Journal of Food Composition and Analysis 128 (2024) 105995

Contents lists available at ScienceDirect

Journal of Food Composition and Analysis

journal homepage: www.elsevier.com/locate/jfca

Recent advances in the extraction of bioactive compounds from plant matrices and their use as potential antioxidants for vegetable oils enrichment

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ARTICLE INFO

Keywords:
Flavoring
Bioactive compounds
Oxidative stability
Phenolic compound
Vegetable extracts

ABSTRACT

Oxidation of vegetable oils is a phenomenon that alters the nutritional value of the oil and produces unpleasant flavors, it is also the main factor limiting the quality of vegetable oils during storage. Synthetic antioxidants (SA) are known for their performance to improve vegetable oil oxidative stability. However, owing to their harmful effects on human health, the edible oil industry is seeking safe and healthy natural antioxidants. Indeed, such natural antioxidants, recovered from different plant matrices, demonstrate a potential effect, even better than SA in some cases, to preserve oils from oxidation and to satisfy consumer demand for natural foods. The enrichment of these oils using antioxidants from natural sources is proposed as a solution. Starting with a literature search using “enrich”, “oxidation”, “shelf-life”, “extract”, “antioxidant”, and “aromatic and medicinal plant” as key-words, the present paper provides a detailed overview of all extraction techniques for bioactive compounds and possible methods for enriching vegetable oil mentioning the advantages and drawbacks of each one.

1. Introduction

Vegetable oils play a prominent role directly or indirectly, as a catalyst, in human metabolic processes. They contain essential fatty acids, tocopherols, and fat-soluble vitamins, as structural components (Combe and Rossignol-Castera, 2010). Fatty acids, the building blocks of the fat in the human body, are divided into saturated and unsaturated. High levels of unsaturated fatty acids and low amounts of antioxidant compounds in oils result in the production of hazardous by-products and lead to a deterioration of the nutritional value of the oil. This happens especially after high-temperature frying (Viana da Silva et al., 2022). Preventing light, molecular oxygen, and high temperatures, optimizing oil extraction conditions, and adding antioxidant compounds are some methods that can be used to increase the oxidative stability of the oil during processing and storage (Grosshagauer et al., 2019). Butylated hydroxyanisole (BHA), butylated hydroxytoluene (BHT), tertiary butylhydroquinone (TBHQ), and propyl gallate are a few synthetic antioxidants used in the vegetable oil industry to retard oxidation reactions (Fadda et al., 2022; Gharby et al., 2022). However, it has been proved that these antioxidants can be associated with certain side effects. Thus, the demand of consumers for natural alternatives to synthetic additives has grown over the past decade. To meet this demand, natural antioxidants have been proposed as an option to synthetic additives in preventing oxidative degradation of oils (Xu et al., 2021). However, the ability of plant antioxidants to prevent oils from deterioration has been the subject of several research projects (Bodoira et al., 2017; Ghorbani Gorji et al., 2016; Iqbal et al., 2008). Thus, this study aimed to review the strengths and limitations of extraction techniques of bioactive compounds of plant origin, as well as some enrichment processes of edible oils with natural antioxidants, to upgrade their oxidative stability.

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https://doi.org/10.1016/j.jfca.2024.105995
Received 28 November 2023; Received in revised form 11 January 2024; Accepted 13 January 2024
Available online 18 January 2024
0889-1575/

1.1. Review methodology

To obtain an overview of the relationships among oxidative stability, oil, enrichment and natural antioxidants, the data and information were collected using the Scopus online platform. Recently published documents were searched using various keywords such as “enrich”, “enhance”, “improve”, “ameliorate”, “oxidative stability”, “oxidation”, “shelf-life”, “natural”, “extract”, “oil”, “phenol”, “antioxidant”, “aromatic and medicinal plant”, “medicinal and aromatic plant”, “AMP”, “MAP”, and “plant”. Once the appropriate database was selected, 109 documents were retrieved that were published between 1999 and 2022 (Fig. 1A). The most cited article (254 times) found was titled “Effect of storage on refined and husk olive oils composition: Stabilization by addition of natural antioxidants from Chemlali olive leaves” by Bouaziz et al. published in 2008 in Food Chemistry (Bouaziz et al., 2008).

The search results were tabulated in Excel format and analyzed using R Studio software. The results were visualized using OriginPro 2022 software and Term maps were produced using VOSviewer software. During the review, the data was tabulated with column headings such as year, author, type of publication, countries or territories of origin, etc. The author ‘Selin Sahin’ published the highest number of findings in this field (Fig. 1B). The most productive countries/territories and affiliations are shown in Figs. 1C and 1D, respectively. With regard to countries and territories, Iran was the most productive country, followed by India and the United States and Gorgan University of Agricultural Sciences and Natural Resources was the most cited affiliation. Fig. 2A depicts the distribution of documents by type according to the Scopus database. It includes mainly “research article” with 82%, followed by “review article” with 23%, and “conference paper” with 2%.

To assist in the creation of the bubble map, words/terms appearing at least five times in the publications were examined and visualized. The results are presented in the form of network visualizations and density diagrams. Based on the terms and density maps, three groups are generated (Fig. 2B). Group 1, in red, includes terms such as "antioxidant activity", "plant extract", "lipid oxidation", "polyphenol", and "flavonoid". Group 2, in blue, contains keywords such as "shelf life", "plant extract" and "plant leaf". The green group 3 includes the following keywords: "lipid peroxidation", "oils, volatile", "cytotoxicity", "oxidation-reduction", "essential oil" (Fig. 2B).

2. Extraction methods of natural compounds

Extraction is a method for recovering components from a solid mixture or solution. It is an indispensable part of sample preparation, experimental research, and qualitative analysis of bioactive compounds (Huang et al., 2013). Pre-extraction is the initial step in the extraction of any substance. It is the step for the preparation of plant samples to preserve the plant biomolecules before extraction. Fresh or dried samples can be used for pre-extraction (Zahran and Najafi, 2020). In medicinal studies of plant samples, fresh samples are fragile and tend to deteriorate more rapidly than dried samples (Kozłowska and Gruczyńska, 2018). The comparison between fresh and dried sample analysis in *Moringa oleifera* leaves revealed no significant effect on total phenolic compounds but flavonoid content was higher in the dried sample where ground or powdered samples were used (Vongsak et al., 2013). Moreover, in dry samples, the reduction in particle size improves the surface contact between the samples and the extraction solvents. Grinding results in more homogeneous and smaller samples, which leads to better surface contact with the extraction solvents. Similarly, in a study of *Centella asiatica* powder from nanoparticles produced by a mill, the

