

# Optimization of Yield for Extraction of an Essential Oil from Cinnamon Using Microwave-Assisted Extraction

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## Abstract

Essential oil is concentrated hydrophobic liquid which containing volatile aromatic compounds extracted from flowers, leaves, stems, roots, seeds, barks, resins, or fruit rinds. These oils are often used for their flavor and their therapeutic or odoriferous properties, in a wide selection of products such as foods, medicines, and cosmetics. Extraction of essential oils is one of the most time and effort consuming processes. Cinnamon is an important herbal medicine with good effects of health promotion and disease prevention. In this work, the volatile compounds of cinnamon bark were extracted by Hydrodistillation and Microwave-assisted extraction (MAE) techniques. Gas chromatography/mass spectrometry was used to identify and quantify the volatile compound composition. The results indicated that cinnamaldehyde was the major component with the highest % area of 89.324% in the volatile oils extracted by MAE while in hydrodistillation method cinnamaldehyde was about 68%. MAE has been recognized as a technique with several advantages over other extraction methods, such as reduction of costs, extraction time, energy consumption, and CO<sub>2</sub> emissions. MAE technique is a green technique which is highlighted by increased extraction yield, decreased time and solvent consumption, moreover the reproducibility is better. For this purpose, various process parameters such as solid loading, water volume, microwave power and extraction time were studied in detail and optimized using the Taguchi method and Artificial neural network. The optimized extraction conditions were obtained at, solid loading of 30 g, water volume of 250 ml, microwave power of 700 W and extraction time of 25 min. Under optimized conditions, 4.169% (w/w) yield of essential oil was obtained.

**Keywords:** Essential oil; Therapeutic; Green technique; Taguchi method

## Introduction

Essential oils are concentrated volatile aromatic compounds produced by plants, the easily evaporated essences that give plants their wonderful scents. Essential oils are frequently referred to as the "life force" of plants. Unlike fatty oils, these "essential" oils are volatile, highly concentrated, substances extracted from flowers, leaves, stems, roots, seeds, bark, resin or fruit rinds. The amount of essential oils found in these plants can be anywhere from 0.01% to 10% of the total. The traditional technologies pertaining to essential oil processing are of great significance and are still being used in many parts of the globe. Water distillation, water and steam distillation, steam distillation, cohobation, maceration and enfleurage are the most traditional and commonly used methods. Maceration is adaptable when oil yield from distillation is poor. Distillation methods are good for powdered almonds, rose petals and rose blossoms, whereas solvent extraction is suitable for expensive, delicate and thermally unstable materials like jasmine, tuberose, and hyacinth. Water distillation is the most favored method of production of citronella oil from plant material.

## Microwave-assisted extraction

Microwave radiation interacts with dipoles of polar and polarizable materials. The coupled forces of electric and magnetic components change direction rapidly (2450 MHz). Polar molecules try to orient in the changing field direction and hence get heated. In non-polar solvents without polarizable groups, the heating is poor (dielectric absorption only because of atomic and electronic polarizations). This thermal effect is practically instantaneous at the molecular level but limited to a small area and depth near the surface of the material. The rest of the material is heated by conduction [1-5].

Thus, large particles or agglomerates of small particles cannot be heated uniformly, which is a major drawback of microwave heating.

It may be possible to use high power sources to increase the depth of penetration. However, microwave radiation exhibits an exponential decay once inside a microwave absorbing solid.

## Material & Methods

### Plant material

Cinnamon bark was used as experimental raw material in this study. 1 kg material was collected as samples from local stores. In this study, chemical usage was limited only for analysis purposes in order to ensure that the extracted essential oil is environmental friendly [6,7].

### Pre-treatment

The raw cinnamon bark contained some dirt particles and other adhering substances such as small sand particles. Cleaning was conducted on the material in order to remove these substances as much as possible. For this process, the fresh cinnamon bark samples were washed in running distilled water for about 60 min and dried in open air for 1 day at a temperature of about 25–30°C and relative humidity of about 30–40%. After this, approximately 100 g of dried cinnamon bark was crushed and grinded to reduce the sample particle size. After grinding, the samples were sieved using a mechanical sieve shaker to

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obtain less than 100 µm size of cinnamon bark powder. Essential oil, being volatile in nature, came out from the oil glands and along with water vapor it rose towards the condenser. The vapor mixture of water and oil was then condensed using a condenser. Upon condensing, the mixture was directed towards the collector, where essential oil got separated from the water. Moisture was removed from the essential oil and yield of oil was measured. The oil was stored at 0°C. The maximum temperature of the system was the boiling point of mixture and the system was operated at atmospheric pressure. To check the accuracy and reproducibility of the process, all the experiments were performed in duplicate. Yield was calculated by following formulas (Figure 1).

