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## Subcritical water extraction of Thymol from Ajowan seeds by Taguchi experimental design

Neda Badrkhani, Maryam Khajenoori and Ali Haghghi Asl\*

School of Chemical Gas and Petroleum Engineering, Semnan University, Semnan, Iran

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### Abstract

In this study, the essential oil of Ajowan seeds was extracted by subcritical water extraction process (SWE). Thymol as the main component of the extracted essential oils was quantified by GC and GC-mass spectrometry. This extraction has been performed using Taguchi design approach under following conditions: temperature (100, 150 and 175 °C), mean particles size (0.25, 0.5 and 1 mm) and water flow rates (1.0, 2.0 and 4.0 mL/min). The optimum conditions as obtained from signal-to-noise ratio analysis in terms of amount Thymol were as follows: 175 °C, 0.25 mm and 2.0 mL/min. The SWE efficiency at optimized conditions was compared with the other conventional methods such as hydrodistillation and Soxhlet extraction. The SWE results showed a higher amount of Thymol (25.248 mg/g dried sample) than hydrodistillation (13.992) and Soxhlet extraction (7.209).

**Keywords:** Subcritical water extraction, Ajowan seeds, Thymol, Taguchi design.

### 1. Introduction

Ajowan, with scientific name *Trachyspermum ammi*, is a grassy annual plant belonging to family Apiaceae. It grows with white flowers and small and brownish seeds in Iran. Ajowan has been shown to possess antiviral, anti-inflammatory, antifungal, antipyretic, analgesic and antioxidant activity effects [1]. Therapeutic uses of Ajowan fruits include; stomachic, carminative and expectorant, antiseptic and amoebiasis; it is also used as Antipyretic, febrifugal and in the treatment of typhoid fever [2]. Ajowan seeds contain about 2–4% essential oil which is a rich source of Thymol (40–65%). Thymol is greatly used as fragrances in cosmetics, flavoring food additives, active ingredients in some old drugs and intermediates in the synthesis of perfume chemicals [2–4]. Thymol is a widely known antimicrobial agent. It is commonly incorporated in mouth washes because of its bactericidal action against oral bacteria. It is used in a variety of drugs [5, 6].

Nowadays, new extraction methods have been developed to reduce the use of organic solvent and to improve the extraction methods of constituents of plant materials, such as microwave assisted

extraction (MAE), supercritical fluid extraction (SFE), accelerated solvent extraction (ASE) and pressurized liquid extraction (PLE) [7–11]. Recently, subcritical water extraction (SWE) has been suggested as a green alternative method to extract natural products from herbal plants and foodstuffs [12–15]. SWE is performed at temperatures between 100–374 °C and pressures high enough to keep water in liquid state [12]. Subcritical water has singular properties such as acceptable selectivity property due to wide tunable polarity, high solubility, low viscosity, density, and dielectric constant due to increased temperature. Thus, desired polar or slightly polar components, that are not soluble well in water at ambient conditions, can be much more soluble in subcritical water. Based on the research works published, SWE shows a number of advantages including shorter extraction time, simplicity, cheaper, higher quality of the extracts and environmental-friendly than the conventional extraction methods [12, 13, 15].

The design of experimental has been done in some published articles related to the SWE of plant species. It seems SWE and optimization using Taguchi testing method of *Ajowan* essential oil was

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\*. Corresponding Author: Email: ahaghghi@semnan.ac.ir, Tel: +98231-3366914.

not studied previously and this is the first report announcing. The purpose of this research work was to investigate the effects of different factors (temperature, mean particles size and flow rate) on the SWE efficiency. Taguchi L<sub>9</sub> orthogonal array design was used to minimize number of experiments. The results of optimized SWE of *Ajowan* seeds were compared with extracts obtained by conventional techniques such as hydrodistillation and Soxhlet extraction.

