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FACILE METHODS FOR THE EXTRACTION OF ESSENTIAL OIL FROM THE PLANT SPECIES - A REVIEW

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Essential oil, Seeds, flowers, fruits, Rhizomes, and leaves, etc., of Plants, Different Extracting Techniques, Merits and Demerits

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ABSTRACT: Essential oils are a diverse group of natural products. Oils are the important source of aromatic and flavoring chemicals in food, industrial, and pharmaceutical products. Essential oil is a concentrated hydrophobic liquid containing volatile aroma compounds derived from the different parts of the plants. Various extraction methods are used in the manufacture and extraction of essential oils from the plant materials. Essential oils are produced using several techniques like water distillation, steam distillation, hydro diffusion, maceration, solvent-free microwave extraction, solar distillation and CO_2 supercritical fluid extraction, etc. These extraction and expression methods are used to remove the oils from the dried/fresh plants (or) machines to squeeze the oil out of the plants. Out of all the extraction methods, supercritical carbon dioxide method is more efficient. The present paper is an attempt to list out the various methods of extraction of essential oil from the different parts of the plants and their merits and demerits.

INTRODUCTION: Essential oils are the sweet-smelling hydrocarbons present in many medicinal plant systems [1]. A variety of plants yield essential oils are used as perfumes, food flavoring agents, medicines, and as a fragrant and antiseptic additives, etc. Essential oils have been used for thousands of years for body massage in siddha and homeopathy medicines [2]. The aroma oils interrelate with the human body by four distinct modes of action - pharmacological, physiological, psychological and spiritual [3, 4]. In ancient era of mesopotamia, more than 5,000 years ago, had mechanical machines for obtaining essential oils from medicinal plants.

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In nature, essential oils play an important role in the protection of the plants as antibacterial, antiviral, antifungal and insecticides activities [5, 6]. Essential oils are extracted from various aromatic plants generally localized in temperate to warm countries like Mediterranean and tropical countries where they represent an important part of the traditional pharmacopoeia. They are liquid, volatile, and rarely colored, lipid soluble and soluble in organic solvents, etc. Essential oils are the main source of perfumes for the ancient human-beings of Egypt, India, Greece, and Rome, etc. Essential oils may be found in roots, flowers, leaves, fruit, seeds or bark of the plant [7]. Growing and harvesting conditions are optimized for the production of the best fragrances.

There are several methods for extracting the essential oils from the natural sources. These may include use of liquid carbon dioxide (or) microwaves, and mainly low (or) high pressure distillation employing boiling water (or) hot steam. Due to their bactericidal and fungicidal properties, pharmaceutical and food uses are more and more wide-spread as alternatives to synthetic chemical products to protect the ecological equilibrium. Owing to the new attraction for natural products like essential oils, despite their wide use and being familiar to us as fragrances, it is important to develop new, cheap and facile methods for essential oil extraction. The review article mainly focused on the different extracting methods for essential oils from the seeds and nuts of medicinal plants.

Different Extraction methods:

Distillation: Distillation is the extracting oil process, converts volatile liquid (essential oils) into vapor state and then condenses the vapor into a liquid state. The extracting method is cost to produce essential oils. Under this distillation process; three category

(i) Water Distillation

(ii) Steam Distillation

(iii)Hydro diffusion [8-23]

i. **Water Distillation**: In the case of water distillation process, the botanic material is completely immersed in water and the still is brought to the boil. It is used to protect the oils to certain degree, since the surrounding water acts as a barrier to prevent overheating. When the condensed material cools down, the water and essential oil gets separated and the oil is decanted to use as an essential oil. The water separated in the above mentioned process is used as a "floral waters" in the world fragrance market.

