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TECHNIQUES FOR EXTRACTION OF ESSENTIAL OIL FROM ORANGE PEELS: A REVIEW

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Abstract: <u>Background:</u> In recent years, variety of Extraction techniques has been introduced for the recovery of organic compounds. Pure essential oils are derived from various part of the plants and fruits. These essential oils have a very high commercial value due to its properties. They are widely used in the various fields of industries, such as perfumery industries and pharmaceuticals. The essential oils for are primarily used in the perfumery industry and have a very high commercial value due to its therapeutic properties.

Objective: To study the techniques available to extract the essential oils from Orange peels and study the efficiency and the selectivity for this techniques.

Results: The essential oils of the plant are composed of heat-sensitive chemical compounds, the use of steam distillation and Microwave-assisted extraction (MAE) techniques would inevitably inflict thermal degradation to the natural fragrance. In this experimental work, solvent extraction method was employed due to its mild extracting condition and lower operating cost. Ethanol was used as the solvent due to its high availability in the market. The extract compositions were compared using UV analysis.

Conclusion: Extraction of essential oils out by different techniques and Innovative methods avoid shortcomings of content optional techniques friendly to environmental to avoid chemical risk, extraction time, consumption the solvents and obtain yield quality of essential oils.

Index Terms - Essential oil; Extraction method; Conventional methods; Non-Traditional extraction

I. INTRODUCTION

Essential oils are plants and fruits based volatile oils with strong aromatic components that are made up of different chemical compounds. For example, alcohols, hydrocarbons, phenols, aldehydes, esters and ketones are some of the major components of essential oil (Younis *et al.*, 2008). Oils from hundreds of plant species are available commercially (Formaceck and Kubeczka, 1982). Numerous studies have demonstrated the efficiency of Essential Oils in low doses in the fight against bacterial pathogens (Oussalah *et al.*, 2006, 2007) even against multi-resistant bacteria (Mayaud *et al.*, 2008; Burt *et al.*, 2004). The effectiveness of these procedures has beenattributed mainly to the presence of active phytochemicals or bioactive compounds in plants. Given the scope of searching new antimicrobial agents (Seoussen *et al.*, 2016), antimicrobials derived from peels materials are often regarded as natural and safe compared to industrial chemicals. Of late, plant-based medicine has becomemore popular due to the increasing concern of consumers with regard to the use of synthetic chemical preparations and use of artificial antimicrobial preservatives, especially in modern food protection practices. The worldwide market for essential oil growth rapidly and nowadays a lot of scientific research presently focused on the industrial development together with environmental preservation by used different techniques as Hydrodistillation (HD), Supercritical Fluid Extraction (SFE), Microwave-Assisted Hydrodistillation (MAHD) and Ultrasound-assisted extraction (UAE).

1. Essential Oils:

Essential oils are concentrated volatile aromatic compounds produced by plants - the easily evaporated essences that give plants their wonderful scents. Each of these complex precious liquids is extracted from a particular species of plant life. Each plant species originates in certain regions of the world, with particular environmental conditions and neighboring fauna and flora. Essential oils are frequently referred to as the "life force" of plants and fruits. Unlike fatty oils, these "essential" oils are volatile, highly concentrated, substances extracted from flowers, leaves, stems, roots, seeds, bark, resin or fruit peels. The amount of essential oils found in these plants can be anywhere from 0.01 percent to 10 percent of the total. That's why tons of plant and fruits material are required for just a few hundred pounds of oil. These oils have potent antimicrobial factors, having wide range of therapeutic constituents. 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. Beware of imitations. Essential oils cannot be substituted with

synthetics. Only pure oils contain a full spectrum of compounds that cheap imitations simply cannot duplicate. Essential oils are usually colorless, particularly when fresh. Nevertheless, with age essential oil may oxidize which resulting the color becomes darker. Therefore, essential oil needs to be stored in a cool, dry place tightly stoppered and preferably full in amber glass

