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Optimisation of biologically active compounds ultrasound assisted extraction from potatoes using response surface methodology

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Abstract. Potato (*Solanum tuberosum* L.) is source of phenolic compounds and from plant matrixes can be extracted by several methods. In recent years ultrasound assisted extraction has become more popular due to its efficiency for recovery of phenolic compounds and antioxidants and response surface methodology is an effective tool for optimisation of extraction procedure by evaluating different variables and their interaction. The aim of the current research was to optimize ultrasound assisted extraction of biologically active compounds from potatoes by response surface methodology. For experiment purple-flesh potato variety 'Blue Congo' was selected. Control sample was extracted by stirring for 1 hour. Box-Behnken design was used for optimization of extraction conditions from fresh potatoes and as variables were selected: ethanol concentration (% v/v), hydrochloric acid concentration (molarity) and time (min). For extracts as responses total phenolic, total flavonoid, total anthocyanin content and antioxidant activity (DPPH, ABTS+ scavenging activity) were determined using a spectrophotometric methods. Significant models were obtained for antocyanins, total phenols and DPPH radical scavenging activity. Optimisation of extraction showed that for maximising all responses optimal HCl concentration is 2.5M, ethanol concentration 79.4% and extraction time 60 minutes, resulting in following responses: 57.41 mg 100 g^{-1} of anthocyanins, 238.52 mg 100 g^{-1} of TPC, 24.58 mM TE 100 g^{-1} of DPPH scavenging activity and 12.99 mM TE 100 g^{-1} of ABTS scavenging activity. Conventional extraction method showed significantly lower results. It could be concluded that ultrasound assisted extraction is effective method for recovery of phenolic compounds and solvents and extraction time is significant parameter influencing efficiency.

Key words: purple-flesh potatoes, phenolics, ultrasound assisted extraction, response surface methodology, Box-Behnken design.

INTRODUCTION

Potato (*Solanum tuberosum* L.) is widely grown and consumed crop and it contains many vitally important elements that can benefit human diet (Leo et al., 2008).

Significant and valuable nutrient group in potatoes are phenolics which are secondary metabolites with health promoting effect (Velioglu et al., 1998; Espin et al., 2000; Manach et al., 2004). Potatoes are so important source of phenolics that they range

as a third consumed crop right after apples and oranges which are good source of phenolics as well (Chun et al., 2005).

Phenolics show antioxidant, anticarcinogenic, antibacterial, anti-inflammatory, antiglycemic, antiviral and vasodilatory qualities (Duthie et al., 2000; Cai et al., 2004; Reyes et al., 2005; Tsao & Deng, 2005; Mattila & Hellstrom, 2006; Leo et al., 2008; Berghe, 2012; Kazeem et al., 2012; Konaté et al., 2012; Lolayekar & Shanbhag, 2012). They also present a positive impact on human longevity, ocular organs, mental health as well as cardiovascular system (Parr & Bolwell, 2000; Manach et al., 2004; Scalbert et al., 2005), and phenolics usage in human diet protects from degenerative diseases (Pourcel et al., 2007; Im et al., 2008). Studies show that in terms of safety as natural antioxidant in form of extract phenolics are not mutagenic (Sotillo et al., 2007).

Analysis of potatoes have shown significant correlation between existence of phenolics and total antioxidant level lighting up the fact that higher phenolics amount bring higher antioxidant levels (Andre et al., 2007).

While all potatoes contain phenolics, the amounts differ in varieties, i.e., red and purple flesh potatoes might contain approximately twice as much total phenolics compared to white flesh potatoes (Ezekiel et al., 2013). It may be explained with high amount of anthocyanins that are pigments in those varieties (Im et al., 2008; Al-Weshahy & Rao, 2009). Preparation process for different coloured potatoes for consumption purposes also affects level of phenolics in different ways (Reyes & Zevallos, 2003; Brown et al., 2005). The phenolics appear in the whole tuber in potatoes, still the skin has highest level of phenolics (Lewis et al., 1999; Nara et al., 2006).

Phenolics extraction optimisation is important to reach most accurate analysis, therefore response surface methodology can be used as an effective tool for this purpose. It is used as an alternative to classical optimization methods, and is more time saving, cheaper and helps in data evaluation process (Myers et al., 2004; Amado et al., 2014). This methodology may help to evaluate the effect of the variables and their interactions (Wettasinghe & Shahidi, 1999; Farris & Piergiovanni, 2009; Asfaram et al., 2015). Methanol and ethanol are one of the most widely used solvents for phenolics extraction from potatoes (Singh & Rajini, 2004; Mohdaly et al., 2010; Amado et al., 2014). Conventional way of phytochemical extraction includes maceration and Soxhlet extraction, and these methods have quite high organic solvent consumption which limits bioactive extract usage range because of solvent toxicity, as well as time required for extraction is long and consumed energy in the process is high (Da Porto et al., 2012). The alternative and modern method, called ultrasound assisted extraction, has recently gained more and more popularity (Bendicho et al., 2012) which shortens the time and energy spent on the process and it also limits final costs. This technology is sustainable as it protects the environment and consumers health as well as saves time and money (Armenta et al., 2015).

