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Study the Quantity and Quality of Cumin Seed Essential Oil Extraction with a Modified Steam Extraction System

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ABSTRACT

Essential oils are widely used across multiple industries, including cosmetics, personal care, and aromatherapy, pharmaceuticals, and food flavorings. Since extraction methods critically influence essential oil quality, steam distillation remains the most common approach. In this study, an innovative steam distillation apparatus and conventional hydro-distillation device (Clevenger) were compared to investigate their effects on the yield and quality of cumin seed (*Cuminum cyminum L.*) essential oil. Extraction duration (60, 90, and 120 min) and steam temperature (100, 110, and 120°C) were selected as independent variables and analyzed via Response Surface Methodology (RSM). The results showed that increasing the extraction time significantly enhanced the essential oil yield, reaching a maximum of 1.8% at 120 min—an 11.6% improvement over the best yield obtained using the Clevenger apparatus. Gas chromatography (GC) and gas chromatography–mass spectrometry (GC–MS) identified 22 compounds, with β -pinene, p-cymene, γ -terpinene, cumin aldehyde, α -terpineol-7-al, and γ -terpinene-7-al collectively accounting for over 94% of the total essential oil. In essential oil extraction using a modified steam distillation system, increased extraction temperatures elevated β -pinene and p-cymene percentages, while lower temperatures favored higher cumin aldehyde and γ -terpinene-7-al percentages. The optimal temperature for obtaining more cumin aldehyde was found to be at 100°C.

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INTRODUCTION

Herbal medicinal products have gained global significance due to their health benefits. The demand for these products has increased because consumers have developed a growing preference for natural products, often perceived as safer and more cost-effective than synthetic drugs. According to the World Health Organization (WHO), approximately 80% of the population in developing countries still rely on herbal medicines for their primary healthcare needs. Many food and pharmaceutical companies are active in this field, believing that the market for medicinal plants has promising growth potential. This demonstrates the substantial demand for the use of medicinal plants (Mamun & Khan, 2020).

Essential oils, also known as volatile aromatic oils, are fragrant oily liquids extracted from various parts of plants, such as leaves, bark, flowers, buds, and seeds. These oils can be obtained from plant materials using various methods, including water distillation, steam distillation, solvent extraction, supercritical fluid extraction, and others. Among all methods, steam distillation is widely used on an industrial scale (Cassel & Vargas, 2006; Lira et al., 2009). The method used for extracting essential oils usually depends on the plant materials and their physical form. The extraction method is one of the main factors determining the quality of the essential oil. Choosing an inappropriate method can lead to damage or alteration of the essential oil's chemical composition, resulting in the loss of biological activity and natural properties. Additionally, in severe cases, it can cause changes in color, odor, and taste, as well as physical changes such as increased viscosity (Felegary et al., 2023; Tongnuanchan & Benjakul, 2014).

Steam distillation is the most widely used method for extracting essential oils from plants. Approximately 93% of essential oils are extracted using steam distillation (Masango, 2005). Considering the cellular structure and mechanisms of mass and heat transfer in plant tissues, new methods for extracting active

ingredients have been developed. These methods include pressurized solvent extraction, ohmic heating extraction, supercritical fluid extraction, ultrasonic-assisted extraction to induce cell vibrations, increase temperature, and disrupt cell walls, and microwave-assisted extraction. These methods aim to increase yield, reduce extraction time, and improve the quality of essential oils. The reduction in extraction time with microwaves is likely due to the simultaneous mass and heat transfer. Additionally, the extraction speed using this method depends on the dielectric constant of the plant material and the power of the microwave device. Ultrasound waves in extraction create small shear forces and cavitation bubbles that disrupt cell walls, allowing effective oil release and mass transfer, thereby reducing extraction time and temperature. In ohmic-assisted hydro distillation (OAHD), the dielectric constant of the plant material is used to rapidly increase its temperature, which enhances extraction speed and reduces extraction time (Gavahian et al., 2020).

Steam distillation is a widely utilized method for extracting essential oils due to its cost-effectiveness and minimal environmental impact, as it employs water vapor instead of organic solvents (Masango, 2005). However, this technique often results in lower yields and longer extraction times, leading to increased resource consumption (Machado et al., 2024). Interestingly, laboratory experiments have demonstrated that the oil content in plant materials is higher than what is typically obtained through industrial steam distillation. This discrepancy suggests that industrial steam distillation equipment could be optimized to enhance oil yield, improve quality, and reduce extraction durations (Chemat et al., 2006).

