

INTRODUCTION

The word perfume derives from the latin "per fumum" meaning through smoke, is fragrant liquid that is sprayed or rubbed on the skin or clothes to give a pleasant smell. Extraction of perfume from various plants resources is of ancient origin. In fact, the natives from different tropical regions of the globe have long been extracting oil from numerous oil-bearing plants. Human since the ancient time have known how to extract oil from their natural resources. Vegetable oils are naturally occurring esters of higher fatty acids and glycerol. They are widely distributed in nature and were first consumed as food. Later oils were discovered to be used as renewable raw materials for variety of non-food production, for instance perfumes, disinfectants, inks to mention but a few.

STATEMENT OF THE PROBLEM

There is a high demand of essential oils for various purposes such as medicinal, perfumery, soap making, insecticides to mention but a few have opened up wide opportunities for global warming. Imported essential oils are very expensive to meet the demand of our local consumer industries, therefore it becomes necessary to source and extract these oils from local source. In particular perfumes that are usually imported can be produced locally from a vast variety of oil-bearing plants yet to be explored. It has also been observed that high concentration of perfumes in the epidermal tissues can cause skin irritation or peeling due to poor formulation. This problem will be explored on this project. Solvent extraction is the safest method for extracting high quality oil because some herbs and spices cannot be extracted from enfleurage method but it has the disadvantage of having residual solvents in the essential oils.

The research is conducted in order to use hydro distillation as one of the applicable methods to extract essential oils. Hydro distillation has the advantage of no solvent residues as an alternative to conventional extraction techniques. This research will reveal the difference in yields by the methods.

OBJECTIVE OF THE RESEARCH

The main objective of this research is to extract essential oil from lemon grass (*Cymbopogon citrasus*) using solvent extraction, enfleurage, and hydro distillation and formulating the perfume.

SOURCES OF PERFUMES

➤ Aromatics sources

Plant sources

Plants have long been used in perfumery as a source of essential oils and aroma compounds. These aromatics are usually secondary metabolites produced by plants as protection against herbivores, infections, as well as to attract pollinators. Plants are by far the largest source of fragrant compounds used in perfumery. The sources of these compounds may be derived from various parts of a plant. A plant can offer more than one source of aromatics, for instance the aerial portions and seeds of coriander have remarkably different odors from each other. Orange leaves, blossoms, and fruit zest are the respective sources of petitgrain neroli, and orange oils.

Bark: Commonly used barks include cinnamon and cascarilla. The fragrant oil in sassafras root bark is also used either directly or purified for its main constituent, safrole, which is used in the synthesis of other fragrant compounds.

Flowers and blossoms: Undoubtedly the largest and most common source of perfume aromatics. Includes the flowers of several species of rose and jasmine, as well as osmanthus, plumeria, miosa, tuberose, narcissus, scented geranium, cassie, ambrette as well as the blossoms of citrus and ylang-ylang trees. Although not traditionally thought of as a flower, the unopened flower buds of the clove are also commonly used. Most orchid flowers are most commercially used to produce essential oils or absolutes, except in the case of Vanilla, an orchid, which must be pollinated first and made into seed pods before use in perfumery.

Fruits: Fresh fruits such as apples, strawberries, cherries unfortunately do not yield the expected odors when extracted; if such fragrance notes are found in a perfume, they are synthetic. Notable exceptions include litsea cubeba, vanilla, and juniper berry. The most commonly used fruits yield their aromatics from the rind; they include citrus such as oranges, lemons, and limes. Although grapefruit rind is still used for aromatics, more and more commercially used grapefruit aromatics are artificially synthesized since the natural aromatic contains Sulfur and its degradation product is quite unpleasant in smell.

Leaves and twigs: Commonly used for perfumery are lavender leaf, patchouli, sage, violets rosemary, and citrus leaves. Sometimes leaves are valued for the "green" smell they bring to perfumes, examples of this include hay and tomato leaf.

Resins: Valued since antiquity, resins have been widely used in incense and perfumery. Highly fragrant and antiseptic resins and resincontaining perfumes have been used by many cultures

as medicines for a large variety of ailments. Commonly used resins in perfumery include labdanum, frankincense, myrrh, Peru balsam, gum benzoin. Pine and fir resins are a particularly valued source of terpenes used in the organic synthesis of many other synthetic or naturally occurring aromatic compounds. Some of what is called amber and copal in perfumery today is the resinous secretion of fossil conifers.