Yield (% w/w) = Weight of obtained essential oil / Weight of taken Cinnamon powder \* 100

Design of experiments (DOE) was adopted for optimization of various process parameters. The experimental planning was done with the help of the Taguchi method. In the present study, the important factors selected for optimization purpose were: solid loading, solvent quantity, microwave power and extraction time (Table 1) [8-10].

## Results & Discussion

### Effect of solid loading on yield

In the present study, solid loading was varied from 20 to 35 g, while solvent quantity, microwave power and extraction time was taken as per generated table data respectively. An increase in yield of essential oil was observed with an increase in the solid loading. However, this yield exhibited a decreasing trend as the solid loading was increased above 30 g (Figure 2). As solid loading was increased, microwave incident per particle reduced at a fixed power which in turn resulted in low dielectric heating and consequently reduced effect of microwave radiation. Also, the material residing in the internal part of the flask absorbs fewer microwaves in comparison with the material near the surface of the flask (Table 2) [11].

### Effect of solvent quantity on yield

Polar compounds are affected the most by microwave radiation; hence cinnamon powder was soaked in water. Enough quantity of water was added to ensure that the solid is being immersed and can be swelled during the extraction process. Volume of water was varied from 200 to 350 ml, while solid loading, microwave power level and extraction time was taken as per generated table data. An increase in yield of essential was observed with an increase in water volume up to 250 ml. At 300

ml of water volume, a decrease in yield was observed. The highest yield of cinnamon oil was observed at 250 ml solvent quantity (Figure 3) [12,13].

### Effect of microwave power level on yield

Effect of microwave power on cinnamon oil yield at different solvent quantity, solid loading, and extraction time of is shown in Figure 4. Four different microwave power levels were used in the extraction which are 280, 420, 490 and 700 W. From Figure 4, it can be seen that when the microwave power increases, the yield of extracted cinnamon oil also increased for all categories of the extraction power. Noteworthy is the highest yield of 4.169% which was obtained when cinnamon oil extracted was conducted at 700 W for a period of 25 min. In a similar research which was carried out by other researchers using the conventional method of hydrodistillation, report showed that the highest yield which was obtained from Cinnamon was about 2.38%. This indicates that the highest extraction yield (4.169%) obtained from MAE was considerably high compared to conventional methods (Figure 4).

### Effect of extraction time on yield

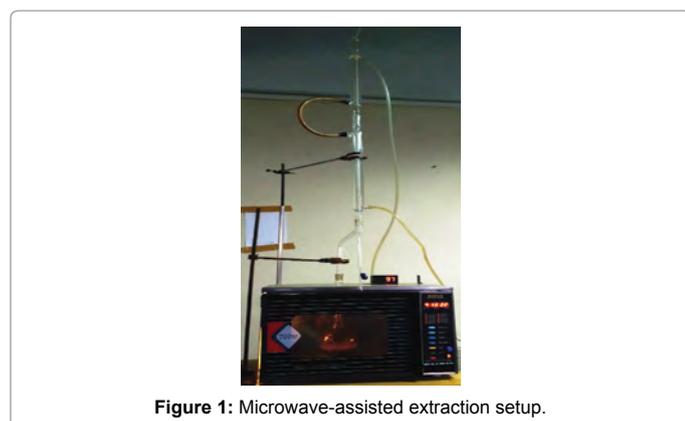
The extraction time was varied to examine the effect of radiation for distinctive time duration and in turn to observe an influence on mechanism of interaction between microwave, plant cell structure and quality of oil. Yield of oil was found to increase with increase in extraction time. Figure 5 shows that the yield of essential oil extracted from Cinnamon at different extraction time at a microwave power of 280-700 W, and at solvent quantity 200-350 ml. From the graph, it can be seen that the amount of yield increases as the extraction time was increased from 15 min to 30 min. The yield obtained at 25 min is 4.169% w/w. However, as the extraction time was increased beyond 25 min, the yield can be seen to be notably reduced [14].