The aim of the current study was to optimize ultrasound assisted extraction of biologically active compounds from potatoes by response surface methodology.

MATERIALS AND METHODS

Plant material

Purple-flesh potato variety 'Blue Congo', grown at the test fields of Institute of Agricultural Resources and Economics, was selected for the experiment. Harvested tubers were kept in the storage facility at 4 °C and at 80 ± 5% relative air humidity until analysis. Potatoes were homogenized before extraction experiment.

Extraction of phenolic compounds

The homogenized potato samples (2.0 g) were extracted with 20 mL solvent (according to optimisation model described in experimental design) in an ultrasonic bath YJ5120-1 (Oubo Dental, USA) at 35 kHz for certain time (according to optimisation model described below).

Extraction solvent was acidified ethanol (ethanol: HCl solution 85:15 (v/v)). Ethanol concentration and molarity of HCl were varied in the experiment. The extracts were then centrifuged in a centrifuge CM-6MT (Elmi Ltd., Latvia) at 3,500.00 rpm for 5 min.

For comparison, extraction methodology used in previous studies was tested (Kampuse et al., 2016). Phenolic compounds extraction from potatoes - the homogenized samples were extracted with ethanol (80/20 w/w) in a conical flask with a magnetic stirrer (magnet 4.0 cm × 0.5 cm) at 700 rpm for 1 h at room temperature (20 ± 1 °C). The extracts were then filtered (paper No.89).

Experimental design

A response surface methodology using Box-Behnken design (Design Expert) was used for optimization of extraction conditions of anthocyanins, total phenols and antioxidants from fresh potatoes and variables were selected as follows: ethanol concentration (% v/v), hydrochloric acid concentration (molarity) and time (min) (Table 1).

Table 1. Independent variables, their levels and responses

Run order	A HCl, M	B Ethanol, %	C Time, min	Anthocyanin, mg $100g^{-1}$	TPC, mg $100g^{-1}$	DPPH, mM TE $100g^{-1}$	ABTS, mM TE $100g^{-1}$
1	0.5	50	40	29.55	218.36	13.46	20.96
2	2.5	50	40	44.76	218.99	20.99	14.01
3	0.5	90	40	28.35	189.24	14.64	14.79
4	2.5	90	40	34.95	175.70	24.92	17.76
5	0.5	70	20	42.38	258.84	15.04	20.26
6	2.5	70	20	49.75	243.06	22.16	13.97
7	0.5	70	60	42.73	263.07	14.97	21.01
8	2.5	70	60	58.27	256.88	22.52	13.70
9	1.5	50	20	54.76	243.06	15.31	20.06
10	1.5	90	20	32.78	205.01	20.69	25.02
11	1.5	50	60	31.16	178.84	18.56	18.20
12	1.5	90	60	44.08	204.09	22.15	12.88
13	1.5	70	40	29.77	248.22	18.70	15.89
14	1.5	70	40	29.97	248.68	18.76	15.36
15	1.5	70	40	30.70	248.22	18.95	16.13

Experiment was performed by 15 runs with three replicates of central run. Estimation of error was performed by 3 runs of central points. Optimisation process was based on evaluation of responses of samples designed according to model. Coefficients of response function was calculated, predicting response of fitting model. Statistical significance was examined by analysis of variance (Anova), lack of fit, pure error, adeq.precision was tested to check models adequacy.

Optimisation was made by both numerical and graphic analysis using contour curves and desirability functions. In current experiment different maximum response variable values were obtained.

Analytical methods

The total phenolic content (TPC) of the potato extracts was determined according to the Folin-Ciocalteu spectrophotometric method (Singleton et al., 1999). The absorbance was measured at 765 nm and total phenols were expressed as the gallic acid equivalents (GAE) 100 g^{-1} dry weight (DW) of plant material. The TFC was measured by a spectrophotometric method (Kim et al., 2003).

The absorbance was measured at 415 nm and total flavonoids were expressed as catehin equivalents (CE) 100 g^{-1} DW of the sample. Antioxidant activity of the plant extracts was measured on the basis of scavenging activities of the stable 2,2-diphenyl-1-picrylhydraziyl (DPPH$^{\cdot}$) radical as outlined by Yu et al. (2003) and 2,2′-azino-bis(3-ethylbenz-thiazoline-6-sulfonic) acid (ABTS$^{\cdot+}$) radical cation assay (Re et al., 1999). Antioxidant activity was expressed as TE 100 g^{-1} DW of plant material. Total anthocyanins were determined by method described by Mane et al. (2015). The pH shift method (pH 0.6 and pH 3.5) for determination of anthocyanins in potatoes extracts were used based on the absorbance at 700 nm and 520 nm. Results were calculated as pigment cyanindin-3-glucoside equivalents and expressed to dry matter.

