

Multi Response Optimization of Process Parameters in the Extraction of Bioactive Components from Dill: Taguchi-MOORA Methodology

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Abstract – The aim of this study was to obtain an extract rich in phenolic compounds with high antioxidant activity from dill (*Anethum graveolens* L.). To optimize the ultrasound-assisted extraction process, a Taguchi (L9) orthogonal matrix which consists of three levels and three factors including solute: solvent ratio (A; 1:100, 3:100 and 5:100), sonication temperature (B; 30, 45, 60 °C) and sonication time (C; 5, 10, 15 min) was used. In the single response optimization based on the Taguchi method, the optimum ultrasonic extraction parameters for total phenolic content and antioxidant activity (DPPH) were determined as A₁B₃C₃ and A₁B₁C₁, respectively. In this study, MOORA (Multi-Objective Optimization by Ratio Analysis) was utilized to convert a two-response problem into a single response. As a result of this optimization, the solute: solvent ratio, sonication temperature, and sonication time variables were determined as 1:100, 60 °C and 15 min, respectively (Run 3), for the extract with the highest total phenolic and antioxidant content (A₁B₃C₃). According to the results obtained, the solute: solvent ratio was found to be the parameter that most significantly affected the ultrasonic extraction process for both response variables. The Taguchi-MOORA hybrid optimization technique was successfully applied to the ultrasonic extraction of bioactive components from dill.

Keywords – Total Phenolic Content, DPPH, Ultrasonic Extraction, Sonication

I. INTRODUCTION

Dill (*Anethum graveolens* L.) is an important herbal plant belonging to the *Apiaceae* family. While it can be cultivated in various regions around the world, it is used as a local spice, particularly in Central and Southwest Asia, Southeast Europe, and Mediterranean regions. This aromatic plant has been used in traditional medicine in Asian culture for centuries. The essential oils and bioactive components in dill have been proven to have pharmaceutical effects with antioxidant, antimicrobial, diuretic, carminative, and appetizing effects [1]. Its aromatic properties and essential minerals such as phosphorus, potassium, and magnesium, make it a valuable ingredient in various culinary applications, such as

salads, sauces, soups, seafood, and pickles [2]. Dill is known for its high content of phenolic acids, flavonols, and flavanones [3]. Different extraction methods, including solvent, ultrasound [1], microwave [4], and pulsed electric field [5], using water, ethanol, or methanol solvents, have been reported for extraction of bioactive components from dill.

The Taguchi method, developed by Genichi Taguchi, is a tool for improving product quality in industrial processes. Taguchi method promises to reach optimal conditions with fewer experiments compared to the traditional optimization methods, resulting in both time and cost savings [6]. However, one limitation of this method is its primary focus on single-response industrial

processes. Multi-criteria decision-making methods are often used when more than one characteristic needs to be optimized [7]. There are different multi-criteria decision-making methods, such as Analytical Hierarchy Process (AHP), Technique For Order Preference By Similarity To An Ideal Solution (TOPSIS), Gray Relational Analysis (GRA), Vlse Kriterijumska Optimizacija Kompromisno Resenje (VIKOR), and Multi-Objective Optimization Based on Ratio (MOORA) [8]. In this study, ultrasonic extraction was aimed to obtain an extract rich in phenolic compounds with high antioxidant activity from dill. Furthermore, the process was optimized by the Taguchi-MOORA hybrid technique.

II. MATERIALS AND METHOD

The dried dill used in the analysis was obtained in powder form from a local seller in Sakarya, Türkiye. The samples were kept in their packages in a cool and dry place until analysis. Level-2 and level-3 headings can be used to detail main headings.

A. Extraction of bioactive compounds

The dill sample was mixed with water in different solute: solvent ratios (100:1, 100:3, and 100:5 (w/w)) in a beaker, which was then sonicated at different temperatures (30, 45, and 60 °C) and various times (5, 10 and 15 min). The ultrasonic water bath (CALISKAN, CHINA) had a frequency of 40 kHz and a power of 150 kW. The temperature of the water bath was balanced with ice packs. The extracted samples were filtered through Whatman No:1 filter paper with a water trombe. The samples were then filtered through a 0.45 µm membrane filter and stored in a brown glass bottle at +4 °C until analysis.

