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The effects of ultrasound-assisted extraction on polyphenolics compounds obtained from *Physalis angulata* using response surface approach

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ABSTRACT. Total polyphenols content (TPC), including flavonoids (rutin, mangiferin and kaempferol) and phenolic acids (gallic, caffeic and ellagic acid) from *Physalis angulata* were recovered by Ultrasound-Assisted Extraction and quantified by UV–vis and HPLC analysis. Process parameters were assessed through a Central Composite Rotatable Design (CCRD) and a model with regression coefficient equal to 0.9640, was used to establish the optimum conditions together with its respective response surfaces. The increase of ethanol percentage and solid-liquid ratio promoted a decrease on TPC but, on the other hand, the increase in the temperature led to an increase in the extraction of these compounds. Experimental results indicated a maximum amount of total polyphenols of 1.039 mg Gallic Acid Eqivalent (GAE) g^{-1} of extract, 104.88, 4.04, 8.37, 58.28, 13.26 and 1.87mg.L^{-1} for gallic acid, caffeic acid, ellagic acid, rutin, mangiferin and kaempferol, respectively.

Keywords: *Physalis*; ultrasound-assisted extraction; phenolic acids; flavonoids; HPLC analysis; total polyphenols.

Efeito da extração assistida por ultrassom sobre a obtenção de compostos fenólicos de *Physalis angulata* usando metodologia de superfície de resposta

RESUMO. Fenóis totais, incluindo flavonóides (rutina, mangiferina e kaempferol) e ácidos fenólicos (ácido gálico, ácido elágico e ácido caféico) de *Physalis angulata* foram obtidos por meio de extração assistida por ultrassom e quantificados por espectroscopia UV-VIS e cromatografia líquida de alta eficiência. Os parâmetros de processo foram acessados através de um delineamento composto central rotacional e um modelo, com coeficiente de regressão igual a 0.9640, foi usado para estabelecer as condições ótimas de extração juntamente com suas respectivas superfícies de resposta. O aumento na percentagem de etanol e razão sólido-líquido promoveu um decréscimo na quantidade de polifenóis totais extraídos enquanto que a temperatura, quando aumentada, promoveu um aumento na quantidade dos compostos. Os resultados experimentais indicaram um teor máximo de polifenóis totais de 1.039 mg EAG g^{-1} de extrato. Em paralelo, os valores máximos de ácido gálico, ácido caféico, ácido elágico, rutina, mangiferina e kaempferol foram de 104.88, 4.04, 8.37, 58.28, 13.26 e 1.87 mg L^{-1}, respectivamente.

Palavras-chave: *Physalis*; extração assistida por ultrassom; ácidos fenólicos; flavonoides; CLAE; polifenóis totais.

Introduction

Natural phenols has been reported excellent properties as food preservatives (colorants, antioxidant, antimicrobial), in the production of paints, paper and cosmetic (Ignat, Volf, & Popa, 2011) as well as to have an important role in the protection against pathological disturbances, such as atherosclerosis, brain dysfunction and cancer (Nair, Panneerselvam, Gopi, & Hong-bo, 2013).

Polyphenols are divided into several classes according to the number of phenol rings. In this class of compounds, flavonoids and phenolic acids may be highlighted. Flavonoids may be considered as antioxidants due to the ability to scavenge free radicals and reduce the risk of heart disease (Wang et al., 2017). Additionally, phenolic acids such as gallic, caffeic and chlorogenic acid, are a large group of hydrophilic polyphenols commonly found in fruits and vegetables important antioxidant properties (Padayachee et al., 2012).

Physalis angulata (PA) is a branched annual shrub that belongs to the *Solanaceae* family and widely used for treatments of diseases (Kusumaningtyas, Laily, &

Limandha, 2015). In the Amazon region, PA is popularly known as 'camapu' and its juice is used as sedative, depurative, anti-rheumatic and relief of earache (Bastos, Silveira, Salgado, Picanço-Diniz, & Nascimento, 2008; Hseu et al., 2011).

In order to obtain the highest possible amount of bioactive compounds from vegetable raw, effective extraction procedures are required (Tušek et al., 2016), commonly carried out by solid-liquid separation. From this approach, the Ultrasound-Assisted Extraction (UAE) may be coupled to the process aiming to avoid/minimize the unsuccessful recovery of these compounds. Indeed, the propagation of ultrasound waves results in the cavitation phenomena, which accelerates the diffusion and consequently the mass transfer (Hu et al., 2012). Currently, there are many researches involving the use of ultrasound in the extraction of polyphenols (Silva, Garcia, & Franciscato, 2016; Guerra, Garcia, & Silva, 2016; Lazar, Talmaciu, Volf, & Popa, 2016; Jovanović et al., 2017) however, it is desirable more discussion about variables involved in the extraction process (as solid-liquid ratio, pressure, temperature, solvent concentration, ultrasound power) to contribute with the results at separation process field. Within this framework, we studied the effects of process parameters on the recovery of total polyphenols and specific compounds (flavonoids and phenolic acids) from *Physalis angulata* obtained by ultrasound-assisted extraction.