RESULTS AND DISCUSSION

The response surface methodology using Box-Behnken is widely used for optimisation of extraction (Espada-Bellido et al., 2017). Extraction variables were HCl molarity (A), ethanol concentration (B) and time (C). Results of analysis of variance for the quadratic model of responses are shown in Table 2 for total anthocyanins, in Table 3 for total phenols, in Table 4 for DPPH radical scavenging activity and Table 5 for ABTS scavenging activity. Models for response were significant for anthocyanins ($P = 0.0310$), total phenols ($P = 0.0231$), DPPH radical scavenging activity ($P = 0.0018$) but not for ABTS scavenging activity ($P = 0.1274$). Coefficient of variation ranged between 5.2 until 8.4.

The second order polynomial equation express relationship between tested factors their interaction and anthocyanins fitted model is:

$$\text{Anthocyanins} = 30.15 + 3.54A - 0.46B - 0.43C + 1.95AB + 2.04AC + 8.72BC + 3.87A^2 - 3.72B^2 + 14.72C^2$$

Current equation could be used for prediction of responses in the range of tested factors.

Table 2. Analysis of variance for the quadratic model of extracted anthocyanins

Source	Sum of squares	Degree of freedom	Mean square	*F* value	*P*-value	Coefficient estimate
Model	1,313.42	9	145.94	6.03	0.0310	30.15
A-HCl	100.14	1	100.14	4.14	0.0975	3.54
B-Ethanol	1.67	1	1.67	0.0691	0.8031	-0.4572
C-Time	1.48	1	1.48	0.0612	0.8144	-0.4301
AB	15.25	1	15.25	0.6308	0.4631	1.95
AC	16.66	1	16.66	0.6891	0.4443	2.04
BC	304.41	1	304.41	12.59	0.0164	8.72
A^2	55.30	1	55.30	2.29	0.1909	3.87
B^2	51.08	1	51.08	2.11	0.2059	-3.72
C^2	751.36	1	751.36	31.07	0.0026	14.27
Residual	120.91	5	24.18			
Lack of Fit	120.43	3	40.14	165.84	0.0060	
Pure Error	0.4841	2	0.2421			
Cor. Total	1,434.33	14				
Adeq. precision	7.24					

Based of coefficient estimate showed at Table 3 the second order polynomial equation express relationship between tested factors their interaction and total phenol fitted model is:

$TPC = 248.37 - 8.08A - 6.93B - 5.89C + 3.90AB + 2.40AC + 15.82BC - 3.76A^2 - 51.48B^2 + 10.85C^2$

Table 3. Analysis of variance for the quadratic model of extracted TPC

Source	Sum of squares	Degree of freedom	Mean square	*F* value	*P*-value	Coefficient estimate
Model	1,2870.42	9	1,430.05	6.94	0.0231	248.37
A-HCl	521.91	1	521.91	2.53	0.1724	-8.08
B-Ethanol	384.48	1	384.48	1.87	0.2302	-6.93
C-Time	277.24	1	277.24	1.35	0.2985	-5.89
AB	60.75	1	60.75	0.2948	0.6105	3.90
AC	23.00	1	23.00	0.1116	0.7519	2.40
BC	1,001.56	1	1,001.56	4.86	0.0786	15.82
A^2	52.25	1	52.25	0.2536	0.6360	-3.76
B^2	9,783.96	1	9,783.96	47.48	0.0010	-51.48
C^2	434.75	1	434.75	2.11	0.2061	10.85
Residual	1,030.35	5	206.07			
Lack of Fit	1,030.20	3	343.40	4,845.27	0.0002	
Pure Error	0.1417	2	0.0709			
Cor. Total	1,3900.76	14				
Adeq. precision	7.6617					

Also for DPPH antioxidant activity the second order polynomial equation expresing relationship between tested factors their interaction fitted model is:

$DPPH = 18.80 + 3.27A + 2.55B + 0.63C + 2.28AB + 0.11AC - 0.44BC - 1.20A^2 - 0.69B^2 + 1.07C^2$

For ABTS antioxidant activity developed model was not significant it was not used for optimisation procedure.

Table 4. Analysis of variance for the quadratic model of extracted DPPH

Source	Sum of squares	Degree of freedom	Mean square	*F* value	*P*-value	Coefficient estimate
Model	174.15	9	19.35	21.34	0.0018	18.80
A-HCl	85.31	1	85.31	94.08	0.0002	3.27
B-Ethanol	52.16	1	52.16	57.52	0.0006	2.55
C-Time	3.13	1	3.13	3.45	0.1224	0.6254
AB	20.70	1	20.70	22.83	0.0050	2.28
AC	0.0467	1	0.0467	0.0515	0.8295	0.1080
BC	0.7916	1	0.7916	0.8730	0.3930	-0.4449
A^2	5.29	1	5.29	5.84	0.0604	-1.20
B^2	1.77	1	1.77	1.96	0.2209	-0.6929
C^2	4.19	1	4.19	4.62	0.0842	1.07
Residual	4.53	5	0.9068			
Lack of Fit	4.50	3	1.50	87.16	0.0114	
Pure Error	0.0344	2	0.0172			
Cor. Total	178.69	14				
Adeq. precision	14.9679					

