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Optimization of ultrasound-assisted extraction conditions of total phenolics and anthocyanins from purple onion peel

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ABSTRACT

Purple onion (*Allium cepa L.*) peels**,** an abundant agro-industrial by-product, are particularly rich in flavonoids and anthocyanins with strong antioxidant potential. The effective recovery of these biologically active compounds enables the conversion of plant residues into high value-added products, supporting both clean-label ingredient development approaches and circular bioeconomy strategies. This study aimed to optimize ultrasound-assisted extraction (UAE) conditions to maximize the yield of total phenolic content (TPC) and total monomeric anthocyanins (TMAC) from purple onion peel s. A central composite design based on response surface methodology (RSM) was employed to systematically investigate the effects of four independent variables: ethanol concentration (0-100%)**,** solvent-to-solid ratio (10-60 mL/g)**,** extraction temperature (20-60 °C)**,** and extraction time (30-120 min)**.** The results indicated that solvent composition, temperature, and duration significantly influenced bioactive compound recovery. TPC ranged from 237.06 to 1230.96 mg GAE/100 g, while TMAC varied between 1645.84 and 12357.50 mg/kg. Multi-response optimization identified 95% ethanol, 55 mL/g solvent-to-solid ratio, 40 °C, and 105 min as optimal conditions, which were subsequently validated through confirmatory experiments. Extracts obtained under these optimized conditions exhibited desirable color parameters**,** low water activity values (0.20-0.52), indicating high stability and functional applicability in various formulations. Overall, the integration of UAE with RSM offers an efficient, scalable, and environmentally friendly approach for the extraction of natural pigments and antioxidants from purple onion peels, emphasizing their value as a sustainable ingredient source for food, nutraceutical, and related applications.

1. Introduction

Onion (*Allium cepa L.)* is one of the most important vegetable crops worldwide, with an estimated global production exceeding 98 million tons annually (FAO, 2021). In addition to its nutritional role, onion is recognized as a functional food due to its rich phytochemical profile. It contains a wide spectrum of bioactive compounds, such as flavonoids (notably quercetin and kaempferol), phenolic acids, anthocyanins, organosulfur compounds, and carotenoids, which are associated with diverse biological activities, including antioxidant, anti-inflammatory, antimicrobial, antidiabetic, and anticancer effects (Slimestad et al., 2007; Lee et al., 2014; Nile et al., 2018). Quercetin and its glycosides, for instance, have been linked to cardiovascular protection and anti-hypertensive effects, whereas anthocyanins and betalains are reported to modulate oxidative stress, protect against neurodegenerative disorders, and contribute to the prevention of metabolic diseases (Prakash et al., 2007; He & Giusti, 2010).

During onion processing, substantial quantities of dry outer peels and non-edible layers are generated as waste. These by-products pose environmental and economic challenges but also represent a valuable and underutilized source of phenolic acids and natural pigments. Several studies have demonstrated that onion peels contain significantly higher levels of phenolic compounds than the edible bulb, with purple onion varieties being particularly rich in anthocyanins, flavonols, and betalains (Benitez et al., 2011; Chadorshabi et al., 2022). Consequently, the valorization of onion peel waste has attracted increasing attention as part of global strategies aimed at reducing food waste, enhancing sustainability, and promoting the transition toward a circular bioeconomy (Umeda & Jorge, 2021).

The efficient recovery of bioactive compounds from plant matrices is strongly influenced by the extraction method and operational conditions employed. Conventional extraction

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techniques, including maceration, Soxhlet extraction, and solid-liquid extraction, are often associated with extended processing times, high solvent consumption, and the risk of thermal degradation of heat-sensitive compounds (Bitwell et al., 2023). In contrast, ultrasound-assisted extraction (UAE) has emerged as a promising green extraction technology. UAE utilizes acoustic cavitation to disrupt plant cell walls, enhance solvent penetration, and improve mass transfer, resulting in higher extraction yields with reduced solvent and energy requirements (Shen et al., 2023). Moreover, UAE can be conducted under mild temperature conditions, which helps preserve the stability of phenolic compounds and pigments such as anthocyanins and betalains that are prone to degradation during prolonged thermal treatment (Kaderides et al., 2015; Saikia et al., 2015).

Although the application of UAE has been widely studied in agro-industrial by-products such as pomegranate peel, grape pomace, and fruit peels, systematic optimization for purple onion peel s remains limited. Previous studies have mainly focused on compositional analysis or conventional extraction approaches (Nuutila et al., 2003; Benitez et al., 2011). However, to maximize recovery, it is essential to evaluate the combined effects of multiple process variables, such as solvent type, concentration, temperature, extraction time, and solvent-to-solid ratio, rather than optimizing parameters individually. Response surface methodology (RSM) is a powerful statistical tool that allows such multivariate optimization with minimal experimental runs, enabling the development of predictive models and the identification of optimal extraction conditions (Montgomery, 2013).

The present study addresses this gap by applying RSM to optimize UAE conditions for purple onion peel s. Four independent variables, ethanol concentration, liquid-to-solid ratio, extraction temperature, and extraction time, were systematically evaluated for their effects on total phenolic content (TPC) and total monomeric anthocyanins (TMAC) content. In addition, the physicochemical and functional properties of the optimized extracts, including color parameters, water activity, water- and oil-holding capacity, carr index, were analyzed to assess their stability and potential applicability as natural colorants and functional food ingredients. By integrating an eco-friendly extraction technology with statistical optimization, this study contributes to the sustainable valorization of onion processing by-products. The findings provide insights into the efficient recovery of phenolics and pigments from purple onion peel s, supporting their use in food, nutraceutical, and pharmaceutical applications as natural colorants.

2. Materials and Methods

2.1. Materials

Purple onions (*Allium cepa* L.) were purchased from local markets in Bursa, Turkey, in August 2024. The dry outer peels, considered as agro-industrial by-products, were carefully separated by hand. Non-edible residues such as roots and stems were discarded. The collected peels were thoroughly washed with distilled water to remove surface impurities and then dried in a laboratory oven (MEMMERT-UN55, Germany) at 45 °C for 2 h. The dried samples were subsequently ground into a fine powder using a laboratory grinder and stored in airtight containers until further use. All chemicals and standards used in the analyses were of analytical grade and purchased from reputable suppliers.