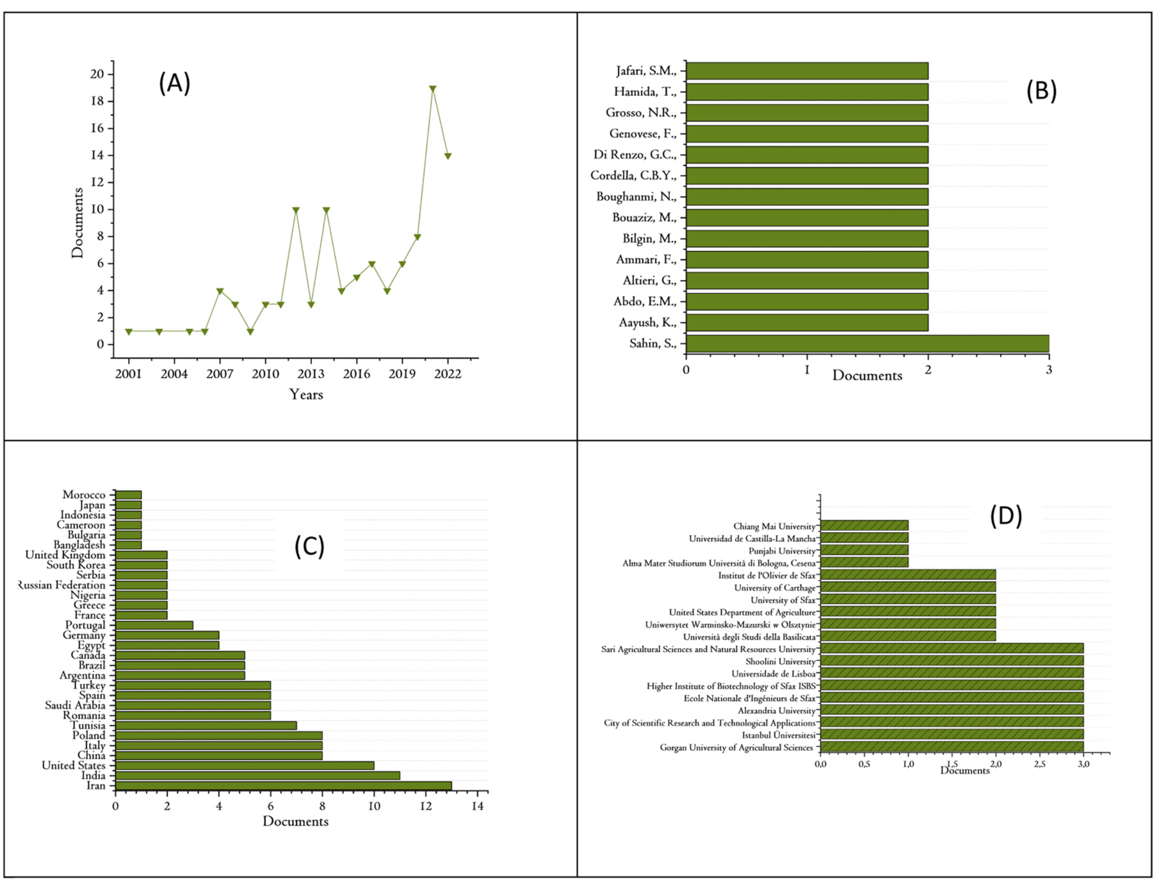

Fig. 1. Trend of no. of publications related to antioxidants across the year from the results of Scopus search databases (A), authors with highest no. of publication (B), countries and territories with highest productivity (C), and affiliations with highest no. of publication (D) The next figure provides an outline of the classification of the type of paper, of which the articles take the first rank (82%) followed by reviews (23%) (Fig. 2).

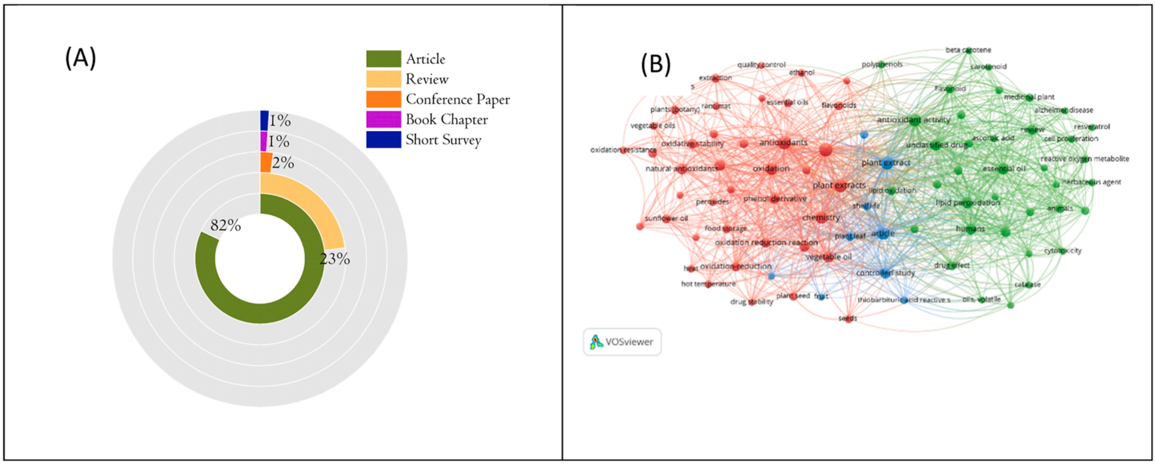

Fig. 2. Classification of documents according to their type (A) and terms map based on the generated by the VOSviewer software showing publication density (B). Term Map is a set of results visualized as a density map or bubble map created with Vosviewer version 1.6.18 and consists of extracting significant terms from abstracts and titles of publications. For the present bibliometric study, 1843 keywords were determined and plotted on a term map (Fig. 2B).

authors found a higher yield (by 82%) compared to the micro powder obtained by maceration in 90% methanol for 3 days (Borhan et al., 2013). Other methods of sample preparation such as air drying, microwave drying, oven drying and freeze drying can also be carried out (Abdullah et al., 2012; Kaufmann and Christen, 2002). However, air-drying takes longer than microwave- and freeze-drying and may be subjected to unstable temperature conditions.

The actual extraction process can be started after preparing samples. For the extraction, besides the sampling method (wet or dry), other factors such as the type and concentration of the extraction solvent, extraction temperature, extraction time, and extraction pH also influence the efficiency of the extraction process (Nguyen et al., 2016). Among them, solvent concentration is one of the most important and antioxidants have been extracted from foods and medicinal plants using a variety of solvents (Ait Bouzid et al., 2022; Lefebvre et al., 2021; Ranveer et al., 2013; Zanqui et al., 2021; Zeroual et al., 2022). The chemical composition and polarity of the antioxidant chemicals to be extracted need to be considered while choosing a solvent concentration (Nguyen et al., 2016). The majority of phenolic, flavonoid, and anthocyanin antioxidants are water-soluble. Water, ethanol, methanol, propanol, acetone, and their aqueous mixes are a few examples of polar and moderately polar solvents that are often used for extraction (de Camargo et al., 2016). Standard organic solvents, such as combinations of hexane with acetone, ethanol, methanol or ethyl acetate with acetone, ethanol, and methanol, have been applied for the extraction of fat-soluble antioxidants such as carotenoids (Faidi et al., 2016; Strati and Oreopoulou, 2011).

Conventional (hot water bath, maceration and Soxhlet extraction) and non-conventional procedures are the two most widely used extraction methods (Giacometti et al., 2018; Wang and Weller, 2006). The former is solvent-intensive methods and small-scale research or small production companies can use it. The later (non-conventional) is modern methods which use high energy input/high processing capacity to boost extraction efficiency and/or selectivity and is used in ultrasound, microwave, pressurized liquids, enzymatic hydrolysis, supercritical fluids, high hydrostatic pressure and pulsed electric field (Xu et al., 2017).

2.1. Conventional extraction methods

2.1.1. Maceration, infusion, percolation, and decoction

Maceration involves submerging the plants (coarse or powdered) in an airtight container filled with a solvent (called menstruum). Those samples can be kept at room temperature for at least 3 days and require stirring regularly until the soluble matter is dissolved (Manousi et al., 2019). The resulting mixture is then filtrated to remove impurities (Jovanović et al., 2017).

Infusion and decoction use the same principle as maceration; both are plunged into cold or boiled water until the soluble material is dissolved (Manousi et al., 2019). However, the maceration period is shorter in the case of infusion. For decoction, the sample is boiled in a specific volume of water for a specific time. It is only appropriate for the extraction of thermostable compounds, hard plant materials and other substances. Percolation is another extraction method with a similar basic principle to decoction. In percolation, dried and powdered material is transferred to boiling water and macerated for 2 h. This process is usually conducted at a moderate rate and it is recommended to finalize the extraction before evaporation to get a concentrated extract (Akram, 2018).

