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Review Paper

Methods to extract Essential oils

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Abstract

Essential oils are low boiling oils, also known as volatile oils mainly obtained from plants and they possess a strong aroma. Due to their wide applications, they are attracting attention in recent times. The components present in them does not allow the use of extreme condition for their extraction from plant sources. This chapter covered the maximum possible extraction methods used by different researcher and industrial groups to extract the essential oils. The methods like solvent extraction, steam distillation, hydrodistillation, hydrodiffusion, solvent free microwave, and cold press methods have been discussed.

Keywords: Essential oils, volatile oils, extraction, isolation, solvent extraction, steam distillation.

Introduction

Essential oils are low boiling oils, also known as volatile oils that are obtained from plant species. They are odoriferous and aromatic oily liquids. These liquids constitute very small portion of total plant constitution. These oily liquids have been mainly extracted from barks, seeds, peels, leaves, buds, flowers, and other parts of the plants[1-3].

The aroma of essential oils is owing to the complex and variable composition having several hundred components, that mainly consists of hydrocarbons like terpenes and sesquiterpenes and different classes of oxygenated compounds (Figure-1)[2].

The quantity of extracted essential oils is region and plant specific. This varies from plant to plant and even from different parts of same plants.

Hence challenges are associated with the extraction and quantification of essential oils. The essential oils find applications in many day-to-day items and is in high demand (Figure-2)[4].

The anti-oxidative and anti-microbial properties possessed by essential oils make them a natural additive in foods and food products[5]. They are better alternatives of synthetic preservatives used to avoid rancidity of food and to increase the shelf-life of food products.

The use of essential oils makes the packaging materials 'active or smart packaging material'[6].

A market analysis estimated the demand of essential oils at 247.08 kilotons in 2020 with annual growth of about 7.5% which self explains its utility[7]. This manuscript discusses the classical methods of essential oil extraction.

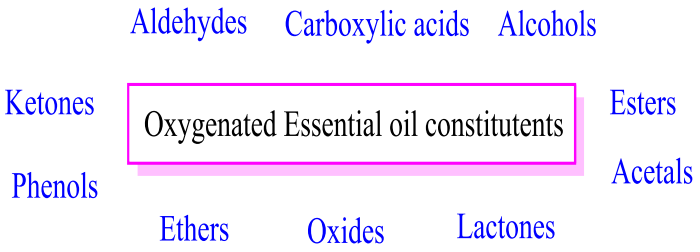

Figure-1: Different types of oxygenated compounds present in essential oils.

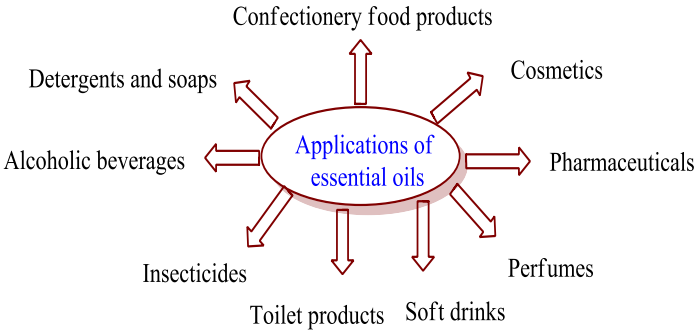

Figure-2: Different applications essential oils.

Extraction of Essential Oils

The quality and quantity of essential oils is entirely dependent upon the methods of extraction[8,9]. Apart from this, the form of botanical material used, the geographical area from where, the material has been collected also play important part in deciding the quality and quantity of essential oils. The different methods used to extract the essential oil include solvent extraction, steam distillation, hydrodistillation, hydrodiffusion, solvent free microwave and combination methods (solvent and steam both).

