Bartın Orman Fakültesi Dergisi | Journal of Bartin Faculty of Forestry
2023, 25(3): 492 – 513 | DOI: 10.24011/barofd.1303285

A Review of Extraction Methods for Obtaining Bioactive Compounds in Plant-Based Raw Materials

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Article History

Received: 27.05.2023
Accepted: 05.09.2023
Published: 15.12.2023

Review Article

Abstract - In recent years, the usage of lignocellulosic biomasses in food supplements has become popular. The studies about the development of reliable and short-term methods in the extraction of these active components in lignocellulosic biomasses and the usage of eco-friendly solvents have increased gradually. Traditional extraction methods take a long time and use a large amounts of organic solvents. In addition, the damages of used organic solvents to the environment are high considerably. For this reason, the need for technologies that reduce the extraction time and reduce the use of organic solvents and environmentally friendly "green chemistry" solvents is increasing day by day. In this review, traditional and modern extraction methods were examined and general application areas of these methods were introduced. Traditional extraction methods include maceration, pressing, infusion, digestion, percolation, decoction, reflux, tincture, distillation, and soxhlet methods, and modern extraction methods including pressurized liquid extraction, supercritical fluid extraction, ultrasonic assisted extraction, microwave assisted extraction, pulsed electric field extraction, enzyme assisted extraction methods, and their variation parameters were investigated. It has been seen that modern extraction methods are performed in less amount of sample, lower volume of solvent, lower energy consumption, and shorter extraction times compared to traditional methods. Within the scope of the study, considering all these, it has been determined that modern extraction methods are more applicable to nature-friendly methods than traditional methods.

Keywords: *Extraction, PLE, SFE, UAE, MAE*

Bitki Bazlı Hammaddelerdeki Biyoaktif Bileşiklerin Elde Edilmesi İçin Ekstraksiyon Yöntemlerinin İncelenmesi

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Makale Tarihçesi

Gönderim: 27.05.2023
Kabul: 05.09.2023
Yayım: 15.12.2023

Derleme Makale

Öz - Son yıllarda lignoselülozik biyokütlelerin takviye gıda ürünlerindeki kullanımı popüler hale gelmiştir. Lignoselülozik biyokütlelerdeki bu etkin bileşenlerin ekstraksiyonunda güvenilir ve kısa süreli yöntemlerin geliştirilmesi ve doğa dostu çözücülerin kullanılması yönünde yapılan çalışmalar artış göstermektedir. Geleneksel ekstraksiyon yöntemlerinin süreleri oldukça uzun olmakta ve çok miktarda organik çözücü kullanılmaktadır. Ayrıca kullanılan organik çözücülerin çevreye olan zararı da oldukça fazladır. Bu sebepten ekstraksiyon süresini düşüren, organik çözücü ve doğa dostu "yeşil kimya" çözücülerin kullanım miktarını azaltan teknolojilere ihtiyaç günden güne artmaktadır. Bu derleme çalışmasında, geleneksel ve modern ekstraksiyon yöntemleri incelenmiş ve bu yöntemlerin genel uygulama alanları ortaya konulmuştur. Maserasyon, presleme, infüzyon, çürütme, süzülme, kaynatma, reflüks, tentür, distilasyon ve sokslet metotlarının olduğu geleneksel ekstraksiyon yöntemleri ile basınçlı sıvı ekstraksiyon, süperkritik sıvı ekstraksiyon, ultrasonik destekli ekstraksiyon, mikrodalga destekli ekstraksiyon, darbeli elektrik alan ekstraksiyonu, enzim destekli ekstraksiyon metotlarının yer aldığı modern ekstraksiyon yöntemlerinin çeşitleri ve değişkenlik gösteren parametreleri araştırılmıştır. Yapılan incelemeler neticesinde modern ekstraksiyon yöntemlerinin geleneksel yöntemlere nazaran, daha az miktarda örnek, daha düşük hacimde çözücü, daha düşük enerji sarfiyatı ve daha kısa ekstraksiyon sürelerinde gerçekleştirildiği görülmüştür. Çalışma kapsamında, tüm bunlar göz önünde bulundurulduğunda modern ekstraksiyon yöntemlerinin geleneksel yöntemlere göre daha uygulanabilir doğa dostu yöntemler olduğu belirlenmiştir.

Anahtar Kelimeler: *Ekstraksiyon, PLE, SFE, UAE, MAE*

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1. Introduction

There are many herbal medicines used in public health from past to present. In general, about 80% of the world's population relies on the therapeutic effect of herbs (Daud et al., 2022; Kant and Kumar, 2022).

Considering the plant diversity we have and the existence of more detectable plants, studies on plant contents have been increased continuously. In addition, technological developments continue in the development of methods in order to carry out the works in a nature-friendly and high efficiency (Dönmez et al., 2016; Mousavi et al., 2022).

Lignocellulosic biomass is the residues of industrial woods and many plant species. Pine species, which have an important place as forest cutting residues, are extracted from widely used industrial woods such as oak, beech, and alder. Forest felling residues, also known as foliage, are bark, leaves, needles, flowers, cones, fruits, shoots and all twigs up to 0.6 cm in length (Vurdu, 1989; Alma et al., 2002). As herbaceous plants, endemic species found in the regions are known as plants that have been used for therapeutic purposes in public health from the past to the present. In studies with these plants, parts of the plants such as roots, stems, leaves, flowers and seeds are widely used (Dönmez et al., 2016).

Free radicals are reactive oxygen formed in the anatomical processes of the human body. Increases in reactive oxygen can cause many negative consequences in living things. Among the diseases that can be given as examples are cancer and diabetes, which have become very common. The most important effect of the recent increase in such diseases is the use of artificial antioxidants and synthetic drugs by moving away from natural (Borneo et al., 2009). In order to get rid of the negative effects of synthetic drugs and harmful chemical-containing foods, natural product orientation has increased considerably. It has been our primary effort to make use of our forest lands, which is the biggest heritage we have, in order to meet the consumption of natural products (Avan, 2021). Not only wood raw materials, but also many non-wood products and derivatives can be obtained from forest lands. The wood raw material wastes ıt obtain from forests and the bark, root, stem, shoot, leaf, fruit, etc. of herbaceous plant parts can be used. Studies on the determination of the components in the extracts obtained from different parts of herbaceous and woody plants show that these plants are very rich in phenolic compounds (Yusoff et al., 2022). However, the ratios of phenolic compounds in plants may vary according to the parts of the plants used. Apart from these, there are many determinant parameters such as the production of phenolics in the plant, genetic properties, extraction methods, and environmental conditions (Şahin, 2019; Agregan et al., 2021).

