



Physicochemical Characterization of Potato Peel Flour (*Solanum tuberosum* L.) and Optimization of the Ultrasound-Assisted Extraction Process (UAE) of the Phenolic Compounds Using Response Surface Methodology

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Abstract

The objective of this research was to characterize potato peel flour, evaluate which solvent was best for extracting the phenolic compounds (PC), and optimize the ultrasound-assisted extraction by investigating the linear and squared effects and their interaction in recovering these compounds using an RCCD experimental design and response surface methodology associated with a desirability graph. The potato peel was donated by a local restaurant in the city of Goiânia (GO), and the results obtained for the physicochemical composition confirmed to those expected for this residue. Six solvents were used to extract the PC (ether, ethanol, water, methanol-acetone-water, 60% ethanol in water, and 10% ethanol in water), and the results showed that 10% ethanol in water gave the best results for total PC and antioxidant activity. A second RCCD experimental design was then used to obtain the ideal parameters for the ultrasound, and it was observed that, to obtain a fitted model that predicted the ideal extraction conditions with confidence, one must use three variables in the design: time, temperature, and the solid-liquid ratio. According to the response surface and desirability graphs, the ideal conditions to maximize the extraction of phenolic compounds from potato peels were: a time of 66.9 min, a temperature of 44.9 °C, and a solid-solvent ratio of 1:46.82 m/v. Thus, both the ultrasound and the experimental design helped maximize the recovery of PC from the potato peel flour.

Keywords Cavitation · Design · Desirability · Ideal conditions · Tuber · Ultrasound

Introduction

The potato (*Solanum tuberosum* L.), of the Solanaceae family, is a very important crop, ranking after wheat, corn, and rice, with more than 5000 varieties. The edible part of the plant is the tuber, which can be consumed in various ways, for example, after boiling, baking, or frying, among other options (EMBRAPA 2024; Sampaio et al. 2020).

In Brazil, this tuberous vegetable has an annual production of approximately 3.6 million tons (EMBRAPA 2024), and its global production exceeded 383 million tons in 2023 (FAOSTAT 2023). Due to its large-scale consumption, millions of tons of potato peels are generated by potato-processing industries, which are considered one of the largest food processing sectors in the world. Most of these residues are disposed of in landfills, leading to environmental consequences (Kumari et al. 2017; Sampaio et al. 2020).

Potato peel is a byproduct with great nutritional value, composed mainly of carbohydrates, starch, dietary fiber, and proteins, and it is a good source of mineral elements. It is also considered a source of functional and bioactive compounds, such as phenolic compounds, which provide health benefits due to their antioxidant properties—ten times higher in the peel than in the pulp (Silva-Beltrán et al. 2017).

Phenolic compounds are among the most important groups of natural antioxidants and help control free radicals by transferring hydrogen atoms or electrons to them, thereby preventing the oxidation of macromolecules or cell structures (Araújo 2001). Thus, phenolic compound-rich extracts could be used to preserve and fortify foods as substitutes for some synthetic antioxidants, which have toxic and carcinogenic properties (Kumari et al. 2017).

Due to the production of large volumes of residues during potato processing, the peel represents a low-cost, accessible, and promising resource for recovering these compounds, reducing environmental impact, since potato processing generates between 70,000 and 140,000 tons of peel annually on a global scale (Chang 2019; Javed et al. 2019).

Phenolic compounds are mainly extracted using conventional extraction (solid-solvent extraction) with mechanical agitation, employing different solvents such as water, ethanol, methanol, and acetone—the latter two being considered toxic and environmentally inappropriate (Lee et al. 2024). Therefore, several studies have been carried out to obtain these phenolic compounds using alternative (green), low-cost, and non-toxic solvents (Lee et al. 2024; Venturi et al. 2019; Chakraborty et al. 2020; Brahmi et al. 2022).

There are also non-conventional methods for extracting phenolic compounds, such as ultrasound-assisted extraction (UAE), a simple, low-cost technique that is easy to implement on an industrial scale. The sonication produced by ultrasound enhances mass transfer of bioactive constituents to the solvent through three effects: cavitation, which produces bubbles in the liquid/solid extraction medium that then collapse, generating localized pressure that ruptures plant cells and improves the release of intracellular substances into the solvent; mechanical agitation; and the heat generated by this agitation (Pereira et al. 2024).

Statistical tools have been employed to optimize the parameters for phenolic compound extraction (Živković et al. 2018; Riciputi et al. 2018; Chakraborty et al. 2020). Response surface methodology is a tool that produces response surface graphs, which can help predict the ideal extraction parameters to maximize the recovery of bioactive compounds from plant matrices and investigate the effects of different parameters on one or more response variables (Chatzimitakos et al. 2024).

Desirability graphs associated with these tools help solve multifactorial problems and those with multiple responses for the same parameters. That is, they can identify conditions capable of maximizing all the response variables studied in the experiment simultaneously (Apel et al. 2020).

Thus, this research aimed to characterize potato peel flour, evaluate which solvent best extracted the phenolic compounds, and optimize the ultrasound-assisted extraction by investigating the linear and squared effects and their interaction in the recovery of these compounds using an RCCD experimental design and response surface methodology associated with desirability graphs.

Material and Methods

Raw Material

The potato peels were donated by a local restaurant in Goiânia (Goiás, Brazil). The peel strips were washed under running water to remove dirt and impurities, then sanitized in a 200-ppm sodium hypochlorite solution for 10 min. Afterward, they were placed on an aluminium tray and dried at 50 °C for 48 h. Once dried, the peel strips were ground in a mill (Tec mil rotor mill, model TE-633, Tecnal brand, Piracicaba, SP, Brazil) to produce a 20-mesh powder, referred to as potato peel flour (Fig. 1). The flour was then placed in plastic bags and stored in a freezer (model FE26, Electrolux brand, Curitiba, PR, Brazil) at −18 °C.

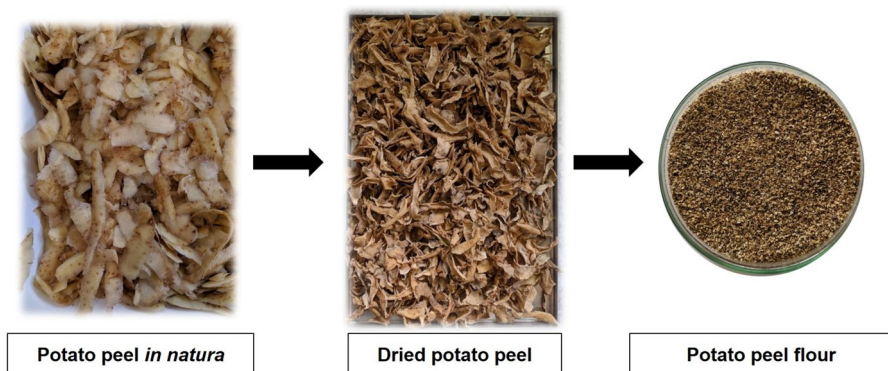


Fig. 1 Potato peel before and after drying and the resulting flour

Physicochemical Analysis of Potato Peel Flour

The moisture content was determined using a gravimetric method, according to AOAC methods 925.10 (2023a). Approximately 5 g of the sample was weighed in previously dried and tared porcelain crucibles. The samples were placed in an oven (model 402/D, Ethik Technology brand, Vargem Grande Paulista, SP, Brazil) at 105 °C and dried until constant weight was achieved (approximately 4 h). After drying, the samples were transferred to a desiccator to cool to room temperature before reweighing. The difference between the initial and final weights represents the amount of water that evaporated from the sample.

The ash content was determined according to AOAC method 923.03 (2023b). Approximately 2 g of the sample were first carbonized on an electric plate (Magnetic stirrer with heating, model RH basic 1, IKA, Staufen im Breisgau, Germany) to remove most of the organic matter. The partially carbonized material was then incinerated in a muffle furnace (model EDGCON 3P, EDG Equipamentos, São Carlos, SP, Brazil) at 550 °C until light gray ash was obtained, corresponding to the mineral residue. The difference between the initial and final weights represents the amount of mineral residue (ash).

Water activity (aw) was determined using a LabSwift-aw meter (LabSwift model, Novasina, Lachen, Switzerland). A small amount of the sample was placed in the sample cup, and the chamber was sealed and maintained at 25 °C until equilibrium was reached. The instrument measures the equilibrium relative humidity (ERH) in the headspace above the sample using a capacitive sensor and automatically converts this value to water activity (aw).

Crude protein content was determined according to AOAC method 920.87 (2023c) using the Kjeldahl method. Approximately 0.5 g of the sample was digested, distilled, and titrated to determine the total nitrogen content. This classical method does not measure protein directly but quantifies the nitrogen present in the sample, which is then converted to crude protein using a standard conversion factor of 6.25.

