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ORIGINAL ARTICLE

Ziziphus lotus (L.) Lam. plant treatment by ultrasounds and microwaves to improve antioxidants yield and quality: An overview

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Abstract

The purpose of this review is to compile the literature published about different aspects of microwave-assisted extraction (MAE) use and ultrasound-assisted extraction (UAE) applied on jujube worldwide and to compare the results on the antioxidant activity obtained for each extraction method. As a result of the increased consumers demand for natural products, as well as for those of agro-food, nutraceutical, cosmetic industries, and green extraction techniques are nowadays trending to be potential alternatives that can improve antioxidant yield and its quality from an economical and environmental point of view by reducing time, energy, and solvent consumption. Ultrasounds and microwaves are widely used methods in the extraction of active principles due to their cavitation and dipolar rotation effect, respectively. These two techniques provide efficiency of extraction while minimizing the time and preserving the quality of the food matrix, overcoming the disadvantages of conventional techniques characterized by their consumption of large quantities of solvents and providing a sparse quantity of extraction. Jujube, a shrub with a high antioxidant potential, which can be affected by various extraction conditions can be the target of UAE and MAE to increase the antioxidant extraction yield. Exploiting the beneficial properties such as the antioxidant activity can lead to an industrialization process, replacing therefor synthetic antioxidants with natural compounds. These can also help in the development of new nutraceuticals and can be used, for instance, in agro-food industries as preservatives.

Keywords : Microwave-assisted extraction (MAE), ultrasound-assisted extraction (UAE), antioxidants, *Ziziphus lotus* (L.) Lam plant.

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1 Introduction

Ziziphus genus is a spiny shrub belonging to the family of Rhamnaceas, it is disseminated in tropical regions such as Asia, America, South of Europe, and the northern part of Africa as in Algeria [1]. There are several species of this genus (*Ziziphus vulgaris* Lam, *Ziziphus lotus* Lam, *Ziziphus Spina-christi* (L.) Wild, and *Ziziphus mauritiana* Lam), depending on the soil and climate [2, 3]. The fruits have been edible for millennia [2, 3]. In Algeria, *Z. lotus* (L.) Lam is very abundant [4]. Locally named (Sedra) and the fruit is called 'Nbag' [5]. Several botanists have described the morphological features of the jujube plant (*Ziziphus lotus L.*) which has a perianth pentamer, and the fruit is a drupe the size of a pea or an olive. The leaves are alternate, coriaceous, and accompanied each of two spines straight or crooked. In the most common species, the leaves are small (15 x 10 mm). It is a shrub or a tree frequent in the hot countries, it is cultivated for its fruits. Jujube is located in several regions of Algeria such as in Kabylie region and the southern part (Djelfa, Biskra, and M'sila), as well as in other Mediterranean countries such as Morocco and Tunisia [6].

For decades, researchers and industrial food companies have been increasingly interested in natural antioxidants, due to their properties in food preservation and their significant value for the prevention of diseases related to oxidative stress [7, 8]. The consumer's demand for a natural diet to counteract synthetic antioxidants is the main reason for this search [9-12]. In general, the first process of treatment of several plant materials is the extraction of their crude pigments [13]. Extraction of natural products can be done by various extraction techniques. For several years, conventional extraction methods, including maceration, solvent extraction, Soxhlet extraction, and alembic distillation, all basically utilized in food, medicine, and perfumery [14] have been used. However, many non-conventional methods including ultrasound-assisted extraction (UAE), supercritical fluid extraction (SFE), microwave-assisted extraction (MAE), and enzyme-assisted extraction (EAE) have been proposed due to their enhanced extraction efficiency and environmental friendliness [15-17].

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Based on the literature, there has been no review on the extraction techniques of bioactive compounds and antioxidant activity of *Z. lotus* (L.) Lam. Therefore, this work aims to provide a comprehensive review of green innovative extraction methods such as UAE and MAE in comparison with traditional methods having as target two important molecule types, polyphenols, and polysaccharides, from different parts of *Z. lotus* (L.) Lam. The effect will be evaluated on health-promoting human food and disease prevention, taking into account their antioxidant activity.

In recent years, the physiological function of foods including fruits, vegetables, and food components such as phytochemicals has received much attention [18, 19]. Possible correlations between the biologically active compounds and human health have generated interest in *in vitro* and *in vivo* studies about these biological activities. The major class of phytochemicals found in plants is related to phenolic compounds which contain a large variety of derivatives including simple flavonoids, tannins, phenols, phenylpropanoids, benzoic acid derivatives, lignans, and lignins [20, 21]. According to Croteau *et al.* [22], the classification of bioactive compounds from plant materials is divided into terpenes, alkaloids, and phenolic compounds. These categories contain a minimum of 8000 types of compounds approximately. Azmir *et al.* [23] suggested that shikimic acid and malonic acid are the pathways of the synthesis of phenolic compounds. While, alkaloids and terpenes come from mevalonic acid and non-mevalonate pathways, respectively. On the other hand, it has been found that polysaccharides represent a vital category as they exhibit numerous pharmacological and biological potential such as antioxidant, anti-inflammatory, and anticancer [24].

