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Optimization of extraction of phenolic acids from a vegetable waste product using a pressurized liquid extractor

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ARTICLE INFO

Article history:
Received 20 September 2011
Received in revised form
1 June 2012
Accepted 4 June 2012
Available online 20 July 2012

Keywords:
Potato (*Solanum tuberosum* L.)
Phenolic acids
Pressurized liquid extractor
HPLC analysis
Extraction condition optimization

ABSTRACT

This study identified the phenolic acids in potato peels, tuber, and developed an optimized method for extraction of phenolic acids from potato peels using a pressurized liquid extractor (PLE). Caffeoylquinic acid isomers were the predominant phenolic acids present in potato peels as identified by HPLC and LC–MS analysis. The phenolic acids extracted from potato peels were over tenfold higher than that of the tubers. A variety of PLE parameters were investigated to optimize the extraction conditions. The results indicated that the optimum yields of phenolic acids from the potato peels were extracted with methanol/water (90:10, v/v) at 160 °C with a solid-to-solvent ratio of 250 mg with 20 mL of solvent using a pressurized liquid extractor. Although static time variations (5–15 min), pressure (500–2000 psi), and flush volume (10–100%) variations did not significantly improve the extraction yields of phenolic acids, optimization of the above three parameters will result in significant reduction of the resources used and waste generated, thereby impacting the overall operation cost. The results presented in this manuscript suggest that optimization of extraction parameters is critical for accurate quantification of phytochemicals present in foods and other plant based products.

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1. Introduction

Potato (*Solanum tuberosum* L.) serves as a primary food source worldwide, offering a rich source of nutrients and energy to its consumers. Potato is the world's fourth largest crop, according to the total mass of generated fresh produce (http://www.newworldencyclopedia.org/entry/Potato, April 30, 2012). It is the USA's leading vegetable crop with a per capita consumption of approximately 60 kg (National Potato Council, 2005; Reddivari, Hale, & Miller, 2007). Potatoes, like other plant derived foods, are known to accumulate a variety of nutrients and phytochemicals, including carbohydrates, proteins, minerals, phenolic acids, anthocyanins, glycoalkaloids, phytoalexins, and vitamins (Casanas, Gonzalez, Rodriguez, Marrero, & Diaz, 2002; Kolasa, 1993; Leo et al., 2008).

There has been significant interest in phenolic phytochemicals during the past few decades as numerous studies suggest that the consumption of diets rich in fruits and vegetables may play a role in reducing the risk of cardiovascular and neurodegenerative diseases and certain forms of cancers (Criqui & Ringel, 1994; Maxwell, 1995). The health benefits of fruits and vegetables have been partially ascribed to the presence of phenolic phytochemicals.

The interest in phenolic phytochemicals has resulted in the publication of a number of research articles related to the analysis and composition of potato polyphenols (Ahmed, Akter, & Eun, 2011; Del Mar Verde Mendez, Rodriguez Delgado, Rodriguez, & Diaz Romero, 2004). Chlorogenic acid is the predominant phenolic acid present in potatoes. Several isomeric forms of chlorogenic acids, 3-*O*-caffeoylquinic (*n*-chlorogenic

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1756-4646/$ - see front matter Published by Elsevier Ltd.
http://dx.doi.org/10.1016/j.jff.2012.06.001

acid), 4-caffeoylquinic (*crypto*-chlorogenic acid), and 5-*O*-caffeoylquinic (*neo*-chlorogenic acid), have been identified from potatoes (Ieri, Innocenti, Andrenelli, Vecchio, & Mulinacci, 2011; Leo et al., 2008).

As wide range of extraction solvents, techniques, and conditions have been described in peer-reviewed published literature, it is challenging and difficult to identify a single best optimized procedure for extracting all phenolic acids (Stalikas, 2007; Luthria, 2006; Luthria, Mukhopadhyay, & Kwansa, 2006). This is critical for accurate quantification and evaluation of health beneficial properties of bioactive phytochemicals in edible food parts and peels that are often discarded as waste. In this study, we have evaluated influence of different extraction parameters such as solvent composition, extraction time, particle size, flush volume, temperature, pressure, and solid-to-solvent ratio using a pressurized liquid extractor to optimize extraction of phenolic acids from potato peels. This will allow researchers to accurately quantify the amount of phenolic acid present in potato peels and precisely evaluate the health beneficial properties of f phenolic acids present in potato peels.