## 2. Materials and procedures

### 2.1. Chemicals

Seeds of *Ajowan* were purchased from farm of Barij Essence Pharmaceutical Company (Kashan, Iran) in October 2011. Thymol (Merck, Darmstadt, Germany, 99.99% purity) was used as a standard component. NaCl, Na<sub>2</sub>SO<sub>4</sub> and n-pentene (Merck, Darmstadt, Germany) were used as an emulsion breaker, a drying agent and an extractor, respectively, in the liquid-liquid extraction step of the aqueous extracts. The doubly distilled, de-gassed water purified through a Milli-Q de-ionizing unit (Millipore, USA) used as extractant. Hexane (Merck, Germany, HPLC grade) was used as the diluting solvent before gas chromatography (GC).

### 2.2. Sample preparation

*Ajowan* seeds were dried in shadow at room temperature for two days and then were stored in polyethylene bags at -4 °C in refrigerator (HARRIS Co., Germany) until the process of analysis. The moisture content of the seeds was 5% (dry basis). Before performing the experiments, the dried samples were crushed in a laboratory mill and the powder was prepared using suitable sieves and a shaker in three desired sizes. To prevent from increasing the temperature of the samples, the grinding time was short. The granulated samples were stored in 3 glass container.

One hundred grams of dried *Ajowan* seeds were hydrodistilled with 1 L of water for 3 h in a Clevenger-type apparatus (Ashk-e shisheh Co.) in accordance with the description of the British Pharmacopoeia [16]. After hydrodistillation, around 3.2 mL of essential oil was obtained. The essential oil was dried over anhydrous Na<sub>2</sub>SO<sub>4</sub> and stored in a dark glass bottle at 4 °C until analysis.

### 2.3. Soxhlet extraction

Soxhlet extraction was carried out using standard apparatus (Ashk-e shisheh Co., Iran) for 6.0 h on 15.0 g of *Ajowan* seeds with 250 mL hexane [17]. The organic extract was concentrated by using a rotary evaporator (Rotovapor R-210, Water bath B-491, Buchi, Switzerland) to about 20 mL, and the final concentration step to about 1.5 mL for analysis was carried out by N<sub>2</sub> stream. This ready extract for chromatography was stored at 4 °C until analysis.

### 2.4. Subcritical water extraction system

SWEs were carried out in a laboratory-built apparatus (Semnan University, Semnan, Iran) shown in Figure 1. De-ionized water, as the only solvent, filled into a 5 L Pyrex feed tank was first purged for 2 h with N<sub>2</sub> to remove dissolved O<sub>2</sub>. A HPLC pump (SY-8100 series, BFRL Co., Germany) was used to deliver the water through the system. The HPLC pump output could be checked using a burette equipped in the inlet pipelines.

To avoid heat losses of essential oil, a double pipe heat exchanger (tube side: 30.48 mm i.d.×45.72 mm o.d., cooling surface area: 75 cm<sup>2</sup>) cooled with water with about 15 °C and 3 L/min flow rate was used to cool the extract coming out from the oven to a temperature close to 20 °C. After the wanted extraction, the pump and heater were stopped and the pressure was released to atmospheric pressure.

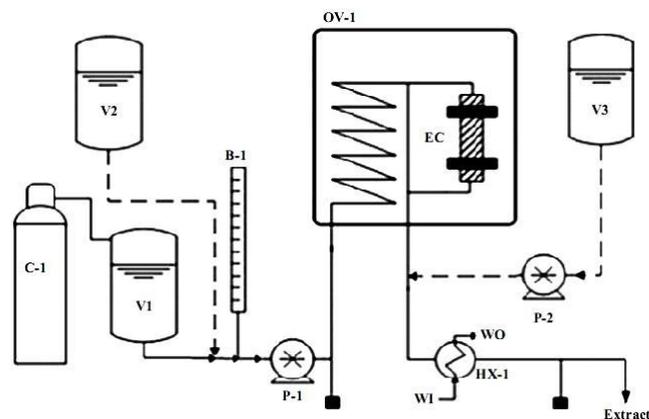


Figure 1. Schematic diagram of SWE system, B-1: Burette, C-1: Nitrogen cylinder, EC: Extraction cell, OV-1: Oven, P-1, 2: Pumps, V-1: Water tank, V-2: Solvent tank, V-3: Rinsing solvent tank, WI: Water inlet, WO: Water outlet.