Essential oil is used in perfumery, aromatherapy, cosmetics, incense, medicine, household cleaning products and for flavoring food and drink. They are valuable commodities in the fragrance and food industries. More than 250 types of essential oils. A number of countries produce different kinds of essential oils. India ranks second in the world trade of essential oils (Rao et al., 2005). Essential oils are derived from various sections of plants. An essential oil is usually separated from the aqueous phase by a physical method that does not lead to significant change in its chemical composition (Figure 1). Essential oils could be then subjected to an appropriate further treatment. Essential oils are oily aromatic liquids extracted from aromatic plant materials. They could be biosynthesized in different plant organs as secondary metabolites (A. El Asbhanni et al., 2015).

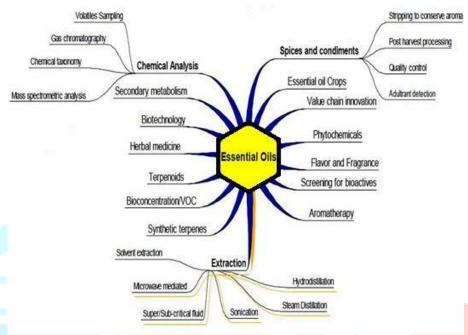


Fig. 1: Show the copious branching of specializations in the work of consequential oils.

2. How Essential Oil Works in Aromatherapy?

An Essential Oil is inhaled and directly by the olfactory system to the limbic System of the Brain. In true, the brain responds to the particular scent affecting our emotions and chemical balance. Essential Oils also absorbed by the skin and carried throughout the body via the circulatory system to reach all internal organs. By carefully choosing one or more oils, you can experience benefical effects promoting overall health - and even specific targets. Benefits depend upon the unique nature of each person's response to an aromatic stimulus.

3. Pharmacological Properties of Essential Oils

- œ Antiseptics: Essential oils have antiseptic properties and are active against a wide range of bacteria as well as on antibioresistant strains. Moreover, they are also known to be active against fungi and yeasts (Candida). The most common sources of essential oils used as antiseptics are: Cinnamon, Thyme; Clover; Eucalyptus; Culin savory; Lavender. Citral, geraniol, linalool and thymol are much more potent than phenol.
- œ Expectorants and diuretics: When used externally, essential oils like (L'essence de terebenthine) increase microcirculation and provide a slight local anaesthetic action. Till now, essential oils are used in a number of ointments, cream and gels, whereby they are known to be very effective in relieving sprains and other articular pains. Oral administration of essential oils like eucalyptus or pin oils, stimulate ciliated epithelial cells to secrete mucus. On the renal system, these are known to increase vasodilation and in consequence bring about a diuretic effect.
- œ Spasmolytic and sedative: Essential oils from the Umbellifereae family, Mentha species and verbena are reputed to decrease or eliminate gastrointestinal spasms. These essential oils increase secretion of gastric juices. In other cases, they are known to be effective against insomnia.
 - œ Others: Cholagogue; anti-inflammatory; cicatrizing

Chemical constituents of Essential Oils:

They are complex mixtures of volatile compounds such as terpenes (mostly monoterpenes and sesquiterpenes), phenolics and alcohols (Lucchesi et al., 2004). However, the essential oils are highly complexand may include oxygenated compounds. The essential oil is so called because they were believed to represent the quintessence of odor and flavor from the flower kingdom - differ in composition properties from fatty or fixed oils, which consist for the most part of glycerides and from mineral or hydrocarbon oils. A scientific definition of the term essential or volatile oils is not possible, although several practical definitions exist. The most common one defines an essential oil as a more or less volatile material isolated from an odorous plant of a single botanical species by a physical process (see Table 1).

They are oxygenated derivatives of hydrocarbon terpenes such as aldehydes, ketones, alcohols, phenols, acids, ethers and esters (Bakkali et al., 2008). Some terpenes are potent drugs against diseases such as heart disease (Liebgott et al., 2000), malaria (Parshikov and Netrusov, 2012) and cancer (Ebada et al., 2010).