2.1.2. Hydro-distillation

Hydrodistillation is a traditional method for extracting bioactive constituents and essential oils from plants. In this method, the plant material is first placed in a still compartment, and then enough water is added and boiled. It is also possible to inject steam directly into the plant sample (Vankar, 2004). The major factors affecting the release of bioactive substances from plant tissue are warm water and vapor. In this process, the steam and oil mixture is condensed by cooling water and oil and bioactive compounds are automatically separated from water (Silva et al., 2005). In addition, there are three different kinds of hydrodistillation: Direct steam distillation, water and steam distillation, and water distillation (Azmir et al., 2013). The major drawback of hydrodistillation is that at the high extraction temperature, some volatile components may be lost, which limits its application, particularly for the harvesting of thermolabile compounds.

2.1.3. Soxhlet extraction

Soxhlet extractor is the most popular technique for separating bioactive compounds from plants (Bimakr et al., 2011). This apparatus is used to dissolve poorly soluble compounds from solid matrices. To do this, the Soxhlet apparatus's thimble chamber is filled with the finest sample, which is then placed inside a porous bag or "thimble" made of sturdy filter paper or cellulose (Fig. 3). Heating of the lower vial causes the extraction solvents to evaporate into the sample cartridge, condense into the condenser, and then drain. The procedure continues when the liquid contents empty back into the lower vial at the siphon arm (Fig. 3, Bimakr et al., 2011). In a Soxhlet extractor, several factors need to be considered such as matrix properties and particle size, and required

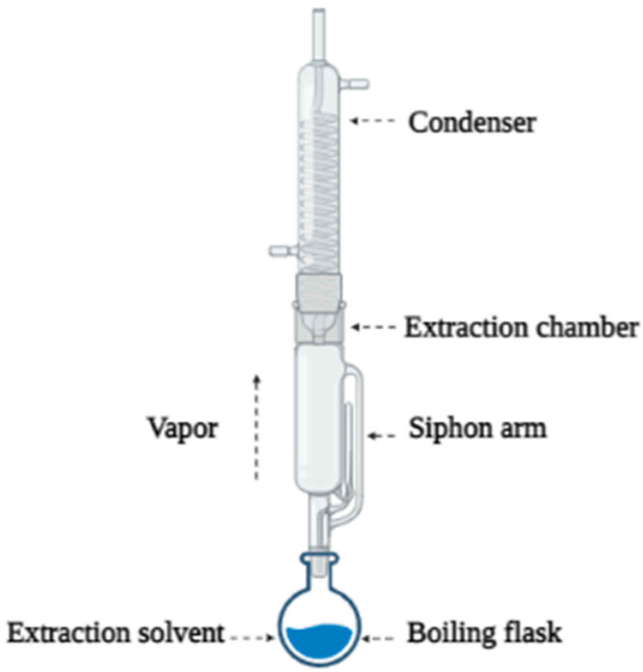

Fig. 3. Soxhlet extraction apparatus.

qualities of the solvent including chemical reactivity, selectivity, solvation, recoverability, density, and interfacial tension. To make the liquid phase more polar, a cosolvent may be added (Redfern et al., 2014). The advantage of using a Soxhlet extractor is the sample is repeatedly contacted with fresh solvent, which facilitates the shift in transfer equilibrium. In addition, as the system operated at a relatively high temperature, it allows for higher accuracy of extraction (Luque de Castro and Priego-Capote, 2010). However, Soxhlet extraction has several drawbacks such as long extraction time (6 h), and exposure to hazardous and flammable liquid organic solvents with potential toxic emissions during the extraction. To minimize this risk, the solvents used in the extraction system must be of high purity, which can increase the operating costs (Naudé et al., 1998). The ideal sample for Soxhlet extraction is also limited to a dry, finely separated solid and many factors such as temperature, solvent-to-sample ratio, and stirring speed must be considered for this method (Azwanida, 2015).

2.2. Non-conventional extraction methods

2.2.1. Microwave assisted extraction (MAE)

Microwave-assisted extraction (MAE) is a non-conventional method in chemical laboratories, first described in 1986 by Gedye, and is used to speed up the extraction of analytes from the sample matrix into the solvent (Vinatoru et al., 2017). In this process, the dipoles of polar and polarizable materials, such as solvents and samples, interact with microwave radiation by heating the materials close to the surface and transferring heat through conduction. The hydrogen bonding between molecules is broken by the electromagnetic microwave-induced dipolar rotation, which also encourages the movement of dissolved ions and the penetration of the solvent into the matrix (Kaufmann and Christen, 2002). In non-polar solvents, the heating is low because the energy is translated by dielectric absorption only (Handa et al., 2008). The benefit of this method is that it uses less solvent and requires less time for extraction than the standard approach (maceration and Soxhlet extraction). This method has the drawback of working only with small-molecule phenolic compounds, such as quercetin, isoflavin, and *trans*-resveratrol, which can withstand microwave heating conditions of up to 100 °C for 20 min (Boryana et al., 2007). Polar molecules and solvents with high dielectric constants are preferred by EAWs, which are regarded as selective methods (Azwanida, 2015).

2.2.2. Ultrasound-assisted extraction (UAE)

An ultrasound-assisted extractor (UAE) is a single type of sound wave that is above the normal range of human hearing. In chemistry, it is broadly between 20 kHz and 100 MHz (Azmir et al., 2013). Over the past three decades, food and pharmaceutical industries have widely used UAE as an efficient extraction technique (Esclapez et al., 2011). In this method, the cavitation phenomenon serves as the foundation and through a series of compression and rarefaction waves, ultrasound travels through liquid systems. This process has the potential to create cavitation bubbles in the fluid (Chemat et al., 2012; Soria and Villamiel, 2010). Until a critical threshold is reached, the diameter of these bubbles dilates for a few cycles. Then, they collapse and release a tremendous amount of energy, resulting in extreme temperatures (5000 K) and pressures (1000 atmospheres) at room temperature. The high temperature and pressure used during the extraction of bioactive components from plant materials would cause the plant cell walls to burst, making it easier for bioactive compounds to escape and improve mass transport. Also, frequency and yields of extraction are directly influenced by ultrasound frequency, intensity, temperature, and duration, (these parameters are fixed in a generator, Fig. 4). For an effective extraction, other factors have to be considered including solvent types, solvent volume, and sample characteristics like moisture content and particle size (Çevik et al., 2023).