The total carbohydrate content was determined by the phenol–sulfuric acid colorimetric method, as described by Dubois et al. (1956). This method is based on the reaction of carbohydrates with concentrated sulfuric acid and phenol, forming a yellow-coloured complex whose intensity is proportional to the sugar concentration. Absorbance was measured at 490 nm. For the analysis, 1 g of the sample was diluted in approximately 150 mL of distilled water, heated at 80 °C for 5 min under stirring on an electric plate (magnetic stirrer with heating, model RH basic 1, IKA, Staufen im Breisgau, Germany), and then cooled in an ice bath before colorimetric measurement.

Total lipid content was determined according to the method of Bligh and Dyer (1959), a classical cold extraction procedure widely used for wet biological samples due to its speed and efficiency. Approximately 2 to 3.5 g of the sample, depending on its fat content, were used. Lipid extraction was performed using a ternary solvent system consisting of 10 mL of chloroform, 20 mL of methanol, and 8 mL of water. The mixture was homogenized for 30 min, followed by the addition of 10 mL of chloroform and 10 mL of 1.5% sodium sulfate solution to promote phase separation. After standing for 24 h, the lower chloroform phase (5 mL) was carefully collected,

the solvent evaporated, and the dried lipid residue weighed to determine the total lipid content.

The pH was determined according to AOAC method 945.27 (2023d). For this, 5 g of the sample was homogenized in 50 mL of distilled water and stirred for 30 min. The mixture was then filtered through a nylon mesh, and the pH was measured in the resulting filtrate.

Titrate acidity was determined according to AOAC method 942.15 (2023e), using the soluble alcohol acidity method. A known mass of the sample (2.5 g) was extracted with neutralized ethanol (80–95%), and the filtered extract was titrated with 0.1 N sodium hydroxide (NaOH) solution to an endpoint of pH 8.2, detected either by phenolphthalein indicator or potentiometrically. The volume of NaOH consumed was used to calculate the total acidity, expressed as the equivalent of the predominant acid.

The starch content was determined by acid digestion in a microwave oven (model BMP40ESBBB, Brastemp brand, São Bernardo do Campo, SP, Brazil), according to the method described by Cereda et al. (2004). Approximately 2.5 g of the sample was hydrolyzed with a strong acid in sealed digestion flasks under microwave heating to convert starch into glucose. After neutralization, the released glucose was quantified by a colorimetric method, and the result was converted to starch content using a stoichiometric factor of 0.9.

Mineral Composition

The mineral composition of potato peel flour was analyzed at the Soil and Leaf Analysis Laboratory of the School of Agronomy, Federal University of Goiás (UFG), Brazil, according to the methodology described by Silva (2009). The contents of nitrogen (N), phosphorus (P), potassium (K), calcium (Ca), magnesium (Mg), boron (B), copper (Cu), iron (Fe), manganese (Mn), and zinc (Zn) were determined, and all analyses were performed in triplicate, using analytical blanks and calibration standards.

Calibration curves were prepared for each mineral element using analytical-grade standard solutions with known concentrations. Serial dilutions were performed to obtain at least five concentration levels covering the expected range of each element in the potato peel flour samples. Blank solutions containing all reagents, but no sample or standard, were used to correct background signals.

Calcium (Ca) and magnesium (Mg) Calcium and magnesium were extracted using a 1.0 mol L⁻¹ potassium chloride (KCl) solution. Approximately 10 g of potato peel flour were transferred to 125-mL Erlenmeyer flasks, and 100 mL of KCl solution was added. The suspensions were shaken for 5 min on a horizontal shaker and allowed to stand overnight for decantation. An aliquot of 25 mL of the extract was titrated by complexometric titration with 0.0125 mol L⁻¹ EDTA, using Eriochrome Black T as the indicator. The volume of EDTA consumed was recorded, and the combined Ca²⁺+Mg²⁺ content was calculated according to the equivalence between EDTA volume and metal concentration. Results were expressed as mg kg⁻¹ of sample.

Phosphorus (P) Phosphorus was extracted using Mehlich-1 solution (0.05 mol L⁻¹ HCl+0.0125 mol L⁻¹ H₂SO₄). Approximately 10 g of sample was mixed with 100 mL

of the extractant, shaken for 5 min, and allowed to decant overnight. Phosphorus determination was performed by the molybdenum blue colorimetric method. An aliquot of 5 mL of extract was reacted with ammonium molybdate solution and ascorbic acid, forming a phosphomolybdenum blue complex. After 60 min of colour development, absorbance was measured at 660 nm using a UV–Vis spectrophotometer (UV-1800, Rayleigh, Beijing, China).

A calibration curve was constructed using standard phosphorus solutions prepared at concentrations ranging from 1 to 4 mg L⁻¹. Absorbance values were plotted against phosphorus concentrations, generating a linear regression equation ($y = ax + b$). Phosphorus content in the samples was calculated by interpolation from the calibration curve, taking into account the dilution factor, and results were expressed as mg kg⁻¹ of sample.

Potassium (K) Potassium was determined in the Mehlich-1 extract by flame photometry (PFP7 model, Jenway, Almada, Portugal). Instrument calibration was performed using potassium standard solutions with concentrations ranging from 0.1 to 0.4 mmol L⁻¹. Calibration curves were obtained by plotting emission intensity against potassium concentration. Sample concentrations were determined by interpolation from the calibration curve and corrected for the extraction dilution factor. Results were expressed as mg kg⁻¹ of sample.

Micronutrients (Cu, Fe, Mn, Zn) Copper, iron, manganese, and zinc were extracted using Mehlich-1 solution at a sample-to-extractant ratio of 1:5. Approximately 5.0 g of potato peel flour was shaken with 25 mL of extractant for 5 min and filtered through Whatman No. 42 filter paper.

The concentrations of Cu, Fe, Mn, and Zn were determined by atomic absorption spectrophotometry (Shimadzu® AA-6300, Tokyo, Japan), using element-specific hollow cathode lamps. Calibration curves were prepared using certified standard solutions for each element, and concentrations were calculated from linear regression equations. Results were expressed as mg kg⁻¹ of sample.

Boron (B) Boron was extracted using a barium chloride solution under microwave oven (model BMP40ESBBB, Brastemp brand, São Bernardo do Campo, SP, Brazil) heating. Approximately 10 g of sample was mixed with 20 mL of BaCl₂ extractant and subjected to microwave heating. After cooling and filtration, boron was determined by spectrophotometry using the azomethine-H method.

Following 30 min of colour development in the dark, absorbance was measured at 420 nm. A calibration curve was constructed using boron standard solutions at known concentrations. Absorbance values were plotted against boron concentration, and boron content in the samples was calculated by interpolation from the linear regression equation and expressed as mg kg⁻¹ of sample.

Nitrogen (N) Total nitrogen content was determined by the Kjeldahl method, as previously described, and converted to nitrogen content using a factor of 6.25.

Total Tannin Content

The total tannin content was determined according to the methods of Swain and Hillis (1959) and Singleton and Rossi (1965), using the Folin-Denis reagent. Approximately 2.5 g of potato peel flour was extracted with 100 mL of distilled water heated to 80 °C and stirred for 5 min while maintaining constant temperature. After cooling, the mixture was filtered through filter paper, and the filtrate was used for analysis.

For the determination, aliquots of the extract were mixed with 0.5 mL of Folin-Denis reagent and 1.0 mL of saturated sodium carbonate solution. The reaction occurs in an alkaline medium, where the phenolic hydroxyl groups of tannins reduce the phosphomolybdic-phosphotungstic acid complex, forming a blue colouration. After standing for 30 min at room temperature, absorbance was measured at 760 nm using a spectrophotometer (UV-1800 model, Rayleigh, Beijing, China).

A standard curve was prepared from a freshly prepared tannic acid solution containing 20 mg of tannic acid dissolved in 200 mL of distilled water. Aliquots of this solution, at different concentrations, were subjected to the same analytical procedure as the samples. Absorbance readings were plotted against concentration, and the resulting calibration equation was used to calculate the total tannin content, expressed as a percentage (%).

Conventional Extraction of Phenolic Compounds from Potato Peels

Preparation of Potato Peel Extracts

Six extracts were obtained to evaluate the most effective solvent for extracting phenolic compounds from potato peel flour. The solvents used were the following: ethyl ether, 95% ethanol, distilled water (Zielinski and Kozłowska 2000), a mixture of 50% methanol: 70% acetone:distilled water (2:2:1) (Rufino et al. 2010), 10% ethanol in water (Venturi et al. 2019), and 60% ethanol in water (Mendes et al. 2021).