Table 5. Analysis of variance for the quadratic model of extracted ABTS

Source	Sum of squares	Degree of freedom	Mean square	*F* value	*P*-value	
Model	107.27	6	17.88	2.38	0.1274	No
A-HCl	35.26	1	35.26	4.70	0.0621	significant
B-Ethanol	1.59	1	1.59	0.2116	0.6578	
C-Time	22.87	1	22.87	3.05	0.1191	
AB	20.90	1	20.90	2.78	0.1338	
AC	0.2625	1	0.2625	0.0350	0.8563	
BC	26.40	1	26.40	3.52	0.0976	
Residual	60.07	8	7.51			
Lack of Fit	59.76	6	9.96	65.11	0.0152	
Pure Error	0.3060	2	0.1530			
Cor. Total	167.35	14				
Adeq. precision	5.0943					

Results are visualized in nine response surface graphs which provide visual representation of the relationship between responses and levels of each variable and the type of interactions between two test variables in each case. Circular or elliptical form of the contour plots show significance level of the interactions between the variables (Fig.1).

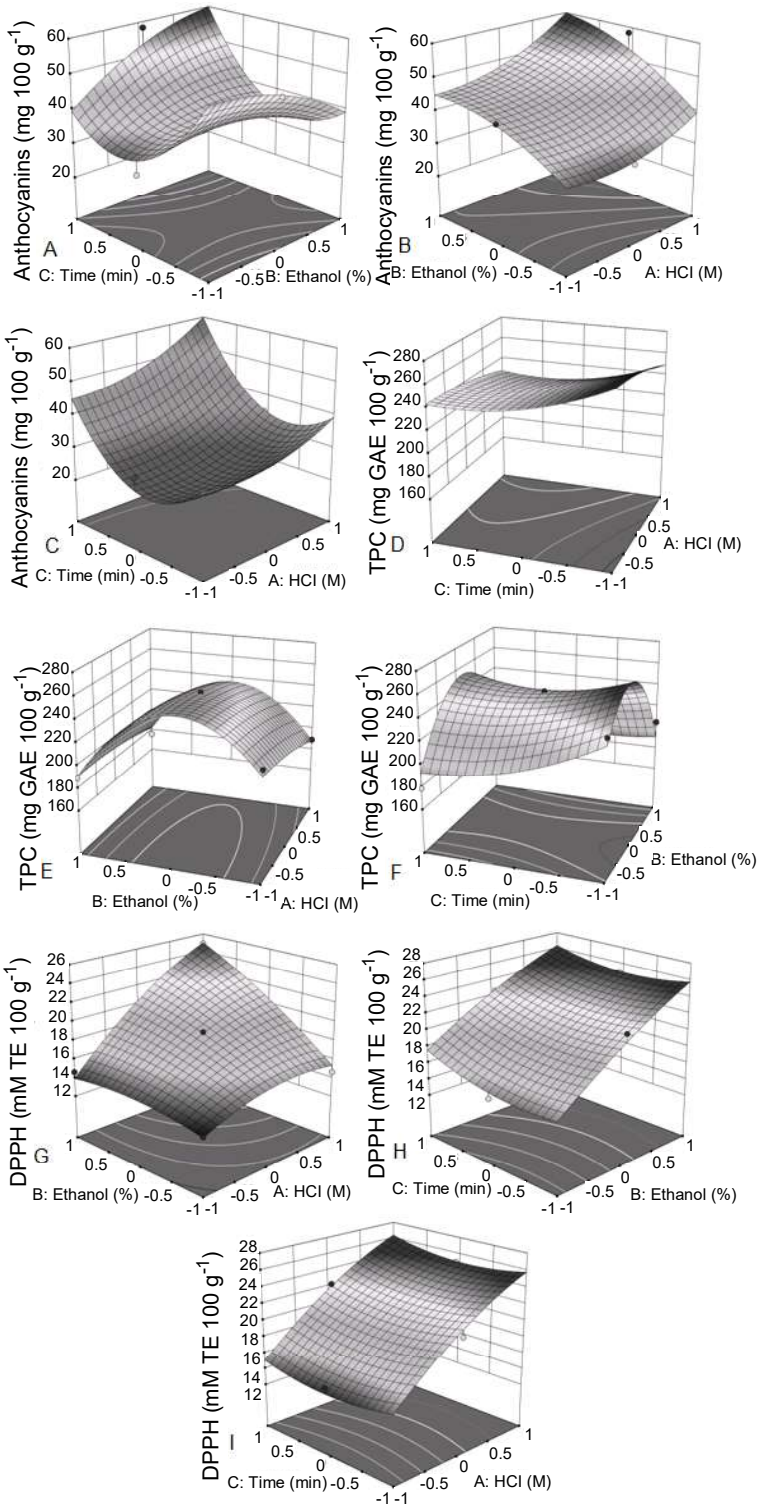

Figure 1. Response surface 3D plots (A (Anthocyanins), F (TPC), H (DPPH)) presenting the effect of extraction time and extraction ethanol concentration; (B (Anthocyanins), E (TPC), G (DPPH)) extraction ethanol and HCI concentration; (C (Anthocyanins), D (TPC), I (DPPH)) extraction time and HCI concentration.