Z. lotus (L.) is known for its richness in primary metabolites mainly, protein 19.11%, carbohydrate 40.87%, and lipids 32.92% [25, 26]. For secondary metabolites (Table 1 and Figure 1), *Z. lotus* (L.) demonstrated the presence of many biologically active molecules [1], such as polyphenols (flavonoids and tannins), triterpenes, anthraquinones, alkaloids (cyclopeptides and isoquinolines), and saponosides, everything depends on parts of the vegetable matrix (leaf, root, fruit, and seeds) [27, 28]. The leaves are a source of flavonoids, tannins, alkaloids, and saponins [29-31]. The fruits contain flavonoids, tannins, and saponins [32]. Likewise, the roots are a source of flavonoids, tannins, and alkaloids [33]. Besides containing a higher amount of secondary metabolites, both seed and fruit reveal the presence of important minerals such as magnesium, calcium, and potassium [1]. These compounds are valued for their contribution to a healthy diet and also as ingredients for designing new foods [34, 35]. Among the most isolated compounds from *Z. lotus* (L.), the phenolic acids due to considerable amounts of caffeic acid, gallic acid can be mentioned [34, 35]. Flavonoids like rutin, epicatechin, taxifolin, and catechin can be extracted with organic solvent or mixtures in all parts of the jujube tree. Elsewhere, these compounds may well explain the biological activity, which can be used as control drugs in most pharmaceutical formulations [35]. Table 2 mentioned some isolated compounds from the *Ziziphus* genus and the part of the plant from where they were isolated.

Besides the nutritional composition, jujube has been a dietary food that appears in list A of the medicinal plants of French Pharmacopeia [36]. Several *in vitro* and *in vivo* studies on phytochemical and pharmacological effects have clearly revealed that *Z. lotus* (L.) contains some active molecules responsible for its beneficial effects depending on the part of the plant (root, leaf, seed, pulp, or fruit) mainly as antifungal, antibacterial, antiulcer, anti-inflammatory, antioxidant, and immunostimulant properties [1]. Based on the literature, flavonoid, polysaccharide, protein, and triterpenic acid are the main active molecules responsible for its biological effects. Both flavonoids and polysaccharides are found in both seed and pulp are known for exhibiting antioxidant, antimicrobial, and immunomodulatory properties [1]. However, the triterpenic acids, abundant in leaves, were proposed to be the main active ingredients for the effect on anti-inflammatory and anticancer activities [37]. While, proteins are found in seeds and pulps known for their functional properties such as emulsifying activity, emulsion stability, and water holding capacity [34]. However, the major organoleptic characteristics of plant-derived food (color, taste, ...) are represented in particular by phenolic compounds [38]. Additionally, they are known for their capacity to reduce oxidation reactions by controlling and quenching the reactive oxygen species (ROS) including peroxides, hydroxyl radicals, superoxide, and nitrous oxide that damages food and can be linked to various diseases [39]. The antioxidant activity of *Z. lotus* (L.) extracts is well documented [16, 40-47]. Many methods have been used to evaluate the antioxidant effect of extracts; the most commonly developed method is 2,2-diphenyl-1-picrylhydrazyl (DPPH) which is based on the inhibitory action of vegetable extracts on the free radical activity of ROS. This method is reproducible and time efficient, other methods are also used 2,2-azinobis (3-ethyl-benzothiazoline-6-sulfonic acid) (ABTS), ferric reducing antioxidant power (FRAP), and trolox equivalent antioxidant capacity (TEAC) [48]. According to Bakhtaoui *et al.* [43], *Z. lotus* (L.) fruits showed stronger scavenging free radicals effect when compared to other morphological parts (leaves, root, and stem) [1]. This is influenced by several factors, including their concentration, temperature, type of solvent, ratio, and frequency, as well as the presence of prooxidants and synergists [49]. In parallel to the conventional methods, green extraction processes such as ultrasound and microwave methods on *Ziziphus* species have found to give different antioxidant effects, these differences are well discussed in the next section.

2 Conventional extraction methods of antioxidant from jujube fruit

Several studies have shown the large choice of traditional extraction methods of antioxidants compounds from plant materials, such as maceration, hydro-distillation, and Soxhlet extraction [23]. Generally, this is based on the application of temperature treatment and the use of different solvents depending on the compound to remove or to improve extraction. The most common processes used for the extraction of compounds from plants are either physical or chemical [50]. In addition, the conventional extraction method allows the transfer of heat from the outside to the inside of the sample through the heating medium. Maceration is very used in homemade preparation of tonic for a long time, which is inexpensive, based on the mixture of solvent with the surface area to get bioactive compounds. Hydro-distillation and Soxhlet extraction techniques are generally used for the extraction of essential oils. They are used for bioactive compounds, thus allowing automatic separation of these compounds from water due to the flows from the condenser to a separator of the condensed mixture [51].

Many studies have been reporting on the extraction of secondary metabolites from the *Z. lotus* (L.) plant using conventional methods. Indeed, Borgi *et al.* [27] reported the extraction of antioxidants in root barks from *Z. lotus* (L.) with different solvents. Borgi *et al.* [52] used Soxhlet for extraction of saponin and flavonoid fractions from the leaves and root bark of *Z. lotus* (L.). Borgi *et al.* [4] extracted bioactive compounds from the leaves and root barks of *Z. lotus* (L.) by maceration method. On the other hand, the effect of *Z. lotus* (L.) root barks extracts' on antiulcerogenic activity using Soxhlet extractor as demonstrated

Table 1: Chemical composition of *Ziziphus lotus* (L.) in different part of jujube

Fraction	Fruits	Pulp & peels	Seeds	Leaves	Root bark	References
Moisture content (%)	-	12.27	6.05	-	9.11	
Carbohydrates (%)	-	65.90	40.87	8720 (mg/100 g)	8.71	
Crude protein (%)	-	3.80	19.11	-	3.18	
Crude fat (%)	-	1.32	-	-	-	
Crudefibe (%)	-	8.41	-	-	47.90	
Ash (%)	-	3.28	1.05	-	2.69	
Pectin (%)	-	3.78	-	-	-	
Vitamin C	5.67	190.65	31.24-170.84	63.40	47.20	
Calorific values Kj/g	-	16.341	-	-	-	1,5, 25, 40, 101
Oleic acid (%)	-	88.12	61.93	-	-	
Elaidic acid (%)	-	7.88	-	-	-	
Linolenic acid (%)	-	-	-	9.15	-	
Saponins (mg/100 g)	-	-	-	340	219	
Polyphenols (mg/100 g)	297–4078.2	325	14.68	664	2009	
Total flavonoids (mg/100 g)	122	173	-	133-199	120	
Total tannins (mg/100 g)	33	929	-	39	156 (Proanthocyanidins)	