Apart from phenolic compounds, terpenes and alkaloids in plants are also important components. Terpenes, which are usually found free in plant tissues, are one of the largest classes of natural products. Apart from its free state, it is sometimes combined with proteins, sometimes as glycosides or esters of organic acids. These compounds are important for their odour. Alkaloids in the content of medicinal plants used in the treatment of diseases from past to present have an important place. Alkaloids are active, nitrogen-containing basic compounds. Alkaloids have the ability to be used as insecticides for defence against insects (Tring et al., 2021).

The main aim of this review; extraction methods, used for determining the active chemical compounds known as second metabolites of the plant materials, have been searched and discussed. In addition to performing the extraction process, which is the first and most important step in research, with the highest efficiency, its suitability in terms of issues that are gaining more and more importance day by day has been taken into consideration. Also, traditional and modern extraction technics have been compared and advantages and disadvantages have been stated.

2. Extraction

All of the process steps, which constitute the most important stage of such studies, and which ensure the separation of bioactive components from the inert and inactive parts of plant tissues, are called extraction.

Several steps are required to obtain bioactive components. First, the solvent is combined with the ground solid and the solvent enters the matrix of the sample. Finally, the dissolved substances pass into the solvent. When all these stages are examined, every element that accelerates the transport and solubility of the mass increases the quality of the extraction (Oprescu et al., 2022). Extracts from plants; it is expressed as the active ingredient obtained by applying according to an extraction method from various parts of herbaceous and woody plants such as leaves, flowers, roots, stems, seeds, fruits or bark, or as the sum of these components (Radosevic et al., 2015).

The most important step is to evaluate the extraction method of the samples in order to obtain qualitative and quantitative results in scientific studies. Since extraction is the first stage of scientific study, it has a significant impact on the final result of the study. As a result of choosing the most suitable one among the extraction methods, it will be easier to obtain the best data. Over time, very different methods have been developed for the extraction of the desired components in foods (Fu et al., 2021). Extracts of flavonoid and phenolic compounds found in herbaceous and woody plants can be obtained by using different extraction methods. In choosing the most suitable method among the different extraction methods, many features such as the chemical structure of the plant we will study, the variability of the component amounts, the heterogeneity of the matrices, and the polarities of the components are taken into consideration (Azmir et al., 2013).

In order to carry out a good extraction process, it is necessary to choose a cheaper, easier, faster, and environmentally friendly method and to obtain the desired bioactive components with higher efficiency. For this reason, there have been developments in methods such as solvent selection and sample preparation, which are in parallel with the developing technology (Pawliszyn, 2003). Some modern extraction methods such as pressurized liquid extraction, supercritical fluids, ultrasonic assisted extraction, and microwave assisted extraction have advantages in obtaining bioactive compounds from samples. Important parameters affecting the efficiency of the extraction process; temperature, time, solvent type, pH, particle size, and solid-liquid ratio. There are two different extraction method titles, classical (traditional) and modern methods (Calinescu et al., 2021).

2.1. Traditional Extraction Methods

2.1.1. Maceration

In the maceration method, a simple extraction method, the herbaceous or woody plant sample is cut into small pieces and then taken into a container with the solvent. The prepared mixture can be kept at room temperature or in an incubator depending on the desired time and temperature. After the specified time has elapsed, the extraction process is completed by applying the filtration process. In order to obtain more effective results in the maceration process, agitating the container with the mixture will allow the solvent to penetrate the sample better (Zhang et al., 2018). In other words, the maceration process takes place in 3 stages (Çopuroglu, 2013);

- The sample and solvent are mixed and stored for a certain period of time, usually at room temperature.
- Afterwards, the liquid is filtered from the mixture.
- The final stage is filtered into a clearer liquid (Figure 1).

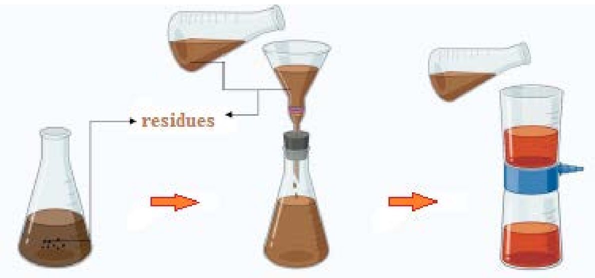

Figure 1. Maceration (Kumar et al., 2023)

Different phytochemicals such as polyphenols, flavonoids, alkaloids, tannins, coumarins, terpenoids, polypeptides, glycosides, steroids, quinones and saponins can be obtained by maceration (Sasıkala and Sundaraganapathy, 2017). Bioactive components that can be obtained from plants by maceration method may vary depending on the solvent used. Glycosides, alkaloids, glycosides and carbohydrates can be obtained by ethanol solvent extraction; terpenoids, alkaloids, glycosides and carbohydrates can be obtained by water solvent extraction; phenolic compounds, flavonoids, tannins, glycosides and amino acids can be obtained by methanol extraction (Abegunde and Ayodele-Oduola, 2015).

As with other extraction methods, the maceration method has both positive and negative aspects. Being an easily applicable method is one of the positive aspects of the simple experimental design. However, long extraction times and low productivity are disadvantageous. In addition, the maceration method can generally be applied to samples with heat-resistant components (Handa et al., 2008).

2.1.2. Pressing

The pressing method is based on the technique of filtering a solid or liquid substance by applying pressure to the liquid. There are two techniques, cold pressing, and hot pressing. In the distillation method used in the process of obtaining essential oil from herbaceous and woody plants, one of the reasons for preference is to obtain essential oil by cold pressing, since the temperature damages the volatile components (İmer and Taşan, 2018).

In cold press method, the sample is not treated with any chemicals and is not exposed to heat. In order to obtain oil from oil seeds in industrial establishments, the cold press method is completed in 4 stages. These; pre-cleaning, drying, grinding and, as the last step, pressing (Campbell et al., 2016).

In the simple pressing technique, sometimes the filtered plant material is placed in cloth bags and compressed with the help of hydraulic presses that will apply mechanical pressure to be applied on it, and oil extraction is carried out in this way. Sometimes it is carried out in the form of obtaining oil by means of a mechanical mechanism, by means of the pressure exerted on the plant sample by rotating the piston, which will help to apply pressure in the closed cabin (Figure 2).