For extraction of anthocyanins the best conditions are with higher concentration of ethanol and longer extraction time (Fig. 1, A), and also with higher concentration of HCl and ethanol (Fig. 1, B). Analysis of purple sweet potatoes showed that significant factors influencing anthocyanins extractability are temperature, ethanol concentration and ultrasound power, but time is not significant that is opposite to results obtained in current experiment (Cai et al., 2016). It is confirmed that anthocyanins are not stable in alkaline and neutral environment, and for improvement of their stability and extractability hydrochloric acid addition is beneficial to reduce pH up to 2 til 2.3 (He et al., 2016). Anthocyanins extraction yield and composition of sweet potatoes are also dependent on extraction method and ultrasound assisted extraction could result in higher impurities of other phenolics (Cai et al., 2016). In current experiment it is not disadvantage because coextraction is tested.

Analysing tendencies for extraction of TPC, higher values wereobtained extracting shorter time, and with lower concentration of HCl as solvent (Fig. 1, D), ethanol concentration and HCl interaction showed that higher values were obtained by medium ethanol concentration and the result was not dependent on HCl concentration (Fig. 1, E). For extraction of phenolics from potatoe peels, use of acidified ethanol resulted in pure and stable extracts avoiding side reactions (Maldonado et al., 2014). Extraction of phenolic compounds from eggplant showed that acidified solvent gave better yield, and it decreased by the increase of pH (Ferarsa et al., 2018). Whereas analysing interaction of time and ethanol, higher results were obtained by medium concentration of ethanol and shorter time (Fig. 1, F). Extraction efficiency of phenolics from eggplants increased by increasing water content in ethanol up to 50% (Ferarsa et al., 2018). It could be explained by polarity of solvents and addition of water to organic solvent enhances extract separation efficiency (Ferarsa et al., 2018). Comparing the two extracts (in water and ethanol), water was less effective than ethanol for the extraction of phenolic compounds. This difference can be explained by the polarity of both solvents. It can also be seen that the yields of aqueous ethanol extracts (50 and 75%) are higher than those of pure ethanol (100%) and pure water (0%). These results indicate that adding water to the organic solvent enhances extraction yield (Ferarsa et al., 2018). For DPPH activity the maximal values analysing ethanol and HCl concentration was obtained using highest concentrations tested (Fig. 1, G), higher activity was observed with higher concentration of HCl (Fig.1.H), and ethanol (Fig. 1, I), and in both cases time was not significant factor. Ultrasound assisted extraction of sweet potatoes resulted in samples with high radical scavenging activity (Cai et al., 2016).

Box Behnken methodology gives possibility to analyse interaction of different factors for obtaining desirable results. In current experiment possibility to make one extraction for testing all responses were tested. If all responses were maximised then optimal parameters for extraction were following: HCl concentration 2.5M, ethanol concentration 79.4% and extraction time 60 minutes, and as a result can be obtained 57.41 mg 100 g^{-1} of anthocyanins, 238.52 mg 100 g^{-1} of TPC, 24.58 mM TE 100 g^{-1} of DPPH scavenging activity and 12.99 mM TE 100 g^{-1} of ABTS scavenging activity. It is also possible to optimise one parameter and if content of anthocyanins is maximised then optimal HCl concentration is 2.42M, ethanol concentration 88.8% and extraction time 59.8 min, as a result can be obtained 58.31 mg 100 g^{-1} of anthocyanins, but results of other parameters are lower – 210.57 mg 100 g^{-1} of TPC, 25.91 mM TE 100 g^{-1} of DPPH scavenging activity and 12.69 mM TE 100 g^{-1} of ABTS scavenging activity. For

extraction of antocyanins from mulberries were 76% MeOH in water at pH 3 (Espada-Bellido et al., 2017) that is according to our results that higher concentration of HCl gives better results. Whereas for extraction of anthocyanins from purple sweet potatoes maximal yield was obtained at 50 °C, 45 minutes using 90% (v/v) ethanol as the solvent (Cai et al., 2016).