2. Materials and methods

2.1. Sample preparation

Potatoes were washed with warm water and air-dried. The potato skins were then peeled (~1–2 mm) and stored in a −70 °C freezer overnight. The frozen potato peels were freeze-dried (Labconco freeze-dryer, Kansas City, MO, USA) and ground into a powder using a coffee grinder (Mr. Coffee Grinder, Sunbeam Products, Inc., Hattiesburg, MS, USA). The powdered potato peels sample was sieved through standard mesh size sieves and five different particle size fractions (fraction 1: >2.0 mm, fraction 2: 2.0 mm and >0.85 μm, fraction 3: <0.85 μm and >0.425 μm, fraction 4: <0.425 μm and >0.25 μm, and fraction 5: <0.25 μm) were collected. All ground samples were stored under an inert nitrogen atmosphere at −70 °C prior to extraction and analysis to minimize oxidation of phenolics in the powdered potato peel samples.

2.2. Chemicals

HPLC grade methanol, ethanol, and 20–30 mesh Ottowa sand were purchased from Fisher Chemicals (Fair Lawn, NJ, USA). De-ionized water (18 MΩ cm) was prepared with a Millipore Milli-Q purification system (Millipore Corp., New Bedford, MA, USA). HPLC grade acetone was purchased from EMD Chemicals (Gibbstown, NJ, USA). Compressed nitrogen was bought from Airgas Inc. (Randor, PA, USA).

2.3. Extraction procedure

All extractions were conducted using an accelerated solvent extractor (ASE, Model ASE 200, Dionex Corporation, Sunnyvale, CA, USA). ASE extraction cells were prepared by first placing two circular cellulose filters (size 1.983 mm, Dionex Corporation, Sunnyvale, CA, USA) at the bottom of each 11 mL stainless steel cell (Dionex Corporation Sunnyvale, CA, USA) to prevent fine suspended particles from entering the solvent lines and collection vials. Approximately, 2–3 g of 20–30 mesh Ottawa sand was poured into the cell followed by 200 ± 1 mg of the ground potato peels sample. Void volume of the cell was filled with Ottawa sand and topped with one circular cellulose filter to prevent sand particles from entering the solvent injection needle. Both extraction cells and collection vials were properly arranged in the two designated carousels of the instrument. The extracts were collected in 60 mL amber collection vials fitted with Teflon coated rubber caps (I-CHEM Brand Products, New Castle, DE USA).

PLE extractions were optimized by altering the following parameters: temperature, particle size, pressure, static time, solid-to-solvent ratio, flush volume, and solvent composition. A preliminary study was conducted to determine the optimal solvent composition that would yield the highest extraction yield of phenolic acids from potato peels. Extractions were initially carried out with the purified, methanol and ethanol solvents. Additional extractions were performed with several different proportions of methanol:water, mixtures. The optimal solvent mixture was used in the optimization of the other PLE extraction parameters. The default conditions for extraction were: <25 μm particle size, 40 °C, 1000 psi, 5 min static time, 200 mg sample, and 75% flush volume. One parameter or variable was altered at a time. Resulting extracts were each transferred to a 25 mL volumetric flask and diluted to 25 mL with the appropriate solvent. The extracts were filtered through a 0.45 μm PVDF syringe filter (National Scientific Company, Duluth GA, USA) prior to assay for phenolics by HPLC analysis.

2.4. HPLC and LC–MS analysis

All extracts were analyzed on a HPLC-DAD system (High Performance Liquid Chromatography, Beckman Coulter, System Gold, Series 166 Detector, Series 508 Autosampler, Fullerton, CA, USA) with 32 Karat operation software (Beckman Coulter Fullerton, CA, USA). Extraction samples of 20 μL were injected. A reversed phase C_{18} Luna column (150.0 × 4.6 mm; particle size 5 μm, Phenomenex, Torrence, CA, USA), preceded by a guard column (4.0 × 3.0 mm, Phenomenex, Torrence, CA, USA) of the matching stationary phase was used. The column and guard column were thermostatically controlled at 40 °C and the flow rate was set to 0.5 mL/min. The mobile phase consisted of two solvents: acetonitrile with 0.1% formic acid (labeled A) and water with 0.1% formic acid (labeled B.) The solvent gradient in volumetric ratios of solvents A to B were 0 to 30 min, 10% A to 30% A; 30 to 50 min, 30% A to 60% A; 50 to 55 min, 60% A to 100% A; 65 to 70 min, 100% A to 10% A; 75 min 10% A. Dual wavelengths (270 and 350 nm) were used and HPLC chromatograms were detected with the use of a photodiode array UV detector. HPLC area of the identified phenolic acids was used for comparison of extraction efficiency of phenolic acids from potato peels. The mass spectral analysis was carried out using a Agilent 1100 LC system coupled with a diode array and MSD (SL) detectors (Agilent, Palo Alto, CA, USA) using electrospray ionization in the positive and negative ionization (PI and NI) modes at low (70 V) and high (250 V) fragmentation voltages as described in our earlier communication (Lin & Harnly, 2007; Lin & Harnly, 2008).