Material and methods

Material and sample preparation

Samples of physalis were obtained and prepared according to Carniel et al., (2016). In addition, the following materials were used in this work: ethanol (95% of purity, Dinamica, Diadema/Brazil); flavonoids, phenolic acids and Folin-Ciocalteu's reagent (Sigma-Aldrich, St. Louis, MO, USA) and Na_2CO_3 (Dinamica, Diadema/Brazil).

Extraction procedure

Ultrasound-Assisted Extraction (UAE) was carried out according to Filippi, Bilibio, Bender, Carniel, & Priamo, (2015) using an ultrasonic bath (Unique, USC-1800A) with frequency of 40 kHz and power output of 154 W. Frequencies in the range 18-40 kHz may promote the cell wall disruption, which facilitates solvent access to the cell content (González-Centeno et al., 2014). The extraction procedure was developed as previously described by Conte et al., (2016) but in the current work, the system was kept in contact during 120 min and at end, the tubes were centrifuged (4000 rpm 5 min^{-1}) and the supernatant used to further analyze. Assays were performed in triplicate.

Experimental methodology and analysis

Central Composite Rotatable Design (CCRD), shown in Table 1, consisted of a full 2^3 factorial design (8 experiments), axial points (6 experiments) and replicated central points (6 experiments), totaling 20 experiments (Rodrigues & Iemma, 2005; Pereira, Molina, Arruda, & Pastore, 2016). All assays were performed at maximum ultrasound power and in random order to minimize the effects of unexpected variability in the observed responses (Pereira et al., 2016). Data were analyzed using Statistica software 5.0 (Statsoft, Oklahoma, USA) considering a significance level ($p \leq 0.05$) by analysis of variance (ANOVA).

Table 1. Experimental design, total polyphenols content and individual compounds.

Assay	Temperature (°C)	Solid-solvent ratio (g mL^{-1})	Ethanol percentage (v v^{-1})	TPC (mg g^{-1})	Individual phenolic compounds (mg g^{-1})					
					Gallic acid	Ellagic acid	Caffeic acid	Rutin hydrate	Kaempferol	Mangiferin
1	-1 (35)	-1 (0.03)	-1 (30)	0.627 ^{b}	0.1049 ^{a}	N.I	0.0040 ^{a}	0.0075^{bc}	N.I	0.0088^{c}
2	+1 (45)	-1 (0.03)	-1 (30)	0.549 ^{c}	0.0782^{b}	N.I	0.0036^{b}	0.0064^{c}	N.I	0.0133 ^{a}
3	-1 (35)	+1 (0.09)	-1 (30)	0.191 ^{hi}	0.0343^{g}	N.I	0.0013^{ef}	0.0024^{de}	N.I	0.0030^{g}
4	+1 (45)	+1 (0.09)	-1 (30)	0.242 ^{fg}	0.0483^{dc}	0.0033^{b}	0.0019^{d}	0.0034^{de}	0.0009^{b}	0.0517 ^{c}
5	-1 (35)	-1 (0.03)	+1 (76)	0.483 ^{d}	0.0511^{d}	N.I	0.0033^{c}	0.0083^{b}	N.I	0.0105^{b}
6	+1 (45)	-1 (0.03)	+1 (76)	0.453 ^{d}	0.0469^{dc}	N.I	0.0034^{c}	0.0065^{c}	N.I	0.0108^{b}
7	-1 (35)	+1 (0.09)	+1 (76)	0.160 ^{hij}	0.0166^{hij}	0.0084 ^{a}	0.0013^{efg}	0.0024^{de}	N.I	0.0041^{f}
8	+1 (45)	+1 (0.09)	+1 (76)	0.158 ^{hij}	0.0157^{ij}	N.I	0.0011^{gh}	0.0024^{de}	N.I	0.0038^{f}
9	-1.68 (32)	0 (0.06)	0 (53)	0.262 ^{f}	0.0350^{g}	N.I	0.0020^{d}	0.0035^{d}	N.I	0.0062^{d}
10	+1.68 (48)	0 (0.06)	0 (53)	0.319 ^{e}	0.0378^{fg}	N.I	0.0019^{d}	0.0041^{d}	0.0019^{a}	0.0057^{dc}
11	0 (40)	-1.68 (0.01)	0 (53)	1.039 ^{a}	0.0668^{c}	N.I	N.I	0.0583 ^{a}	N.I	N.I
12	0 (40)	+1.68 (0.11)	0 (53)	0.129 ^{j}	0.0135^{j}	N.I	0.0007^{i}	0.0017^{e}	N.I	0.0026^{g}
13	0 (40)	0 (0.06)	-1.68 (14)	0.198 ^{gh}	0.0436^{ef}	N.I	0.0015^{e}	0.0029^{de}	N.I	0.0052^{e}
14	0 (40)	0 (0.06)	+1.68 (91)	0.117 ^{j}	0.0111^{j}	N.I	N.I	0.0030^{de}	N.I	N.I
15	0 (40)	0 (0.06)	0 (53)	0.139 ^{j}	0.0215^{hi}	N.I	0.0011^{gh}	0.0028^{de}	N.I	N.I
16	0 (40)	0 (0.06)	0 (53)	0.143 ^{ij}	0.0209^{hi}	N.I	0.0011^{fgh}	0.0039^{d}	N.I	N.I
17	0(40)	0(0.06)	0(53)	0.142 ^{j}	0.0205^{hi}	N.I	0.0010^{h}	0.0030^{de}	N.I	N.I
18	0(40)	0(0.06)	0(53)	0.138 ^{j}	0.0210^{hi}	N.I	0.0010^{gh}	0.0031^{de}	N.I	N.I
19	0(40)	0(0.06)	0(53)	0.136 ^{j}	0.0218^{h}	N.I	0.0012^{efgh}	0.0035^{d}	N.I	N.I
20	0(40)	0(0.06)	0(53)	0.137 ^{j}	0.0209^{hi}	N.I	0.0011^{gh}	0.0027^{de}	N.I	N.I

