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Optimization of the extraction process of phenolic compounds from eggplant peel

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Abstract

In this study, the extraction process of phenolic compounds from eggplant peels, an important industrial waste, with the help of ultrasound was optimized by Taguchi method. Experiments were carried out according to Taguchi L_9 (3^3) experimental matrix. Ethanol ratio (50, 70, 90% (v/v)), sonication time (15, 30, 45 min) and sonication temperature (30, 40, 60 °C) were selected as independent variables. In this study, which aimed to maximize the total phenolic matter (TPM) content of the extracts, the optimum conditions were determined as 70% ethanol ratio, 45 min sonication time and 60 °C sonication temperature. ANOVA results showed that ethanol ratio (83.06%) and sonication temperature (14.38%) were the most significant parameters contributing to the process. According to the validation experiments carried out to verify the optimum conditions, 66.02% improvement was achieved in the TPM content obtained under optimum conditions compared to the initial process conditions.

Key Words: *Ultrasound, Taguchi, phenolic, ANOVA, optimization*

Patlıcan kabuklarından fenolik bileşen ekstraksiyon sürecinin optimizasyonu

Özet

Bu çalışmada önemli bir endüstriyel atık olan patlıcan kabuklarından fenolik bileşenlerin ultrases yardımıyla ekstraksiyon süreci Taguchi yöntemiyle optimize edilmiştir. Deneyler Taguchi L9 (3^3) deney matrisine göre gerçekleştirilmiştir. Bağımsız değişkenler olarak etanol oranı (A; %50, 70, 90 (v/v)), sonikasyon süresi (B; 15, 30, 45 dak) ve sonikasyon sıcaklığı (C; 30, 40, 60 °C) olarak seçilmiştir. Ekstraktların toplam fenolik madde (TFM) içeriğinin maksimize edilmesinin amaçlandığı bu çalışmada optimum koşullar %70 etanol oranı, 45 dak sonikasyon süresi ve 60 °C sonikasyon sıcaklığı olarak belirlendi. ANOVA sonuçları proses sürecine en fazla katkı sağlayan parametrelerin etanol oranı (%83.06) ve sonikasyon sıcaklığı (%14.38) olduğunu gösterdi. Optimum koşulların kontrolü amacıyla gerçekleştirilen doğrulama deneylerine göre optimum koşullarda elde edilen TFM içeriğinde başlangıç proses koşullarına göre %66.02 oranında iyileşme sağlanmıştır.

Anahtar Kelimeler: *ultrases, Taguchi, fenolik, ANOVA, optimizasyon*

Introduction

Eggplant (Solanum melongena L.) is an important vegetable crop due to its nutritional and economic values (Ferarsa et al., 2018). Eggplant fruits, which are mostly produced in subtropical and tropical regions, are widely consumed annual vegetables worldwide (Zhang et al., 2020). According to FAO data for 2022, around 59 million tons of eggplant is produced in the world. Nearly half of this (~23 million tons) is produced by China.

A significant amount of waste is generated as a result of the further processing of agricultural foods in food facilities (Philippi et al., 2016). Eggplant peels, which are considered as a waste in food processing, can be a potential threat to the environment if not properly utilized. Recent studies have focused on the extraction of bioactive components from plant by-products using different extraction techniques (Kainat et al., 2023).

Traditional methods such as soxhlet and maceration with low extraction yields are widely used in the recovery of phenolic compounds from industrial by-products. The relatively long extraction time causes degradation/loss of heat sensitive components in these methods. In addition, high energy costs and solvent costs are among the most important disadvantages of traditional extraction methods (Ferarsa et al., 2018). The development of eco-friendly and sustainable extraction methods for natural products has become a prominent area of research in food applications. These innovative techniques, known as clean or green technologies, aim to minimize or eliminate the use of harmful solvents, safeguard the environment, and decrease energy usage (Popovic et al., 2022). Recently, eco-friendly methods like ultrasonically-assisted extraction (UAE), microwave-assisted extraction, and enzymatic-assisted extraction have been developed to extract bioactive compounds from plant sources (Hong et al., 2023). Ultrasound-assisted extraction, in particular, has several benefits over traditional methods, including reduced extraction time, lower solvent usage, and higher yields (Güldane, 2023). In this study, it was aimed to improve the extraction process of phenolic substances from eggplant peel, which is considered as a waste, with the help of ultrasound. The optimal levels of process parameters in the extraction process were determined using Taguchi method.

Material and Methods

Material

Eggplants used in this study were obtained from a local market. Eggplant peels were cut with a peeling apparatus with a thickness of 1 cm and dried in an oven at 40 °C until constant weight. The peels were then ground with a blender and passed through a 0.2 mm sieve and stored at room temperature until the analysis stage.