The ether extract was prepared by homogenizing 2.5 g of potato peel flour in 50 mL of ethyl ether (1:20 m/v), stirring for 1 h at room temperature in the absence of light. The solution was then filtered through filter paper, and the volume was adjusted to the original volume in a volumetric flask using ethyl ether. The residue was dried, weighed, and used to obtain the alcoholic extract with 95% ethanol, following the same procedure. Afterward, the dried residue was again weighed and used to obtain the aqueous extract with distilled water using the same method.

For the 50% methanol:70% acetone:distilled water (2:2:1) extract, 2.5 g of the sample was first left to rest in 20 mL of 50% methanol for 1 h. The solution was then centrifuged (model 5403, Eppendorf brand, Hamburg, Germany), and the supernatant was transferred to a 50-mL volumetric flask. An aliquot of 20 mL of 70% acetone was added to the residue, left to rest for 1 h, and centrifuged (model 5403, Eppendorf brand, Hamburg, Germany) again. The supernatant was transferred to the same volumetric flask, and the volume was completed to 50 mL with distilled water.

The 60% ethanol in water extract was obtained by homogenizing 2.5 g of the sample in 50 mL of 60% ethanol in water, stirring for 1 h at room temperature in the absence of light. The solution was then filtered through filter paper, and the volume was adjusted to 50 mL with the same solvent. The 10% ethanol in water extract was prepared using the same procedure.

All extracts were stored in amber flasks and kept in a freezer (model FE26, Electrolux brand, Curitiba, PR, Brazil) at -18°C until analysis.

Determination of Total Phenolic Compound Content

The total phenolic compound (TPC) content of the potato peel flour extracts was determined using the Folin-Ciocalteu method as described by Zielinski and Kozłowska (2000). This colorimetric method is based on the redox reaction in which phenolic groups reduce the phosphomolybdic-phosphotungstic acid complex of the Folin-Ciocalteu reagent, producing a blue colouration proportional to the phenolic concentration.

For the standard curve, gallic acid solutions were prepared by dissolving 50 mg of gallic acid in a 500-mL volumetric flask with distilled water. Aliquots ranging from 0.0 to 1.0 mL of this solution were transferred to 10 mL flasks containing 5.0 mL of distilled water, followed by the addition of 1.0 mL of Folin-Ciocalteu reagent and 1.0 mL of 25% sodium carbonate solution. The volume was adjusted with distilled water, and the mixtures were allowed to stand for 30 min before measuring absorbance at 700 nm.

For the samples, 2.0 mL of extract was mixed with 5.0 mL of distilled water, 1.0 mL of Folin-Ciocalteu reagent, and 1.0 mL of sodium carbonate solution, following the same procedure as for the standard curve. Absorbance readings were performed using a spectrophotometer (UV-1800 model, Rayleigh, Beijing, China) at 700 nm.

Results were calculated from the gallic acid calibration curve and expressed as milligrams of gallic acid equivalents per 100 g of sample (mg GAE/100 g).

Antioxidant Activity

Antioxidant activity was evaluated for all extracts using the DPPH, ABTS, and ferric reducing antioxidant power (FRAP) assays.

DPPH The antioxidant activity was determined using the DPPH (2,2-diphenyl-1-picrylhydrazyl) free radical method, as described by Rufino et al. (2010). This method is based on the ability of antioxidant compounds to reduce the purple-coloured DPPH• free radical to its yellow-coloured reduced form (DPPH-H), with the decrease in absorbance being proportional to the sample's antioxidant capacity.

A 0.06 mM DPPH solution was prepared by dissolving 2.4 mg of DPPH in 100 mL of methanol and used on the same day of analysis. In a dark environment, 0.1 mL of the extract at different dilutions was added to 3.9 mL of the DPPH solution, homogenized, and kept at rest. Absorbance was measured at 515 nm at 5-min

intervals until stabilization using a spectrophotometer (UV-1800 model, Rayleigh, Beijing, China).

A standard curve was prepared from DPPH solutions ranging from 10 to 60 μM , and the corresponding absorbance values were used to obtain the calibration equation. The absorbance corresponding to half of the initial control absorbance was used to calculate DPPH consumption. Results were expressed as EC_{50} (g sample/g DPPH), representing the amount of sample required to reduce 50% of the initial DPPH radical concentration.

ABTS The ABTS (2,2'-azinobis-(3-ethylbenzothiazoline-6-sulfonic acid)) assay was also conducted according to Rufino et al. (2010). The $\text{ABTS}^{\bullet+}$ radical was generated by reacting 5 mL of ABTS stock solution (7 mM) with 88 μL of potassium persulfate (140 mM) and keeping the mixture in the dark at room temperature for 16 h. Before analysis, the solution was diluted in ethanol to an absorbance of 0.70 ± 0.05 at 734 nm.

A standard curve was prepared using Trolox solutions ranging from 100 to 2000 μM , where 30 μL of each standard solution was mixed with 3.0 mL of $\text{ABTS}^{\bullet+}$ radical solution. After 6 min of reaction in the dark, absorbance was measured at 734 nm.

For the samples, 30 μL of extract at different dilutions was added to 3.0 mL of $\text{ABTS}^{\bullet+}$ radical solution, following the same procedure as the standard curve. Absorbance was measured 6 min after mixing using a spectrophotometer (UV-1800 model, Rayleigh, Beijing, China). Results were expressed as μmol of Trolox equivalents per gram of sample (μmol Trolox/g).

FRAP The FRAP assay was conducted following Rufino et al. (2010). This method is based on the reduction of the ferric (Fe^{3+})–TPTZ [2,4,6-tri(2-pyridyl)–1,3,5-triazine] complex to the ferrous (Fe^{2+})–TPTZ blue complex, whose absorbance is proportional to the reducing power of the sample.

The FRAP reagent was prepared by mixing 25 mL of acetate buffer (0.3 M, pH 3.6), 2.5 mL of TPTZ solution (10 mM in 40 mM HCl), and 2.5 mL of ferric chloride solution (20 mM). The standard curve was obtained using Trolox solutions ranging from 104 to 800 μM , by mixing 90 μL of each standard solution with 270 μL of distilled water and 2.7 mL of FRAP reagent. After incubation in a water bath at 37 °C for 30 min, absorbance was measured at 595 nm.

For the samples, 90 μL of extract at different dilutions was added to 270 μL of distilled water and 2.7 mL of FRAP reagent, following the same procedure as for the standard curve. Absorbance was measured 30 min after mixing using a spectrophotometer (UV-1800 model, Rayleigh, Beijing, China). Results were also expressed in μmol of Trolox equivalents per gram of sample (μmol Trolox/g).

Experimental Design for Ultrasound-Assisted Extraction (UAE) of Phenolic Compounds from Potato Peels

The rotational central composite design (RCCD) was selected to determine the ultrasound-assisted extraction parameters for phenolic compounds from potato peel flour.

This approach investigates the linear and squared effects, as well as the interaction between extraction time (X_1 : 15, 35, and 55 min) and temperature (X_2 : 30, 45, and 60 °C). These variables were chosen as the independent factors, each with five coded levels (−1.41, −1, 0, 1, 1.41) (Table 1).

The experimental design resulted in 13 trials, including five central points to evaluate the repeatability of the data. A second-order polynomial equation (Eq. 1) was used to assess the interaction between the independent variables and the response variable (phenolic compound content). In this equation, Y represents the predicted response for phenolic compound content, b_0 is the constant term, b_1 and b_2 are the linear coefficients, b_{11} and b_{22} are the squared coefficients, b_{12} is the interaction coefficient, and X_1 and X_2 correspond to the independent variables (extraction time and temperature).

$$Y = b_0 + b_1X_1 + b_2X_2 + b_{11}X_1^2 + b_{22}X_2^2 + b_{12}X_1X_2 \quad (1)$$

The experimental design was validated through analysis of variance (ANOVA) for the response variable (total phenolic compound content) at a 95% confidence level. This analysis was conducted to evaluate the effect of each factor. The determination coefficient (R^2), the F value calculated for the regression, and the F value for the lack of fit were used to assess the adequacy of the regression model.

The extraction of phenolic compounds followed the procedure outlined by Apel et al. (2020), with adaptations. The solvent used was the most efficient one in recovering these compounds during the conventional extraction step. For the extraction, 2.5 g of the sample was mixed with 50 mL of the chosen solvent and placed in an ultrasound bath (model LS2800, Logen Scientific brand, Diadema, SP, Brazil) with an operational frequency of 40 kHz, to perform the sonication and extraction at the specified time and temperature for each trial, as outlined in the experimental design. After sonication, the samples were centrifuged (model 5403, Eppendorf brand, Hamburg, Germany) at 6000 rpm for 10 min and filtered through filter paper. The resulting extracts were stored in amber flasks and kept at −18 °C until analysis.