Solvent extraction: Solvent extraction is a solubility dependent process in which a particular compound is soluble in a particular solvent only. This method is also known as liquid-liquid extraction or partitioning method. This method is utilized to extract any compound from any sources based on the solubility[10]. In some occasions, the combination of solvents or two immiscible solvents can also be used. Some of the solvents that are commonly used for extraction of essential oils include petroleum ether, hexane, acetone, methanol, and ethanol etc[1]. This technique is used to extract fragile or delicate flower materials that are thermally labile (e.g., from blossom). Some of the plants like narcissus, jasmine, mimosa, carnation, etc have been subjected to solvent extraction to isolate essential oil from them[11].

In the solvent extraction method, the plant material is subjected into a solvent in that it has to be extracted. Then slight heat with stirring is given to extract the essential oil followed by filtration. After filtration, residue is discarded while filtrate is concentrated by the evaporation of the solvent to get either concrete or resinoid. Concrete or resinoid contains a mixture of wax, fragrance, and essential oil. Concrete is melted by warming and then mixed with pure ethanol to dissolve essential oil, some waxes, fixed oils, and fats. This further steam distilled under reduced pressure to get the aromatic substance from alcoholic solution. This aromatic substance is the true aroma of the plant and known by the name 'absolute'. This has its demand in the perfume industry.

Solvent extraction method of essential oil extraction is also associated with some disadvantages like long extraction period, high solvent requirement and unsatisfactory reproducibility[10] which makes the oils more expensive than other methods[11]. Since a lower temperature is required in solvent extraction in comparison to steam distillation, so it decreases the risk of chemical changes which may be possible in the presence of high temperatures.

Distillation: Steam distillation or water distillation is one of the earliest and most widely applied methods for the extraction of essential oil from plants[12]. This method is so much versatile that about 93% essential oils were extracted using steam or water distillation[13]. This method of extraction is suitable where the constituents of essential oil get decomposed at their boiling point[14].

In the distillation method for the extraction of essential oil, the plant material is subjected to steam supplied from steam generator without maceration in water. The injected steam moves through the plant material from which essential oil is to be extracted. The heated steam in combination with gentle pressure leads to the bursting of microscopic protective sacs. This gives essential oils or aromatic components[15,16]. As the mixture of steam and essential oils flows through the condenser, they are collected and separated where essential oil present in upper layer[17,18]. The hands-on positive outcome of water distillation are that the assembly (also known as still) is inexpensive, easy to construct. This method finds its suitability for field operation also.

The exposure of essential oils constituents to boiling water (steam) for longer period of time leads to change in the composition of the essential oil. For example, the esters can be hydrolysed to acids and alcohols during distillation. An innovative steam distillation extraction technique was developed by Masan go developed to increase the yields of isolated essential and reduce the amount of wastewater produced during the extraction process. In his system, he placed the plant sample above the steam source and allowed only steam to pass through the bed of plants. The plant materials are not directly mixed with boiling water, in this method. Thus, less steam was required resulting in reduction in the amount of water in the distillate[13].

Hydro-distillation (HD): Hydro-distillation (HD) is discovered by Avicenna, and considered as the oldest and easiest method for oil extraction from natural sources[19]. This is mainly applied to obtain the essential oil from flowers like saffron, marigold, rose, jasmine, pandanus or wood (like sandal wood). This method is suitable for the high-boiling natural product compounds. Rose was the first plant extract used and purified by this method. The principle behind HD is similar to the steam distillation (SD). In other words, the HD can be considered as the modified version of SD to obtain essential oils from dried plants. The water immersion and direct vapor injection are two important processes which either alone or in combination used in HD. Different types of plant materials requires different time to release the essential oils using the above-mentioned method. One of the limitations of HD is the presence of oxidation products and undesirable compounds in the essential oil if the HD process is prolonged[19].

Extraction with Supercritical fluids (SF): Conventional extraction methods (like steam distillation and solvent extraction) have some shortcomings such as long preparation time, low yield, loss of volatile compounds, long extraction times, low extraction efficiency, degradation of unsaturated compounds, toxic solvent residues in the extract[20-24], and excess amount of organic solvents used[25]. These shortcomings forced the natural product chemists to look for the alternative extraction techniques that can overcome these problems. Supercritical fluid extraction (SFE) is an answer to all these problems[26].