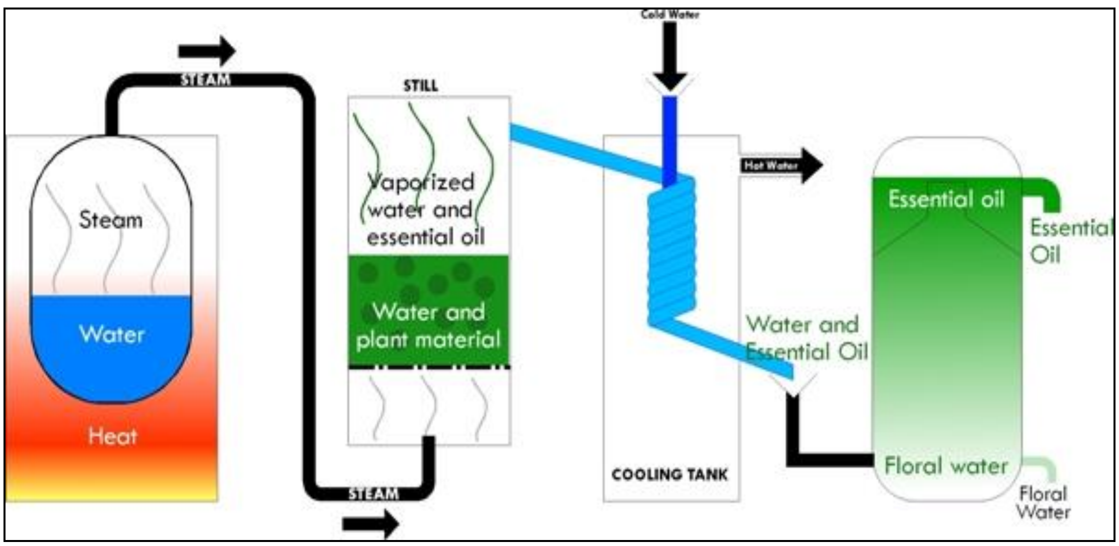


FIGURE 1: WATER DISTILLATION UNIT

Water distillation can be done at reduced pressure (under vacuum) to reduce the temperature to less than 100°C, which is useful in protecting the botanical material for obtaining the essential oils. In case of lavender, the plant material should not be exposed to hot water, because it may lead to decompositions. The plant material containing high amounts of esters cannot be extracted with water distillation extraction method, since the extended exposure to hot water will start break down the esters into the resultanting alcohols and carboxylic acids.

Merits: It is simple and easy to operate and extracts oil from the plant species.

Demerits:

a. However, due to use of heat in this Method, it may not be used on very fragile plant material, because major therapeutic characteristics would be adversely affected, (or) where the Method is employed with great difficulty.

b. Oil components like esters are sensitive to hydrolysis while others like acyclic monoterpene hydrocarbons and aldehydes are susceptible to polymerization (since the pH of water is often reduced during distillation, hydrolytic reactions are facilitated).

c. Oxygenated components such as phenols have a tendency to dissolve in the still water, so their complete removal by distillation is not possible.

d. As water distillation tends to be a small operation (operated by one or two persons), it takes a long time to accumulate much oil, so good quality oil is often mixed with bad quality oil.

e. The distillation process is treated as an art by local distillers, who rarely try to optimize either oil yield (or) quality.

f. Water distillation is a slower process than either water and steam distillation (or) direct steam distillation.

Application: This method is used to extract the essential oil from the plant species like orchid flowers, Cassia flowers, etc.

ii. **Steam Distillation:** In steam distillation method [24-28], the botanical material is placed in a still and steam is forced over the material. The hot steam is used to release the aromatic molecules from the plant material. The steam forces to open the pockets and then the molecules of these volatile oils, escape from the plant material and evaporate into the steam. The steam contains the essential oil, is passed through a cooling system to condense the steam, which forms a liquid form of essential oil and then water is then separated. The steam is produced at greater pressure than the atmospheric pressure and therefore boils at above 100^{o}C which is used to the remove the essential oil from the plant material.