2.2. Optimization of purple onion peel powder

The optimization of extraction conditions of purple onion (*Allium cepa* L.) peel powder was prepared using the Central Composite Design (CCD) based on Response Surface Methodology (RSM) through the Design-Expert software (Version 11.0.5.0, Stat-Ease Inc., Minneapolis, USA). The design type is a quadratic (second-degree) model, consisting of 27 randomized runs without blocks, in which four numerical variables, ethanol concentration (A), temperature (B), time (C), and solvent-to-solid ratio (D), were investigated at five levels. The coded and actual values of the variables and design model are presented in Table 1. The experimental design includes factorial, axial, and center points, allowing for the determination of both linear and quadratic (second-degree) effects of the variables as well as their interactions. In this study, total phenolic content (TPC) and total anthocyanin content were evaluated as response variables. The suitability and statistical significance of the model were assessed by analysis of variance (ANOVA); the significance level of the coefficients was determined based on the criterion of $P<0.05$. Response surfaces and optimization graphs were generated using the Design-Expert (v11.0.5.0) software. Briefly, purple onion peels were dried, milled, and sieved, and 5 g of the resulting peel powder was mixed with acidified aqueous ethanol at concentrations ranging from 0-100%, v/v as specified by the experimental design. The volume of solvent was adjusted in each run to achieve the desired solvent-to-solid ratio (10-60 mL/g), while extraction temperature (20-60 °C) and time (30-120 min) were varied according to the central composite design. The suspensions were placed in 250-mL Erlenmeyer flasks and subjected to ultrasound-assisted extraction (UAE) in an ultrasonic bath (WiseClean WUC-D06H, Daihan, South Korea) at 40 kHz. At the end of each extraction period, samples were rapidly cooled to room temperature (25 ± 1 °C) and centrifuged at 8694 × *g* for 10 min in a refrigerated centrifuge (Nüve NF 3000R, Ankara, Türkiye). The supernatants were vacuum-filtered at 25 °C to obtain clear extracts. Following clarification, the liquid extracts were frozen at -20 °C and subsequently freeze-dried using a laboratory lyophilizer (TeknoSEM, Turkey) to obtain dry extract powders. The lyophilized extracts were stored in amber glass bottles at 5 ± 1 °C until analysis.

Table 1. The coded and actual values of the variables

Factor	Name	Units	Type	Minimum	Maximum	Coded Low	Coded High	Mean	Std. Dev.
A	Concentration	%	Numeric	0	100	-1 ↔ 5.00	+1 ↔ 75.00	48.15	23.94
B	Temperature	°C	Numeric	20	60	-1 ↔ 30.00	+1 ↔ 50.00	40.00	9.61
C	Time	Min	Numeric	30	120	-1 ↔ 52.50	+1 ↔ 97.50	75.00	21.62
D	Solvent:Solid Ratio	-	Numeric	10	60	-1 ↔ 22.50	+1 ↔ 47.50	35.00	12.01

2.3. Determination of bioactive compounds in purple onion peel extracts

Extractable fractions of purple onion peel powder were prepared for the determination of bioactive compounds following the procedure described by Singleton et al. (1999), with minor modifications. Briefly, 1 g of powdered sample was mixed with 20 mL of methanol-water (70:30, v/v) and shaken on an orbital shaker at 20 °C for 2 h. The mixture was then centrifuged at 2500 × *g* for 10 min, and the supernatant was collected as the extractable fraction.

Total phenolic content (TPC) was determined using the Folin–Ciocalteu method (Singleton et al., 1999). Briefly, diluted extract samples or gallic acid standards were reacted with an alkaline copper solution, followed by the addition of diluted Folin-Ciocalteu reagent. After incubation in the dark at room temperature, the absorbance was measured at 750 nm using a UV-Vis spectrophotometer (Rigol/3660UV, China). Distilled water was used as the blank, and gallic acid solutions (5-500 mg/L) were employed to construct the calibration curve. TPC values were expressed as milligrams of gallic acid equivalents (mg GAE) per gram of dry weight.

The total monomeric anthocyanin content (TMAC) of purple onion peel extracts was quantified using the pH differential method (AOAC, 2005) with slight modifications. In this approach, the samples were diluted separately in two buffer systems: potassium chloride buffer (25 mM, pH 1.0) and sodium acetate buffer (400 mM, pH 4.5). Absorbance measurements were recorded at 520 and 700 nm using a UV-Vis spectrophotometer (Inesa L6S, Shanghai, China). TMAC was expressed as cyanidin-3-glucoside equivalents, based on the Eq. 1:

$$\mathrm{TMAC}\ \left(\frac{\mathrm{mg}}{\mathrm{L}}\right) = \frac{\mathrm{A}*\mathrm{Mw}*\mathrm{DF}*1000}{\varepsilon*\mathrm{l}} \quad (1)$$

where A represents the absorbance difference calculated as; $A = (A\lambda520 - A\lambda700)\mathrm{pH}\ 1{,}0 - (A\lambda520 - A\lambda700)\mathrm{pH}\ 4{,}5$; M_W is the molecular weight of cyanidin-3-glucoside (449.2 Da); DF is the dilution factor; ε is the molar extinction coefficient (26,900 1/M cm); and l is the path length (1 cm). Results were reported as mg cyanidin-3-glucoside equivalents per g of dry weight.

2.4. Characterization of purple onion peel extracts

The moisture content of the freeze-dried purple onion peel extracts was determined gravimetrically. Samples were dried in a convection oven at 105 °C until a constant weight was achieved, and the results were expressed as a percentage of dry matter (AOAC, 2005). Water activity (aw) of the purple onion peel extracts was measured at 25 °C with a water activity meter (Novasina-Labmaster, Sweeden). The visual and instrumental color properties of the powders were assessed using a colorimeter (PCE-CSM3, PCE Deutschland), recording L^* (lightness), a^* (red-green), and b^* (yellow-blue) values. These parameters provide quantitative information on the pigment intensity and hue.