### 2.5. Subcritical water extractions

For all subcritical water extractions, the extractor was filled with 1.0 g of ground *Ajowan* seeds. To prevent moving of the particles from the fixed bed, two

pulpy and cloth-filters sandwiched in between two stainless steel filter were inserted on both sides of the fixed bed. After the sample was replaced in the extraction vessel, the top and bottom of vessel was closed with two earring couplings (3/4 inch). In the oven, the closed vessel was connected to input and output of solvent pipes with two quick-open junctions (3/4 inch).

It is well known that effect of pressure on the SWE process is negligible [18, 19]. Therefore, for all SWEs, the pressure was selected to be 2 MPa to maintain the water as a liquid at the extraction temperatures. While terminal valve and back-pressure regulator were closed, HPLC pump (BFRL Co., SY-8100 series, Germany) was started to reach to the required pressure. Then, the pump was turned off, the by-pass valve was closed and the oven was brought up to the required temperature. At that time, the de-gassed and de-ionized water was then delivered at the adjusted flow rate with the pump to a 3-m stainless steel capillaries preheating section installed in the oven to heat it to the required temperature before passing through the extraction vessel. The pressure of the system was adjusted using the back-pressure regulator valve at the out let coil. The other operating conditions to be optimized were chosen as extraction temperature, mean particle size and water flow rate in the range of 100–175 °C, 0.25–1.0 mm and 1.0–4.0 mL/min, respectively and 150 min of extraction time. The extractions were dynamic and were performed to collect a certain extract volume, which depended on the extraction time and flow rate.

Regarding the selected flow rate and a void volume of system between the extractor and collection vessel (~40mL), in the all runs, around 30 mL of the water coming out of the system was discarded. Using GC analysis, it was observed that this amount of the extracts was clean and no peak was detected. There after that, the collection of the extract in a separating funnel began.

At the end of extraction time, a liquid–liquid extraction step using n-pentene was carried out. The volumetric ratio of n-pentene to extract was 1:2 in all experiments and extractions were completed by two equal volumes of solvent in two steps. Around 8 g NaCl was added to facilitate the breaking of the emulsion. The essential oil was stored in 1 mL hexane before the GC analysis. For the kinetic experiments, the collection vial was replaced at 30 min time intervals.

## 2.6. Essential oil analysis

The essential oil was analyzed with a gas chromatography (ACME 6100, 6000 series). The GC unit was equipped with a TRB-WAX capillary column (60 m×0.25 mm i.d.) with a poly ethylene glycol of 0.5 µm film. In temperature profile, at first the temperature of oven was static on 50 °C for 2min and then the temperature was increased to 230 °C with the temperature increase speed of 4°C/min. Carrier gas was Helium at a flow rate of 1 mL/min (constant flow). The both of detector and injector temperatures

were 240 °C and the volume of the sample was 1 µL. The essential oil sample was injected using splitless mode injection. Software supplied by Chrompack was used to handle the data compounds of essential oil

generated by GC. The gas chromatography was calibrated by an external standard calibration method.

GC-mass spectrometry (MS) analysis was conducted on a Varian Saturn model 3400. The GC–MS system was equipped with a DB-5 fused silica column (30 m × 0.25 mm i.d., film thickness 0.25 µm) and interfaced with a Varian ion trap detector. The GC–MS conditions were: oven temperature from 40 °C to 200 °C with the temperature increase speed of 4°C/min; injector and transfer line temperature, 210 °C and 220 °C; carrier gas, helium at a flow rate of 1.0 mL/min; and splitting ratio, 1:13. The detector temperature was maintained at 240 °C. The MS conditions were: ionization energy, 70 eV; mass range, 40–400 amu and scan mode electron impact (EI). The components were identified by comparing their retention times and mass spectra with those of pure reference components. The mass spectra were also compared with those in the National Institute of Standards and Technology, WILEY5 and TERPENOIDES mass spectra libraries and our own created library.

