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MICROWAVE RADIATION, ITS INFLUENCE ON SOLUTIONS AND USE FOR EXTRACTION OF COMPONENTS OF PLANT MATERIAL

1-st Report. The systems of microwave-assisted extraction and their application for extraction of natural compounds

Abstract. The review discusses the basic principles of microwave extraction and its difference from convection extraction methods. Microwave extraction systems and their application for distinguishing various classes of natural compounds are considered.

Key words: microwave radiation, natural compounds, microwave extraction.

Introduction. Extraction is the main step in the production of plant components. Conventional extraction methods include infusion, soaking, maceration, extraction in a Soxhlet apparatus and other procedures that require a long extraction time and which involve the risk of thermal decomposition of thermolabile active compounds. In recent decades, intensive development has been directed towards improving the processes of complex processing of plant raw materials and creating highly efficient methods for the isolation of biologically active substances, one of which is microwave-assisted extraction (MAE) [1-4].

1. The basic principles of MAE. Microwave extraction (MAE), originally introduced in 1994 by Paré and co-workers [1] as the "microwave-assisted process" (MAP). Currently, it is one of the areas of analytical "green" chemistry, which quickly gained the position of one of the most effective methods for isolating compounds.

The effect of microwaves on lots of liquids and solids, which consisting polar molecules and ions leads into their heating. The reason for this phenomenon is the interaction of the electrical component of the electromagnetic field with molecules of the irradiated substance. This interaction contains several physical effects. There are two mechanisms that cause heat generation, namely, dipole rotation and ionic conductivity.

The ability of a material to convert electromagnetic energy into heat is characterized by the tangent of the dielectric loss angle tan α and is calculated by the following formula (1):

$$tg\ \delta = \varepsilon''/\varepsilon' \qquad (1)$$

where ε'' is the coefficient of dielectric losses, which characterizes the efficiency with which the energy of the electromagnetic field is converted into heat, and ε' is the dielectric constant.

The values of these quantities depend on nature and state of the substance, the frequency of the electromagnetic field, and temperature. Usually, with increasing frequency, ε 'decreases, and ε "passes through a maximum. The value of *tan δ* has maximum values in the microwave range of electromagnetic radiation.

The contribution of dipole polarization or ionic conductivity is largely determined by the temperature. For water molecules and other solvents, the dielectric losses due to the contribution of dipole rotation are decreased with increasing temperature, and the dielectric loss due to ionic conductivity, on the contrary, increases with increasing sample temperature [9-11].

When extracting, dry plant materials are usually used, but plant cells contain small traces of moisture that serve as a target for microwave heating. Thanks to the microwave effect, when moisture is heated inside the plant cell, it evaporates and creates enormous pressure on the cell wall from inside. Pressure pushes the cell wall from within, stretches and eventually destroys it, which helps the components from the destroyed cells to escape into the surrounding solvent.

This process can be more intense if the plant matrix is impregnated with solvents with a higher heating efficiency under the action of microwaves (i.e., with a higher *tan δ* value). The higher temperature achieved throughout this process can hydrolyse the cellulose ether bonds that are present in the cell wall and reduce its mechanical strength, and this in turn helps the solvent to have easier access into the cell. During the rupture process, rapid release of chemical compounds from the cell to the surrounding solvent occurs. This microwave extraction mechanism, based on the exposure of analytes to the solvent by cell rupture, differs from the convection extraction mechanism, which depends on a series of permeability and solubilization processes to remove the analytes from the plant matrix. In addition, the transfer of dissolved ions enhances the penetration of the solvent into the matrix, which facilitates the release of substances. A microwave heating strongly depends on the dielectric susceptibility of both the solvent and the solid plant matrix. Basically, the sample is placed in one solvent or a mixture of solvents that absorb microwave energy. Temperature helps increase the penetration capacity of the solvent into the matrix and, as a result, the components are released into the environment of the hot solvent. However, in some cases only selective heating of the sample matrix is achieved by immersing the sample in a transparent solvent (hexane, chloroform). This approach is particularly useful for thermolabile components to prevent their degradation.

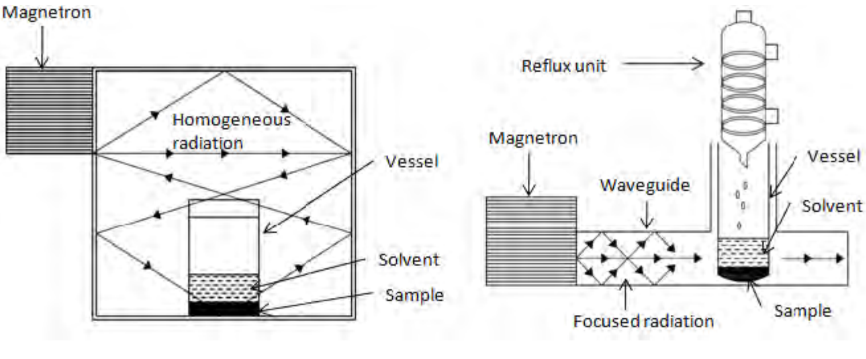
In conventional solvent extraction, mass transfer from the inside to the outside occurs, while heat transfer occurs in the opposite direction. In MAE, these transport phenomena occur in one direction, and the increase in the extraction rate can be due to a synergistic combination of two transport phenomena: mass and heat, which act in one direction. At MAE, heat is dissipated to the entire volume inside the irradiated medium, while in thermal extraction, heat is transferred from the coolant through the outer part of the sample to the inner part. This leads to an important difference between conventional and microwave heating. Heat transfer depends on the thermal conductivity, and in the case of liquids, it is due to convection currents. As a result, a fairly slow increase in temperature is observed.