2.5. Statistical analysis

All statistical analyses were conducted using PROC GLM in SAS® v 9.1.2 (SAS Institute, Inc., Cary, NC, USA) software. For each extraction procedure, a one-way ANOVA was conducted with subsequent means comparisons to identify differences among extraction methods. All extractions and analyses were either carried out in triplicate.

3. Results and discussion

3.1. Analysis of phenolic acids

Table 1 shows that different researchers have used various methods for extracting phenolic acids from potato samples. The methodology and the conditions used effects the amount of phenolic compounds extracted and quantified from potato

Table 1 – A brief summary of extraction methods used by different researcher for extraction of phenolics from potato samples.

Sr. No.	Method	Extraction solvent	Extraction technique	Reference
1	10 g of fresh tissue pooled from tuber halves	85% aqueous MeOH	Omni mixer	Ramamurthy, Maiti, Thomas, and Madusudanan (1992)
2	2 g fresh (0.1 g freeze-dried) homogenized in 7 mL of MeOH with 2 g/L BHA and 10% acetic acid	MeOH, BHA, 10% acetic acid (85:15, v/v)	Heidolph diax 900 homogenizor	Mattila and Hellstrom (2007)
3	1 g freeze-dried powder extracted at room temp. under stirring, twice with 30 mL of 70% EtOH adjusted to pH 2.0 by HCOOH(1 h total time)	70% EtOH acidic	Stirring	Ieri et al. (2011)
4	Tissue ground in Liquid N, alkaline hydrolysis after MeOH extraction. Antioxidant 2,6-ditertbutyl-B cresol during alkaline hydrolysis. N_2 bubbled through sample after NaOH addition	MeOH	Alkaline hydrolysis	Cvikrova, Eder, Sukhova, and Korableva (1995)
5	1 g defatted flour extracted 6 times in Polytron	70% MeOH/70% acetone (1:1, v/v), diethyl ether:ethyl acetate (1:1, v/v)	Homogenize	Sosulski, Krygier, and Hogge (1982)
6	200 mg freeze-dried powder with 50% MeOH, 2.5% meta phosphoric acid, 1 mM EDTA. 500 mg glass beads, concentrated in a speed vac	50% MeOH, 2.5% metaphosphoric acid, 1 mM EDTA	BeadBeater	Shakya and Javarre (2006)
7	1 g of tuber peel macerated with mortar and pestle then suspension in 80% EtOH. Subject to charcoal treatment and residue re extracted twice with 80% EtOH	80% EtOH	Ultrasonication Centrifuge	Singhai, Sarma, and Srivastava (2011)
8	0.5 g sample per cell, 30 °C, and adjusted volume 20 mL	65% aqueous MeOH, 0.1% formic acid	ASE 200	Soltoft et al. (2010)
9	0.1 g sample extracted three times with 20 mL of 75% MeOH and filtered. Concentrated in a rotary vacuum evaporator. Samples were diluted	75%MeOH	Not defined	Ahmed et al. (2011)
10	Undamaged tubers of 20–80 g mass. For analysis 5 tubers were used, every study was done 3 times	MeOH, 3% metaphosphoric acid	Method cited in cross references.	Hejtmankova, Pivec, Trnkova, Hamouz, and Lachman (2009)
11	Roots were chopped in food processor then ground in 15 mL boiling 80% ethanol. Centrifuged, extracted 2 more times, adjusted volumes then filtered	80% EtOH	Centrifuge Tekmar Tissuemizer	Troung et al. (2007)
12	150 mg of dry tuber epidermis extracted with 1 mL of methanol/0.1% HCl solution in an ultrasonic bath for 15 min. After centrifuge, supernatant was filtered and dried. Polyphenols were suspended in 1 mL of water	MeOH/0.1%HCl solution	Ultrasonication Centrifuge	Lukaszewicz et al. (2004)

samples. In order to accurately evaluate the health beneficial properties of phenolic compounds present in foods and its waste products (peels) it is vital to optimize extraction procedures to accurately quantify the amount of phenolic compounds in different food matrices. Peels that are often discarded as waste may provide a good source of bioactive phytochemicals.