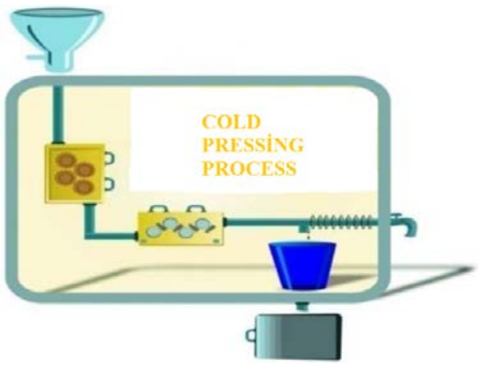

Figure 2. Cold press process (Geramitcioski et al., 2018)

One of the most important advantages of the cold press method is that it requires low energy and does not use solvents. Polyphenols, which are the most important components in essential oils, are not damaged, as there is no solvent usage and operation at high temperatures as in other extraction methods. Phenolic compounds are of great importance because they cause high antioxidant activity in plants and prevent oxidative degradation caused by free radicals. For this reason, the cold press method, which provides the opportunity to obtain oil with high content of phenolic components, has also been preferred (Maier et al., 2009). In hot pressing, cold pressing can be preferred since the structural decomposition of the polyphenols in the sample will cause undesirable changes in the color, smell, and taste of the oil, since a heat treatment will take place in the mechanical process. However, the efficiency of oils obtained by cold pressing method is lower than other extraction methods. This disadvantage can cause difficulties in the production capacity of the product in its use in the industry (Karabaş, 2013).

2.1.3. Infusion

The infusion extraction method is another traditional method similar to maceration. In the infusion method, the solvent is applied hot or cold. The plant sample to be extracted is ground into powder. The powdered plant sample is placed in a clean container and hot or cold extraction solvent is added (Figure 3). Depending on the intended use of the extract, the ratio of plant sample and solvent is usually 1:4 or 1:16. The plant mixed with the solvent is left for a short time. This method is favored in plant samples where easily soluble bioactive components are present. It is a widely used traditional method of preparing the extract practically even before use (Ergen et al., 2018; Abubakar and Haque, 2020).

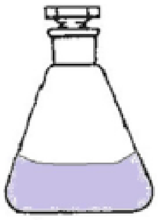

Figure 3. Infusion method (Belwal et al., 2018)

2.1.4. Digestion

Digestion is also a traditional method similar to the maceration method in which heat in the range of 40-60 °C is applied during the extraction process. Since the temperature in the specified range is not objectionable, it is preferred. The principle of the method is to add powdered plant material to the extraction solvent taken in a clean container. The mixture is then placed in an oven or water bath at around 50 °C. Temperature is used to reduce the viscosity of the solvent and for easier separation of secondary metabolites from the plant sample. During the extraction process, the mixture can be stirred by hand agitation, mechanical or magnetic stirrers. After 8-12 hours, the mixture is filtered and the extract is separated from the solvent. In order to obtain a fully efficient extract from the plant sample, the filtration process is completed by adding fresh solvent (Rasul, 2018; Hussain et al., 2019; Jha and Sit, 2022).

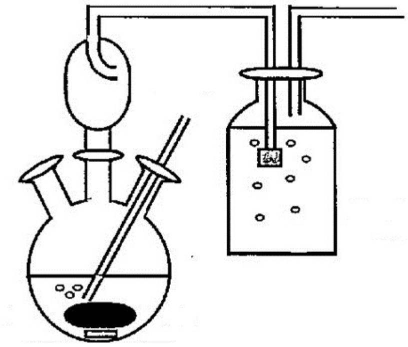

Figure 4. Digestion apparatus (Ashton et al., 1999)

2.1.5. Percolation

It is one of the most commonly used traditional methods for obtaining active ingredients in the preparation of liquid extracts. In the percolation process, a glass material in the form of a narrow cone open at both ends, known as a percolator, is used. Dried plant sample; ground and pulverized. Then, the plant material is moistened for 4 hours by mixing with the extraction solvent in a clean container. At the end of 4 hours, the mixture is transferred to a sealed percolator and kept at room temperature for 24 hours (Figure 5) (Majekodunmi, 2015).

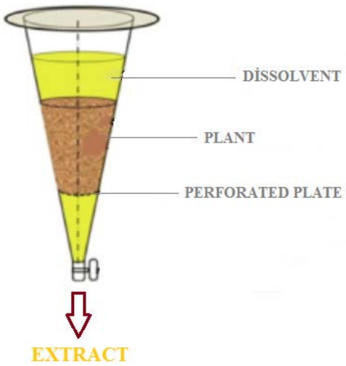

Figure 5. Percolation apparatus (Alara et al., 2021)

The mixture is fully saturated by adding solvent and the bottom of the percolator is opened to allow the liquid to drip slowly. Gravity is utilized to leach the solvent from the mixture by adding some solvent until a high percentage of extract remains. The extract is then separated by filtration method (Belokurov et al., 2019; Zhang et al., 2018).

2.1.6. Decoction

It is based on the procedure of boiling the plant sample in a certain volume of water. The plant sample to be extracted is powdered after drying and grinding. The powdered plant sample is boiled in a certain volume of water for 15-20 minutes. Depending on the intended use of the extract, the ratio of plant sample and solvent is usually 1:4 or 1:16. Heat is applied during the extraction period for the process to be fast and extraction to take place. With the applied heat, there is a decrease in the solvent volume due to evaporation during the extraction process. After the boiling process is completed, the mixture is cooled and then filtered (Chemat et al., 2017; Ingle et al., 2017; Hussain et al., 2019).

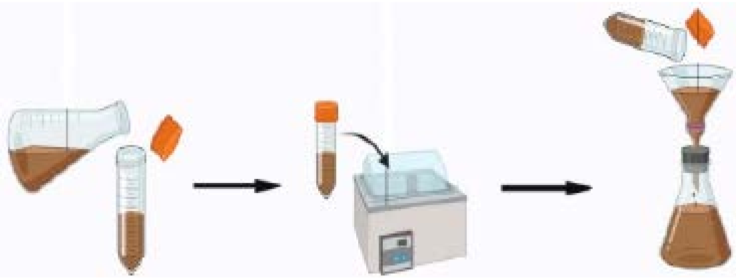

Figure 6. Decoction extraction techniques (Kumar et al., 2023)

In a study, phenolic compounds and antioxidant activities of *Teucrium polium* plant extracts obtained by decoction and infusion methods were investigated. The components of luteolin-7-O-glycoside, luteolin-5-O-glycoside, fumaric acid, and pelargonin in the extracts obtained by decoction method were determined as 1167.0; 835.2; 2060.1; 829.9 mg/kg dry plant, respectively. The components of luteolin-7-O-glycoside, luteolin-5-O-glycoside, fumaric acid, and pelargonin in the extracts obtained by infusion method were determined as 431.1, 278.4, 1456.2, 312.5mg/kg dry plant, respectively. 2,2-diphenyl-1-picrylhydrazyl (DPPH) free radical scavenging, β-carotene linoleic acid assays and cupric (Cu^{2+}) ion reducing power assay (CUPRAC) analyses were performed. The antioxidant activities of the extracts obtained by decoction and infusion methods were high (Özer et al., 2018).