Table 1: Some compounds of essential oils with physical properties and biological applications.

EO	Molecular structure	Chemical	Molecular	Boiling point	Refractive index	Plant	Some biological	References
components	Wiolecular structure	Formula	Weight	C_0	$(20 \mathrm{C}^0)$	source	application	references
Ketones alcohols Camphor	X	C10H16O	152.23	204	-	Lavendula stoechas	Antispasmodic, sedative, diuretic antirheumatic, anti- inflammatory, anti-anxiety	(Braden et al., 2009)
Monoterpenes D-Limonène		С10Н16	136.23	175.4	1.473	Citrus limon	Antifungal, Antioxidant	(Singh et al., 2010)
g-Terpinène		C10H16	136.23	183	1.474	Origanum vulgare	Antioxidant	(Ruben Olmedo, 2014)
Terpenic oxides 1,8-Cineole	H ₃ C CH ₃	С10Н18О	154.25	176	1.457	Eucalyptus poly bractea	Antiinflammatory activity (asthma)	(Juergens et al., 2003)
Oxygenated sesquiterpenes a-Bisabolol	□ OH	C ₁₅ H ₂₆ O	222.37	153	1.496	Matricaria recutita	Anti-irritant, anti inflammatory, antimicrobial	(Joseph M. Mwaniki et al., 2015)
Terpenic oxides Cis-Rose oxide	CH ₃	C ₁₀ H ₁₈ O	154.25	70–71	1.454	Rosa damascena	Antiinflammator y, Relaxant	(Nonato et al., 2012; Boskabad y et al., 2006)
Cinnamaldehyde		С9Н8О	132.16	248– 250	1.621	Cinnamom um, Zeylanicu m	Bactericide, fungicide, insecticide	(Ye et al., 2013)

5. Essential oils extraction methods:

Essential oils are used in a wide variety of consumer goods such as detergents, soaps, toilet products, cosmetics, pharmaceuticals, perfumes, confectionery food products, soft drinks, distilled alcoholic beverages (hard drinks) and insecticides. The world production and consumption of essential oils and perfumes are increasing very fast. Production technology is an essential element to improve the overall yield and quality of essential oil. Essential oils are obtained from plant raw material by several extraction methods (Wang and Weller, 2006)(Dick and Starmans, 1996).

5.1 Classical and Conventional Methods:

There are several by the numbers methods of extraction behavior of essential oils. The timid technologies about essential oils processing are of abundant significance and are still overused in copious parts of the globe. Hydrodistillation (HD), Steam distillation (SD), Solvent extraction, are the roughly traditional and generally used methods.

5.1.1 Hydrodistillation (HD):

Hydrodistillation is a traditional method for removal of essential oils. Water or hydrodistillation is one ofthe oldest and easiest methods (Meyer-Warnod et al., 1984). Being used for the extraction of essential oils. Hydrodistillation normally used to isolation essential oils from the aromatic and medicinal plant. The conventional method for the extraction of essential oils is hydrodistillation (HD), in which the essential oils are evaporated by heating a mixture of water or other solvent and plant materials followed by the liquefaction of the vapors in a condenser. The setup comprises also a condenser and a decanter to collect the condensate and to separate essential oils from water, respectively (Figure 2). The principle of extraction is based on the isotropic distillation. In fact, at atmospheric pressure and during extraction process (heating), water or other solvent and oils molecules. Hydro-distillation (HD) is a variant of steam distillation, which is bespoke by the French Pharmacopoeia for the extraction of Essential oils from dried plants and the quality control of essential oils in the lab. There are three types of hydrodistillation: with water immersion, with direct vapor injection and with water immersion and vapor injection. It is a multilateral process that can be utilized for large or small industries. The distillation time depends on the plant material being processed. Prolonged distillation produces only a small amount of essential oil, but does add unwanted high boiling point compounds and oxidation products.