B. Determination of Total Phenolic Content (TPC)

TPC of the sample extracts was determined by the method proposed by [4] with slight modifications. Briefly, 100 µl sample extract, 1 ml Folin Ciocalteu reagent, and 2 ml distilled water were transferred into a test tube. After 3 min, 1 ml of sodium carbonate solution (20%) was added. The mixture was vortexed and then kept in the dark condition for 1 h. The absorbance value was measured at 765 nm using a UV-VIS spectrophotometer (Shimadzu UV-1240, Japan). The results were determined using an Eq. obtained from the calibration curve, which was prepared

with a gallic acid standard solution and calculated as mg gallic acid equivalent (GAE)/100 g sample dry matter (dm).

C. Determination of Total Antioxidant Activity

The total antioxidant capacity of the samples was determined by measuring DPPH free radical scavenging activity. The analysis was conducted by using the method described by [4]. DPPH values were calculated by following Eq. 1:

$$DPPH (\%) = \left[1 - \frac{A_s}{A_{blank}} \right] \times 100 \quad (1)$$

where A_s and A_{blank} indicate the absorbance of the sample extract with DPPH and the absorbance of the DPPH without a sample, respectively.

D. Experimental Design

D1. Taguchi Method

The Taguchi technique is a statistical analysis tool that uses an efficient design of experiments. It uses an orthogonal array including various process parameters that reduce the number of experiments and the duration of the experiment [9]. In this study, solute: solvent ratio, sonication temperature, and sonication time variables were determined as control parameters in ultrasound-assisted extraction of bioactive components from dill (Table 1). Taguchi L9 orthogonal matrix was utilized to optimize the effect of control variables on total phenolic matter and DPPH scavenging activity responses (Table 2).

Table 1. Process parameters and corresponding levels

Symbol	A	B	C
Parameter	Solute: solvent ratio	Sonication temperature (°C)	Sonication time (min)
A	1:100	30	5
B	3:100	45	10
C	5:100	60	15

To obtain a statistical metric, the signal-to-noise (S/N) ratio is evaluated as an indicator of process performance. The S/N ratio is a logarithmic function, calculated by evaluating the ratio of signal (mean) to noise (standard deviation). In each statistical approach, there are three types of S/N ratio, depending on the desired output quality characteristics: (1) smaller the better, (2) nominal

the better, and (3) larger the better [10]. In this study, as the aim was to maximize the total phenolic matter and total antioxidant activity responses in ultrasonic extraction of dill, the "larger the better" function was selected and the results were assessed according to the following Eq. (2):

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

Table 2. Taguchi L9 orthogonal design matrix and response values

Run	Control parameters			Responses			
	A	B	C	TPC	S/N	DPPH (%)	S/N
1	1:100	30	5	2127.78	66.56	91.05	39.19
2	1:100	45	10	2533.33	68.07	85.40	38.63
3	1:100	60	15	2794.44	68.93	84.77	38.57
4	3:100	30	10	1276.84	62.12	57.84	35.24
5	3:100	45	15	1382.39	62.81	57.24	35.15
6	3:100	60	5	1252.77	61.96	61.30	35.75
7	5:100	30	15	741.11	57.40	47.76	33.58
8	5:100	45	5	46.77	33.40	725.56	57.21
9	5:100	60	10	43.05	32.68	736.67	57.35

D2. Multi-Objective Optimization by Ratio Analysis (MOORA) Technique

Multi-objective optimization is the process of simultaneously optimizing two or more parameters that contradict each other under certain limitations. The MOORA technique is a multi-objective optimization method that can be successfully employed to solve a variety of complex decision-making problems. This methodology consists of 5 stages [11]:

1. Creation of decision matrix (D)

The decision matrix was created as follows:

$$D = \begin{Bmatrix} X_{1,1} & x_{1,2} & \dots & x_{1,n} \\ X_{2,1} & x_{2,2} & \dots & x_{2,n} \\ \vdots & \vdots & & \vdots \\ X_{m,1} & x_{m,2} & \dots & x_{m,n} \end{Bmatrix} \quad (3)$$

2. Normalization of the decision matrix

The matrix values were normalized by Eq.