Water- and oil-holding capacity (WHC and OHC) of the purple onion peel extracts were determined using a gravimetric method. Briefly, 50 mg of powdered extract was placed into a Falcon tube and weighed. To assess WHC, 1.5 mL of distilled water was added to the tube, whereas for OHC, 1.5 mL of sunflower oil was used. The mixtures were vortexed for 20 s and incubated in a water bath at 30 °C for 30 min. Subsequently, the tubes were centrifuged at 9000 rpm for 10 min at 40 °C. After centrifugation, the supernatant was carefully removed, and the tubes were weighed again. The water- and oil-holding capacities were calculated using the following equation (Eq. 2 and Eq. 3, respectively), and the results were expressed as grams of liquid retained per gram of powder (Elsebaie & Essa, 2018).

$$\mathrm{WHC}(\%) = \frac{\text{Mass of water absorbed (g)}}{\text{Mass of sample(g)}} * 100 \quad (2)$$

$$\mathrm{OHC}(\%) = \frac{\text{Mass of oil absorbed (g)}}{\text{Mass of sample(g)}} * 100 \quad (3)$$

Purple onion peel extracts were analyzed for bulk and tapped density as described by Turchiuli et al. (2005). Powder cohesiveness and flowability based on Carr index (Eq. 4),

$$\text{Carr index} = \frac{\text{Tapped density} - \text{Bulk density}}{\text{Tapped density}} * 10 \quad (4)$$

2.5. Statistical Analysis

The experimental data were statistically evaluated using response surface methodology (RSM) for optimization purposes with the aid of *Design-Expert* software (version 11, Stat-Ease Inc., Minneapolis, MN, USA). The statistical significance of all terms in the polynomial model was assessed at $P<0.05$. The adequacy of the fitted model was verified by calculating the coefficient of determination (R^2). The relationships between the responses and the independent variables were further illustrated through response surface plots.

3. Results and Discussion

3.1. Model performance and influence of extraction parameters

In this study, a Central Composite Design (CCD) involving four independent variables (ethanol concentration, temperature, time, and solvent-to-solid ratio) was applied to maximize the recovery of phenolic compounds and anthocyanins from purple onion peels, and a total of 27 conditions were evaluated. The resulting data were analyzed using second-degree polynomial models, and models with high explanatory power ($R^2 > 0.95$) were obtained for both total phenolic content (TPC) and total monomeric anthocyanin content (TMAC). The significance of the models was confirmed by ANOVA, and the non-significant lack-of-fit values for all responses ($P>0.05$) indicated that the model fit well with the experimental data. According to the ANOVA results, which examined the individual and interaction effects of the parameters, the most decisive factor was ethanol concentration. Depending on the response type, certain interactions among temperature, extraction time, and solvent-to-solid ratio followed. In particular, the significant interactions of ethanol × temperature and ethanol × time suggest that optimal extraction is determined by a complex mechanism involving solvent polarity, mass transfer, and compound stability, rather than by a single variable. The complete experimental matrix and response outcomes obtained from Design-Expert are summarized in Table 2.

Table 2. The complete experimental matrix and analysis results

RUN	A: Concentration	B: Temperature	C: Time	D: Solvent:Solid Ratio	Anthocyanin Matter (mg/kg)	Total Phenolic Matter (mg GAE/100 g)
1	75	50	52.5	47.5	10636.80	1070.00
2	100	40	75	35	12357.50	1062.18
3	25	30	52.5	47.5	2928.48	428.137
4	25	50	97.5	22.5	3673.46	337.503
5	50	60	75	35	6572.26	546.648
6	50	40	75	35	5424.97	532.558
7	50	20	75	35	6399.26	467.679
8	25	30	97.5	47.5	4499.81	385.626
9	75	30	52.5	47.5	6788.18	705.284
10	75	30	52.5	22.5	7325.51	623.264
11	25	50	97.5	47.5	4699.69	448.636
12	25	30	52.5	22.5	3951.12	393.58
13	75	30	97.5	47.5	8376.95	671.234
14	25	50	52.5	47.5	4871.82	397.251
15	25	30	97.5	22.5	2660.88	423.818
16	75	50	97.5	47.5	11535.5	1230.96
17	50	40	75	60	7251.99	655.066
18	75	50	52.5	22.5	5512.26	465.102
19	50	40	120	35	6402.19	610.006
20	50	40	75	10	3957.3	292.617
21	50	40	75	35	6413.29	595.915
22	0	40	75	35	1645.84	237.058
23	25	50	52.5	22.5	2572.96	260.508
24	50	40	30	35	5211.05	509.293
25	75	50	97.5	22.5	7462.65	546.398
26	50	40	75	35	5808.37	605.343
27	25	30	97.5	22.5	2660.88	552.426

Comparable patterns have been documented across different plant by-products. For example, Kaderides et al. (2015) reported that ethanol concentration and extraction duration were the key factors controlling phenolic yield from pomegranate peel, while González-Centeno et al. (2015) emphasized the importance of solvent polarity and extraction time in grape pomace extraction, particularly for flavonols and proanthocyanidins. Similarly, Pinelo et al. (2005) observed that solvent polarity critically affected both yield and selectivity in grape skin phenolic extraction, indicating that optimization cannot be achieved without careful solvent adjustment. These studies collectively corroborate the findings of the present work, suggesting that bioactive recovery from onion peels follows trends comparable to those observed in other phenolic-rich residues.