Zhang et al. analyzed the hydrolysis of flavonoid glycosides in the extract of Danggui Buxue Tang (DBT) herbal mixture obtained by decoction method. They found that two flavonoid glycosides, ononinin and calycosin-7-O-β-d-glucoside, could be obtained by decoction extraction (Zhang et al., 2014).

2.1.7. Reflux

The reflux method is a solid-liquid extraction method similar to the soxhlet apparatus. It is a process in which the plant sample is repeated at a constant temperature with a boiling solvent for a certain period of time without loss of solvent. The plant sample and solvent are placed in a glass flask and a condenser is placed on the glass flask (Figure 7). This device vaporizes the solvent and then condenses it. Reflux extraction is widely used in the plant industry because it is practical, efficient, and cost-effective (Zhang et al., 2018; Chu et al., 2016).

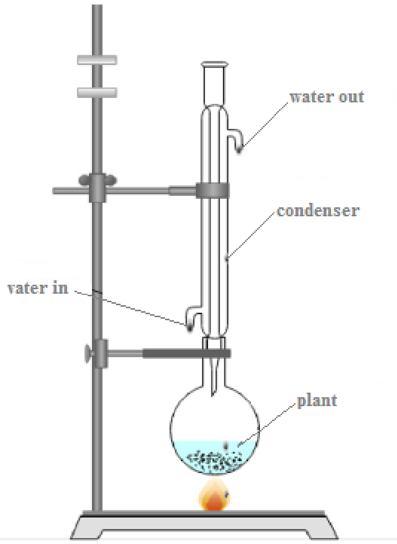

Figure 7. Reflux extraction techniques (Abd Aziz et al., 2021)

In their study, Kongkiatpaiboon and Gritsanapan extracted the root of Stemona collinsiae by soxhlet, maceration, reflux, ultrasonic and percolation methods. Didehydrostemofolin, a bio-insecticidal component, was found in the extracts obtained from S. collinsiae root. It was determined that the highest didehydrostemofolin content in extracts obtained with 70% ethanol was obtained from reflux and ultrasonic methods. However, the reflux is simple, cheap, and more suitable for upgrading to industrial processes. Therefore, it should be recommended for the extraction of high-quality extracts from the roots of S. collinsiae (Kongkiatpaiboon and Gritsanapan, 2013).

2.1.8. Tincture

It is an extraction method by treating one or more plant samples with alcohol. In this method, the fresh plant sample is generally mixed with ethyl alcohol in a certain ratio. The extract mixed with alcohol (tincture) can be kept at room temperature without deterioration due to the alcohol content (Rasul, 2018).

Tinctures, which are liquid alcoholic extracts from dry plant or animal drugs, are manufactured by a method similar to that used in the production of resinoids. Drugs used for some extracts are subjected to pre-treatments such as grinding, enzyme inactivation or degreasing. It is obtained by maceration, percolation or any other suitable method using alcohol in appropriate concentration. The alcohol content in the tincture produced is kept in the range of 20-60% by the manufacturers. In some tinctures, this ratio reaches 95% (Öztekin and Soysal, 1998).

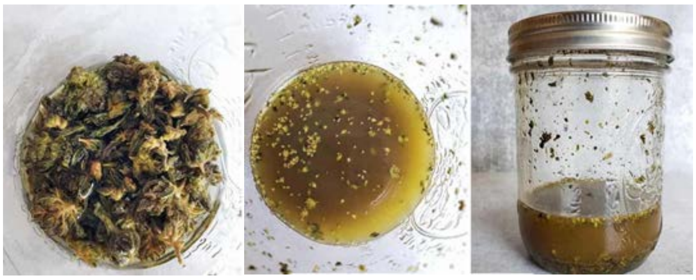

Figure 8. Tincture extraction techniques (Homestead and chill, n.d.)

2.1.9. Distillation

Working principle of the distillation method; It is based on the process of separating two or more liquid components using the difference of their boiling points or volatility (Figure 9). By heating the mixture consisting of substances with different boiling points to their boiling points, the substance with a low boiling point is primarily vaporized (Kaya and Ergönül, 2015). Afterward, the vaporous substance is condensed by the cooling unit of the distillation apparatus. It is easier to separate substances with large differences in volatility levels. The main purpose of this method; is to remove a volatile liquid or liquids of different volatility levels from a non-volatile substance (Cellat, 2011; Elguea-Culebras et al., 2022).

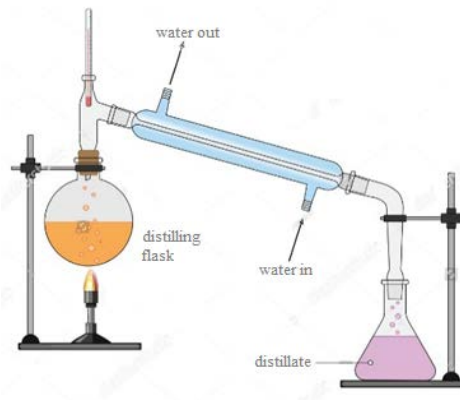

Figure 9. Simple distillation setup (Gavahian and Farahnaky, 2018)

2.1.9.1. Water distillation

It is one of the most widely used traditional methods for obtaining volatile compounds. It can be applied to dry or fresh plant samples that do not deteriorate when they reach the boiling point. In general, it shows high efficiency in small-size or powdered plant samples (Gavahian and Farahnaky, 2018). In this method, plant specimens in direct contact with water in a glass balloon are boiled for 2-8 hours with the help of a heater. With the realization of the boiling process, the oil molecules in the sample come with the evaporated water and then the condensed molecules are separated from the water thanks to the cooling unit of the mechanism (Akdağ and Öztürk, 2019). Small-scale studies in the laboratory can be performed with the Clevenger apparatus in Figure 10. However, in enterprises with industrial production, distillation boilers are used to operate in large volumes (Elguea-Culebras et al., 2022).