Fig. 1 shows a typical chromatogram of the phenolic acids extracted from potato peel extracts. The major peak (Peak # 3) eluting at retention time 23.1 min showed UV spectra with absorption bands at 324, 294sh, 238 and 218 nm. The three other major peaks (1, 2, and 4) also showed similar UV spectral pattern, thus indicating that they may be isomeric or related compounds possessing a similar chromophore. The mass spectral data of the major peak (# 3) in the negative ion mode showed a molecular ion $[M-H]^-$ at *m/z* 353 and fragment ions at mass *m/z* 179. Comparison of mass and UV spectral data with the previously published literature indicated that major peak (# 3) was caffeyolquinic acids. The two additional peaks (2 and 4) were also identified as isomeric caffeyolquinic acids (Lin & Harnly, 2008; Friedman, 1997) as they showed similar UV and mass spectral data. Based on the previously published literature data on the order of elution of three isomeric caffeyolquinic acids on four different columns, the compound eluting first (Peak 2, retention time 16.4 min) was tentatively identified as 3-caffeoylquinic acid. The most abundant isomer eluting at retention time 23.2 min (Peak 3) was determined as 5-caffeoylquinic acid, and the compound eluting at (Rt 25.2 min, peak 4) was assigned as 4-caffeoylquinic acid (Lin & Harnly, 2008; Friedman, 1997). The compound eluting at retention time 9.2 min (Peak 1) also showed similar UV absorption spectrum as that of caffeic acid and a fragment ion at mass *m/z* 179 in a negative ion mode (equivalent to the mass of deprotonated caffeic acid). Thus the unidentified peak 1was tentatively suggested to be a derivative of caffeic acid.

In recent studies by Im et al. (2008), chlorogenic acid and caffeic acid were identified in potato peels. Chlorogenic acid and its isomer contributed 96–98% of the total phenolic acids extracted from potato peel samples. Similarly in the study by Al-Weshahy and Rao (2009), the major phenolic acid was identified as chlorogenic acid. In other recent publication by Singh and Sladana, chlorogenic acid was the major phenolic acid extracted with methanol. However, when extractions were carried out with subcritical water as 160–180 °C at 6 MPa for 60 min, chlorogenic acid and gallic acid were two major phenolic acids extracted and identified. The same authors also reported trace quantities of five additional phenolic acids namely, gallic acid, protocatechuic acid, syringic acid, ferulic acid, and coumaric acid. These differences in phenolic acid composition in different potato samples may be attributed to variation in cultivars, growing conditions, and extraction solvents and techniques used by different researchers (Luthria, 2006; Naczk & Shahidi, 2004). Furthermore, the focus of the current research was to determine conditions for optimum extraction of phenolic acids from foods and its waste product (peels).

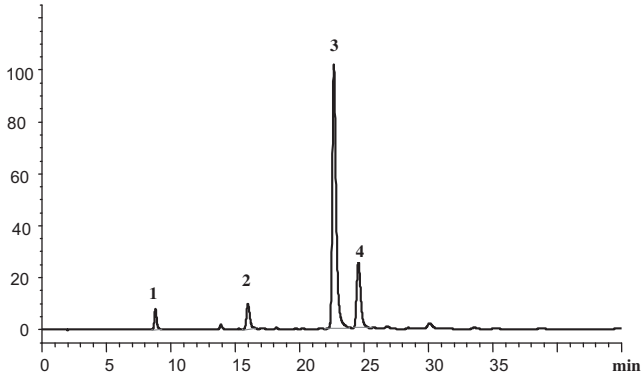

Fig. 1 – A typical HPLC chromatogram of phenolic acids extracted from potato peels. Peaks 2, 3, and 4, were identified as 3-caffeoylquinic acid, 5-caffeoylquinic acid, and 4-caffeoylquinic acid.

For calculation of extraction efficiency, combined areas of three most abundant identified caffeoylquinic acid isomers (peaks 2, 3, and 4) were used as the HPLC profile did not change significantly with varying extraction conditions. The extraction condition that yielded optimum recoveries of three combined caffeoylquinic acid isomers was assigned a value of 100% for each set of experiment. This value was then used to determine the relative percent extraction efficiency with other extraction conditions within the same set.