Optimization of Ultrasound-Assisted Extraction (UAE) of Phenolic Compounds

A second rotational central composite design (RCCD) was conducted with three factors as the independent variables: extraction time (X_1 : 20, 37, and 55 min), temperature (X_2 : 30, 45, and 60 °C), and solid-solvent ratio (1: X_3 : 20, 30, and 40 m/v). Each factor had five coded levels (−1.41; −1; 0; 1; 1.41) (Table 2). The experimental design followed the same procedure described above, but this time with 19 trials and 5 central points. The optimal ultrasound-assisted extraction conditions for obtaining the maximum amounts of phenolic compounds were selected based on the response surface and desirability graphs.

Table 1 Factors and levels of the rotational central composite design (RCCD)

Factors	Code	−1.41	−1	0	1	1.41
Time (min)	X_1	6.7	15	35	55	63.3
Temperature (°C)	X_2	23.8	30	45	60	66.2

Table 2 Factors and levels of the rotational central composite design (RCCD)

Factors	Code	−1.68	−1	0	1	1.68
Time (min)	X_1	6.7	15	35	55	63.3
Temperature (°C)	X_2	23.8	30	45	60	66.2
Solid-solvent ratio (m/v)	1: X_3	13.18	20	30	40	46.82

The extraction of phenolic compounds followed the procedure outlined by Živković et al. (2018), with adaptations. The solvent used was the most efficient one in the conventional extraction. Therefore, 2.5 g of each sample was mixed with the solvent at the different solid/solvent ratios and placed in an ultrasound bath (model LS2800, Logen Scientific brand, Diadema, SP, Brazil) for the time and temperature specified in each trial, according to the experimental design. After sonication, the samples were centrifuged (Centrifuge 5403) at 6000 rpm for 10 min and filtered through filter paper. The extracts were stored in amber flasks and kept at -18°C until analyzed.

Determination of Total Phenolic Compound Contents and Antioxidant Activities of the Samples Obtained Using the Experimental Design

The total phenolic compound contents were evaluated as described above for all trials of the two experimental designs. Regarding antioxidant activity, the assays (DPPH, ABTS, and ferric ion reduction—FRAP) were applied only to the extract obtained under the optimum conditions, as determined by the response surface and desirability graph.

Scanning Electronic Microscopy (SEM)

The morphology of the potato peel flour was analyzed using a scanning electron microscope (Model JSM-6610, JEOL brand, Tokyo, Japan, equipped with energy dispersive spectroscopy (EDS) with a Thermo Scientific NSS Spectral Imaging detector) in the Multiuser Laboratory of High-Resolution Microscopy (LabMic) at the Physics Institute of UFG. The morphology was evaluated after conventional extraction (without ultrasound) and after extraction using the optimum conditions obtained from the design (with ultrasound) to assess the effect of ultrasound on the potato peel flour. For this, the samples were defatted according to Bligh and Dyer (1959), dried in an oven (model 402/D, Ethik Technology brand, Vargem Grande Paulista, SP, Brazil) at 45°C , and the morphology was evaluated at magnifications of $\times 100$ and $\times 10,000$.

Statistical Analysis

All analyses were performed with three repetitions and in triplicate, and the results were subjected to ANOVA using Student's *t*-test with the statistical program

Sisvar® (Ferreira 2019). Values of $p < 0.05$ were considered statistically significant. The experimental design was analyzed using the Protomiza Experimental Design software, and the graphs and response surface were obtained using the same software. The desirability graph was generated using STATSOFT STATISTICA 12.5 software.

Results and Discussion

Physicochemical Composition

Tables 3 and 4 present the nutritional values found in the potato peel flour. Table 3 shows the values obtained for the proximate composition, where the values for moisture, ash, protein, lipid, and carbohydrate contents were close to those reported by Kumari et al. (2017), Elkahoui et al. (2018), and Helal et al. (2020) for potato peels. This confirms that the results obtained in the present study were within the expected range for this product.

Moisture content is an important parameter for evaluating changes in food quality during storage, as excess moisture can lead to the growth of microorganisms and enzyme reactions (Cogliatti et al. 2014). According to Resolution-RDC n°. 263 of the Brazilian National Health Surveillance Agency (BRASIL 2005), the maximum permitted moisture content for flours, cereal starches, and brans is 15% (g/100 g). Therefore, the moisture content obtained in the present study was within the limits established by the legislation.

Water activity is a parameter that evaluates the amount of water bound to the food components and is important for assessing food stability. The higher the water activity,

Table 3 Physicochemical composition of potato peel flour (*Solanum tuberosum* L.)

Parâmetros	Potato peel flour*	Kumari et al. (2017)	Elkahoui et al. (2018)	Helal et al. (2020)
Moisture (%)	7.49 ± 0.05	6.98	4.46	6.99
Water activity (a_w)	0.49 ± 0.004	-	-	-
Ash (%)	7.73 ± 0.15	7.24	6.69	7.37
Lipid (%)	1.27 ± 0.017	2.09	0.81	2.60
Protein (%)	11.70 ± 0.23	11.17	15.99	10.21
Carbohydrate (%)	72.40 ± 2.77	72.53	72.00	65.47
Starch (%)	44.85 ± 1.39	-	-	-
Tannins (%)	0.33 ± 0.015	-	-	-
pH	6.89 ± 0.04	-	-	-
Acidity (%) **	1.46 ± 0.22	-	-	-

*Mean and standard deviation of three replicates. The values of the constituents were determined after drying the sample

**Acidity in % NaOH mL 100 g⁻¹ of citric acid (v/w)

Table 4 Macro and micro-mineral content of potato peel flour

Sample	Macro-minerals (g/kg)				
	N	P	K	Ca	Mg
	20.12 ± 0.63	4.61 ± 0.06	31.0 ± 0.60	0.64 ± 0.05	1.22 ± 0.03
Potato peel flour	Micro-minerals (mg/kg)				
	B	Cu	Fe	Mn	Zn
	25.63 ± 0.06	5.87 ± 0.06	149.73 ± 6.05	-	239.90 ± 7.10

Mean and standard deviation of three replicates. The values of the constituents were determined after drying of the sample

the greater the availability of water for microbial growth. A water activity below 0.90 inhibits the growth of certain microorganisms, and in the present study, the water activity was approximately 0.49, indicating that the potato peel flour was highly stable to microbial deterioration (Silva and Marsaioli 2003; Picanço et al. 2018).

The most abundant component of potato peels is carbohydrates, with starch accounting for 30 to 52% of the total content (Ramaswamy et al. 2013; Gaudino et al. 2020). In the present study, the starch content of the potato peel flour was 44.85%, and 27.55% was composed of other non-starchy carbohydrates (polysaccharides without starch).

The tannin content of the potato peel flour was also evaluated. The determination of tannins is related to their antinutritional effects at concentrations above 5%, which can negatively affect palatability and reduce protein digestibility (Makkar 1994; Cruz et al. 2007). However, the tannin content in the potato peel flour (0.33%) (Table 3) did not present an antinutritional effect due to its low concentration.

The hydrogen ion potential (pH) and acidity are parameters related to food quality control, its processing, and its state of conservation (Cecchi 2003). The pH value and acidity of the potato peel flour in this study were 6.89 and 1.46%, respectively, indicating a low concentration of organic acids. It should be noted that while these values make the sample susceptible to microbiological contamination, the low moisture content and water activity of the flour would reduce the growth rates of any microorganisms present (Batista et al. 2020).

Table 4 shows the quantification of six macro-minerals (N, P, K, Ca, and Mg) and five micro-minerals (B, Cu, Fe, Mn, and Zn) in the potato peel flour. According to Bethke and Jansky (2008), potassium (K) is the main mineral present in potato peels and was the most abundant in the potato peel flour used in the present study, followed by nitrogen (N), which also showed a high content. Similar values were reported by Vaitkevičienė (2019), who evaluated the peels of different potato cultivars and found K values ranging from 24.3 to 33.3 g/kg.

According to Vaitkevičienė (2019), phosphorus (P) is another important mineral found in potatoes. In his study, phosphorus levels varied from 2.90 to 4.54 g/kg in the peel, a value similar to that found in the present study (4.61 g/kg). Removing vegetable peels before consumption can contribute to a deficiency of certain minerals, such as calcium (Ca), magnesium (Mg), copper (Cu), iron (Fe), and zinc (Zn) (White and Broadley 2009).