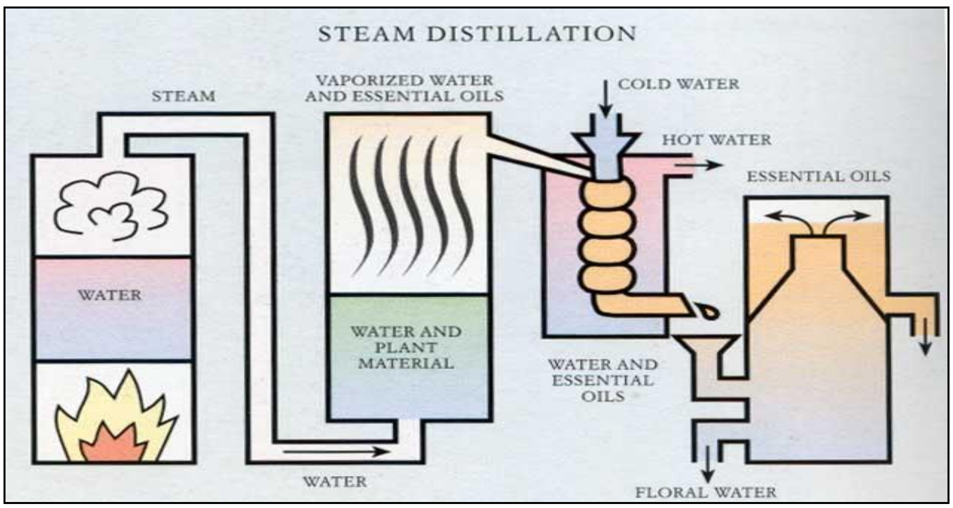


FIGURE 2 STEAM DISTILLATION UNIT

Merits: The major advantage of steam distillation is that the temperature never goes above 100C so temperature sensitive compounds can be distilled.

Demerits: The disadvantage is that not many compounds can be steam distilled - usually aromatic ones.

Application: This method is used to extract the essential oil from the plant *thymus vulgaris* has six different chemo-types main component like timol, carvacrol, linalool, geraniol, tuyanol-4, terpineol.

iii. **Hydro diffusion:** Hydro diffusion method [29] is similar like steam distillation process. The main difference between these two methods, steam is introduced into the still. In the case of hydro diffusion the steam is fed into the top onto the botanical material instead of from the bottom as in normal steam distillation. The steam contains the essential oil, is passed through a cooling system to condense the steam, which forms a liquid form of essential oil and then water is then separated. The condensation of the oil containing steam mixture occurs below the area in which the botanical material is held in place by a grill.

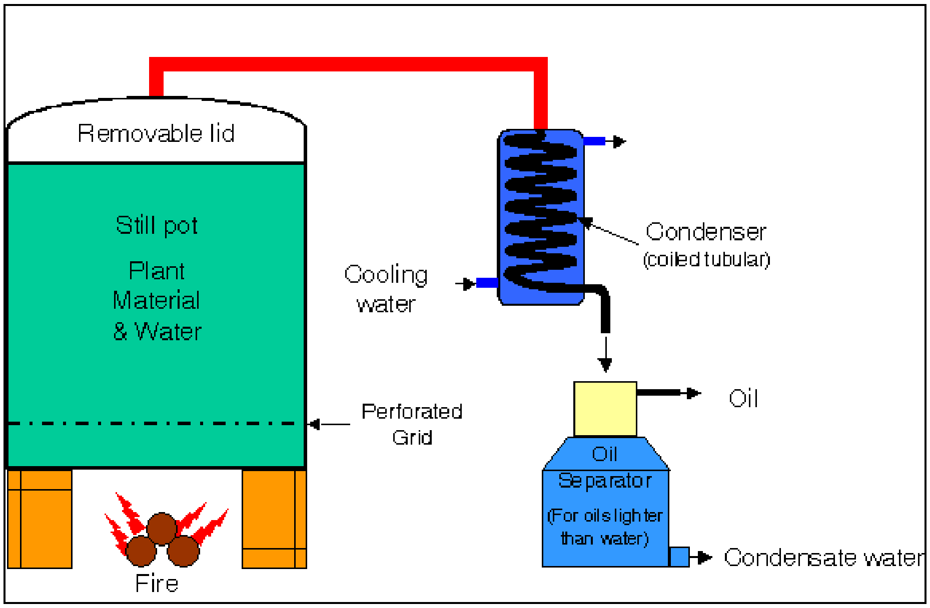


FIGURE 3: HYDRO DIFFUSION UNIT

Merits: The main advantage of this method is that less steam is used, shorter processing time and a higher oil yield.

Demerits: In this method the oil-water solution permeates, by osmosis, the swollen membranes and finally reaches the outer surface, where the oil is vaporized by passing steam.

Application: It is used to extract the essential oil from the Floral attars of flowers (such as saffron, marigold, rose, jasmine, pandanus) in sandal wood.

Expression methods:

Sponge extraction process: Most of the citrus essences are extracted using expression methods. The fruit pulp was removed and then the rind and pith were soaked in warm water to make the rind more pliable, since the pith of the fruit absorbed the water.