Roots, rhizomes and bulbs: Commonly used terrestrial portions in perfumery include iris rhizomes, Vetiver roots, various rhizomes of the ginger family.

Seeds: Commonly used seeds include Tonka bean, carrot seed, coriander, caraway, cocoa, nutmeg, mace, cardamom, and anise.

Woods: Highly important in providing the base notes to a perfume, wood oils and distillates are indispensable in perfumery. Commonly used woods include sandalwood, rosewood, Agarwood, birch, cedar, juniper, and pine. These are used in the form of macerations or rectified (distilled) forms.

Animal sources

Ambergris: Lumps of oxidized fatty compounds, whose precursors were secreted and excreted by the sperm whale. Ambergris should not be confused with yellow amber, which is used in jewelry. Because the harvesting of ambergris involves no harm to its animal source, it remains one of the few animalic fragrance agents around which little controversy now exists.

Castoreum: Obtained from the odorous sacs of the North American beaver.

Civet: Also called Civet Musk, this is obtained from the odoriferous sacs of the civets, animals in the family Viverridae. The World Society for the Protection of Animals investigated African civets caught for this purpose.

Hyraceum: Commonly known as "Africa Stone", is the petrified excrement of the Rock Hyrax.

Honeycomb: From the honeycomb of the honeybee. Both beeswax and honey can be solvent extracted to produce an absolute. Beeswax is extracted with ethanol and the ethanol evaporated to produce beeswax absolute.

Deer musk: Originally derived from the musk sacs from the Asian musk deer, it has now been replaced by the use of synthetic musk sometimes known as "white musk".

Other natural sources

Lichens: Commonly used lichens include oakmoss and treemoss thalli.

"Seaweed": Distillates are sometimes used as essential oil in perfumes. An example of commonly used seaweed is *Fucus vesiculosus*, which is commonly referred to as bladder wrack. Natural seaweed fragrances are rarely used due to their higher cost and lower potency than synthetics.

➤ Synthetic sources

Aroma compound

Many modern perfumes contain synthesized odorants. Synthetics can provide fragrances which are not found in nature. For instance, Calone, a compound of synthetic origin, imparts a fresh ozonous metallic marine scent that is widely used in contemporary perfumes. Synthetic aromatics are often used as an alternate source of compounds that are not easily obtained from natural sources. For example, linalool and coumarin are both naturally occurring compounds that can be inexpensively synthesized from a terpenes. Orchid scents (typically salicylates) are usually not obtained directly from the plant itself but are instead synthetically created to match the fragrant compounds found in various orchids.

One of the most commonly used classes of synthetic aromatic by far are the white musk. These materials are found in all forms of commercial perfumes as neutral background to the middle notes. This musk is added in large quantities to laundry detergents in order to give washed clothes a lasting "clean" scent.

FORMULATION OF PERFUMES

Perfume oils usually contain tens to hundreds of ingredients and these are typically organized in a perfume for the specific role they play. These ingredients can be roughly grouped into four groups:

Primary scents (Heart):- can consist of one or a few main ingredients for a certain concept such as —rose|. Alternatively, multiple ingredients can be used together to create an —abstract| primary scent that does not bear a resemblance to a natural ingredient. For instance, jasmine and rose scents are commonly blended for abstract floral fragrances.

Modifiers: These ingredients alter the primary scent to give the perfume a certain desired character for instance fruit esters may be included in a floral the cherry scent in cherry cola can be considered a modifier.

Blenders: A large group of ingredients that smooth out the transitions of a perfume between different layers or bases. These themselves can be used as a major component of the primary scent. Common blending ingredients include linalool and hydroxycitronellal.

Fixatives: they are used to support the primary scent by bolstering it. Many resins, wood scents, and bases are used as fixatives. The top, middle, and base notes of a fragrance may have separate primary scents and supporting ingredients. The perfume's fragrance oils are then blended with ethyl alcohol and water aged in tanks for several weeks and filtered through processing equipment to respectively allow the perfume ingredients in the mixture to stabilize and to remove any sediment and particles before the solution can be filled into the perfume bottles.

MATERIAL AND METHODS

SAMPLE PREPARATION

Fresh Lemongrass sample was collected from the garden in Caritas University Enugu in Enugu State. The sample was allowed to dry for about three days in the laboratory. The leaves were later cut into slices to reveal the tighter inner stem until when ready for use.



