).

2.4. Scanning electron microscope (SEM)

Micro-structures were observed by a Mira II (Tescan SEM, Czech Rep.) of Iranian Research Organization for Science and Technology (IROST). The samples were coated with a thin layer of gold and were scanned with acceleration tension of 7 kV.

2.5. ICPD texturing equipment

A laboratory-scale ICPD apparatus (Iranian Research Organization for Science and Technology, IROST) was designed and fabricated, Fig. 1. It consisted of an insulated 2.5 L SS304 processing vessel and a 10 L SS 304 jacketed vacuum tank connected to a water vacuum pump. There are three manual valves for injection of steam (6), instant pressure drop in between the vacuum tank and processing vessel (7), and the last one for vacuum tank drain (8). Saturated steam was supplied by a mini-steam generator (SilTer, 3.5 L, Turkey). The ICPD experiments were implemented in 5 steps as follows:

1. Initial vacuum in processing vessel which contains pomegranate peel. It gives rise to removing air from the vessel.
2. Injection of high pressure saturated steam (up to 3 bar) into that vessel,
3. Abrupt pressure drop toward the vacuum tank,
4. Repeating steps (b) and (c) in multi-cycles mode,
5. Releasing to atmospheric pressure.

All parts of the apparatus which were in contact with processing materials were made from stainless steel.

2.6. Experimental design

RSM (Minitab, ver. 17) was used for the experimental design and optimization of ICPD parameters. The goal was to maximize total phenols and antioxidant extraction from pomegranate peel. In this work, ICPD experiments were carried out considering three factors including

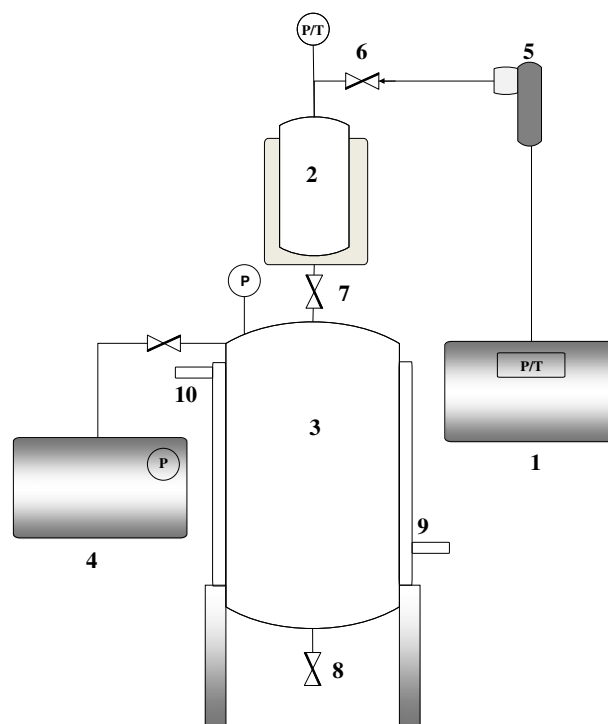


Fig. 1. Schematic diagram of ICPD apparatus: 1. Steam generator, 2. Processing vessel, 3. Vacuum tank, 4. Vacuum pump, 5. Steam injection valve, 6. Handy valve, 7. Pressure drop valve, 8. Drain, 9. Water inlet, 10. Water outlet.

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