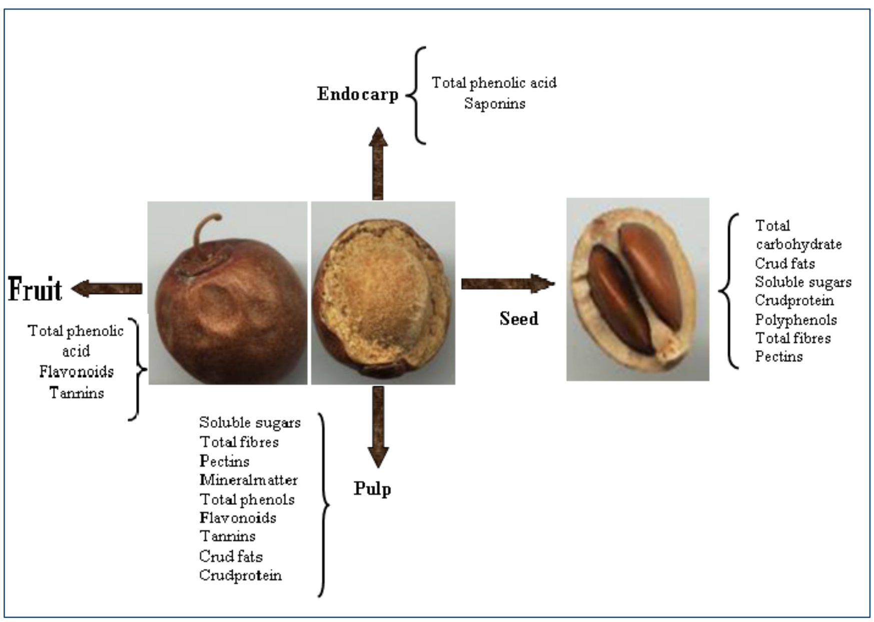

Figure 1: Anatomy of *a Z. lotus* (L.) Lam. fruit

Table 2: Some classes of polyphenols isolated from *Ziziphus* species

Bioactive compounds	*Ziziphus* species	Fruit	Pulp & peel	Seed	Leave	Stem bark	Branche	References
			Phenolic acids					
Gallic acid	*Z. lotus* *Z. jujuba*	+	-	+	-	-	-	102, 103
p-Hydroxybenzoic acid	*Z. jujuba*	-	-	-	+	-	-	29
Syringic acid	*Z. jujuba* *Z. lotus*	+	-	-	+	-	-	29, 102
p-coumaric	*Z. jujuba* *Z. lotus*	+	-	+	-	-	-	29, 102
Ferulic acid	*Z. jujuba* *Z. lotus* *Z. jujuba*	+	-	+	+	-	-	29, 102, 103
Caffeic acid	*Z. jujuba* *Z. jujuba*	+	-	+	+	-	-	29, 103
			Acylatedquinic acid derivatives					
Quinic acid	*Z. lotus*	+	+	-	-	-	-	
3-O-caffeoylquinic acid (cholorogenic acid)	*Z. jujuba* *Z. jujuba*	+	+	+	+	-	-	29, 103
Flavonoid aglycones								
Luteolin	*Z. lotus* *Z. jujuba*	+	-	-	-	-	-	102
Quercetin	*Z. jujuba* *Z. lotus* *Z. jujube* *Z. mauritania* *Z. mistol*	+	+	+	+	-	-	29, 102-109
Catechin	*Z. jujuba* *Z. jujuba* *Z. lotus* *Z. jujube* *Z. mauritania* *Z. joazeiro*	+	+	+	-	-	-	29, 102-105, 107, 108
Procyanidin trimer	*Z. jujube*	+	-	-	-	-	-	107
			Flavonoid glycosides					
Kaempferol-3-O-glucoside	*Z. lotus* *Z. jujuba*	+	+	-	-	-	-	107
Kaempferol-3-O-robinobioside	*Z. lotus* *Z. jujuba*	+	+	-	-	-	-	107
Vitexin	*Z. jujuba*	-	+	-	-	-	-	109
Quercetin -3-O-glucoside	*Z. mauritania* *Z. lotus*	+	+	-	-	-	-	110
Quercetin-3-O-robinobioside	*Z. jujuba* *Z. mauritania*	+	-	-	-	-	-	107, 110
Quercetin-3-O-rutinoside	*Z. jujuba* *Z. jujuba* *Z. mistol* *Z. mauritania* *Z. lotus*	+	+	+	+	-	-	29, 103, 104, 106, 110
Quercetin-3-O-rutinoside-7-O-pentoside	*Z. jujuba*	+	-	-	-	-	-	107

by Wahida *et al.* [53]. Similarly, Naili *et al.* [54] studied the antimicrobial and antioxidant activities of *Z. lotus* (L.) plants growing in the south part of Libya, which showed high content of polyphenols and alkaloids using different solvents, and considered them a source of phenolic antioxidants and antimicrobials. Benammar *et al.* [55] used the antioxidant effect of *Z. lotus* (L.) root, leaf, stem, fruit pulp, and seed extracted with decoction and the role of different crude extracts plant on human T-lymphocyte proliferation, they have found that the seed extract showed the most potent immunosuppressive effects on T cell proliferation. In addition, Bakhtaoui *et al.* [42] found that the use of bioactive compounds extracted from *Z. lotus* (L.) fruit of Morocco by Soxhlet using methanol enhanced the anti-helicobacter pylori, gastro-protective, and antioxidant properties. More recently, Marmouzi *et al.* [56] studied the effect of phenolic compounds extracted from *Z. lotus* (L.) fruit and leave by infusion, it showed a very important antioxidant, antidiabetic, and derma protective potential. Furthermore, the identification of these compounds using HPLC-DAD-QTOF-MS showed a high yield in gallic acid with 2715 mg/kg in the leaves and 15000 mg/kg in fruits. In another study conducted by Ghalem *et al.* [44] the antioxidant activity of *Z. lotus* (L.) root from Algeria extract using the Soxhlet method with the use of beta-carotene bleaching test confirmed the antioxidant capacity of these extracts.