In ultrasound-assisted extraction (UAE) systems, the interaction between ethanol concentration, temperature, and time becomes even more crucial due to the dual role of ultrasound: cavitation-induced cell disruption and enhanced solvent penetration. Chemat et al. (2017) demonstrated that cavitation efficiency is closely related to solvent properties, particularly ethanol fraction and vapor pressure, which influence bubble dynamics. At lower ethanol concentrations, cavitation intensity increases, but reduced phenolic solubility may limit recovery. Conversely, higher ethanol contents improve solubility yet decrease cavitation efficiency, highlighting the importance of identifying a balance point. Similar findings were reported by Nayak et al. (2015) for citrus sinensis peel, where optimal ethanol ratios enhanced flavonoid yield without compromising cavitation effects.

Temperature and extraction time also play a decisive role, not only in facilitating mass transfer but also in determining compound stability. Moderate heating (40-50 °C) has been shown to enhance phenolic solubilization (Rodríguez-Juan et al., 2021), whereas prolonged exposure to higher temperatures accelerates the degradation of anthocyanins, flavonols, and catechins (Tian et al., 2021). In the current study, this trade-off was reflected in the decline of total phenolic content under harsher conditions, consistent with previous observations in pomegranate peel (Viuda-Martos et al., 2012) and tea leaves (Tian et al., 2021). These findings highlight the delicate balance between maximizing extraction efficiency and preventing thermal or oxidative degradation of labile compounds.

The findings of this study demonstrate that the efficient recovery of phenolic compounds from purple onion peels requires a balanced regulation of ethanol concentration, extraction temperature, and duration, together with their interactive effects. In line with observations from other agro-industrial by-products such as grape pomace, citrus peels, and pomegranate residues, onion peels exhibit considerable potential for valorization when subjected to carefully optimized UAE conditions. Moreover, the application of RSM proved valuable not only for determining optimal extraction parameters but also for elucidating parameter interactions, thereby providing a robust framework for the development and scale-up of sustainable extraction processes aimed at functional food and nutraceutical applications.

3.2. Total phenolic content (TPC)

The TPC values obtained under different extraction conditions ranged from 237.06 to 1230.96 mg GAE/100 g, clearly demonstrating that the parameters have a strong effect on the solubility and stability of phenolics. The highest TPC value was obtained with 75% ethanol, at 50 °C, for 97.5 min, and at a solvent-to-solid ratio of 47.5. This result indicates that moderate temperatures combined with moderately polar solvent compositions increase both the solubility and diffusion rate of phenolic compounds as such conditions enhance mass transfer and solvent–solute interactions while preventing thermal degradation of phenolic structures (Cacace & Mazza, 2003).

ANOVA analysis (Table 3) revealed that ethanol concentration had a highly significant effect on TPC ($P<0.0001$). The solvent-to-solid ratio was also determined to be an important factor; although the individual effects of temperature and time were limited, significant interaction terms made them decisive within the process. Since phenolic compounds are generally known to dissolve more effectively in solvents with medium to high polarity, ethanol mixtures of 50-75% provided optimal polarity. Completely aqueous solvents limited the solubility of phenolic compounds, while pure ethanol solvents did not provide a suitable environment for polar phenolic acids. Based on the ANOVA results, in which ethanol concentration (A) and solvent-to-solid ratio (D) as well as the interactions A×B (Figure 1a), A×D (Figure 1b), and B×D (Figure 1c) were found to be significant for the TPC response, the graphs particularly illustrate the combined effects of these variables on the response. On 3D surfaces created for TPC, it was observed that an increase in ethanol concentration significantly enhanced the solubility of phenolics, while a decrease in the solvent-to-solid ratio facilitated mass transfer and increased extraction efficiency. The ethanol × temperature interaction surface demonstrated that moderate temperatures (40-50 °C) maximized TPC by enhancing the solvent effectiveness of ethanol. In the ethanol × solvent-to-solid ratio graph, it was found that a high percentage of ethanol combined with a low solvent volume synergistically increased phenolic recovery. Benítez et al. (2011) reported 1200-1350 mg GAE/100 g in dry onion peel extracts obtained using aqueous ethanol, while Nuutila et al. (2003) documented slightly lower values (~900 mg GAE/100 g) using conventional maceration. Moreover, the phenolic content of purple onion peels was higher than that reported for red cabbage leaves (approximately 850-1000 mg GAE/100 g) (Podsedek et al., 2008) and comparable to pomegranate peel (1100-1500 mg GAE/100 g) (Li et al., 2006). This evidence highlights purple onion peel s as a competitive and low-cost source of dietary phenolics with substantial potential for functional food and nutraceutical applications. The results obtained in this work align well with established findings in related research. Sasongko et al. (2020) reported that increasing temperature up to 40 °C enhanced TPC, while further increases led to declines due to degradation of heat-sensitive compounds. Similarly, Kaderides et al. (2015) observed that UAE of pomegranate peel benefited from moderate heating (25-35 °C), but higher temperatures reduced yields by weakening cavitation intensity. Rodríguez-Juan et al. (2021) further demonstrated degradation of quercetin glycosides above 50 °C, while Zeng et al. (2016) reported catechin losses under prolonged heating in tea leaves. These findings explain the decrease in TPC under harsher conditions in the present study. Moreover, ethanol concentration exhibited a bell-shaped effect, consistent with Spigno et al. (2007), who showed that intermediate-to-high ethanol fractions (50-80%) maximize flavonoid solubility, while excessive concentrations (>90%) reduce extraction due to limited solubility of polar phenolic acids. The role of ultrasound cavitation should also be emphasized. Cavitation enhances cell wall disruption, solvent penetration, and mass transfer, resulting in higher phenolic recovery compared with conventional techniques (Chemat et al., 2017). However, its efficiency is strongly influenced by solvent composition, vapor pressure, and temperature, which explains the sensitivity of TPC to process parameters in the present study.