Extraction of phenolics from eggplant peels

Ethanol ratio, sonication time and sonication temperature were selected as independent variables for the extraction of phenolic compounds from eggplant peel (Table 1). For extraction, 5 g of eggplant peel powder was transferred to a beaker and 100 ml of solvent containing different ratios of ethanol (50%, 70% and 90%, v/v) was added. The beaker was placed in an ultrasonic water bath (Creworks, PS-30A, China) operating at constant frequency (40 kHz) and power (180 W), pre-set to a certain temperature (30, 45 and 60 °C) and sonicated for different times (15, 30 and 45 min). After extraction, solid particles were removed from the mixtures by filtration with Whatman No:1 filter paper.

Determination of total phenolic matter (TPM) content

The method proposed by Singleton et al. (1999) was partially modified and used to determine the TPM content of the samples. For this purpose, 100 µL Folin reagent was added to 100 µL extract and vortexed. After 3 min, 4 mL of 1 M sodium carbonate (Na_2CO_3) solution was added to the test tube. The samples were kept at 45 °C for 5 min, then cooled rapidly and their absorbance at 760 nm was determined with a spectrophotometer (Shimadzu, UVmini1240, Japan). TPM contents of the samples were calculated as mg gallic acid equivalent (mg GAE/g) using the calibration curve graph equation prepared with standard gallic acid solutions (0-1 mg/mL).

Experimental design

Taguchi L^9 (3^3) experimental design presented in Table 1 was used for the extraction of phenolic compounds from eggplant peel. In the process optimization, the effect of solvent ethanol content ((50%, 70% and 90%, v/v), sonication time (15, 30 and 45 min) and sanication temperature (30, 45 and 60 °C)) variables on total phenolic matter (TPM) extraction was investigated.

Table 1. Taguchi L_9 (3^3) design matrix

Run	Ethanol ratio (%)	Sonication time (min)	Sonication temperature (°C)	TPM (mg GAE/g)	S/N (dB)
1	50	15	30	4.62	13.29
2	50	30	45	5.82	15.30
3	50	45	60	6.23	15.89
4	70	15	45	6.95	16.84
5	70	30	60	7.64	17.66
6	70	45	30	6.64	16.44
7	90	15	60	4.74	13.52
8	90	30	30	4.04	12.13
9	90	45	45	4.48	13.03

In the Taguchi experimental matrix, each column represents the selected parameters and each row represents the corresponding parameter levels. In this method, experimental data are converted into signal (S)/noise (N) ratios. Here "S" represents the targeted output and "N" represents the variance. There are 3 types of S/N ratio norms in Taguchi optimization: "Larger is better", "nominal is best" and "smaller is better". The main objective is to maximize the selected norm (Tansel et al., 2022). In this study, the following Equation 1 was preferred since it was aimed to maximize the TPM response in ultrasound-assisted extraction of phenolic compounds.

$$S/N = -10log\left[\frac{1}{n}\sum_{i=1}^{n}\frac{1}{{y_i}^2}\right] \quad (1)$$

Where; y_i: results for i^{th} experiment, n: total number of scenario.

Statistical analysis

Minitab 17.0 software was used to obtain the Taguchi experiment matrix and to evaluate and predict the experimental results.

Results and Discussion

Taguchi Optimization

The Taguchi optimization method was employed to assess the impact of different parameters (ethanol ratio, sonication time, and sonication temperature) on the total phenolic matter (TPM) in the ultrasound-assisted extraction of phenolic compounds. The experiments followed the Taguchi L9 experimental design, and the results, along with the corresponding signal-to-noise (S/N) ratios, are presented in Table 1. The S/N ratios graph of the experimental results is shown in Figure 1. The graphical representation of the results shows the main effects of the independent factors on the response. These interactions are used to estimate the effect of each parameter on the TPM during the extraction process.

Water and ethanol mixtures were used to extract phenolic compounds from eggplant peel. Ethanol was preferred for extraction because it is both easily available in high purity and widely used in food applications (Ferarsa et al., 2018). Figure 1 shows that when the ethanol content in the solution was increased up to a certain level, an increase in extraction efficiency was observed. The extraction efficiency of the solution containing 70% EtOH was found to be better than the solution containing 50% and 90% EtOH. This can be explained by the ability of two solvents of different polarity to extract different phenolic compounds. In the study, the amount of phenolic substances extracted increased as the sonication time applied to the samples increased. Similar data were obtained for sonication temperature. As a result of the increase in sonication temperature, TPM yield increased. These results are closely related to the data reported by Ferarsa et al. (2018) in the ultrasound-assisted extraction of anthocyanins from eggplant peels. Similar observations were also made by Dranca & Oroian (2016). These studies revealed that increasing the temperature improved extraction efficiency by lowering the viscosity of the solvent, allowing it to penetrate the sample cells more effectively. As a result, the solubility and desorption of phenolic compounds were enhanced. Based on the extraction parameters, the optimal conditions for extracting phenolic compounds from eggplant peels were identified as using a solvent with 70% ethanol, a duration of 45 minutes, and a temperature of 60°C ($A_2B_3C_3$).