## 2.7. Taguchi design

Subcritical water extraction depends on different factors, therefore to reduce the number of tests, it is necessary to design of the experiment. In this experimental research, in order to design of the experiment, we have used Taguchi method because of facility and simplicity of use. In general, Taguchi design can reduce the costs and economize the time. Generally in the process of the subcritical water extraction of the herbal essence, the most important factors concerning the total yield of extraction and the concentration of the essence in the subcritical water fluid are: pressure and the temperature of the extraction, mean particles size, flow rate.

For Taguchi design and results analysis, the software named as Qualitek-4 (Version 15.6.1) was used. In Table 1, the efficient factors on quantity and quality of extractive essence in subcritical water extraction manner and total yield of extraction process consist of: temperature, mean particles size, flow rate, each one is seen in three different levels. L9 array has been prepared based on Table 1.

Table 1. Factors and their levels for the experimental design.

Factors	Description	Levels		
		1	2	3
A	Temperature (°C)	100	150	175
B	Flow rate (mL/min)	1.0	2.0	4.0
C	Particle diameter (mm)	0.25	0.5	1.0

The other parameters implicated in the extraction of extracts were kept constant, namely the pressure,

extraction time, weight sample and sample to co-packing ratio were 2 MPa, 150 min, 1 g and 1:1.5, respectively.

### Results and discussion

In using SWE, hydrodistillation and Soxhlet extraction methods to isolate the essential oil from Ajowan seeds, it was clear that Thymol was the main and important component. It was chosen as the key component to find the best SWE operating conditions. Based on experiments designing in the framework of

Table 2. The results of orthogonal (L9) test<sup>a</sup>.

Run	Trial conditions			Mean value (mg Thymol/g dried sample)	S/N ratio
	T (°C)	Q (mL/min)	dp (mm)		
1	100	1.0	0.25	11.230	20.970
2	100	2.0	0.50	7.671	17.672
3	100	4.0	1.00	7.969	18.013
4	150	1.0	0.50	6.661	16.410
5	150	2.0	1.00	9.071	19.139
6	150	4.0	0.25	17.966	25.085
7	175	1.0	1.00	8.096	18.155
8	175	2.0	0.25	25.248	28.044
9	175	4.0	0.50	12.073	21.635

<sup>a</sup> P < 0.05.

### 2.8. Optimization of SWE parameters

Analysis using S/N ratio is highly recommended since the results of experiments involved multiple runs. In its simplest form, the S/N ratio is the ratio of the mean response (signal) to the standard deviation (noise) [20]. The analysis of S/N ratio for each of the experiments will give the better combination of parameters levels that ensures a high level of extraction yield according to the experimental set of data. Taguchi defined three categories of quality characteristics in the analysis of (S/N) ratio, i.e. lower is better, nominal is best, and higher is better [21]. In this study, based on selecting the extraction yield as quality characteristic, the higher is better criterion was applied when evaluating the S/N ratio of the various extraction parameters is given by:

$$\frac{S}{N} = -10 \log \frac{\left( \frac{1}{Y_1^2} + \frac{1}{Y_2^2} + \dots + \frac{1}{Y_n^2} \right)}{n} \quad (1)$$

Where n is the number of runs under the same experimental conditions, and Y is the experimental yields of agglomerate for each run. The analysis begins with calculation of average of the S/N ratio of each parameter at a certain level using the following formula:

Taguchi method and according to array L<sub>9</sub>, and considering the change of conditions and different parameters such as temperature, flow rate, mean particles size, nine times of test and just one repetition, we have done in sum 18 main tests. The Thymol yield was defined as its value per value of used dried sample. The mean relative standard deviation of all the runs was calculated to be 7.46%. The designed experimental runs, their extraction results and signal-to-noise (S/N) ratio data are shown in Table 2.

$$(M)_{\text{parameter}=1}^{\text{level}=i} = \frac{1}{n_{1i}} \sum_{j=1}^{n_{1i}} \left[ \left( \frac{S}{N} \right)_{\text{parameter}=1}^{\text{level}=i} \right]_j \quad (2)$$

Where  $(M)_{\text{parameter}=1}^{\text{level}=i}$  represents mean of the S/N ratio of parameter 1 at level i,  $\left[ \left( \frac{S}{N} \right)_{\text{parameter}=1}^{\text{level}=i} \right]_j$  represents S/N ratio of parameter 1 at level i and its appearance sequence in Table 2 is the jth value in Table 3 and  $n_{1i}$  represents number of appearances of parameter 1 in level i. The results of calculation of S/N ratio for each parameter at level 1, 2 and 3 are presented in Figure 2.