3.2. *Extraction solvent composition*

The structural diversity, interaction with other cellular components, and existence of multiple aglycon and conjugated forms of phenolic phytochemicals, present a significant challenge in optimizing a method for extraction of phenolic acids from any food matrix (Antolovich, Prenzler, Robards, & Ryan, 2000; Escarpa & Gonzalez, 2001; Luthria, 2006; Naczk & Shahidi, 2004). As depicted in Table 1, a wide range of aqueous alcohols (methanol and ethanol) solvent mixtures have been used by different researchers for extraction of phenolic acids from potato tuber and peel samples. Addition of water to organic solvents is known to cause the plant material to swell thereby allowing the organic solvent to penetrate more easily into the sample matrix which in turns results in increased extraction efficiency of phytochemicals. In the present study, an initial comparison with methanol and ethanol solvent showed that methanol had a better extraction efficiency than ethanol. Therefore a systematic comparison of different aqueous methanol (20, 40, 60, 80, 90, and 100%) solvent mixtures on extraction efficiency of phenolic acids was carried out. The results indicated that the extraction efficiency of phenolic acids in different methanol:water mixtures varied with proportion of methanol in aqueous alcohol mixtures (Fig. 2A). Optimal extraction efficiency of total phenolic acids was obtained when extractions were performed with methanol/water mixtures (90:10, v/v). Very similar extractions yields (99.3%) were obtained when extractions were performed with methanol/water (80:20, v/v). The percent relative extraction efficiency was calculated using methanol/water (90:10, v/v) as 100%. Only 91.1% of the total phenolic acids were recovered with 100% methanol. The extraction efficiency of the total phenolic acids decreased by 18.2% when extractions were carried out with 60:40 (v/v) methanol/water. Extraction