In contrast, microwave heating, due to the heating effect affecting the entire volume. It can be obtained much more quickly. The rise of temperature will depend on the power of the microwaves and the dielectric loss factor of the material that is irradiated. Evaporation occurs, when microwaves come into contact with tiny traces of moisture within the cell matrix. It creates intense pressure on the cell wall, which is ruptured and causes release of the active components. Higher throughput can be achieved by carefully selecting conditions such as temperature, since a rise in temperature promotes a faster penetration of the solvent into the matrix of cells. Among other parameters that affect the performance of MAE, the solid-liquid ratio, extraction time, microwave power, character of the samples and mixing should be noted.

The effect of microwaves on extraction is to increase the speed and increase the degree of extraction of components. This is due to a specific microwave effect. It consists in raising the temperature of the extraction system and in selective action on plant material and the solvent used. This is achieved by selective heating of the phase or individual components, as well as in reducing the risk of thermal decomposition of thermolabile active compounds [5-9].

2. The systems of microwave-assisted extraction and its main components. Since the first publication of Adu-Samra et al. [12] on the use of microwave treatment in chemistry, which used a household microwave oven for the laboratory analysis of trace amounts of metals in biological media, the technique and technology of microwave extraction has been further developed. Commercial reactors have been developed for conducting MAE as in both closed reactors as and in atmospheric systems [13].

In general, MAE systems are classified into a multi-mode system and a focused-mode system (mono-mode) according to the type of distribution of microwave energy in the resonator [13-15]. Schematically, according to [16, 17], both systems are shown in figure.



A B

Types of microwave systems [16, 17]:
A) microwave system of "closed" type; B) microwave system of "open" type

Multimode and focused microwave devices contain four main components, namely: a microwave generator or a "magnetron" that generates microwave energy; waveguide used to propagate microwaves from a source to a microwave cavity; an applicator that is a device for placing a sample that can be multimode in which the microwaves are randomly distributed or the waveguide and circulator itself being a three- or four-port device, usually consisting of magnets and a ferrite material used to control the direction of the signal flow in the microwave system.

When microwaves enter the multimode furnace chamber, they are reflected from its walls. When reflecting from the chamber walls in three directions standing stationary waves – modes are generated. Usually, in the chamber of a household furnace, from 3 to 6 such modes are created, providing uniform heating. However, the field intensity in the multimode chamber is not the same, which contributes to the formation of so-called "hot" and "cold" zones, in which the degree of sample heating can vary significantly, especially if the sample is small in size. In addition, the non-uniform distribution of electromagnetic energy is facilitated by the periodic operation of the magnetron. Part of the energy is absorbed by the sample, the other part is dissipated as heat to the environment. To equalize the energy density throughout the volume, the chamber is provided with dissectors and rotating pallets [13].

If the multimode system produces a random dispersion of microwave radiation in the cavity, the monomode system allows focusing of microwave radiation in a confined zone and the energy is directed directly to the object being processed through the waveguide. In a monomode system, only one type of frequency is generated in the cavity, allowing only one resonance mode to be excited. In this case, the sample must be placed in a certain position in order to obtain the maximum of the microwave energy. This method provides direct and efficient use of microwave energy of high density by the sample matrix, inducing rapid heating of the medium with rapid extraction of the analyte. However, monomodule systems are very limited in the volume and quantity of the used sample, which can be extracted. On the other hand, the multimode systems can be used for a larger quantity of raw materials using several different configurations of the extraction vessel. The energy losses of monomode reactors are minimal, so it is characterized by lower power consumption compared to the multimode system.

Microwave systems can operate under pressure or at atmospheric pressure. The first experiments on the extraction of organic compounds were carried out in a household microwave oven (multimode system) in closed vessels without pressure control.

Typically, a multi-mode system is associated with high pressure, while a monomode system is used at atmospheric pressure, but can also operate at high pressure. To avoid confusion in the MAE classification, the terms "closed system" and "open system", which operates under pressure and at atmospheric pressure, are used to designate the system used, respectively [16, 18-20].

In a closed MAE system, the extraction is carried out in a sealed vessel with different microwave conditions. Extraction is usually carried out with uniform microwave heating. High operating pressure and system temperature ensure fast and efficient extraction. The pressure within the extraction vessel is controlled in such a way that it does not exceed the working pressure of the vessel, while the temperature can be adjusted above the normal boiling point of the extraction solvent. The results of research in this direction led to the development of a nitrogen-protected microwave-assisted extraction (NPMAE) technique [20].

3. Advantages and disadvantages of the "closed" and "open" system of microwave-assisted extraction. The advantages of a "closed" system include the following:

1) higher temperatures can be achieved in them than in an open vessel, because the increased pressure within the vessel increases the boiling point of the solvents used;

2) reducing the time required for microwave treatment;

3) the loss of volatile substances is practically eliminated;

4) less solvent is needed, since evaporation does not occur and there is no need to constantly add a solvent;

5) reduced the risk of contamination in the air;

6) the vapors formed during the acid microwave treatment are contained inside the vessel;

As weaknesses it is possible to note:

1) high pressure inside the vessel reduces the safety of the process;

2) a limited amount of a sample that can be processed;

3) the procedure excludes the addition of reagents during operation;

4) cooling the vessel before opening, to avoid loss of volatile components;

5) the materials used must withstand high temperatures.

The "open" system reduces some of the security problems, since it operates under milder conditions and is considered more suitable for extracting thermolabile connections. This system has a higher sample throughput, and more solvent can be added to the system at any time. The "open" MAE system operates at atmospheric pressure, and only part of the vessel is directly exposed to the propagation of microwave radiation. The upper part of the vessel is connected to a reflux condenser to condense the solvent or substance.

