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АКЦИОНЕРЛІК ҚОҒАМЫ

# ҚАЗАҚСТАННЫҢ ХИМИЯ ЖУРНАЛЫ

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## ХИМИЧЕСКИЙ ЖУРНАЛ КАЗАХСТАНА

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**"GREEN" DIRECTIONS  
MICROWAVE EXTRACTION  
IN CHEMISTRY OF NATURAL COMPOUNDS  
2-nd Report: Environmental factors of microwave extraction**

**Abstract.** The last decade marked by intensive use of microwave radiation for solving various problems of analytical chemistry, which one of the directions is extraction. At present, microwave extraction (microwave-assisted extraction (MAE)) has been taking the position of one of the most effective methods for the isolation of natural and biologically active compounds. The current trend of its development is the development and implementation of innovative approaches to the extraction of biologically active substances that comply the principles of "green chemistry". Factors affecting the environmental friendliness of microwave extraction include those associated with hazardous reagents, the amount of waste, safety, energy consumed and environmental impact.

**Keywords:** natural compounds, microwave extraction, environmental factors.

**Introduction.** Since the first using of microwave process in chemistry, when in 1975 Adu-Samra et al. [1] it used for the laboratory analysis of trace amounts of metals in biological media the microwave chemistry has leaped forward. Microwave radiation has found practical use in sample preparation [2, 3], for extraction [4-8], drying plant objects [9], thermal decomposition of plant biomass [10], in particular hydrolysis of plant biomass in order to produce biofuel (bioethanol), and also for enzymatic [11] and acid hydrolysis [12, 13], wood pyrolysis [14], delignification, such as rice husk [15] and beech wood [16]. After appearance of the first works of using microwave radiation in organic synthesis, in 1986 [17, 18], the intensive studies of microwave processing on the course of chemical reactions and chemical modification of plant materials were initiated.

At present, microwave extraction (microwave-assisted extraction (MAE)) has been taking the position of one of the most effective methods for the isolation of natural compounds. The trend of its development is to develop and implementation of innovative approaches in accordance with the principles of "green chemistry" [19,20]. One of its areas of development is the reduction or abandonment of the use of hazardous and toxic chemicals, the use of cleaner and less polluting processes, the prevention of pollution in the initial stages of process planning and the responsibility for the products produced [21, 22].

## DISCUSSION

**1. Environmental factors of microwave-assisted extraction.** Assessing the friendliness of microwave extraction to the "green" factors affecting the environmental friendliness of MAE should be attributed to those associated with

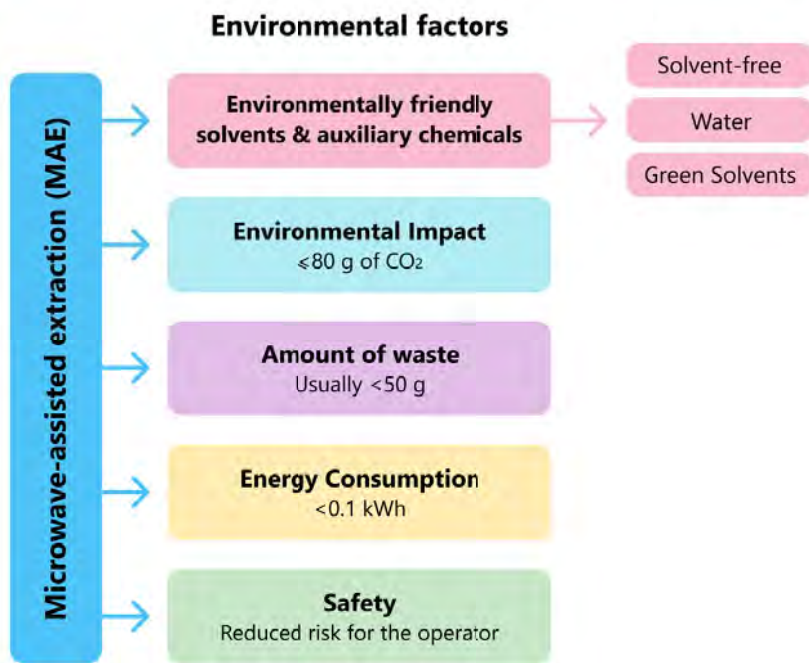


Figure 1 – Environmental factors of microwave-assisted extraction

hazardous chemicals (solvents and auxiliary chemicals), the amount of waste, safety, energy consumption and environmental impact (figure 1).