2.2.3. Supercritical fluid extraction (SFE)

Compared to other methods, supercritical fluid is a precise method for recovering valuable compounds, especially from the perspective of environmental protection. Additionally, since CO_2 is gas at ambient pressure after the application of SFE, no additional separation step is necessary, making it simple to recover the extractant. CO_2 can also be recycled to counteract the greenhouse effect (Yen et al., 2015). The solvating properties of supercritical fluid (SF), whose characteristics are attained by using pressure and temperature that are higher than the fluid's critical point, are the foundation of SFE operation. SFE

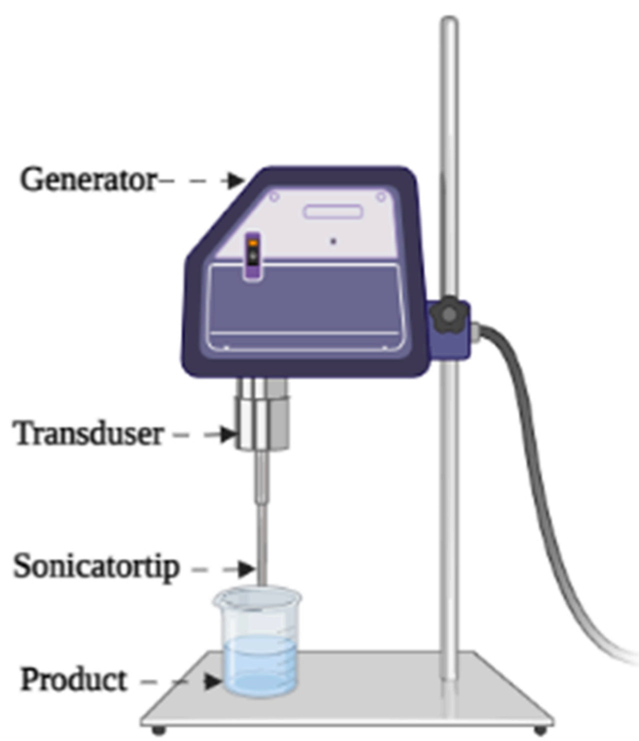

Fig. 4. Ultrasonic assisted extraction equipment.

outperform conventional extraction methods, offering greater flexibility through the control of parameters, the avoidance of solvents that cause environmental harm, and less energy consumption for solvent recovery after extraction (Crampon et al., 2011). Therefore, due to the numerous experimental results that have been reported, CO_2 extraction from various matrices is currently a topic of great interest (Mouahid et al., 2013). Moreover, in a study by Cuco et al. (2019), the simultaneous extraction of seed oil and active compounds from pumpkin rind, using subcritical and supercritical CO_2 as a solvent, was evaluated. This pressurized CO_2 extraction resulted in higher quality lipid extracts in terms of phenolic compounds, tocopherols, and phytosterols content, as well as thermal stability and antioxidant activity (Cuco et al., 2019). Also, the fundamental design of the procedure can be easily transferred from an experimental scale to an industrial scale (Reverchon and De Marco, 2006).

2.2.4. Pressurized liquid extraction (PLE)

Pressurized Liquid Extraction (PLE) is used for extracting specific components from a variety of matrices by using solvents under high pressure and temperature (Mustafa and Turner, 2011). The solubility of analytes can be supported, and the mass transfer rate can be increased by raising the solvent's temperature above its normal boiling point by increasing pressure. The temperature range for this extraction can be from room temperature to 200 °C and pressures ranging from 35 to 200 bar. Interesting results have been reported by Jaski et al. (2023) who extracted bioactive compounds from *Olea europaea* L. leaves with pressurized propane at the following conditions: temperature (60 °C), pressure (12 MPa), volumetric flow rate (1.5 $mL.min^{-1}$) and extraction time (60 min) (Jaski et al., 2023). Subcritical water extraction (SWE) is the name given to the PLE because water can also be used as a solvent (SWE). As water's dielectric constant (ε) decreases from 80 to 30 to 25, which is close to the dielectric constant of some organic solvents like ethanol or methanol, when heated to 200 to 250 °C in SWE, it can be stored in the liquid state (Herrero et al., 2015). As advantages, this extraction technique reduces the use of solvents, extraction time, operation steps and the presence of light and oxygen (Cai et al., 2016). It allows the extraction of a wide variety of antioxidants from plant materials such as carotenoids and anthocyanins (Bozan and Altinay, 2014; Breithaupt, 2004; Setyaningsih et al., 2016). Also, this method provides a short extraction time, elimination of post-processing steps, and a high potential for promoting the products' healthiness (Jaski et al., 2022).

2.2.5. Enzyme-assisted extraction (EAE)

Enzyme-assisted extraction (EAE) is a green potential extraction option as the extraction conditions are soft and practically do not have negative consequences on the environment (Xu et al., 2017). Enzymes have the characteristics of being extremely specific and effective, as well as having the capacity to break down compositions and destroy the integrity of the plant cell membrane, which facilitates the release of bioactive compounds. The enzymes most frequently used in EAE are cellulase, pectinase, hemicellulose, and β-glucosidase. These particular enzymes can be derived from various materials such as bacteria, fungi, fruit and vegetable extracts, or animal organs (Liu et al., 2014). Similar to PLE, EAE techniques have demonstrated the capacity to significantly improve the extraction efficiency of antioxidants, including phenols, flavonoids, carotenoids, and anthocyanins (Dinkova et al., 2014; Mushtaq et al., 2015; Ranveer et al., 2013). Key factors affecting extraction efficiency and yield include enzyme types, concentration, pH, incubation temperature and time, liquid/solid ratio, and particle size (Liu et al., 2016). Optimized EAE (enzyme concentration 2.24%, pH 6.58, 51.8 °C, 30 min) provides a 3-fold increase in the release of phenolic antioxidant compounds versus solvent extraction (Puri et al., 2012). However, there are some potential commercial and technical restrictions for the application of EAE; for example, the price of enzymes is relatively high, which impacts application, and enzyme activity fluctuates with environmental triggers such as temperature and nutrient availability (Liu et al., 2014).

2.2.6. High hydrostatic pressure extraction (HHPE)

High Hydrostatic Pressure Extraction (HHPE) is a new technique that is currently used for processing food at elevated pressure for the extraction of active ingredients from natural biomaterials (Shouqin et al., 2005). It should be mentioned that this elevated hydrostatic pressure signifies a high cold isostatic hydraulic pressure that varies from 100 MPa to 800 MPa or more. The most critical parameters that influence recoveries are the nature and volume of the solvent, pressure, extraction time, number of extraction cycles and temperature (Xi et al., 2011). In a study by da Silva et al. (2023), a number of process variables were used to optimize the extraction of total phenolic compounds from yerba mate leaves using HHPE. The optimum processing conditions obtained were 69% v/v water, 31% v/v glycerol, 50 MPa pressure and 10 min time (da Silva et al., 2023). The time taken for extraction/holding time of HHPE is only 5 to 20 min, which is very low compared to reflux or boiling methods, which take 1 to 3 h. It is also faster than supercritical CO_2 extraction, which requires about 3 h to complete the process (Khan et al., 2019). However, care must be taken with pressure because high hydrostatic pressure can lead to structural changes in structurally sensitive foods, including deformation of cells, denaturation of proteins, and deterioration of the cell membrane (Paul and Morita, 1971).

2.2.7. Pulsed electric field extraction (PEFE)

The use of pulsed electric field extraction (PEFE) has been explored as a non-thermal process to achieve improved extraction of intracellular compounds (Mohamed et al., 2012). In the process of extraction, PEFE is a treatment that involves applying high voltage (kV) pulses of low duration (s-ms) to a biological material situated between two electrodes (López et al., 2009). This voltage causes an electric field whose intensity is related to the voltage delivered and the distance between the electrodes (Zderic and Zondervan, 2016). The PEFE cause electroporation which increases the permeability of the cell membrane to ions and macromolecules (Vallverdú-Queralt et al., 2012). New variables, such as the pulse intensity and the number of pulses used, are integrated during the pulsed electric field pretreatment of the material. The range of pulse intensity used to permeabilize the cell membrane for the study of extraction in organic materials is between 1 and 10 kV, while the number of pulses is between 5 and 50 (López et al., 2009; Vallverdú-Queralt et al., 2012). This technique is indeed economical, non-destructive, and easy to scale up but with two limitations, i.e., the dependence on the composition of the medium (conductivity) (Martínez et al., 2020).