Table 3. ANOVA table for TPC

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1.414E+06	14	1.010E+05	17.30	< 0.0001	significant
A-Concentration	7.310E+05	1	7.310E+05	125.22	< 0.0001	significant
B-Temperature	17469.99	1	17469.99	2.99	0.1092	
C-Time	10674.15	1	10674.15	1.83	0.2012	
D-Solvent:Solid Ratio	2.260E+05	1	2.260E+05	38.72	< 0.0001	significant
AB	57457.43	1	57457.43	9.84	0.0086	significant
AC	143.42	1	143.42	0.0246	0.8781	
AD	96023.94	1	96023.94	16.45	0.0016	significant
BC	10281.70	1	10281.70	1.76	0.2092	
BD	1.138E+05	1	1.138E+05	19.50	0.0008	significant
CD	128.65	1	128.65	0.0220	0.8844	
A^2	8903.37	1	8903.37	1.53	0.2405	
B^2	4882.53	1	4882.53	0.8364	0.3784	
C^2	87.69	1	87.69	0.0150	0.9045	
D^2	11727.10	1	11727.10	2.01	0.1818	
Residual	70048.50	12	5837.37			
Lack of Fit	58644.94	9	6516.10	1.71	0.3592	not significant
Pure Error	11403.56	3	3801.19			
Cor Total	1.484E+06	26				
R^2	0.9528					
Adjusted R^2	0.8977					
Predicted R^2	0.7375					

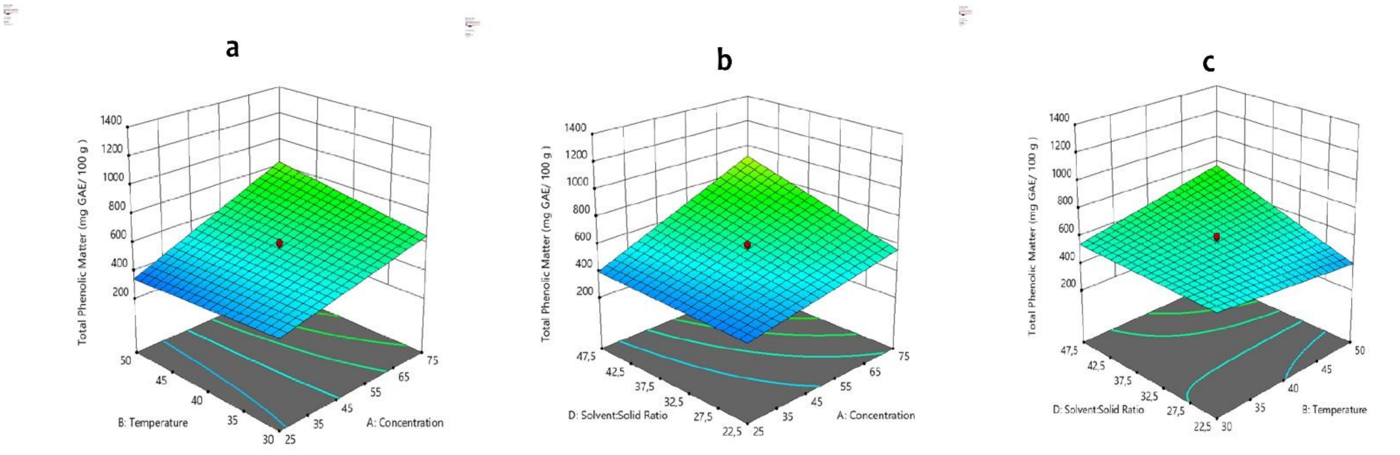

Figure 1. Response surface plots showing the significant effects of extraction variables on Total Phenolic Content (TPC); a) effect of ethanol concentration (%) and temperature (°C), b) effect of ethanol concentration (%) and solvent-to-solid ratio, c) effect of temperature (°C) and solvent-to-solid ratio.

3.3. Total monomeric anthocyanin content

TMAC values ranged between 1645.84 and 12357.50 mg/kg, and it was observed that the extraction yield of anthocyanins was highly sensitive to both the solvent composition and the thermal conditions. The highest TMAC level was obtained with 95% ethanol, at 40 °C, for 75 min, and at a solvent-to-solid ratio of 35 mL/g.

The ANOVA results (Table 4) also showed that ethanol concentration was a determining factor for TMAC (P<0.0001). Due to the anthocyanins exhibiting both polar and semi-polar characteristics, optimal dissolution occurs in a specific ethanol-water mixture. The determination of 95% ethanol as optimal clearly demonstrates the balance between solvent polarity and anthocyanin solubility. In the model for TMAC, only ethanol concentration (A), solvent-to-solid ratio (D), and the interactions A×D (Figure 2a) and B×D (Figure 2b) were found to be significant, so the 3D graphs focused on these factors. It is clearly seen from the graph that anthocyanins are more stable and soluble at high ethanol percentages and at moderate solvent-to-solid ratios. The ethanol × solvent-to-solid ratio graph reveals that a high percentage of ethanol maximizes anthocyanin extraction when solvent volume is low, but as the solvent volume increases too much, pigment stability decreases.

These results are consistent with earlier findings. He et al. (2016) demonstrated that extraction time and temperature critically affect anthocyanin recovery in blueberry by-products, where moderate heating improved solubility but excessive heat accelerated degradation. Similarly, González-de-Peredo et al. (2021) reported that maintaining extraction temperatures between 40-50 °C preserved anthocyanin stability, while higher levels promoted structural breakdown. In line with these studies, our findings highlight that purple onion anthocyanins can be effectively extracted under carefully optimized UAE conditions, particularly at moderate ethanol concentrations and controlled temperatures, supporting their potential valorization as a natural source of bioactive pigments.

Table 4. ANOVA table for TMAC.