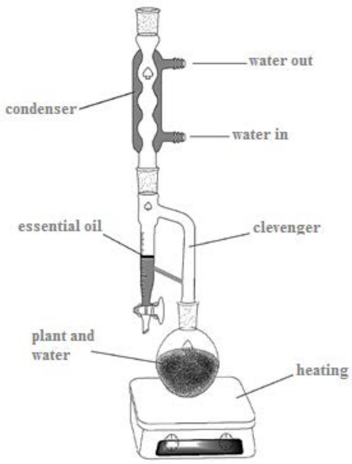

Figure 10. Water distillation device Clevenger (Elguea-Culebras et al., 2022)

The efficiency of essential oils obtained by water distillation method is high. Compared to other distillation methods, the colors of the oils obtained by the water distillation method are darker and their odors are more effective. The need for a large number of plant samples and solvents to obtain essential oil is among the negative features of the method. Although high-efficiency oil is obtained by water distillation, the negative effect of the method is that the long-term boiling at high temperatures causes the decomposition of the volatile components desired to be extracted from the plant sample (Kant and Kumar, 2022; Gavahian and Farahnaky, 2018).

2.1.9.2. Steam distillation

The steam distillation method is one of the traditional methods widely used to obtain oil from medicinal and aromatic plants. Thanks to this method, approximately 93% of the amount of essential oil in plants can be obtained. In steam distillation, the plant from which the essential oil is to be obtained should be selected from plants that have completed their development (Radwan et al., 2020; Antosz et al., 2022).

The steam distillation method is an easy method with a simple system compared to other methods. Another advantage is; is the use of water vapor instead of solvents polluting the environment in steam distillation. In this way, it has been preferred as an environmentally friendly method. Apart from its advantages, there are also situations that cause negative effects on studies and production. The biggest disadvantage; Although the amount of all sources used for steam distillation is high, essential oil is obtained in low yields. In addition, the fact that the distillation process takes place for a very long time can cause both losses of time and deterioration in the structure of the components desired to be obtained from the plant (Machado et al., 2022; Kant and Kumar, 2022).

In steam distillation, steam is produced by the steam generator in a separate place from the distillation boiler. The steam produced passes through the plant placed in the distillation boiler thanks to the mechanism (Xavier et al., 2011). The steam distillation mechanism works according to the distillation principle. Distillation is used to purify liquids at different boiling points and to separate their components. According to the working principle, the liquid mixture is heated until it turns into steam in order to separate and condense the desired components (Kaya and Ergönül, 2015).

There are two liquids in the steam distillation apparatus, immiscible water, and essential oil. The boiling point of the liquid mixture is the temperature at which the vapor pressure is equal to the total atmospheric pressure, according to Dalton's law of partial pressure. A temperature lower than the boiling point of the most volatile component will occur (Guntero et al., 2017).

Figure 11 shows the steam distillation setup. The steam produced comes into contact with the plant. In the closed container, water and essential oil are dragged under pressure and transmitted to the condenser, and condensed there. The water and essential oil mixture accumulated in the collection container as a result of condensation is separated due to the density difference (Cellat, 2011; Kant and Kumar, 2022).

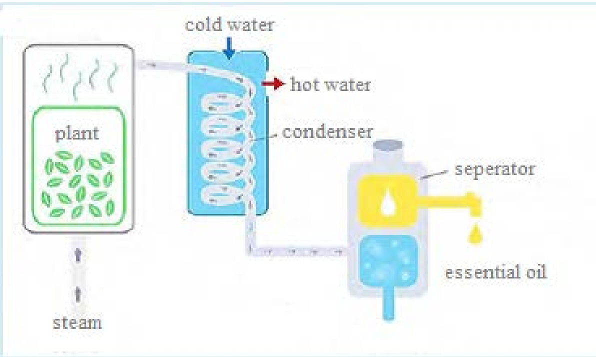

Figure 11. Steam distillation setup (Boukhatem, 2020)

When essential oil production is evaluated in industrial terms, it is a problem to obtain products with lower efficiency compared to laboratory studies. For this reason, industrial organizations have developed modified methods that can eliminate the deficiencies of steam distillation in order to eliminate the negative effects (Hasinah Johari et al., 2020).

Memarzadeh et al. (2020) obtained the essential oils from the Bakhtiari savory plant by using water distillation and microwave-assisted steam distillation methods. In the study, two different distillation methods were compared. As a result, they determined that microwave-assisted steam distillation produces better quality oil with less energy and wastewater in a shorter time than water distillation.

2.1.10. Soxhlet extraction

The soxhlet extraction is carried out in a special device. Soxhlet extraction apparatus; Soxhlet extractor consists of the glass balloon in which the solvent is placed, the cooling system (condenser), the part that provides the flow back to the glass balloon after meeting with the sample. In Soxhlet extraction, the plants are treated above the boiling point of the solvent used for a certain period of time. During the extraction process, when the solvent and the plant are together at the temperature for a very long time, the structure of the active components in the plant content may also deteriorate. Compared to modern extraction methods, soxhlet extraction is not an advantageous method because it uses a large amount of solvent and takes a long time. After the extraction process of the solvent used in excess is completed, the solvent is separated from the obtained solution with the evaporator. Since the solvent recovered in this way is not pure, it is not healthy to use it in the re-extraction method (de Castro and Priego-Capote, 2010; Zhang et al., 2018).

In Soxhlet extraction, applications take place between 6 and 24 hours. Pure organic solvents in the range of 100 to 500 ml can be used as solvents in the extraction. The solid material to be extracted is placed in the soxlet extractor with the help of paper crucibles. The solvent is placed in the solvent bottle and the bottle is placed in the heating system that will contact the heat (Figure 12). Thanks to the heating system, the solvent is treated at a temperature above its boiling point. The solvent, which is heated and evaporates after passing the boiling point, passes to the condenser where condensation will take place, where the gas condenses into liquid form with the effect of the cooling system and drips onto the solid material. In the continuation of this process, as the level of the solvent that meets the solid matter in the Soxlet extractor rises, it reaches the top of the siphon, and then the extractor is completely emptied with the help of the siphon and the solvent is filled back into the solvent bottle. In this way, a change in color is observed due to the constant meeting of the circulating solvent with the solid material during the extraction. However, with the effect of the temperature determined according to the boiling point of the solvent each time, only the solvent will evaporate and then condense and the solid material and the clean solvent will meet (Oluwaseun et al., 2018; George et al., 2019).

Kaya and Ergönül, (2015) used the soxhlet extraction method and the pressurized liquid extraction method in a study they conducted with citrus peels. As a result of the studies, they obtained high-efficiency products with low energy use in a short time with the pressurized liquid extraction method.