Other jujube species components have been extracted using conventional techniques. Indeed, Soxhlet apparatus has been used for extraction of total phenols and flavonoids content from Omani *Z. jujuba* Mill fruits and leaves as well as the antioxidant activity and polarities of jujube crude extracts have been also evaluated [57]. The effect of different extraction solvents using Soxhlet on yield of active metabolites extracted from *Z. jujuba* Mill. leaves was studied by Al-Saeedi *et al.* [58] which confirmed their higher extraction yield and their antimicrobial activities, these results should be considered in pharmacological studies. Furthermore, the phenolic compounds from Apple Kul pulp (*Z. mauritiana* Lam.) were extracted by a Soxhlet extractor using the methanolic as an extraction solvent for 6 hours which was found to be a rich source of polyphenols (52.19 ± 2.38 mg gallic acid equivalents/100 g), tannins (50.20 ± 3.61 mg tannic acid equivalents/100 g), and flavonoids (13.19 ±1.31 mg catechin equivalents/100 g) [59]. Moreover, the *Z. jujuba* Mill. seeds were studied using the conventional method with ethanol/water extracts and analyzed for their bioactive phytochemicals using chromatographic techniques which revealed the presence of many bioactive compounds in which 20 components were identified [60]. On the other hand, Abdula *et al.* [32] used the same method for the extraction of polyphenols from jujube leaves. More recently, Shams *et al.* [61] demonstrated that *Z. jujuba var vulgaris* fruit extracted with maceration method at different extraction conditions give the optimum phytochemical compounds contents using ethanol concentration, pH, extraction time, and extraction temperature of 60%, 3, 180 min, 25°C, respectively. The obtained values were 164.51 mg GAE/g DW, 52.94 mg cy-3-glu 100 g-1 DW, and 137.12 mg LAA 100 g-1 DW for total phenolic, total monomeric anthocyanin, and vitamin C contents, respectively.

Based on what has been cited previously, the conventional extraction methods are characterized by high volumes of solvents and longer extraction time, with a low extraction yield of bioactive compounds. To overcome the limitations of these types of methods, non-conventional extraction methods have been introduced, like microwave and ultrasound -assisted extraction.

3 Ultrasounds and microwaves to enhance bioactive compounds yield and quality

3.1 Application of ultrasound-assisted extraction (UAE) in food research

Ultrasound is a mechanical wave, with frequencies higher than the capacity of the ear to catch [62], which can propagate in material and cause cycles of expansion and compression in the environment. This can create bubbles that surround themselves in a liquid at high speed, called cavitation phenomenon [63]. Ultrasound can also be broadly classified as low-intensity sonication (<1 W/cm^2) and high-intensity sonication (10–1000 W/cm^2) [64]. According to Hielscher *et al.* [65], ultrasound shows a very important expansion in medicine because of its effectiveness. Thus, in medical imaging, ultrasound has been much more interesting compared to other imaging methods [62]. It provides access to quantities such as blood flow mapping as well as their positive impact on human health which is justified in the place of ultrasound in medical diagnostic and therapeutic applications [64].

There are two types of ultrasonic equipment used in laboratories, one is called ultrasound probe; which confirmed a direct contact with the sample to be analyzed, such as the extraction of bioactive compounds from plants in order to accelerate the maceration. Unlike the ultrasonic probe, the second ultrasonic bath is used for homogenization, dispersion, degassing, and cleaning, generally based on the indirect contact and used for enrichment [66, 67].

3.1.1 Cavitation mechanism

Ultrasounds are mainly based on heating. It is the phenomenon of ultrasonic cavitation which is due to the cycles of compression and decompression of water molecules. The mechanical effect of ultrasound at high acoustic pressure forms cavitation bubbles as shown in Figure 2 and allows the acceleration and release of bioactive principles of the plant, via the disruption of cell walls and the intensification of mass transfer [68]. When the medium is introduced under ultrasonic waves, cycles of compressions and rarefactions are formed following the longitudinal displacement of the waves in the particles of the medium. Then, a formation of gas bubbles will take place in these zones of variable pressure while changing their size during the process, this is the cavitation phenomenon, these bubbles will subsequently reach a critical size over a period of a few cycles. Thus, allowing them to collapse violently while releasing large amounts of energy [68]. The size of the cavitation bubble is dependent on the frequency of ultrasound. This cavity can absorb ultrasonic energy more efficiently by expanding rapidly until it can no longer absorb energy when liquid rushes in and the cavity implodes. The cavity containing gas and vapors allows generating enormous local temperatures and pressures creating an environment for a chemical reaction [69].

Due to the beneficial effects of ultrasound in the extraction of bioactive components from plants, it improves the extraction time, by reducing it and giving higher yields [14, 70-71]. Ultrasound is used for plant dehydration [72], drying [65, 73], emulsification, and extraction of bioactive substances [74, 75.] For years, many researchers have demonstrated the importance of ultrasound in the development of agro-food industries [13, 67.] On the economic front, the use of ultrasonic treatment has valuable advantages based on the extraction of materials while preserving the quality of the plant's matrix [70.] In addition, Dalvi-isfahan *et al.* [76] showed that the control of ice nucleation by ultrasound waves is a much better innovative alternative preservation technique in lieu of the freezing foodstuffs, a technique that can alter the nutritional and hygienic quality of the food.