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1.805E+08	14	1.290E+07	19.11	< 0.0001	significant
A-Concentration	1.303E+08	1	1.303E+08	193.06	< 0.0001	significant
B-Temperature	2.494E+06	1	2.494E+06	3.70	0.0786	
C-Time	2.237E+06	1	2.237E+06	3.31	0.0937	
D-Solvent:Solid Ratio	1.568E+07	1	1.568E+07	23.23	0.0004	significant
AB	1.727E+05	1	1.727E+05	0.2560	0.6221	
AC	1.389E+06	1	1.389E+06	2.06	0.1770	
AD	1.265E+06	1	1.265E+06	1.87	0.1960	
BC	3.333E+05	1	3.333E+05	0.4938	0.4956	
BD	7.879E+06	1	7.879E+06	11.67	0.0051	significant
CD	1.519E+05	1	1.519E+05	0.2250	0.6438	
A^2	1.076E+06	1	1.076E+06	1.59	0.2306	
B^2	1.959E+05	1	1.959E+05	0.2903	0.5999	
C^2	1.158E+05	1	1.158E+05	0.1716	0.6860	
D^2	3.286E+05	1	3.286E+05	0.4868	0.4987	
Residual	8.099E+06	12	6.749E+05			
Lack of Fit	7.602E+06	9	8.447E+05	5.10	0.1035	not significant
Pure Error	4.966E+05	3	1.655E+05			
Cor Total	1.886E+08	26				
R^2	0.9571					
Adjusted R^2	0.9070					
Predicted R^2	0.7628					

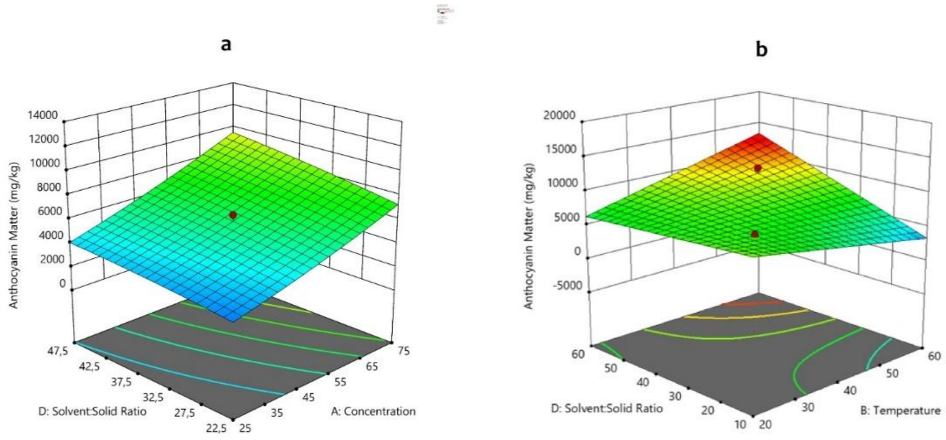

Figure 2. Response surface plots showing the significant effects of extraction variables on Total Monomeric Anthocyanin Content (TMAC); a) effect of ethanol concentration (%) and extraction time (min), b) effect of ethanol concentration (%) and solvent-to-solid ratio

3.4. Optimization and validation

According to constraints that are given in Table 5, 100 solutions were created and a desirability function was used to simultaneously maximize TPC and TMAC, resulting in three different optimum points. These three optimum conditions were evaluated; in particular, the combination of 95% ethanol - 40 °C - 105 min - 55 mL/g yielded the highest overall desirability value (d>0.9).

In the validation experiments, the measured values for both TPC and TMAC fell within the 95% confidence intervals predicted by the model, indicating that the regression model has high predictive power and that the RSM approach can be reliably used in industrial-scale extraction design. This validation demonstrates that, given the interaction-based nature of ultrasonic-assisted extraction involving multiple parameters, the optimization model accurately reflects both the mechanical and chemical processes. Similar interaction patterns have been observed in RSM optimizations of phenolics from grape pomace (Gonzalez-Centeno et al., 2015) and anthocyanins from black currant (Cacace & Mazza, 2003).

3.5. Physicochemical analysis of optimized extracts

Physicochemical analyses were conducted for all experimental combinations; however, only the results obtained under the optimized extraction conditions are presented in Table 6. The evaluation of these properties is essential for assessing product stability and potential industrial applicability. Accordingly, key quality indicators including color parameters, water activity, water- and oil-holding capacity, and flow properties were analyzed, demonstrating the suitability of the purple onion peel extracts as functional food ingredients.

Color characteristics

Color properties are important quality indicators of phenolic extracts, as they are closely related to pigment composition, extraction efficiency, and chemical stability. Color parameters, particularly those associated with anthocyanins and other colored polyphenols, provide rapid insight into oxidative degradation and structural integrity of phenolic compounds (Giusti & Wrolstad, 2001; He & Giusti, 2010). In addition, color characteristics influence the technological applicability of phenolic extracts as natural colorants and functional ingredients, directly affecting formulation compatibility and consumer acceptance in food and cosmetic products (Castañeda-Ovando et al., 2009). Therefore, color evaluation is essential for assessing both functional quality and application potential of phenolic extracts.

The color properties of purple onion peel extracts obtained under different extraction conditions were evaluated using the CIELAB color space (L^*, a^*, b^*). Lightness (L^*) values ranged from 11.32 to 14.76, indicating the formation of intensely colored, dark-hued extracts. The a^* values, representing red-green chromaticity, varied between 9.21 and 14.77, confirming the predominance of red tones typical of anthocyanin-rich matrices. Meanwhile, b^* values (blue-yellow chromaticity) ranged from 1.97 to 3.46, showing minor contributions of yellow hues that may originate from flavonols or oxidized phenolics.

The minimum L^*, a^*, and b^* values were obtained at 97% ethanol, 60 °C, and 100 min, whereas the highest color intensity was observed at 98% ethanol, 54 °C, and 110 min. Statistical evaluation significant differences among the optimization points for all three color coordinates (P<0.05). A gradual increase in lightness (L^*), redness (a^*), and yellowness (b^*) was evident from OP1 to OP3, indicating that moderate extraction conditions favored pigment stability and chromatic enhancement (Table 6). These findings are consistent with the results of Lee et al. (2014), who reported that both ethanol concentration and extraction temperature exert a pronounced influence on the color strength and hue characteristics of onion peel extracts, primarily through their effects on pigment solubility and thermal degradation dynamics.