Figure 1: lemon grass

APPARATUS AND REAGENTS

- 1.A retort stand
- 2.500ml Separation funnel
- 3.250ml and 100ml Beakers
- 4.Electronics weighting balance (V 100)

5. Water bath (DC 1000)
6. Mortar and pestle
7. 500ml Round bottom flask
8. Knife
9. Aluminum foil
10. Electric heater
11. Distilled water
12. N-hexane
13. Ethanol
14. Olive oil
15. Lemon grass

PROCEDURE FOR SOLVENT EXTRACTION METHOD

130g of the dry sample of lemongrass were weighed from the sliced lemongrass sample and placed in a 500ml clean flat bottom flask. 600ml of N-hexane solvent were poured into the 500ml flask and stopped. The flask and content were allowed to stand for 24hrs; this was done to extract all the oil content in the lemongrass and for complete extraction. After which the extract was decanted into another 500ml beaker. 200ml of Ethanol were added to extract the essential oil since essential oil is soluble in Ethanol. The mixture was then transferred to 500ml separating funnel and separated by a process called liquid/liquid separation process. The content of the separating funnel was allowed to come to equilibrium, which separated into two layers (depending on their different density). The lower Ethanol extract and the upper Hexane layer were collected into two separate 250ml beaker and were placed in a water bath at 78°C. This was done to remove the Ethanol leaving only the natural essential oil. The yield of oil was determined by weighing the extract on an electronic weighing balance. The difference between the final weight of the beaker with extract and the initial weight of the empty beaker gave the weight of essential oil.



Figure 2: Solvent extraction separation

PROCEDURE FOR ENFLEURAGE METHOD

130g of the dry sample of lemongrass were weighed out and pounded with mortar and pestle (to reveal the tighter inner stem). The pounded sample was then placed in a 500ml beaker. About 70ml of lightflavored olive oil were warmed and mixed with the mashed lemongrass (to allow for efficient absorption of the essential oil). The beaker was covered with aluminum foil and shaken until the lemongrass was distributed throughout the oil. It was then allowed to stand for 24hours at room temperature for proper absorption. 140ml Ethanol were added to absorb the essential oil leaving behind the light-flavoured olive oil and the lemongrass residue. The Ethanol extract was decanted and placed on a water bath at 78oC to vaporize the Ethanol leaving behind the essential oil. The yield of oil was determined by weighing on an electronic weighing balance. The difference between the final weight of the beaker and the initial weight gave the yield of essential oil.

HYDRODISTILLATION METHOD

130g of fresh lemongrass sample were placed into a 500ml round bottom flask containing 250ml of distilled water. The flask was fitted with a rubber stopper connected to a condenser and heated. Water at 0°C flowed counter currently through the condenser to condense the ensuring steam. When the water reached 100oC it started boiling ripping off the essential oil from the lemongrass. When the lemongrass got heated up, the essential oil that was extracted from the leaf mixed with the water vapour. Both passed through the condenser and the vapour was condensed into liquid. With the use of ice block, cooling was made possible and

volatilization of the essential oil was avoided. The condensate was directly collected using a 500ml beaker and then poured into a separating funnel. This formed two layers of oil and water. The tap of the separating funnel was opened to let out the water while the oil was immediately collected into a 100ml stoppered bottle. The bottle was closed tightly to prevent vaporization of the essential oil. The oil was collected and the volume of oil obtained was weighed.