Table 3. S/N ratio response table.

Parameter/level	$\left[ \left( \frac{S}{N} \right)_{\text{parameter}=1}^{\text{level}=i} \right]_j$			(M)
	j=1	j=2	j=3	
A/1	20.970	17.672	18.013	18.885
A/2	16.410	19.139	25.085	20.211
A/3	18.155	28.044	21.635	22.611
B/1	20.970	16.410	18.155	18.511
B/2	17.672	19.139	28.044	21.618
B/3	18.013	25.085	21.635	21.577
C/1	20.970	25.085	28.044	24.699
C/2	17.672	16.410	21.635	18.572
C/3	18.013	19.139	18.155	18.435

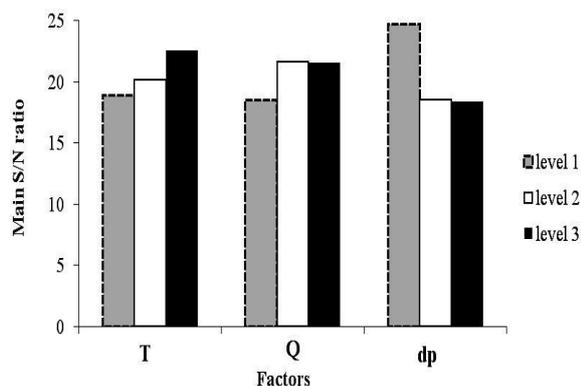


Figure 2. Response graph illustrating the variation of the average S/N ratios plotted against the various extraction parameters. Temperature (100, 150, 175 °C), particle diameter (0.25, 0.50, 1.00 mm), flow rate (1.0, 2.0, 4.0 mL/min).

According to Figure 2, increasing temperature and water flow rate to 2.0 mL/min and decreasing particle size increase the S/N ratio ( $P < 0.05$ ). Higher temperatures in SWE lead to rapid extractions. The physical advantages such as high diffusion rate, low viscosity of the water, low surface tension and low dielectric constant are achieved at elevated temperature conditions [19, 22].

In Figure 2, flow rates (2.0, 4.0 mL/min) have higher the S/N ratios ( $P < 0.05$ ). Increase in flow rate results in increase of superficial velocity and thus quicker mass transfer [23, 24]. Therefore, the best flow rate must be selected considering two important factors including the extraction time and the extract concentration. It is clear that shorter extraction time and more concentrated extracts are desirable. To prevent a slower extraction rate and longer extraction times (at the flow rate of 1.0 mL/min), and large amount of final dilute extracts (at the flow rate of 4.0 mL/min), flow rate of 2.0 mL/min was selected as the optimum value.

As shown as in Figure 2, the S/N ratio (extraction yield) for smaller particles is higher than that for larger particles. It shows that, the extraction process is controlled by mass transfer of the essential oil components on the surface of the solid phase. Smaller particle size means lower mass transfer resistant. Also, the final amount of Thymol extracted from 0.5 mm size particles was near to 1.0 mm particles. It shows that, at least in the selected range of mean particle sizes (0.5–1.0 mm), the extraction process may not be controlled by the mass transfer of Thymol.

Based on the result of Table 2, the optimization related to Taguchi method has been done with qualitek-4 software, and the maximum yield of subcritical water extraction was calculated. Also, the optimum conditions for achieving the maximum extraction yield of subcritical water extraction summarized in Table 4. Taguchi prediction for maximum extraction yield (mg/g dry sample) at this optimum conditions was 24.519 which showed 2.9% error compared to result of run number 8 (25.248). In Figure 3, cumulative extraction efficiencies in nine runs have been presented.

Table 4. Optimum conditions and performance for SWE yield of Ajowan essential oil.