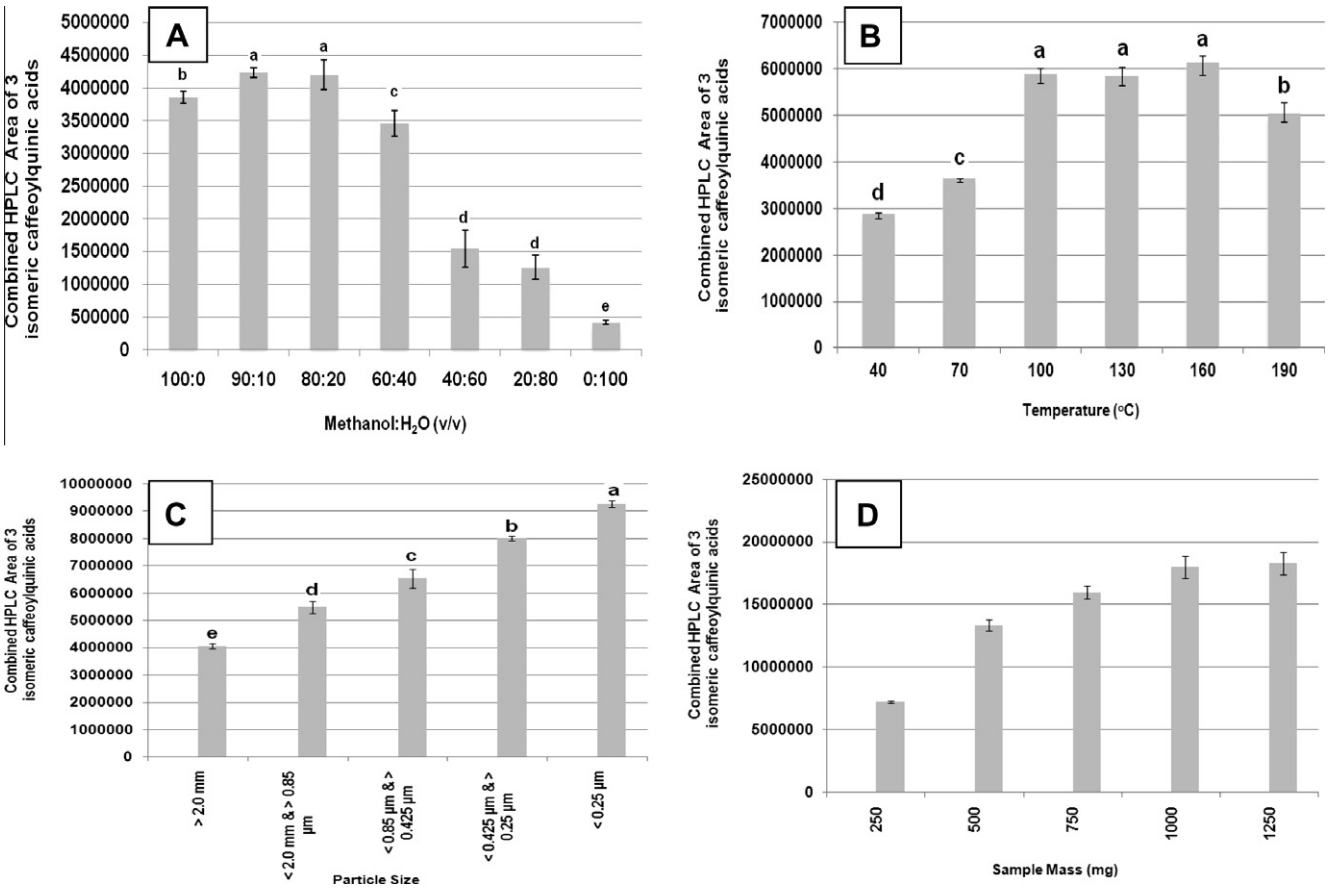

Fig. 2 – Effect of different extraction parameters on assay of phenolic acids from extracted from potato peels. (A) Extraction solvent composition, (B) temperature, (C) particle size, and (D) solid-to-solvent ratio. Column marked by different letters are significantly different from each other ($P \leqslant 0.05$) and columns marked with same letter are not statistically different.

efficiencies further decreased by 64.5, 70.3, and 89.9%, respectively, when extractions were carried out with either 40% methanol, 20% methanol and 100% water, respectively. Similar results in extraction of phenolic acids were observed when chlorogenic acid was extracted from different cultivars of eggplants (Luthria & Mukhopadhyay, 2006). This was slightly different from the black cohosh samples where 60:40 (v/v) methanol/water resulted in increased efficiency of total phenolic acids. The difference in the extraction yield can be attributed to variation in the type of phenolic acids extracted from the plant. Ferulic, isoferulic, sinapic, and caffeic acids were extracted from black cohosh (Mukhopadhyay, Luthria, & Robbins, 2005) as compared to caffeoylquinic acids isomers extracted from potato peels and eggplant samples (Luthria & Mukhopadhyay, 2006).

3.3. *Extraction temperature*

As shown in Table 1, a wide range of temperatures (ambient to reflux temperatures) were used for extraction of phenolic acids from potatoes in previously reported studies by different researchers. In the present study, a systematic investigation of the effect of temperature on the extraction efficiency of phenolic acids from potato peels was carried out as it is known that temperature impacts both the equilibrium (solubility) and the mass transfer rate (diffusion coefficient) (Mukhopadhyay et al., 2005). Extractions were carried out between temperature ranges of 40–190 °C with systematic increments of 30 °C (Fig. 2B). Higher yields of total phenolic acids were obtained when extractions were carried out between 100 and 160 °C. Optimum extraction yields were obtained at 160 °C which were marginally higher (<5%) than the yields obtained at 100 and 130 °C. Further increase of temperature to 190 °C resulted in a decrease (18.0%) in extraction yield of phenolic acids which may be due to partial degradation of phenolic acids at higher extraction temperature. The yields of total phenolic acids decreased significantly by 53.2 and 40.8%, respectively, at lower extraction temperatures of 40 and 70 °C. Thus indicating the benefits of extracting phenolic acids at higher temperature (100–160 °C) under an inert nitrogen atmosphere by using pressurized liquid extractor. Such high temperature extractions are not easily possible with classical extraction methods.

3.4. *Particle size*

The influence of particle size on extraction efficiency of phenolic acids from potato peels has not been studied previously. To evaluate the influence of particle size on extraction efficiency of total phenolic acids, dried ground skins were sieved

through different sized multiple stacked sieves. The sieves were vigorously shaken manually for 10 min. Five different particle size fractions (fraction 1: >2.0 mm, fraction 2: <2.0 mm and >0.85 μm, fraction 3: <0.85 μm and >0.425 μm, fraction 4: <0.425 μm and >0.25 μm, and fraction 5: <0.25 μm) were collected. Each particle size fraction was separately extracted under identical extraction conditions and phenolic acid extracts were assayed by HPLC analysis. The results