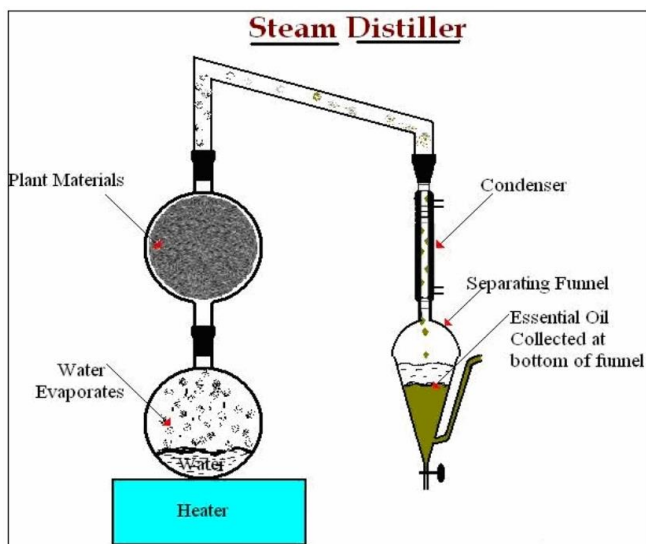


Figure 3: Extraction of essential oil using water distillation

FORMULATION OF PERFUME WITH LEMON GRASS ESSENTIAL OIL PRODUCED

- 1.Apparatus and reagents
- 2.Pipette
- 3.Funnel
- 4.50ml and 120ml beakers
- 5.Perfume bottle
- 6.Fixatives (Surprise and Dream)
- 7.Methanol
- 8.Distilled water
- 9.Lemongrass essential oil

PROCEDURE

10ml of lemongrass essential oil extract were measured and placed in a 120ml beaker containing 5ml of Methanol. 5ml of the Fixatives were added to the mixture (to improve the longevity of the perfume). The solution were shaken and poured into a 50ml bottle.

RESULTS AND DISCUSSIONS

SOLVENT EXTRACTION METHOD

Result obtained by solvent extraction is shown in Table 1 below.

Weight of oil (g)	Time (min)
0.2	240
0.3	480
0.7	720
0.72	960
0.78	1200

The amount of essential oils obtained by solvent extraction method was 2.7g of essential oil per 130g of dry lemongrass sample. This gave 2.08% yield of essential oil per 130g of dry lemongrass. The temperature used was 780C i.e. the boiling point of ethanol. The volume of essential oil was measured at every 4hr interval to determine the oil yield at varying time. As the time increases the Ethanol solvent reduces thereby leaving the essential oil in the mixture.

ENFLEURAGE EXTRACTION METHOD

Table 2 weight of oil with respect to time

Weight(g)	Time (min)
0.31	240
0.41	480

0.55	720
0.58	960
0.70	1200

The essential oil produced by enfleurage method is 2.55g weight of essential oil per 130g of dry lemongrass sample thereby producing 1.96% oil yield at 780C.

HYDRO DISTILLATION METHOD

Table 3 weight of oil yield with respect to time

Weight (g)	Time(min)
0.10	240
0.14	480
0.26	720
0.35	960
0.38	1200

The result of hydro distillation process was 1.23g per 130g of lemongrass sample giving 0.95% yield of oil.

Physical and Chemical Properties of lemongrass oil

The essential oil produced was pale yellow, with an aromatic camphoraceous odour, pungent and cooling taste. Because of its high volatility, it was stored in an air-tight container protected from light in cool place. The essential oil is insoluble in water, miscible in alcohol and in oil.

Result of Essential oil Extraction

Method of extraction	% yeild
Solvent Extraction	2.08
Enfleurage	1.96
Hydro distillation	0.95

From the experiment carried out it was observed that the best method used in extraction is solvent extraction method because it yielded more oil than any other method. This conforms to works done by other researchers.

Enfleurage method, yielded less oil when compared to the solvent extraction this could be because most volatile content gets lost during the pounding process while hydro distillation gave low yield. This could be because the extraction of the essential oil was not always complete due to variable rate of distillation cause by heat.

GENERAL OBSERVATION ON THE PREFUME PRODUCED

- 1.The quantity of essential oil has the highest yield in solvent extraction.
- 2.The mixture is pale yellow.
- 3.Has a clear lemongrass fragrance.
- 4.It is volatile and has a cooling effect in the skin.

CONCLUSION

Solvent extraction, enfleurage and hydro distillation methods are effective and efficient means of extracting essential oils. Solvent extraction is the most common and most economically technique for extracting oil in modern perfume industry because of its simplicity. Extraction by enfleurage was commonly used when distillation was not possible because some fragrant compounds denature through high heat. This technique is not commonly used in modern industries because of its primitive cost. The essential oil extracted by hydro distillation has strong odor characteristics of the raw material from which they were produced. When compared with other methods of extraction. It is cheaper because the cost involved is that of energy used in heating water to generate steam. Water itself is the commonest material easily available from nature supply or other alternative sources. There is high demand for essential oils for various purposes such as medicinal, perfumery, soap making, insecticides to mention but a few. Imported essential oils are very expensive to meet the demand of our local consumer industries, therefore it becomes necessary to source and synthesis these oils from local sources, in particular lemon grass. With essential oils made from lemon grass, perfume can be produced locally using different methods of extraction, thereby creating employment.

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