Most of the research to find more “green” methods of MAE, aimed at making chemical processes more environmentally friendly, emphasize the need to use safer, less toxic and more benign solvents, or the elimination of solvents, as well as reducing the use of reagents and auxiliaries. Other actions include reducing energy consumption through the use of milder conditions, preference for substrates based on renewable sources.

**2. Environmentally friendly solvents and auxiliary chemicals.** One of the main environmental problems in analytical chemistry is the need to use solvents. Since the application of methods without solvents [23] is not always possible, solvents should be selected taking into account environmental requirements.

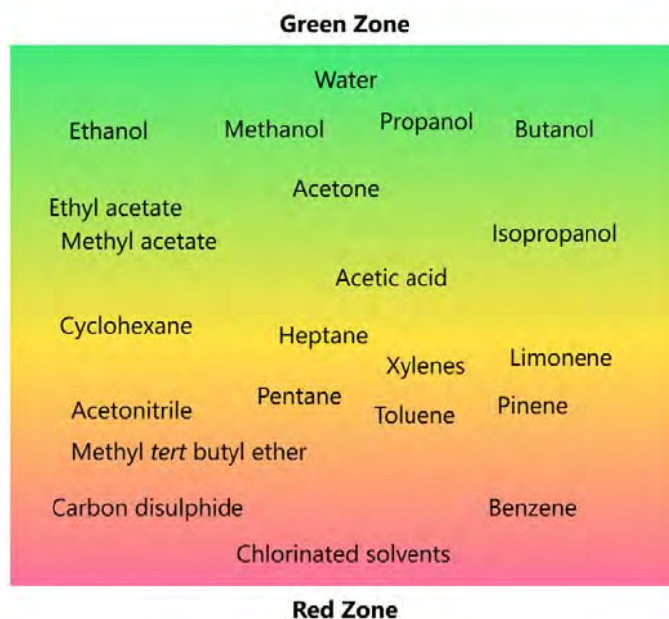
Currently, there are several guidelines developed by pharmaceutical companies on the choice of solvents [24-26], which can provide some tips for optimization techniques from the point of view of solvents. The key provisions adopted in these guidelines are based on environmental, health and safety criteria. The result of the assessment is the categorization of the solvent as “recommended”, “recommended or problematic?”, “Problem”, “problematic or dangerous?”, “Dangerous” and “highly dangerous”. Usually the “recommended” solvents are water, alcohols and some esters. Hazardous or very dangerous hydrocarbons, chlorinated solvents and some esters. There are some ways to combine them into one direc-

tory and include “less common” solvents [27]. The advantage of using these solvent selection guides is that it is easy to interpret the result of the evaluation. The main disadvantage is that these results are available for a limited amount of solvents. For example, in the publication Prat D. with co-authors [27] a selection guide of common solvents has been elaborated, based on a survey of publically available solvent selection guides. In order to rank less classical solvents, a set of Safety, Health and Environment criteria is proposed, aligned with the Global Harmonized System (GHS) and European regulations. A methodology based on a simple combination of these criteria gives an overall preliminary ranking of any solvent. This enables in particular a simplified greenness evaluation of bio-derived solvents.

Another solvent-based approach is based on cluster analysis [28], which grouped 151 solvents into three clusters according to their similarity in properties. The first and least green cluster consists of "non-polar and volatile" solvents, the second cluster is intermediate from the point of view of "greenness" - these are non-polar and non-volatile solvents and the third, consisting of more "green" polar solvents. Unlike other solvent selection guides, this is the most flexible manual because it allows the user to select solvents for analysis, evaluation criteria, and their relative importance.

For example, in publications [27, 29-32] discussed the environmental characteristics of the solvents used, and in table1 summarizes the "greenness" of the solvents used in analytical applications.