The use of atmospheric pressure provides significant advantages over pressure vessels:

1) increased safety performance;

2) the possibility of adding reagents;

3) the use of vessels of various materials (glass and quartz);

4) ease of removal of excess solvent;

5) the possibility of processing more material;

6) no requirements for cooling or sealing;

7) low cost of necessary equipment;

8) suitable for heat-labile joints.

Despite their numerous advantages, the "open" type systems also have some disadvantages:

1) the bandwidth is lower, since most open systems can not process multiple samples at the same time, while systems with a closed type can handle 8-14 samples at a time;

2) the operating time required to obtain results similar to systems of "closed" type usually is longer [21].

4. Application of the "closed" and "open" system of microwave-assisted extract for the isolation of natural compounds. Some examples of the use of the "closed" and "open" MAE system for the extraction of natural compounds are presented in table 1 and 2.

Table 1 – The use of a "closed" MAE system for the extraction of natural compounds

N	Plant material	Compounds	Equipment	Extraction parameters	Ref.
1	2	3	4	5	6
1	Citrus mandarin peels	Phenolic acids	Microwave extractor CW-2000, 800 W (Xintuo Technology, Shanghai, China)	Solvent - 66% methanol, liquid / solid ratio substance - 16 ml/g, power - 152 W, time - 49 s	22
2	Peanut peels	Phenolic compounds	Microwave system MES-1000, 950 W	Solvent -30% ethanol, liquid / solid ratio substance - 25 ml/g, power - 855 W, time-30 s	23
3	Dactylis glomerata, Festucarubra L., Festuca ovina L., Bromusinermis и Bromus marginatus, Hypericum perforatum, Thymus vulgaris L., Tilia cordata, Uncaria tomentosa	Phenolic compounds	Microwave system Ethos SEL (Millestone, Sorisole, Italy)	Solvent - aq. HCl (2 mol / l HCl), time - 20 min, T = 70 ° C.	24
4	Bark, core and roots of apple Malus domestica	Phenolic compounds	Microwave system MARS-X, 1500 W (CEM, Mathews, NC, USA).	Solvent - ethanol: water 60:40 vol. / vol.) liquid / solid ratio substance - 20:0.1 ml/g, time - 20 min., T = 100 ° C.	25

Continuation of table 1

1	2	3	4	5	6
5	Leaves Stevia rebaudiana Bertoni	Phenolic compounds	Microwave system Multiwave 3000 SOLV (Anton Paar, Graz, Austria).	Solvent - 75% ethanol, liquid / solid ratio substance - 10 ml/g, time- 15 min, T = 100 ^{o}C.	26
6	Leaves Cyclocarya paliurus (Batal.)	Flavonoids	Microwave system MDS-2002AT (Shanghai Sineo Microwave Chemical Technology Co., Shanghai, China).	Solvent - 63.2% ethanol, liquid / solid ratio substance - 21.4:1 ml/g, time -15 min, T = 76.8 ° C.	27
7	Leaves Pistacia lentiscus var.	Phenolic compounds and flavonoids	Microwave system Start D (Milestone, Sorisole, Italy).	Solvent - water, liquid / solid ratio substance - 12: 0.6 ml/g power - 400 W, time - 60 min, T = 100 °C.	28
8	Roots Radix astragali	Flavonoids	Microwave system Ethos*T (Millestone, Sorisole, Italy)	Solvent - 90% ethanol, liquid / solid ratio substance - 25 ml/g, power - 1000 W, time - 25 min, 2 extraction cycles (total - 50 min.), T = 110 °C	29
9	Citrus unshiu	Hesperidin	Microwave system MicroSYNTH labstation, 1000 W (Millestone, Sorisole, Italy)	Solvent - 70% ethanol, liquid / solid ratio substance - 10 ml/g, time - 8 min,T = 140 °C	30
10	Purple corn (Zea mays L.)	Anthocyanins (cyanidin-3-glucoside, pelargonidine-3-glucoside, neonidine-3-glucoside)	Microwave extractor NJL07-3, 1100 W (Jiequan microwave equipment)	Solvent - 15 M HCl: 95% ethanol in a ratio of 15:85, liquid / solid ratio substance - 20 ml/g, power - 555 W, time - 19 min	31
11	Grape skins	Anthocyanins	Microwave extractor Ethos 1600, (Millestone, Sorisole, Italy)	Solvent - 40% methanol, liquid / solid ratio substance - 2.5 ml/g, power - 500 W,time - 5 min, T = 100 °C	32

Continuation of table 1

1	2	3	4	5	6
12	Leaves Pigeonpea (Cajanus cajan (L.) Millsp.)	3-Hydroxy-4-phenyl-methoxy-stilbene-2-carboxylic acid, pinostobin	Microwave system MARS-II, 1000 W (CEM, Corp. (Mathews, NC, USA)	Solvent - 80% ethanol, liquid / solid ratio substance - 30 ml/g, power - 300 W, time - 1 min 2 extraction cycles (total – 2 min.), T = 65 ° C	33
13	Roots Morinda citrifolia	Anthraquinones	Microwave system MARS 5, 1200 W (CEM Corp. (Mathews, NC, USA)	Solvent - 80% aqueous ethanol, liquid / solid ratio substance - 100 ml/g, power - 720 W, time - 15 min, T = 60 ° C.	34
14	Flowering part Melilotus officinalis L.	Coumarin, *o*-coumaric acid, melilotic acid	Microwave system Discover®LabMate, CEM Corp. (Matthews, NC, USA)	Solvent - 50% aqueous ethanol, liquid / solid ratio substance - 20 ml/g, power - 100 W, time - 5 min, 2 extraction cycles (total- 10 min)	35
15	Fruit Pastinaca sativa	Coumarins	Microwave system Plazmotronika UniClever BMZ I (Wroclaw, Poland)	Solvent - 80% methanol, power - 240 W, time- 1 min power - 360 W, time-30 min (total - 31 min.)	36
16	Ganoderma atrum	Triterpene saponins	Microwave extractor MDS 3003 (Shanghai Xinyi company, Shanghai, China)	Solvent - 95% ethanol, liquid / solid ratio substance - 25 ml/g, power - 800 W, time - 5 min 2 extraction cycles (total - 10 min.), T = 78 °C	37
17	Roots *Panax ginseng*	Saponins	Microwave extractor Microdigest 3.6, 250 W, (Prolabo, Fontenay-sous-Bois, France)	Solvent - 60% ethanol, liquid / solid ratio substance - 10 ml/g, power - 162 W, time - 30 s	38