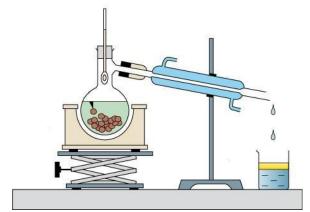


Fig. 2: The schematic subsidize apparatus for hydrodistillation.

5.1.2 Steam Distillation:

Steam distillation is a type of distillation (a separation or extraction process) for a temperature-sensitive plant such as natural aromatic compounds. It once was a popular laboratory method for purification of organic compounds but has become obsolete by vacuity distillation. Steam distillation still important in certain industrial sectors (Fahlbusch et al., 2003). Steam distillation is one of ancient and official approved methods for isolation of essential oils from plant materials. The plant materials charged in the alembic are subjected to the steam without maceration in water. The injected steam passes through the plants from the base of the alembic to thetop. Steam distillation is a method where steam flows through the material as shown in Figure 3. This steam functions as agents that break up the pores of the raw material and release the essential oil from it. The system yields a mixture of a vapor and desired essential oil. This vapor is then condensed further and the essential oil is collected (Rai R. and Suresh B., 2004). The principle of this technique is that the combined vapor pressure equals the ambient pressure at about 100 °C so that the volatile components with the boiling points ranging from 150 to 300 °C can be evaporated at a temperature close to that of water. Furthermore, this technique can be also carried out under pressure depending on the essential oils extraction difficulty.

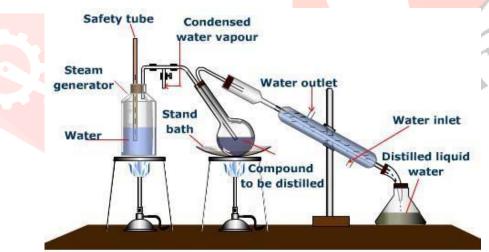


Fig. 3: The schematic subsidize apparatus for Steam distillation

5.1.3 Solvent extraction:

Solvent extraction, also known as Liquid-liquid extraction or partitioning, is a method to separatea compound based on the solubility of its parts. This is done using two liquids that don't mix, for example, water and an organic solvent (Figure 4). In the Solvent-Extraction method of Essential Oils recovery, an extracting unit is loaded with perforated trays of essential oil plant material and repeatedly washed with the solvent. Solvent extraction is used in the processing of perfumes, vegetable oil, or biodiesel. Solvent extractionis used on delicate plants to produce higher amounts of essential oils at a lower cost (Chrissie et al., 1996). The most frequently applied sample preparation procedure in plant material analysis. The quality and quantity of extracted mixture are determined by the type of extra heat applied because of the method is limited by the compound solubility in the specific solvent used. Although the method is relatively simple and quite efficient, it suffers from such disadvantages as long extraction time, relatively high solvent consumption and often unsatisfactory reproducibility (Dawidowicz et al., 2008).

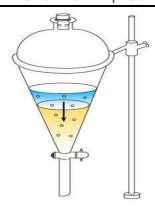


Fig. 4: Solvent Extraction

5.1.4 Soxhlet Extraction:

A Soxhlet extractor is a piece of laboratory apparatus (Harwood, Laurence M. and Moody, Christopher J, 1989) invented in 1879 by Franz von Soxhlet. (Soxhlet et al., 1879) It was originally designed for the extraction of a lipid from a solid material (Figure 5). Typically, a Soxhlet extraction is used when the desired compound has a limited solubility in a solvent, and the impurity is insoluble in that solvent. It allows for unmonitored and unmanaged operation while efficiently recycling a small amount of solvent to dissolve a larger amount of material. Soxhlet extraction involves solid-liquid contact for the removal of one or several compounds from a solid by dissolution into a refluxing liquid phase. In a conventional soxhlet device, the solid matrix is placed in acavity that is gradually filled with the extracting liquid phase by condensation of vapors from a distillation flask. When the liquid reaches a preset level, a siphon pulls the contents of the cavity back into the distillation flask, thus carrying the extracted analytes into the bulk liquid (Schantz et al., 1998). This procedure is repeated until virtually complete extraction is achieved. There are several advantages of Soxhlet extraction. The most important are that the sample is repeatedly brought into contact with fresh portions of the solvent. This procedure prevents the possibility of the solvent becoming saturated with extractable material and enhances the removal of the analyte from the matrix. Moreover, the temperature of the system is close to the boiling point of the solvent. This excess energy in the form of heat helps to increase the extraction kinetics of the system. Soxhlet extraction has several disadvantages, including it requires several hours or days to perform; the sample is diluted in large volumes of solvent, and due to the heating of the distillation flask losses due to thermal degradation and volatilization have been observed.