Table 1 – The scheme showing greenness of solvents commonly used in analytical chemistry [28, 33]



Traditionally used solvents for extraction, including extraction by the Soxhlet apparatus, include ethanol, methanol, acetone, hexane, dichloromethane, tetrahydrofuran, acetonitrile, xylene, toluene, chloroform, toluene, ethyl acetate, diethyl acetate, benzene, and their mixtures in different proportions. The choice of solvent for MAE depends on the solubility of the analyzed analyte, the interaction of the solvent and the plant matrix and the microwave absorbing properties of the solvent. Accordingly, the solvent must have a high selectivity, eliminating the extraction of undesirable components. The key role in microwave extraction is played by the dielectric properties of the solvent, since both the efficiency and selectivity of MAE strongly depend on the dielectric constant of the extracting solvent or its mixture [3-5, 34]. As a rule, when MAE in most cases, solvents or solvent mixtures are used with good heating efficiency under the action of microwaves (with a high  $\tan \delta$  value). For MAE, the use of water is described, for example, for the extraction of phenolic compounds and flavanoids from the leaves of *Pistacia lentiscus* var. [35], alkaloids (corilagin, geraniin) from *G. Sibiricum* Linne [36]], alcohols, esters, fatty acids and ketones from

*Vernonia amygdalina* leaves [37], as well as aqueous organic solvents. In particular, acetone, ethanol and methanol and their aqueous-organic systems are more often used for extraction than the corresponding monocomponent solvent systems. Thus, the use of aqueous methanol is described for the extraction of phenolic acids, anthocyanins, coumarins, alkaloids, aqueous ethanol is used for the extraction of phenolic compounds, flavonoids, anthraquinones, coumarins, saponins, alkaloids, genepoidic and chlorogenic acids, fatty acids, diterpenes, catechins. The efficiency of extraction is also influenced by the percentage ratio of alcohol-water of the extracted solvent mixture [5, 38, 39].

Solvent toxicity is also evaluated when choosing a suitable solvent for MAE. When extracting oleanolic acid from *Gymnema sylvestre*, ethanol is not toxic, so it was chosen in favor of n-butanol, despite the fact that using the latter has a higher yield [40]. In general, ethanol is by far the most used solvent and a good microwave absorber, which is suitable for extracting many active compounds from plants [41].

In the case of using the NEMI pictogram in assessing the environmental friendliness of the MAE, it is necessary to take into account the requirements that none of the chemicals are on the list of persistent, bioaccumulative and toxic chemicals and that none of the chemicals are listed in the applicable procedure in the D, F, P or U hazardous waste lists (Resource Conservation and Recovery Act (RCRAs)) (<https://www.epa.gov/>).

In MAE natural compounds, it is also possible to use acids and bases as various auxiliary chemicals. In addition to water and water-organic mixtures, acidic water or water-alcohol mixtures are used in MAE [42, 43]. So, for the separation of phenolic compounds from *Dactylis glomerata*, *Festuca rubra* L., *Festuca ovina* L., *Bromus inermis* and *Bromus marginatus*, *Hypericum perforatum*, *Thymus vulgaris* L., *Tilia cordata*, *Uncaria tomentosa* used an aqueous

solution of HCl (2 mol/l HCl) [42], for the extraction of anthocyanins from Purple corn (*Zea mays* L.) used a mixture of 15 M HCl:95% ethanol in a ratio of 15:85 [43], and for phenolic compounds from the peel of grapes - a mixture of ethanol – water 50:50 (v/v) with 0.8% (v/v) HCl [44], 140 ml of water at pH 5 (adjusted with citric acid) with Enzyme concentration of 1.82% v/v [45] were used to extract the oil from *Isatis indigotica* seeds using the MAAEE method (Microwave assisted aqueous enzymatic extraction).

Also at MAE, it is possible to use solutions of alkalis and ammonia. In the article [46], described using the hexane:ethanol (1:3) as solvent mixture with 0.05 mol/L of NaOH solvent for extraction of Solanesol from tobacco leaves. The authors of study [47] used 50-60% ethanol with 1-2 % ammonia solution to extract glycyrrhizic acid from licorice root. The results of the assessment of the "greenness" of some acids and bases used in analytical chemistry are presented in table 2.

Table 2 – Greenness of acids and bases used in analytical chemistry.  
Based on the GSK selection guide [33, 48].