Wszelaki et al. (2005) evaluated mineral contents in *Solanum tuberosum* and found 1.44 g/kg Ca, 1.71 g/kg Mg, 16.7 mg/kg Cu, 778.7 mg/kg Fe, and 37.8 mg/kg Zn, values similar to those found in the present study. Similarly, Vaitkevičienė (2019), when evaluating the peels of different potato species, found values ranging from 0.66 to 1.10 g/kg for Ca, from 1.19 to 1.60 g/kg for Mg, from 4.01 to 4.55 mg/kg for Cu, and from 151.1 to 320.5 mg/kg for Fe, which are also comparable to those observed in the current study. However, the same author found values ranging from 9.20 to 11.2 mg/kg for boron (B) and from 9.04 to 18.3 mg/kg for Zn, which were lower than the values found in the present study (Table 4).

The mineral contents of potato peels can vary due to various factors, such as genotype, which determines the thickness of the peels, the cultivar, cultivation conditions and location, nitrogen and potassium fertilization, the inclusion or absence of other micronutrients, and the production system (organic or conventional) (Sampaio et al. 2020). These factors help explain the variation in mineral contents found in the present study compared to the studies cited above.

Effect of the Parameters on the Conventional Extraction of Phenolic Compounds

Table 5 shows the total phenolic compounds obtained and the antioxidant activity of the potato peel flour extracts obtained using different solvents. The total phenolic compounds showed significant differences ($p < 0.05$) between the solvents used for extraction. The 10% ethanol in water solvent extracted more phenolic compounds than the other solvents, with an increase of 79.8% compared to 95% ethanol, 10.6% more than water, and 27.7% more than the 60% ethanol in water solvent.

Water, when added to other solvents, allows the vegetable material to swell, increasing the solvent contact area and facilitating the extraction of phenolic compounds as well as the hydrophilic fraction of glycosylated phenolic compounds (Jovanović et al. 2017; Brahmi et al. 2022). According to Sharmila et al. (2016), a

Table 5 Total phenolic compound content and antioxidant activity of potato peel flour after conventional extraction using different solvents

Solvent	Analysis			
	TPC (mg GAE/100 g)	DPPH (EC ₅₀ -g sample/g DPPH)	ABTS (μM Trolox/g sample)	FRAP (μM Trolox/g sample)
Ether	2.25 ± 0.30 ^f	2.73 ± 0.03 ^b	96.20 ± 0.1 ^c	40.69 ± 0.32 ^c
Ethanol	57.22 ± 1.01 ^c	9.71 ± 0.62 ^a	31.45 ± 0.89 ^f	36.39 ± 0.01 ^c
Water	253.10 ± 1.91 ^b	0.99 ± 0.02 ^c	985.15 ± 13.9 ^b	444.47 ± 4.46 ^b
MAW	169.54 ± 1.85 ^d	0.88 ± 0.01 ^c	768.2 ± 2.29 ^c	191.17 ± 0.25 ^d
60% ethanol	204.90 ± 0.21 ^c	0.77 ± 0.01 ^c	664.04 ± 9.88 ^d	413.05 ± 5.85 ^c
10% ethanol	283.51 ± 1.90 ^a	0.66 ± 0.03 ^c	1073.00 ± 6.78 ^a	648.90 ± 2.67 ^a

Means ± standard deviation of three repetitions. Equal letters in the columns do not differ in Student's *t*-test with significance level $p < 0.05$. TPC, total phenolic compounds; GAE, gallic acid equivalent; ether, ethyl ether; ethanol, 100% alcohol; water, distilled water; MAW, methanol 50% + acetone 70% + distilled water; 60% ethanol, 60% ethanol in water solvent; 10% ethanol, 10% ethanol in water solvent

greater proportion of water in relation to methanol improved the extraction of these compounds, as a more polar medium breaks the hydrogen bonds, making the extraction easier. Zielinski and Kozłowska (2000) also stated that the addition of water to organic solvents facilitates extraction since sugars and proteins bound to the phenolic compounds are more soluble in water.

Ethanol plays an important role in the extraction process as it breaks the bonds between the solutes and the vegetable matrix, facilitating the mass transfer of constituents into the solvent (Brahmi et al. 2022). The results of the present study suggest that the phenolic compounds in the potato peel flour have relatively high polarity and are preferentially extracted by a mixture of 10% ethanol in water.

Abreu et al. (2021) evaluated the phenolic compounds in *seriguela* seeds using different methods and solvents and found that a ternary mixture (50% methanol + 70% acetone + distilled water) gave better results than solvents like ether, ethanol, water, and 70% ethanol in water. This ternary mixture was more effective because it acted directly on the cell walls and membranes, aiding in the extraction of these compounds. Therefore, the extraction of phenolic compounds is influenced by several factors, including the nature of the vegetable material, the compound structure, functional groups, type of solvent, and extraction method, among others, all of which significantly affect their recovery (Abreu et al. 2021).

The antioxidant activity of the different potato peel flour extracts was determined using the DPPH, ABTS, and FRAP methods (Table 5). These methods evaluate the ability of antioxidant phenolic compounds to stabilize DPPH and ABTS free radicals and reduce iron (FRAP) (Rufino et al. 2010).

The DPPH method is based on the theory that a hydrogen-donating compound is considered an antioxidant (Moon and Shibamoto 2009). In the present study, there was no significant difference ($p > 0.05$) between the water, MAW, 60% ethanol in water, and 10% ethanol in water extracts. These four extracts showed satisfactory results (less than 1 g sample/g DPPH), considering that antioxidant activity is expressed in EC₅₀, i.e. the lower the amount of sample required to reduce 50% of the DPPH radical, the greater the antioxidant activity (Rufino et al. 2010). When compared with the other extracts, the 95% ethanol extract showed the lowest antioxidant activity, requiring 9.71 g of sample to reduce 50% of the DPPH radical.

The results obtained using the ABTS and FRAP methods expressed the antioxidant capacity of the compounds in the extracts. The higher the result, the greater the amounts of compounds with antioxidant activity. Using the ABTS method, the results for the different extracts were significantly different ($p < 0.05$), with the 10% ethanol extract showing the greatest antioxidant activity. According to Moon and Shibamoto (2009), the ABTS method is widely used to evaluate compounds with both lipophilic and hydrophilic natures, further suggesting that the phenolic compounds in the potato peel flour are of a more polar nature.

The results obtained using the FRAP method were expressed similarly to the ABTS method. However, the values obtained using FRAP were smaller than those obtained using ABTS for all the extracts, as FRAP evaluates the capacity of compounds to reduce ferric ions (Fe^{3+} , the most oxidized form) to ferrous ions (Fe^{2+} , the more stable form of iron). Therefore, the FRAP method did not fully express the

antioxidant potential of the samples. Table 5 shows that the 10% ethanol in water solvent was able to extract the largest amounts of compounds that reduced iron.

Effect of the Parameters on the Ultrasound-Assisted Extraction (UAE) of Phenolic Compounds

Of the results obtained from conventional extraction, the 10% ethanol in water solvent presented the best results and was therefore used as the solvent in the experimental design for ultrasound-assisted extraction of phenolic compounds. Table 6 shows the levels used and the experimental assays obtained using the RCCD design.

As mentioned previously, the design provided 13 trials associated with the independent variables (time and temperature), which were applied in the ultrasound bath, and the variable response (total phenolic compounds). The design also included five central points to evaluate the repeatability of the data and estimate the experimental error.

As shown in the results obtained for the different trials (Table 6), the total phenolic compound contents varied from 228.48 to 376.89 mg GAE/100 g, representing an increase of 19.4 to 24.8% compared to the 10% ethanol in water extract obtained by the conventional method (Table 5). This increase was expected, as the ultrasound facilitated the cavitation phenomenon, resulting in cell rupture and particle breakage due to the currents generated in the medium, which enhanced the mass transfer rate between the sample and the solvent (Paniwnyk et al. 2009).

From the results obtained, the effects of the factors on the response (total phenolic compounds) were analyzed, as shown in Table 7. It was observed that only the linear and squared terms of the temperature variable were statistically significant at a 5% significance level ($p < 0.05$), indicating that this parameter significantly influenced the total phenolic compound content.