Gallicacid (280 nm): 16.02 min; Ellagicacid (280 nm): 28.38 min; Rutinhydrate (210 nm): 28.39 min; Quercetin (210 nm): 32.41 min; Caffeicacid (320 nm): 23.34 min; Kaempferol (360 nm): 33.00 min; Mangiferin (257 nm): 24.38 min; N.I: notidentified.* Mean of the concentration of TPC and individual compounds in each extraction condition. Same response followed by the same letter (s) are not significantly different based on Tukey's HSD.

Characterization of compounds

Total polyphenol content (TPC) was determined by Folin-Ciocalteu colorimetric method as previously described by Carniel et al., (2016). The concentration of phenolic acids and flavonoids in the extracts were evaluated by HPLC-DAD (within the range of 1-100 μg mL^{-1} for both compounds) according to analytical conditions reported by Nayak et al., (2015) and Carniel et al., (2016).

Results and discussion

TPC

Firstly, it was developed an experimental design 2^2 (data not shown) aiming to assess the influence of ethanol percentage (0 to 100%) and solid-liquid ratio (0.01 to 0.1 g mL^{-1}). The results indicated a maximum TPC (0.74 mg GAE $g^{-1}$of extract) obtained at 0.01 g $mL^{-1}$and 50% of ethanol. Considering the negative effect for both variables as well as the operational limitation (mass measurement), a new experimental design was adopted and presented together with the individual quantification of compounds. It can be seen that the content of polyphenol ranged from 1.039 to 0.117 mg GAE g^{-1} of extract, obtained at 40°C, 0.01 g mL^{-1} and 53% of ethanol and 40°C, 0.06 g mL^{-1} and 91% of ethanol, respectively. From Pareto Chart (Figure 1), all independent variables shown a significant effect on TPC. Only the linear interaction between T and EP was not significant. The increase of EP and SLR promoted a decrease on TPC but, on the other hand, the increase in the T led to an increase in the extraction of these compounds.

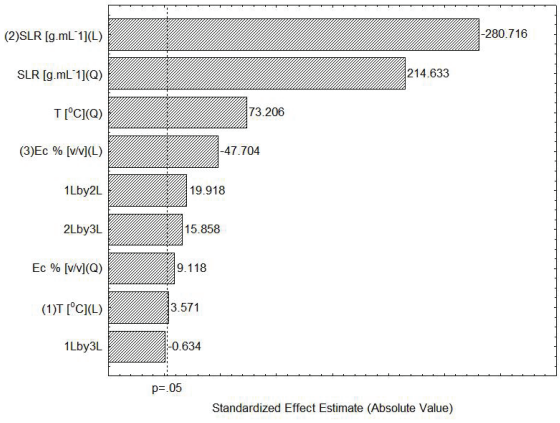

Figure 1. Pareto chart of the standardized effects of independent variables on total phenolic compound (TPC).

