



## Analytical Methods

# Continuous extraction of phenolic compounds from pomegranate peel using high voltage electrical discharge



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## ABSTRACT

Pomegranate peel, a waste generated from fruit processing industry, is a potential source of phenolic compounds that are known for their anti-oxidative properties. In this study, a continuous high voltage electrical discharge (HVED) extraction system was for the first time designed and optimized for phenolic compounds from pomegranate peel. The optimal conditions for HVED were: flow rate of materials 12 mL/min, electrodes gap distance 3.1 mm (corresponding to 29 kV/cm of electric field intensity) and liquid to solid ratio 35 mL/g. Under these conditions, the experimental yield of phenolic compounds was  $196.7 \pm 6.4$  mg/g, which closely agreed with the predicted value (199.83 mg/g). Compared with the warm water maceration, HVED method possessed higher efficiency for the extraction of phenolic compounds. The results demonstrated that HVED technique could be a very effective method for continuous extraction of natural compounds.

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## 1. Introduction

Pomegranate (*Punica granatum* L., Fig. 1a), a member of Punicaceae family, is one of the fruits containing important bioactive phenolic ingredients and has been widely used as botanical ingredients in herbal medicines and dietary supplements (Song, Li, & Li, 2016). Pomegranate fruit is rich in many nutritional and bioactive compounds, comprising organic acids, minerals (such as potassium), vitamins (C, A, and K), folic acid, etc (Akhtar, Ismail, Fraternal, & Sestili, 2015). However, the most valuable aspect of pomegranate is its phenolic compound contents, including hydrolysable tannins (punicalagins and punicalins), condensed tannins (proanthocyanidins), anthocyanins, catechins, phenolic acids (gallic, ellagic and chlorogenic), and so on (Qu, Breksa, Pan, & Ma, 2012). Phenolic compounds, which widely exist in peel, pulp and seed of pomegranate, are very beneficial to health and known for the possession of the remarkable antioxidant properties capable on protecting normal cells from various stimuli-induced oxidative stress and cell death (Nawaz, Shi, Mittal, & Kakuda, 2006). The peel (Fig. 1b) has higher content of phenolic ingredients than the pulp and seed, and can be a good source for producing high-value antioxidants (Shaban, El-Kersh, El-Rashidy, & Habashy, 2013). Pomegranate is increasingly consumed as various processed products, such as juice, wine, jam, jelly and extract, whose consumption

has been motivated by its health benefits derived from its high antioxidant capacity (Abid et al., 2017). In pomegranate juice processing, 1 ton of fresh fruit generates 669 kg by-product pomegranate pomace containing 78% peel and 22% seed (Qu et al., 2009). Large amounts of pomegranate peel are mostly used as animal feed or discarded as useless residue, which is not only an environmental pollution but also a waste of the large raw materials (Shabtay et al., 2008). Therefore, in order to efficiently utilize pomegranate, it is necessary for us to optimize extraction of phenolic compounds from the pomegranate peel.

Several studies have been published to extract phenolic compounds from pomegranate peel with various extraction methods (Kazemi, Karim, Mirhosseini, & Hamid, 2016; Masci et al., 2016; Mushtaq, Sultana, Anwar, Adnan, & Rizvi, 2015; Pan, Qu, Ma, Atungulu, & McHugh, 2012; Ranjbar, Eikani, Javanmard, & Golmohammad, 2016; Sood & Gupta, 2015; Tabaraki, Heidarizadi, & Benvidi, 2012; Çam & Hışıl, 2010). The conventional methods are distillation and organic solvent extraction, which not only are tedious and time consuming but also exert negative impact on environment in terms of emission of organic volatile compounds (Pan et al., 2012). Also, some new and promising techniques such as pressurized water extraction (Çam & Hışıl, 2010), instant controlled pressure drop (Ranjbar et al., 2016), supercritical fluid extraction (Mushtaq et al., 2015), and ultrasound assisted extraction (Kazemi et al., 2016) have been introduced and shown high extraction efficiency, low energy and solvent consumptions. However, little attentions have been presented on the extraction of

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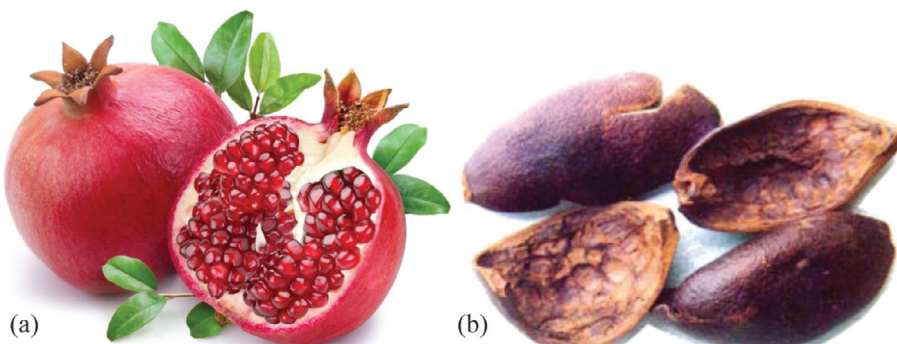


Fig. 1. Pomegranate (a) and its dried peel (b).

phenolic compounds from pomegranate peel using high voltage electrical discharge (HVED).