Completion of table 1

1	2	3	4	5	6
18	Silybum marianum L.	Silymarin	Microwave extractor Ethos-1, 1600 W (Millestone, Sorisole, Italy)	Solvent - 82% ethanol, liquid / solid ratio substance - 38 ml/g, time - 60 min, T = 112 °C	39
19	G. Sibiricum Linne	Alkaloids (corilagin, geraniin)	Microwave extractor (Shanghai Sineo Microwave Chemical Technology Co., Ltd., Shanghai, China)	Solvent--water, liquid / solid ratio substance - 40 ml/g, power - 500 W, time - 20 min, T = 33 °C	40
20	Stephania sinica	Alkaloids (sinoacutine, palmatine, isocorydine, *l*-tetrahydropalmatine)	Microwave system MDS-8 (Shan-ghai Sineo Microwave Chemical Technology Co., Ltd., Shanghai, China)	Solvent - 65% ethanol, liquid / solid ratio substance - 24 ml/g, power - 150 W, time - 90 s, T = 60 °C.	41
21	Leaves Vernonia amygdalina	Hydrocarbons, alcohols, esters, fatty acids and ketones	Microwave extractor Ethos (Milestone, Sorisole, Italy)	Solvent - dist. water, liquid / solid ratio substance - 10 ml/g, power - 416 W, time - 7 min., T = 100 °C.	42

Table 2 – The use of an "open" MAE system for the extraction of natural compounds

№	Plant material	Compounds	Equipment	Extraction parameters	Ref.
1	2	3	4	5	6
1	Dried cortex Eucommia ulmodies oliv.	Geneposide and chlorogenic acid	Modified microwave oven LG, 700 W	Solvent - 80% ethanol (for geneposidic acid), 20% ethanol (for chlorogenic acid), liquid / solid ratio substance - 20 ml/g, power - 350 W, time - 30 - 40 s	43
2	Leaves of tobacco	Solanesol	Modified household microwave oven with magnetic stirrer, temperature control and time.	Solvent -hexa-ne: ethanol (1: 3), NaOH 0.05 mol / L, liquid / solid ratio substance - 10 ml/g, power - 700 W, time - 40 min., T = 60 °C	44

Continuation of table 2

1	2	3	4	5	6
3	Tribulus terrestris	Steroid Saponins	Microwave system MAS-II (Sineo, 900 W)	Solvent - 70% ethanol, liquid / solid ratio substance - 20 ml/g, power - 500 W, time - 5 min.	45
4	Roots Radix Bupleuri	Saponins (saikosaponin A, C, D)	Microwave system made to order	Solvent- 47 - 50% ethanol, liquid / solid ratio substance - 30 ml/g, power - 360-400 W, time - 6 min, T = 73-74 °C	46
5	Rhizomes Rodgersia aesculifolia Batal	Fatty acids (oleic, linoleic and palmitic acid), steroids	Modified microwave oven LG, 800 W	Solvent - 80% ethyl ether, liquid / solid ratio substance - 2 ml/g, power - 320 W, time - 40 s	47
6	Roots Radix astragali	Astragalosides I-IV	Microwave system MARS-II (CEM, 1000 W)	Solvent-80% ethanol, liquid / solid ratio substance - 25 ml/g, power - 700 W, time - 5 min 3 extraction cycles (total - 15 min), T = 70 °C	48
7	Leaves Gymnema svlvestres	Oleanolic acid	Microwave extractor CATAR (Catalyst Systems, 700 W)	Solvent - 90% ethanol, liquid / solid ratio substance- 25 ml/g, power - 500 W, time - 8 min.	49
8	Leaves green tea	Polephenols, caffeine	Modified microwave oven (National, Japan, 700 W) with magnetic stirrer	Solvent -50% ethanol, time - 4 min.	50
9	Roots licorice	Glycyrrhizic acid	Modified microwave oven (National, Japan, 700 W) with magnetic stirrer	Solvent -50-60% ethanol and 1-2% ammonia solution, liquid / solid ratio substance - 10 ml/g, time - 4-5 min.	51
10	Nothapodytes foetida	Camptothecin, 9-methoxycamptothecin	Microwave oven MCG-LG, MG583, (Mumbai, India)	Solvent - 90% methanol, liquid / solid ratio substance - 20 ml/g, power - 100 W, time - 3 min.	52

