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Distillation Methods of Essential Oils

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Abstract: Methods used for obtaining essential oils can be ranged as simple, fractional, vacuum, water-steam distillations and micro-wave assisted hydro distillation. Distillation methods are mainly based on the difference of boiling degree or the solvent and transport effect of water-steam. Vacuum can be used for accelerating the process. The success of the methods is related to presence of undesirable compounds such as waxes, flavonoids, coumarone and deterioration rate of active compounds for whatever reason. Low-temperature water-steam distillation can avoid deteriorations but it cannot block the presence of the undesirable compounds. Micro-wave assisted hydro distillation is a new method resulting with the high degree pureness. But this method has not been placed well in commercial production of essential oils. Essential oils having a high potential to be used in medical care and food industry are also used as a feed supplement with the antimicrobial and antioxidant effects. It is aimed to discuss about the advantages, disadvantages of distillation methods against each other and their effects on quantity and the quality of active substances in this review.

Keywords: Microwave-assisted extraction, supercritical-fluid extraction, medicinal plants, water extraction

Esansiyel Yağlar için Distilasyon Yöntemleri

Öz: Esansiyel yağların elde edilmesi için kullanılan yöntemler basit, ayrımsal, vakum, su buharı destilasyonu ve mikro dalga destekli hidrodestilasyon olarak sınıflandırılabilir. Damıtma yöntemleri temelde kaynama derecesi farkına ve su buharının çözücü ve taşıyıcı etkisine dayanmaktadır. Vakum, sürecin hızlandırılması için kullanılmaktadır. Yöntemlerin başarısı vaks, flavonoid ve coumarin gibi istenmeyen bileşiklerin, elde edilen esansiyel yağ içerisindeki mevcudiyeti ve ne sebeple olursa olsun etken maddelerde meydana gelen hasarlarla ilişkilidir. Düşük sıcaklıkta uygulanan su buharı destilasyonu etken maddelerde meydana gelen hasar ve bozulmaların önüne geçse de istenmeyen bileşiklerin esansiyel yağda bulunmasını engelleyememektedir. Mikrodalga destekli hidrodistilasyon, yeni ve yüksek derecede saflık sağlanabilen bir teknolojidir. Ancak esansiyel yağların ticari üretiminde kullanımı henüz tam olarak oturtulamamıştır. Esansiyel yağlar yalnız sağlık ve gıda alanlarında değil aynı zamanda gıda ve yem katkı maddesi olarak da yüksek bir kullanım potansiyeli taşımaktadır. Bu derleme çalışmasında distilasyon yöntemlerinin birbirlerine karşı avantaj, dezavantajları ve etken madde miktar ve kalitesi üzerine etkilerinin tartışılması amaçlanmıştır.

Anahtar Kelimeler: Mikrodalga destekli distilasyon, su distilasyonu, süper kritik sıvı ekstraksiyonu, tıbbi bitkiler

1. Introduction

Essential oils, as an important compound, are oily liquids obtained from the stem, stock and leaf of plants, can be called as volatile, ethereal or essential oils. Essential oils, consisting of complicated mixtures of very low amounts volatile compounds, are present at different odor and aroma in several plants. Area of usage of essential oils is medicine, food, pharmacology and animal husbandry with the antioxidant and antimicrobial effects.

The most prominent feature of these components, giving the herbaceous odor and taste, is to be volatile and fragrant at room temperature. Essential oils densely contain terpenoids, acids, alcohols, aldehydes, ketones, acyclic esters, lactones and rarely nitrogen and sulfur compounds, homologues of coumarins and phenylpropanoids (Grassman vand Elstner, 2003).

Maceration and soxhlet extraction methods have been most commonly used to extract essential oils from medicinal and aromatic plants. Studies continue to work on increasing the quantity and quality of essential oils in plants. Efforts are being made to develop a more efficient extraction method than traditional methods by using modern technologies (Tongnuanchan and Benjakul, 2014). Steam distillation, water distillation, hydrolysis, microwave assisted water distillation and some extraction methods continue to be used traditionally in the supply of poultry feeds, pharmaceutical industry and acquisition of essential oils used in many fields. However, it is also known that the volatile active compounds of essential oils are extremely sensitive to heat and vulnerable to degradation. Some volatile compounds losses, low extinction efficiency, degradation of heat-affected unsaturated or ester components and toxic component residues in essential oil have prompted researchers to develop alternatives to traditional methods (Lucchesi et al., 2004). Among these alternative methods, supercritical fluids, ultrasonic and microwave technologies, which use less chemical and energy have lower cost, can be said to be the foreground (Azwanida, 2015). Microwave-assisted (MAE), ultrasound-assisted extraction (UAE) and supercritical fluid extraction (SFE) applications can be shown as new methods to get more efficiency at lower cost.