The correlation between extraction conditions and chromatic attributes can be explained by the solubility and stability behavior of anthocyanins. Moderate ethanol levels (around 90-95%) promote efficient pigment extraction by facilitating the solubilization of both polar and semi-polar anthocyanins, whereas excessive temperatures accelerate pigment degradation through hydrolysis and oxidation (He & Giusti, 2010; Saikia et al., 2015). Similarly, Lee et al. (2014) observed that anthocyanin-rich extracts from red onions exhibited maximal color intensity at moderate ethanol concentrations and low thermal exposure. These results collectively suggest that controlling solvent polarity and thermal input is essential to preserving the chromatic quality of anthocyanin-containing extracts.

In comparison with other natural pigment sources, the a* values recorded in this study are comparable to those reported for red cabbage (10-16) (Cacace & Mazza, 2003) and higher than those for purple corn (8-10) (Yang et al., 2009), confirming that purple onion peel s possess a high intrinsic color potential suitable for natural colorant applications.

Table 5. Optimization constraints and the top-ranked solutions generated by the desirability function.

Constraints				
Name	**Goal**	**Lower Limit**	**Upper Limit**	**Importance**
Concentration	is in range	0	100	3
Temperature	is in range	20	60	3
Time	is in range	30	120	3
Solvent:Solid Ratio	is in range	10	60	3
Anthocyanin	Maximize	1645	15000	5
Total Phenolic Content	Maximize	237	2000	5

Solutions

Solution	**Conc. (%)**	**Temp (°C)**	**Time (min)**	**S:S Ratio (mL/g)**		**Predicted**	**95% PI**	**Observed**	**Agreement with prediction interval**
2	95	40	105	55		14397.96	10992.10 – 16167.86	12398.10	✓ Within PI
27	98	55	93	40	**Anthocyanin**	13906.17	11298.76 – 16513.58	12577.24	✓ Within PI
39	97	60	100	60		17882.97	14185.95 – 21579.98	13682.82	✓ Within PI
2	95	40	105	55		1355.02	981.81 – 1500.30	1432.34	✓ Within PI
27	98	55	93	40	**Total Phenolics**	1399.19	1137.99 – 1660.40	1086.20	✓ Within PI
39	97	60	100	60		1964.76	1594.40 – 2335.11	1538.60	✓ Within PI

Table 6. Physicochemical analysis results of optimized extracts.

Parameters		**Solution 2 OP1**	**Solution 27 OP2**	**Solution 39 OP3**
	L*	11.32±0.38[c]	13.34±0.23[b]	14.76±0.13[a]
Color	***a****	9.21±0.33[c]	10.97±0.18[b]	14.77±0.11[a]
	b*	1.97±0.79[c]	2.17±0.03[b]	3.46±0.01[a]
Dry matter		89.46±0.18	89.91±0.01	89.82±0.29
a_w		0.34±0.02[b]	0.52±0.01[a]	0.20±0.01[c]
WHC		0.40±0.11[c]	0.64±0.05[a]	0.46±0.22[b]
OHC		1.04±0.18[b]	1.31±0.02[a]	0.99±0.15
Carr index		37.51±0.01[a]	35.05±0.05[b]	33.34±0.02[c]

Dry matter content

Purple onion peel extracts exhibited dry matter contents ranging from 89.29% to 98.52% depending on extraction conditions. The lowest dry matter value was obtained at 97% ethanol, 60 °C, and 100 min, while the highest was at 95% ethanol, 40 °C, and 105 min. These results confirm that lower ethanol concentration and milder temperatures enhance dry matter retention, likely due to reduced solubility of non-polar compounds and lower volatilization of extraction solvent, which prevents mass loss during drying. This finding aligns with Çalışkan & Dirim (2013), who reported that lower extraction temperatures minimized degradation of polysaccharides and proteins, enhancing dry matter yield in fruit and vegetable powders. Similarly, Bae & Lee (2008) indicated that mild extraction preserves structural integrity, reducing the release and loss of solids. The dry matter levels are also consistent with anthocyanin-rich residues such as grape skins and pomegranate peels, which typically range between 90-97% (Spigno et al., 2007), indicating that UAE is an efficient method to concentrate solids while preserving quality.

Water activity (a_w)

Water activity (a_w), a key indicator of microbial and oxidative stability, ranged from 0.20 to 0.52 across all extraction conditions. The lowest value was obtained at 98% ethanol, 54 °C, and 110 min, whereas the highest occurred at 95% ethanol, 40 °C, and 105 min. Statistical analysis revealed significant differences among optimization points ($P<0.05$). All aw values remained below 0.60, indicating that the dried extracts are microbiologically stable and resistant to enzymatic and oxidative degradation.

The low aw values observed here are consistent with previous findings for dehydrated plant extracts, including onion and tomato peels (Çalışkan-Koç & Dirim, 2013; Rodríguez-Roque et al., 2015), where values below 0.60 effectively inhibited microbial growth and maintained pigment stability. Therefore, the extracts obtained under optimal UAE conditions are expected to maintain their quality and stability during long-term storage.

Water- and Oil-Holding Capacity (WHC and OHC)

Water- and oil-holding capacity are key functional properties of phenolic extracts, as they influence hydration behavior, fat binding, and overall performance when incorporated into food systems. High water-holding capacity contributes to texture stability, moisture retention, and shelf-life improvement, while oil-holding capacity enhances flavor retention and lipid stabilization, particularly in fat-rich formulations. These properties are especially relevant for phenolic-rich powders intended as functional ingredients, as they affect processability and product quality (Elleuch et al., 2011).

The water-holding capacity (WHC) of purple onion peel powders ranged from 0.40 to 0.64 g water/g powder, while the oil-holding capacity (OHC) varied between 0.99 and 1.31 g oil/g powder, depending on the extraction conditions. The highest WHC (0.64±0.05 g/g) and OHC (1.31±0.02 g/g) were recorded at OP2, which corresponds to relatively milder extraction parameters, particularly lower ethanol concentration and temperature. This behavior can be attributed to the preservation of structural integrity and surface-active components such as proteins, polysaccharides, and phenolic complexes, which provide hydrophilic and hydrophobic binding sites for water and oil molecules, respectively. Under moderate extraction conditions, reduced solvent polarity and limited thermal degradation prevent the collapse of the porous matrix and maintain the accessibility of these binding domains. In contrast, higher ethanol concentrations and elevated temperatures may lead to partial collapse of the porous matrix and denaturation of hydrophilic biopolymers, ultimately reducing WHC and OHC. This behavior is consistent with findings by Bae & Lee (2008), who reported that intense extraction conditions disrupt the structural integrity of plant-based powders, diminishing their fluid-binding capabilities.