Continuation of table 2

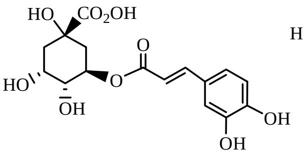
1	2	3	4	5	6
11	Roots Salvia miltiorrhiza	Diterpenes (tanshinone I, tanshinone IIA, cryptotanshi none)	Modified microwave oven (National, Japan, 700 W) with magnetic stirrer	Solvent - 95% ethanol, liquid / solid ratio substance - 10 ml/g, time - 2 min.	53
12	Leaves Pistacia lentiscus L.	Polyphenol compounds	Modified microwave oven (NN-S674MF, Samsung, Malaysia)	Solvent - 46% ethanol, liquid / solid ratio substance - 28 ml/g, power density - 17.86 W / ml, time - 60 s	54
13	Leaves and fruits Prunus laurocerasus	Chlorogenic acid, 3-glucoside of quercetin, 7-glucoside luteolin, 7-glucoside apigenin, 3-glucoside kaempferola and naringenin	Modified microwave oven «Samsung» (M1712N, Malaysia)	Solvent - methanol, liquid / solid ratio substance - 10 ml/g, power - 300 W, time - 4 min, total - 15 min.	55
14	Terminalia chebula Retz.	Flavonoids	Modified microwave oven R-219T, (SHARP, Japan)	Solvent - water, liquid / solid ratio substance - 40: 1 ml/g, time - 1 min, T = 100 °C.	56
15	Peel Citrus sinensis	Phenolic compounds	Modified microwave oven «Samsung» model: NN-S674MF, Kuala Lumpur, Malaysia)	Solvent - 51% aqueous acetone, liquid / solid ratio substance - 25 ml/g power - 500 W, time - 122 sec.	57
16	Grape peels	Phenolic compounds	Microwave system Microdigest 301d with a maximum power of 200 W (Prolabo, France)	Solvent -50:50 (v/v) ethanol-water with 0.8% (v / v) HCl, liquid / solid ratio substance - 10 ml/g, power - 140 W, time - 10 min.	58
17	Fruit Morus alba L.	Gallic acid, chlorogenic acid, p-coumaric acid, ferulic acid, rutin, (+) catechin	Microwave system Microdigest (Soxwave 100, Prolabo, France)	Solvent - 40% ethanol, liquid / solid ratio substance - 25 ml/g, power - 210 W, time - 8 min.	59

Continuation of table 2

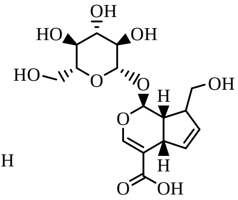
1	2	3	4	5	6
18	Raspberries Rubus Coreanus Miq.	Anthocyanins, phenolic compounds	Microwave system Microdigest (Soxwave 100, Prolabo, France).	For anthocyanins: solvent - 27.5% ethanol, liquid / solid ratio substance - 10 ml/g, power - 219 W, time - 5.9 min.	60
				For phenolic compounds: solvent - 24.4% ethanol, liquid / solid ratio substance -10 ml/g, power - 166 W, time - 4.5 min.	
19	Cherry Pomace	Phenolic compounds	Modified microwave oven Ethos D, 1000 W (Millestone, Italy)	Solvent - ethanol-water, 1: 1 v/v), liquid / solid ratio substance - 20 ml/g, power - 700 W, time is 12 min.	61
20	Prunus laurocerasus L.	Phenolic compounds	Modified microwave oven «Samsung», type M1712N (Malaysia)	Solvent -methanol, liquid / solid ratio i substance - 20 ml/g, power - 550 W, time - 25 min.	62
21	Hawthorn fruits Crataegus pinnatifida Bge.	Chlorogenic acid, procyanidin B and C, epicatechin, rutin	Microwave system XH-100B, 850 Вт. (Xianghu Science and Technology Co., Ltd., Beijing, China)	Solvent -83% ethanol liquid / solid ratio substance - 20 ml/g, power - 440 W, time -13 min.	63
22	Dimocarpus Longan Lour.	Phenolic compounds	Microwave system XH-100A (Beijing XiangHu Science and Technology Development Co., Ltd., Beijing, China)	Solvent -95% ethanol, liquid / solid ratio substance - 10 ml / g, power - 500 W, time - 30 min., T = 80 ° C.	64
23	Памело Citrus grandis (L.) Osbeck	Polyphenolic compounds	Microwave system (MAS-II; Shanghai Xinyi Microwave Chemistry Technology Co., Ltd., Shanghai, China)	Solvent -85% ethanol, liquid / solid ratio substance - 15 ml/g, power - 1000 W, time - 45 min.	65
24	Clinacanthus nutans	Polyphenolic compounds	Microwave system CEM Discover®, 300 W (CEM Corporation, Matthews, NC, USA).	Solvent - 50% ethanol, liquid / solid ratio substance - 14 ml/g, power - 300 W, time - 15 s	66

Completion of table 2

1	2	3	4	5	6
25	Bark Quercus robur L.	(+) - Catechin, (-) - epicatechin, (-) - epicatechin gallate, p-coumaric acid, naringenin	Microwave system NEOS-GR, 230V-50 Hz, 900 W (Milestone, Italy)	Solvent - 33% ethanol, pH 10.75, liquid / solid ratio substance - 20 ml/g, power - 45 W, time - 60 min, particle size 0.5 cm.	67
26	Curcuma longa L.	Curcumin, dimethoxycurcumin bis-dimethoxy curcumin	CEM Discover system equipped with a 2455 MHz magnetron andinfrared fiber optic probe. (CEM Corporation, Matthews, NC,USA).	Solvent - ethanol, 75% ethanol, methanol, 75% methanol IL: 0.3 mol / l 1-octyl-3-methyl-imidazolium bromide Optimum conditions: liquid / solid ratio substance - 30:0.5 ml / g, time - 2 min, T = 70 ° C.	68



Chlorogenic acid



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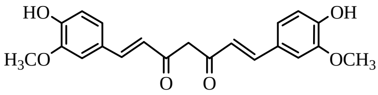

