

Extraction and Characterization Study of Aniseed Oil

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Abstract: - Anise belongs to Umbelliferae family originates from Asian countries. The essential oil is extracted from the fruits but roots are also used. Extraction of Oil from Aniseed was performed by using Steam Distillation Method. Aniseed Oil which has now replaced the fruits for medicinal and flavoring purposes. Chemical composition was done by GC. Physical analysis of Aniseed Oil such as saponification value, iodine value, acid value, specific gravity, etc. was done in laboratory. Yield of Aniseed Oil obtained for steam Distillation process varied with respect to temperature, time heating and quantities of raw Material. The temperature range for effective separation of essential oil and water was 60-70°C.

Major component identified in the extracts were anethole, methylchavicol and *Cis-pseudoisoeugenyl* 2-methylbutyrate. Quality parameter of Oil extracted with Steam Distillation are not much different but only anethole content is more than any other methods and were in range of 89-92% content.

Keywords: Aniseed oil, GC, acid value, saponification value, etc.

I. INTRODUCTION

Anise (*Pimpinella anisum*), also called **aniseed**, is a flowering plant in the family Apiaceae native to the eastern Mediterranean region and Southwest Asia. Its flavor has similarities with some other spices, such as star anise, fennel, and licorice. Anise is an herbaceous annual plant. The leaves are simple, long and shallowly lobed; the flowers are white, produced in dense umbels. The fruit is a dry long, usually called "aniseed". Composition of Aniseed Moisture: 9-13%, Protein: 18%, Fatty oil: 8-23%, Essential oil: 2-7%, Starch: 5%, N-free extract: 22-28%, Crude fibre: 12-25% Aniseed contains 2-3% Oil. Main constituent of Oil is Anethole 80-90 % [1]. But it may also contain lipids rich in fatty acids. Aniseed oil is used in food processing industry to impart flavor to cakes. Anethole is used for synthesis of Drug chloral an anticonvulsion agent and pentobarbital. We are all familiar with the pleasant odors coming out from flowers, spices and many trees. The essence or aromas of plants are due to volatile oils present in them. These smelling volatile oils present in plants are called essential oils. Essential oils contain highly volatile substances that are isolated by a physical method or process Oils are used in the embalming process, in medicine and in purification rituals.

Analysis of Essential Oil is done by using Gas Chromatography with Mass Spectrometer. The qualitative and quantitative analysis is done to know the constituents in the

oil and the percentage of components present in the oil. Therefore Objective of this work was to study Extraction and characterization of raw Aniseed Oil at different condition.

II. METHOD OF EXTRACTION

Several techniques had been implemented for the extraction of oils like turbo distillation, super critical fluid extraction [1], Solvent extraction, hydro distillation, use of superheated and supercritical water and other such as microwave assisted processes, Steam distillation [5] etc. Out of these techniques the Steam distillation is one of the promising techniques for extraction of oil.

Aniseed, on steam distillation, yields an essential oil, known as 'Oil of Aniseed', which has now replaced the fruits for medicinal and flavoring purposes. Aniseed oil is a colorless or pale-yellow liquid having the characteristic odor and taste of the fruit. The yield of oil generally varies from 1.9 to 3.1 percent. Higher values up to 6 per cent have been reported from Syrian aniseed. Crushing of fruits prior to distillation gives better yields of oil. The material should be distilled soon after the crushing to prevent any loss of oil due to evaporation. Aniseed oil is a highly refractive liquid, which solidifies on cooling. The congealing point depends much on the anethole content and is a valuable criterion for evaluating the oil. Exposure of the oil to air causes polymerization, and some oxidation also takes place with the formation of anisaldehyde and anisic acid. The chief constituent of aniseed oil is anethole, which is present to the extent of 80 to 90 per cent and is mainly responsible for the characteristic flavor of the oil. The oil also contains methyl chavicol, p-methoxyphenyl acetone, and small amount of terpenes and sulfur containing compounds of disagreeable odor.

Experimental setup: Fig no.1 Clevanger apparatus



Clevanger apparatus [1] consists of a steam generator connected to the round bottom flask through a glass inlet tube. The flask is connected to a water condenser through a glass outlet tube. Condenser is further attached to a receiver through an adaptor.

III. PROCEDURE OF STEAM DISTILLATION

1. Take about 750 ml of water in the steam generator and start heating to produce steam.
2. In the round bottom flask take crushed sauff.
3. A vigorous current of steam from steam generator is passed through the round bottom flask.
4. A part of the steam condenses in the round bottom flask. As more and more steam is passed, the steam volatile components of sauff pass through the condenser along with steam. These contents on condensation are collected in the receiver.
5. The contents in the round bottom flask may be heated by a heating mental to prevent excessive condensation of steam.
6. The process of steam distillation is continued for about half an hour.
7. Transfer the distillate to a separating funnel.
8. Wait for one day allow settling down to found two layer of phases of different density, raffinate and extract phases.
9. Separate the two layers.
10. Find the weight of the extracted essential oil. Note the color, odor and weight of the essential oil.

IV. RESULTS AND DISCUSSION

Batches:

1) first batch of distillation and results:

Quantity of aniseed = 150 gm
 Water quantity = 500 ml
 Temperature = 70°C
 Oil recovery = 2 ml

2) Second batch of distillation

Quantity of aniseed = 150 gm
 Water quantity = 400 ml
 Oil recovery = 3 ml

3) Third batch of distillation

Quantity of aniseed = 250 g
 Water quantity = 300 ml
 Oil recovery = 2 ml

4) Fourth batch of distillation

Quantity of aniseed = 350 gm
 Water quantity = 350 ml
 Oil recovery = 4 ml



Fig no. 2 Collected Oil & water

V. GAS CHROMATOGRAPHY

Gas Chromatography (GC) is a method that combines the features of gas- liquid chromatography and to identify different substances within a test sample. Applications of GC-MS include drug detection, fire investigation, environmental analysis, explosives investigation, and identification of unknown samples.

VI. INSTRUMENTATION

The GC-MS is composed of two major building blocks: the gas chromatograph and the mass spectrometer. The gas chromatograph utilizes a capillary column which depends on the column's dimensions (length, diameter, film thickness) as well as the phase properties (e.g. 5% phenyl polysiloxane).

Details of column used CP-Sil 8 CB (30 m long, 0.53 mm i.d. and 0.35 µm inner film thickness). Z

Sr. no.	Parameter	Results
1	Methylchavicol	2.5
2	p-anisaldehyde	2.1
3	anethole	90.43
4	himachalene	0.70
5	Cis-pseudoisoeugenyl 2-methylbutyrate	2.3
6	Trans pseudoisoeugenyl 2-methylbutyrate	0.45
7 4)	Fourth batch of distillation	1.52

VII. ANALYSIS OF PHYSICAL PARAMETER

The color of Anisee oil was measured using spectrophotometer. The Saponification value of oil is determined by laboratory method using HCl, KOH chemical. Acid value determines free fatty acid content in oil by laboratory method. Refractive Index was measured by using Refractometer at 25 C.

Properties of Aniseed Oil:

Sr. no.	Parameter	Results
1	Color	Pale Yellow
2	Specific gravity	0.987
3	Saponification value	168.3
4	Acid value	2.55
5	Iodine value	99
6	Refractive index	1.55
7	Odor	Sweet like Anethole

VIII.CONCLUSION

It was observed that very less work was done in obtaining Oil from Aniseed by Steam distillation method. Volume of Essential Oil increases with increases in time of heating keeping the temperature constant. On decreasing the pressure we can extract Essential Oils at relatively less temperature and within less time of heating.

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