Star Anise Essential Oil Extraction by Hydro distillation Techniques

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Abstract: In the recent years, there have been growing interests on Star Anise due to its antiflu properties. Traditionally the fruit had been used as carminative, digestive, dyspepsia, antispasmodic, and stimulant, anti-rheumatic and diuretic. It is also the primary source of Shikimic acid used to produce Oseltamivir Phosphate, sold under the brand name, Tamiflu. Many research studies have been carried on Star Anise in regard to its preparation, health benefits, nutritional values, pharmacognostic and phytochemical study of the oil. Despite numerous studies on the botanical fruit and its oil, there is no report on comparative study of Indian Star Anise.

This study centred on the extraction of Star Anise using hydrodistillation. Extraction parameters (extraction time, extraction power, solid loading, water volume, particle size) were used to determine which parameter are suitable or recommendable for optimum extraction of sample oil. Extraction time, power and particle size where the dominating factors. In order to study the compositions of the oil extracted constituents were analyzed by GC-MS method. In GCMS, Anethole was found as a dominant contributor.

Keywords: Anethole, Essential oil, GCMS, Hydrodistillation, Star Anise.

I. INTRODUCTION

Essential oil is a concentrated hydrophobic liquid containing volatile aroma compounds from plants. They are also known as volatile oil, ethereal oils, aetherolea or simply as the oil of the plant. An oil is essential in the sense that it contains the essence of plant’s fragrance. Essential oil can be extracted from plant, leaves, barks, flowers, stem, and seeds. Essential oil bring a wide range of health benefits, unlike modern drugs they have no side effects. Mostly, essential oil are obtained by distillation although other methods include expression, solvent extraction, absolute oil extraction, resin tapping and cold pressing[1].

In this research study, botanical fruit Star Anise. Illicium verum Hook. f. (Illiciaceae) is an aromatic evergreen tree bearing purple-red flowers and anise-scented star-shaped fruit. It grows almost exclusively in southern China, Vietnam and India[10]. Its fruit (star anise) is an important traditional Chinese medicine as well as a commonly used spice.

The characteristically shaped fruit is listed in Chinese Pharmacopoeia and has been applied as a traditional Chinese medicine to treat vomiting, stomach aches, insomnia, skin inflammation and rheumatic pain[8]. Traditionally, the essential oil of star anise is used topically for rheumatism and as an antiseptic. It is also the primary source of Shikimic acid used to produce Oseltamivir Phosphate, sold under the brand name, Tamiflu[2,7].

There are several methods in the extraction of oil. Conventionally, in the production/extraction of essential oil, steam distillation and hydro distillation has been recognised as the most advanced process in distillation due to its highly sensitive to heat. It is specially used for temperature sensitive materials like resins and oils that are not miscible with water and possibly give way when attaining their boiling point [3]. Apart from been economical, it safe time and energy when compared to other distillation methods. Series of studies has been reported on essential oil of Star Anise, there has not been any report in relation to the extraction of this oil with the use hydro distillation of India Star Anise. Therefore, this study is aimed at bridging this gap by employing steam distillation method in the extraction of essential oil from Star Anise.

II. MATERIAL AND METHOD

A. Plant Material

Samples of botanical fruits Star Anise was collected from Spice Market, Kappalandimukku, Mattancherry, Kochi, Kerala (India) in January 2019. The identity of the Star Anise was confirmed by plant agriculture scientist Chandra Sekhar Azad University, Kanpur, UP (India). The botanical fruit were then made to get dried in a dark room under ambient conditions (20-30°C) for five days placed on a large transparent tray. It was packaged in a poly ethylene bag, kept for further work.
B. Hydrodistillation

Samples of the botanical fruits were crushed to powder by Mortar and pestle. Then sieved by differential sieve method and separated according to size (10mm i.e. petals, 4.76mm i.e. 4/7mesh, 2.83mm 7/25mesh, 0.707mm i.e. 25 mesh). Weighed sample of 25gm was placed in a round bottom flask (1000mL) containing 250mL of distilled water which was attached to a Clevenger type-apparatus. The heat was permitted to go through the samples filled with water. The both liquid (oil+water) were fed through the condenser having an inlet and outlet water ways to the 3-way tap where each liquid are separated by skimming it off the top. Then oil was dried with Sodium Sulphate Anhydrous. The oils were then kept in amber vials and weighed on a portable digital weighing balance. The extraction time was 3 hours.