<b>Green Zone</b>	
Hydrochloric acid	Carbonates & biocarbonates
Hydrobromic acid	Potassium & sodium acetates
Acetic acid	Sodium hydroxide
Phosphoric acid, diluted	Potassium hydroxide
Sulfuric acid, diluted	
Benzoic acid	Pyridine
Sulfuric acid	
Phosphoric acid	Ammonia
Formic acid	Triethylamine
Perchloric acid	Sodium amide
Nitric acid	Dicyclohexylamine
	Imidazole
<b>Red Zone</b>	

When assessing environmental friendliness using the NEMI pictogram, the third requirement is that the pH of the sample is in the range of 2–12 to avoid severe corrosion during the entire analytical process, therefore this factor of “greenness” of the auxiliary chemicals used should also be considered.

Most of the solvents used are included in the lists of hazardous or toxic wastes (Toxic Release Inventory (TRI) and Resource Conservation and Recovery Act (RCRA) F, K, P, and U lists. (<https://www.epa.gov>) for which good disposal

methods are needed. For most of the solvents included in the analysis, distillation is useful. Spent solvents widely used in analytical chemistry, such as acetonitrile, tetrahydrofuran, acetone, acetic acid, cyclohexane or toluene, must be distilled. Wastes resulting from methanol use, ethanol, pentane, hexane or heptane, should be processed by incineration [28].

One of the MAE options is Solvent-free microwave extraction. In SFME procedures, water or other reagents are not added as extractants. SFME is based on the fact that water naturally contained in plant cells, heats up under the action of microwaves, and thus, direct internal change leads to an increase in pressure inside the plant cells, causes heat to be transferred from the inside of the plant cell, causing the cell to expand and break down walls, which ultimately causes the release of target analytes. Another focus is the use of green solvents, such as water, ILs, DESs, and SUPRASs.

**3. Amount of waste.** From the point of view of analytical chemistry, the amount of waste depends on the amount of reagents used. The amount of solvent used and the characteristics of the plant matrix are critical to the effectiveness of MAE. The volume of 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 may be effective with conventional extraction methods, however, with MAE, a higher ratio may result in lower sample recovery, which may be due to inadequate mixing of the solvent using microwaves.

Such characteristics of the matrix as the size of the sample particles and the state in which it is presented for the MAE can have a tremendous effect on the extraction of compounds. Fine powders can increase the extraction process, providing a large surface area, which provides better contact with the solvent, as well as more allow for improved or significantly used. As a rule, studies provide data on the size of extractable particles of the plant matrix and/or the liquid/solid ratio.

If we consider the data presented in [39] on average, the ratio of liquid/solid is in the range from 10 ml/g to 20-25 ml/g. However, depending on the raw material used and the MAE method, this ratio may vary and in analytical procedures the volume of solvent used may be from 5 to 140 ml [49]. The typical volume used is in the range of 5-50 ml, at which about 50 g of waste is generated, which is consistent with the fourth requirement in assessing the environmental performance of the NEMI profile ([www.nemi.gov/](http://www.nemi.gov/)), which is that less than 50 grams of waste.

**4. Energy consumption.** Energy is a complex parameter that needs to be assessed. It is included in the Rayne and Driver [50] profile and Eco-scale by Aken K.V., Strekowski L., Patiny L. [51]. Energy costs are among the Penalty points in Eco-Scale and are estimated as the energy consumption per sample (as kWh) (table 3).

Table 3 – Penalty points applied for the calculation of final analytical Eco-Scale score [52]

	Sub-Total Panaly Points	Total Panaly Points
Energy	Instruments	
	< 0.1 kWh per sample	0
	< 1.5 kWh per sample	1
	> 1.5 kWh per sample	2

The values of the Panaly Points indicator are calculated based on the data in the table from 0.1 to 1.5 kWh in order of increasing penalties and decreasing “greenness”. Analysis of the literature data showed that most of the presented research works on the MAE extraction did not provide data on energy costs. As an example, one can cite the data on solvent-free microwave extraction (MAF) extraction with MAE [53-55]. This method of extraction is specifically designed to obtain essential oils from plant material [55]. The main advantage of this method is that it includes a shorter extraction time and low energy consumption. In addition, the yield of essential oil and its composition are maintained near the control values obtained by the classical method of hydrodistillation (HD). The temperature of the material does not exceed 100 °C, and the forced evaporation of water has two main effects: the dehydrating effect due to evaporation and the change in the strength of cell walls, which contributes to their destruction [53]. Water as a solvent is particularly interesting in the extraction of tea polyphenols, since it is directly related to the procedure for consuming tea infusions [56]. Comparison of indicators with conventional water extraction and MAE for the extraction of polyphenols, and it was shown that the extraction time was reduced from 45 to 15 minutes, and the energy consumption, in the case of MAE, was 3 times smaller.