The aforementioned methods have so far been developed on a laboratory scale. Due to high initial costs, excessive energy consumption, and limitations in manufacturing at the industrial scale, they have not yet reached industrial development. On an industrial scale, much simpler and widespread methods such as steam distillation are used. Investing in and adopting the

advanced methods involves high investment risks. On the other hand, industrial methods currently in use, like steam distillation, have drawbacks, including incomplete extraction of essential oils, which leads to significant economic losses and energy consumption. Therefore, efforts to improve existing methods are essential and of great importance.

This study examined the impact of an improved steam distillation system on the quality of essential oil extracted from cumin, the production of steam at appropriate temperatures to enhance the distillation process, and the complete extraction of essential oil compounds from the plant at a faster rate compared to conventional distillation methods.

MATERIALS AND METHODS

Preparation of Cumin Seeds

Cumin seeds (*Cuminum cyminum*) used in this study were procured in February 2024 from the online herbal store "Giyahine," located in Isfahan, Iran. Upon receipt, the seeds were meticulously sieved to remove any impurities and foreign materials. The botanical authenticity of the purchased seeds was subsequently verified by a qualified botanist. Following this verification, the cleaned seeds were stored in airtight plastic bags under controlled conditions until they were required for the experimental procedures (Figure 1).



Figure 1. Cumin seeds used in this Study

Essential oil extraction systems

In this research, an innovative version of steam distillation apparatus was used where the steam generation chamber was separated from the product chamber. This separation helps protect the product from temperature fluctuations and makes controlling the steam flow easier. Initially, water was boiled in a separate chamber, and the produced steam was transferred through a pipe to the product chamber. Then, the steam was directed to a condenser to condense, and finally, it was separated in a decanter. To regulate and control the temperature of the outgoing steam and determine the final steam temperature, a thermomanometer (pressure gauge and thermometer) and an adjustable needle valve were installed on the boiler lid. The reason for using this type of valve is that, instead of a disk, it has a long conical part at the end of the main valve stem, which reduces the seating area compared to globe valves and increases precision in flow control. Given the constant volume and using the Gay-Lussac law (Equation 1), these tools provided precise temperature control in this study. The schematic of the system used in this research is shown in Figure 2.

$$T_1 P_1 = T_2 P_2 \quad (1)$$

In this relationship, (P_1) and (P_2) represent the initial and secondary pressures (Pa), and (T_1) and (T_2) represent the initial and secondary temperatures (K) of the fluid, respectively.

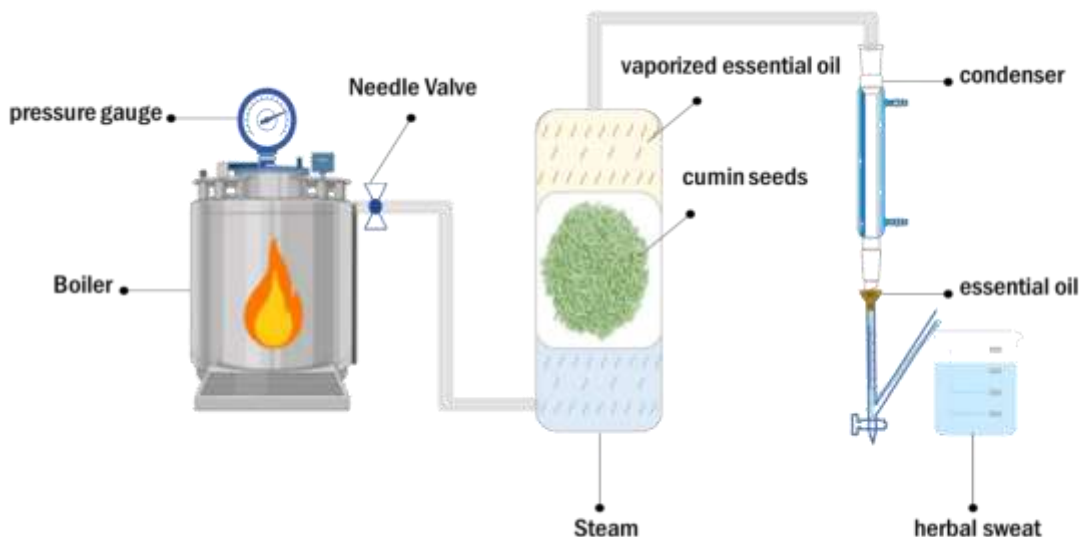


Figure 2. Schematic of the Innovative Steam Distillation System for Essential Oil Extraction