2.2.8. High voltage electrical discharge extraction (HVEDE)

The high voltage electrical discharge (HVED) technique was developed as an electrical processing technology for the removal of biomolecules from various products (Boussetta and Vorobiev, 2014). Under the action of a high current/high voltage electrical discharge, a plasma channel is created between two submerged electrodes and the energy is injected directly into an aqueous solution (Rajha et al., 2015). It leads to physical processes and chemical reactions, such as the formation of shock waves, the generation of high intensity UV light and the creation of hydroxyl radicals by photodissociation of water. Still, this method is associated with some negative outcomes, like producing tiny particles, which can complicate the subsequent solid-liquid separation step (Bouras et al., 2016).

2.2.9. Combined techniques

Carpentieri et al. (2021) talked in their article about "Synergism between Solvents and Technology", which consists of combining solvents and some of the extraction techniques mentioned above, highlighting their affinity with the type of solvent, the matrix and the target bioactive compounds (Carpentieri et al., 2021).

For higher efficiency, combinations of extraction techniques can also be adapted, as Patil et al. (2021) included in their review where they combined EAE and SFE while converging recent advances in the field that outperformed conventional extraction processes. Also highlighting several experimental conditions affecting EAE and SFE processes individually and together in order to get an overview of the overall efficiency evaluation of the combined approach (Patil et al., 2021). Similarly, Teles et al. (2021) combined enzyme-assisted extraction and high hydrostatic pressure for the recovery of phenolic compounds from grape marc (Cascaes Teles et al., 2021). The extraction process by pulsed electric field followed by an ultrasonic extraction process has also been adopted by Ziagova et al. (2022) for the isolation of antioxidants from aromatic and medicinal plant extracts.

3. Enrichment methods of vegetable oils using natural compounds

Vegetable food oils are typically rich in unsaturated fatty acids, which are subject to oxidation, the major cause of oil damage (Viana da Silva et al., 2022). The most common edible added oils were refined oils, including soybean and sunflower oils, but also virgin oils such as extra virgin olive oils as shown in Table 1. To overcome this problem several studies have resorted to the addition of natural source anti-oxidants or what is known as enrichment (Sousa et al., 2015). In addition to its effect on improving the oxidative stability of oils, the incorporation of flavors into oil affects several properties, including the sensory attributes of oils (Moczkowska et al., 2020). But their presence should be maintained at low or moderate levels in terms of sensory acceptance in order to avoid over-flavoring, especially in the case of certain intense spices, their quality and shelf life may also be affected, as the inclusion of antioxidant and/or pro-oxidant substances impacts the stability of the oils (Kandylis et al., 2011; Matsakidou et al., 2010). This part gives an idea of the set of enrichment techniques that can be used once the antioxidants are extracted.

3.1. Enrichment by maceration, infusion

The enrichment by maceration or infusion facilitates the extraction of fat-soluble active ingredients by simply incorporating and then blending plant extracts in a fatty substance (the oil to enrich) that acts as a sort of organic solvent (Sousa et al., 2015). Compared to other enrichment methods, this is the easiest to carry out even if it can take much longer (the longer the plant is left to macerate, the better the mixture is enriched) (Ayadi et al., 2009; Caponio et al., 2016). Gambacorta et al. (2007) used some herbs (hot pepper, garlic, oregano and rosemary) for the enrichment by maceration of the extra virgin olive oil and they subsequently reported that during the 7 months of storage of the flavored oils, all the herbs and spices showed antioxidant activity at all the concentrations tested since they stopped the formation of primary oxidation products (Caponio et al., 2016). Similarly, using the maceration process, rosemary and thyme were added to the soybean oil, in order to evaluate the effect of this enrichment; these samples were subjected to 24 h of heating at 180 °C and then analyzed every 6 h. After 24 h of warming, the incorporation of these oils by maceration resulted in a 69% reduction in polar compounds for rosemary and 71% for thyme compared to the control. In addition, the use of the two extracts allowed the preservation of approximately 50% of the total tocopherol content up to 18 h for rosemary and 24 h for thyme in the flavored oils. Volatile compounds known for their antioxidant activity were also identified in the developed oils (Saoudi et al., 2016).

3.2. Enrichment by co-processing

Co-processing, also called co-extraction or enrichment during extraction, is a method that allows the production of flavored or enriched oils. It is the addition of ingredients, such as fruit or aromatic and medicinal plants, during vegetable oil extraction (Baiano et al., 2009). This technique allows the selection of the nature of seeds (e.g. cultivar and stage of ripening), the ingredient(s) with the greatest aromatic and/or bioactive potential, and the extraction conditions

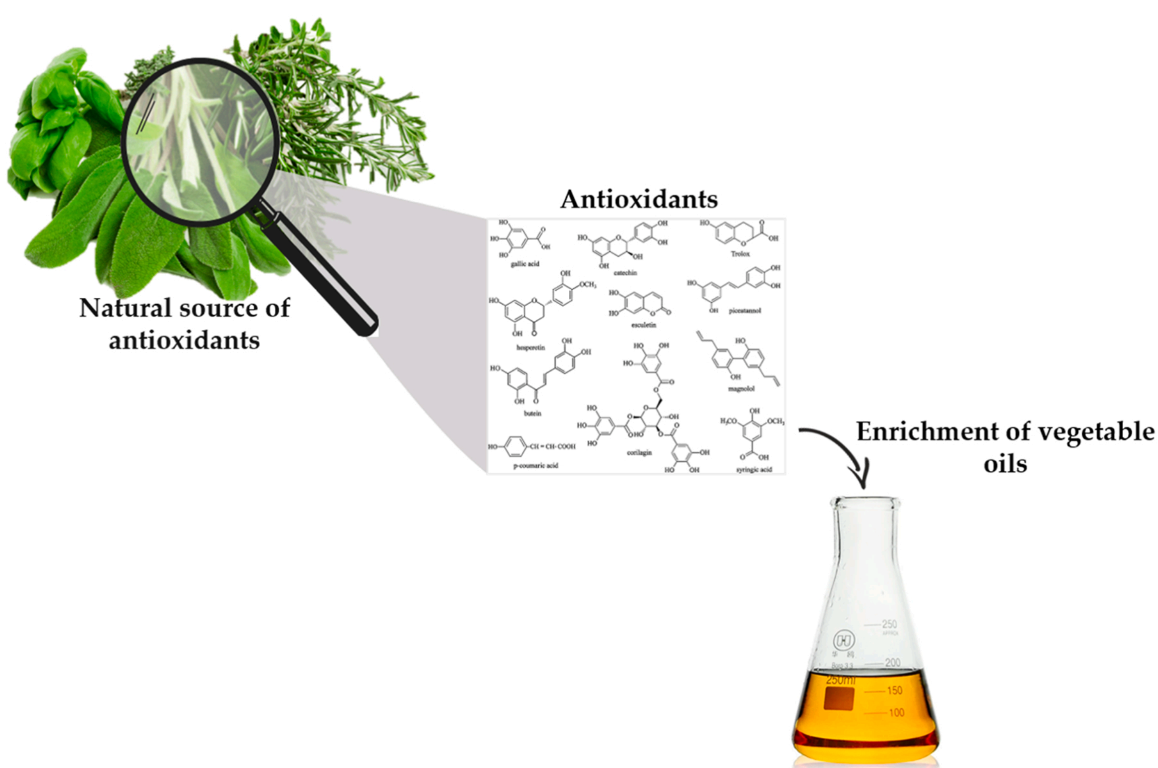

Fig. 5. Enrichment of vegetable oils using natural sources of antioxidants.

(Caponio et al., 2016). Such enrichment is useful to increase the antioxidant activity and the nutritional value of the oil (Bendini et al., 2015; Peres et al., 2021; Sacchi et al., 2017). Caponio et al. (2016) evaluated the influence of aromatization technique on the chemical and sensory quality of olive oils flavored with dried basil, dried chilli pepper and a combination of them. Their results showed that such a mixing method is effective in extracting phenolic compounds (Caponio et al., 2016).