Literature reports similar behaviors which corroborates the results assessed in this work. For example, the positive influence of high temperature, also observed by Silva et al., (2016) and Guerra et al., (2016), may be due the increase of solubility and diffusivity as well as, a decrease in the viscosity of the phenolic compounds. Both effects improve the mass transfer and accelerate the extraction rates.

Regarding the ethanol percentage, Silva et al., (2016) and Guerra et al., (2016) also found a maximum amount of polyphenols extracted using 50% (v v^{-1}) however, with positive effect of this variable probably due the different range studied. Finally, the cited authors found a maximum yield of polyphenol of 1225.7 and 2125.31 mg GAE 100 g^{-1}, however, was adopted a concentration lower than this work (around 0.005 and 0.0025 g mL^{-1}, respectively). By the way, the solid-liquid ratio may be greatly increasing the polyphenols extraction but, on the other hand, the amount of solvent is an important factor that should be highlighted, specially to reduce the process costs and residual material. To endorse, Pradal, Vauchel, Decossin, Dhulster, & Dimitrov, (2016) mention that the volume of solvent should be sufficient to permit a good hydration and swelling of the solid phase. In terms of yield, Yue, Shao, Yuan, Wang, & Qiang, (2012) obtained 13.26 mg GAE g^{-1} of unripe apple at 30 min, power of 519.39 W, 50°C and ethanol concentration of 50%. Haminiuk, Maciel, Plata-Oviedo, & Peralta, (2012) reported an interesting review about phenolic content present in other fruits ranging from 112.00 to 1515.90 mg GAE 100 g^{-1} of fresh weight.

Equation 1 shows a quadratic model (TPC as a function of the coded independent variables) based on the analysis of the effects:

$$TPC = 0.1391 + 0.0027 \times T + 0.0538 \times T^2 - 0.2118 \times SLR + 0.1578 \times SLR^2 - 0.0360 \times Ec + 0.0067 \times Ec^2 + 0.0196 \times T \times SLR - 0.0006 \times T \times Ec + 0.0156 \times SLR \times Ec \quad (1)$$

Analysis of variance indicated that the $F_{calculated}$ was nine times higher than the $F_{tabulated}$ value and the regression coefficient (R^2) was 0.9640, i.e., based on this value, 3.60% of total variation was not explained by the model. Therefore, as the value for R^2 showed high significance the model was used to predict the optimum conditions of extraction process.

Figure 2A and B show the effects of EP *versus* SLR and T *versus* SLR on TPC recovery, respectively. In both cases, at low solid-liquid ratios and intermediates values of EP, high amount of TPC may be recovered, in accordance with the mass transfer principles. Although the temperature has shown a low effect (positive) on the TPC recovered,

an intermediate value (about 40°C) may be adopted, preserving therefore, these thermolabile compounds.

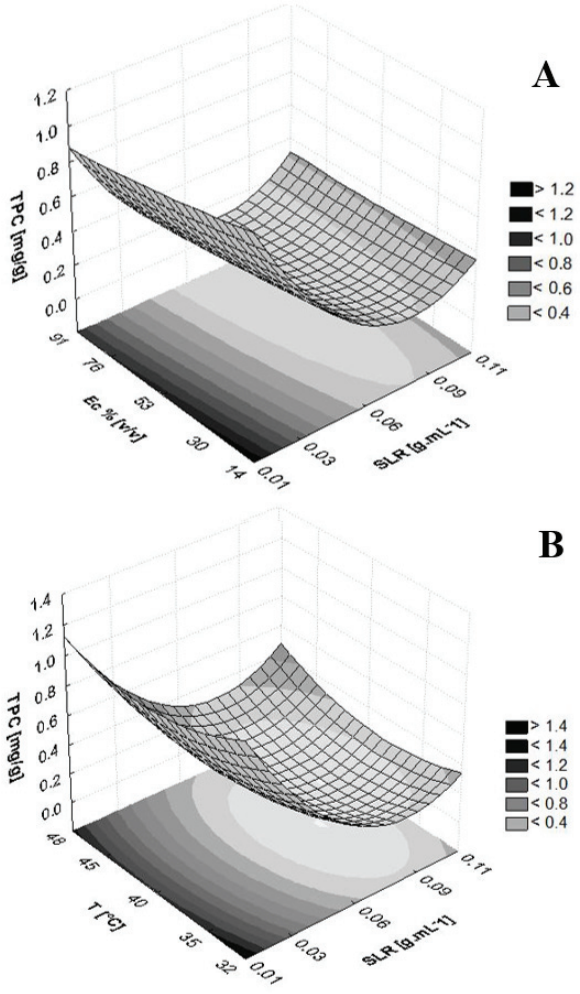

Figure 2. Three-dimensional response surface curves of TPC as a function of: A) temperature *versus* solid-liquid ratio and B) ethanol concentration *versus* solid-liquid ratio.