The existing apparatus in the Agricultural Engineering Properties Laboratory at Tarbiat Modares University was utilized to treat cumin seeds. This apparatus consists of a separate steam generator, a product placement chamber, silicone connecting hoses, a condenser, and a separating burette. To initiate the treatment, 100 g of cumin seeds were placed on a mesh inside the product placement chamber. The steam generator was then heated with a gas stove flame, and the temperature was carefully adjusted until it reached a steady state. Once the desired temperature was achieved, the essential oil extraction process began and continued for the predetermined duration, allowing for the collection of the essential oil in the separating burette.

For the extraction of the conventional sample essential oil, a Clevenger apparatus was utilized. Specifically, 50 g of cumin seeds were placed in a Clevenger flask, followed by the addition of 600 mL of distilled water. The mixture was then subjected to hydrodistillation under standard operating conditions until the essential oil was fully extracted (Wany et al., 2014). The experiment was conducted over four different time durations: 60, 90, 120, and 150 min. The cumin essential oil was then collected.

Essential Oil Percentage and Composition

Upon completing the essential oil extraction process in the extraction system, the collected essential oil was dehydrated by adding sodium sulfate (Na_2SO_4). The weight of the dehydrated essential oil was then measured using a scale with an accuracy of 0.0001 g. The percentage of extracted essential oil was calculated using the following Equation (Zhao et al., 2019).

$$\text{Percentage of Essential Oil} = \frac{\text{Amount of Essential Oil (g)}}{100 \text{ grams of cumin}} \times 100 \quad (2)$$

The components of the essential oils obtained in this study were determined using gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). The analyses were performed using an Agilent 7890B gas chromatograph equipped with a flame ionization detector (FID) and an HP-5 capillary column (30 m in length, 0.25 mm internal diameter, and 0.25 μm film thickness). The temperature program was set at 60°C for 2 min, then increased to 280°C at a rate of 5°C per minute.

The GC-MS analysis was performed using a Thermoquest-Finnigan gas chromatograph (Figure 3) equipped with an HP-5 column (60

meters $\times$ 0.25 mm, 0.25 μ m film thickness) and a mass spectrometer. Helium was used as the carrier gas at a flow rate of 1.1 mL per minute, with a split ratio of 1:100. The ionization voltage was set at 70 electron volts (eV), the ion source temperature at 200°C, and the interface temperature at 280°C. The mass range was set from 45 to 456 atomic mass units (AMU). The same temperature program as the GC was used. The essential oil components were identified by matching the mass spectra of each compound with those in the Wiley 7 and Adams reference libraries.



Figure 3. Gas Chromatograph used for qualitative analysis of cumin seed essential oil samples

Response Surface Methodology

In this study, the Response Surface Methodology (RSM) was used to analyze the data and implemented this approach using the Design Expert software. Specifically, the software's factorial design and Central Composite Design (CCD) test with 5 central point replications test were utilized to analyze the relevant data, examine the effects of independent variables on responses, and reduce the number of required treatments. Table 1 shows the selected levels of the independent variables for this study.

Table 1. Selected Levels of Independent Variables in RSM

Independent Variables	Coded Levels		
	-1	0	1
Steam Temperature (°C)	100	110	120
Duration of Essential Oil Extraction (min)	60	90	120

RESULTS AND DISCUSSION

Conventional Extraction Results

In this study, the quantity and quality of the essential oil extracted from cumin seeds using the Clevenger apparatus were examined over four time intervals: 60, 90, 120, and 150 min. According to Table 2, as the essential oil is gradually extracted from the surface channels of the cumin seeds, it was observed that with an increase in extraction time, the percentage of extracted essential oil increased as expected. However, the extraction rate decreased over time.

Table 2. Percentage of Essential Oil Extracted from conventional Samples at Different Time Intervals

Duration of Essential Oil Extraction (min)	Extracted Essential Oil (%)
60	1.3
90	1.5
120	1.5
150	1.6

In the analysis of the conventional samples' components using gas chromatography (GC), a total of 36 compounds were identified. The main compounds, which accounted for more than 94% of the essential oil content, included β -Pinene (4.9%), p-Cymene (3.2%), γ -Terpinene (9.5%), Cumin aldehyde (22.4%), α -Terpineol-7-al (9%), and γ -Terpinene-7-al (45%).