In a paper [57] describes the use of the organic acid extracting system (PMAE). Extraction of active ingredients from Rosemary (*Rosmarinus officinalis* L.) using the PMAE method (30 g of raw materials, extraction time 15 minutes, power - 100 W, at atmospheric pressure) and hydrodistillation (500 g of 3 h in a conventional apparatus (until no more essential was obtained) showed that these methods were respectively 2 kW h for HD, and 0.1 kW h for PMAE.

Some examples found in the literature show an estimated energy consumption of about 0.75-2.5 kWh for conventional extraction, 0.75-2 kWh for hydrodistillation (HD), and 0.04±0.25 kWh for SFME, water extraction (microwave-assisted water extraction (MWE) and microwave-assisted aqueous extraction (MAAE)) [5,53,56,57].

Energy is associated with time. Extraction time is another factor whose influence must be taken into account. The long process of extraction requires additional energy costs. Optimization of the extraction time is an important factor in the efficiency of the process, since the extraction time can vary depending on



the part of the plant used and the type of microwave extraction. The dielectric properties of the solvent also affect the exposure time. Solvents such as ethanol, methanol and water can be very hot with longer exposure, thereby increasing the risk of destruction of the thermolabile components.

Thus, it was shown in [39] that the extraction time varies from 15 sec to 60 min, but on average, 10-20 min is sufficient for extraction. For example, it took 60 min to extract the active ingredients from *Pistacia lentiscus* var. [35], silymarin from *Silybum marianum* L. [58] or phenolic compounds from *Quercus robur* L. [59], and in a number of studies, good results were obtained in 15-90 sec [60-67], for example, 30-40 sec it was sufficient to extract phenolic compounds from peanut peel [61], genepozidnoe and chlorogenic acids [64], oleic, linoleic and palmitic acids and steroids from *Rodgers aesculifolia* Batal [65], 49-60 sec to extract phenolic acids from mandarin peel [60], pinostorobin from leaves of *Pigeonpea* (*Cajanus cajan* (L.) Millsp.) [62], polyphenols from *Pistacia lentiscus* L. [66], flavonoids from *Terminalia chebula* Retz [67], 90 sec to extract alkaloids from *Stephania sinica* [63].

As a rule, the time used for MAE is much lower than in the traditionally used methods of extraction. Microwave heating has a completely different nature than convection and its important advantages include: heating occurs throughout the volume of the material, and the thermal conductivity of the material does not matter; the change in temperature of the material being heated occurs without inertia in accordance with the change in input power; the possibility of dynamic temperature control during the experiment; the ability to quickly raise the temperature of the material to the specified values; the absence of a heat transfer agent eliminates the possibility of sticking to the material.

An analysis of the SFME method, presented in [53], showed that the SFME requires only a few minutes of heating and less than 30 min. extracts from the same material. The SFME needs less than 0.5 kWh.

Most of the data on the comparative analysis of traditional methods of extraction and MAE confirms that the MAE method consumes significantly less energy than traditional approaches.

**5. Environmental impact.** Another factor affecting the environmental friendliness of microwave extraction is its environmental impact. As an indicator, a number of studies provide data on the amount of CO<sub>2</sub>.

The amount of CO<sub>2</sub> released into the atmosphere can be calculated on the assumption that 800 g of CO<sub>2</sub> is released into the atmosphere to produce 1 kWh as a result of burning fossil coal or fuel [56].

There are several publications that evaluate this factor [53, 56, 57]. Regarding environmental impact, in [53] it was shown that it was (3600 g CO<sub>2</sub>/g of essential oil) than for SFME (200 g CO<sub>2</sub>/g of essential oil). The article [57] reports that the amount of carbon dioxide emitted into the atmosphere during rosemary extraction is significantly higher with conventional distillation (1600 g) than with the use of portable microwave assisted extraction apparatus (PMAE) (80 g).