SFE has advantages over other conventional solvent extraction methods. This method mainly performed for thermally sensitive compounds because the process requires low temperature. At low temperature, even the chemical constituents of the essential oil remain intact. The high diffusivity of supercritical fluid along with low viscosity yellows solvent to enter porous solid materials more efficiently. This helps in rapid extraction with faster mass transfer. In this way, this method is better in

comparison to liquid solvents extraction method[27]. Also, the use of more volatile and harmful organic solvent is avoided. SFE can be scaled according to application from laboratory to industrial scale[23]. This process has been used in continuous, semi batch, and batch modes. For efficient and successful SFE, various factors like extraction time, thermodynamic conditions employed such as temperature and pressure, flow rate, sample and fluid type, play important role[24].

Supercritical Carbon dioxide extraction: The supercritical carbon dioxide is the carbon dioxide gas under pressure. This is a supercritical fluid solvent and usually applied as solvent due its modest critical conditions[21,28-31]. In the presence of 200 atmosphere of high-pressure, CO_2 changes its state from gas to liquid. This have been used as green and safe solvent for extraction of secondary metabolites including the aromatic molecules from plant species. After extraction process is complete at normal atmospheric pressure and temperature, the liquid CO_2 again changes its state from liquid to gas and evaporates from the obtained secondary metabolites leaving behind no solvent residue. This is an excellent choice due to its non-toxic, inert, and odourless properties[32-35]. This method has been successfully utilized for the extraction of essential oil from plants like rosemary, ginger, calendula, marjoram, etc. One of the noticeable limitations is the use of high pressure, heavy-duty stainless-steel equipment that makes the process slight expensive.

The Algerian rosemary leaves was extracted with supercritical CO_2 to yield 0.95-3.52g of essential oil/ gram of dry rosemary where the compound camphor (at 48.89%wt) was identified as the main constituent of essential oil[36].

Subcritical liquid extraction: The subcritical stage of a liquid reached when any liquid is at higher pressure than the critical pressure and at lower than the critical temperature or *vice-versa*[37]. This type of liquid has also been used for the extraction of essential oils. Continuous subcritical water extraction technique uses water as an extractant to extract essential oils at temperatures between 100^{o}C and 374^{o}C and pressure high enough to keep water in the liquid state[38]. Under these conditions, polarity of water decreases due to the breaking of intermolecular hydrogen bonds present in water. It makes water, a more effective solvent for several organic compounds[37]. This method of essential oil extraction is more efficient in terms of oil production per one gram of plant used[21]. This is a faster method than the traditional extraction techniques, sometimes three hours of extraction was completed in fifteen minutes only[39]. This is also an environmentally compatible technique with better essential oil quality[40-42].

Microwave-assisted extraction: Microwave-assisted extraction is an important another method utilized to extract essential oils from herbal materials[43,44]. This process is advantageous in terms of time reduction, higher selectivity, higher yield, and reasonable cost[45]. The microwave-heating in combination with other processes like steam distillation, hydro-distillation, hydro diffusion and gravity become more useful for the essential oil extraction[46-49].

The microwave extraction techniques have been used by many natural product scientists to obtain essential oils from herbs as early as in the 80's[50,51]. More recently, Mohammad-Taghi et al. also isolated the essential oil from *Thymus vulgaris*[52]. The microwave-assisted extraction method was also applied to get polyphenols from wild sage[53].

Solvent-free microwave extraction (SFME) is another alternative process for essential oil production that overcome the limitations like loss of some volatile compounds, use of toxic solvents, low efficiency, and so on[54,55]. In this method, essential oil is extracted using water present in the plant material without adding any solvent[56]. This method was developed by Cheat and co-workers[57,58] by integrating dry distillation and heating energy of microwave.