ANOVA Results for the Essential Oil Extracted by Steam Distillation System

The ANOVA results for the percentage of essential oil extracted are presented in Table 3. The findings from this analysis indicate that the model is significant and the lack of fit is not

significant. Therefore, the selection of the model and the analyses are reliable and valid. The effect

of the extraction duration (Factor A) is significant at the 1% level.

Table 3. ANOVA Table for the Extracted Essential Oil of Cumin Seed

Source of Variation	Sum of Squares	dF	Mean Square	F Value	P Value
Model	0.1218	5	0.0244	9.68**	0.0010
Time(A)	1.0130	1	1.0130	40.25**	0.0001
Temperature(B)	0.0033	1	0.0033	1.30	0.2770
A × B	0.0023	1	0.0023	0.8968	0.3640
A × A	0.0065	1	0.0065	2.57	0.1372
B × B	0.0077	1	0.0077	3.05	0.1086
Residual	0.0277	11	0.0025		
Lack of Fit	0.0252	7	0.0036	5.71	0.0558
Pure Error	0.0025	4	0.0006		

**Significant at the 1% probability level

Utilizing the RSM, a complete second-order polynomial model with a R^2 of 81.5 was selected to estimate the percentage of extracted essential oil by varying the coded values of the independent variables. A quadratic function was used to model the percentage of essential oil. The proposed real model is a second-order polynomial function as shown in Equation (3).

$$R1 = +7.29316 - 0.012785 * A - 0.098889 * B + 0.000079 * A * B + 0.000043 * A^2 + 0.000426 * B^2 \quad (3)$$

Effect of Extraction Duration on the Essential Oil

As shown in Figure 3, the overall trend in the percentage of essential oil increases with the extraction duration. It is observed that from 60 min to 80 min, the extracted essential oil increases slightly. However, extending the duration to 120 min results in reaching the maximum percentage of extracted essential oil, with a steep increase in the graph. In this study, the increase in the extraction duration led to an increase in the percentage of extracted essential oil, which is consistent with previous research in this field (Hamid et al., 2021; Malaka et al., 2017).

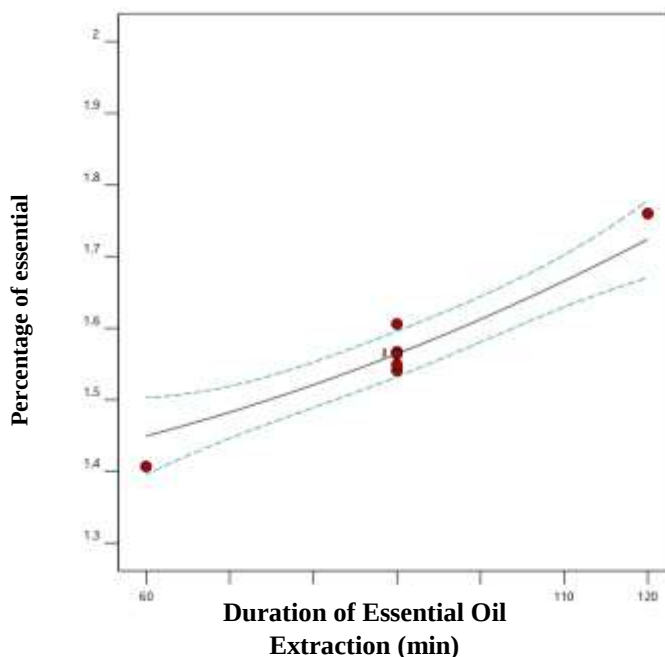


Figure 3. Percentage of essential oil extracted in different extraction time periods

Analysis of Cumin Seed Components

Based on the results of the analysis of cumin seed essential oil components, the extraction

temperature had the greatest impact on the essential oil components of cumin seeds. The summary of these results is presented in Table 4.