For maximal TPC extraction the optimal HCl concentration is 0.82M, ethanol concentration is 68.2% and extraction time is 27.2 min, as a result 37.87 mg 100 g^{-1} of anthocyanins, 263.58 mg 100 g^{-1} of TPC, 15.52 mM TE 100 g^{-1} of DPPH scavenging activity and 20.03 mM TE 100 g^{-1} of ABTS scavenging activity can be obtained. The optimum conditions for extraction of total phenolic compounds from berries were 61% MeOH in water at pH 7 (Espada-Bellido et al., 2017), that is also in accordance with our results showing that lower concentration of acid gives higher TPC.

To maximise DPPH antioxidant activity, the optimal HCl concentration would be 2.46M, ethanol concentration 89.2% and extraction time 48.4 minutes, as a result 41.92 mg 100 g^{-1} of anthocyanins, 192.90 mg 100 g^{-1} of TPC, 25.10 mM TE 100 g^{-1} of DPPH scavenging activity and 15.17 mM TE 100 g^{-1} of ABTS scavenging activity can be obtained.

For comparison of results, conventional extraction by stirring for one hour was tested and results obtained were significantly lower, namely, TPC 200.56 mg 100g^{-1}, DPPH scavenging activity 6.27 mM TE 100g^{-1}, ABTS 12.77 mM TE 100g^{-1}. Obtained results are in accordance with results reported in literature showing efficiency of ultrasound for extraction for anthocyanins (Mane et al., 2015; Espada-Bellido et al., 2017) and phenolic compounds (Espada-Bellido et al., 2017).

CONCLUSIONS

In current experiment coextraction of total phenols, anthocyanins and antioxidants characterised by antiradical activity were tested. Significant models were obtained for antocyanins, total phenols and DPPH radical scavenging activity. Optimisation of extraction showed that for maximising all responses, the optimal HCl concentration is 2.5M, ethanol concentration 79.4% and extraction time 60 minutes, resulting in following responses: 57.41 mg 100 g^{-1} of anthocyanins, 238.52 mg 100 g^{-1} of TPC, 24.58 mM TE 100 g^{-1} of DPPH scavenging activity and 12.99 mM TE 100 g^{-1} of ABTS scavenging activity. Results of conventional extraction showed significantly lower results. If one of the responses are maximised, content of others reduces significantly. For extraction of anthocyanins, the optimal HCl concentration is 2.42M, ethanol concentration is 88.8% and extraction time is 59.8 minutes, whereas for TPC optimal HCl concentration is 0.82M, ethanol concentration is 68.2% and extraction time is 27.2 minutes. For obtaining extracts with higher DPPH activity optimal HCl concentration is 2.46M, ethanol concentration is 89.2% and extraction time is 48.4 minutes. It could be concluded that ultrasound assisted extraction is effective method for recovery of phenolic compounds and solvents and extraction time is significant parameter influencing efficiency.

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REFERENCES

Al-Weshahy, A. & Rao, V.A. 2009. Isolation and characterization of functional components from peel samples of six potatoes varieties growing in Ontario. *Food Research International* **42**(8), 1062–1066.

Amado, I.R, Franco, D, Sánchez, M. & Vázquez, J.A. 2014. Optimisation of antioxidant extraction from Solanum tuberosum potato peel waste by surface response methodology. *Food Chemistry* **165**, 290–299.

Andre, C.M., Ghislain, M., Bertin, P., Oufir, M., Herrera, M.R., Hoffmann, L., Hausman, J.F., Larondelle, Y. & Evers, D. 2007. Andean potato cultivars (*Solanum tuberosum* L.) as a source of antioxidant and mineral micronutrients. *Journal of Agricultural and Food Chemistry* **55**(2), 366–378.

Armenta, S., Garrigues, S. & De Guardia, M. 2015. The role of green extraction techniques in Green Analytical Chemistry. *Trends in Analytical Chemistry* **71**, 2–8.

Asfaram, A., Ghaedi, M., Goudarzi, A., Soylak, M. & Langroodi, S.M. 2015. Magnetic nanoparticle based dispersive micro-solid-phase extraction for the determination of malachite green in water samples: optimized experimental design. *New Journal of Chemistry* **39**(12), 9813–9823.

Bendicho, C., De La Calle, I., Pena, F., Costas, M., Cabaleiro, N. & Lavilla, I. 2012. Ultrasound-assisted pretreatment of solid samples in the context of green analytical chemistry. *TrAC Trends in Analytical Chemistry* **31**(8), 50–60.

Berghe, W.V. 2012. Epigenetic Impact of Dietary Polyphenols in Cancer Chemoprevention: Lifelong Remodeling of Our Epigenomes. *Pharmacological Research* **65**(6), 565–576.

Brown, C.R., Wrolstad, R., Durst, R., Yang, C.P. & Clevidence, B. 2005. Breeding studies in potatoes containing high concentrations of anthocyanins. *American Journal of Potato Research* **80**(4), 241–249.

Cai, Y., Luo, Q., Sun, M. & Corke, H. 2004. Antioxidant activity and phenolic compounds of 112 traditional Chinese medicinal plants associated with anticancer. *Life Sciences* **74**(17), 2157–2184.