3.1.2 Ultrasound-assisted extraction (UAE) of jujube antioxidants

The application of UAE has been widely used for the extraction of plant materials with high-added value. It seems to be an effective extraction method of antioxidants for jujube fruit. A number of authors have evaluated and optimized ultrasound extraction conditions. Boulanouar *et al.* [45] showed that the extraction efficiency of phenolic compounds from *Z. lotus* hydro-alcoholic extracts under sonication was for 81.44 ± 5.64 mg/g, dry weight which exhibited a good antioxidant effect against ABTS, chelating, DPPH, inhibiting lipoxygenase, reducing superoxide radicals, and ORAC assays with a highest EC_{50} value of 110.64 ± 39.71 µmol TE g^{-1} (d.w) by ORAC assay. Additionally, Hammi *et al.* [43] studied the effect of the independent variables under ultrasound extraction, including ethanol concentration (0-100%), sonication time (5-45 min), ratio of solvent to solid (10-70 mL/g), and sonication temperature varying from ambient temperature to 65°C. The authors reported that the use of ultrasound with high intensity improves significantly the phenolic extraction yield from *Z. lotus* pulp and peel. The results showed that increasing the amplitude and the extraction time leads to a higher extraction yield using a lower temperature. The optimum extraction conditions were found using ethanol concentration of 50%, ratio of solvent to solid of 67 g/mL at 25 min and 63ºC. Under these conditions, the extraction yield was for 40.782 mg GAE/g DM with significant antioxidant properties mainly by DPPH (IC_{50} of 0.289 mg/mL) and TAA (IC_{50} of 75.981 mg GAE/g DM) in a shorter extraction time. Moreover, the effects of UAE (20 kHz, 80-95°C, 1-4 h, 20-40 g/mL) on polysaccharide recovery with its antioxidant activities from *Z. lotus* pulp and peel were evaluated by Mkadmini-Hammi *et al* .[77]. The authors reported that the direct UAE process led to the highest yield of polysaccharides (18.88%) and six polysaccharides with an average molecular weight of 2720 kDa were identified (arabinose, rhamnose, glucose, fructose, galactose, and xylose). However, at the optimal conditions of 3h 15min, 91.2°C and water to solid ratio of 39 mL/g, the polysaccharide extract showed a significant DPPH (IC_{50} of 0.518 mg/ml), FRAP (614.39 µmol/L), and anti-lipid peroxidation effects at 50% of 2.417 mg/mL. Similarly, Adeli *et al.* [41] investigated the effect of UAE on yield of water-soluble polysaccharide extracted from *Z. lotus* fruit while obtaining a maximum yield of 13.398 ± 0.019% under optimized conditions as follows: 88.77 W, 29.96 min, 77.73°C and water to raw material ratio 24.44 mL/g with highest antioxidant activities for DPPH (78%) and hydroxyl radical-scavenging (91%).

There are few reports on the extraction of bioactive compounds from the *Z. lotus* plant using UAE. While several reports were found from other *Ziziphus* species demonstrating the good use of innovative extraction techniques as shown in Table 3. Qu *et al.* [78] studied the application of UAE in polysaccharide extraction from *Z. jujuba* Mill. using different solvents and results showed that UAE produces a higher yield of extraction with good antioxidants activity against OH scavenging assay with 68%. Furthermore, UAE enhanced the extraction of polysaccharides from *Z. jujuba cv. Muzao* (ZMP) by UAE using both 29% ethanol and 15% $(NH_4)2SO_4$, the authors used jujube powder with liquid-to-solid ratio (mL/g) of 30 under a power of 70 W for 38 min at 48°C. Following these conditions, the experimental extraction yield of ZMP was 8.18% with a high antioxidant potential compared to DPPH (29.68%) and ABTS radical scavenging (21.45%) at a concentration of 2.5 mg /mL [79]. In another study conducted by Lin *et al.* [80] that utilized UAE for the recovery of polysaccharides from *Z. jujuba* Mill. var. *spinosa* seeds showed a higher yield of polysaccharides (1.05 ± 0.08%) at 52.5 °C, 21.2 min, 134.9W, and ratio of liquid to solid 26.3 mL/g as applied conditions. These results are significantly equated to 0.93 ± 0.14% of 6 hours using the heating water extraction method. The seeds extract scavenged more rates of ABTS (33.41%), superoxide anion (41.72%), and hydroxyl radicals (69.78%), while its chelating capacity of Ferrous ion was up to 42.70%. Similarly, Zemouri-Alioui *et al.* [81] extracted phenolic compounds using UAE on jujube leaves and evaluated their antioxidant activity. RSM study has been used under some extraction conditions including solvent concentration (25-100%), solid/solvent ratio (1/50-1/300), extraction time (1-15 min), and ultrasound intensity (25-100%). The authors demonstrated the positive use of UAE providing 6 g GAE/100g for total phenolic content, under methanol 60%, 75% intensity, time of 10 min and ratio of 1/200. The extract showed a significant correlation with the antioxidant activities against DPPH (3.886 g ascorbic acid equivalents/100g) and FRAPS (2.587 g ascorbic acid equivalents/100g). More recently, in our previous work, we found that phenolic compounds extracted from jujube seeds using UAE under a RSM study have shown a higher yield (2383.10 ± 0.87 mg GAE/100g) at applied conditions of 29.01 °C, 15.94 min, ethanol 50.16%, and liquid to solid ratio of 34.10:1 mL/g. This yield was significantly correlated with the antioxidant activities tested against DPPH (EC_{50} of 0.39 µg/mL) and FRAP (1670.42 ± 6.5 mg/100 g) [82].