Table 4. Major Components of Cumin Seed Essential Oil at Different Extraction Temperatures

Compounds	RI	100°C	110°C	120°C
β -Pinene	935	5.3	5.9	7.1
p-Cymene	1030	3.8	4.2	4.5
γ -Terpinene	1064	9.5	11.3	14.0
Cumin aldehyde	1258	26.0	24.5	22.3
Terpinen-7-al < α ->	1300	6.7	6.2	7.9
Terpinen-7-al < γ ->	1308	43.7	42.1	38.9

The extraction temperature had the most significant impact on the components of cumin seeds, which is also confirmed by the other research studies (Felegary et al., 2023; Gogus et al., 2005). The increase in extraction temperature in this study led to a higher percentage of β -pinene and p-cymene in the extracted cumin seed essential oil, similar to the findings which using supercritical water extraction (Eikani et al., 2007). However, using higher steam

temperatures in this study resulted in a decrease in the percentages of cumin aldehyde and γ -Terpinene-7-al due to the thermal degradation of these heat-sensitive molecules. Over the past three decades, many scientific studies have reported the instability of essential oils and their aromatic compounds under the influence of extraction temperature. At high operational temperatures during distillation, these heat-sensitive metabolites typically decompose through carbocation intermediates, radical

intermediates, or pericyclic reactions (McGraw et al., 1999; Turek & Stintzing, 2013).

According to the International Organization for Standardization (ISO 9301:2003), the quality of cumin seed essential oil is reported in Table 5.

Table 5. Quality of Cumin Essential Oil

Compound	Minimum (%)	Maximum (%)
β -Pinene	7	20
p-Cymene	3	17
γ -Terpinene	14	32
Cumin aldehyde	15	46
Terpinen-7-al < α ->	1.5	16
Terpinen-7-al < γ ->	2.8	22

According to ISO 9301:2003

Optimization of Essential Oil Extraction Process

To optimize the essential oil extraction process using a modified steam distillation system, the

yield of essential oil must be maximized. According to the essential oil standard table (Table 5), cumin seed should have the β -Pinene and γ -Terpinene contents reach their maximum levels, as their concentrations are lower than the standard values. Additionally, the percentages of p-Cymene, Cumin Aldehyde, and α -Terpinen-7-al should remain unchanged, while the γ -Terpinen-7-al content should be minimized to align with the standard composition of cumin essential oil. Based on these criteria, the boundary conditions for the optimization process were established in this study.

One of the key aspects of the optimization process is assigning weights to the objective function variables. Given the higher significance of essential oil yield compared to other factors, a weight of 2 was assigned to this parameter. The remaining variables, which equally influence the essential oil quality, were each assigned a weight of 1. The optimal conditions determined using the software are presented in Table 6.

Table 6. Optimal Points of Independent Variables and Predicted Values of Dependent Variables by RSM

Time (min)	Temp (°C)	Essential oil (%)	β -Pinene (%)	p-Cymene (%)	γ -Terpinene (%)	Cumin aldehyde (%)	Terpine n-7-al < α -> (%)	Terpine n-7-al < γ -> (%)
1	115	112.23	1.71	6.56	4.31	12.18	24.70	7.38
2	115	112.14	1.71	6.56	4.31	12.18	24.70	7.38
3	115	112.39	1.71	6.56	4.31	12.17	24.69	7.38
4	115	112.07	1.71	6.56	4.31	12.18	24.71	7.38
5	115	112.48	1.71	6.55	4.30	12.17	24.69	7.37

In the cumin samples used in this study, the β -Pinene and γ -Terpinene contents in the Conventional group were lower than the standard values, while the γ -Terpinen-7-al content exceeded the standard range. The application of the constructed and optimized steam distillation system in this research led to an increase in β -Pinene and γ -Terpinene levels in some treatments and a reduction in γ -Terpinen-7-al content in certain treatments. However, the system was

unable to fully align these values with the standard composition.

Nevertheless, variations in chemical composition can be attributed to environmental factors, plant variety, harvesting stage, storage conditions, and essential oil extraction methods (Behera et al., 2004; Rebey et al., 2012). Among these factors, the extraction method plays a significant role in determining the composition percentages. This study successfully introduced beneficial modifications in this regard.

CONCLUSIONS

1. The duration of the extraction had the most pronounced influence on the yield of cumin seed essential oil, with the highest extraction yield occurring at 120 minutes.
2. An increase in extraction temperature led to elevated percentages of β -pinene and p-cymene in the essential oil.
3. The highest percentages of cumin aldehyde and γ -terpinene-7-al were observed at lower extraction temperatures; the relative proportions of these compounds declined as the temperature increased.

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