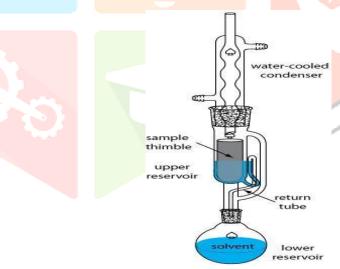


Fig. 5: Soxhlet Extraction

5.1.5 Cold Pressing method:

The term cold pressed theoretically means that the oil is expeller-pressed at low temperatures and pressure..Cold pressed method is one of the best methods to extract essential oils. Cold pressing is used to extract the essential oils from citrus rinds such as orange, lemon, grapefruit and bergamot. This method involves the simple pressing of the rind at about 120 degrees F to extract the oil. The rinds are separated from the fruit, are ground or chopped and are then pressed. The result is a watery mixture of essential oil and liquid which will separate given time. Little, if any, alteration from the oil's original state occurs – these citrus oils retain their bright, fresh, uplifting aromas like that of smelling a wonderfully ripe fruit. It is important to note that oils extracted using this method have a relatively short shelf life, so make or purchase only what you will be using within the next six months.

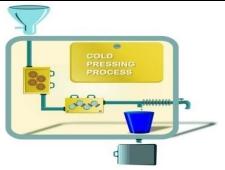


Fig. 6: Cold Pressing Method

5.2 Innovative Techniques Of Essential Oils Extraction (Non-Traditional):

One of the disadvantages of conventional techniques is related with the thermolability of Essential oils components which undergo chemical alterations (hydrolyse, isomerization, oxidation) due to the high applied temperatures. The quality of extracted Essential oils is therefore extremely damaged particularly if the extractiontime is long. It is important that extraction methods could maintain Essential oils chemical composition and natural proportion at its original state. Since economy, competitiveness, eco-friendly, sustainability, high efficiency and good quality become keywords of the modern industrial production, the development of essential oils extraction techniques has never been interrupted. Strictly speaking, conventional techniques are not the only way for the extraction of essential oils. Novel techniques, for example, abide by green extraction concept and principles have constantly emerged in recent years for obtaining natural extracts with a similar or better quality to that of official methods. New extraction techniques must also reduce extraction times, energy consumption, solvent use and CO2 emissions.

Traditional methods of extraction of essential oils have been discussed and these are the methods most widely used on a commercial scale. However, with technological advancement, new techniques have been developed which may not necessarily be widely used for commercial production of essential oils but are considered valuable in certain situations, such as the production of costly essential oils in a natural state without any alteration of their thermosensitive components or the extraction of essential oils for micro-analysis.