Factors	Level	L. description
Temperature (°C)	3	175
Flow rate (mL/min)	2	2.0
Particle diameter (mm)	1	0.25
Expected result (extraction yield) at optimum conditions = 24.519		

## 2.9. Comparison with conventional techniques

The total extraction yields found for the essential oil of Ajowan seeds were 5.68, 2.95 and 3.65% (w/w) based on the dry weight for SWE at optimum conditions, hydrodistillation and Soxhlet extraction, respectively. The highest value for the total extraction yields was for the SWE method and the lower value was for hydrodistillation. Hydrodistillation has a distinct mechanism of extraction (mainly distillation), whereas SWE and Soxhlet extraction are mainly dissolution and/or solubilization of the essential oil in the solvent (extraction process). The comparison among the amount of Thymol (mg/g dried sample) by SWE at optimum conditions, hydrodistillation and Soxhlet extraction is shown in Table 5.

The amount of valuable oxygenated component in the SWE method is significantly higher than hydrodistillation and Soxhlet extraction. As hexane is a non-polar solvent, non-oxygenated components are enhanced compared to subcritical water [25]. On the other hand, in general, non-oxygenated components present lower vapor pressures compared to oxygenated components, and in this sense, its content in hydrodistilled extracts are increased. Because of the significant presence of the Thymol component, these facts showed that extract using the SWE method has more valuable component selectively and quickly.

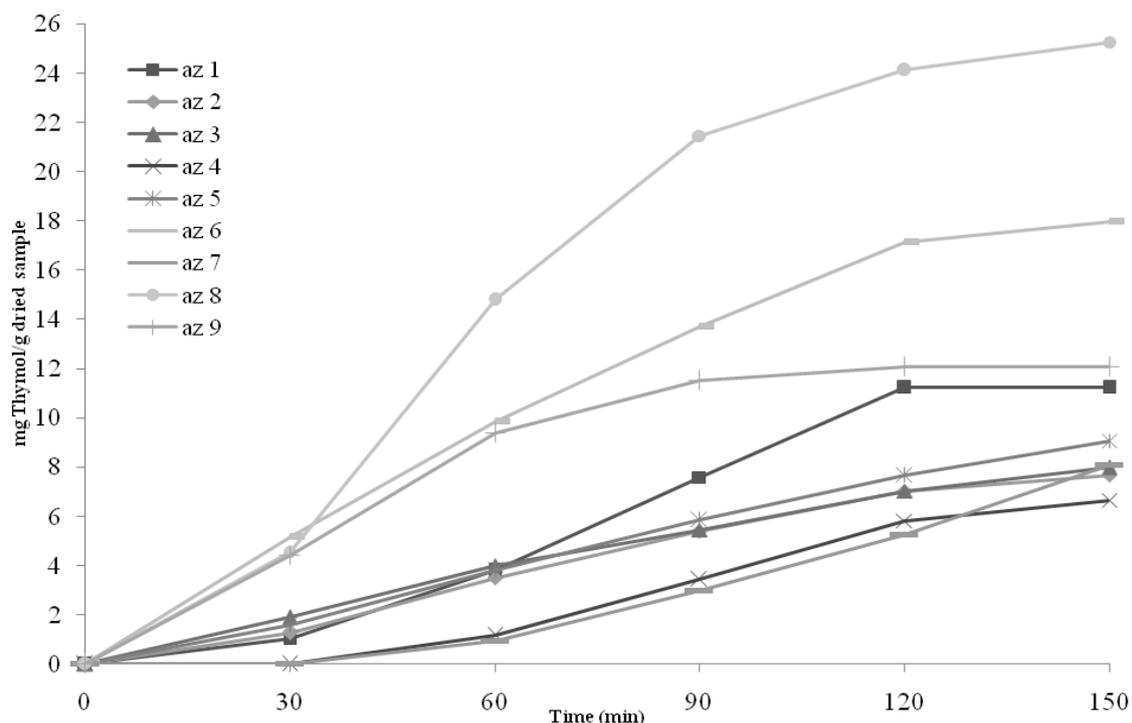


Figure 3. Cumulative extraction efficiencies (mg Thymol/g dried sample) from 1.0 g of Ajowan at  $L_9$  Orthogonal array scheme using Taguchi design of experiment.