Thus, from the available data it can be concluded that CO<sub>2</sub> emissions for hydrodistillation are on average 1600-3600 g [53, 57], 600 g CO<sub>2</sub> for conventional water production [56], 200 g CO<sub>2</sub> for the MWE method [56] and 80 g CO<sub>2</sub> using PMAE [57] was released into the atmosphere.

**6. Safety.** The initial use of household appliances or their modified systems for microwave extraction was associated with a number of factors affecting safety. So, when using a “closed” system, the pairs formed during acidic microwave processing are contained inside the vessel, and high pressure inside the vessel reduces the safety of the process and creates the risk of an explosion, the procedure eliminates the addition of reagents or solvents during operation, and the working vessel must be cooled to its opening to avoid loss of volatile components. In addition, when using microwave energy, there are requirements for the materials used that can withstand high temperatures. An “open” system operating at atmospheric pressure, in contrast to a “closed” system, reduces some of the security problems, since it operates under milder conditions and is considered more suitable for extracting thermolabile compounds. This system has a higher sample throughput, and more solvent can be added to the system at any time. However, in both cases control over temperature and pressure was excluded, the use of mixing and regulation of the power used was limited, etc. All the shortcomings of using household appliances for research purposes led to the creation of more convenient and safer microwave installations. The rapid development of microwave chemistry prompted the company to develop improved microwave systems and related tools for the process of extraction, synthesis, sample preparation, drying, etc.

For more than 20 years of experience in the field of microwave chemistry instrumentation and the development of the latest industrial technology, the modern instrumentation of the MAE method provides a number of undoubted advantages over the use of conventional or modified household microwave systems.

Compared with household microwave ovens, laboratory options provide the ability to control the temperature, pressure of the reaction mixture and the power of the radiation used, they create a uniform and stable field and have mixing systems. Some systems can in real time; directly display the workflow, be equipped with a sensor for the presence of solvent vapors in the working chamber and other functions. This provides the possibility of uniform heating of the reaction mixtures. Rapid heating and rapid cooling system at the end of the process allows you to control the heating time, which results in reproducible results. Much attention is paid to the safety of equipment. In addition to special extraction vessels for extraction, the devices have a safe external control from a personal computer; easy to use program window interface [68-73].

**Conclusion.** With regard to safety issues, the use of microwave energy can pose a risk to inexperienced analysts, especially when working with toxic organic solvents and aggressive concentrated acids. As a rule, small weights of raw materials and volumes of solvent or their mixtures, short-term use of microwave

energy are used in MAE analytical procedures. In the case of a carefully planned experiment performed according to the technical information provided on the instruments, the extraction protocols used, the amount of reagents and the practical implementation of the experience under the supervision of an experienced specialist, the safety risks are negligible.

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**Резюме**

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**«ЗЕЛЕНЬЕ» НАПРАВЛЕНИЯ МИКРОВОЛНОВОЙ ЭКСТРАКЦИИ  
В ХИМИИ ПРИРОДНЫХ СОЕДИНЕНИЙ**

Сообщение 2: Факторы экологичности микроволновой экстракции

Рассмотрены факторы экологичности микроволновой экстракции, которые связаны с опасными реагентами, количеством отходов, безопасностью, потребляемой энергией и воздействием на окружающую среду.

**Ключевые слова:** природные соединения, микроволновая экстракция, факторы экологичности.

**Резюме**

*Т. В. Харламова*

**ТАБИҒИ ҚОСЫЛЫСТАР ХИМИЯСЫНДАҒЫ МИКРОТОЛҚЫНДЫ  
ЭКСТРАКЦИЯЛАУДЫҢ «ЖАСЫЛ» БАҒЫТТОРЫ**

2-ші хабарлама: Микротолқынды экстракцияның экологиялық факторлері

Микротолқынды экстракцияның экологиялық факторлері қауытты реагенттерге, қалдықтар мөлшеріне, қауіпсіздігіне, пайдаланған энергияға және қоршаған ортаға әсер ету байланысылығы қарастырылғын.

**Түйін сөздер:** табиғи қосылыстар, микротолқынды экстракция, экологиялық факторлар.