5.2.1 Supercritical Fluid Extraction (SFE):

Supercritical Fluid Extraction (SFE) is the process of separating one component (the extractant) from another (the matrix) using supercritical fluids as the extracting solvent. Extraction is usually from a solid matrix, but can also be from liquids. Supercritical fluids have been used as solvents for a wide variety of applications such as essential oil extraction and metal cation extraction (Figure 7). In practice, more than 90% of all analytical supercritical fluid extraction (SFE) is performed with carbon dioxide (CO2) for several practice reasons. Apart from having relatively low critical pressure (74 bars) and temperature (32Co), CO2 is relatively non-toxic, nonflammable, noncorrosive, safe, available in high purity at relatively low cost and is easily removed from the extract (Rozzi et al., 2002). The main drawback of CO2 is its lack of polarity for the extraction of polar analytes (Pourmortazavi et al., 2007). These essential oils can include limonene and other straight solvents. Carbon dioxide (CO2) is the most used supercritical fluid, sometimes modified by co-solvents such as ethanol or methanol. It was found that extracts prepared by SFE yielded a higher antioxidant activity than extract prepared by other methods (Fadel et al., 1999). This extraction method produces higher yield, higher diffusion coefficient, and lower viscosity. Many essential oils that cannot be extracted by steam distillation can be obtainable with carbon dioxide extraction. Nevertheless, this technique is very expensive because of the price of this equipment for this process is very expensive and it is not easily handled. Supercritical extracts proved to be of superior quality, with better functional and biological activities (Capuzzo et al., 2013). Furthermore, some studies showed better antibacterial and antifungal properties for thesupercritical product.

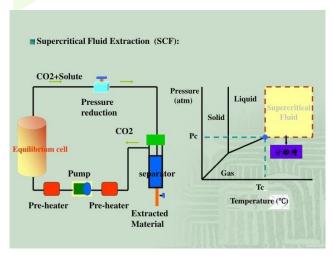


Fig. 7: Supercritical Fluid Extraction (SFE)

5.2.2 Microwave-Assisted Hydrodistillation (MAHD):

Microwave-assisted hydrodistillation is an advanced hydrodistillation technique utilizing a microwave oven in the extraction process. (Golmakani et al., 2008) reported some recently published studies have successfully utilized a microwave oven for the extraction of active components from plants. The efficiency of Microwave- assisted hydrodistillation is strongly dependent on the dielectric constant of water and the sample (Brachet et al., 2002).

Conventional techniques for the extraction of active constituents are time and solvent consuming, thermally unsafe and the analysis of numerous constituents in plant material is limited by the extraction step (Mandal et al., 2007). High and fast extraction performance ability with less solvent consumption and protection offered to thermolabile constituents are some of the attractive features of this new promising microwave-assisted hydrodistillation technique (Figure 8). Application of Microwave-assisted hydrodistillation in separation and extraction processes has shown to reduce both extraction time and volume of solvent required, minimizing environmental impact by emitting less CO2 in atmosphere (Lucchesi et al., 2004; Ferhat et al., 2006) and consuming only a fraction of the energy used in conventional extraction methods (Farhat et al., 2009). The use of Microwave-assisted hydrodistillation in industrial materials processing can provide a versatile tool to processmany types of materials under a wide range of conditions.

Microwave-assisted hydrodistillation is a current technology to extract biological materials and has been regarded as an important alternative in extraction techniques because of its advantages which mainly are a reduction of extraction time, solvents, selectivity, volumetric heating and controllable heating process.

The principle of heating using Microwave-assisted hydrodistillation is based upon its direct impact with polar materials/solvents and is governed by two phenomenon's: ionic conduction and dipole rotation, which in most cases occurs simultaneously (Letellier et al., 1999).

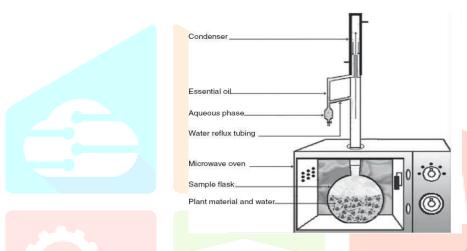


Fig. 8: Microwave-assisted hydrodistillation

5.2.3 Ultrasound-assisted extraction (UAE):

Ultrasound-assisted extraction (UAE) is a good process to achieve high valuable compounds and could Involved to the increase in the estimate of some food by-products when used as sources of natural compounds or plant material (Bhaskaracharya et al., 2009). The major importance will be a more effective extraction, so saving energy, and also the use of mean temperatures, which is beneficial for heat-sensitive combinations. This technique was developed in 1950 at laboratory apparatus (Vinatoru et al., 2001). Ultrasound allows selective and intensification of essential oils extraction by release from plant material when used in combination with other techniques for example solvent extraction and hydrodistillation(Figure 9). Ultrasound technology has been Featured as a valuable method in food engineering processes and plants (Bhaskaracharya et al., 2009), and become this field from the techniques active. In these applications the power ultrasound increases the surface wetness evaporation average and causes oscillating velocities at the interfaces, which may affect the diffusion boundary layer and generate rapid series of alternative expansions of the material, affecting cluster transfer (Garcı'a-Pe'rez et al., 2006).