ANOVA data are utilized to express the degree of influence of independent parameters on TFP. The percentage contribution of each parameter to the result is calculated by taking into account the contribution of the Adj SS value of the relevant process parameter to the total Adj SS value. The results are given in Table 2. According to the ANOVA with high reliability (R^2=0.99; R^2_{adj}=0.98; R^2_{pred}=0.92) the effect of ethanol ratio and sonication temperature on TPM response was found to be significant at 83.06% and 14.38%, respectively ($p<0.05$). However, sonication time had no significant effect on the response variable ($p>0.05$).

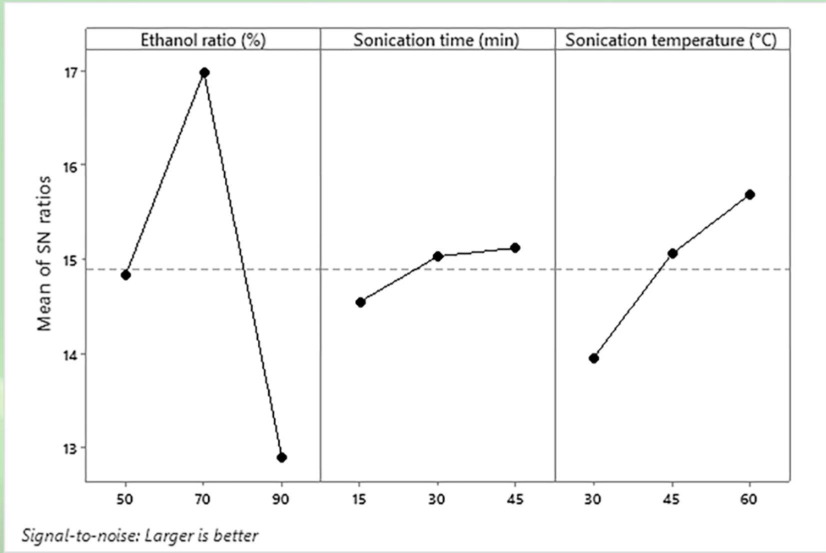

Figure 1. S/N ratio graph

Table 2. ANOVA results for TPM response

Source	DF	Adj SS	Adj MS	F-Value	P-Value	Contribution(%)
Ethanol ratio	2	10.6603	5.33014	216.87	0.005*	83.06
Sonication time	2	0.2800	0.14001	5.70	0.149	2.18
Sonication temperature	2	1.8454	0.92268	37.54	0.026*	14.38
Error	2	0.0492	0.02458			0.38
Total	8	12.8348				100

*$p<0.05$; R^2=99.62%; R^2_{adj}=98.47%; R^2_{pred}=92.24%

The mathematical equation developed to explain the relationship between the independent variables (Ethanol ratio (A), sonication time (B) and sonication temperature (C)) and the output parameter (TPM) was obtained with Minitab 17.0 software. The regression equation (Equation 2) for the ultrasound-assisted extraction of phenolic compounds from eggplant peels was established.

$$TPM=5.68-0.13*A_{50}+1.39*A_{70}+1.26*A_{90}-0.25*B_{15}+0.15*B_{30}+0.1*B_{45}-0.58*C_{30}+0.07*C_{45}+0.52*C_{60} \quad (2)$$

Confirmation experiments

In order to verify the optimization results, validation experiments with 3 replications were performed. The results are given in Table 3. When the results were examined, it was seen that the experimental result was close to the result predicted by the Minitab. In addition, as a result of the optimization, 66.01% improvement was achieved compared to the initial parameter levels.

Table 3. Confirmation test results

	Initial process parameters	Optimum conditions	
		Prediction	Experiment
Parameter levels	$A_1B_1C_1$	$A_2B_3C_3$	$A_2B_3C_3$
TPM (mg GAE/g)	4.62	7.69	7.67±0.03
Improvement (%)			66.02

Conclusions

In this study, ultrasound was utilized as a novel extraction method to obtain phenolic compounds from eggplant peel. Experiments were conducted using the Taguchi L9 orthogonal array for optimization. Various ethanol ratios were tested as solvents, with 70% ethanol found to be the most effective. Additionally, the results indicated that both extraction time and temperature positively influenced phenolic content. Statistical analysis revealed that the ethanol ratio was the most significant parameter affecting the extraction process.

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