Table 6 RCCD experimental design matrix of two independent variables and the response of total phenolic compound content

Assays	Time (min) X_1	Temperature (°C) X_2	TPC (mg GAE/100 g)
1	15 (−1)	30 (−1)	354.64
2	55 (1)	30 (−1)	374.90
3	15 (1)	60 (1)	337.53
4	55 (−1)	60 (1)	348.20
5	6.7 (−1.41)	45 (0)	352.41
6	63.3 (1.41)	45 (0)	352.66
7	35 (0)	23.8 (−1.41)	362.52
8	35 (0)	66.2 (1.41)	228.48
9 (C)	35 (0)	45 (0)	376.89
10 (C)	35 (0)	45 (0)	364.76
11 (C)	35 (0)	45 (0)	363.75
12 (C)	35 (0)	45 (0)	370.94
13 (C)	35 (0)	45 (0)	373.17

TPC total phenolic compounds, (C) centre point, solvent, 10% ethanol in water

Table 7 Analysis of the effects of the factors of RCCD experimental design of two independent variables for total phenolic compound content (variable response)

Factors*	Regression coefficient	Error	<i>t</i> -value	<i>p</i> -value
Mean	369.90	11.56	32.01	0.0000
(X_1) Time (L)	3.91	9.14	0.43	0.6815
(X_1^2) Time (Q)	− 1.23	9.80	− 0.13	0.9033
(X_2) Temperature (L)	− 29.17	9.14	− 3.19	0.0152
(X_2^2) Temperature (Q)	− 29.17	9.80	− 3.04	0.0189
(X_1 , X_2) 1L by 2L	− 2.40	12.92	− 0.019	0.8580

*Bold for $p < 0.05$. L, linear; Q, quadratic. 95% significance ($p < 0.05$)

Using multiple regression analysis, an equation was fitted to the data, considering only the coefficients that were significant at $p < 0.05$, to obtain the fitted model. This equation described the phenolic compound contents predicted by the method as a function of the coded variables (Rodrigues and Iemma 2014). Thus, the fitted model was characterized by the following equation (Eq. 2):

$$TPC \text{ (mg GAE/100 g)} = 369.90 - 29.17X_2 - 29.17X_2^2 \quad (2)$$

In order to validate the experimental model regarding the total phenolic compound content, the data were evaluated using analysis of variance (ANOVA) (Table 8). The results showed that the model was statistically significant, with R^2 being sufficient, $F_{\text{regression/residues}} \geq F_{\text{tabulated}}$ and $F_{\text{lack of fit/pure error}} \leq F_{\text{tabulated}}$.

The coefficient of determination (R^2) determines the proportion of variation explained by the regression equation in relation to the total variation in the responses. It evaluates how well the predicted data from the statistical program correlate with the experimental data. The higher the R^2 value, the better the model fits the experimental data (Rodrigues and Iemma 2014).

Using this experimental design with two independent variables, an R^2 of 75.52% was found, meaning the equation explained only 75.52% of the total variation in the responses. Upon evaluating the analysis of variance (Table 8), the value for F calculated by the regression ($F_{\text{regression/residues}}$) was 15.4, higher than the $F_{\text{tabulated}}$ value of 4.10. The F value for the lack of fit ($F_{\text{lack of fit/error}}$) was 81.2, higher than the $F_{\text{tabulated}}$

Table 8 Analysis of variance (ANOVA) of the RCCD experimental design with two independent variables for the content of total phenolic compounds

Variation source	Sum of squares	Degree of freedom	Mean square	* F_{cal}	* F_{tab}
Regression	9204.5	2	4602.3	15.4	4.10
Residuals	2983.6	10	298.4	-	-
Lack of fit	2959.3	6	493.2	81.2	6.16
Pure error	24.3	4	6.1	-	-
Total	12,188.2	12	-	-	-

* F_{cal} , calculated values of F ($F_{\text{regression/residues}}$ or $F_{\text{lack of fit/pure error}}$); F_{tab} , tabulated values of F at $p < 0.05$; $R^2 = 75.52\%$

value of 6.16. Since the calculated F value for the lack of fit was greater than the tabulated F and the R^2 was not very high (about 75.52%), it was concluded that the model did not fit the experimental data very well. The phenolic compound contents obtained were very similar across all trials, indicating that within the studied range, the independent variables did not have a strong effect on the responses.

Optimization of the Ultrasound-Assisted Extraction (UAE) of Phenolic Compounds

A second rotational central composite design (RCCD) was conducted to improve the experimental model with respect to the variable response (total phenolic compounds). Three independent variables (time, temperature, and solid-solvent ratio) were used, resulting in 19 trials with five central points to evaluate the repeatability of the data and estimate the experimental error. Table 9 shows the levels used and the experimental assays obtained using this design.

Using this design, greater variation was observed between the responses of the trials, ranging from 333.9 to 450.4 mg GAE/100 g, representing an increase of 15.09 to 37.05% compared to the 10% ethanol extract obtained by the conventional method (Table 5). With the addition of one more independent variable (solid-solvent ratio), the variation increased from 16.32 to 31.57%, compared to the results obtained with the experimental design involving only two variables. Regarding the central points, a small variation was observed between the five assays, indicating good repeatability of the process.

The factors presented in Table 10 were evaluated, and it was found that the linear term (L) of the variable time, as well as the linear and squared (Q) terms of the variables temperature and solid-solvent ratio, were statistically significant at a 5% significance level ($p < 0.05$). This indicates that all the independent variables had an influence on the extraction of the phenolic compounds. Therefore, the fitted model with the statistically significant terms was characterized by the following equation (Eq. 3):

$$TPC \text{ (mg GAE/100 g)} = 403.96 + 9.93X_1 - 5.47X_2 - 8.09X_2^2 + 33.07X_3 - 4.80X_3^2 \quad (3)$$

The experimental model with three variables was validated by an analysis of variance, presented in Table 11, with a coefficient of determination $R^2 = 96.315\%$ (indicating that the model explained 96.315% of the total variation in the responses). The F value calculated for the regression $R^2 = 67.9$ was greater than the F tabulated of 3.03, and the value for F lack of fit of 3.06 was smaller than the value F tabulated of 6.00.

Thus, the model was considered valid within the 95% confidence interval and fitted the experimental data well, suggesting satisfactory agreement between the obtained responses and the predicted values. Therefore, the equation of the fitted model could reliably predict the ideal conditions for the extraction of phenolic compounds.

Figure 2, which presents the experimental values alongside those predicted by the fitted model, confirms the model's adequacy. It shows good agreement between the two, with the points being very close to the fitted line.

Table 9 RCCD experimental design matrix of three independent variables and the response of total phenolic compound content

Assays	Time (min) X_1	Temperature (°C) X_2	Solid-solvent ratio (g/mL) $1: X_3$	TPC (mg GAE/100 g)
1	20 (−1)	30 (−1)	20 (−1)	355.3
2	55 (1)	30 (−1)	20 (−1)	381.2
3	20 (−1)	60 (1)	20 (−1)	342.5
4	55 (1)	60 (1)	20 (−1)	350.0
5	20 (−1)	30 (−1)	40 (1)	417.4
6	55 (1)	30 (−1)	40 (1)	441.2
7	20 (−1)	60 (1)	40 (1)	408.6
8	55 (1)	60 (1)	40 (1)	417.5
9	8.0 (−1.68)	45 (0)	30 (0)	383.4
10	66.9 (1.68)	45 (0)	30 (0)	424.7
11	37.5 (0)	19.8 (−1.68)	30 (0)	382.3
12	37.5 (0)	70 (1.68)	30 (0)	383.4
13	37.5 (0)	45 (0)	13.18 (−1.68)	333.9
14	37.5 (0)	45 (0)	46.82 (1.68)	450.4
15 (C)	37.5 (0)	45 (0)	30 (0)	404.5
16 (C)	37.5 (0)	45 (0)	30 (0)	401.2
17 (C)	37.5 (0)	45 (0)	30 (0)	411.2
18 (C)	37.5 (0)	45 (0)	30 (0)	398.9
19 (C)	37.5 (0)	45 (0)	30 (0)	403.4

TPC, total phenolic compounds; (C), centre point; solvent, 10% ethanol in water

Table 10 Analysis of the effects of the factors of RCCD experimental design of three independent variables for total phenolic compounds content (variable response)

Factors*	Regression coefficient	Error	<i>t</i> -value	<i>p</i> -value
Mean	403.96	3.40	118.91	0.0000
(X_1) Time (L)	9.93	2.06	4.82	0.0009
(X_1^2) Time (Q)	−0.60	2.06	−0.29	0.7784
(X_2) Temperature (L)	−5.47	2.06	−2.66	0.0262
(X_2^2) Temperature (Q)	−8.09	2.06	−3.93	0.0035
(X_3) Solid-solvent ratio (L)	33.07	2.06	16.07	0.0000
(X_3^2) Solid-solvent ratio (Q)	−4.80	2.06	−2.33	0.0445
(X_1, X_2) 1L by 2L	−4.16	2.69	−1.55	0.1560
(X_1, X_3) 1L by 2L	−0.09	2.69	−0.03	0.9748
(X_2, X_3) 1L by 2L	1.44	2.69	0.53	0.6059