Table 5. The amount of Thymol (mg/g dried sample) of the essential oil of Ajowan seeds, extracted by SWE at optimum conditions, Hydrodistillation and Soxhlet extraction.

Component	SWE <sup>a</sup>	Hydrodistillation <sup>b</sup>	Soxhlet extraction <sup>c</sup>	RI <sup>d</sup>
Thymol	25.248	13.992	7.209	1289

<sup>a</sup> Sample weight = 1 g; particle size = 0.25 mm; flow rate = 2.0 mL/min; temperature = 175 °C; pressure = 2 MPa and extraction time = 150 min.  
<sup>b</sup> Extraction time = 180 min.  
<sup>c</sup> Extraction time = 360 min.  
<sup>d</sup> Retention indices (RI) on the DB-5 column

### 3. Conclusions

In this study, for the first time, the extraction of Thymol from *Ajowan* seeds using subcritical water was investigated. Also, using a three level Taguchi experimental design ( $L_9$ ) the experimental parameters for oil agglomeration were optimized. The optimized extraction efficiency of Thymol, 25.248 (mg/g dried sample) was obtained at flow rate 2.0 mL/min, temperature of 175 °C and mean particle size 0.25 mm and pressure of 2 MPa and extraction time of 150 min. The optimum SWE was compared with conventional methods, hydrodistillation and Soxhlet extraction. An interesting result was that the total yield of essential oil and amount of Thymol extracted in SWE method were significantly higher than those in hydrodistillation and Soxhlet extraction.

### 4. Acknowledgements

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### Nomenclature

ASE	Accelerated solvent extraction
dp	Particle diameter
GC	Gas chromatography
GC-MS	GC-mass spectrometry
MAE	Microwave assisted extraction
P<0.05	Significant level 95%
PLE	Pressurized liquid extraction
S/N	Signal-to-noise ratio
SFE	Supercritical fluid extraction
SWE	Subcritical water extraction
T	Temperature
Q	Flow rate

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## استخراج با آب فوق گرم تیمول از دانه‌های زنیان با کمک طراحی آزمایش تاگوچی

ندا بدرخانی، مریم خواجه نوری و علی حقیقی اصل\*

دانشکده مهندسی شیمی نفت و گاز، دانشگاه سمنان، سمنان، ایران

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### چکیده:

در این مطالعه، اسانس دانه‌های زنیان با فرآیند استخراج آب دمای زیر بحرانی (SWE) استخراج شد. تیمول به عنوان ترکیب اصلی اسانس استخراج شده با کروماتوگرافی گازی و کروماتوگرافی گازی-طیف سنجی جرمی تعیین مقدار شد. این استخراج با استفاده از روش طراحی تاگوچی تحت شرایط زیر: دما (۱۰۰، ۱۵۰ و ۱۷۵ درجه سلسیوس)، اندازه ذرات (۰/۲۵، ۰/۵ و ۱/۰ میلی‌متر) و دبی‌های جریان آب (۱/۰، ۲/۰ و ۴/۰ میلی‌لیتر بر دقیقه) انجام شد. شرایط بهینه بدست آمده از آنالیز نسبت سیگنال به نویز بر حسب مقدار تیمول ۱۷۵ درجه سلسیوس، ۰/۲۵ میلی‌متر و ۲/۰ میلی‌لیتر بر دقیقه بود. بازده SWE در شرایط بهینه با دیگر روش‌های متداول مانند هیدروتقطیر و استخراج سوکسله قابل مقایسه بود. نتایج SWE مقدار بالاتری از تیمول (۲۵/۲۴۸ میلی‌گرم بر گرم نمونه خشک شده) نسبت به هیدروتقطیر (۱۳/۹۹۲) و استخراج سوکسله (۷/۲۰۹) نشان داد.

**کلمات کلیدی:** استخراج با آب فوق گرم، دانه‌های زنیان، تیمول، طراحی تاگوچی.