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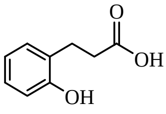
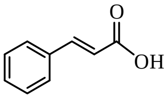
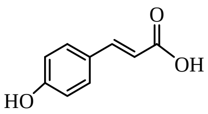


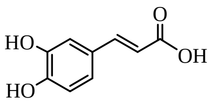
Melilotic acid



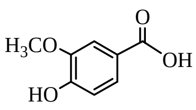
Cinnamon acid



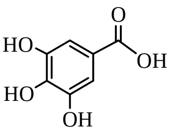
n-Cumaric acid



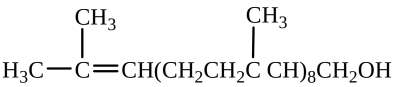
Caffeic acid



Vanillic acid

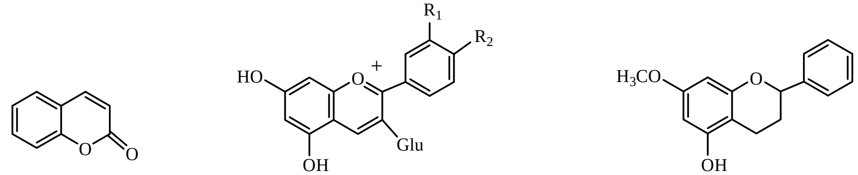


Gallic acid

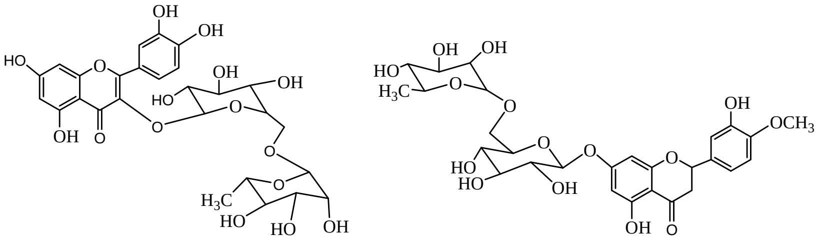


$H_3C{-}\overset{CH_3}{\overset{|}{C}}{=}CH(CH_2CH_2\overset{CH_3}{\overset{|}{C}}\,CH)_8CH_2OH$

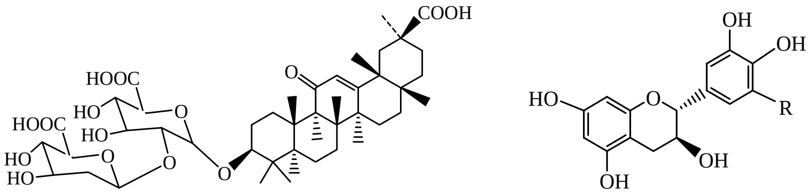
Solanesol



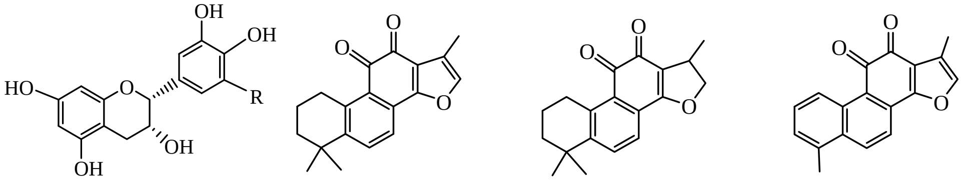
Coumarin
Cyanidin-3-glucoside (R_1, R_2-OH)
Pelargonidine-3-glucoside (R_1-H, R_2-OH)
Neonidine-3-glucoside (R_1-OCH_3, R_2-OH)
Pinastorbine



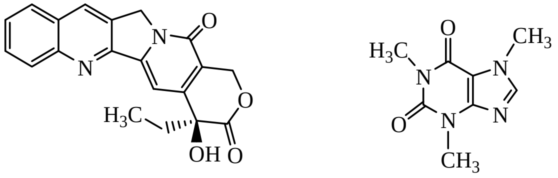
Rutin
Hesperidin



Glycyrrhizic acid
(+) catechin (R-H)



(-) epicatechin (R-H)
(-) epicatechinhallate (R-OH)
Tanshinon IIA
Kryptotanshinon
Tanshinon I



Camptothecin
Caffeine

Figure 2 – Chemical structures of some derivatives presented in table 1 and 2

The choice of the extraction solvent is crucial for MAE and depends on the solubility of the analyte being studied. The interaction of the solvent and the plant matrix, and the microwave absorbing properties of the solvent. Accordingly, the solvent should be highly selective, eliminating the removal of unwanted components. A key role in microwave extraction is played by the dielectric properties of the solvent, since both the efficiency and the selectivity of MAE strongly depend on the dielectric constant of the extracting solvent or its mixture. For example, in the case of extraction of furanocoumarins from plant tissues of Pastinaca sativa [36], petroleum ether is usually used, while more polar coumarins containing hydroxyl groups are extracted with methanol. Since the use of petroleum ether in MAE is ineffective, 80% methanol was used, however, the yields of furanocoumarins at use were significantly lower than when extracted in a Soxhlet apparatus and with ultrasonic extraction. Typically, in MAE, in most cases, solvents or solvent mixtures with good microwave heating efficiency (with a high $\tan \delta$ value) are used. Water-organic solvents, in particular acetone, ethanol and methanol, are most often used for extraction than the corresponding mono-component solvent systems [69]. This is confirmed by the data given in tables 1, 2. Thus, the use of aqueous methanol is described for the extraction of phenolic acids [22], anthocyanins [32], coumarins [35, 36], alkaloids [52]; aqueous ethanol was used to extract phenolic compounds [23, 25-27, 50, 52], flavonoids [27, 29, 33, 58], anthraquinones [34], saponins [37, 38, 45, 46], alkaloids [41], geneposidic and chlorogenic acids [43], fatty acids [47, 49], diterpenes [53], catechins [67]; and the largest yield of polyphenols from Citrus sinensis [57] was recorded using 50% aqueous acetone. In addition, the use of water [28, 40, 42, 67], acidified aqueous alcoholic mixtures [24, 31], 50-60% ethanol with 1-2% ammonia solution, for example, for the isolation of glycyrrhizic acid [51] or the use of ionic liquids (IL) (1-octyl-3-methylimidazolium bromide [68]) was reported.