3.3. Enrichment with essential oils

The enrichment using essential oils is also used for the aromatization of vegetable oils. The extraction method used for the recovery of these essential oils is hydrodistillation (Dorman et al., 2003; Milos et al., 2002). Besides the remarkable smell generated by the addition of these essential oils, it has been shown that they have a positive role in the preservation of oils against oxidation (Ascrizzi et al., 2019).

Moreover, lower values of lipid oxidation indicators [K232, K269, peroxide value (PI) and anisidine value], were presented in olive oil enriched with oregano oil (Asensio et al., 2013). Similarly, Benkhoud et al. (2022) found the effects of mixing six essential oils to form an extra virgin olive oil (EVOO) with volatile and sensory qualities, oxidative stability, fatty acid composition and nutritional value. This finding confirmed that essential oils can be successfully used to aromatize EVOO. Also, flavored EVOO has great potential as food, pharmaceutical and cosmeceutical utilities (Benkhoud et al., 2022).

3.4. Enrichment with specific extracts

Using different methods previously mentioned in the section (extraction methods of natural compounds), bioactive compounds (flavonoids, carotenoids, polyphenols, etc.) are extracted from different sources such as aromatic and medicinal plants, fruits, vegetables, herbs and, food by-products (Blasi and Cossignani, 2020a; Fadda et al., 2022). Several examples of enrichment have been reported, including the work of Ribeiro and Jorge (2017) who prepared phenolic extracts of coffee (*Coffea arabica* L.) for addition to soybean oil to boost its oxidation stability (Ribeiro and Jorge, 2017). The antioxidant capacity of garlic extracts was investigated in the stabilization of sunflower oil and demonstrated significant results (Iqbal and Bhanger, 2007). In another study, the antioxidant activity of spinach extract was used for the stabilization of sunflower oil under accelerated storage conditions (Tehseen et al., 2021). In a different work, a carotenoid extract was prepared from mango skin to be used in the enrichment of sunflower oil (Sánchez-Camargo et al., 2019).

3.5. Enrichment by ultrasound-assisted maceration

Ultrasonic maceration has also been developed as a method of enrichment. The first direct enrichment using continuous extraction assisted by ultrasound, was developed in 2008 by Luque de Castro and collaborators (Japón-Luján et al., 2008). Ultrasound has been applied to improve the extraction of natural products from plant materials, thanks to the cavitation phenomenon, and also to accelerate the release of bioactive plant components through rupture of the cell wall and the intensification of mass transfer (Vinatoru, 2001). To make it happen, this approach involves the introduction of bioactive compounds (plant, spice, etc.) and the oil to be enriched into the ultrasonic apparatus. In this process, a rotating blade is used to homogenize the mixture. The mixture is then filtered to remove the traces of leaves before analysis (Achat et al., 2012). While comparing two enrichment techniques; conventional maceration and ultrasonic maceration, treatment time is reduced from hours or days to minutes (Veillet et al., 2010).

3.6. Enrichment by microwave-assisted infusion

Lately, the utilization of microwave energy has sparked some consideration as an appropriate strategy for the rapid extraction of volatile compounds from aromatic plants (Farhat et al., 2009). Benmoussa et al. (2016) enriched olive oil with rosemary leaves using microwave-assisted infusion rather than traditional infusion. To fast-track the diffusion of volatile compounds from rosemary into the olive oil, the mixture was heated by microwaves. The infusion of dried rosemary leaves in olive oil has greatly accelerated by this method: yielding flavored olive oil in just a few minutes. Moreover, all quality parameters and fatty acid compositions were well within the limits. This can be explained by the antioxidants provided by the compounds infused into the olive oil, thus improving the relative heat resistance and stability against the high temperature used during MAI. Ultimately, this enrichment method proved a viable and rapid infusion method to effectively flavor olive oil with dried rosemary leaves (Benmoussa et al., 2016).

4. Benefits and drawbacks of enrichment

Based on many studies, the enrichment of vegetable oils using safe, soft and natural additives has many advantages that are very well demonstrated especially in increasing the oxidative stability and antioxidant activity of oils (Baiano et al., 2009; Hernández-Hernández et al., 2019; Luo et al., 2019), extending the oil's shelf life (delaying the initiation of peroxidation) (Abenoza and Sánchez-Gimeno, 2021), promoting human health (Odeh et al., 2021; Rubió et al., 2012), replacing synthetic antioxidants (Fadda et al., 2022; Taghvaei and Jafari, 2015) and enhancing the bioavailability of phenolic compounds, which are recognized to be vulnerable to degradation throughout the gastrointestinal tract (Delgado-Adámez et al., 2014; Şahin et al., 2021; Suárez et al., 2011). Moreover, the study of Chougui *et al.* has shown that the addition of antioxidants from natural sources can present much better results than those given by synthetic additives (Chougui et al., 2015). Economically, the profit of the incorporation of vegetable oils and natural sources of natural compounds has good markets and can be marketed as aromatic and medicinal plants, which will subsequently offer several jobs. As well, the use of food waste (olive leaves, tomato pomace, etc.) as a source of natural antioxidants will help to recover and recycle these by-products (Rao et al., 2021).

However, more studies are needed that optimize the various critical parameters and simultaneously safeguard the extracts from thermal harm during the high-temperature treatments that occur during frying and cooking (Fadda et al., 2022).

The major shortcoming of enrichment is the absence of a viable analytical method to evaluate the bioactive agents responsible for the enhancement of oxidative stability. Notably, European legislation has authorized the inclusion of health claims on extra virgin olive oil (EVOO) labels ''*Olive oil polyphenols contribute to the protection of blood lipids against oxidative stress*'' (Sanmartin et al., 2019), such an initiation will be very necessary for the rest of the matrixes. Furthermore, a number of chemical compounds have been discovered and isolated from various plant parts, but there are no reports on the presence of harmful or antinutritional components in these natural extracts. Therefore, quantifying and identifying anti-nutritional factors can assist scientists and nutritionists in deciding how much extract to use (Blasi and Cossignani, 2020b). The absence of standards that specify the concentrations that should be added may cause toxicity and other issues.

5. Conclusion

The enrichment of oils is a good opportunity for oil producers as a kind of solution for the oxidation problem and a natural and safer new food to offer the consumers. Naturally derived antioxidants have been suggested as an alternative to synthetic supplements to protect edible oils from oxidative damage and thus improve their nutritional value. Apart from that, the combination of these two matrices (oils and antioxidant sources) will also have other economic benefits; the use of

Table 1
Major investigations of natural antioxidant-enriched oils, including information on the type of extract used, the amount of extract added to the oils, and some key findings.