### GC-MS analysis of cinnamon oil

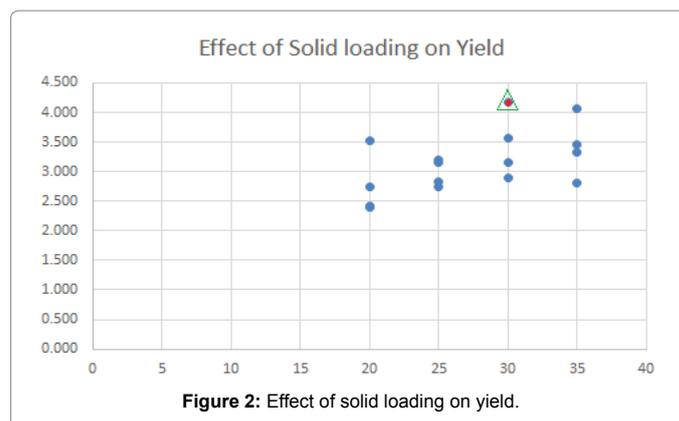
Gas Chromatography-Mass Spectrometry analysis was conducted in order to analyze the constituents which are present in the extracted oil. The extracted oil samples were analyzed with the aid of a GC-

Factors	Levels			
	1	2	3	4
A Solid Loading (g)	20	25	30	35
B Solvent quantity (ml)	200	250	300	350
C Microwave power level (W)	280	420	490	700
D Extraction time (min)	15	20	25	30

**Table 1:** Factors and their levels.



**Figure 1:** Microwave-assisted extraction setup.



**Figure 2:** Effect of solid loading on yield.

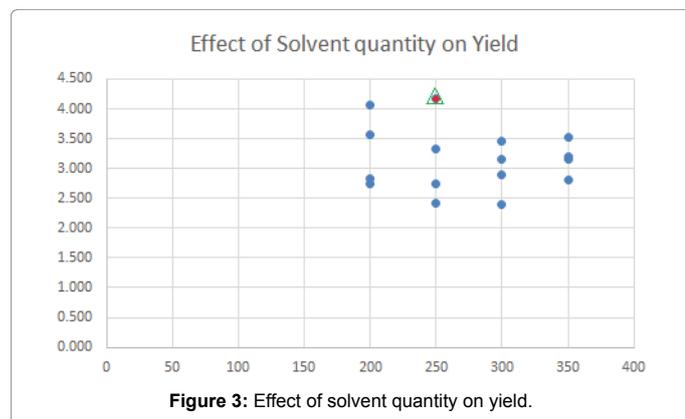


Figure 3: Effect of solvent quantity on yield.

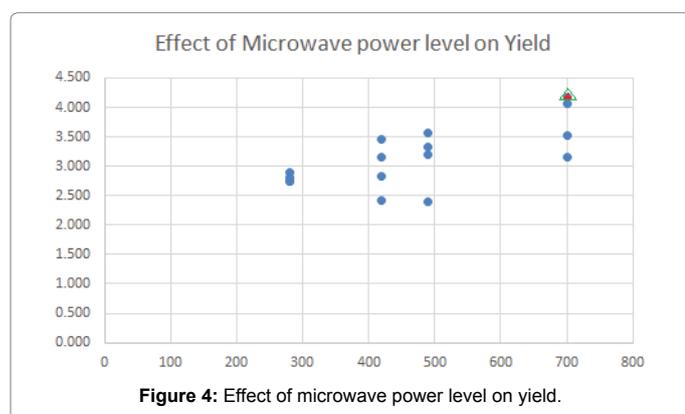


Figure 4: Effect of microwave power level on yield.