Samaram et al. (2013) used soxhlet extraction and ultrasound assisted extraction methods to obtain essential oils from the seeds of the melon fruit plant. When they compared the methods as a result of the studies, ultrasonic assisted extraction; it has been determined that it is more advantageous to be short in terms of time, to be effective in terms of physicochemical properties, and to provide high efficiency.

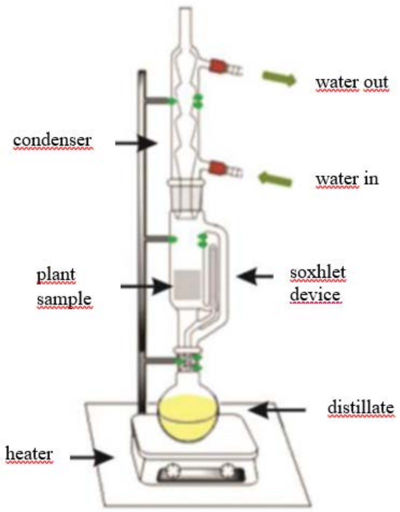

Figure 12. Soxlet assembly (Guntero et al., 2017)

2.2. Modern Extraction Methods

Encountered in classical extraction methods; Extraction techniques need to be developed in order to eliminate the negative effects such as high energy consumption, high cost, long extraction time, need for large amounts of solvent, the necessity of evaporation of the solvent, and decrease in efficiency as a result of the degradation of temperature-sensitive components (Büyüktuncel, 2012).

Today, the features sought in modern extraction methods developed to eliminate the disadvantages of classical extraction methods are; shortening extraction times, less solvent need, using environmentally friendly solvents, preventing energy consumption, reducing costs, preventing pollution, using renewable samples, and obtaining high-efficiency bioactive components from samples (Wen et al., 2018).

2.2.1. Pressurized liquid extraction (PLE)

The method, which Richter et al. found in 1996, is now referred to in the literature as high-pressure solvent extraction, accelerated solvent extraction or advanced solvent extraction. It has equipment that allows high temperatures to be used for traditional solvents and to extract the prepared sample in a high-pressure environment without leaking. By creating high pressure, it is ensured that the solvent remains liquid even at very high temperatures (Richter et al., 1996).

The pressurized liquid extraction method has been developed to eliminate the disadvantages of traditional extraction methods. Compared to conventional extraction methods, its advantages are that it is suitable for automation, short extraction time, less solvent requirement in the range of about 10-40 ml and different solvents can be preferred. It is aimed to extract the targeted analyte from semi-solid and solid matrices by keeping the solvent below the critical period thanks to the high pressure and temperature used in the pressurized liquid extraction method (Kaufmann and Christen, 2002).

The sample is loaded into the extraction chamber made of stainless steel and the solvent is pumped into the extraction chamber (Figure 13). Static transfer of the analyte from the matrix to the solvent takes place by adjusting the determined temperature and pressure values and keeping the values constant during the determined extraction time of the sample. After the extraction time is over, the pressure is released. Afterward, the obtained extract comes to the collection chamber of the device, and the chamber is washed with a clean solvent in order to take the remaining extracts into the pressure chamber where the extraction takes place (Mustafa and Turner, 2011).

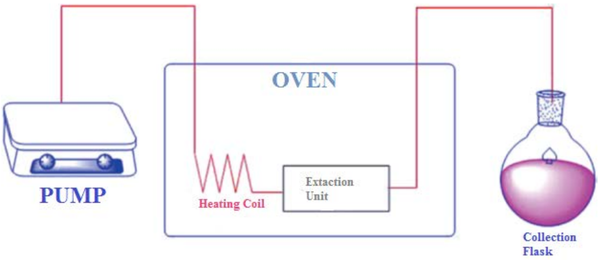

Figure 13. Pressurized liquid extraction system (Alara et al., 2021)

2.2.2. Supercritical fluid extraction (SFE)

A supercritical fluid is a substance, mixture, or element that can be applied at levels above its critical temperature and pressure used during extraction. Supercritical fluids are between gaseous and liquid states. The increase in temperature or pressure of supercritical fluids in a single phase does not turn into liquid or steam (Zougagh et al., 2004).

Supercritical fluids have low viscosity like gas, zero surface tension, high density like liquid and dissolving power in terms of their properties. Having these properties of gas and liquid provides high diffusion for analytes to which supercritical fluid is applied. It is easier to penetrate solid porous materials due to its low viscosity and high diffusion coefficients. For this reason, the fact that the spreading power and dissolving ability are higher than the liquid causes faster reaction formations (Başer, 2010).

Since supercritical fluids are compressible compared to normal fluids, their densities can be varied in wide ranges. The higher density of the supercritical fluid makes it capable of dissolving. In this regard, temperature and pressure adjustments are made so that density and other properties can be adjusted and changed as desired. Supercritical fluids can be preferred for matrices in different samples because they provide an enormous extraction environment. In addition, the possibility of changing the temperature and pressure for the dissolving power makes this method more preferable than the classical extraction methods (Büyüktuncel, 2012; Kant and Kumar, 2022).

In the supercritical fluid extraction system, there is a pressure cell in which the sample is contained, a pressure maintenance device for controlling the pressure in the system, a pump for CO_2 injection and a collection container (Figure 14) (Hussain et al., 2019).

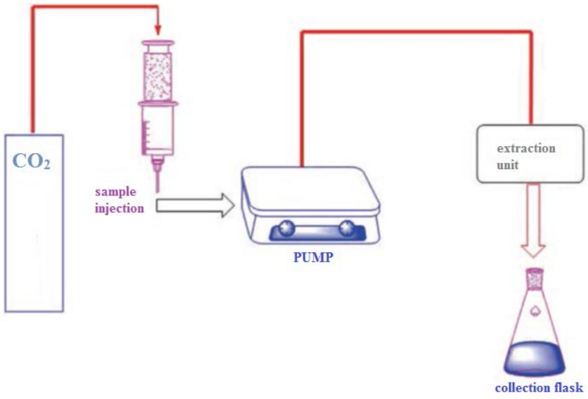

Figure 14. Supercritical fluid extraction system (Hussain et al., 2019)

Formation of supercritical fluid; It is carried out with an equipment where pressure, temperature and flow rate can be controlled. The device, which can operate in dynamic, static or static/dynamic modes, is generally operated in dynamic mode extraction. The extract obtained after the extraction process accumulates in the trap unit of the device or in the specified solvent. In the system, which needs solvent in the range of 10 to 20 ml, the extraction takes place in the range of 20-60 minutes. Compared to traditional extraction methods, it can be carried out in a short time and with less solvent. Compared to soxhlet extraction, higher efficiency results were obtained in terms of extraction time and solvent usage (Eisenmenger and Dunford, 2008).