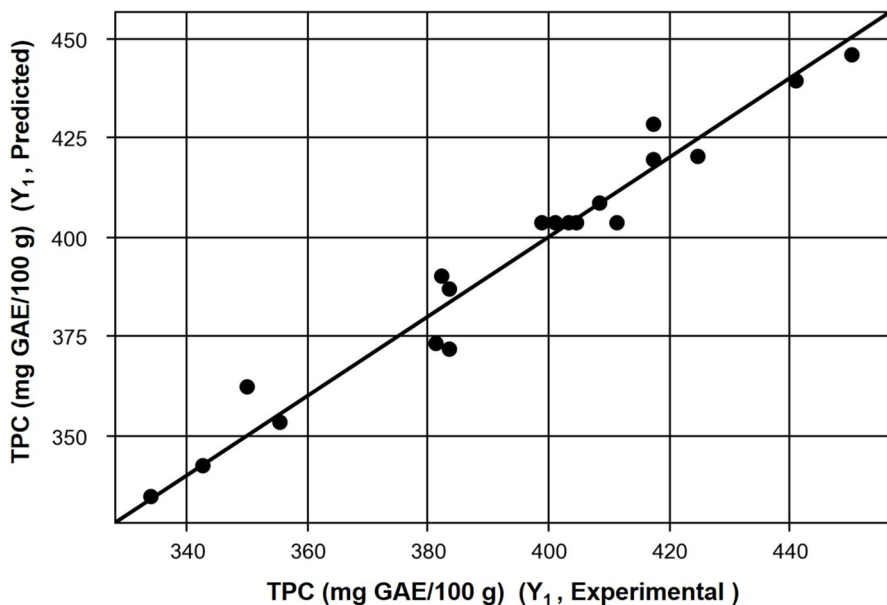
*Bold for $p < 0.05$. L, linear; Q, quadratic. 95% significance ($p < 0.05$)

Table 11 Analysis of variance (ANOVA) of the RCCD experimental design with three independent variables for the content of total phenolic compounds

Variation source	Sum of squares	Degree of freedom	Mean square	* F_{cal}	* F_{tab}
Regression	17,776.03	5	3555.20	67.9	3.03
Residuals	680.615	13	52.35	-	-
Lack of fit	594.44	9	66.05	3.06	6.00
Pure error	86.17	4	21.54	-	-
Total	18,456.64	18	-	-	-

* F_{cal} , calculated values of F ($F_{regression/residues}$ or $F_{lack\ of\ fit/pure\ error}$); F_{tab} , tabulated values of F at $p < 0.05$; $R^2 = 96.315\%$

Figure 3 presents the response surfaces for the total phenolic compound contents as a function of time, temperature, and solid-solvent ratio based on the model proposed by Eq. 3. This analysis allows for the determination of the optimized process conditions to extract the greatest phenolic compound contents from the potato peel flour. Figure 3A shows the effects of time and temperature on the total phenolic compound content, where the longest extraction time and a temperature ranging from 40 to 50 °C in the ultrasound bath (model LS2800, Logen Scientific brand, Diadema, SP, Brazil) resulted in the highest amounts of phenolic compounds extracted.

**Fig. 2** Experimental values and those predicted by the fitted model for the total phenolic compounds (TPC) response

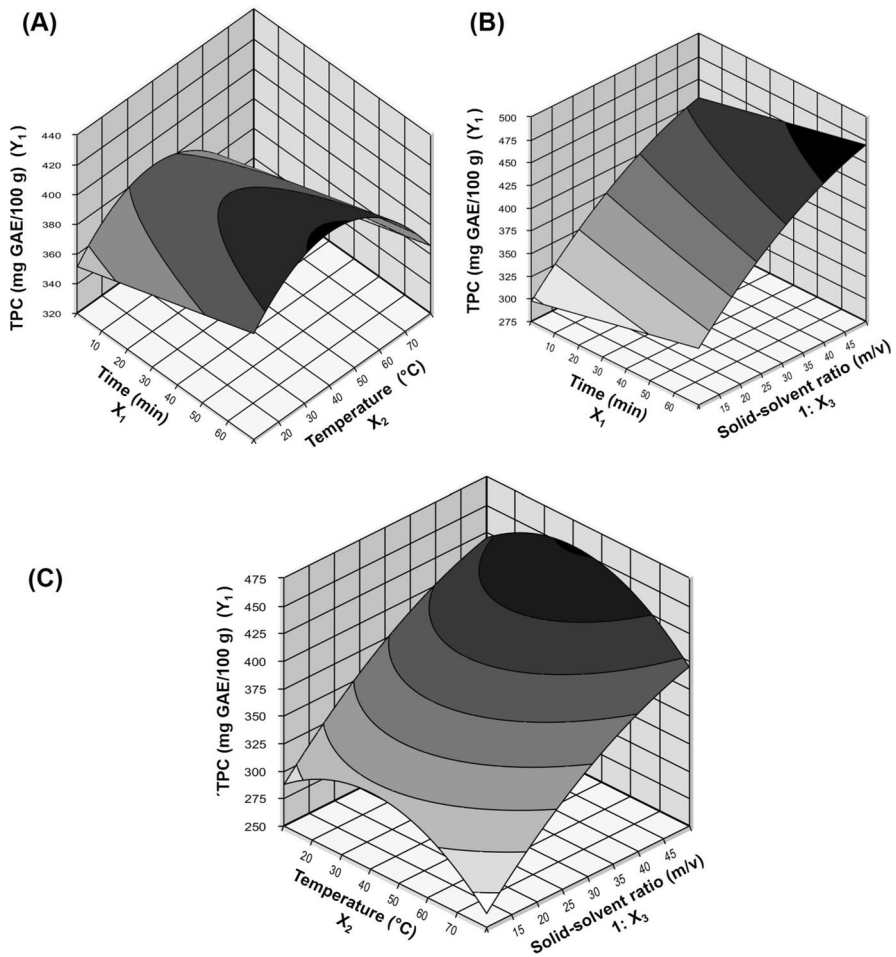


Fig. 3 Response surface plots showing the effect of (A) time versus temperature, (B) time versus solid-solvent ratio, and (C) temperature versus solid-solvent ratio on total phenolic compounds content (TPC)

According to Kazemi et al. (2016), in addition to the ultrasound effect, a longer extraction time would also contribute to the rupture of cell walls, improving the penetration of the solvent into the cells and facilitating the release of larger amounts of phenolic compounds. Wang et al. (2020) evaluated the ultrasound-assisted extraction of phenolic compounds from potato peels and observed that a longer extraction time produced a greater extraction yield, as the release of compounds is a diffusion-controlled process and thus time-dependent.

Živković et al. (2018) stated that temperature also influenced extraction, as higher temperatures weaken the interactions between phenolic compounds and proteins and polysaccharides, thus increasing the rate of diffusion.

Figure 3B shows that to obtain greater extraction of phenolic compounds, a longer extraction time in the ultrasound bath (model LS2800, Logen Scientific brand, Diadem, SP, Brazil) and a higher solid-solvent ratio are necessary. The solvent directly influenced the extraction of phenolic compounds, as a larger amount of solvent penetrated the vegetable cells (Živković et al. 2018). Paleologou et al. (2016) stated that the higher the solid-solvent ratio, the greater the extraction yield, as mass transfer is affected by the concentration gradient between the solid and liquid. A smaller amount of solvent would hinder the transfer of phenolic compounds due to the various equilibria occurring in the solution, which creates resistance to transfer to the solvent.

Figure 3C confirmed the results mentioned above, showing that a temperature range of 40 to 50 °C and a higher solid-solvent ratio resulted in greater extraction of phenolic compounds.

The desirability graph is another tool used to optimize the extraction conditions for recovering phenolic compounds. This graph indicated the combination of factor levels required to achieve the desired optimization, specifying the necessary adjustments to obtain the best process response (Paleologou et al. 2016).

In the present case, the process desirability (Fig. 4) indicated that the maximum phenolic compound content would be achieved with a time of 66.9 min, a temperature of 44.9 °C, and a solid-solvent ratio of 1:46.82 m/v.

To validate the prediction model, and to confirm the desired optimization in relation to the phenolic compounds, ultrasound-assisted extraction was carried out under the predicted optimized conditions. The results are presented in Table 12.

There was a significant increase in phenolic compound extraction when compared with the results obtained using the experimental design with three variables, validating the precision of the model. The phenolic compound content was within the range of values determined by the desirability graph (Fig. 4).