Following the ban on the use of antibiotics as growth promoters in poultry nutrition in European Union countries, the focus has been on working on alternative growth promoters. For this purpose, many medicinal and aromatic products and essential oils (thyme, rosemary, sage, laurel, garlic, propolis) emerged with antimicrobial, antioxidant, anti-coccidial and immunomodulatory properties (Brenes and Roura, 2010; Basmacıoğlu et al., 2010; Erener et al., 2010; Konanc and Ozturk, 2016; Küçükyılmaz et al., 2017; Sacildi and Ozturk 2018; Sahin and Ozturk, 2018). In this review, the methods of obtaining essential oils used in animal nutrition are discussed by comparing the effects on the amount and quality of active compounds.

Comparison of methods used for obtaining essential oils

Obtaining essential oils from plants are traditionally made by using water distillation, steam distillation and solvent

extraction methods. Although these processes appear to be advantageous at low cost, they have negative effects such as hydrolysis and heat-induced degradation. The use of solvents is not recommended because of the residues in the essential oils and the loss of essential oil from the solvent at the time of the evaporation (Charles and Simon, 1990). To overcome this disadvantage, supercritical fluids method has been developed and proposed (Mostafa et al., 2004). Some of the methods used to obtain essential oils can be ranged as pressurized liquid extraction, pressurized hot water extraction, membrane-supported solvent applications, solid-phase microextraction and ultrasound applications. Recently, microwave-assisted extractions have been used very efficiently because of the possibility of rapid heating of aqueous samples (Kosar et al., 2005; Chemat et al., 2006).

Super critical liquid extraction

Using water as solvent between 100 and 374 °C and applying a pressure that is high enough to protect the liquid. It has been suggested that it is a powerful alternative to the extraction of solid materials and its use in practice is promising (Basile et al., 1998; Luque de Castro et al., 1999).

Water distillation

Water distillation, known as the oldest method (Figure 1), is the most appropriate method to apply to fresh and dry plant parts which are not damaged by heating. The application is carried out by boiling water added to the top of the vessel where the plant is placed, transportation of essential oil by vapor and accumulating in the water due to density difference when it reaches the collecting vessel called Florentine Container (Başer et al., 1998).

Steam distillation

The distillation method in which steam with a certain amount of moisture is sprayed on the plant material placed on the grid in a similar system to water distillation and steam transfer of the essential oils is known as (Figure 1) steam distillation. Steam distillation is essentially a process of distilling plant material with steam generated by a boiler. In this method, the material is placed on a perforated plate above the steam inlet. It is easy to control how much steam is generated in the steam generating mechanisms. Furthermore, since the steam generator is outside of the distillation unit, the ambient temperature at which the material to be distilled is located is kept below 100° C and the occurrence of impairments due to the heat effect can be prevented or reduced (Öztekin and Soysal, 1998). The biggest problem of the steam distillation is the vapor pressure and the degradation which can occur when the flow rate is high.