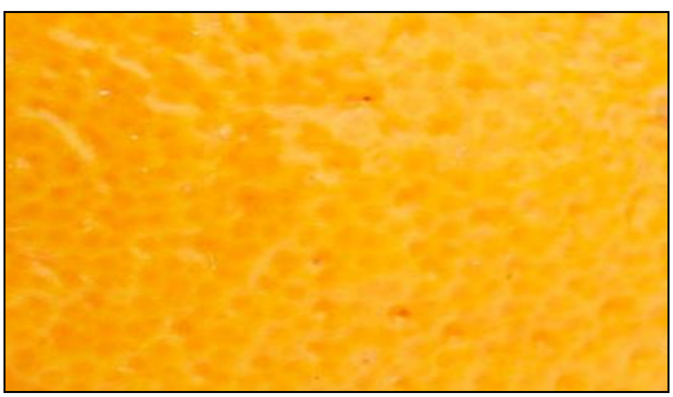


FIGURE 4 SPONGE EXTRACTION PROCESS

After the fruit has absorbed the water and become more elastic. It was inverted which will help to rupture the oil cells and then the sponge placed next to the rind.

It was then squeezed to release the volatile oil, which is collected directly into the sponge material. In the final stage, the sponge became saturated with oil, was squeezed and the essential oil collected is directly collected from the reaction vessel.

Application: In this method is particularly used for the separation of essential oil from the citrus plant species.

***Ecuelle a piquer* Method:** It is one of the types of expression extraction methods, which is mainly used, in case of citrus species to obtain essential oils. This is more modern way of essential oil extraction referred as *ecuelle e piquer* process.

The fruit is placed in a device and rotated with spikes on the side puncturing the oil cells in the skin of the fruit. This cause the oil cells to rupture and the essential oil, and other material such as pigment, to run down to the center of the device, which contains plant residues.

The liquid is separated and the essential oils are removed from the water-based parts of the mixture.

FIGURE 5: *ECUELLE A PIQUER* METHOD

Machine abrasion: Machine abrasion is similar like ecuelle a piquer method. It is mostly used in the manufacture of citrus fruit essential oils. In the case of machine abrasion, machine strips off the outer peel, which is then removed by running water and then fed into a centrifugal separator. Using the centrifugal separation, the plant residues and the essential oils gets separated completely in a different layer.

Solvent extraction:

1. **Maceration:** The flower (or) petal part of the plant materials are slightly ruptured and placed in warm fat. The process is repeated for several times until the fat becomes saturated with essential oil and then bathed in alcohol, which evaporates leaving essential oil.

Application: Certain plant materials require maceration in warm water before they release their essential oils, as their volatile components are glycosidically bound. For example, leaves of wintergreen (*Gaultheria procumbens*) contain the precursor gaultherin and the enzyme primeverosidase; when the leaves are macerated in warm water, the enzyme acts on the gaultherin and liberate free methyl salicylate and primeverose. Other similar examples include brown mustard (sinigrin), bitter almonds (amygdalin) and garlic (alliin).

2. **Solvent-free microwave extraction** [30-32]**:** Solvent-free microwave extraction is used to separate the essential oil from the plant material. The method involves placing the sample in the microwave reactor, without any addition of the organic solvent (or) water. The internal heating of the water within the sample distends its cells and leads to rupture of the glands and oleiferous receptacles. This process thus frees essential oil, which is evaporated by the *in-situ* water of the plant material. A cooling system outside the microwave oven continuously condenses the vapors which are collected on specific glassware. The excess of water is refluxed back to the extraction vessel in order to restore the *in-situ* water to the sample.

FIGURE 5 MICROWAVE EXTRACTION

Merits: The microwave isolation offers net advantages in terms of yield and selectivity, with better isolation time, essential oil composition, and is environmental friendly.

Demerits: In this method the low boiling point hydrocarbon compounds may undergoes decomposition.

Application: Solvent-Free Microwave Extraction (SFME) technology for rapid extraction of essential oils from aromatic herbs, spices and dry seeds.

3. **Solar distillation** [33-35]: It is a new type of distillation method to extract the essential oils from the plant material. Scheffler fixed focus concentrator was used for solar distillation system. The system comprises of a primary reflector, secondary reflector, stainless steel boiler and condenser unit and florentine flasks as shown in **figure 6**.