The extraction of antioxidants mainly polyphenols and polysaccharides from different parts of the jujube plant by conventional and non-conventional methods have been the subject of several studies. All these results are indicated in Table 3.

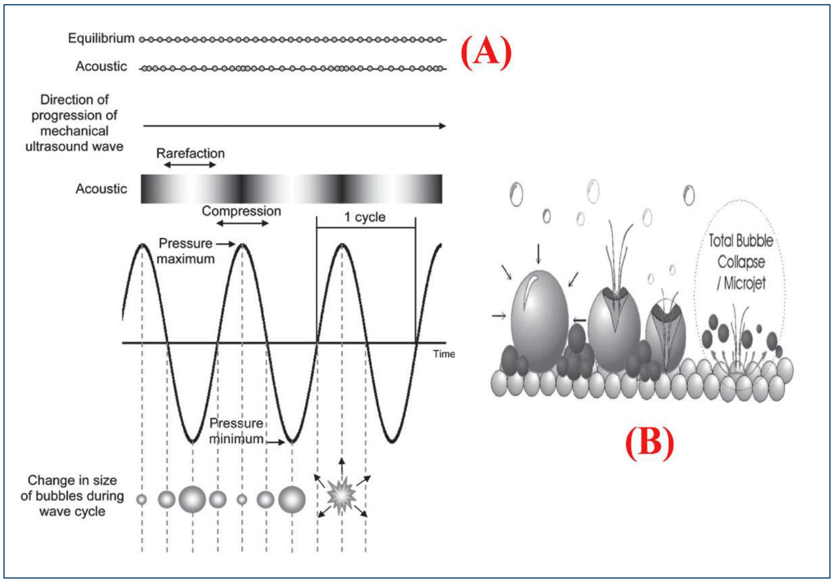

Figure 2: (A) Development and collapse of cavitation bubbles, and (B) schematic depicting classically thought bubble collapse at the solid surface [112]

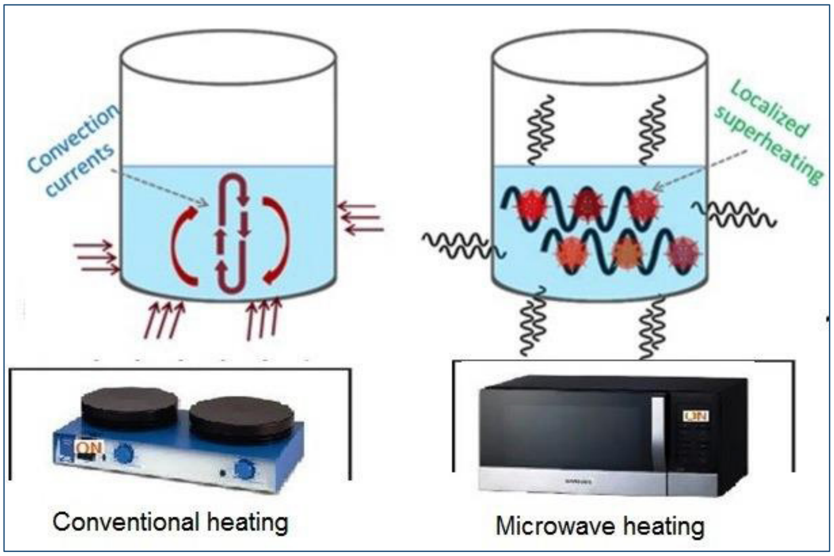

Figure 3: Conventional and microwave heating mechanisms

3.2 Application of microwave assisted extraction (MAE) in food research

The use of microwaves began to appear in the 1950s, the literature reveals that the first microwave oven was introduced in 1955 by Tappan. While during the 1970s and 1980s the widespread use of domestic microwave ovens occurred [83]. Its first application was in chemical synthesis and it was published in 1986 [84], it was used in several domains, such as food processing and drying on industrial process and domestic purposes [85]. Microwaves are a non-ionizing electromagnetic energy [86], with frequencies ranging from 0.3 to 300 GHz. They can be transmitted, absorbed, and reflected, thanks to the laws of optics. Domestic microwave units generally operate at a frequency of 2450 MHz in comparison to industrial applications (915 MHz)[87].

3.2.1 Theory of MAE

Microwave has the capacity to convert a part of plant materials absorbed by electromagnetic energy to heat energy. Microwave heating of plant materials is mainly characterized by the rotation dipole and the ionic conduction [88]. The mechanism of dipole rotation is based on the principle that any molecule under microwave irradiation which generates heat must have a dipole

Table 3: The application of green techniques in the extraction of antioxidants from Zizyphus plant