Individual compounds

According to statistical analysis it was verified that the process variables may be modulated aiming to reach the desirable amount of phenolic acid and flavonoids ($p \leq 0.05$). The minimum and maximum amount of gallic acid was 0.0111 mg g^1 (assay 14, carried out at 40°C, 91% of ethanol and 0.06 g of physalis per mL of solvent) and 0.1049 mg g^{-1} (assay 1, carried out at 35°C, 30% of ethanol and 0.03 g of physalis per mL of solvent), representing a decrease around 9.4 times. Ellagic acid was observed only in two assays and the composition ranged from 0.0033 (assay 4) to 0.0084 mg g^{-1} (assay 7). Finally, the content of caffeic acid ranged from 0.0007 (assay 12) to 0.0040 mg g^{-1} (assay 1).

Regarding flavonoids, both showed different extraction behavior. Quercetin was not detected in any extracts (similar results found by Nayak et al., 2015 and Carniel et al., 2016). Kaempferol showed similar extraction behavior to Ellagic acid and it was only found in 2 assays where ranged from 0.0009 (assay 4) to 0.0019 mg g^{-1} (assay 10). Rutin and mangifer in showed the minimum values of 0.0017 and 0.0026 mg g^{-1} (assay 12), whereas the maximum values were 0.0583 and 0.0133 mg g^{-1} for each compound, respectively. By comparison, Carniel et al., (2016) adopted the microwave-assisted extraction and found maximum values of TPC, phenolic acids (gallic, ellagic and caffeic acid) and flavonoids (rutin and mangiferin) equal to 3.74 mg GAE g^{-1}, 7.77, 0.55, 0.26, 0.86, and 2.43 mg L^{-1}, respectively. Although the TPC herein found was lower than using microwave-assisted extraction, it should be noted that amount extracted of each compound was considerably higher. Fu et al., (2011) reported a research about the antioxidant capacities and total phenolic contents of 62 fruits and maximum amount of phenolic acids (gallic, ellagic and caffeic acid) and flavonoids (rutin and kaempferol) were 50.25, 2.85mg.100g^{-1}, 2.16, 1.89 and 6.67 mg 100 g^{-1}, respectively.

Vu, Scarlett, and Vuong (2016) mentioned that the recovery yields of phenolic compounds, flavonoids, pro-anthocyanidins and antioxidant properties were affected by the extraction parameters. Optimal extraction conditions were found to be at ultrasonic temperature of 30°C, ultrasonic time of 5 min, ultrasonic power of 150 W, sample to solvent ratio of 8:100 g mL^{-1} and acetone concentration of 60%. Under these optimal conditions, 23.49 mg of phenolic compounds, 39.46 mg of flavonoids from 1g of banana peel could be extracted (Tsai, Chou, Liu, & Hsieh, 2014) optimized ultrasound-assisted extraction of phenolic compounds from *Phyllanthus emblica*. Under optimal conditions (15 min of extraction time, 60°C of extraction temperature, 70% of ethanol concentration, 56 kHz of ultrasonic frequency and a 1:50 solid to solvent ratio), the leaching-out rate of phenolic compounds was up to 55.34 mg g^{-1}. Finally, Batiston et al., (2015) showed the total phenolic content of ten fruits ranging from 58.97 to 675.73 mg 100 g^{-1}.

The results found may be considered as complementary to those present in the scientific literature. Besides, show relevant and provides subsidies to the purification/separation field once these compounds show well-known pharmacological properties and indicate promising future applications.

Conclusion

Ultrasound-assisted extraction was performed to recover total polyphenols from *Physalis angulata*. Optimized condition (1.039 mg GAE g^{-1} of extract at 40°C, 0.01 g mL^{-1} and 53% of ethanol) promoted an increase about 1.48 times greater than conventional extraction (without ultrasound-assisted, 0.74 mg GAE g^{-1} of extract at 0.01 g mL^{-1} and 50% of ethanol). Overall, it was observed yields and differences about phenolic acids and flavonoids extracted, depending on the experimental conditions, i.e., this research allows us to know the extraction behavior and to set specific conditions as function of the target compound aiming different applicability.

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