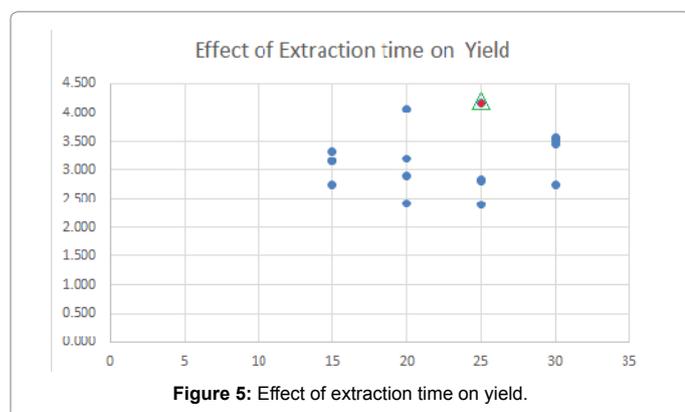


Figure 5: Effect of extraction time on yield.

MS instrument (Agilent 5975C inert, DDIT-ShahSchulman Surface Science & Nanotechnology Lab. Nadiad, Gujarat) coupled with a mass spectrometer. The GC system (model-Agilent 7890A) was paired with a DB-5 MS column with the dimensions of 30 m length  $\times$  0.25 mm internal diameter  $\times$  0.25  $\mu$ m film thickness. The mass selective detector was operated in the electron impact mode with electron energy of 70 eV (source temperature 230°C to evaluate the constituent of the oil extracted from the cinnamon bark and analyze its quality (Figure 6) [15-19].

## Conclusion

By GC-MS analysis different chemical components were identified in the essential oil of cinnamon bark, main component Cinnamaldehyde (89.324%) of the essential oil extracted from cinnamon bark, and this compound most responsible for the cinnamon's aroma, fragrant odor.

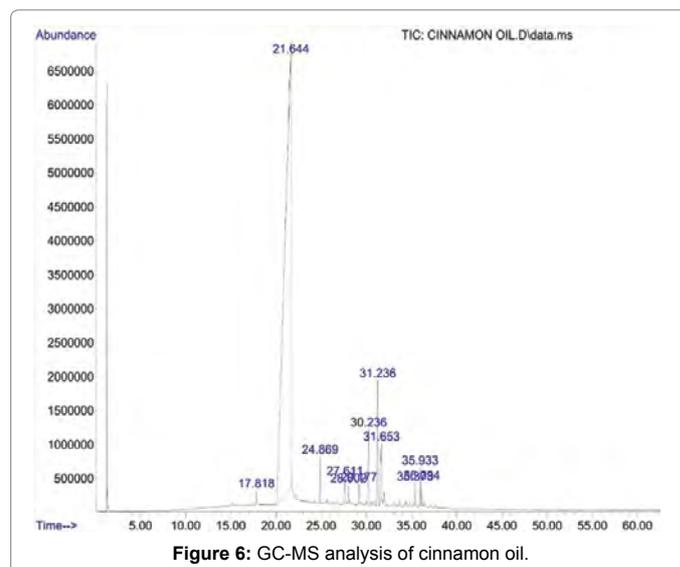


Figure 6: GC-MS analysis of cinnamon oil.

Sr. no	Solid loading (g)	Solvent quantity (ml)	Microwave power level (W)	Extraction time (min)	Yield (% w/w)
1	20	200	280	15	2.741
2	20	250	420	20	2.416
3	20	300	490	25	2.395
4	20	350	700	30	3.516
5	25	200	420	25	2.826
6	25	250	280	30	2.734
7	25	300	700	15	3.163
8	25	350	490	20	3.194
9	30	200	490	30	3.558
10	30	250	700	25	4.169
11	30	300	280	20	2.892
12	30	350	420	15	3.159
13	35	200	700	20	4.056
14	35	250	490	15	3.318
15	35	300	420	30	3.449
16	35	350	280	25	2.817

Table 2: Experimental layout using L16 orthogonal array and the responses.

HD method gives cinnamaldehyde component about (68%). So, MAE method gives better yield of cinnamaldehyde compared to HD method and MAE also gives better overall yield of cinnamon oil compared to HD method. The optimized extraction conditions were obtained at, solid loading of 30 g, water volume of 250 ml, microwave power of 700 W and extraction time of 25 min. Under optimized conditions, 4.169% (w/w) yield of essential oil was obtained. Observations from this research shows that MAE technique produced more oxygenated compounds, is more cost effective and environmental friendly. These results indicate that cinnamon oil extracted through MAE method exhibit better properties especially in terms of quality when with conventional HD technique. It also suggests that MAE is suitable for extracting volatile oils from cinnamon barks without necessarily causing any adverse change to the chemical composition of the oil.

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