Supercritical fluid extraction is used in food analysis, pharmaceutical polymer, and environmental fields. It has a wide range of uses in the crude vegetable oils industry. In oil production, refining should be carried out before consumption in order to remove unwanted compounds in it (de Azevedo et al., 2008).

2.2.3. Ultrasonic assisted extraction (UAE)

It is also called sound waves-assisted liquid extraction. In the ultrasonic assisted extraction method, the extraction process is carried out by spreading the sound waves to the environment with the help of devices that produce ultrasonic sound. These devices generally operate with frequencies in the range of 20-50 kHz. The sound waves created in the basic working principle of the ultrasonic extraction process cause the mechanical deformation of the cell wall in the sample and thus the material to pass into the sample (Figure 15). This method is preferred over traditional methods in order to ensure that the active components in the cell come into contact with the solvent and pass into the solvent easily (Avşar et al., 2014; Daud et al., 2022).

In ultrasonic assisted extraction studies in foods, two different types of ultrasound are used, namely high and low energy. High-energy ultrasonic applications are preferred for the termination of microbial and enzymatic activity in foods. Low-energy ultrasonic application is preferred in determining the physicochemical properties such as composition, hardness, and maturity of the food. Because a small amount of energy output will not cause chemical and physical degradation in the material affected by the wave (Ercan and Soysal, 2011; Dedebaş et al., 2021).

Ultrasonic assisted extraction is a method that uses solvents in the range of 20 to 200 ml and takes 2-20 minutes. The ultrasonically assisted extraction system can be a stable or an active system. In the active extraction method, it is advantageous because the active ingredients in the sample are removed as soon as they pass into the solvent. Another benefit of dynamic extraction is that the sample is constantly exposed to fresh solvent. The transition from the sample matrix to the solvent, which is constantly treated with fresh solvent, is greater (Tadeo et al., 2010; Yusoff et al., 2022).

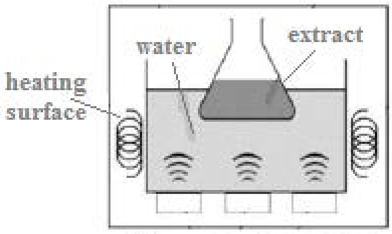

Figure 15. Ultrasonic assisted extraction system (Samaram et al., 2013)

Ultrasonic assisted extraction method is used to improve food quality, minimize nutrient loss in food and partially preserve vitamins. Sound waves can be used in extraction, emulsion, crystallization, drying, fermentation, accelerating oxidation in the food industry, filtering and degassing in frozen foods, and inhibition of enzyme activity (Chemat et al., 2011).

2.2.4. Microwave assisted extraction (MAE)

Microwaves are devices that can generate electromagnetic waves in the range of 300 to 300 000 MHz.
The basic element in the heating process in the microwave assisted extraction method is to ensure that the microwave has a direct effect on the molecules by means of ion transport (Büyüktuncel, 2012).

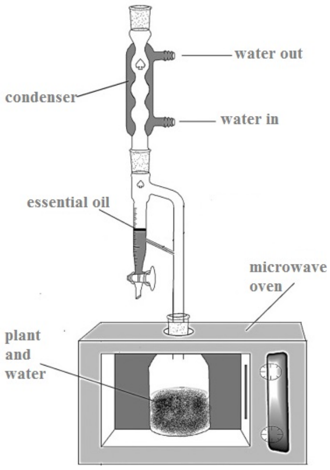

Figure 16. Microwave assisted extraction (Elguea-Culebras et al., 2022)

In many studies using the extraction method, ion transport and dipole rotations take place. The situation expressed as ion transport is the electrophoretic migration of ions under the effect of the magnetic field. Again, the realignment of the dipoles in the magnetic field is called dipole rotation. The resistance of the mixture formed by the sample and the solvent in the extractor to ion transport causes it to heat up (Kumar et al., 2016; Rodrigues et al., 2020).

Processes such as thawing frozen foods, cooking foods, heating, drying, pasteurization and extraction can be carried out with microwave devices. In the microwave assisted extraction system, microwave processing is carried out with the help of solvent in order to obtain the target components in the plant samples (Yılmaz et al., 2018).

In the microwave assisted solvent extraction method, there are two types of systems: the closed vessel model with controllable temperature and pressure, and the open vessel model at atmospheric pressure. While the temperature setting in the open vessel system is determined by the boiling point of the solvent, the pressure and temperature applied in the closed vessels can be increased as desired. While the cells are irradiated separately with the open cup method, all the cells are irradiated at the same time in the studies carried out in closed containers (Bouras et al., 2015).

When the studies on open and closed systems are evaluated, it is thought that the open vessel system gives better results than the closed vessel system in the extraction processes of essential oil. However, in some cases, the closed system has a disadvantage compared to the open system (Figure 16). In closed systems, after the extraction process is completed, cooling must occur in order to open the container. This cooling phase also causes the time to be extended by about 20 minutes (Saim et al., 1997; Famiyeh et al., 2021).

Microwave extraction can also be used for liquid samples, but it provides more efficient work for solid samples. Generally, 20 to 50 ml of solvent are needed to carry out the extraction process. The extraction time is around 20 minutes on average. It is highly preferred over traditional methods because of the high-efficiency extraction in a short time using a small amount of solvent (Tokkan et al., 2012).

Spigno ve Faveri (2009), the phenolic compounds in black tea were extracted using microwave assisted extraction and the brewing method, which is one of the traditional methods. In the microwave assisted extraction method, which lasted for 210 seconds, 43% higher phenolic content was obtained compared to the brewing method.

Alara et al. (2018) conducted a study in which they used microwave assisted extraction and soxhlet extraction to determine the total phenolic content of V. amygdalina leaf. Microwave assisted extraction (MDE) method, which lasted for 10 minutes, obtained a higher yield and more phenolic substances than the soxhlet extraction, which took 480 minutes.

2.2.5. Pulsed electric field (PEF) Extraction

In the extraction processes in the food industry, new technological methods that do not use heat instead of heat treatment have started to gain importance. One example is pulsed electric field extraction. Pulsed electric field extraction increases mass transfer by destroying the plant matrix. In this way, extraction time decreased and efficiency increased. The PEF study procedure depends on several parameters. These parameters are pulse number, process temperature, extraction efficiency, field strength and energy input. Since there is no heat treatment in PEF extraction, the degradation rate of compounds in plants that are not resistant to heat is reduced (Ağçam et al., 2014; Diaconeasa et al., 2023).