The efficiency of extraction is influenced by the percentage of alcohol-water extractable mixture of solvents. Among the various ethanol concentrations, the use of 95% ethanol showed the best optimal results for the MAE tanshinone (tanshinone IIA, cryptotanshinone, tanshinone I) from the roots of Salvia miltiorrhiza [52], and the use of 80% aqueous methanol was optimal for extraction of chlorogenic and gneoposide acid [43]. When studying the concentration of ethanol to isolate flavonoids from Cyclocarya paliurus (Batal.) [27], it was shown that the yield of flavonoids increased when the concentrations of aqueous ethanol ranged from 30% to 70% and at an ethanol concentration above 70% the yield decreased. Similar patterns were also observed with MAE saponins from Panax ginseng [38]; an increase in the yield of the derivatives was observed with varying the concentration of ethanol from 30% to 60%, and a yield reduction using 75% and 90% ethanol, as with a microwave power of 88 W, and as of at 162 W. In the case of extraction of phenolic compounds from Morus alba L. [59], the highest yield of the derivatives was achieved with 40% ethanol, while using 30, 40, 50 and 60% ethanol, the quantitative yield was lower. In MAE an increase in the yield of the derivatives of phenolic compounds from Stevia rebaudiana, was observed with a variation in the ethanol concentration from 0 to 75%, and the use of 100% ethanol reduced indices were obtained [26]. A study of the concentration of ethanol for the isolation of glycyrrhizic acid [51] showed that an increase in the concentration of ethanol from 0 to 50-60% leads to an increase in the degree of recovery, whereas at concentrations from 60 to 100%, the yield is sharply reduced. To improve recovery, the use of 1-2% ammonia with 50-60% ethanol was tested and it was shown that the yield of glycyrrhizic acid was significantly increased, due to the formation of a salt having better solubility in the employed solvent system. Extraction using 0.3 mol/l of 1-octyl-3-methylimidazolium bromide was successfully used to extract curcumin and its derivatives from Curcuma longa L. [68].

The decisive factor for the effectiveness of MAE is the volume of the used solvent and the characteristics of the plant matrix. The volume of the solvent must be sufficient to ensure that the plant matrix is completely immersed in the solvent during the entire extraction time. In most cases, a higher ratio of solvent volume to solid matrix can be effective in conventional extraction methods. However, in MAE, a higher ratio may result in lower sample recovery, which may be due to inadequate solvent mixing when exposed to microwaves. Such characteristics of the matrix as the particle size of the sample and the state in which it is presented for MAE can have a tremendous effect on the extraction of compounds. Fine powders can increase the extraction process, providing a larger surface area, which provides better contact with the solvent, but it should also take into account the ability of the plant material to swell and the ability to mix the extracted raw material during extraction, as well as the subsequent processing, especially at the filtration stage. Typically, the studies provide data on the size of the extractable particles of the plant matrix and/or the liquid/solid ratio. Thus, the study [27] showed that the yield of flavonoids tended to increase (from 14.56 ± 0.22 to 15.42 ± 0.45 mg/g) with an increase in the ratio of solvent to material from 10:1 to 20:1, but the recovery rate quickly decreased at a ratio above 20:1. This is probably due to inadequate stirring of the solvent under microwave irradiation. On average, the liquid/solid ratio is in the range of 10 ml/g [26, 30, 38, 42, 44, 51, 53, 55, 60, 64] to 20-25 ml/g [22, 23, 27-29, 31, 32, 37, 41, 43, 45, 48, 49, 52, 54, 57, 61-63, 65].

Another factor, the influence of which must be taken into account, is the extraction time. Typically, by increasing the extraction time, the number of extracted substances can increase, but the risk of degradation of the derivatives should be taken into account. Thus, from the data in Tables 1 and 2, it can be seen that the extraction time varies from 15 s to 60 min, but on the average, 10-20 min is sufficient for extraction [24-27, 31, 34, 35, 37, 40, 55, 61-63]. Thus, in a number of studies good results in 15-90 s were obtained [22, 23, 33, 41, 43, 47, 54, 56, 57, 66]; however it was demonstrated that, for example, for the extraction of active components from Pistacia lentiscus var in [28], silymarin from Silybum marianum L. [39] or phenolic compounds from Quercus robur L. [67] extraction took 60 min. Optimizing the extraction time is an important factor in the efficiency of the process, since the extraction time may vary depending on the part of the used plant and the kind of microwave extraction. The time of irradiation is also affected by the dielectric properties of the solvent. According to Chan et al. [20] and Veggi et al. [70] thermal degradation and oxidation of sensitive compounds are related to the time of microwave irradiation. Solvents such as ethanol, methanol and water can be strongly heated by prolonged exposure, thereby increasing the risk of destruction of thermolabile components. Thus, in the study of MAE compounds from Morus alba L. [59] it was shown that when the irradiation time was extended from 1 to 7 min, the maximum values of the polyphenols and flavonoids were recorded, but longer heating reduced the yield of the derivatives due to the thermal decomposition of the polyphenolic compounds.