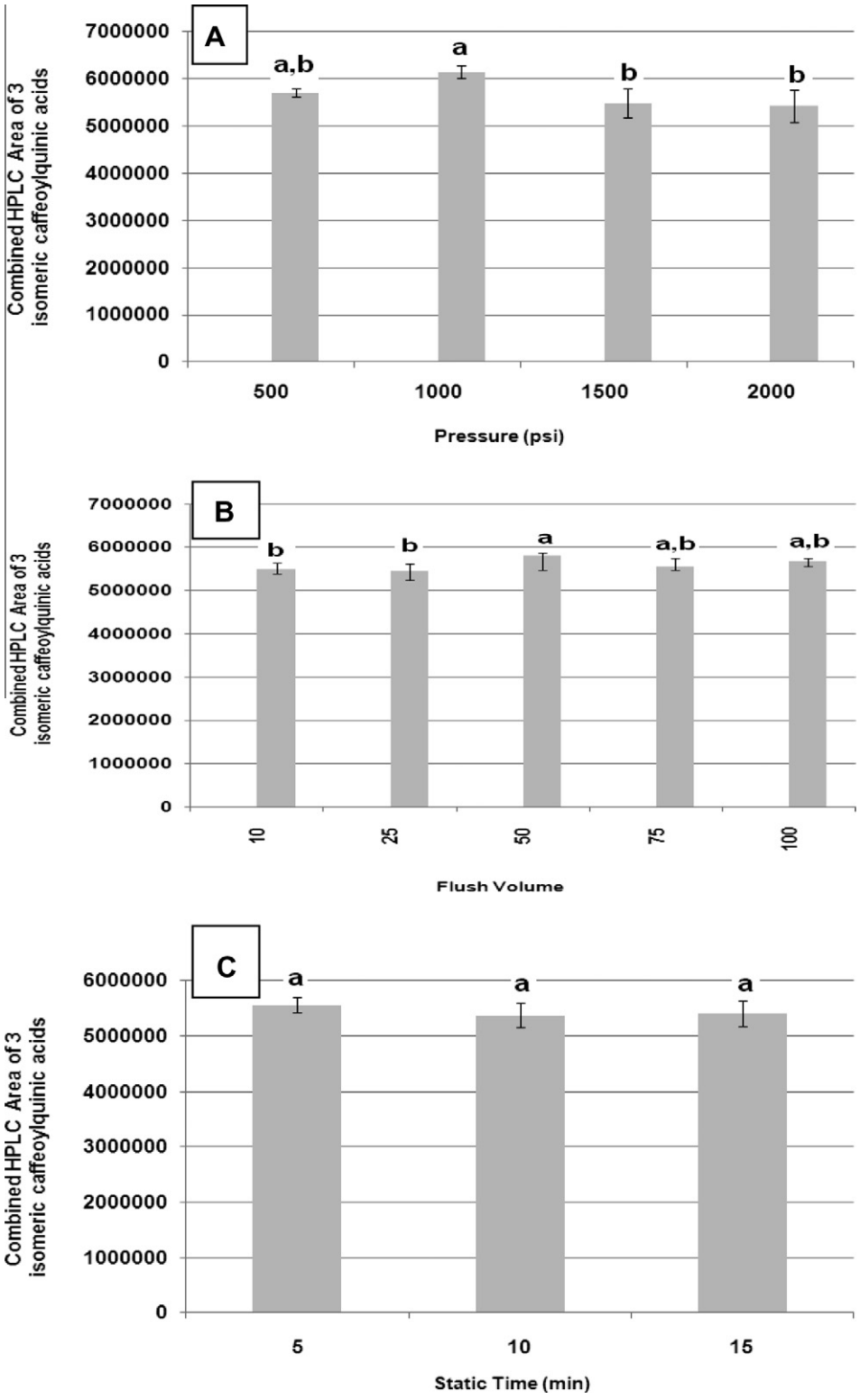

Fig. 3 – Effect of different extraction parameters on assay of phenolic acids from extracted from potato peels. (A) Pressure, (B) flush volume, and (C) static time. Column marked by different letters are significantly different from each other ($P \leqslant 0.05$) and columns marked with same letter are not statistically different.

indicated that particle size significantly influenced the extraction yields of phenolic acids (Fig. 2C). This is primarily due to the well know fact that particle size directly affects solvent–solute interactions thereby influencing extraction efficiency. It is known that the surface area per unit mass increases with decrease in particle size, so optimum extraction was obtained with the smallest particle size (<0.25 μm). Only 43.8% of the phenolic acids were extracted when the particle size was >2 mm. The extraction efficiency varied between 59.3 and 86.5% with other intermediate size particles of potato peels. Similar results of increased extraction efficiency of phenolic yields were obtained with decreased particle size in our earlier studies (Luthria & Mukhopadhyay, 2006; Mukhopadhyay et al., 2005).