Extraction method	Category	Vital Products	Matrix	Conditions	Model	Yield	Antioxidant effect	Ref.
UAE	Polysaccharides	Fruit	*Z. jujuba Mill*	15 min 40 °C 80 W	OH scavenging assay.	ND	The extract revealed 68% inhibition. OH scavenging	78
	Polyphenols	Fruit	*Z. lotus*	6 min 20 kHz 1g/ 7 mL of a hydro-alcoholic solution (70%)	In vitro ABTS, ORAC, DPPH, chelating, superoxide radicals and inhibiting lipoxygenase were assayed.	81.44 ± 5.64mg/g, dry weight	*Zl* extract exhibited IC_{50} values of 0.049 ± 0.002, 1.406 ± 0.023, 0.042 ± 0.018, 0.138 ± 0.005, 0.001 ± 0.006, 0.129 ± 0.011 mg/ml and 110.64 ± 39.71 µmol TE g^{-1} (d.w.) against ABTS, chelating, DPPH, inhibiting lipoxygenase, reducing, superoxide radicals and ORAC assays, respectively	45
	Polyphenols	Pulp and peel	*Z. lotus*	Ethanol 50% 25 min 63°C 67 mL/g	*In vitro* DPPH and TAA assays	40.782 mg gallic acid equivalents/g dry matter	The extract revealed IC_{50} values of 0.289 mg/mL and 75.981 mg GAE/g DM for DPPH and TAA tests, respectively	43
	Polysaccharides	Pulp and peel	*Z. lotus*	3h 15min 91.2°C 39 mL/g	DPPH scavenging ability, reducing power and anti-lipid peroxidation assays	18.88%	The *Zl* extract revealed potent IC_{50}values of 0.518 mg/ml), 614.39 µmol/L and 2.417 mg/mL at 50% for DPPH, FRAP and anti-lipid peroxidation tests	77
	Polysaccharides	Fruit	*Z. lotus*	88.77 W 29.96 min 77.73°C 24.44 mL/g	DPPH and hydroxyl radical-scavenging activities	13.39±0.019%	The polysaccharide extract revealed an antioxidant effect of 78 and 91% for DPPH and hydroxyl radical-scavenging tests, respectively	41
	Polyphenols	Leave	*Z. jujuba Mill*	Methanol 60% 75% intensity 10 min 1/200 mg/mL	FRAP and DPPH assays	6 g GAE/100g	Antioxidant activities against DPPH and FRAP were for 3.886 and 2.587 g ascorbic acid equivalents/100g, respectively	81
	Polysaccharides	Fruit	*Z. jujube cv. Muzao*	29% of ethanol 15% $(NH_4)2SO_4$ 30 mL/g 70 W 38 min 48°C	ABTS and DPPH assays	8.18%	The extract revealed to have a moderate antioxidant activity for both DPPH (29.68%) and ABTS (21.45%)	79
	Polysaccharides	Seeds	*Z. jujuba Mill* var. spinosa	52.5 °C 21.2 min 134.9W 26.3 mL/g	ABTS, superoxide anion, hydroxyl radicals and chelating capacity of Ferrous ion	1.05 ± 0.08%	The extract showed an ABTS, superoxide anion, hydroxyl radicals and chelating capacity of ferrous ion of 33.41, 41.72, 69.78 and up to 42.70%, respectively	80
	Polyphenols	Seeds	*Z. lotus*	Ethanol 50.16% 29.01 °C 34.10:1 mL/g 15.94 min	DPPH and FRAP tests	2383.10 ± 0.87 mg GAE/100g	*Zl* extract exhibited EC_{50} of 0.39 µg/mL for DPPH test and 1670.42 ± 6.5 mg/100 g by FRAP	82
MAE	Polysaccharides	Pulp and peel	*Z. lotus*	600 W 40 min 26.69 mL/g	ABTS, DPPH and FRAP tests	13.98 ± 1.55%	The *Zl* extract revealed a good scavenging capacity against ABTS. + (70.45%), DPPH*. (66.02%), and FRAP (A = 0.63)	16
	Polysaccharides	Fruit	*Z. jujuba Mill*	4 min 300 W	OH scavenging assay.	ND	The extract revealed 52% inhibition. OH scavenging test	78
	Polysaccharides	Peels	*Z. jujuba Mill*	400W 75°C 60 min 30 g water/g powdered jujube	FRAP and DPPH assays	9.02%	Jujube extract showed a scavenger effect against DPPH arround 65 to 75% and FRAP (A= 0.63)	99

moment where the molecule is charged (+ or -) like water (H_2O) which according to the polarity of the field, it tries to align with the electromagnetic field by rotary motion (Figure 3). This last causes friction heat [86, 89].

When an electromagnetic field is applied, ionic compounds move at an accelerated rate producing ionic polarization. As the movement of the ions increases, kinetic energy is converted quickly into the thermal energy of the solution [86]. The ability of given material to interact with an electric field by converting energy into heat depends largely on its dielectric properties. Dielectric constant and dielectric loss factor are the parts of dielectric properties. Dielectric constant (ε'), meaning the ability to store electrical energy, while dielectric loss (ε"), describes the material's ability to convert electrical energy into heat [90, 91], according to the following equation:

$$\varepsilon' = \varepsilon'' \, tan \, \delta Eq(1)$$

The dissipation factor (tan δ) is an indicator of the efficiency of the dissipation or absorption of electrical energy in the form of heat by microwave which is described by:

$$p_v = kf\varepsilon' E2 \, tan \, \delta Eq(2)$$

Where:

P = absorbed microwave power (W/cm3)
f = microwave frequency (GHz)
ε"= dielectric loss factor of material
k = a constant
E = electric field intensity for a given volume (volts/cm).

Numerous studies have been published by several authors for microwave applications acquired and tested in the food industry, as shown by Smith *et al.* [93], including drying, moisture determinations, safety guidelines, economy, automation, and robotics Routray *et al.* [86] showed that several factors can affect microwaves extraction of bioactive compounds (gives an example of flavonoids), as suggested by the authors, both polar and non-polar solvents can be used for extraction with respecting substances nature of extraction in each used solvent. On the other hand, the power level, temperature, and time of extraction may affect positively the extraction process and increases the solubility due to the interaction molecules with the opening cell-matrix and the liberation of bioactive compounds. More recently, Chemat *et al.* [13] demonstrated the extraction mechanism of MAE which was supposed to involve several stages that are based on the effect of microwave radiation, which increases the temperature and the pressure of the microwaves during the extraction, these will allow the diffusion of the solvent in the sample matrix and will thus release the active ingredients of the this last. Due to these effects, the advantage of using microwaves is very important because it not only guarantees an efficient extraction allowing the recovery of a maximum of bioactive compounds more quickly compared to the conventional extraction processes but also considered as green technology by reason of the less use of organic solvents [94].