Assembly of SFME consists of a microwave connected to dry-distillation setup at atmospheric pressure[59]. In these processes, the plant material was moistened with the help of water by soaking it in some amount of water for 1 to 2 h in the beginning. The soaking duration is plant dependent. The water was drained off after soaking and moistened material is separated and further subjected to the microwave oven. The essential oil released under the influence of microwave and get collected using a condenser. Using the control panel of the instrument, the parameter of the extraction condition like temperature, irradiation power, and duration of the extraction were fixed. The obtained essential oils contain moisture which requires removal. This moisture content was removed with the help of anhydrous sodium sulphate. The essential oil passed through the anhydrous sodium sulphate at 4^{o}C in the dark, stored and used.

Cold Expression: Expression (or cold-pressed or expeller-pressed or scarification extraction method) is the oldest extraction method. This method is used solely for the essential oils extraction from the rinds of citrus fruits like oranges, bergamot, grapefruits, lemons, lime mandarin etc[60-62]. It is a physical process in which the peels and cuticles are pricked either manually or with the help of a machine to puncture the cells containing the essential oils. It produces a watery emulsion from which the essential oil is separated by centrifugation[61].

A method known as '*spugna* method' is used specifically in Sicily in ancient times to extract essential oil. In this method, citrus fruits were cut into two half and pulp was removed with a sharpened spoon-knife. The peels of the fruits were imperiled to pressing between hard object made up of baked clay (*concolina*) that is located under a large natural sponge. Sometime the peel was also bended into the sponge. Further squeezing of the oil emulsion absorbed by the sponge into the *concolina* or some other container gave the desired essential oil. This type of essential oil possesses fruit odour. The odour of the essential oil

obtained by this process is similar to the fruits from which extraction was done. The essential oil extracted with other methods usually do not show this type of property. Another ancient times process is called equaling or the *scodella* process. In this process copper or brass bowl which is shallow is being used. This shallow bowl contains a hollow tube in the middle similar to a shallow funnel that utilizes the whole citrous fruits.

Now days a machine is used to pierce whole citrus fruits, so that essential oil sacs get open up and oil and pigments flow down into a collection area. The juice and oil are allowed to squeeze out which is further separated. This method gives 100% pure essential oil retaining the properties of fruit used or plant used. Li et al studied the isolation of essential oils from lemon using the cold-pressing method[63]. The lemon essential oil showed excellent antioxidant properties. Lu-Martínez et al. reported the extraction of Prunus serotine oil, a *Prunus serotine* essential oil using the cold-pressing method[64].

Ultrasound-assisted extraction: Ultrasound contains high-frequency waves of sound. Ultrasound-assisted extraction (UAE) uses high-frequency waves of sound whose range varies from 20 kHz to 2000 kHz. UAE works on the phenomena of cavitation. The microscopic bubbles are produced and breakdown during the process. Ultrasonication increases the permeability of cell walls and produces cavitation. When the size of the bubble's increases, they collapse violently and give rise to micro-jets. These micro-jets induce mechanical forces which further damage the cell membrane[65] and facilitate release of plant essential oils. The ultra-sonication method showed many advantages. Some of the advantages includes a reduction in extraction time, and higher yield of extracted materials. In this method, the use of organic solvents has also been avoided or minimized[66]. The UAE also helped in the enhanced rate of extraction.

Kumar et al. reviewed the UAE of essential oils and other bioactive compounds from various fruits and vegetables[67].*Carum carvi* seeds have been used to extract essential oil using UAE and gave excellent yield of 80% within 30 min[68]. The UAE method was also used for the essential oil extraction from papaya seed[69] and winter melon seed[70].

Conclusion

Essential oils are plant-based odoriferous volatile oils which also known by other names like ethereal oils or volatile oils and possess a strong aroma. Due to their unlimited applications, they are attracting attention in recent times. The components present in them does not allow the use of extreme condition for their extraction from plant sources. This chapter covered the maximum possible extraction methods used by different researcher and industrial groups to extract the essential oils. The methods like solvent extraction, steam distillation, hydrodistillation, hydrodiffusion, solvent free microwave, and cold press methods have been discussed.

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