Enriched oil	Plant	Method of enrichment	Operating parameters	Main Results	References
Refined soybean oil *(Glycine max* L.*)*	Olive leaves(*Olea europaea* L.)	Enrichment by ultrasound-assisted maceration	Extract preparation: 100 g of powder, methanol (80%), 12 h, agitation in dark (25 °C). After filtration, the solvent was recovered in a rotary evaporator. Oil enrichment parameters: 1000 ppm and 1500 ppm	PV after 21 days of incubation: 94.42 mEq O_2/kg for the control and just 60.20 mEq O_2/kg for the enriched oil at 1500 ppm. Para anisidine value of the control reached a maximum level of 31.77 ± 0.89, and just 20.10 for the enriched oil at 1500 ppm. Enriched oil with 1500 ppm showed greater efficiency.	(Zahran and Najafi, 2020)
Refined olive oil (*Olea europaea* L.)	Tomato peels (*Solanum lycopersicum* L.)	Enrichment with extracts	Extract preparation: 5% (w/v) of ground tomato peels in hexane, agitation at 200 rpm, 25 °C, overnight. After filtration, the solvent was recovered in a rotary evaporator (45 °C), Oil enrichment parameters: 2000, 1000, 500 and 250 μg/g	Enriched refined olive oil (250 μg/g and 500 μg/g) presented lower PV than the control sample at all time points. Enriched refined olive oil presented lower acidity compared to the control. Enriched oil at 250 had the lowest K232 values followed by enriched olive oils 500, 1000 and 2000, respectively.	(Kehili et al., 2018)
Corn oil (*Zea mays* L.)	Olive leaves and pomace (*Olea europaea* L.)	Enrichment with extracts (phenolic extracts)	Extract preparation:12% (w/v) of alperujo or leaves in ethanol, microwave extraction (a total of 500 g of alperujo and 300 g of leaves) at 400 W for10 min, centrifugation at 855 g for 5 min, the extract was concentrated in a rotary evaporator at 35 °C. Oil enrichment parameters: 200 and 400 μg/mL	Increase stability of all enriched oils (from 60 to 90%) as compared with their nonenriched oil. The PV of the control surpassed a value of 6 mEq O_2/kg, while enriched corn oil did not exceed 5 mEq O_2/kg. Increase in the induction time from 2.5 h for the control oil to more than 5 h for enriched oils at 120 °C.	(Sánchez de Medina et al., 2011)
Olive oil (*Olea europaea* L.)	Oregano (*Origanum vulgare* L.)	Enrichment with flavored oils	Flavored oils preparation: 1 kg of oregano was added to 5 L of extra virgin olive, stored at room temperature and in the dark, with daily shaking. Oil enrichment parameters: 10, 20 and 40 g/L	Enriched oils showed six times lower PV at all concentrations compared to the control. For the sensorial characteristics, the oils flavored at concentrations of 20 and 40 g/L were more highly regarded at the end of storage than the control and the oil flavored at the lowest concentration.	(Gambacorta et al., 2007)
Soybean oil *(Glycine max L.)*	Thyme (*Thymus vulgaris* L.)	Enrichment with extracts	Extract preparation:10 g of dried plant with ethanol (70%), in an oil bath for 10 h at 45 °C, after filtration, the solvent was removed to dryness in a rotary evaporator at 40 °C. Oil enrichment parameters: 0.01%, 0.03% and 0.07%	Enhancement of the oxidative stability; (From t_{ON} of 145.86 ± 0.47 for control to 156.86 ± 0.84 at 0.07% for unenriched oil).	(Kozłowska and Gruczyńska, 2018)
Hemp oil	Rosemary (*Rosmarinus officinalis* L.)	Enrichment with extracts (antioxidants extracts)	Extract preparation: 30 g of rosemary powder, Soxhlet extraction with 320 mL methanol, at 80 °C for 3 h. The solvent was removed using a vacuum oven at 50 mbar and 35 °C. The extract obtained was flushed with nitrogen gas Oil enrichment parameters: The extract was directly added to the oil (20 mg/100 g). The mixture was blended thoroughly for 2 min at 15 000 rpm at room temperature.	Enhancement of the oxidative stability; From a PV of 105.93 ± 0.12 mEq O_2/kg to 98.70 ± 0.50 mEq O_2/kg for enriched hemp oil. During storage, the colour of samples changed, lightness and yellowness were increased for all groups, but the intensity of the green colour was decreased for all groups.	(Moczkowska et al., 2020)
Soybean oil *(Glycine max L.)*	Rosemary (*Rosmarinus officinalis* L.)	Enrichment with Extracts (antioxidants extracts)	Commercial rosemary extract with a very high carnosic acid content of 70% was used.	Enhancement of the oxidative stability; From an induction period of 2.2 ± 0.22 h to 3.4 ± 0.18 h. From a PV of 23.72 ± 0.51 mEq O_2/kg 17.32 ± 0.15 mEq O_2/kg for enriched soybean oil.	(Yang et al., 2016)
Rice bran oil				Enhancement of the oxidative stability; From an induction period of 3.83 ± 0.07 to 6.22 ± 0.21 h at 120 °C. From a PV of 29.45 ± 0.61 mEq O_2/kg 19.00 ± 0.19 mEq O_2/kg for enriched rice bran oil.	
Cottonseed oil			Oil enrichment parameters: Extract is added directly to the oil (400 mg/kg)	Enhancement of the oxidative stability; From an induction period of 1.88 ± 0.2 for oil control to 3.35 ± 0.15 h for enriched olive oil at 120 °C. From a PV of 19.47 ± 0.18 mEq O_2/kg 16.53 ± 0.24 mEq O_2/kg for enriched cottonseed oil	
Olive oil (*Olea europaea* L.)	Laurel (*Laurus nobilis* L.)	Enrichment with essential oil	Essential oil preparation: Commercial *L. nobilis* essential oil obtained through hydro-distillation. Oil enrichment parameters: 0.01% (v/v)	Enhancement of the oxidative stability; From an acidity of 0.54 ± 0.00 for oil control to 0.50 ± 0.03 g/100 g for enriched olive oil. From a PV of 0.35 ± 0.02 for oil control to 0.20 ± 0.00 mEq O_2/kg for enriched olive oil.	(Taoudiat et al., 2018)

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Table 1 (*continued*)