Microwave power and extraction time are two factors that greatly affect each other. The use of low and medium microwave power with longer exposure to the extracted material may be a more reasonable approach. As a result rapid destruction of the cell wall occurs at a higher temperature and a higher power. Along with the desired components, impurities pass into the solvent. At low power levels, the cell wall rupture can occur gradually, which makes it possible to carry out selective MAE. Thus, the extraction of saponins using the MAE method [38], took 30 s at a power of 250 W and 180 s at a power of 88 W, whereas for a conventional thermal extraction at 80 °C took 3 h. In a closed vessel system, the selected power settings depend on the number of samples to be extracted, since up to 12 vessels can be used during one extraction run. A reasonable choice of the used power avoids an excessive temperature, which can lead to sample degradation and excessive pressure inside the vessel, in the case of a closed MAE system. In [27], data on the yield of flavonoids from C. Paliurus on the effect of temperature (T = 40, 50, 60, 70, 80 and 90°C), at a fixed extraction time of 5 minutes, the solvent to material ratio 20:1 and using 90% ethanol as the solvent are presented. At a solvent temperature of 40 to 80 ° C, the extraction efficiency (from 6.17 ± 0.32 to 12.99 ± 0.25 mg/g) was significantly increased, which may be due to the fact that a higher temperature leads to an increase in the molecular interaction and increased solubility. However, higher extraction temperatures T = 80-100 ° C showed no significant improvement in extraction, and further heating may have negative consequences caused by degradation or conversion of the analytes.

Microwave power and temperature are related to each other and require special attention, especially when working with a closed vessel system. In a closed vessel system, the temperature can be much higher than the boiling point of the used solvent. This increased temperature leads to an increase in recovery efficiency due to an increase in the desorption of the sample from the active sites in the matrix. Moreover, solvents have a greater ability to solubilize the analytes at higher temperatures. At the same time, the surface tension and viscosity of the solvent are reduced, which improves the wetting of the sample and increases the ability of the solvent to penetrate into the matrix. The efficiency of the solvent heating is also important, since the evaporation of the solvent will depend on how quickly it heats under the action of microwaves. Therefore, optimization of this parameter is also of paramount importance. An investigation of the influence of different temperature regimes on the extraction of flavonoids in [27] showed that the yield first rapidly increased with increasing temperature of the solvent from 40 to 80 °C, and then slowed down at T> 80 °C. Such effect can be explained by the fact that the initial increase in temperature leads to an increase in solubility, and higher temperatures have negative consequences caused by degradation or conversion of the analytes. The optimum temperature T = 100 °C was determined for MAE flavanoids from Stevia rebaudiana [26], and the process at T = 125 °C and T = 150 °C reduced the yield of the derivatives.

The use of the MAE method significantly reduces the process time compared to conventional extraction methods, as well as by ultrasonic extraction. Thus, the MAE method of extraction of phenolic compounds from Pistacia lentiscus var. [28] took 60 min, whereas during extraction in the Soxhlet apparatus it took 3-4 h and 3 extraction cycles. When extracting coumarin, o-coumaric acid and melilotic acid from Melilotus officinalis (L.), it took 8 h in the Soxhlet apparatus, 60 min for ultrasound extraction, whereas for MAE it took only 5 min and two extraction cycles. In MAE, tanshinones from the roots of Salvia miltiorrhiza is required only 2 min, and extraction at room temperature, ultrasound extraction and extraction in the Soxhlet apparatus requires 24 h, 75 min and 90 min, respectively [52]. In the case of extraction of polyphenolic compounds from the leaves of Pistacia lentiscus [54], the MAE process took only 60 s, the ultrasonic extraction was 15 min, and the extraction using heating was 120 min, while the total yield of polyphenolic compounds was higher with MAE than with ultrasound and heating.

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Резюме

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МИКРОВОЛНОВОЕ ИЗЛУЧЕНИЕ, ЕГО ВЛИЯНИЕ НА РАСТВОРЫ И ИСПОЛЬЗОВАНИЕ ДЛЯ ЭКСТРАКЦИИ КОМПОНЕНТОВ РАСТИТЕЛЬНОГО СЫРЬЯ

Сообщение 1. Системы микроволновой экстракции и их использование для экстракции природных соединений

В обзоре обсуждаются основные принципы микроволновой экстракции и ее отличительные особенности. Рассматриваются системы микроволновой экстракции, их преимущества и недостатки. Проведен анализ использования «закрытой» и «открытой» системы микроволновой экстракции для извлечения различных классов природных соединений.

Ключевые слова: микроволновое излучение, природные соединения, микроволновая экстракция.

Резюме

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МИКРОТОЛҚЫНДЫ СӘУЛЕЛЕНУ, ОНЫҢ ЕРІТІНДІЛЕРГЕ ӘСЕР ЕТУІ ЖӘНЕ ӨСІМДІК ШІКІЗАТТЫҢ КОМПОНЕНТТЕРІНІҢ ЭКСТРАКЦИЯ ҮШІН ҚОЛДАНУЫ.

1-ші хабарлама. Микротолқынды экстракция жүйелері және олардың түрлері табиғи қосылыстарды алу үшін қолданыңыз

Зерттеуде микротолқынды экстракцияның негізгі принциптері және оның ерекшеліктері қарастырылады. Микротолқын шығару жүйелері, олардың артықшылықтары мен кемшіліктері қарастырылады. Табиғи қосылыстардың әртүрлі сыныптарын алу үшін «жабық» және «ашық» микротолқынды экстракция жүйесін пайдалануды талдау жүргізілді.

Түйін сөздер: микротолқынның сипаттамасы**,** табиғи қосылыстар, микротолқынмен қыздыру.