FIGURE 6: SOLAR DISTILLATION PROCESSES

The primary reflector rotates along with an axis parallel to the earth axis of rotation by a precise photovoltaic tracking mechanism. It keeps the reflected beam aligned with the fixed secondary reflector as the sun moves. The secondary reflector further reflects the beam radiation to targeted distillation bottom for the steam distillation. The system is equipped with thermocouples and Pyranometer to control and optimize the distillation processes. Different glass Florentine flasks were used to separate the oil from water.

In order to assess the continuous performance of the Scheffler concentrator during distillation experiments, three connections of K-type and T-type thermocouples were used to record receiver inside the reaction vessel temperatures, water temperature and steam temperature of the distillation unit. All the three connections were attached to computer via data recording machine. The intensity of beam radiations was recorded with the help of Pyranometer.

Merits and Demerits: The solar distillation process for essential oil extraction utilized approximately same heat energy per unit weight of the herb. The results show that completes distillation process for essential oils extraction can be done successfully using Scheffler fixed focus concentrator.

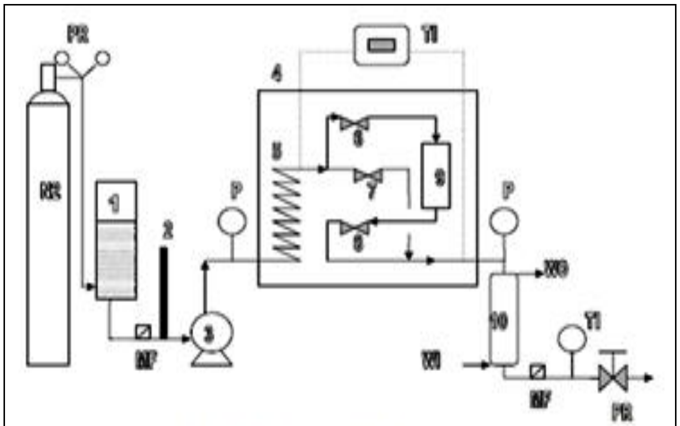
Application: Different herbs like Melissa, Peppermint, Lavender, Fennel seeds, Cumin, Basil, Cloves buds and rosemary etc were processed successfully and extracted essential oil by using solar distillation system.

4. **Subcritical water extraction** [36-38]: The subcritical water extractions were carried out in a laboratory-built apparatus shown in **Figure 7**. The de-ionized water filled into a 5 L stainless steel feed tank was first purged for 2 h with N_2 to remove dissolved O_2. A Dosapro Milton Roy (H_9 series obtained from USA) high pressure metering pump was used to deliver the water through the system at a constant flow rate of 1, 2 and 4 ml/min.

   The pump output could be adjusted by stroke knob at the required flow rates and be checked using a burette equipped in the inlet pipelines. A coil made from 3m length stainless steel tubing (3mm I.D · 6.35 O.D.) was used for preheating the water. The extractor consisted of a stainless steel cylindrical extraction chamber (103mm · 16mm I.D). The solid bed inside the extractor was fixed with ring screws at both ends in order to permit the circulation of the water through it. Input and output of the water was carried out through two side-connected quick-open high pressure valves. The main body of the extractor was closed with screw caps at both ends.

The flow direction was top to bottom. After the preheating coil, a three way line was made by using three 1/4 inch (6.35 mm) high-pressure heat-resistant needle valves. The needle valves 6 and 8 were inserted on the inlet line to the extractor and outlet line from it, respectively. The needle valve 7 was used as by-passing line. In this manner, the water flow stream could be selected either to the extractor or by-passing it. The preheating coil, the extractor and the needle valves were placed in a fanequipped temperature-controlled oven (Teb Azma Co., Tehran, Iran), designed to work at up to 200^oC.

In order to avoid heat losses of essential oils, a double pipe heat exchanger (tube side: 10.20 mm I.D · 13.22mm O.D., cooling surface area: 240 cm^2) cooled with water with about 15^oC and 3ml/min flow rate was used to cool the extract coming out from the oven to a temperature close to 20^oC. A 1 m length stainless steel tube (1 mm I.D· 3.2 mm O.D) was applied before a 1/8 inch (3.18mm) pressure regulator (Hoke Co., USA). In this manner, maintaining the desired pressure in the system was performed precisely. The outlet was inserted in a collection vial. All parts which were in contact with the extractant water made from stainless steel 316. Two 140 lm micro-filter (SS316, Nupro Co., USA) were used to protect the high pressure pump and pressure regulator, respectively. After the pump, a safety valve (50bar, SS316) was used to control the maximum allowable pressure in the system. The experimental setup of Subcritical water extraction process as shown in **figure 7**.