Regarding OHC, the values observed in this study are comparable to those reported for other anthocyanin-rich plant by-products. Kaderides et al. (2015), for instance, found OHC values between 1.10 and 1.35 g/g for pomegranate peel powders, attributing these to the presence of surface-active compounds and the structural arrangement of polysaccharide-protein networks. Moreover, Szulc & Lenart (2013) emphasized that powders with a balanced composition of hydrophilic and lipophilic groups are effective in fat-rich or emulsified systems, where they contribute to emulsion stability, lipid binding, and textural enhancement. These results indicate that the extraction conditions play a decisive role in modulating the hydration and lipid absorption properties of purple onion peel powders. Milder UAE parameters appear to preserve the porous microstructure and functional groups necessary for fluid interaction, thereby improving WHC and OHC. The high OHC values, in particular, highlight the potential use of these powders as functional ingredients in food formulations such as meat analogues, baked products, or emulsified sauces, where fat absorption and moisture retention are critical for product quality.

Powder Flowability (Carr Index)

The flowability of purple onion peel powders, evaluated via the Carr index, varied between 33.34% and 37.51% under different UAE conditions. According to the Carr classification, values between 31-38% indicate poor flowability, while values above 38% are classified as very poor (Schlick-Hasper et al., 2022). In this study, all optimized extraction points yielded Carr index values exceeding 30%, with the lowest value observed at OP3 (33.34±0.02%), and the highest at OP1 (37.51±0.01%), indicating that none of the samples demonstrated good flow properties.

The limited flowability is primarily attributed to the fine particle size of the powders obtained after ultrasonic-assisted extraction. As particle size decreases, surface area increases, which enhances cohesive forces such as van der Waals interactions and electrostatic charges. These forces result in greater interparticle friction, leading to reduced bulk flow (Schlick-Hasper et al., 2022). In addition, high surface energy in fine powders promotes agglomeration, further restricting movement under gravity.

Similar flowability limitations have been reported in various biopigment-rich plant powders. For example, Mutavski et al. (2025) documented Carr index values ranging from 29.87±0.87 to 39.91±0.76 in spray-dried black elderberry by-product extracts containing 60-100% maltodextrin, underscoring the decisive role of carrier concentration in modulating powder cohesion and dispersibility. Similarly, Liu et al. (2025) investigated freeze-dried navel orange peel powders formulated with various wall materials and recorded Carr index values of 31.07 for unencapsulated samples, 38.33 with gum arabic, 29.45 with maltodextrin, and 29.07 with corn starch, demonstrating that the choice of encapsulating agent profoundly influences flowability and bulk density. Moreover, studies on aronia fruit powders produced under different drying regimes revealed Carr index values between 61.57 and 32.20, indicating that both drying intensity and the resulting particle morphology critically affect interparticle friction and cohesive forces (Taşova et al., 2024). Taken together, these findings highlight that the flowability of polyphenol-rich powders is predominantly determined by the interplay of drying method, particle size distribution, and carrier matrix composition, all of which collectively define surface roughness, particle packing behavior, and overall bulk flow properties. Furthermore, the present study's findings are consistent with Szulc & Lenart (2013), who emphasized that natural pigment powders, due to their fine nature and hydrophilic surface characteristics, often exhibit cohesion-related flow problems. As a mitigation strategy, they recommended the use of carrier agents or flow aids such as maltodextrin, silicon dioxide, or modified starch, which can reduce interparticle interactions by increasing particle size and reducing hygroscopicity.

Despite the unfavorable flow properties, the powders developed in this study exhibited high bioactive retention, low water activity, desirable color parameters, and enhanced hydration–lipid interaction capabilities, all of which are critical attributes for their potential application as functional natural additives in food systems. These properties are essential not only for nutritional and sensory quality but also for processing stability and shelf life (Rodríguez-Roque et al., 2015).

4. Conclusion

This study demonstrated that purple onion peels, a commonly discarded agro-industrial by-product, are a valuable and underutilized source of phenolic compounds and anthocyanins with significant functional and technological potential. The combined application of ultrasound-assisted extraction (UAE) and response surface methodology (RSM) enabled the systematic optimization of key process variables, including ethanol concentration, temperature, extraction time, and solvent-to-solid ratio, maximizing bioactive compound recovery while preserving extract integrity. Optimal conditions (95% ethanol, 55 mL/g solvent-to-solid ratio, 40 °C, and 105 min) resulted in high total phenolic (1230.96 mg GAE/100 g) and total anthocyanin contents (12398.1 mg/kg), comparable or superior to those reported for other phenolic-rich by-products such as pomegranate peel, grape pomace, and red cabbage. The robustness and predictive accuracy of the RSM model were statistically validated, confirming its suitability for process design and potential industrial scale-up.

The physicochemical characterization revealed favorable water- and oil-holding capacities, low water activity, and intense chromatic properties, indicating good powder stability and suitability for incorporation into diverse food formulations. Although limited flowability associated with fine particle size was observed, consistent with similar biopigment powders, these handling challenges can be addressed through formulation strategies such as carrier addition or agglomeration without compromising functional performance. Overall, this study highlights the dual role of purple onion peels as a rich source of bioactive compounds and a promising functional ingredient. The integration of green extraction technologies with robust statistical optimization provides an efficient and environmentally sustainable approach for agro-industrial by-product valorization, supporting circular bioeconomy principles and enabling clean-label applications in food, nutraceutical, and cosmetic industries.

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Conflicts of Interest

The authors state that they have no conflicts of interest.

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