Cai, Z., Qu, Z., Lan, Y., Zhao, S., Ma, Z., Wan, Q., Jing, P. & Li, P. 2016. Conventional, ultrasound-assisted, and accelerated-solvent extractions of anthocyanins from purple sweet potatoes. *Food Chemistry* **197**(A), 266–272.

Chun, O.K., Kim, D.O., Smith, N., Schroeder, D., Han, J.T. & Lee, C.Y. 2005. Daily consumption of phenolics and total antioxidant capacity from fruit and vegetables in the American diet. *Journal of the Science of Food and Agriculture* **85**(10), 1715–1724.

Da Porto, C., Porretto, E. & Decorti, D. 2012. Comparison of ultrasound-assisted extraction with conventional extraction methods of oil and polyphenols from grape (*Vitis vinifera* L.) seeds. *Ultrasonics Sonochemistry* **20**(4), 1076–1080.

Duthie, G., Duthie, S. & Kyle, J. 2000. Plant polyphenols in cancer and heart disease: implications as nutritional antioxidants. *Nutrition Research Reviews* **13**(1), 79–106.

Espada-Bellido, E., Ferreiro-González, M., Carrera, C., Palma, M., Barroso, C.G. & Barbero, G.F. 2017. Optimization of the ultrasound-assisted extraction of anthocyanins and total phenolic compounds in mulberry (*Morus nigra*) pulp. *Food Chemistry* **219**, 23–32.

Espin, J.C, Rivas, C.S., Wichers, H.J. & Viguera, C.G. 2000. Anthocyanin based natural colorants: a new source of antiradical activity for foodstuff. *Journal of Agricultural and Food Chemistry* **48**(5), 1588–1592.

Ezekiel, R., Singh, N., Sharma, S. & Kaur, A. 2013. Beneficial phytochemicals in potato – a review. *Food Research International* **50**(2), 487–496.

Farris, S. & Piergiovanni, L. 2009. Optimization of manufacture of almond paste cookies using response surface methodology. *Journal of Food Process Engineering* **32**(1), 64–87.

Ferarsa, S., Zhang, W., Moulai-Mostefa, N., Ding, L., Jaffrin, M.Y. & Grimi, N. 2018. Recovery of anthocyanins and other phenolic compounds from purple eggplant peels and pulps using ultrasonic-assisted extraction. *Food and Bioproducts Processing* **109**, 19–28.

He, B., Zhang, L., Yue, X., Liang, J., Jiang, J., Gao, X. & Yue, P. 2016. Optimization of Ultrasound-Assisted Extraction of phenolic compounds and anthocyanins from blueberry (*Vaccinium ashei*) wine pomace. *Food Chemistry* **204**, 70–76.

Im, H.W., Suh, B.S., Lee, S.U., Kozukue, N., Ohnisi-Kameyama, M., Levin, C.E. & Friedman, M. 2008. Analysis of phenolic compounds by high-performance liquid chromatography and liquid chromatography/mass spectrometry in potato plant flowers, leaves, stems, and tubers and in home-processed potatoes. *Journal of Agricultural and Food Chemistry* **56**(9), 3341–3349.

Kampuse, S., Tomsone, L., Krūma, Z., Šabovics, M. & Skrabule, I. 2016. Effect of lovage phenolics to formation of acrylamide in French fries. *Agronomy Research* **14**(S2), 1315–1327.

Kazeem, M.I., Akanji, M.A., Hafizur, R.M. & Choudhary, M.I. 2012. Antiglycation, Antioxidant and Toxicological Potential of Polyphenol Extracts of Alligator Pepper, Ginger and Nutmeg from Nigeria. *Asian Pacific Journal of Tropical Biomedicine* **2**(9), 727–732.

Kim, D., Jeong, S.W. & Lee, C.Y. 2003. Antioxidant capacity of phenolic phytochemicals from various cultivars of plums. *Food Chemistry* **81**(3), 321–326.

Konaté, K., Hilou, A., Mavoungou, J.F., Lepengué, A.N., Souza, A., Barro, N., Datté, J.Y., M'Batchi, B. & Nacoulma, O.G. 2012. Antimicrobial Activity of Polyphenol-Rich Fractions from *Sida alba* L. (Malvaceae) against Cotrimoxazol-Resistant Bacteria Strains. *Annals of Clinical Microbiology and Antimicrobials* **11**(1), 1–6.

Leo, L., Leone, A., Longo, C., Lombardi, D.A., Raimo, F. & Zacheo, G. 2008. Antioxidant compounds and antioxidant activity in early potatoes. *Journal of Agricultural and Food Chemistry* **56**(11), 4154–4163.

Lewis, C.E., Walker, J.R.L. & Lancaster, J.E. 1999. Changes in anthocyanin, flavonoid and phenolic acid concentrations during development and storage of coloured potato (*Solanum tuberosum* L.) tubers. *Journal of the Science of Food and Agriculture* **79**(2), 311–316.