Schematic diagram of superheated water extraction system: 1, water reservoir; 2, burette; 3, pump; 4, oven; 5, preheater; 6, inlet water; 7, bypass stream; 8, outlet water; 9, extraction cell; 10, heat exchanger; MF, micro filter; P, pressure indicator; PR, pressure regulator; TI, temperature indicator; WI, cooling water in; WO, cooling water out.

FIGURE 7: SUBCRITICAL WATER EXTRACTION

5. **Supercritical fluid carbon dioxide extraction method:** The extraction of plant essential oil was conducted in an own built dynamic extraction apparatus, which mainly consists of a high pressure extraction vessel and a separator, as shown in **Figure 8**. The extraction vessel was 19mm in inner diameter and 360mm in length, giving a total of 100 mL in volume, while the separator with a volume of 25mL had a flat bottom and was connected with a back pressure regulator. In a typical run, 5grams of ground medicinal plant material were placed between two layers of stainless steel balls (dp-2.5mm) in a high-pressure stainless steel extraction vessel to provide particle voidage of 0.3.

The extraction vessel was immersed in a water bath controlled by an electrical heater (Thermo Haake, model DC_{10}, Germany) to within ± 0.1 K. When the water bath temperature reached the desired temperature, carbon dioxide was continuously delivered at a volumetric flow rate of 1 mL/min into the system by an HPLC pump (Jasco, model PU-980, Japan) until the system pressure was achieved. The system pressure was determined by a pressure transducer (Druck, model PDCR961, Switzerland).

The CO_2 was depressurized by a back pressure regulator (Jasco, model 880-81, Japan) and the amount of CO_2 consumed was determined using a wet gas meter (Alaxander Wright & Co., model DM_3A, USA). The essential oil extracts were collected at one hour intervals and the extraction process was terminated when it was visually observed that no plant material oil was being collected in the vial and has reached what is termed exhaustive extraction.

The oil collected was gravimetrically determined using a balance (Mettler Toledo, model AG204, USA) with an accuracy of ± 0.0001 g.

Effect of pressures, temperatures of the extraction process, and particle sizes were investigated in this study. The schematic diagram of CO_2 Supercritical fluid extraction is shown in **figure 8**.

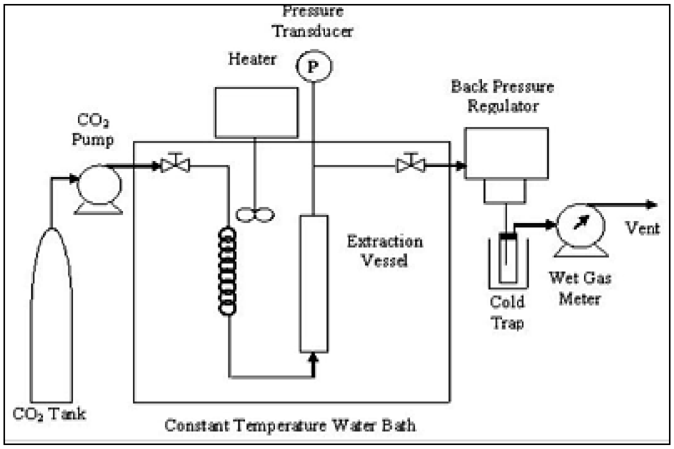


FIGURE 8: SUPERCRITICAL FLUID CARBON DIOXIDE

CONCLUSION: The final conclusions were drawn based on the complete review on extraction of essential oil from the plant species using different extraction methods (Conventional and Non-conventional methods).

Comparison of all the above mentioned extraction methods, carbon dioxide super critical fluid extraction method is more enhanced rather than other. The main advantage of the CO_2 SCFE method is;

A. CO_2 is non-hazardous

B. Less expensive

C. Bulk Quantification

D. No Organic Solvent usage

E. Environmental friendly.

But in the case of other methods;

A. Highly expensive

B. Cost-effective

C. Usage of organic solvents

D. Non eco-friendly.

The plant materials produce essential oils from the different part of the plant material which are extracted by several processes to make perfumes (or) essences for the applications of various industries. All the fragrances are well accepted for various purposes in our daily routine for making natural harmony with nature.

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