3.2.2 Microwave-assisted extraction (MAE) of jujube antioxidants

The conventional extraction methods, compared to MAE, occurs from the outside to the inside of the substrate, and the heat is not transferred as the same [86]. During this extraction, there is stable conduction due to the concentration of solute in interaction with the solid varies, and this according to the solvent penetrated the matrix, the solubilization of the components and the migration solute from the outside to the solution as well as. The extraction efficiency is not a linear function of extraction time [95].

Several studies demonstrated the efficiency of the MAE process compared to the UAE and the feasibility of using the MAE process at an industrial scale, and it has been very used in green extraction of bioactive compounds from plants and industrial by-products [96]. A number of authors have evaluated and optimized conditions of extraction of bioactive contents using MAE, Dahmoune *et al.* [97] used MAE of *Citrus limon* (L.) Burm. f. peels and compared to UAE and CSE for the recovery of total phenolic compounds. The optimized result for MAE was 48%, 28:1 mL/g, 123s, and 400 W for ethanol as extraction solvent, solvent: solid ratio, irradiation time, and power, respectively. Results show that the maximum predicted TPC recoveries under the optimized conditions for MAE was 15.74 mg GAE/g model. In comparison to UAE and CSE, MAE showed better results in terms of yield and antioxidant activities against DPPH and reducing power. Dahmoune *et al.* [98] used the MAE of total phenolic compounds from the leaves of *Pistacia lentiscus* L. which generates better extraction yield (185.69 ± 18.35 mg GAE/gdw) with higher antioxidant activities in comparison to UAE and CSE with optimal conditions as 46% ethanol, extraction time 60 s, potency density 17.86 W/mL, and liquid/solid ratio 28:1. This is due to the rapid energy-saving heating rates with deep penetration of organic solvent in raw material, leadings to very short extraction times as shown in Figure 3.

More recently, several studies have been carried out for other species of jujube. The influence of microwave heat treatment on jujube plant in terms of storability and quality has been studied. The response surface methodology (RSM) was used to evaluate and optimize MAE in polysaccharide recovery from *Z. jujuba* Mill. peels. For this purpose, jujube fruits were treated by MAE until reaching a temperature level of 45-85°C, microwave powers (250-450 W), extraction time (30-70 min), and solvent to solid ratio (10-70 mL/g). The authors reported that the use of microwave with high intensity improves significantly the yield of polysaccharides of *Z. jujuba* Mill. fruit (9.02% of polysaccharide) at 400 W, 75°C, 60 min, using 30 g water/g powdered jujube with a good antioxidant effect against DPPH (around 65 to 75%) and FRAP (A= 0.63) [99].

For instance, there is no report about the use of MAE to enhance the extraction of phenolic compounds from the *Z. lotus* (L.) species. However, our previous work focused on polysaccharide extract from *Z. lotus* (L.) pulp and peel using MAE under RSM study. The effect of the independent variables, including microwave power (200-600 W), irradiation time (20-40 min), and a liquid/solid ratio (20-40 mL/g) have shown that MAE improved significantly the polysaccharide extraction yield from *Z. lotus* (L.) pulp and peel at 600 W, 40 min, and 26.69 mL/g [16]. Under these conditions, the *Z. lotus* extract exhibited a good antioxidant capacity against $ABTS^+$ (70.45%), $DPPH^·$ (66%), and FRAP (A of 0.63) [16].

3.3 Comparison between conventional methods with the green extraction processes

There are several conventional extraction techniques such as maceration, water distillation, steam distillation, combined water and steam distillation, and enfleurage. Comparing these techniques to newer extraction methods, they consist of low-cost solvent extraction that uses heat and/or agitation. Generally, these conventional techniques make it possible to increase the solubility of the target compounds while improving the mass transfer during the extraction stage, several factors are important in the solvent system, which concern volatility, selectivity, density, toxicity, reactivity, miscibility with aqueous media, viscosity, and purity. On the other hand, these conventional methods result in low selectivity and high consumption of organic solvents, in addition to contamination and loss of analytes [40]. However, green technologies such as ultrasound and microwaves are increasingly studied, the latter combines both ecological and economic aspects in order to contribute to the process of sustainable development of the world population by seeking new (modern) means. Extract valuable compounds from plants, herbs, algae, and other organizations (e.g., bioactive compounds such as essential oil, antioxidants, oil, and dyes). According to several researchers, this is the best strategy for 2030 allowing the greatest reduction in waste while valuing the by-products rejected by reusing them in order to reduce energy, especially since the majority of the extractions used consume enormous quantities of energy and solvents with a high environmental impact. So-called green techniques are a better alternative to conventional methods for good respect for the environment and effective sustainability [13, 66]. Furthermore, Chemat *et al.* [67] has developed some principles of innovative extraction, in particular the reduction of large volumes of solvents, energy consumption, extraction time, production of by-products instead of waste with high added value and sourcing reasoned the recovery of a safe natural extract. In addition, on an industrial scale, these principles of green extraction methods have been able to improve the sustainability of production compared to conventional methods.

4 Conclusions

This brief review paper described the uses of emerging technologies mainly ultrasounds and microwaves to enhance antioxidant extraction yields and quality from *Z. lotus* (L.) Lam plant. In order to substitute synthetic antioxidants and palliate the inconvenience of conventional techniques which consume large quantities of solvent and time versus a lower yield, the UEA and MAE extraction technologies could be used. This would present a significant economic gain for the industry. The interest of this review was to show the benefits of antioxidants in protecting humans against free radicals and the benefits of using new environmentally friendly extraction technologies. Furthermore, the valorization of this local plant which could be used as an inhibitor agent of oxidation phenomena, the possibility of exploitation of these antioxidants at an industrial scale, and its commercialization is indicated in this mini-review. Finally, in the next few years, these green methods including UAE and MAE could provide an innovative approach to increase the production of specific compounds extracted from the *Ziziphus* plant for use as nutraceuticals or as ingredients in the field of modern food engineering.

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