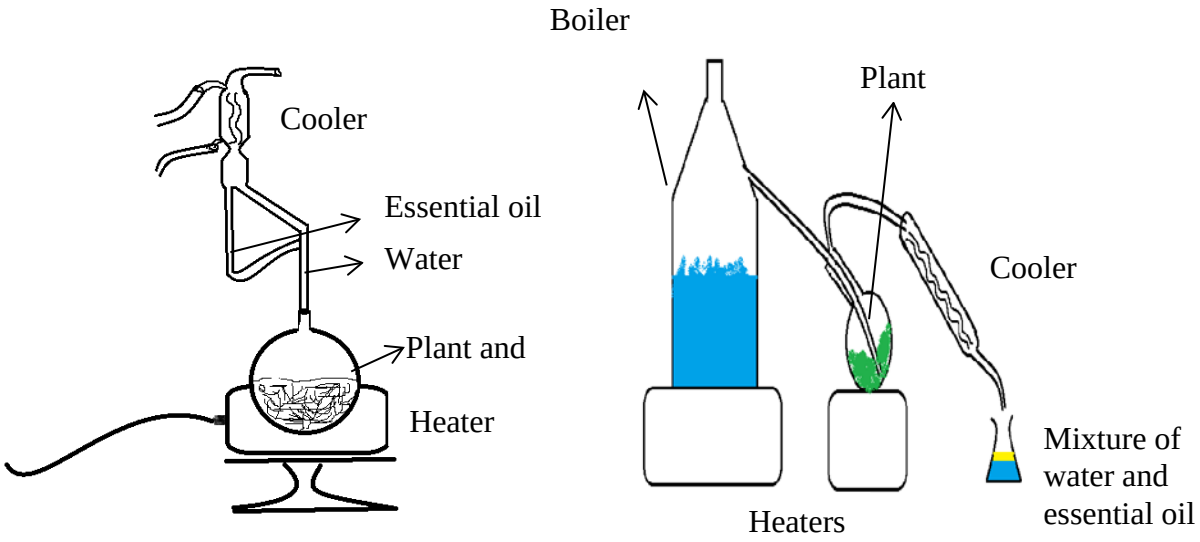


Figure 1. Water and steam distillation

Vacuum distillation

The boiling points of some compounds are quite high. However, they can be distorted even at temperatures below the boiling point. It is a difficult and inadequate method to extract these compounds under atmospheric pressure. It is therefore more effective to reduce the pressure rather than increase the temperature to avoid deterioration and this method is called vacuum distillation. As it is known, if the boiling point decreases as the outer pressure decreases the pressure is lowered below the vapor pressure of the compound and boiling with distillation begins (Kılıç, 2008).

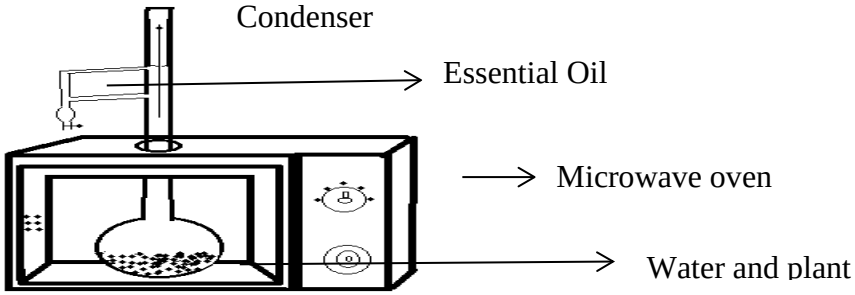
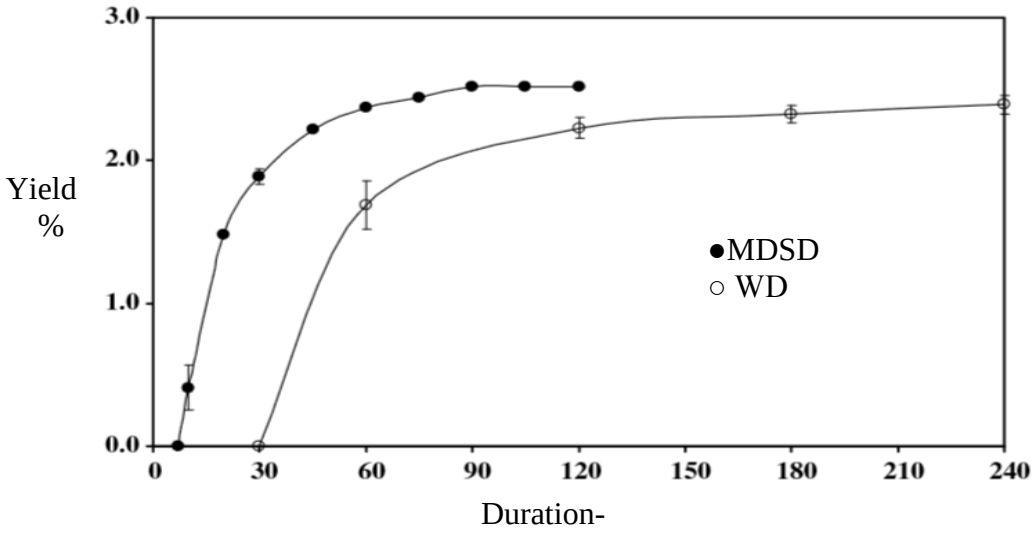


Figure 2. Microwave assisted water distillation

Microwave assisted water distillation

Microwave assisted water distillation, supercritical fluid extraction and ultrasonically assisted extraction methods have been developed and used to increase the quality of essential oils obtained by shortening the extraction period, increasing the efficiency and reducing the cost (Kaufmann and Christen, 2002). Microwave

assisted water distillation has previously been reported to be used in plants such as *Cuminum cyminum*, *Zanthoxylum bungeanum* and *Xylopia aromatica* but many more important plants such as thyme is needed to be studied (Stashenko et al., 2004; Wang et al., 2006).