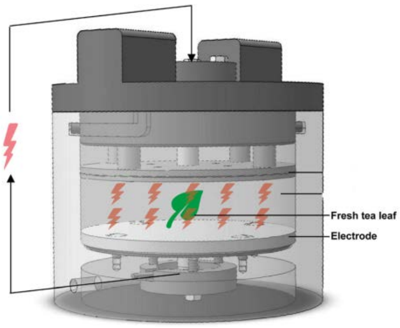

Figure 17. Pulsed electric field extraction schematic diagram (Liu et al., 2019)

Cannabis plant (hemp) oil was extracted from using pulsed electric field (PEF) extraction method. In this study, the effect of PEF electric field intensity and screw press speed on the physicochemical properties of hemp oil was investigated. The researchers stated that the PEF extraction method is a new, clean, environmentally friendly, efficient, and fast system especially for the petroleum industry (Haji-Moradkhani et al., 2018).

In another study, three different Sideritis scardica, Crocus sativus, and Vitis vinifera plants were extracted using pulsed electric field (PEF) extraction method and their polyphenols were investigated. An attempt was made to optimize the PEF procedure for the determination of total polyphenol content. For the three plants used in the study, PEF extraction was found to be an effective method for the extraction of total polyphenols.

The electric field intensity up to 1.4 kV/cm according to the PEF procedure showed that polyphenol content can be obtained with high efficiency (Lakka et al., 2021).

2.2.6. Enzyme assisted extraction (EAE)

Compared to traditional extraction methods, EAE is a more specialized method. In order to improve the shortcomings of the conventional method, the EAE method provides low energy, low chemical or water use, and fast action. This method is used to recover products such as peptides, chitin or pigments with high added value. In addition, high-quality chitin is obtained by chemical processes that ensure depolymerisation of the polysaccharide (Olaiya et al., 2022).

In the EAE method, enzymes that can hydrolyze components in the cell wall and matrix are advantageous. In this way, intracellular macromolecules in natural products are obtained with high efficiency. In EAE, α amylase, cellulose, and pectinase are generally used (Song et al., 2020; Mohan et al., 2022).

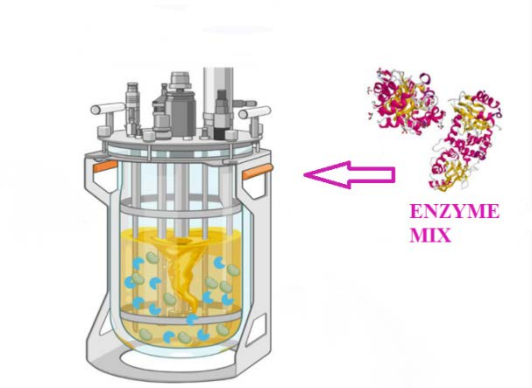

Figure 18. Enzyme assisted extraction (EAE) method (Diestra et al., 2020)

Chen et al. (2014) extracted Astragalus membranaceus plant by EAE method using glucose oxidase, amyloglucosidase, hemicellulase, bacterial amylase, fungal amylase, pectinase, cellulose and vinozyme enzymes. Polysaccharide content was investigated in the extracts obtained from the plant. Glucose oxidase showed the best efficiency among the enzymes used in the EAE method. Also, the polysaccharide yield in the EAE method using glucose oxidase was more than 250% compared to the method without enzyme.

In a study, the yield of chlorogenic acid in the extract obtained from the leaves of Eucommia ulmoides plant by EAE method using ionic liquids and cellulase was high (Liu et al., 2016).

Strati et al. (2015) obtained extracts using cellulase and pectinase enzymes and EAE method in their study with tomato wastes. Lycopene and carotenoid efficiencies in the obtained extracts were obtained at high values. Lycopene and carotenoid components were obtained with 10 times higher efficiency with the extraction method using cellulase and pectinase enzymes compared to the solvent extraction method without enzymes.

3. Conclusion

Lignocellulosic biomass has been used as a flavoring or protective in the public health and food industry with the existence of human beings on earth. Along with the developing technology, they wanted to get away from synthetic drugs and additives that started to pose a health threat by becoming conscious of people. For this, they started to search for alternative treatment methods with extracts and oils obtained from herbal products by different methods. For this reason, many scientists in the field continue their studies by considering different parameters.

Various extraction methods can be preferred to obtain the desired components from the plants used in the studies. There are parameters to be considered in the selection of extraction. These; it is an effective technique for the components we want to extract from the plant, an easy-to-apply method, cheap, short-term, environmentally friendly, energy-efficient, safe, less solvent needed, and repeatable. As a result of these researches, as a result of the increase of bioactive compounds and every sector where these compounds can be used, improved and improved extraction methods will be obtained to meet the demand.

Today, bioactive compounds are highly preferred in the pharmaceutical, cosmetic and food industries. Recently, in obtaining bioactive components; modern extraction methods such as pressurized liquid extraction, supercritical fluid extraction, ultrasound assisted extraction and microwave assisted extraction are preferred. The purpose of preferring modern extraction methods is that it can be carried out in a shorter time compared to traditional extraction methods, with low energy consumption and using less solvent, with an economic and environmentally friendly sensitivity. In addition, the fact that there are systems that facilitate the transfer of bioactive components during extraction and ensure that their structures are obtained without decomposition increase efficiency and quality.

When looking at the relationships between modern extraction methods, they are similar in many ways, but differ from each other with minor differences. Ultrasonic-assisted and microwave-assisted extractions were found to be more cost-effective than pressurized liquid and supercritical fluid extractions. It is concluded that ultrasonic-assisted extraction requires more solvent usage than pressurized liquid, microwave assisted and supercritical fluid extractions. In the supercritical extraction method, there is a problem in extracting polar analytes. Among the widely used modern extraction methods, microwave-assisted extraction, which is low solvent use, cheap, small amount of raw material requirement, short extraction time, polar analytes and almost everything can be extracted, has been observed as the most environmentally friendly method.

In recent years, human beings; it has become a common demand to turn to nature in order to eliminate the physical and psychological ailments experienced as a result of climate change, the deterioration of the balance of nature, synthetic drugs, and the negative effects of living conditions. In order to meet this demand, “green technology”, which will enable the continuation of works with more environmental awareness, will develop by trying to make more space for itself in the sector.

Author Contributions

Didem VEREP: Designed the study, conducted a literature review, and wrote the article.

Saim ATEŞ: Literature review and control

Eyyüp KARAOĞUL: Inspection and control

Conflict of Interest

No potential conflict of interest was reported by the authors.

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