Lolayekar, N. & Shanbhag, C. 2012. Polyphenols and Oral Health. *RSBO* **9**(1), 74–84.

Maldonado, A.F.S., Mudge, E., Gänzle, M.G. & Schieber, A. 2014. Extraction and fractionation of phenolic acids and glycoalkaloids from potato peels using acidified water/ethanol-based solvents. *Food Research International* **65**(A), 27–34.

Manach, C., Scalbert, A., Morand, C., Remesy, C. & Jimenez, L. 2004. Polyphenols: food sources and bioavailability. *American Journal of Clinical Nutrition* **79**(5), 727–747.

Mane, S., Bremner, D.H., Tziboula-Clarke, A. & Lemos, M.A. 2015. Effect of ultrasound on the extraction of total anthocyanins from Purple Majesty potato. *Ultrasonics Sonochemistry* **27**, 509–514.

Mattila, P. & Hellstrom, J. 2006. Phenolic acids in potatoes, vegetables, and some of their products. *Journal of Food Composition and Analysis* **20**(3–4), 152–160.

Mohdaly, A.A.A., Sarhan, M.A., Smetanska, I. & Mahmoud, A. 2010. Antioxidant properties of various solvent extracts of potato peel, sugar beet pulp and sesame cake. *Journal of the Science of Food and Agriculture* **90**(2), 218–226.

Myers, R.H., Montgomery, D.C., Vining, G.G. & Borror, C. 2004. Response surface methodology: a retrospective and literature survey. *Journal of Quality Technology* **36**(1), 53–78.

Nara, K., Miyoshi, T., Honma, T. & Koga, H. 2006. Antioxidative activity of bound form phenolics in potato peel. *Bioscience, Biotechnology, and Biochemistry* **70**(6), 1489–1491.

Parr, A.J. & Bolwell, G.P. 2000. Phenols in the plant and in man. The potential for possible nutritional enhancement of the diet by modifying the phenols content or profile. *Journal of the Science of Food and Agriculture* **80**(7), 985–1012.

Pourcel, L., Routaboul, J.M., Cheynier, V., Lepiniec, L. & Debeaujon, I. 2007. Flavonoid oxidation in plants: from biochemical properties to physiological functions. *Trends in Plant Science* **12**(1), 29–36.

Re, R., Pellegrini, N., Proteggente, A., Pannala, A., Yang, M. & Rice-Evans, C. 1999. Antioxidant activity applying an improved ABTS radical cation decolorization assay. *Free Radical Biology and Medicine* **26**(9–10), 1231–1237.

Reyes, L.F. & Zevallos, L.C. 2003. Wounding stress increases the phenolic content and antioxidant capacity of purple-flesh potatoes (*Solanum tuberosum* L.). *Journal of Agricultural and Food Chemistry* **51**(18), 5296–5300.

Reyes, L.F., Miller, J.C. & Zevallos, L.C. 2005. Antioxidant capacity, anthocyanins and total phenolics in purple and red fleshed potato (*Solanunm tuberosum* L.) genotypes. *American Journal of Potato Research* **82**(4), 271–277.

Scalbert, A., Manach, C., Morand, C., Remesy, C. & Jimenez, L. 2005. Dietary polyphenols and the prevention of diseases. *Critical Reviews in Food Science and Nutrition* **45**(4), 287–306.

Singh, N. & Rajini, P. 2004. Free radical scavenging activity of an aqueous extract of potato peel. *Food Chemistry* **85**(4), 611–616.

Singleton, V.L., Orthofer, R. & Lamuela-Raventos, R.M. 1999. Analysis of Total Phenols and Other Oxidation Substrates and Antioxidants by Means of Folin-Ciocalteu Reagent. *Methods Enzymology* **299C**(1), 152–178.

Sotillo, D.R., Hadley, M. & Wolf-Hall, C. 2007. Potato peel extract a nonmutagenic antioxidant with potential antimicrobial activity. *Journal of Food Science* **63**(5), 907–910.

Tsao, R. & Deng, Z. 2005. Separation procedures for naturally occurring antioxidant phytochemicals. *Journal of Chromatography* B **812**(1–2), 85–99.

Velioglu, Y.S, Mazza, G., Gao, L. & Oomah, B.D. 1998. Antioxidant activity and total phenolics in selected fruits, vegetables, and grain products. *Journal of Agricultural and Food Chemistry* **46**(10), 4113–4117.

Wettasinghe, M. & Shahidi, F. 1999. Evening primrose meal: A source of natural antioxidants and scavenger of hydrogen peroxide and oxygen-derived free radicals. *Journal of Agricultural and Food Chemistry* **47**(5), 1801–1812.

Yu, L., Perret, J., Harris, M., Wilson, J. & Haley, S. 2003. Antioxidant properties of bran extracts from 'Akron' wheat grown at different locations. *Journal of Agricultural and Food Chemistry* **51**(6), 1566–1570.