MDSD: Microwave assisted water distillation, WD: Water distillation

Figure 3. Comparing water distillation and microwave assisted water distillation of obtaining thyme essential oil (Golmakani and Rezai, 2008)

Table 1. Essential oil amounts of Laurel obtained by different distillation methods (Flamini et al., 2007)

Method	Yield, %
Water distillation	0.784^{b}
Microwave assisted water distillation (200W)	0.813^{ab}
Microwave assisted water distillation (300W)	1.132^{a}
Pulsed microwave water distillation	0.654^{b}

It has been reported that microwave power is very important in using this method which is much more effective than the classical methods in which the boiling point is reached earlier in the microwave supported water distillation and the volatile compounds are obtained with higher efficiency (Lucchesi et al., 2004; Flamini et al., 2007). It has also been determined that the amount and quality of essential oil

obtained by microwave-assisted water distillation in 30 minutes is equivalent to the amount and quality of essential oil obtained in 4 hours and thirty minutes by water distillation, which is the most preferred method due to its low cost (Golmakani and Rezai, 2008).

When methods, advantages and disadvantages were discussed, are compared together, following conclusions can be reached. Essential oils are used for many purposes in the pharmaceutical industry, as a preservative in the food industry, an incentive and intestinal regulator to grow by taking advantage of antimicrobial and antioxidant properties in the feeding of animals. It has been found that water and vapour distillation are the most commonly used methods for obtaining essential oils used in many fields. Solvent extraction methods have been found to cause both residual formation and essential oil losses. It has been determined that steam distillation is due to the fact that the boiler is out of the system and the internal temperature is lower that the water distillation deterioration rate is less, but the main problem of this method is determining the steam flow rate and pressure. Microwave-assisted water distillation has been developed since the total amount of extraction required to reach the boiling point in water distillation has negative effects on both yield and quality of obtaining essential oil. In the microwave-assisted water distillation method, which reached the boiling point much faster, both yield and duration of extraction. It has been reported that microwave power is very important in using this method which is much more effective than the classical methods in which the boiling point is reached earlier in the microwave supported water distillation and the volatile compounds are obtained with higher efficiency (Lucchesi et al., 2004; Flamini et al., 2007). It has also been determined that the amount and quality of essential oil obtained by microwave-assisted water distillation in 30 minutes is equivalent to the amount and quality of essential oil obtained in 4 hours and thirty minutes by water distillation, which is the most preferred method due to its low cost (Golkamani and Rezeai, 2008).

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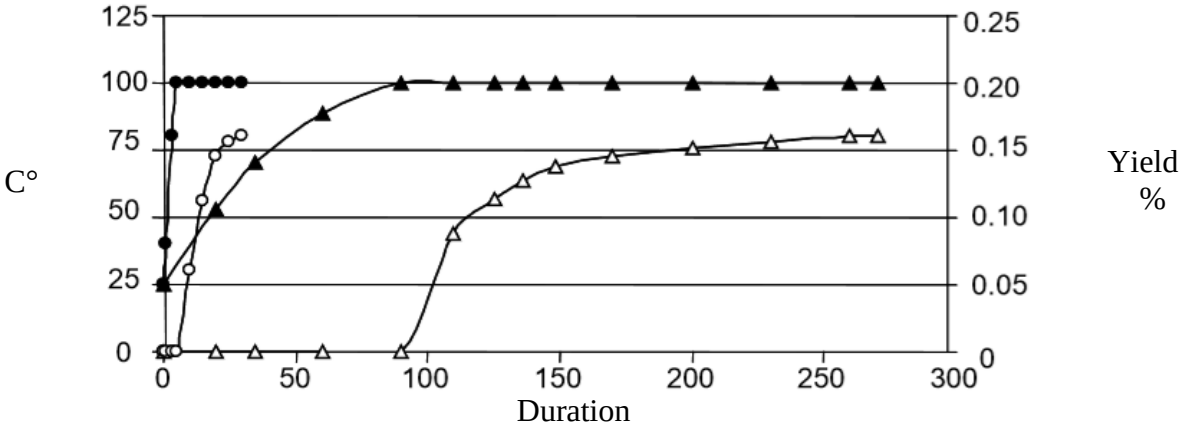

Heat profile ● : MAWD▲: WD, Yield; ○= MAWD Δ=WD, MAWD: Microwave assisted water distillation, WD: Water distillation

Figure 4. Comparing of microwave-assisted water distillation and water distillation (Lucchesi et al., 2004)

2. Result and Recommandations

Determination of the distillation method which provides the highest efficiency with minimum cost is the most important subjects that need to be emphasized for the sectors that process additives, food extracts, food and feed additives sectors and researchers doing research on this subject.

Extracts as a food and feed additive are an important cost factor in the production process. Minimizing this cost is directly proportional to the production of maximum extract at least cost. For this reason, technological advancements in distillation methods necessitate taking into account the high efficiency at low cost in unit time as well as its suitability for industry and bioavailability.

According to the results of this study, it is suggested that the microwave assisted extraction method should be applied in practice by paying attention to microwave power, more studies should be done in order to reduce cost and systems of vapour pressure and flow rate control, which are problems of steam distillation, should be integrated into existing systems.

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