Kumari et al. (2017) extracted phenolic compounds from potato peels using ultrasound-assisted extraction with 80% methanol, obtaining values varying from 217 to 328 mg GAE/100 g. In contrast, Venturi et al. (2019) extracted phenolic compounds using 10% ethanol in water, obtaining 395 mg GAE/100 g, which was lower than the content obtained in the present study.

The antioxidant activity of the extract obtained under the optimized conditions was determined using the DPPH, ABTS, and FRAP methods. As mentioned previously, due to the different mechanisms involved in antioxidant processes and the fact that the methods evaluate different compounds, more than one method should be used to draw a more comprehensive conclusion about antioxidant activity (Albishi et al. 2013).

In the case of the DPPH method, the result remained satisfactory (close to 1 g sample/g DPPH), as only 1.04 g of sample was required to reduce 50% of the DPPH radical. The ABTS method is widely used to evaluate complex mixtures of antioxidant compounds such as plant extracts, beverages, and biological fluids and can estimate the activity of pure compounds, both lipophilic and water-soluble compounds (Nenadis et al. 2004). The quantity of antioxidant compounds determined by the ABTS method showed a 15% increase compared to the extract obtained by conventional means.

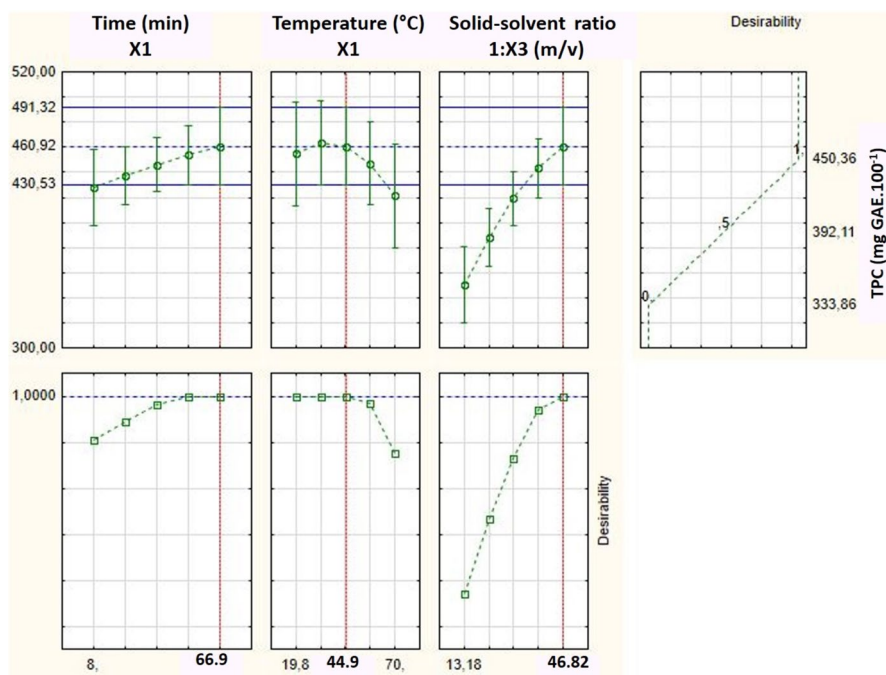


Fig. 4 Profiles for predicted values and desirability for higher total phenolic compounds' content (TPC)

Table 12 Optimal conditions and the experimental response value under these conditions

Optimal conditions			Analysis			
Time (min)	Temperature (°C)	Ratio (g/mL)	TPC	DPPH	ABTS	FRAP
66.9	44.9	1:46.82	461.72 ± 0.82	1.04 ± 0.01	1254.18 ± 1.47	406.71 ± 0.64

Means ± standard deviation of three repetitions. Ratio, solid-solvent ratio (1:X); TPC, total phenolic compounds (mg GAE/100 g); DPPH, EC_{50} (g sample/g DPPH); ABTS, μ M Trolox/g sample; FRAP, μ M Trolox/g sample

Safdar et al. (2017) extracted phenolic compounds from kinnow (*Citrus reticulata* L.) using ultrasound-assisted maceration extraction techniques and obtained 212.9 μ M Trolox/g sample of antioxidant activity by the FRAP method using a 50% ethanol in water solvent. This value was lower than the one found in the present study.

Morphological Analysis Using SEM

Scanning electron microscopy (SEM) was used to observe the morphology of the potato peel flour, the residue obtained after extracting the compounds by

conventional means, and that obtained after ultrasound-assisted extraction under the optimized conditions using a 10% ethanol in water solvent. The results are shown in Fig. 5. This analysis was carried out to observe the effect of ultrasound on the cells of the potato peel flour.

Figure 5A and B show the structures of untreated potato peel flour, revealing the presence of starch granules (red arrows). These granules have an elliptical shape typical of potato starch, with rough, irregular surfaces and variable sizes and forms. According to Kaur et al. (2022), the broken structures observed in Fig. 5A and B indicate an altered protein structure and fragmented crude fiber, which contribute to the release of significant amounts of phenolic compounds.

In Fig. 5B, the surfaces of the potato peel flour cells appear more closed and smooth. However, the residues obtained after extraction by conventional methods (without ultrasound) in Fig. 5C are rougher and exhibit some cracks. Comparing these results with those from the optimized ultrasound-assisted extraction (Fig. 5D), ultrasound caused changes in the cells, forming cavities and increasing surface roughness. This likely facilitated solvent penetration, thereby accelerating the release of phenolic compounds from the potato peels. Such findings are consistent with Wang et al. (2020), who observed that ultrasound-induced cavitation damages cells, creating hollow openings that enhance the extraction process.

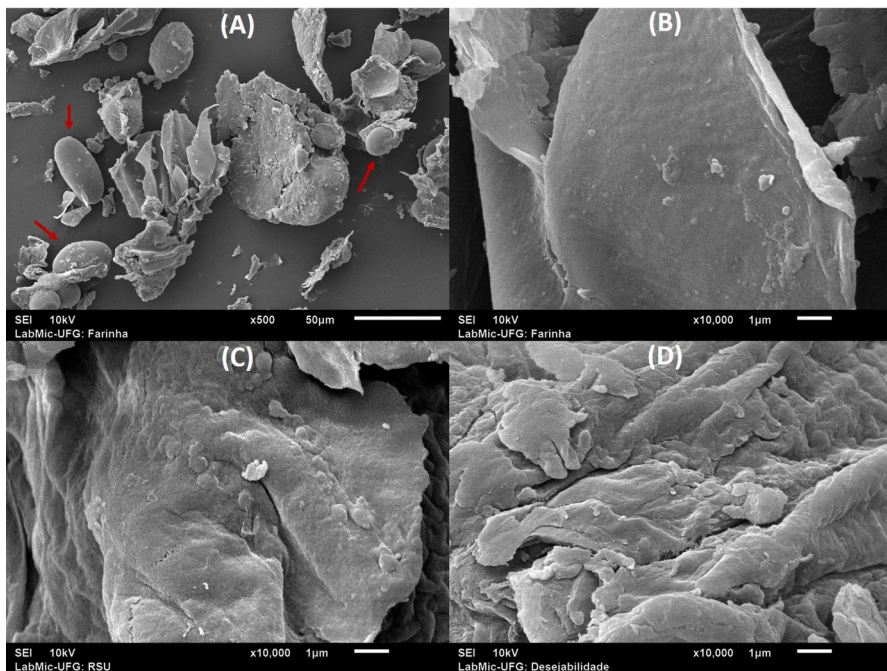


Fig. 5 Scanning electron microscopy (SEM) of potato peel flour (A) and of the residue after extraction (B) without ultrasound (C) and with ultrasound (D)

Conclusions

The present study demonstrates that potato peel can be considered a valuable source of phenolic compounds. The use of 10% ethanol in water as a solvent proved to be the most efficient for extraction. This solvent is ecologically sound, non-toxic, inexpensive, and readily available, making it an ideal choice for sustainable extraction processes.

The response surface methodology (RCCD) experimental design was an effective tool for determining the optimal conditions for phenolic compound extraction. The inclusion of the solid-solvent ratio as a third variable, along with time and temperature, improved the model's fit. Ultrasound-assisted extraction significantly increased extraction efficiency, as evident from the SEM images, which showed that ultrasound caused alterations to the potato peel cells.

The response surface analysis indicated that higher phenolic compound content could be obtained with longer extraction times, a greater solid-solvent ratio, and a temperature range of 40 to 50 °C. These results were validated using a desirability graph and confirmed by the predicted model. This study underscores the potential of ultrasound-assisted extraction to recover valuable phenolic compounds from potato peel residues, offering a promising technology to add value to food processing by-products.

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Data Availability The data generated and/or analyzed during the current study are available from the corresponding author upon reasonable request.

Declarations

Competing Interests The authors declare no competing interests.

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