![Fig 1 Photograph Clevenger Hydrodistillation](image)

III. IDENTIFICATION OF COMPOUNDS

The components of the Star Anise essential oil were identified base on the comparison of their retention indices and mass spectra with those standards, database of the GC/MS system and published data.

| S.No. | Compound               | Analyzed oil | Literature ref[5] |
|-------|------------------------|--------------|-------------------|
| 1     | Anethole               | 93.96        | 74.96             |
| 2     | Benzaldehyde           | 1.89         | 0.06              |
| 3     | D-Limonene             | 1.2          | 1.94              |
| 4     | 1,3,3-trimethyl-1,3,7-Octatriene | 0.47 | - |
| 5     | Estragole              | 0.47         | 5.15              |
| 6     | Pyridine               | 0.37         | -                 |
| 7     | Eucalyptol             | 0.26         | -                 |
| 8     | α-Terpineol            | 0.22         | 0.13              |
| 9     | Terpinen-4-ol          | 0.19         | 0.24              |
| 10    | Octasiloxyxane         | 0.19         | -                 |
| 11    | Silicic acid           | 0.18         | -                 |
| 12    | α-Pinene               | 0.11         | 0.94              |
| 13    | 3-Carene               | 0.11         | 0.11              |
| 14    | 2-Propanone            | 0.1          | -                 |
| 15    | Cyclotrisiloxane       | 0.09         | -                 |
| 16    | 2-Pentanone            | 0.07         | -                 |
| 17    | p-Cymene               | 0.07         | 0.20              |
| 18    | Cyclotrisiloxane       | 0.04         | -                 |
IV. RESULT

In this study, Star Anise was extracted using hydro distillation method. The graph below summarizes the extraction parameters (percentage oil extracted with time taken for extraction on variation of particle size and heater power) hydrodistillation techniques.

A. Variation of Particle Size

Uncrushed petal i.e. 10.000mm  
4/7 Mesh i.e. 4.760mm  
7/25 Mesh i.e. 2.830mm  
25 Mesh undersize i.e. 0.707mm

1) Fixed Parameter

a) Sample loading=25g  
b) Water quantity=250ml  
c) Heater power=300w

B. Variation of Power

150 watt, 300 watt, 600 watt, 1000 watt

1) Fixed Parameter

a) Sample loading=25g  
b) Water quantity=250ml  
c) Particle size=25 mesh (0.707mm)
The weight of oil extracted was calculated based on the equation (1)
\[ W_{sa} = W_f - W_o \]  
Where:
- \( W_{sa} \) = weight of extracted star anise (gm)
- \( W_o \) = empty weight of bottle (gm)
- \( W_f \) = weight of bottle with oil (gm)

The percentage extracted oil yield is the amount of oil extracted in percentage [2] and was calculated as given below:
\[ \text{Yield (\%)} = \frac{W_{sa}}{Wi} \times 100 \]  

V. DISCUSSION

From fig 2, 25g of sample of different particle size were used for the hydrodistillation methods.

When sample was complete petal of mean particle diameter was 10mm was used in hydrodistillation, then essential oil yield was 6.99% in 4 hour.

Similarly 4/7 mesh, particle diameter was 4.760mm was used in hydrodistillation, then essential oil yield was increased to 7.38% in 4 hour.

Similarly 7/25 mesh and 25 mesh, particle diameter was 2.830mm and 0.707mm was used in hydrodistillation, then essential oil yield was same for both 8.16%. But 7/25 mesh takes 4 hours whereas 25 mesh takes only 3 hours.

From fig 3, 25g (25 mesh) of sample was hydrodistilled at different heater power.

When heating mantle was to 150watt, essential oil yield was 7.38% in 4 hours.

Similarly, when heating mantle was to 300watt, 600watt and 1000wattt, then essential oil yield was 8.16% for all cases. But 1000watt takes least time i.e. 2 hours.

VI. CONCLUSION

The following conclusion(s) can be drawn:
The botanical fruit (Star Anise) is heat sensitive which give rise in higher oil yield at short extraction time on increasing power of heater. At 1000watt, yield was 8.16% in 2 hours. Star anise essential oil is directly proportional heater power.

On decreasing particle size, star anise essential yield increases. Star anise essential in inversely proportional to the optimum parameter were found are 25 mesh, 1000 w for 2 hours gives yield of 8.16%.

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