4.

$$x_{ij} = \frac{x_{ij}}{\sqrt{\sum_{i=1}^n x_{ij}^2}}, i = 1, \dots, m \text{ and } j = 1, \dots, n \quad (4)$$

3. Calculation of weighted normalized values

The weighted decision matrix (V) was estimated using Eq. 5 and 6, respectively.

$$V = [Y_{ij}]_{m \times n}, i = 1, \dots, m \text{ and } j = 1, \dots, n \quad (5)$$

$$Y_{ij} = X_{ij}^* W_j \quad (6)$$

4. Obtaining of composite design and ranking the score for each alternative.

The composite score for the alternatives was calculated using Eq. 7. Subsequently, the scores were ranked from highest (run 1) to lowest (run 9).

$$Y_i^* = \sum_{j=1}^t Y_{ij} - \sum_{j=t+1}^n Y_{ij} \quad (7)$$

5. Identification of optimum parametric combination

The composite design values were evaluated in the Taguchi approach. The optimal levels for each extraction parameter were determined.

III. RESULTS AND DISCUSSION

III.A. Single Response Optimization

The effects of process variables on TPC and DPPH responses in ultrasonic extraction of dill samples were investigated using a Taguchi L9 orthogonal design matrix. The results of the experiments and the corresponding S/N ratio values are presented in Table 2. In this study, experimental data were converted to S/N ratios to determine the effects of control parameters on each single response. The main effect plots of S/N ratios for each response parameter are depicted in Fig. 1 and 2. In both graphs, an increase in the solute: solvent ratio had a detrimental impact on the extraction process. Conversely, an increase in sonication temperature and time had a positive effect on the extraction of phenolic substances. However, it was observed that this increase in temperature and time resulted in a reduction in the antioxidant activity values of the phenolic compounds obtained.

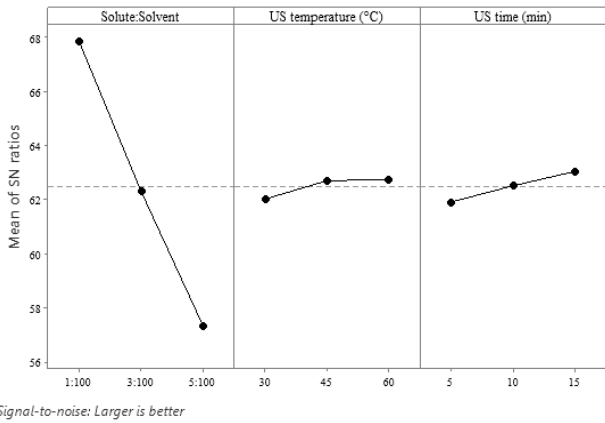


Fig. 1. Main effects plot for S/N ratio of TPC response

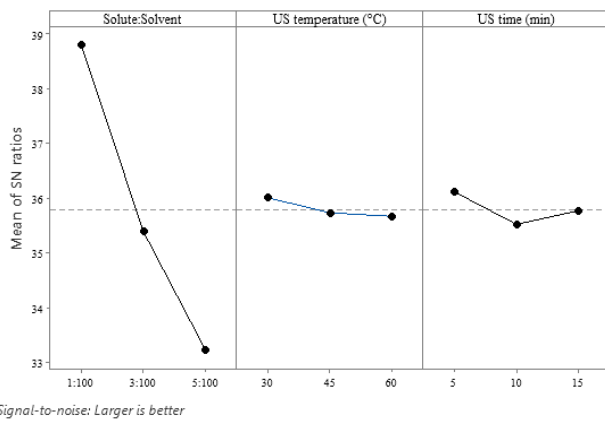


Fig. 2. Main effects plot for S/N ratio of DPPH (%) response

Table 3 illustrates the S/N ratios obtained for both TPC and DPPH responses. In this table, the highest levels of the control parameters for the response variables are considered as the optimum conditions. When analyzing the data from both Figs. 1 and 2, as well as Table 3, it can be seen that the highest TPC value could be achieved with a 1:100 solute: solvent ratio at 60 °C sonication temperature and 15 minutes sonication time (A₁B₃C₃). Additionally, it was determined that the highest antioxidant activity value could be obtained with a solute: solvent ratio of 1:100, sonication temperature of 30 °C, and sonication time of 5 min (A₁B₁C₁). Furthermore, when assessing the "delta" values, which represent the difference between the highest and lowest S/N ratios for each control parameter, it was observed that the most influential factor in the ultrasonic extraction process for both response variables was the "solute: solvent ratio". Overall, the results indicated that different combinations of sonication temperature and

sonication time parameters yield optimal solutions for both response variables.