Enriched oil	Plant	Method of enrichment	Operating parameters	Main Results	References
Soybean oil *(Glycine max* L.)	Rosemary (*Rosmarinus officinalis* L.)	Enrichment by maceration	Oil enrichment parameters: The rosemary leaves were dried and introduced at a rate of 6% (w/w) in glass bottles containing soybean oil. The mixture was kept in the darkness for 7 days. After maceration. The oil was filtered and stored at 4 °C until analysis.	50% of preservation of the total tocopherol content until 18 h. From an anisidine value of 90.32 for oil control to 38.51 for enriched olive oil.	(Saoudi et al., 2016)
Olive oil (*Olea europaea* L.)	Thyme(Thymus mastichina L.)	Enrichment by co-processing	Oil enrichment parameters: 0.5 kg of olives with five levels for thyme and water concentration (0.4% and 4.6% (w/w) and between 8.3% and 19.7%, respectively). Water and thyme were added at the beginning of the malaxation step, carried out at 28 − 30 °C for 30 min. A centrifugation was performed at 3500 rpm for 1 min, the water traces in the oil were removed with anhydrous sodium sulfate, which was removed by filtration through a cellulose filter	From an induction period of 10.3 h for oil control to 16.6 h for enriched oil, at 120 °C. From an acidity of 0.20 g/100 g for oil control 0.17 g/100 g for enriched oil.	(Peres et al., 2021)
Argan oil (*Argania spinosa* L.)	Saffron (*Crocus sativus* L.)	Enrichment by maceration	Plant preparation: Dried saffron stigmas were well cleaned, grounded, and sieved to a particle size of 125 μm. Oil enrichment parameters: The enrichment was carried out using three concentrations of saffron (0.2%, 0.5%, and 1%). The mixtures were maintained in agitation for 48 h	Improvement of argan oil oxidative stability; The oxidation parameters of argan oil significantly decreased after enrichment as compared to the control. The initial PV of unenriched argan oil was 2.3 mEq O_2/kg decreased to 2.17 in enriched argan oil (1%) The decrease in oxidation parameters was dose-dependent. Argan oil enrichment did not induce significant changes in fatty acid and sterol profiles. Sensory analysis results indicate that the enrichment process showed low rancidity but high scores of saffron smell, flavor, and color.	(Oubannin et al., 2023b)
Argan oil (*Argania spinosa* L.)	Thyme (*Thymus vulgaris* L.) leaves	Enrichment by co-processing	Oil enrichment parameters: In the malaxation step, 2.5 kg of argan kernels are mixed with thyme leaves at 3 different concentrations (2%, 5%, and 10%). At the end of extraction, the argan oil-thyme mixture is then filtered using a filtration system.	Improvement of argan oil oxidative stability; Increase of pigments, and antioxidant capacity but a decrease of PV. For fatty acids, there are no significant changes between enriched and unenriched argan oil. Sensory analysis is satisfactory with a reduction of rancidity in enriched argan oil depending on thyme leaves proportion.	(Oubannin et al., 2023a)
Sunflower oil	Spent coffee grounds	Enrichment with extract through maceration	Extract preparation: 50 g of sample was macerated in absolute ethanol (500 mL) for 24 h at ambient temperature (25 ± 2 °C). The extract was isolated using filtration, and the solvent was removed under reduced pressure. Oil enrichment parameters: Different concentrations (0.01%, 0.02%, and 0.03% by weight) were used	Results from the oxidative stability analyses (free fatty acids, peroxide value, conjugated dienes, and conjugated trienes) were in agreement with each other, suggesting the use of 0.03% of spent coffee grounds extract to enhance the oxidative stability of sunflower oil	(Bijla et al., 2024)
Olive oil (*Olea europaea* L.)	Olive oil Olive tree (*Olea europaea* L.)	Enrichment by maceration	Olive mill wastewater (OMWW) was sampled from a three phase extraction system. OMWW as a liquid residue of the olive oil industry, was subjected to total phenolics (TP) extraction. Three doses (100, 300 and 500 ppm of OMWW phenolic extract) were used to enrich olive oil.	The obtained results showed a significant oxidation delay in virgin olive oil enriched with phenolic extracts as demonstrated by low increases in PV, K232, and K270.	El Yamani et al. (2020a)
Olive (*Olea europaea* L.) oil	-Olive oil Olive tree (*Olea europaea* L.) -Carrot (*Daucus carota* L.)	Enrichment by maceration	Carotenoid (CE) and phenol (PE) extracts (from carrot and OMW, respectively) were used at 200 and 400 ppm to enrich olive oil ('Moroccan Picholine' cultivar) obtained from the super-pressure system.	Such natural additives (CE and PE) improve oil oxidative stability by delaying the oxidation process. PE was more effective as compared to CE. Moreover, the studied oil basic quality indices (PV, K232, and K270) decreased with increasing the additive concentration (200-400 ppm).	El Yamani et al. (2020b)
Refined olive (*Olea europaea* L.) oil	Olive leaves Olive pomace	Ultrasonication using lecithin as an emulsifier	Phenolic extracts from both biomasses namely olive leaves (OL) and exhausted olive pomace (EOP) were used to enrich refined olive oil (ROO). 2 g of dry extract/L of oil and diluted using refined olive oil to 0.5 and 1 g/L.	The oxidative stability index of the ROO enriched with OL at 2 g/L extract (70 h) was higher than that of a commercial ROO (46.8 h). Moreover, the oxidative stability index of the refined olive pomace oil (ROPO) enriched with EOP at 2 g/L	Vidal et al. (2022)

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Table 1 (*continued*)

Enriched oil	Plant	Method of enrichment	Operating parameters	Main Results	References
			The change in the content of antioxidant compounds and the antioxidant capacity of the oils were studied over 28 and 50 days of storage at 25, 35, and 45 °C	extract (44.1 h) was higher than that of a commercial ROPO (38.9 h)	
Soybean oil	Apple pomace extract	Enrichment with extracts (antioxidants extracts)	Apple pomace extract (APE) was prepared using ultrasonic-assisted extraction. APE was incorporated into soyabean oil at a concentration of 200 mg/L of oil	Total phenolic content (TPC) and total flavonoid content (TFC) of APE from ultrasound-assisted extraction (UAE) were found to be (5.58 ± 0.27 mg GAE/ g) and (5.42 ± 0.45 mg QE/g) respectively with antioxidant activity of 76% and (9.9 mM As acid/mg) as determined by DPPH and FRAP assay. Soyabean oil enriched with natural antioxidants (APE) shows higher thermal stability and quality regarding physicochemical indices and sensory evaluation as compared to control and TBHQ-enriched oils	Manzoor et al. (2022)
Soybean oil	Extracts from leaves of pomegranate (*Punica granatum* L.), orange (*Citrus sinensis* L.), and beetroot (*Beta vulgaris* L.)	Enrichment with extracts (antioxidants extracts)	Pomegranate/orange/beetroot leaf extract (POB) (1:1:1), as a natural antioxidant, in delaying the thermo-oxidative degradation of the soybean oil during deep-frying. The effect of the POB extract (200, 400, and 800 μg/mL) and the potentiated effect of POB-400 μg/mL on butylated hydroxytoluene (BHT) at 100 μg/mL versus that of BHT-200 μg/mL were investigated by the authors.	The extract at 200 and 400 μg/mL retarded the oxidation. Whereas POB-800 μg/mL and POB/BHT (400/100 μg/mL) inhibited the oil oxidation at the early stages, the peroxide value was < 10 meq O_2/kg oil. POB-800 μg/mL and POB/BHT reduced the total oxidation (TOTOX) by 55% and 64%, respectively, compared to the control (without additives), and decreased the formation of the malondialdehyde by nearly 80%	Abdo et al. (2023)

PV: peroxide value, TPC: total phenolic content, GAE: gallic acid equivalent, QE: quercetin equivalent, TBHQ: tertiary butyl hydroquinone, K232- K270: UV absorption coefficient at wavelength λ = 232 and 270 nm.

Fig. 6. Brief description of advantages and disadvantages of enrichment.

natural antioxidants recovered from aromatic and medicinal plants, food waste or sub-products of the agri-food industries is an innovative approach in the prospect of a circular economy. The study of the literature carried out in this review has brought to light an enormous article highlighting all the conventional and emerging techniques that allow the extraction of these antioxidants and subsequently the possible enrichment methods. In addition, there is a need to develop further enrichment methods, which will both protect the extracts from thermal harm during processing and ensure a controlled liberation of active substances. Advances in nanotechnology for the encapsulation of non-lipophilic antioxidants are moving in this direction. An examination of the doses of added antioxidants is also required to avoid safety issues, highlighting the degree of toxicity of each compound. This study emphasizes the further need for an integrated, interdisciplinary study on the preservation of edible oils with natural antioxidants.

CRediT authorship contribution statement

Laila Bijla: Validation, Investigation. **Mohamed Ibourki:** Software, Formal analysis. **Moussa Nid Ahmed:** Methodology, Investigation. **Devkota Krishna:** Writing – review & editing, Funding acquisition. **Youssef El kharrassi:** Methodology, Investigation. **Maggi Filippo:** Writing – review & editing, Validation. **Abdelhakim Bouyahya:** Writing – review & editing, Validation. **El Hassan Sakar:** Writing – review & editing, Validation, Supervision, Conceptualization. **Caprioli Giovanni:** Writing – review & editing, Validation, Supervision. **Samira Oubannin:** Writing – original draft, Methodology, Formal analysis. **Said Gharby:** Writing – review & editing, Validation, Supervision, Conceptualization.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

No data was used for the research described in the article.

Acknowledgments

This work was performed within the “Project Valorization of Medicinal and Aromatic Plants, 3rd Edition” and financially supported by the National Agency for Medicinal and Aromatic Plants, Taounate and CNRST, Morocco (VPMA3 2021–2024).

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