3.5. *Solid-to-solvent ratio*

As documented in Table 1, a wide spectrum of solid-to-solvent ratios were used by different researchers for the extraction of phenolics from potato extracts. In the current study, a systematic evaluation of the influence of solid-to-solvent ratio on the extraction efficiency of phenolic acids was investigated by carrying out extraction of different quantity of potato peels samples (250, 500, 750, 1000 and 1250 mg) with same amount of extraction solvent as all extractions were performed in a standard 11 mL stainless steel extraction cells. As shown in Fig. 2D, the amount of phenolic acids extracted from potato peels increased as the sample amount increased from 250 to 1250 mg. However, increase in the amount of phenolic acids was not proportionate to the sample quantity. Almost twice the amounts of phenolic acids were extracted when the sample size was increased from 250 to 500 mg. However, when sample amount was further increased to 750 mg, the phenolic acid extract yield was 26.0% less than the optimal extraction yield with 250 mg sample mass. The proportion of extraction of phenolic acids extracted from the 1000 and 1250 mg samples were further reduced significantly to 62.4 and 50.7%, respectively. The reduction in extraction efficiency is due to poor solid-to-solvent interaction possibly due to caking of sample which decreases the solubility of phenolic acids in extraction solvent. The above results indicate the need for optimization of solid-to-solvent ratio for the efficient extraction of phytochemicals from foods and food products for accurate quantification. The optimized solid-to-solvent ratio was 250 mg of powdered peel with 20 mL of extraction solvent. In addition, one can also reduce the amount of extraction solvent used per extraction which directly determines the quantity of waste solvent generated during extraction particularly at commercial scale.

3.6. *Pressure, flush volume, and static time*

Flush volume, static time, and pressure, are parameters specific to accelerated solvent extractors and can be optimized to increase extraction efficiency of analytes or possibly reduce the quantity of consumables. Variation of all the three parameters did not significantly change the efficiency of extraction of phenolic acids from potato peels (Fig. 3A–C). The high pressure allows solvents to be in a liquid state at temperatures above the boiling point of the solvent and also increases the sample to solvent interactions. In the present study, pressure was systematically changed from 500 to 2000 psi with 500 psi increments. The results showed that 1000 psi was optimum for the efficient extraction of phenolic acids. The extraction efficiency at other three pressures (500, 1500, and 2000 psi) did not had major effects on extraction efficiency of phenolic acids from potato peels as extraction yields varied between 88.3 and 92.7% (Fig. 3A). Similar results were obtained with the extraction of polyphenols from parsley flakes at three different pressures in our previous study (Luthria, 2008).

The flush volume determines the quantity of solvent used per extraction cycle. This parameter is specific to the ASE 200. In the present study, the flush volume was systematically changed from 10 to 100%. The efficiency of extraction of phenolic acids from potato peels varied marginally (between 94.3 and 100%) when the flush volume was changed from 10 to 100%. Optimum extraction yields were obtained with the 50% flush volume setting (Fig. 3B). However the quantity of solvent used during extraction was significantly changed with different flush volume settings. With the 10% flush volume setting the quantity of solvent used was ~7 mL and with the 100% flush volume setting the amount of solvent used was more than doubled. Optimization of this parameter will be of significant value when extractions are performed with either expensive and/or toxic solvents.

Static time, also referred to as extraction time, is the amount of time the sample interacts with the solvent per extraction cycle. The total extraction time with a static time setting of 5 min with three cycles will be ~20 min. However, with a 15 min static time setting, the total extraction time for 3 cycles will be ~50 min. Thus, optimization of this parameter will assist in significantly improving the sample throughput using ASE. In addition, operation cost will also be reduced significantly as lesser amount of energy and gas will be utilized. In the current study, extractions were performed with three different static times 5, 10, and 15 min, respectively. The results revealed the increase of static time from 5 min to 15 min, the efficiency of extraction of phenolic acids from potato peel samples varied less than 5.0% (Fig. 3C).

4. Conclusions

The results indicated that the optimum yields of phenolic acids from the potato peels were extracted with methanol/water (90:10, v/v) at 160 °C with a solid-to-solvent ratio of 250 mg with 20 mL of solvent using a pressurized liquid extractor. Although static time variations (5–15 min), pressure (500–2000 psi), and flush volume (10–100%) variations did not significantly improve the extraction yields of phenolic acids, optimization of the above three parameters will result in significant reduction of the resources used and waste generated, thereby impacting the overall operation cost. The process for optimization of an extraction procedure for phenolic acids will not be same for all food matrices. It will be dependent on the type of analytes, its chemical form (aglycon, glycosylated, acetylated, methylated, etc.), the goal of the research project, and on interaction of phytochemicals with the sample matrix. This research illustrates that optimization of sample preparation plays a vital role in accurate quantification of phenolic acids from potato peels.

Acknowledgments

The author will like to thank Mr. Rishi Banerji for carrying out experimentation and Ms. Zurana Taluckder and Ms. Allison Setia for their help in preparation of this manuscript. I will also like to thank Dr. Craig Byrdwell for his help in setting up the LC–MS and Mr. Bruce Richter from Dionex Corporation for providing supplies for ASE extractions. This research was supported by the Agricultural Research Service of the U.S. Department of Agriculture.

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