Table 3. Response Table for S/N ratios

Level	TPC			DPPH		
	A	B	C	A	B	C
1	67.85	62.03	61.91	38.79	36.00	36.11
2	62.30	62.70	62.51	35.38	35.73	35.52
3	57.32	62.74	63.05	33.22	35.66	35.77
Delta	10.53	0.72	1.14	5.57	0.34	0.59
Rank	1	3	2	1	3	2

*Optimal levels for the control parameters

III.B. Multi Response Optimization

The MOORA methodology was employed to optimize the multiple response process of bioactive component extraction from dill. Initially, the design matrix was constructed using Eq. 3, and then it was normalized utilizing Eq. 4. Subsequently, the normalized values were weighted using Eq. 5 considering the weighting factors, which were determined using principal component analysis [12]. The composite score was calculated by using the weighted benefit values in Eq. 6. Since the objective of this study was to maximize both TPC and DPPH responses, no non-benefit value was considered. Finally, the results were ranked from the highest to the lowest value, and alternative "3" with the highest composite score, was identified as the optimal alternative (A₁B₃C₃).

Table 4. The MOORA optimization results

Run	Normalized values		Weighted normalized values		Composite score	Rank
	TPC	DPPH	TPC	DPPH		
1	0,458	0,422	0,229	0,211	0,4399	3
2	0,430	0,502	0,215	0,251	0,4658	2
3	0,427	0,554	0,213	0,277	0,4901	1
4	0,291	0,253	0,146	0,126	0,2720	6
5	0,288	0,274	0,144	0,137	0,2810	4
6	0,309	0,248	0,154	0,124	0,2784	5
7	0,240	0,147	0,120	0,073	0,1936	7
8	0,235	0,144	0,118	0,072	0,1896	8
9	0,217	0,146	0,108	0,073	0,1813	9

*Optimal alternative

As the primary goal of this study was to maximize the composite score, the optimum extraction parameter levels were determined based

on the "larger the better" criterion. According to the results depicted in Fig. 3, it is evident that the optimal process was achieved for sample 3 ($A_1B_3C_3$)

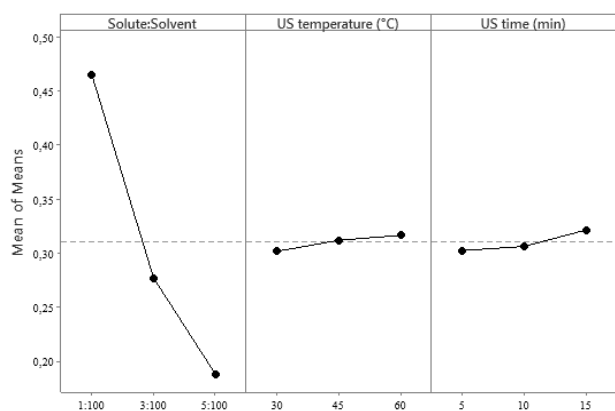


Fig. 3. Response graph for the composite score

IV. CONCLUSION

This study employed the Taguchi-MOORA hybrid optimization technique to optimize the ultrasonic extraction conditions for obtaining bioactive extracts from dill, which is an aromatic plant known for its high phenolic content and strong antioxidant activity. In this technique, the key process variable that significantly influenced the extraction processes was identified as the solute: solvent ratio. Through the MOORA method optimization, the optimal levels for the control parameters, such as solute: solvent ratio, sonication temperature, and sonication time, were determined as 1:100, 60 °C, and 15 min, respectively, in order to maximize both total phenolic content and antioxidant activity responses. The findings of this study showed that ultrasonic extraction of aromatic herbs for food engineering applications can be optimized by the Taguchi-MOORA hybrid optimization technique.

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