HVED is a non-thermal technique for the enhancement of mass transfer of natural ingredients in liquid at ambient temperatures and shorter extraction times by applying pulsed rapid discharge voltages (usually from 20 to 80 kV/cm electric field intensity) through an electrode gap below the surface of aqueous suspensions of natural materials, which is based on the phenomenon of electrical breakdown in liquid (Boussetta, Lebovka et al., 2009; Boussetta et al., 2011; Boussetta, Vorobiev, Reess et al., 2012; Boussetta, Turk et al., 2013; Boussetta, Lanoisellé, Bedel-Cloutour, & Vorobiev, 2009; Boussetta, Lesaint, & Vorobiev, 2013; Boussetta, Vorobiev, Le, Cordin-Falcimaigne, & Lanoisellé, 2012; Boussetta & Vorobiev, 2014; Brianceau, Turk, Vitrac, & Vorobiev, 2016; Liu, Vorobiev, Savoie, & Lanoisellé, 2011; Parniakov, Barba, Grimi, Lebovka, & Vorobiev, 2014; Rajha, Boussetta, Louka, Maroun, & Vorobiev, 2014; Sarkis et al., 2015). Electrical breakdown is the result of an avalanche of electrons turns to a starting point of streamer propagation, from the high voltage needle electrode to the grounded one, which leads to the liquid turbulence and intense mixing, the emission of high-intensity UV light, the generation of hydrogen peroxide ( $H_2O_2$ ), the production of intense shock waves and bubble cavitation, which provoke cell structure damage and particle fragmentation, enhancing the release of intracellular components (Brianceau et al., 2016; Rajha et al., 2014). The HVED has been successfully employed for the recovery of bioactive ingredients from different plant materials including grape by-products (Boussetta, Lebovka et al., 2009; Boussetta et al., 2011; Boussetta, Vorobiev, Reess et al., 2012; Boussetta, Lesaint et al., 2013; Boussetta, Lanoisellé et al., 2009; Boussetta, Vorobiev, Le et al., 2012; Brianceau, Turk, Vitrac, & Vorobiev, 2016; Liu, Vorobiev, Savoie, & Lanoisellé, 2011), flaxseed cake (Boussetta et al., 2013), vine shoots (Rajha et al., 2014), sesame seeds (Sarkis et al., 2015), papaya peels (Parniakov et al., 2014), and so on. These researches show that HVED can achieve less processing time, higher extraction yield, lower power consumption, less extract impurities and no harm to the activity and structure of biological active ingredient (Brianceau et al., 2016; Parniakov et al., 2014; Rajha et al., 2014; Sarkis et al., 2015). However, these investigations have been conducted using batch treatment chambers of low capacity in intermittent mode. Industries application of HVED technique requires evaluating this technique with continuous treatment chambers that allow the extraction process in continuous conditions. Response surface methodology (RSM) is a collection of statistical techniques useful for developing, improving, and optimizing complex processes, the main advantage of which was to reduce number of experimental trials needed to evaluate multiple variables and their interactions (Eren & Kaymak-Ertekin, 2007). Thus, the objective of this work is to develop a continuous

HVED system for extraction of phenolic compounds from pomegranate peel and to optimize the extraction process using RSM.

## 2. Materials and methods

### 2.1. Plant materials and reagents

Pomegranate fruits were purchased from local markets in Chengdu, China. Pomegranate fruits were washed with distilled water, and then manually peeled and their edible portions were carefully separated. The peels were dried in a hot air oven at 40 °C for 48 h, grounded to a fine powder using a grinder machine (KC-1000, Beijing Kaichuangtonghe Technology Development Co., Ltd, Beijing, China) and passed through a 100 mesh sieve, then packaged in polyethylene bags and store in refrigerator at 4 °C until used.

Ethanol used in the experimental work was analytical reagent grade chemicals (Beijing Chemical Reagents Company, Beijing, China). DPPH free radical was purchased from Sigma Chemical Co. (St Louis, USA). Gallic acid, Folin-Ciocalteu's phenol reagent, and  $Na_2CO_3$  were purchased from the Sinopharm Chemical Reagent Co. (Beijing, China). Other reagents were of analytical grade and purchased from Chengdu Chemical Industry (Chengdu, China). All solutions were prepared with analytical chemicals and deionized water was used throughout.

The UV-Vis spectrophotometer (751-GW) was from Shanghai Analytical Instrument Overall Factory (Shanghai, China).

### 2.2. Design of the continuous HVED extraction system

The HVED extraction system (Fig. 2a) was composed of a high-voltage pulse generator, a continuous treatment chamber (Fig. 2b), a data acquisition systems, a fluid handling and cooling system, and voltage and current measuring devices.

The high-voltage pulse generator (TP3020) was obtained from Dalian Teslaman Technology Co., Ltd. (Dalian, China). This pulse generator offered exponentially decaying bipolar triangle waveform pulses with 2  $\mu$ s pulse duration and frequency up to 1000 Hz, which provided 20 kV–10 kA discharges for a few microseconds.

The “converged electric field type” treatment chamber described by Alkhafaji and Farid was used (Fig. 2b), which provided higher electric field intensity in a small volume without increasing the voltage at the electrodes (Alkhafaji & Farid, 2007). The chamber contained a pair of parallel disc mesh electrodes made of stainless steel and an insulating plate with a small hole between the electrodes to create a small orifice (i.e., treatment region) where electric field intensity was highly concentrated. The electric field

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