The plants raw material is immersed in water or another solvent(Methanol or ethanol or anyone from the solvents) and at the same time, it is subjected to the work of ultrasound (Karim et al., 2012). This technique has been used for the extraction of many essential oils especially from the flower, leaves or seeds (Sereshti et al., 2012).

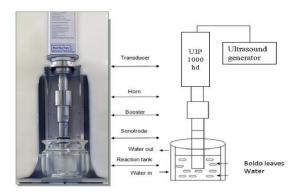


Fig. 9: Ultrasound-assisted extraction (UAE)

COMPARISONS OF EXTRACTION TECHNIQUES

For recovery of an organic compounds from sample of a solid matrices there are comparisons mentioned further for different extraction process.

In Soxhlet type of extraction, the extraction time needed is 6 to 24 hrs per sample for sample mass of about 10gm. Solvents (consumption-150-300ml) Acetone and Hexane, Acetone/DCM are used in 1:1 proportion (vol/vol). For solvents such as toluene and methanol the proportion used is 10:1 per sample (vol/vol). Extraction here is done by heating process. Basically this process is a consecutive process but for multiple assemblies the can be operated continuously. The time needed for development of this method is low, also equipment and operator skills required are very low.

Rather the Extraction time needed for Ultrasonic assisted Method or Sonication process is less comparatively to Soxhlet Extraction Method. For 2-30gm sample mass, extraction time needed is 3-5 min per sample. Here all the combinations of solvents used are of equal proportions. Solvents consumption are of less quantity, approx. 5-20ml. Extraction methods are proceeded with Ultrasound method. Ultrasound Method of Extraction can be performed for Batch and Continuous process both. Like Soxhlet Extraction Sonication method development time, operator skills and equipment cost is low.

The Supercritical Fluid Extraction uses Carbon dioxide as a solvent. For 1-10gm of sample mass to extract, the Extraction time is 30min to 1 hour per sample. Solvent consumed are 10-20ml and extraction method include heat and pressure. Only Batch Operations are performed in this process. Operator skill, cost of the equipment and the time needed to develop this method is high as compared to the above two methods.

For 2-10gm of sample mass, the extraction time needed for Microwave assisted Extraction 50min for up to 40 samples. Solvent consumption is about 25-45ml. Like Supercritical Fluid Extraction, methods of heat and pressure are equipped in this technique. Maximum 40 systems can be extracted simultaneously in this process. Time of development of this process is high as compared to other process. Rather the operator skills and instrument cost are moderate.

CONCLUSION:

Essential oils are natural products which consist of many volatile molecules. They have been used for several applications in pharmaceutical, cosmetic, agricultural and bioactivity example flowers. Extraction of essential oils could be carried out by various techniques. Have Innovative methods avoid shortcomings of content optional techniques to reduced chemical risk, extraction time and high energy input and obtain yield quality of essential oils. Despite their numerous application, except if essential oils are very sensitive to environmental factors used as such. Steam distillation is a special type of distillation or a separation process for temperature sensitive materials like oils, resins, hydrocarbons, etc. which are insoluble in water and may decompose at their boiling point. The temperature of the steam must be high enough to vaporize the oil present, yet not so high that it destroys the plants or burns the essential oils. The experiment as been carried out for the extraction of oil from Orange peels which have high essential oil content. Such Orange essential oil, which have been used as perfume and chemical raw materials for a long time, are now been studied as renewable sources of energy.

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